POST-HARVEST PRESERVATION AND MODELLING OF DRYING ON AN INERT SPHERE FOR SEA BUCKTHORN BERRIES (*HIPPOPHAE RHAMNOIDES* L. SSP. *SINENSIS*)

BY

SUSAN ST. GEORGE

A Thesis

submitted to the Faculty of Graduate Studies

in partial fulfillment of the requirements

of the Degree of

DOCTOR OF PHILOSOPHY

Department of Biosystems Engineering

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Post-Harvest Preservation and Modelling of Drying on an Inert Sphere for Sea Buckthorn Berries *(Hippophae Rhamnoides L. SSP. Sinensis*)

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ABSTRACT

The production of uniform, safe, and effective plant-based functional foods and nutraceuticals is dependent on the optimization or preservation of biologically active (bioactive) compounds throughout the value-added cycle. The scope of this research was to address the quality preservation of sea buckthorn berries (*Hippophae rhamnoides* L. ssp. *sinensis*) in the development of sea buckthorn oils. This was achieved in three phases: I) evaluation of the influence of time of harvest on physical characteristics (moisture content, size, and colour) and bioactive quality (carotenoids, fatty acids, tocols, and sterols) on whole berries; II) evaluation of the influence of forced air drying conditions (50°C at 30.6 and 58.7% RH; 60°C at 24.4 and 57% RH, and 70°C at 20.8 and 57% RH) on colour and bioactive quality of the pulp and peel fraction, and III) development, solution, and validation of a semi-empirical temperature prediction model (based on simple heat and mass transfer theory) for the simulation of thin layer drying on an inert sphere.

Within phase I, berries collected at maturity yielded highest values for berry size, CIELab factor a^*, and total carotenoid content in the fruit fraction. Early maturity berries yielded higher levels of α-tocopherol and β-sitosterol in the fruit fraction. Post-maturity resulted in the lowest quality fruit fraction oil. Seed characteristics and bioactive compounds did not vary significantly with respect to harvest time. In phase II, total carotenoid and phytosterol concentrations remained relatively stable over the range of drying conditions. Fatty acid composition exhibited only minimal changes to palmitoleic acid at 70°C. The lowest colour degradation, occurred at drying conditions of 60°C at

24.4% RH, whereas highest concentrations of major tocols occurred at 50°C. Significant darkening occurred at the higher relative humidity level for each temperature. Although the darkening did not represent a loss in carotenoids, it did coincide with a retention or regeneration of tocols. Within phase III, predicted temperatures were within ~10% of experimental, with a higher accuracy at lower temperatures (i.e. 55 and 65°C) and later stages of drying. Overall, the model showed potential for the prediction of temperature for a material dried in a thin layer on an inert sphere.

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a	Red or green colour factor in the Hunter *Lab* scale
a^*_o	Red or green colour factor in the CIELab scale for fresh control
a^*	Red or green colour factor in the CIELab scale
a_w	Water activity
b	Yellow or blue colour factor in the Hunter *Lab* scale
b^*_o	Yellow or blue colour factor in the CIELab scale for fresh control
b^*	Yellow or blue colour factor in the CIELab scale
d	Diameter of a body, m
d_c	Characteristic dimension, m
d_{ip}	Diameter of the inert particle, m
dm_{H_2O}	Difference in mass of water, kg
dM	Difference in moisture content, dry basis (db), kg/kg
dq_{cond}	Conductive heat transfer, J
dq_{conv}	Convective heat transfer, J
dt	Difference in temperature, °C
dt_{ip}	Difference in temperature of inert particle, °C
$d\tau$	Difference in time, s (Eqns. 2.2, 2.4-2.6, 2.9, 2.34-2.35), h (Eqn. 2.32)
dx	Difference in location in rectangular coordinates, m
h	Heat transfer coeffient, W/(m^2·K)
h_{fg}	Latent heat of evaporation, J/kg
m	Mass of a body, kg
m_f	Mass of dried sample, kg

m_i	Initial mass of sample, kg
m_{ip}	Mass of inert particle or sphere, kg
$m_{pc(i)}$	Mass of press cake at time i-1, kg
$m_{pc(i-1)}$	Mass of press cake, kg
$m_{pc(f)}$	Mass of dried press cake, kg
$m_{pc(o)}$	Initial mass of press cake, kg
$m_{pc\text{-}fat}$	Mass of press cake fat portion, kg
$m_{pc-H_2O(o)}$	Initial mass of press cake water portion, kg
$m_{pc-H_2O(i)}$	Mass of press cake water portion at time i, kg
$m_{pc\text{-}sol}$	Mass of press cake solids portion, kg
$m_{pc\text{-}tot(i)}$	Total mass of press cake components at time i, kg
m_{sys}	Mass of inert sphere, press cake, hook, and sensor system, kg
q	Heat energy, J
q_{cond}	Conductive heat, J
q_{conv}	Convective heat, J
q_{in}	Heat entering a system, J
$q_{sensible}$	Heat being expended by a system in the form of sensible heat, J
q_{latent}	Heat being expended by a system in the form of latent heat, J
r	Distance from centre of body, m
s	Material thickness, m
t	Temperature of a body, °C
t_{air}	Temperature of the drying air, °C
t_i	Temperature of a body at beginning of the drying period, °C

t_{ip}	Temperature of the inert particle or sphere, °C
$t_{ip(i)}$	Temperature of the inert sphere at time *i*, °C
$t_{ip(i-1)}$	Temperature of the inert sphere at time *i*-1, °C
t_{ip-c}	Temperature at the geometric centre of the inert sphere, °C
t_{ip-s}	Temperature on the inside surface of the inert sphere, °C
t_o	Initial temperature of a body, °C
t_{pc}	Temperature of the press cake, °C
$t_{pc-exp(i)}$	Experimental temperature of the press cake, °C
$t_{pc(i)}$	Temperature of the press cake at time i, °C
$t_{pc(i-1)}$	Temperature of the press cake at time i-1, °C
$t_{pc(o)}$	Initial temperature of the press cake, °C
$t_{pc-pred(i)}$	Predicted temperature of the press cake, °C
t_s	Temperature of a body surface, °C
t_{w-b}	Wet bulb temperature of the air, °C
u	Velocity of the drying air, m/s
x	Distance in direction from centre, m

A	Surface area of a body, m^2
A_{ip}	Surface area of an inert particle or sphere, m^2
Cp	Specific heat capacity of a body, J/(kg·K)
Cp_{air}	Specific heat capacity of the drying air, J/(kg·K)
Cp_{ip}	Specific heat capacity of the inert particle or sphere, J/(kg·K)
$Cp_{pc(i)}$	Specific heat capacity of the press cake at time i, J/(kg·K)
$Cp_{pc(o)}$	Initial specific heat capacity of the press cake, J/(kg·K)
D_m	Mass diffusion coefficient, m^2/s
DR	Drying rate, 1/s
L	Lightness factor in Hunter LAB scale
L^*	Lightness factor of sample on the CIELab colour scale
L^*_o	Lightness factor of control sample on the CIELab colour scale
M	Moisture content, db or % wet basis (wb) as noted, kg/kg
$M_{(i)}$	Moisture content at time i (db), kg/kg
M_{cr}	Critical moisture content (db), kg/kg
M_e	Equilibrium moisture content (db), kg/kg
M_o	Initial moisture content (db), kg/kg
MR	Moisture ratio (db), kg/kg
$MR_{(i)}$	Moisture ratio at time i (db), kg/kg
N	Number of observations
R	Radius of a body, m
RH	Relative humidity in decimal form (dec.) or % as noted
V	Volume of a body, m^3

V_{ip}	Volume of an inert particle or sphere, m^3
$X^m_{fat(i)}$	Mass fraction of fat component in press cake, kg/kg
$X^m_{H_2O(i)}$	Mass fraction of water component in press cake, kg/kg
$X^m_{sol(i)}$	Mass fraction of solids component in press cake, kg/kg

GREEK SYMBOLS

α	Thermal diffusivity of a body, m^2/s
Δa	Change in red or green colour in Hunter *Lab* scale
Δb	Change in yellow or blue colour in Hunter *Lab* scale
ΔE	Colour change
$\Delta m_{H_2O(i)}$	Moisture loss at time *i* for time interval $\Delta\tau$, kg
Δt	Temperature difference, °C
$\Delta\tau$	Time interval, s
∇	Symbol of gradient
∇^2	Laplacian operator
∂	Symbol of partial derivative
λ	Thermal conductivity of a body, W/(m·K)
λ_{air}	Thermal conductivity of the drying air, W/(m·K)
λ_n	Eigenvalue roots
ρ	Density of a body, kg/m^3
ρ_{air}	Density of the drying air, kg/m^3
ρ_{ip}	Density of an inert particle or sphere, kg/m^3

Σ	Summation
τ	Elapsed time of drying, s (Eqns. 2.10-2.19), min (Eqn. 2.33), h (Eqns. 2.20-2.31)
τ_i	Time at i in time interval $\Delta\tau$, s (Eqn. 5.2)
τ_{i-1}	Time at i-1 in time interval $\Delta\tau$, s (Eqn. 5.2)
τ_o	Initial time, h
σ	Characteristic time, min
μ	Viscosity of the drying air, (kg/m·s)

COEFFICIENTS

a, b, c, f, g, k, k_o, k_1, n	Drying coefficients or constants
B, C, E	Coefficients for M_e model
m	Dimensionless factor
z	Dimensionless shape factor

STATISTICAL PARAMETER SYMBOLS

CV	Coefficient of variation
p	Probability
r^2	Coefficient of determination
$RMSE$	Root mean squared error
SD	Standard deviation

DIMENSIONLESS NUMBERS

Bi	Biot number
Fo	Fourier number
Pr	Prandtl number
Nu	Nusselt number
Re	Reynolds number

1 INTRODUCTION

In recent years industrialized countries throughout the world have taken steps to change from their reactive approach toward healthcare to a more proactive one (WHO 2004). The need for change arose from increasing health care costs and higher life expectancies. Through significant discoveries by nutrition and health experts, a definite link was made among health, diet and lifestyle (Oomah and Mazza 2000). Incorporating "healthful" compounds into a person's diet along with proper lifestyle choices including exercise, is expected to result in an overall better quality of life with reduced incidence of diet related disease and illness. Although much is known about the benefits of certain compounds (e.g. vitamin C and D and calcium) there are many (e.g. carotenoids, vitamin E tocols, sterols, and fatty acids) for which research into health benefits is still being conducted. Along with this ongoing research is the investigation into plant products that have an abundance of beneficial compounds. This has led to the identification of plants that may have long been respected for their health benefits in ancient and traditional medicines.

The need to provide their community with the opportunity to benefit from health beneficial plants has led governments and experts in the field to introduce them to locations in which they may not be indigenous. To understand the effects of local environment, growing conditions, and crop management on the biologically active (bioactive) quality of these plants, research is required. Sea buckthorn (*Hippophae rhamnoides* L.) is an excellent example of a plant originating from northern locations of Asia that has been introduced throughout the world in similar climatic regions such as in

Europe and Canada (Schroeder and Yao 1995). While research into its bioactive quality has been conducted for many years in Asia and Europe (Kallio et al. 2002a; 2002b; Tang and Tigerstedt 2001; Yang and Kallio 2002a; 2002b; 2001; Yang et al. 2001), information for Canada is limited (Li 2002) especially for the Canadian Prairies.

The extreme cold encountered in the Canadian Prairies allows for winter harvest, whereas this is not commonly practised in warmer climates of Canada. Winter harvest includes the collection of frozen berries at temperatures below -20°C, typically between December and February. Since berries generally begin to ripen in the late summer to early fall (Beveridge 2003a) winter harvest may have an impact on the bioactive quality of the fruit. Providing the Canadian sea buckthorn industry with information regarding optimum time of harvest for quality preservation will enhance the ability to develop a better harvesting protocol, for long term profitability. In addition to harvest issues, researchers (Gutiérrez et al. 2008) are also investigating the post harvest storage and processing of this plant product.

While there has been some research on the drying of whole crushed sea buckthorn berries (Gutiérrez et al. 2008) and leaves (Guan et al. 2005), limited information has been reported on sea buckthorn berry press cake, a by-product of the juice industry. Sea buckthorn berry juice is deemed to be a profitable product for the Canadian market (Beveridge et al. 1999). Utilizing the press cake, a product rich in valuable compounds, can contribute to the development of new functional foods and nutraceuticals as well as reduce the quantity of waste from the juice processing industry. Drying can be used, both to, preserve the highly perishable press cake as well as assist in the separation of seeds

from the pulp and peel portion (Arimboor et al. 2006; Cenkowski et al. 2006). Limited research is available on the effect of forced air drying on bioactive compounds in plant products in general (Rahman 2005; Sablani 2006). Understanding the link between drying conditions and quality characteristics is beneficial in the selection of dryer type and operating conditions (Rahman 2005).

Present trends in drying technology are associated with higher energy efficiency, enhanced drying rates, system compactness, and control for enhanced quality and optimal capacity (Grbavcic et al. 2004). With these trends in mind, fluidized or spouted bed drying with inert particles has shown potential for the production of powders or flakes from liquids, suspensions, slurries, pulps, and pastes (Grbavcic et al. 2004; Orsat and Raghavan 2007). These systems may show potential for sea buckthorn berry press cake. In addition to dryer type and operating conditions, the duration of drying is also an important aspect in final product quality (Kudra and Efremov 2003). Extended drying time can lead to needless quality degradation. Because product temperature is an indicator of drying stage (Konovalov et al. 2003), a temperature prediction model could provide valuable information regarding drying behaviour of the product and control of the operating conditions for optimum quality.

Significant research has been conducted in the area of thin layer drying for olive press cake, another product heterogeneous in both size and composition of particles (Akgun and Doymaz 2005; Celma et al. 2007; Doymaz et al. 2004) and drying of spherical particles such as grains (Pabis et al. 1998). However, limited information is available on the drying of a thin layer on an inert spherical particle. Proposed models range from

theoretical to simplified. Theoretical models are based on a concentric spherical arrangement and unsteady state heat and mass transfer theory (Konovalov et al. 2003; Mikhailov and Özişik 1984). As an alternative to a theoretical analysis, Leontieva et al. (2002) proposed using a simple heat and mass balance model in a micro scale (i.e. individual inert particle), based on conservation laws. This same approach has been employed in a macro scale (i.e. dryer section or drying system) for slurries dried in a fluidized bed (Grbavcic et al. 2004) and suspensions dried in a spout-fluid bed (Costa et al. 2001).

Sea buckthorn berry components are among many plant products that are considered valuable ingredients for functional foods and nutraceuticals. In Canada, currently both functional foods and nutraceuticals are legislated under the Food and Drug Act (Health Canada 1998). Quality control is an important aspect in the production of functional foods and nutraceuticals because products must meet a set of criteria to be marketed as such. Product uniformity, efficacy, and safety are key factors that can be respected through the implementation of guidelines and protocols that are established through research. Investigation of the various stages of the value added cycle (e.g. cultivation through to final packaging and storage) is important so that quality is ensured at each and every step.

The scope of this research program is to focus on the quality preservation of sea buckthorn berries (*H. rhamnoides* L. ssp. *sinensis*) from harvest through to drying. The following are the objectives of each of the independent phases (I, II, and III) of this research:

I. Evaluate the influence of time of harvest on physical characteristics (i.e. moisture content, size, and colour) and levels of oil-based bioactive compounds (i.e. carotenoids, fatty acids, tocols, and sterols) in fresh sea buckthorn berries.

II. Evaluate the influence of forced air drying conditions (i.e. temperature and relative humidity) on physical characteristics (i.e. drying time, colour) and levels of oil-based bioactive compounds (same as phase I) in dried sea buckthorn berry pulp and peel.

III. Develop, solve, and validate a semi-empirical temperature prediction model for the simulation of thin layer drying on an inert sphere using sea buckthorn press cake as a test material.

Within the scope of phase I (Chapter 3), three harvest periods representing initial onset of maturity (early maturity), complete ripeness (maturity), and over-ripeness (post-maturity) were compared. The two investigated fractions, seed and fruit (i.e. juice, pulp, and peel) were discussed individually. Phase II (Chapter 4) allowed for the comparison between fresh and dried pulp and peel as well as between samples dried at six chamber temperature and relative humidity (RH) combinations: 50°C at 30.6 and 58.7% RH, 60°C at 24.4 and 57.0% RH, and 70°C at 20.8 and 57.0% RH. Phase III (Chapter 5) provided an approach to drying modelling that included a time incremental heat and mass balance over a single element which is composed of a concentric sphere (i.e. thin layer on a solid sphere). This approach required the selection of semi-empirical and empirical models for the prediction of material and drying condition characteristics (e.g. moisture ratio, equilibrium moisture content, specific heat, and heat transfer coefficient).

The thesis was written in a publication-manuscript format. The INTRODUCTION (Chapter 1), LITERATURE REVIEW (Chapter 2), GENERAL CONCLUSIONS (Chapter 6), RECOMMENDATIONS FOR FUTURE WORK (Chapter 7), and REFERENCES (Chapter 8) comprise the manuscript portion common to the entire scope of research. The Methods and Results and Discussion sections of research phases I, II, and III are detailed within Chapters 3, 4, and 5, respectively. Chapter 3 entitled, INFLUENCE OF HARVEST TIME ON QUALITY OF OIL-BASED COMPOUNDS IN SEA BUCKTHORN (*H. rhamnoides* L. ssp. *sinensis*) SEED AND FRUIT is a formatted excerpt from a published journal article (St. George and Cenkowski 2007); Chapter 4 entitled, THE INFLUENCE OF DRYING ON LEVELS OF BIOACTIVE COMPOUNDS IN PULP AND PEEL OIL OF SEA BUCKTHORN (*H. rhamnoides* L. ssp. *sinensis*) BERRIES is a formatted excerpt from a recently accepted submission to the Journal of Food Processing and Preservation (St. George and Cenkowski 2008), and Chapter 5, entitled MODEL FOR THIN LAYER DRYING ON AN INERT SPHERE will be submitted for publication in 2008.

2 LITERATURE REVIEW

2.1 Nutraceuticals and functional foods

Traditionally, the role of diet has been to provide energy and essential nutrients to sustain life and growth. However, with aging populations, longer life expectancies, and increasing health-care costs, the developed world is addressing the role of diet and lifestyle in the prevention and management of chronic and degenerative diseases (Oomah and Mazza 2000). Preventable diseases such as certain cancers, heart disease, stroke, diabetes, diseases of the arteries, and osteoporosis have important dietary links (WHO 2004). Incorporation of foods and food products rich in healthful bioactive compounds into a healthy lifestyle, including exercise and a nutritious diet, is part of a strategy to boost health and reduce the risk of disease (Oomah and Mazza 2000).

Several terms are used to describe the many natural products currently being developed for health benefit (e.g. functional foods, nutraceuticals, pharmafoods, designer foods, vitafoods, phytochemicals, phytofoods, medical foods, and foodaceuticals) (Oomah and Mazza 2000; Small and Catling 1999). Although the terms, nutraceutical and functional food, are the most commonly used worldwide, there is no definite consensus on their meaning. To maintain consistency throughout the remainder of this discussion, the definitions recognized by Health Canada (1998) are employed.

2.1.1 Nutraceuticals

A plant-based nutraceutical is a product isolated or purified from plant material and is generally sold in medicinal forms (e.g. capsules, tablets, powders, and potions), not usually associated with foods (Health Canada 1998). To be considered a nutraceutical, a product must also be demonstrated to offer a physiological benefit or assist in the management or prevention against chronic or degenerative diseases or both. Within Canada, nutraceuticals are included in the category of natural health products (NHPs). Other NHPs include: traditional herbal medicines; Chinese, Ayurvedic, and Native North American medicines; homeopathic preparations, and vitamin and mineral supplements.

2.1.2 Functional foods

A functional food appears similar to, or may be, a conventional food that is consumed as part of a usual diet and is demonstrated to have a physiological benefit or assist in the management or prevention against chronic or degenerative disease (Health Canada 1998). A food can be made functional through: 1) the elimination of a compound having a negative physiological effect; 2) increasing the concentration of beneficial compounds; 3) the addition of a new compound observed to offer benefits, and 4) partial replacement of a negative compound by a beneficial one, without adversely affecting the nutritional value of the food (Gibson and Fuller 1998).

2.1.3 Phytochemicals

Plant chemicals (phytochemicals) are the bioactive compounds which contribute to the activity of plant-based nutraceuticals and functional foods (Oomah and Mazza 2000). Plants synthesize both primary as well as secondary metabolites (Webb 2006; Wildman 2001a). Primary metabolites include proteins, amino acids, chlorophyll, membrane lipids, nucleotides, and carbohydrates necessary for the existence of plants. Secondary metabolites have not been linked to plant processes (e.g. photosynthesis, respiration, etc.), and were originally regarded as nonfunctional waste products. However, researchers now recognize that secondary metabolites, which fulfill important functions, may be associated with only certain plant species or taxonomically related groups. Functions include protecting the plant from herbivores, insects, fungi, bacteria, microbial infection, ultraviolet light, also, in the case of colourful pigments and scents, attracting insects and birds for the purpose of pollination and seed dispersal. Secondary metabolites can be divided into three main groups: isoprenoid derivatives, phenolics, and sulfur and nitrogen containing compounds.

Combining primary and secondary plant metabolites enables a simple classification of bioactive compounds based on chemical nature: isoprenoid derivatives; phenolic substances; fatty acids and structural lipids; carbohydrates and derivatives; amino acid-based substances, and elements (Section 2.2.3) (Wildman 2001b).

2.2 Sea buckthorn berries

2.2.1 The shrub

Sea buckthorn (*H. rhamnoides* L.), is a hardy shrub, tolerant of temperatures between -43 and 40°C and drought conditions, yet requires irrigation in regions receiving less than 400 mm of rainfall per year (Li 2003b; Li and Wang 1998). The shrub, 2 - 4 m high, grows best in well-drained soil (e.g. sandy loam) with a pH of 6 to 7, however, can tolerate many soil conditions and pH levels except for extremes. The shrub is known for its extensive root system that develops quickly and is nitrogen fixing. Ripe sea buckthorn berries can be yellow, orange, or red, are spherical to elliptical in shape, and range in size between 3 to 8 mm in diameter (Li 2003b).

During the last 23 years, sea buckthorn has attracted attention as a potential diversification plant by researchers from Asia, Europe, and more recently North America (Schroeder and Yao 1995). The species *H. rhamnoides* is distributed throughout Eurasia (e.g. China, Mongolia, Russia, Kazakastan, Turkey, Romania, Switzerland, France, Britain, Finland, Norway, and Sweden). It grows on hills, valleys, river beds, sea coasts, islands, in isolated or large continuous pure stands or in mixed stands with other shrub or tree species.

2.2.2 Historical uses

The properties of the shrub's bark, leaves, and berries have been known and exploited in Eurasia for centuries. Medicinal value of these components was recorded in the

Tibetan medical classic "'rGyud Bzi" in the eighth century (Li and Guo 1989). In ancient Greece, rapid weight gain and a shiny coat were witnessed in horses fed fodder containing leaves and young branches. This is believed to have led to the Latin name 'Hippo'- horse and 'phaos' – to shine (Rongsen 1992).

Asia

The sea buckthorn industry has been thriving in Russia since the 1940's, when scientists began investigating the shrub's bioactive compounds (Schroeder and Yao 1995). The first Russian sea buckthorn factory developed products utilized in the diet of Russian cosmonauts and as a cream for protection from cosmic radiation. There are currently ~6000 hectares of sea buckthorn plantations in Russia (NRCC 2002).

The sea buckthorn industry in China is more recent, although traditional uses date back many centuries (Schroeder and Yao 1995). Research and plantation establishment were initiated in the 1980's with over 300 000 ha planted by 1995. The shrub has transformed cold deserts in China into a region of sustainable profit. Its acclaim as the "green hope" is because of its ability to reduce soil erosion, provide fuel and fodder, and to produce a crop of berries having significant medicinal and cosmetic value (Rongsen 1992). With the establishment of 150 processing factories, over 200 different products have been developed. The people in the mountainous regions of India are following China's lead (Arimboor et al. 2006).

Europe

The sea buckthorn industry in Germany has a long tradition and is based on a total area of ~300 ha (NRCC 2002). German sea buckthorn products are mainly produced for the valuable supplementary health food market. Significant research has been performed in Finland and Sweden focussing on bioactive compounds, health benefits, and effects of harvest date on quality (Kallio et al. 2002a). Many other European countries are growing sea buckthorn, however, little is reported with regards to these markets.

North America

Sea buckthorn has been used as a landscape and prairie shelterbelt plant for at least thirty years in Canada (Li 1999). It is viewed as a crop that will grow on marginal land and provide excellent soil erosion protection, farmstead protection, an opportunity for land reclamation, shelter for wildlife, and offer commercial potential. In 2003, ~ 182 hectares were reported to be planted in sea buckthorn plantations across Canada, with plans for future expansion (Neish 2003). The variety, subspecies (ssp.) *rhamnoides* cultivar (cv.) *Indian summer* was developed as a hardy and valuable shrub suitable for Canadian production (Li 1999). Originating from China, ssp. *sinensis* has also been tested, however, has not gained wide acceptance as cv. *Indian summer* for a variety of reasons (e.g. different bioactive content, size, and ease of harvest). Lobatcheva et al. (2002) determined that several Russian cultivars of *H. rhamnoides* L. *Elaeagnaceae* (e.g. cv. *Dar Katugne*, *Tchyskaja*, *Orangevaja*, *Maslitchnaja*, and *Tchuiskaja*) are suitable for establishment in Washington State. The following sections include general information

on *H. rhamnoides* L. of different subspecies and cultivars, however, the discussions in Chapters 3 to 5 are restricted to ssp. *sinensis*, unless otherwise noted.

2.2.3 Bioactive compounds

According to Schroeder and Yao (1995), "sea buckthorn berries are among the most nutritious and vitamin-rich fruits found in the plant kingdom". The berries consist of pulp (68%), seed (23%), and peel (8%) (Yang and Kallio 2001; Zadernowski et al. 1997). Sea buckthorn synthesizes and accumulates fat in all morphological parts of the berry, resulting in three types of oils (e.g. seed, pulp, and peel) (Oomah 2003). Generally, the oils from the pulp and peel fractions are combined due to the difficulty involved with separation. Yang and Kallio (2001) reported oil contents of seeds (7.3%), freeze dried pulp and peel (1.7%), and freeze dried whole berry (2.1%) in wild ssp. *sinensis* berries. Compounds within all parts of the berry are discussed within their classification, with special emphasis on oil-based compounds investigated in this research [e.g. carotenoids, fatty acids, tocols (i.e. tocopherols and tocotrienols), and phytosterols].

Isoprenoids

The isoprenoid derivative class of 25 000 different substances, one of the largest categories of secondary metabolites, includes carotenoids, saponins, sterols, and simple terpenes (Wildman 2001b). The carotenoids including carotenes (e.g. γ-,β-,α-,ε-,ζ-carotene, lycopene, etc.) and xanthophylls (e.g. lutein, etc.) are pigments that produce colours of yellow, orange, and red and play a significant role in photosynthesis and photo-protection (Mínguez-Mosquera et al. 2002; Wildman 2001b). All carotenoids must

be consumed as part of a diet either in food or as supplements (Shi et al. 2002). In most plant or animal tissues, only a small number of carotenoids account for more than 80% of the total carotenoid content (Parker 2000). Beta-carotene is the most highly consumed carotenoid due to its prevalence in plant foods (Mínguez-Mosquera et al. 2002).

Carotenoid content is the main parameter by which sea buckthorn oil is traded commercially (Oomah 2003). Carotenoids vary widely depending on the source of the oil; pulp and peel oils are a good source as reflected by their rich colours and seed oil usually contains much lower levels (Beveridge et al. 1999; Xin et al. 1995). As many as 18 carotenoids have been identified in sea buckthorn fruit. The carotenoids possessing provitamin A activity (e.g. β-carotene, γ-carotene, β-zeacarotene, cryptoxanthin and sintexanthin) and lutein account for 48 and 14% of total carotenoids, respectively (Kudritskaya et al. 1989). Mironov (1989) indicated that sea buckthorn berry carotenoids consist of 20% β-carotene, 30% γ-carotene, 30% lycopene, and 15% oxygen-containing carotenoids.

Phytosterols are present in all plants and plant-based foods (Piironen and Lampi 2004). Cereals, vegetables, vegetable oils, and fruits especially berries, are rich in phytosterols. The majority of phytosterols found in foods are β-sitosterol, campesterol, stigmasterol, avenasterols, and stanols. Beta-sitosterol is the main dietary phytosterol (56-79%) of the total dietary phytosterol intake.

The phytosterol content of sea buckthorn berry oil, ranges from 2.2 to 8.8%, with peel, pulp, and seeds contributing 50, 20, and 30%, respectively (Mironov et al. 1989).

Up to 25 phytosterols and triterpenes have been identified in the pulp and peel oil, with β-sitosterol, campesterol, stigmastanol, and the combined isofucosterol (i.e. δ-5 avenasterol) and obtusifoliol contributing 70.6, 1.5, 7.4, and 4.9% of total phytosterols, respectively (Yang et al. 2001). Up to 19 phytosterols and triterpenes have been identified in the seed oil, with β-sitosterol, campesterol, stigmastanol, and the combined isofucosterol (i.e. δ-5 avenasterol) and obtusifoliol contributing 64.8, 2.3, 3.2, and 16.9% of total phytosterols, respectively (Yang et al. 2001). Li et al. (2007) determined that combined lanosterol and β-sitosterol account for 48% of total sterols identified in seed (cv. *Indian summer*) oil extracted using super critical fluid extraction (SCFE).

Phenolics

Phenolics, a class of more than 8000 secondary metabolites includes: cinnamic and benzoic acid derivatives and simple phenols; coumarins; flavonoids and stilbenes; lignans and lignins; suberins and cutins; tannins, and tocopherols and tocotrienols (Shahidi and Naczk 2004). Among the group of flavonoids are flavonols, flavones, flavanols, anthocyanidins, flavonones, and isoflavones (Shahidi and Naczk 2004; Webb 2006). Tocopherol (α-T, β-T, γ-T, and δ-T) and tocotrienol (α-T3, β-T3, γ-T3, and δ-T3) isomers offer vitamin E activity (Packer and Weber 2001; Shahidi and Naczk 2004).

Of the phenolics, sea buckthorn berries contain flavonoids [e.g. leucocyanidin, catechin, flavonol (e.g. isorhamnetin, quercitin, quassin, and camellin), and a trace of flavanone] (Hakkinen et al. 2000), lignans (Yang et al. 2006), and tocols (Oomah 2003). The vitamin E content represented by the presence of tocopherol and tocotrienol isomers,

is higher in pulp and peel oil fractions than in seed or juice oil. Alpha-T is the major tocopherol isomer in pulp and peel and seed oil (Kallio et al. 2002a). Gamma-T is the second most predominant isomer in seed oil. Kallio et al. (2002a) reported low levels of tocotrienols in both pulp and peel and seeds. Levels of vitamin E measured in sea buckthorn berries exceed those found in otherwise rich sources (e.g. wheat embryo, safflower, maize, and soybean) (Lu 1992).

Fatty acids

The fatty acid composition, which differs between plant products, determines the physical properties, stability, and nutritive value of lipids (Kołakowska and Sikorski 2003). Fatty acids are categorized into two broad classes based on chemical structure: saturated and unsaturated fatty acids. Common saturated fatty acids are C12:0 (lauric), C14:0 (myristic), C16:0 (palmitic), and C18:0 (stearic) (Dobson 2002). Important unsaturated fatty acids in nutrition include C16:1*n*7 (palmitoleic acid), C18:1*n*9 (oleic acid), C18:2*n*6 (linoleic acid), C18:3*n*3 (α-linolenic acid), C20:4*n*6 [arachidonic acid (AA)], C20:5*n*3 [eicosapentaenoic acid (EPA)], and C22:6*n*3 [docosahexaenoic acid (DHA)]. Linoleic and α-linolenic acids are considered essential fatty acids, since they cannot be synthesized by the human body and must be consumed in the diet (Shahidi and Senanayake 2006).

Yang and Kallio (2001) determined that a characteristic property of sea buckthorn berry pulp and peel oil is the high content of palmitoleic acid. Berry pulp and peel oil consists of 36% saturated fat (mainly palmitic acid) and 64% unsaturated fat (palmitoleic,

oleic, linoleic, and α-linolenic acids). Although high in concentration in berry pulp and peel, palmitoleic acid is low in seed oil. Seed oil is characterized by high C18 unsaturated fatty acids (linoleic, α-linolenic, and oleic acids) and lower saturated fat content (palmitic and stearic acids).

Carbohydrates and derivatives

Among the carbohydrate and derivatives class, ascorbic acid (vitamin C) is one of the most used functional ingredients. Certain oligosaccharides may function as prebiotics which promote the growth of beneficial bacteria in the GI tract. Another term for non-starch plant polysaccharides is fiber which can be separated into two groups: soluble and insoluble. Included within the insoluble fiber group are cellulose, hemicellulose, and the phenol, lignan (Jalili et al. 2001).

Sea buckthorn berries are rich in sugar (e.g. glucose, fructose, mannitol, sorbitol, xylose, and xylitol), organic acids (e.g. malic, citric, tartaric, succinic, and d-malic), and vitamin C (Beveridge 2003b). Li and Wang (1998) reported that vitamin C content in sea buckthorn berries exceeds levels in strawberry, kiwi, orange, tomato, carrot, and hawthorn.

Proteins and amino acids

The protein and amino acid based group includes intact protein (e.g. soy), polypeptides, amino acids, and nitrogenous and sulfur amino acid derivatives (e.g. capsaicinoids, isothiocyanates, and allyl-s compounds) (Wildman 2001b). The amino

acids, arginine, ornithine, taurine, and aspartic acid have been investigated for their functional activity.

The cloudiness in sea buckthorn juice is attributed to the presence of proteins (Beveridge 2002). Amino acids identified in sea buckthorn berries include aspartic acid, praline, ammonia, threonine, serine, lysine, valine, alanine, phenylalanine, glutamine, isoleucine, glycine, histidine, tyrosine, arginine, cysteine, and methionine (Beveridge 2003b).

Elements

Specific elements (e.g. calcium, potassium, and trace minerals such as copper, selenium, manganese, and zinc) are recognized for their functionality and are being included as nutraceutical and functional food ingredients (Wildman 2001b). Twenty five elements and trace elements have been identified in sea buckthorn berries with the highest concentrations being potassium (497 μg/mL), calcium (143 μg/mL), phosphorus (131 μg/mL), magnesium (70.4 μg/mL), and sodium (76.9 μg/mL) (Tong et al. 1989).

2.2.4 Activity and health benefits

Dobson (2002) has implied a link to a lower incidence of diet related degenerative diseases in individuals and populations that consume a diet high in vegetables and fruits and low in saturated fat. Due to inconclusive results between epidemiological and animal and human case studies, complete evidence may not exist between certain bioactives and health benefits in humans. A discussion of the specifics of case studies and their design

flaws is beyond the scope of this review. Brief mention, however, is made with regards to studies on sea buckthorn oil-based compounds (e.g. fatty acids, carotenoids, tocols, and phytosterols).

Yang and Kallio (2002a) reported that more than 60 publications are available on the link between physiological effects [e.g. anti-inflammation, antimicrobial action, pain relief, the promotion of tissue regeneration (skin and mucosa), boosting of the immune system, and protection against cancer and cardiovascular disease] and sea buckthorn oils. While most papers are available in Chinese or Russian, there are some English publications.

Xing et al. (2002) reported that oral administration of SCFE extracted seed and pulp (ssp. *rhamnoides*) oils had a preventative and curative effect against experimental gastric ulcers in rats. In a small human study (i.e. 12 participants), Johansson et al. (2000) reported beneficial effects of sea buckthorn oil on blood clotting, however, further studies were required on dose-response. Insignificant increases (20%) in plasma high density lipoprotein (HDL) were reported with the consumption of sea buckthorn juice (Eccleston et al. 2002). Yang et al. (2000) reported that although small changes in the skin glycerophospholipids resulted in patients with atopic dermatitis after oral administration of seed oil, fatty acid composition of skin glycerophospholipids were well buffered against short term modifications in diet.

Antioxidant effects

Sea buckthorn oils offer important functions in nutrient and antioxidant (e.g. tocols and carotenoids) delivery. Antioxidants can restrict the effects of oxidation either directly (i.e. elimination of free radicals) or indirectly (i.e. prevention of radical formation) (Packer and Weber 2001; Shahidi and Naczk 2004). Although essential to the body, in excess, free radicals may lead to cyto-toxicity and oxidative damage to healthy tissue (Packer and Weber 2001). Since oxidative stress has been linked to many degenerative diseases including cancer, cardiovascular disease, atherosclerosis, arthritis, and diabetes, antioxidants can potentially play a strong role in disease prevention (Tomaino and Decker 2000). Based on evidence, Packer and Weber (2001) stated that the structural difference between the tocotrienols and tocopherols causes the former to potentially be more mobile, thus increasing their effectiveness over that of the tocopherols.

Effects on skin and mucosa

Tissue regeneration, anti-inflammatory, and anti-microbial effects of topically applied preparations containing sea buckthorn oils are associated with the healing of wounds (Mironov et al. 1983), burns (Lebedeva et al. 1992), and irradiation dermatitis (Zhang et al. 1988) of the skin. Sterols (Lebedeva et al. 1992) and long chain alcohols (Kallio et al. 2001) have been identified as the bioactive compounds responsible for these effects. The fatty acid composition of sea buckthorn oils has been deemed as offering benefits in the oral treatment of atopic dermatitis (Yang et al. 1999). In the treatment of ulcers, sterols are suggested to be one of the compound groups to be the major contributor to healing,

with pulp and peel oils being superior to seed oils (Jiang and Li 1987; Mironov et al. 1989).

Effects on risk factors of cardiovascular diseases

There is evidence that phytosterols and fatty acids can provide protection against cardiovascular disease due to their hypocholesterolemic effects (Dobson 2002; Normén et al. 2004). Preventative effects include: decrease of plasma total and low density lipoprotein (LDL)-cholesterol levels; increase in level of HDL-cholesterol; inhibiting thrombus formation and atherosclerosis, and retarding oxidation of LDL (Eccleston et al. 2002; Jiang et al. 1993; Johansson et al. 2000). The essential fatty acids, linoleic and α-linolenic acids, convert to eicosanoids (e.g. AA, EPA, and DHA) which positively affect physiological reactions ranging from blood clotting to immune response (Shahidi and Senanayake 2006). Oleic acid, a monounsaturated fatty acid, is valued for its cholesterol lowering effect.

Effects on immune function

In an experimental model, increased immune function was reported for mice administered sea buckthorn seed oil (Ren et al. 1992; Wang et al. 1989). The oils and water soluble components (e.g. flavonoids, vitamin C, and lignans) of sea buckthorn juice are also considered as possible contributors to improved immune function of mice (Yang and Kallio 2002a). Seed oil reportedly boosted the immune function of cancer patients undergoing chemotherapy (Li and Tan 1993).

Prevention of cancer

Intraperitoneal injection of sea buckthorn seed oil has been reported to suppress the growth of pre-innoculated tumors (Zhang et al. 1989a). Yang et al. (1989) reported elongated living periods in mice pre-innoculated with tumor cells, when orally administered sea buckthorn press residue oil. In the same publication, it was suggested that sea buckthorn oil may have a cyto-toxic effect on the human leukemia cell line K562.

2.2.5 Harvesting methods

Sea buckthorn berries are a challenge to harvest and store as are most soft fruit. Harvest is complicated by softness of mature berries, lack of abscission layer, and the presence of long spiny thorns (Beveridge 2003a). Due to the lack of abscission layer the berries persist on the shrubs all winter. A variety of harvesting methods have been utilized or researched worldwide, ranging from manual to semi-automated harvesters as summarized by Beveridge (2003a).

Manual methods can be assisted with hand operated devices that comb or brush the fruit off the branch and into a receiving vessel. Hand harvesting is performed in China and requires 1500 person-h/ha (Gaetke and Triquart 1992), which is not practical for the higher-cost labour markets of North America. The various semi-automated systems include: 1) cutting of branches and subsequent berry removal through shaking, screen conveyor and fans, or freezing and beating; 2) shaking of the shrub or branches and

subsequent collection of the berries, and 3) vacuuming (i.e. rapid air flow) (Beveridge 2003a).

Mann et al. (2001) determined that a test branch shaker employed at a frequency and amplitude of 25 Hz and 32 mm, respectively, removed 98% of berries within 15 s. A November harvest time was more suitable than January, since during the latter the branches broke causing damage to the shrub. Certain varieties are more difficult to harvest than others (i.e. ssp. *sinensis* versus ssp. *rhamnoides* cv. *Indian summer*). More knowledge is required on the effects that the shaking process may have on the shrub, specifically with regards to the effects on the roots (e.g. recovery time) and the effect of abrasion of branches (S. Cenkowski, Professor, University of Manitoba). The freezing and beating approach was developed in Germany and includes freezing (i.e. naturally or mechanically at ~-36°C) of fruit laden branches and subsequent beating of the branches on a hard object to remove the solid berries (Wolf and Wegert 1993). Since sea buckthorn sets fruit on second year wood, cutting the branches results in a harvest every two years or longer (Beveridge 2003a). There may be some evidence that improper or continued pruning of the shrubs may expose them to disease.

Berries generally ripen toward the end of August to late September in the Canadian Prairies (Beveridge 2003a). With the lack of current availability of a commercial harvester, berries are harvested at post-maturity at winter temperatures below -20°C (Plate 2.1). In Europe and China, berries are normally harvested from the end of August to middle of September and from end of September to end of November, respectively (Gao et al. 2000; Yang and Kallio 2002b; Yang and Kallio 2001).

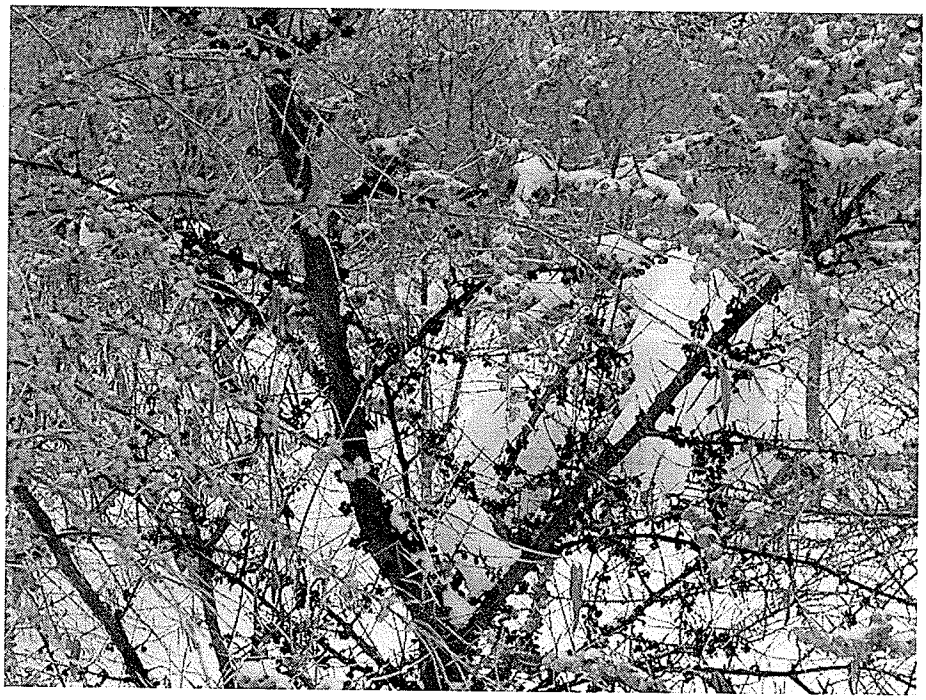


Plate 2.1 Sea buckthorn sinensis berries on shrub in January 2004 at St. Claude, MB.

2.2.6 Processing methods

Hundreds of commercial products (e.g. including pharmaceuticals, nutraceuticals, beverages and foods, cosmetics and skin preparations, sunblocks, fermented products, animal feeds, and pigment) containing sea buckthorn derivatives have been developed in Europe and Asia (Schroeder and Yao 1995) with product development now extending to North America. Once harvested, berries are perishable and must be cooled to 4 - 6°C if they are to be used within a few days (Li 2002). If usage or processing is to be delayed beyond a few days, the berries must be frozen [e.g. individual quick frozen (IQF)]. The berries can be thawed and used or processed when required. Alternatively, berries may be processed immediately and stored as pasteurized or sterilized final products.

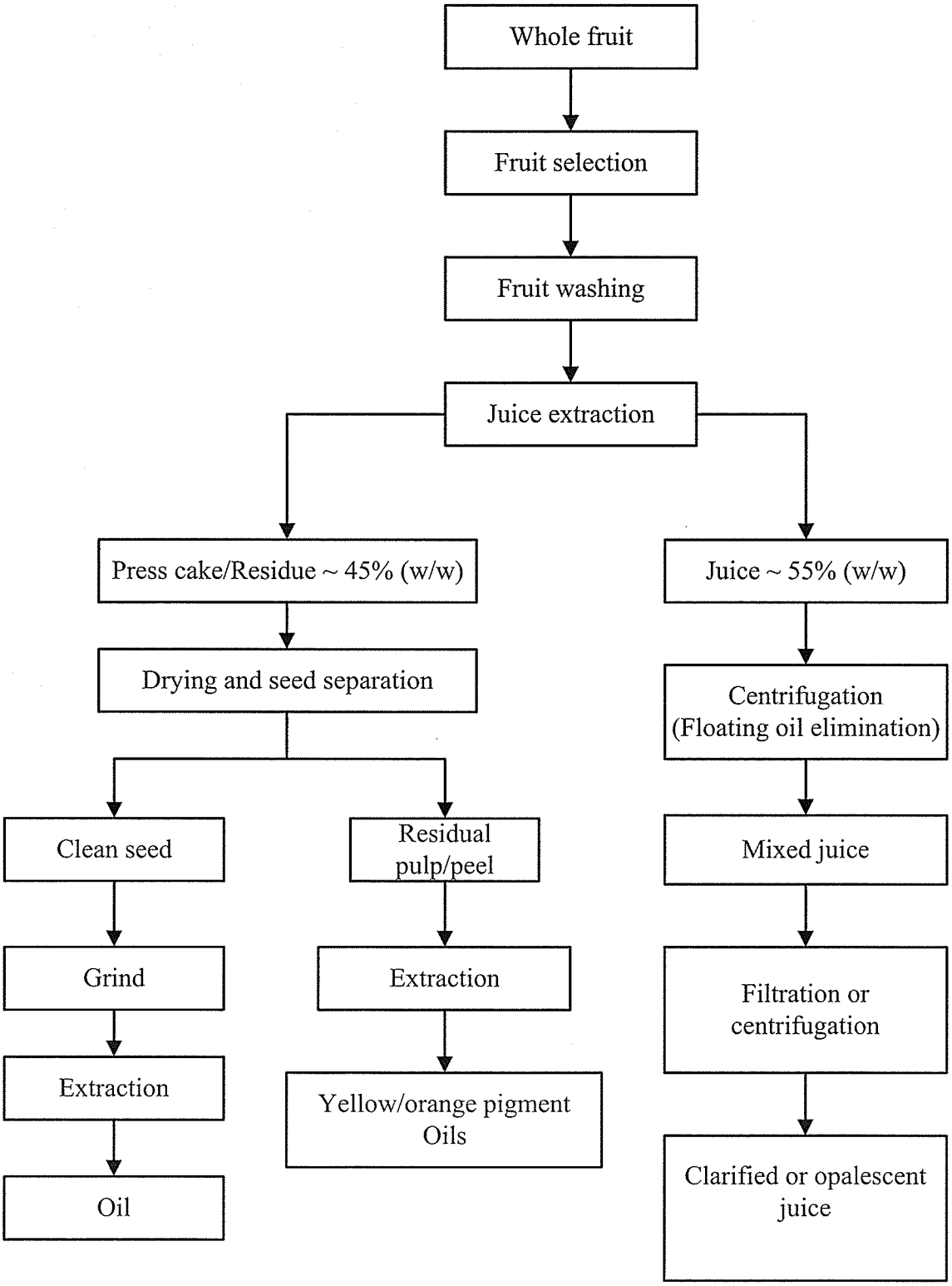


Figure 2.1 Processing plan for sea buckthorn berries. (Adapted from Beveridge et al. 1999. With permission.)

The favoured processing path of sea buckthorn berries is depicted in Fig. 2.1. The preliminary steps prior to juice separation include fruit selection, inspection and washing. Fruit selection and inspection is necessary to remove diseased, damaged, and pest infested berries (Beveridge et al. 1999) followed by washing, recommended for the removal of microorganisms, dust, dirt, and the characteristic "musky" odour (Liu and Liu 1989). The inclusion of berry washing is controversial as it may result in the dilution of soluble solids and the introduction of foreign chemicals or microorganisms (Beveridge et al. 2002).

The investigation into juice extraction has included decanter centrifugation (Zhang et al. 1989c), rack and cloth and serpentine belt pressing (Heilscher and Lorber 1996), and screw pressing (Arimboor et al. 2006). At this point the processing branches off in two directions: 1) juice and 2) press cake processing. The three main resulting products include juice, oils (juice, seed, and pulp and peel), and yellow-orange pigment (pulp and peel) (Beveridge et al. 1999).

Juice

The juice resulting from the press or decanter operation is a turbid product high in suspended solids (Beveridge et al. 1999). The suspended solids in an oil layer leave a ring on the bottle surface. Retaining the juice as is, is not desirable from a North American consumer standpoint, therefore further separation is required (Figure 2.1). One processing path that is recommended includes reduction of the oil layer (< 0.1%) centrifugally, resulting in a "mixed" juice (Zhang et al. 1989c). If allowed to sit for 1 to

2 days, the "mixed" juice separates into a floating particulate phase, a fairly clear middle liquid portion, and particulate sediment. Subsequent centrifugation (Zhang et al. 1989c) or filtration (Liu and Liu 1989) can yield an opalescent or clarified juice, respectively.

An alternative approach to assist with the removal of suspended solids includes treating the extracted juice with pectin methylesterase (Liu and Liu 1989) or commercial hydrolytic enzyme preparations (Beveridge et al. 1999). Heilscher and Lorber (1996) patented a technique to yield a clarified juice using crystalline sugar for sedimentation and subsequent centrifugation. Regardless of the process employed, sea buckthorn juice is commonly developed for the sports or health drink market (Li 2003a). A by-product of the juice clarification process is the suspended solids that can be spray dried and used as a nutrient supplement for foods and nutraceuticals (Beveridge 2003c). Residual products from juice manufacturing can be used in the brewery industry (Li 2003a).

Pigment

Sea buckthorn press cake consists of pulp, peel, and seeds. Drying the press cake facilitates the removal of seeds and enables the subsequent oil extraction from the pulp and peel portion using SCFE (Arimboor et al. 2006; Cenkowski et al. 2006). A yellow-orange pigment (containing flavones, carotenoids, and tocols) can also be extracted using ethanol from a concentrated solution of 11 to 13°Brix (Beveridge et al. 1999). The pigment is suitable for colouring pharmaceutical or cosmetic creams or for addition to foods where yellow-orange colours may be favourable.

Oligomeric proanthocyanidins (Rösch et al. 2004) and lignans such as secoisolariciresinol and matairesinol (Yang et al. 2006), identified in the pulp and peel portion of the press cake also increase its value as a potential commodity for the functional food and nutraceutical industries. Residues from the pulp and peel processing area are used for animal feeds due to the presence (at lower concentration) of compounds (Li 2003a).

Oils

Oils can be extracted from the juice, seeds, and pulp and peel of the press cake or decanter waste product. The seed is separated from either the wet (Beveridge 2003a) or dried press cake (Arimboor et al. 2006), ground, and processed by extraction to remove the oil. Cenkowski et al. (2006) investigated several extraction methods for pulp and peel and seed fractions, including solvent extraction using petroleum-ether, SCFE using carbon dioxide (CO_2), screw pressing, and aqueous extraction. Fatty acid profile of pulp and peel oil did not vary between the extraction methods, however, petroleum-ether extraction resulted in highest content of total carotenoids and major sterols. Petroleum-ether and aqueous extraction methods worked equally well for major tocopherols and tocotrienols. SCFE CO_2 technology provided the best oil quality from seeds (Cenkowski et al. 2006). In a comparison with hexane and cold press extraction methods, Li et al. (2007) also reported higher phytosterol contents in SCFE CO_2 extracted seed oil samples. The residual material from oil extraction can be used for animal feed (Li 2003a).

While sea buckthorn oils are generally used in nutraceuticals and cosmetics (e.g. skin preparations and sunblocks), incorporation of oils into basic foods such as bread, juice, and yogourt represents a movement toward adding functionality to daily foods (Yang and Kallio 2002a). To preserve the stability of sea buckthorn oils and compounds, microencapsulation in modified starches (Partanen et al. 2002) and furcellaran, an extract from red algae (Laos et al. 2007), have been proposed. No matter what the product (e.g. beverage, nutraceutical, or functional food) quality in the form of uniformity, safety, and effectiveness is important from a consumer standpoint.

2.3 Quality of sea buckthorn berry oils

The quality of plant-based bioactive compounds naturally present in foods as well as those added to nutraceuticals and functional foods can be affected by many factors. These factors include: methods and time of harvest, storage (post-harvest or post-processing), processing, and methods of extraction (Tatum and Chow 2000). In general, they can alter the stability of compounds within the product, thus causing chemical reactions to ensue. The bioactive quality and content of oils is a marker for the overall quality of the sea buckthorn berry and its components (i.e. pulp and peel, juice, and seed) (Oomah 2003). As was noted in Section 2.2.6, drying is a fundamental operation in the development of many products including pulp and peel, juice, and oil based products. This section focuses on quality effects pertaining to harvest time and berry maturity and conventional methods of drying (e.g. forced air convective).

2.3.1 Quality assessment

Traditionally food quality has been based on appearance, taste, texture and in the case of dried products, parameters such as shrinkage, colour, rehydration ratio, and bulk density (Perera 2005; Schreiner et al. 2000). Generally a higher quality dried product displays minimal shrinkage, a higher rate of rehydration, and a relatively high bulk density (Van Arsdel et al. 1973). With the development of new extraction methods and compound identification standards, product quality assessment can also include bioactive compound measurement.

Colour

Colour is an important quality marker, since it can be an indicator of the presence of certain bioactive compounds (e.g. carotenoids) (Mínguez-Mosquera et al. 2002); stage of maturity (e.g. loss of chlorophyll) (Tadesse et al. 2002), and degradation of product (e.g. browning) (Perera 2005). Colour evaluation is generally based on the tristimulus approach with the Judd-Hunter *Lab* solid and CIELab systems most often applied to food analysis (Francis 2003). Two principal attributes of colour interpreted by CIELab systems are hue and lightness (Delgado-Vargas and Paredes-López 2003). Hue is the quality normally identified with a colour name (i.e. red, green, yellow, and blue). Lightness represents the light reflected by a surface in comparison to a white surface, under similar conditions of illumination.

Stability of bioactive compounds

The determination of bioactive compound content in a product provides a snapshot of the chemical constituents at a specific instant. Recording the content over a period or between treatments can indicate changes that may be associated with certain conditions. The individual mechanisms by which these changes occur are complex and may be related to 1) fruit development and ripening, 2) pre-harvest climatic conditions, and 3) post-harvest handling and processing (Perera 2005; Schreiner et al. 2000). During fruit development, changes to colour, size, and structure are apparent and it is expected that chemical changes (e.g. biosynthesis) accompany these physical changes (Oomah 2003). In addition to the normal biosynthesis mechanisms that occur in fruit development, climatic and processing conditions can affect the stability of bioactive compounds during pre- and post-harvest stages, respectively.

Many compounds including unsaturated fatty acids, tocols, carotenoids, and phytosterols are susceptible to oxidation and can serve as initiators of oxidative reactions (deMan 1999). Oxidation can occur via three main routes: autoxidation, photooxidation, and enzymatic oxidation (Kołakowska 2003). The sequence of events involved with autoxidation, as described by Tatum and Chow (2000), begins with the oxidation of a fatty acid to a hydroperoxide. Oxygen is required and the process is catalysed by the presence of metals, heat, and light. Hydroperoxides are tasteless and odourless, however, continued oxidation causes them to transform into various short-chain organic compounds. These compounds such as aldehydes, ketones, alcohols, and acids, are responsible for the strong odours and flavours characteristic of rancidity. Certain

oxidation products are also potentially toxic, usually occurring at high levels of oxidation. Pro-oxidants include transition metals (e.g. cobalt, copper, iron, manganese, and nickel). Metals are found in the soil and can be obtained from metallic equipment used in processing or storage. Trace metals are also naturally occurring components of all food tissues and all fluid foods of biological origin as was noted for sea buckthorn berries (Beveridge et al. 1999; deMan 1999; Tong et al. 1989).

Light-induced oxidation or photooxidation results from reactivity of singlet oxygen ($^1O_2$). Normal or ground-state oxygen is triplet oxygen ($^3O_2$). Singlet oxygen results during a reaction of sensitizers (e.g. chlorophyll, hemoglobin, myoglobin, and riboflavin) with atmospheric oxygen. Photooxidation involves the formation of hydroperoxides in a direct reaction of singlet oxygen and unsaturated fatty acids (Kołakowska 2003). Singlet oxygen is short lived and reverts to the ground state with the emission of light. Enzymatic oxidation in plant systems is mediated by a widely occurring group of enzymes (e.g. lipooxygenase) that use oxygen to catalyse the oxidation of lipids. This reaction leads to the formation of conjugated hydroperoxides (deMan 2000) and can initiate oxidation of compounds (e.g. carotenoids, chlorophyll, tocols, and protein) (Kołakowska 2003).

While antioxidants have been noted to restrict oxidative damage *in vivo*, they also contribute to the stability of food products. Photooxidation can be halted by compounds known as singlet oxygen quenchers, such as the carotenoid β-carotene and tocols (Eitenmiller and Lee 2004). Antioxidants (e.g. vitamin C, tocols, and carotenoids) either added or present in the product can reduce the rate of autoxidation by scavenging radicals

(Cohen et al. 2000). With the complexity of food systems, antioxidants can also act synergistically to inhibit or reduce oxidation (Eitenmiller and Lee 2004). In their role as antioxidants, tocopherols become tocopheroxyl radicals and can be regenerated with the assistance of vitamin C and carotenoids thus restoring antioxidant activity. In the presence of high oxidation and in large concentrations, carotenoids can act as pro-oxidants reacting with oxygen to produce radicals, feeding the oxidation process (Palozza 2004). Tocopherols, specifically γ-T can inhibit the pro-oxidant effect of carotenoids and colour changes (Eitenmiller and Lee 2004). Changes in bioactive compounds may signal oxidative mechanisms, however, with the synergistic and regeneration ability of antioxidants the effects of chemical reactions may not be apparent.

2.3.2 Influence of growing conditions and harvest time

Growing conditions

Climatic conditions influence oil accumulation in sea buckthorn berries (Schapiro 1989). Dry and warm periods during the spring and fall increase oil content production. Humid conditions, extended wet and cold weather, and shortened periods of sunshine result in low oil content. Other parameters that affect oil content include genetics, pollen origin, growing altitude, and time of harvest (Oomah 2003). Seed oil content accumulates at a very fast rate from the onset of maturity to a maximum and remains constant or begins to decrease with fruit maturation and ripening. Pulp oil content rises slowly and levels remain constant as the fruit reaches full maturation and ripening. Zadernowski et al. (1997) reported that leaving the berries on the shrub or freezing them did not affect oil content. Content of individual bioactive compounds within the oils is

influenced by climatic conditions, geographical location, berry variety, species, and maturity (Oomah 2003).

Maturity

Level of maturity at harvest has an impact on content and quality of individual compounds prior to processing and can ultimately affect final product quality. In fleshy fruits, the onset of ripening is often associated with colour changes, changes in sugar metabolism, fruit softening and alterations in texture, synthesis of aroma volatiles, and an increased susceptibility to pathogen infections (Barry and Giovannoni 2007). Sea buckthorn fruit are classified as non-climateric, producing only a trace of ethylene during the ripening stage (Harrison and Beveridge 2002). However, contrary to non-climateric fruits, respiration rate, the rate at which stored organic materials (e.g. carbohydrates, proteins, and fats) are broken down, does increase during this period (Zhang et al. 1989b). The variation in content and quality of fatty acids, carotenoids, tocols, and sterols during the ripening stage of sea buckthorn berries has been documented (Gao et al. 2000; Kallio et al. 2002a; Yang and Kallio 2002b; Yang et al. 2001) and will be further discussed in Sections 3.2.6-3.2.9.

Post-maturity

There is a lack of information on berries harvested months after ripening. Berries at post-maturity are exposed to a variety of climatic conditions. Fluctuations and freeze and thaw cycles associated with late fall and winter temperatures can cause extensive crystal formation and physical change, increasing the possibility of oxidation (Erickson 1997).

Extended exposure to air and sunlight can also contribute to photooxidation and subsequent degradation of carotenoids. The effects that late harvest and long-term freezer storage may have on individual compounds have not been reported for sea buckthorn berries.

2.3.3 Influence of forced air convective drying

Drying effects

Drying is a complex process that may involve simultaneous heat and mass transfer, with the subsequent removal of moisture from the product (Cassini et al. 2006). The individual mechanisms involved with moisture removal using forced air convective drying systems are further discussed in Sections 2.5.1-2.5.2. Within the convective drying process exists the potential for enzyme-catalysed reactions, non-enzymatic and Maillard reactions, protein denaturation, and nutrient loss (Roos 2004). These effects can occur as a result of exposure to the drying conditions (e.g. temperature, relative humidity, and airflow) and are dependent on the product's characteristics and drying behaviour. The conditions attributed to product degradation are included in the discussion on the link between drying conditions and quality factors (i.e. colour and bioactive content) in sea buckthorn berry pulp and peel (Sections 4.2.1-4.2.7).

Comparable research

Research into the stability of oil based bioactive compounds and other quality indicators in products, upon exposure to different convective drying conditions, is

limited. Colour as an indicator of carotenoid content, was assessed in rosehips dried convectively at temperatures between 30 and 70°C (Koyuncu et al. 2003). Regier et al. (2005) directly measured carotenoid concentration to determine the effect of convective drying temperatures of 50 to 90°C on the quality of carrot slices. Both colour analysis and carotenoid content were analysed in mango pulp dried in a spout fluidized bed dryer at temperatures of 55 to 75°C (Da Cunha et al. 2006). Minimal colour change and high carotenoid content is generally indicative of quality preservation and enables selection of favourable drying conditions (Da Cunha et al. 2006; Koyuncu et al. 2003; Regier et al. 2005). However, in the production of paprika, red peppers are dried at higher temperatures to invoke caramelization which in turn is correlated with high carotenoid and tocopherol content (Márkus et al. 1999). Although not the typical red to orange colours of carrots, rosehips, and red peppers, convectively dried green sea buckthorn leaves were also evaluated for total carotenoid content (Guan et al. 2005).

Minimal changes in fatty acid composition, was one of the factors used in the determination of the most suitable drying method (e.g. sun, oven, or freeze dried) for brown seaweed (Chan et al. 1997). Only one set of drying conditions was investigated for each method, therefore a comparison of different convective drying temperatures is not provided. Similarly, using only one set of drying conditions each, Gutiérrez et al. (2008) analysed the effect of freeze drying compared to forced air convective drying on total lipid content, oil extractability, and peroxide value (i.e. indication of degree of oxidation) for mashed sea buckthorn berries.

In the above mentioned reports, with the exception of Gutiérrez et al. (2008), determination of the cause of compound loss or retention is not investigated, but is based on knowledge of factors (e.g. oxidation, temperature sensitivity, and browning) that can affect product quality. Details of the results of these studies are further discussed in Sections 4.2.3-4.2.7.

2.4 Drying

Drying may be used as a processing stage: to improve shelf life; for quality preservation (i.e. elimination or reduction of the growth of spoilage micro-organisms and chemical reactions); product enhancement; to simplify handling, storage, and transport, and as a pretreatment for subsequent processes (Cassini et al. 2006; Vega-Mercado et al. 2001). Many drying systems and technologies are suitable for plant products. Selection of the appropriate drying method depends on original product and bioactive compound characteristics, availability of equipment and technology, required quality and characteristics of final product, and economics. Drying methods that are suitable for the processing of plant-based solids for use in the nutraceutical and functional food industries are summarized in Sections 2.4.1-2.4.2.

2.4.1 Applicable drying methods

Technologies applicable to solids drying can generally be divided into two groups: atmospheric, mainly forced air convective (e.g. cabinet, tray, tunnel, belt, conveyor, rotary, vortex, fluidized bed, vibrated fluidized bed, and spouted bed) and sub-atmospheric or alternative drying (e.g. freeze, vacuum, microwave, and osmosis) (Vega-

Mercado et al. 2001). Although sub-atmospheric systems generally offer a higher quality product (Ratti 2001), quality may have to be compromised when considering cost and practicality (Grabowski et al. 2002). Due to sea buckthorn press cake being an industry by-product, forced air convective dryers may offer the most practical solution available. Forced air convective dryers have long been used by the fruit industry with optimization of the drying parameters based on characteristics of the product (Somogyi 2000).

Researchers have determined that degrading reactions associated with forced air convective drying systems (e.g. cabinet, tray, tunnel, belt, and conveyor band dryers) are generally time and temperature dependent (Nindo et al. 2007). Modifications to particle and air interactions (through the implementation of fluidization and movement of the particles within the bed), are offered by fluidized, pulsed fluidized, vibrated fluidized, spouted bed, vortex, and rotary dryers. An increase in exposed surface area of the product offers an alternative for heat and oxygen sensitive products due to faster drying rates and as a result reduced drying times and the potential for lower drying temperatures (Kundu 2004).

Cabinet, tray, and tunnel dryers

Cabinet, tray, and tunnel drying systems employ warm air flowing over the surface of a thin layer of product spread on mesh or solid trays (Sokhansanj and Jayas 2006). These systems are typically used for sliced, chunked, or whole fruit and vegetables or other plant material such as seeds, flowers, roots, and leaves (Orsat and Raghavan 2007; Vega-Mercado et al. 2001). The use of solid trays also allows for the drying of fluids and semi-

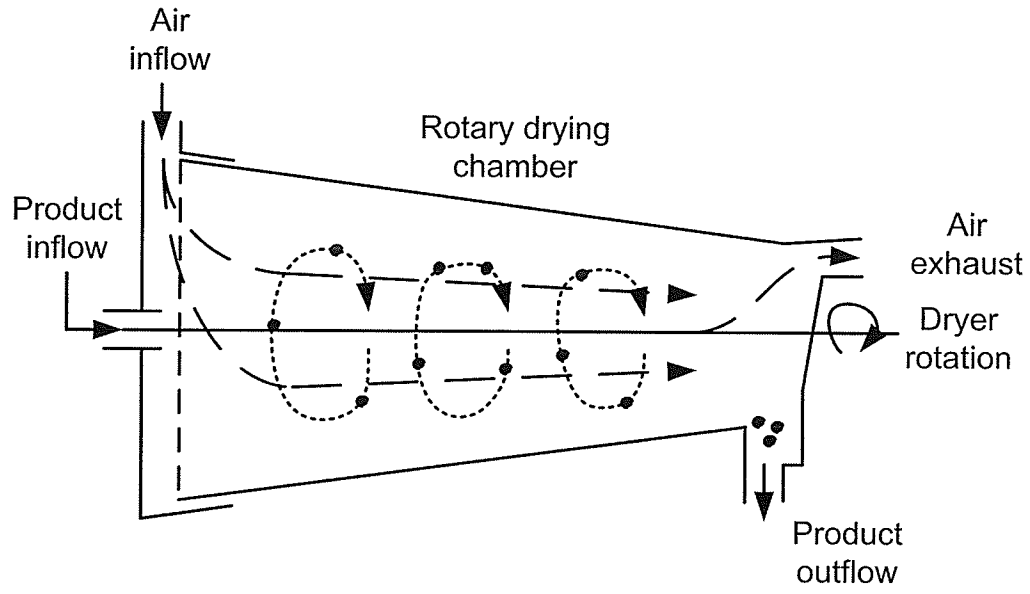
solid products such as slurries, purees, and pomace. The main differences between the batch dryers (tray and cabinet) and continuous tunnel drying systems are configuration and capacity, with the latter being applicable to high volume products.

Belt and conveyor band dryers

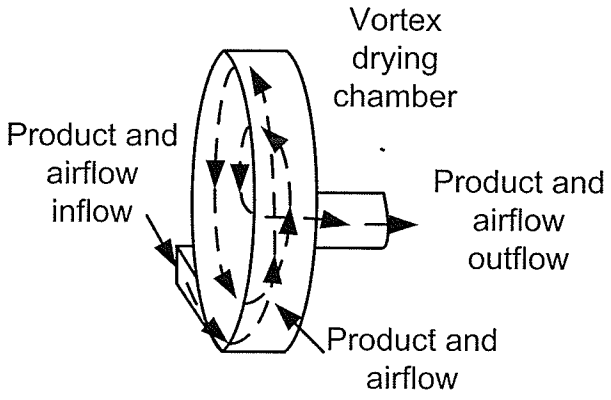
In belt and conveyor band dryers the drying surface moves during the drying cycle and air flow is through rather than over the thin product layer (Sokhansanj and Jayas 2006). Conveyor band dryers offer the flexibility of having separate regions with different air characteristics, to accommodate product requirements throughout the drying cycle, as well as continuous drying (Orsat and Raghavan 2007).

Rotary and vortex dryers

A rotary dryer (Fig. 2.2a) consists of an angled cylindrical rotary chamber turning at a slow speed. Drying air and wet solids are fed continuously at one end of the chamber and are discharged at the other end (Kundu 2004). Due to rotation, the material is continuously churned, leading to uniformity in mixing with air. However, the rotational system leads to an increase in capital investment. Retention time is a function of speed and angle of inclination of the drying chamber. Improved drying performance was reported for some products dried in a countercurrent flow (i.e. air flows in at the material outlet).



a)



b)

Figure 2.2 Schematic of product and air flow in a) a rotary dryer and b) a vortex dryer. (Adapted from Benali 2004; Sokolovskii et al. 1976. With permission.)

This system was traditionally applied to chemical drying but has been used for some agricultural products such as beans, rice, nuts, seeds, cereal grains, and herbs (Benali 2004). Olive oil industry by-products such as pomace, press cake, and bagasse are dried from initial moisture contents of 25-55%, 45%, and >75%, respectively, down to

approximately 6% using concurrent or countercurrent rotary dryers (Doymaz et al. 2004; Friere et al. 2001; Göğüs and Maskan 2006). To minimize the effects on the environment by these potential pollutants, drying is used as a preliminary step in the production of a fuel source, fertilizer, or soil conditioner. Drying temperatures ranging from 125 to 250°C are typically used, however, lower temperatures (50 to 110°C) have been investigated for the quality preservation of press cake oil usable in the soap, animal feed, and high quality fertilizer industries (Akgun and Doymaz 2005; Doymaz et al. 2004; Göğüs and Maskan 2006).

Although the use of a rotary dryer for sea buckthorn press cake has not been reported, Sokolovskii et al. (1976) developed a vortex dryer that was later implemented in Russian factories. A vortex drying chamber (Fig. 2.2b) consists of a hollow disk with product entering tangentially with the incoming air flow. The product and the air move in spirals, while the walls of the disk provide friction and retardation. Due to centrifugal forces that develop, the heavy and moist particles are forced towards the walls while the lighter dry particles flow to the centre of the chamber and are discharged to a collection apparatus. The design was based on initial experiments that determined a concurrent flow vortex dryer (800 mm diameter and 160 mm wide) provided better retention of β-carotene, process stability, and shorter drying times than cabinet, belt, and vibro-fluidized bed dryers. The action of the vortex dryer also contributed to the separation of the seed from the pulp, a step that is subsequently necessary. Rotary and vortex drying systems are not presently in the forefront of drying research for functional foods and nutraceuticals, due to major emphasis being placed on fluidized and spouted bed systems.

Fluidized bed dryers

Fluidized bed dryers (Fig. 2.3) have a perforated floor through which air flows at a specific velocity to fluidize the product (Benali 2004). This dryer type is suitable for granular and solid products such as oilseeds (e.g. mustard, sunflower, soybean, groundnut, and rapeseeds) and has been investigated for many other products including cranberries (Grabowski et al. 2002) and granulated pharmaceuticals (Hlinak and Saleki-Gerhardt 2000).

Systems such as the pulsed fluidized bed (Fig. 2.4) and the vibro-fluidized bed dryers (Fig. 2.5) tested better than the fluidized bed dryer for products difficult to fluidize due to stickiness or poly-dispersity (Benali 2004; Grabowski et al. 2007). The aerodynamic and mechanical functions of the pulsed fluidized bed and vibro-fluidized bed dryers, respectively, can assist in preventing agglomeration. The pulsed fluidized bed dryer, a novel modified version of conventional fluidized bed systems employs gas pulses to provide high-frequency retraction of the particle bed. These methods have been investigated for the drying of particulate solids such as diced carrots, cranberries, blueberries, and onions (Benali 2004; Grabowski et al. 2007; Grabowski et al. 2002). Grabowski et al. (2002) reported that energy efficiency was higher for the pulsed fluidized bed and vibro-fluidized bed dryers over that of the fluidized bed dryer for osmotically pre-treated halved cranberries. Any of the fluidized bed dryers can be either run continuously or in batch mode.

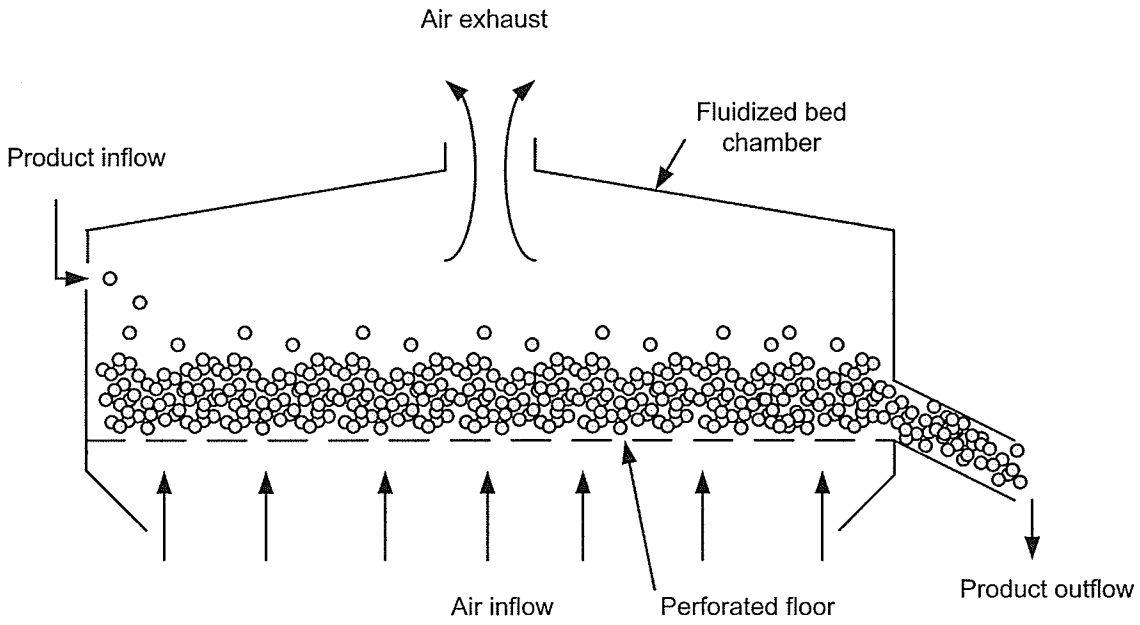


Figure 2.3 Schematic of product and air flow in a fluidized bed dryer.
(Adapted from Benali 2004. With permission.)

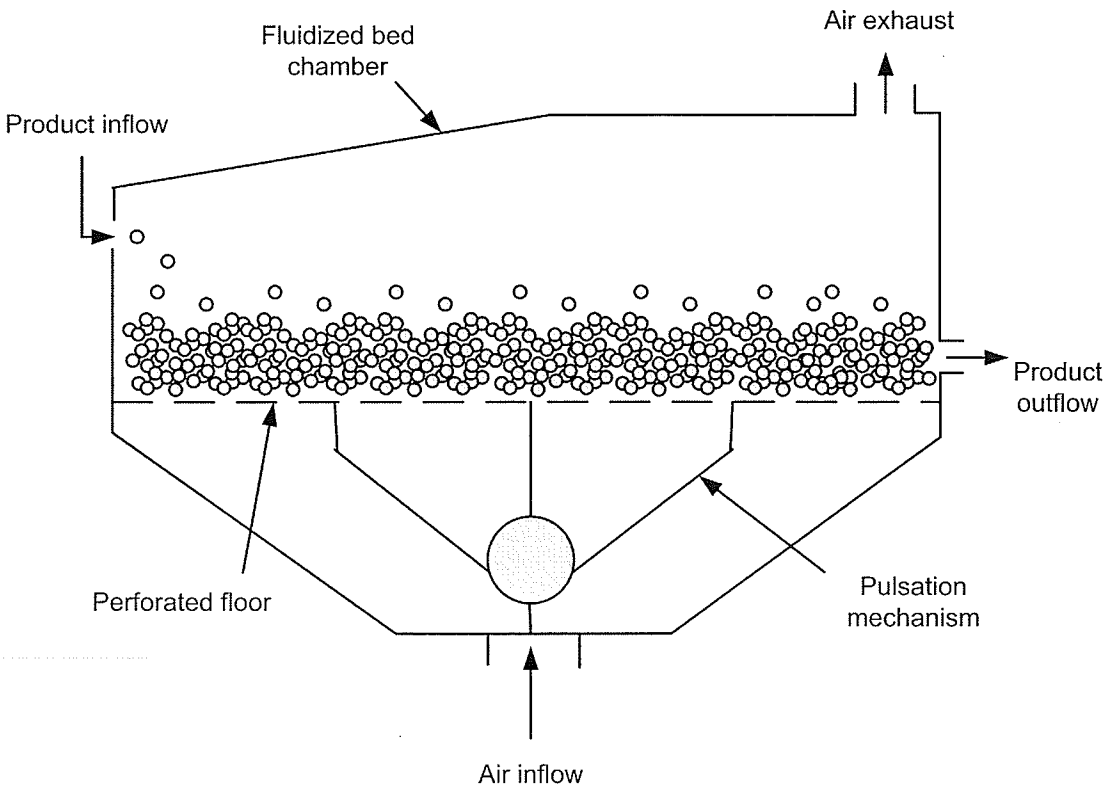


Figure 2.4 Schematic of product and air flow in a pulsed fluidized bed dryer.
(Adapted from Benali 2004. With permission.)

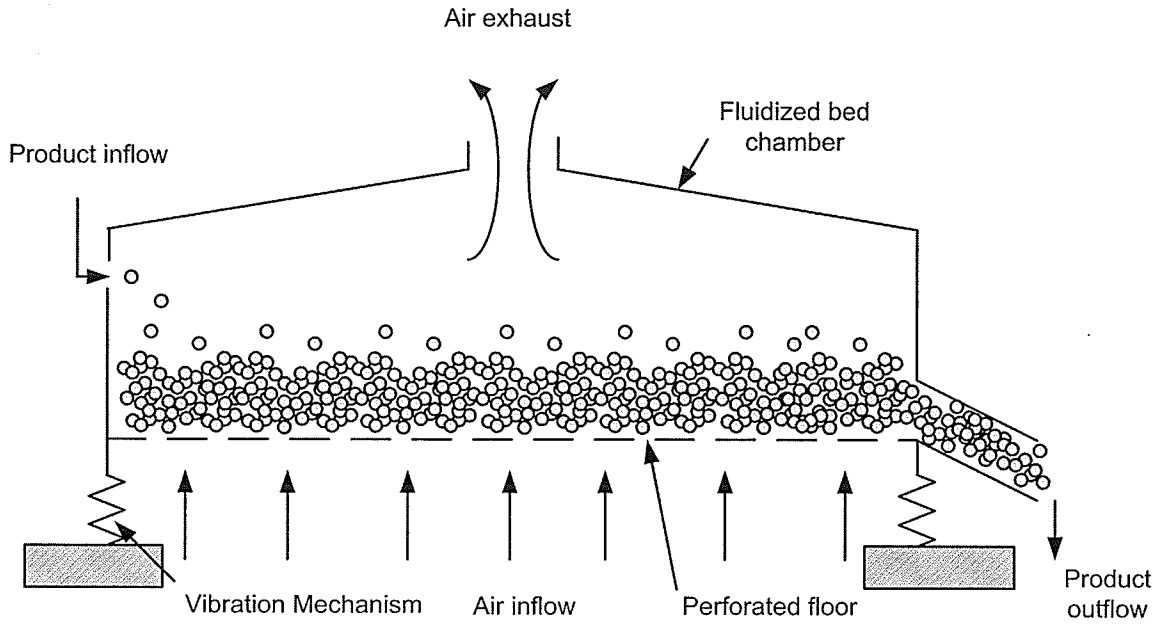


Figure 2.5 Schematic of product and air flow in a vibro-fluidized bed dryer. (Adapted by Benali 2004. With permission.)

Spouted bed dryers

A spouted bed dryer uses a high velocity jet for material agitation. Two distinct zones exist within a spouted bed dryer (Fig. 2.6): 1) a central jet with minimal product and high heat and mass transfer rates and 2) a moving annular bed with lower heat and mass transfer rates. Originally developed for grain drying in the 1960's (Mathur and Epstein 1974), the application has also proven advantageous for sticky or pasty products such as blueberries (Feng et al. 1999) and yeast (Grabowski et al. 1997). Grabowski et al. (1997) determined that pre-drying baker's yeast from 70 to 35% moisture content in a spouted bed dryer provides a more easily fluidized product for finish drying in a fluidized bed. Employing fluidized bed drying for the final drying stage allowed for an energy savings due to a 25% decrease in hot air demand. Currently, spouted bed drying systems

are used mainly for small volumes in batch mode due to difficulties involved with scale-up and continuous systems.

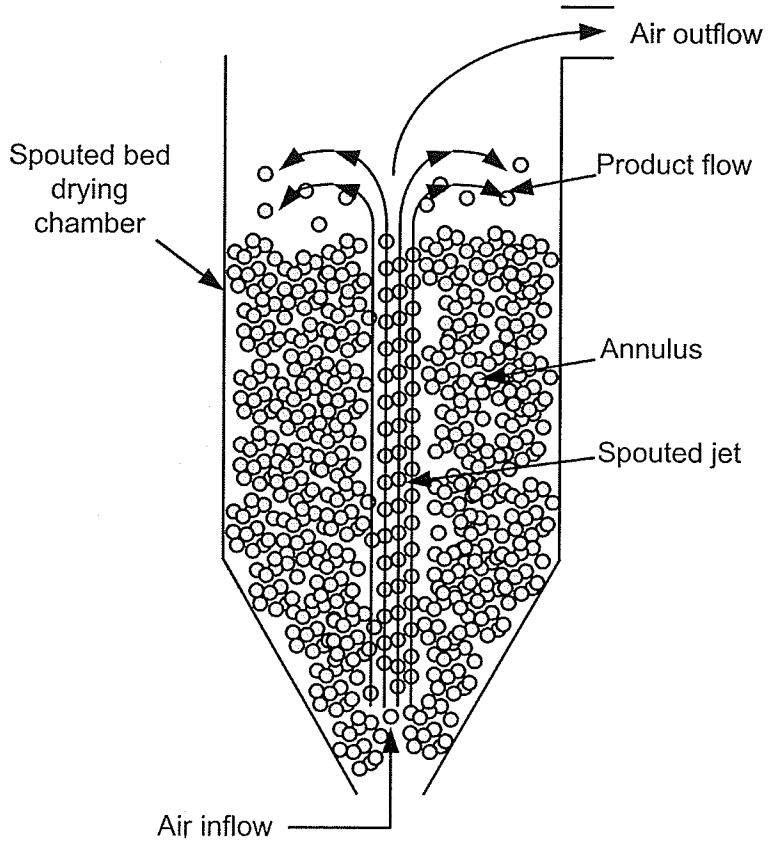


Figure 2.6 Schematic of product and air flow in a spouted bed dryer.
(Adapted from Mathur and Epstein 1974. With permission.)

Modifications to conventional systems include the microwave spouted bed (MWSB) (Feng et al. 1999) and draft-tube spouted fluidized bed (DTSFB) (Marmo 2007) systems. Blueberries dried in a MWSB system experienced a substantial reduction in drying time and improved product quality compared to those freeze, tray, and conventional spouted bed dried (Feng et al. 1999). The addition of fluidization to microwave drying, improved quality with the elimination of charred berries caused by uneven drying. In a conventional spouted bed dryer, dryer dimensions, product characteristics, bed height,

and airflow must be within an acceptable range to achieve and maintain stable spouting (Mathur and Epstein 1974). Marmo (2007) determined that the introduction of a draft tube in the centre of the spouted bed provided improved control of air flow, gas distribution, and solids motion pattern in the drying of olive pomace. The draft tube also provided the opportunity for continuous operation.

2.4.2 Fluidization with inert particles

Although fluidized and spouted bed dryers were originally developed for particulate material, adding uniformly sized inert particles enables the drying of liquids, suspensions, slurries, pulps, and pastes (Orsat and Raghavan 2007). Inert particles can be made from glass, ceramics, and plastics (e.g. Teflon®, polypropylene, and high density polyethylene) and generally range in size from 2 to 8 mm (Costa et al. 2001; Oliviera et al. 2006; Pallai et al. 2001; Zhao et al. 2004). Along with the addition of inert particles to the process, modifications to conventional fluidized bed and spouted bed systems in the form of spout-fluid bed (Costa et al. 2001; Marmo 2007), jet-spouted bed (Benali and Kudra 2002), revolving flow fluidized bed (Zhao et al. 2004), and spouted bed with swirling counter current streams (Kutsakova 2004) have been proposed. Addition of mechanical devices such as a mixer in a fluidized bed (Grbavcic et al. 2004) or an inner conveyor screw to a spouted bed dryer (Pallai et al. 2001) was made to further enhance the mixing and drying uniformity of the system.

The addition of inert particles to fluidized and spouted bed systems was developed as an alternative to spray, drum, and paddle dryers in the production of powders and flakes

and is applicable to the chemical, pharmaceutical, and food processing industries (Grbavcic et al. 2004). Oliviera et al. (2006) reported that compared to spray drying of 9% extractive solutions of Brazilian medicinal plants, spouted bed drying with inert particles resulted in higher concentrations of chemical markers and lower loss on drying values.

Drying of liquids, suspensions, and slurries

Depending on the consistency of the product, the drying process will follow different paths. The path involved with the drying of high moisture products such as liquids, suspensions, and slurries includes: 1) the delivery of the product into the chamber (Fig. 2.7a), 2) adherence of the product to the inert particle, 3) drying and subsequent cracking of the material on the inert particle, 4) peeling of the material caused by collision between inert particles, and 5) entrainment of the powder and flakes in the exhaust air with subsequent separation and collection by a cyclone and bag filter (Grbavcic et al. 2004). Delivery methods of the initial wet product can range from jet spray for a suspension to a screw feeder for a dense paste.

Drying of pulps and pastes

Products high in solids content such as pulps and pastes are fluidized together with the inert particles as shown in Fig. 2.7b and may not necessarily adhere to their surface. The inert particles, however, assist with fluidization of paste agglomerates, prevention of the formation of large agglomerates and caking, and eventual diminishment of dried agglomerates (Grbavcic et al. 2004).

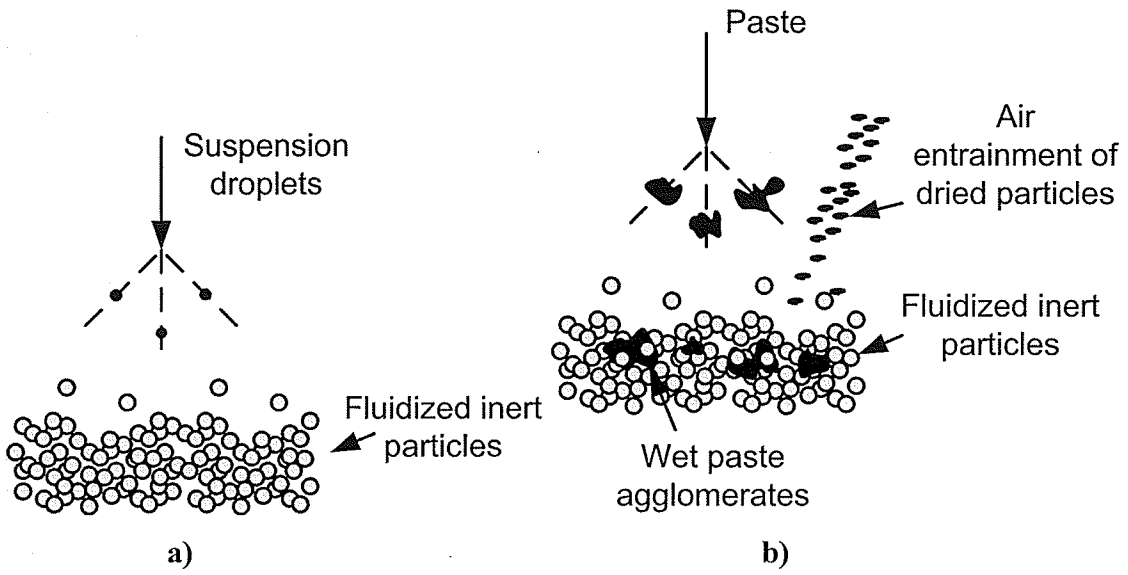


Figure 2.7 Schematic of the process of drying a) liquids, suspensions, and slurries and b) pulps and pastes in a fluidized bed dryer with inerts. (Adapted by Grbavcic et al. 2004. With permission.)

Air separation and collection methods of the dried powder or flakes are the same for the pastes as for the liquids, suspensions, and slurries. Although, there is no reported research on the drying of pomace or press cake with this technology, it may be suitable for sea buckthorn press cake, especially to prevent agglomeration of this sticky product.

2.4.3 Optimization of the drying system

Benali and Kudra (2002) suggested that treating drying as an integrated process is a suitable approach for dealing with slurries of high valued products (e.g. pharmaceuticals, nutraceuticals, and enzymes). This approach considers the dryer (includes ancillary equipment such as feeder, cyclone filter, and discharger) as the core of the process. The next layer is comprised of processes such as heat recovery, control systems, and automation. The final layer is composed of upstream (i.e. dewatering, stabilizing, preheating, and pre-forming) and downstream (i.e. granulation, cooling, screening, and

blending) processes. The ability to assess a drying system using this approach requires preliminary knowledge of the various aspects involved (i.e. product composition and characteristics, drying conditions, and equipment design and configuration). Modelling is a useful tool to assess the effects of different parameters as well as any interactions (positive or negative) that may affect the final product.

2.5 Mathematical modelling of the drying process

One of the approaches to drying modelling includes the analysis of an individual element or a grouping of smaller products (e.g. thin layer) that cannot feasibly be analysed in isolation (Pabis et al. 1998). The data determined through single element and thin layer analyses are especially useful for products that have not been thoroughly researched and for which drying data have not been published. It also provides an opportunity to determine drying behaviour for specific conditions. A review of basics appropriate to single element drying theory (Sections 2.5.1-2.5.3) precedes a discussion of approaches applicable to the analysis of drying of a thin layer of product on an inert sphere (Section 2.5.4).

2.5.1 Drying kinetics

The main method to evaluate the effectiveness with which a dryer removes moisture from a product is to determine the product's drying kinetics. Drying data used to determine drying kinetics for a product are usually expressed as total mass of the material as a function of time as shown in Fig. 2.8 (Kemp et al. 2001). This data can be used to determine moisture content (M) on a dry basis (db) (Eqn. 2.1):

$$M = \frac{m_i - m_f}{m_f} \tag{2.1}$$

where m_i = initial mass of sample and m_f = mass of dried sample.

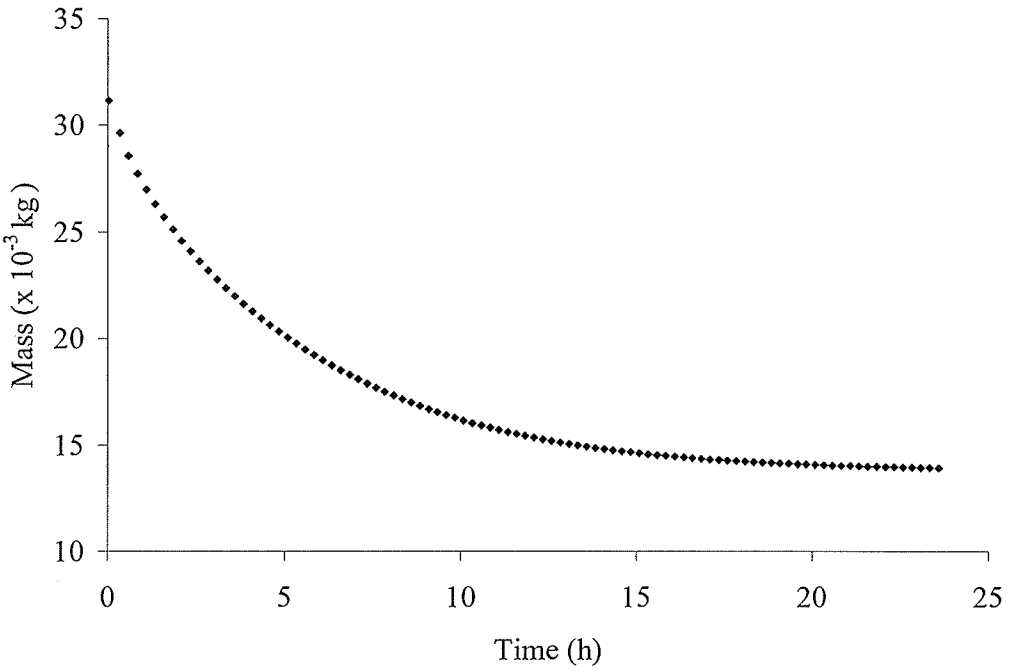


Figure 2.8 Change in mass with drying time.

Drying rate (DR) is based on the difference of moisture content (dM) with respect to a difference in time ($d\tau$) (Eqn. 2.2):

$$DR = -dM/d\tau \tag{2.2}$$

where dM = change in moisture content during time interval $d\tau$. The constant (zone I) and falling (zone II) rates that may occur during the drying process can be visualized with Fig. 2.9, a representation of plots that can be generated for different operating conditions, products, and dryer types (Pabis et al. 1998).

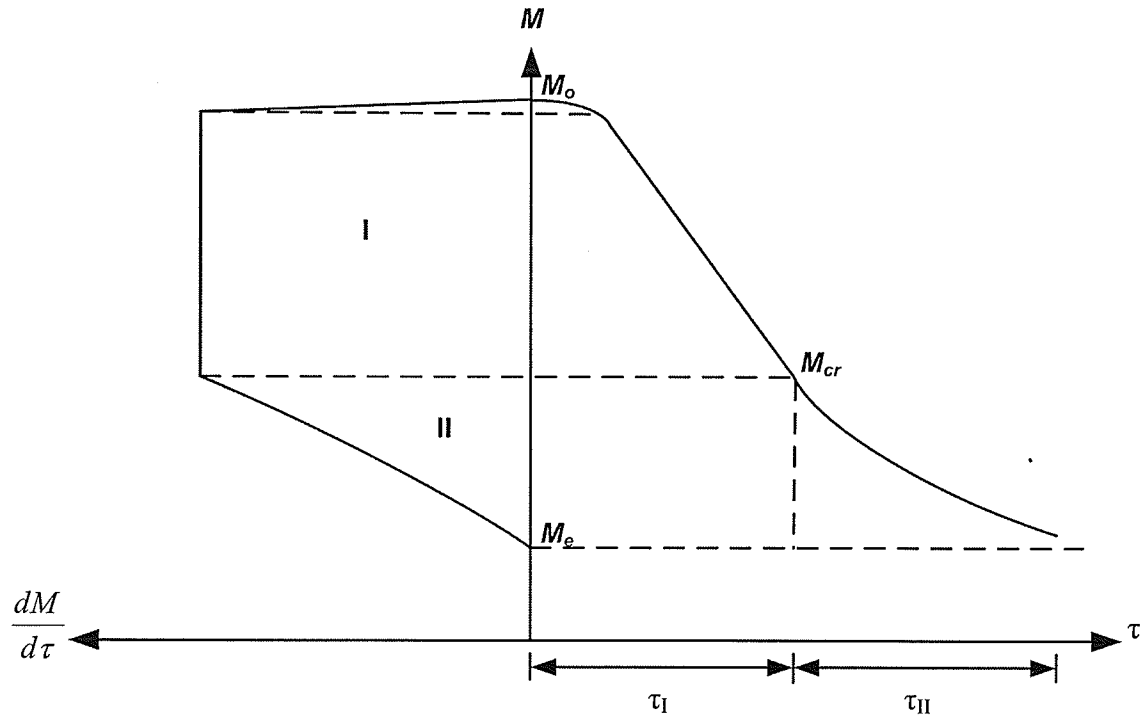


Figure 2.9 Drying rate, $\frac{dM}{d\tau}$, and moisture content, M, of a solid being dried during constant and falling-rate periods. Zone I and II represent the constant and falling rates of drying, respectively. M = moisture content; M_o = initial moisture content; M_{cr} = critical moisture content; M_e = equilibrium moisture content; τ = time; τ_I = time duration for onset of drying to end of constant rate period, and τ_{II} = time duration for falling rate period. (Adapted from Pabis et al. 1998. With permission.)

Constant rate of drying (Zone I)

The initial moisture content (M_o) of a product is greater than the critical moisture content (M_{cr}) (Figs. 2.9 and 2.10). After a short period of heating of the material from its initial temperature (t_o) to the wet-bulb temperature ($t_{w\text{-}b}$), the constant rate of drying begins and represents a constant rate of moisture removal from the surface of the product (i.e. water is supplied to the surface as fast as it evaporates). Within constant rate of drying conditions, body surface temp (t_s) is constant and remains equal to $t_{w\text{-}b}$ (Fig. 2.10).

If heat losses are negligible, then all heat delivered to the solid being dried is used for water vaporization (Pabis et al. 1998).

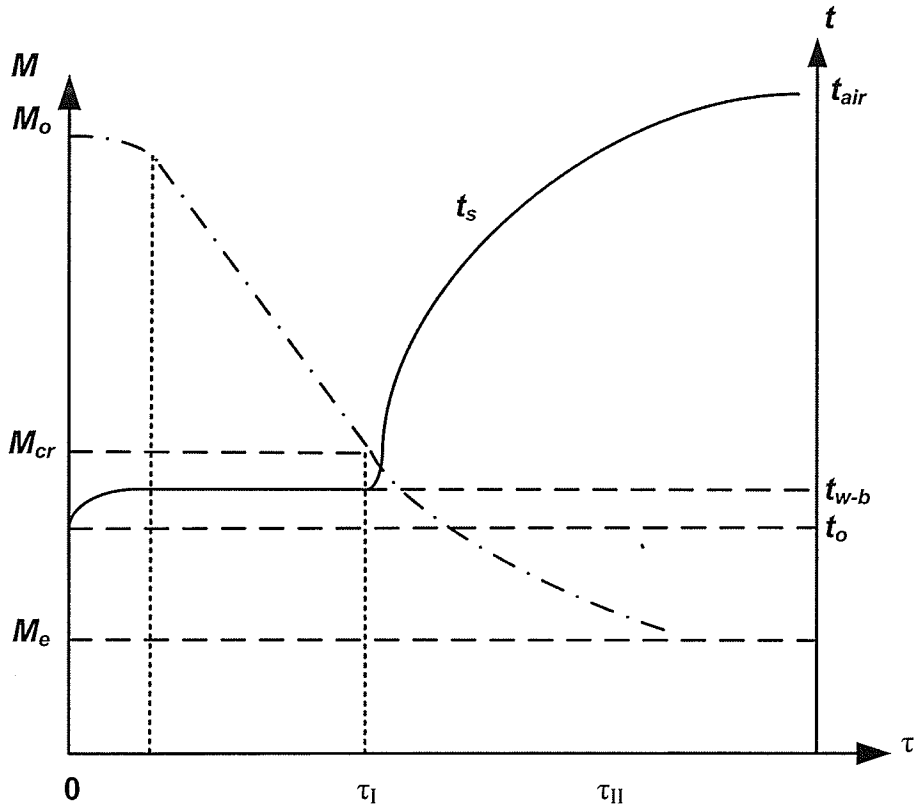


Figure 2.10 Moisture content, M, and surface temperature, t_s, of a solid being dried during constant and falling-rate periods. M = moisture content; M_o = initial moisture content; M_{cr} = critical moisture content; M_e = equilibrium moisture content; τ = time; τ_I = time duration for onset of drying to end of constant rate period; τ_{II} = time duration for falling rate period; t = temperature; t_{air} = drying air temperature; t_o = initial body temperature; t_s = body surface temperature; t_{w-b} = wet-bulb temperature of the drying air. (Adapted from Pabis et al. 1998. With permission.)

Falling rate of drying (Zone II)

If M_o is less than M_{cr}, DR decreases with time of drying until equilibrium moisture content (M_e) is reached (Fig. 2.9). The decrease in rate of drying with time is because of internal resistance to water transfer within the body being greater than the external

resistance to water transfer from the body. At the onset of the falling rate period, t_s continuously increases, and eventually approaches t_{air}, the temperature of the drying medium (e.g. air) (Fig. 2.10). The main mechanism of moisture loss in grain and other commodities such as vegetables and fruit (includes olive cake and pomace), is due to diffusion and occurs during the falling rate period(s) (Jayas et al. 1991; Ramaswamy and Nsonzi 1998). The falling rate period will only be considered for the remainder of this review.

2.5.2 Basic modes of heat transfer

The heat energy (q) (Eqn. 2.3) stored by a product is determined by its mass (m), specific heat (Cp), and temperature (Singh and Heldman 2001):

$$q = m\, Cp\, \Delta t \tag{2.3}$$

where Δt = temperature difference for a specific time period. Heat transfer from or to a product can occur via three modes: convection, conduction, and radiation. Radiation is the mode through which energy, transmitted by electromagnetic waves, is released and converted to heat once it impacts and is absorbed by a contact surface (Singh and Heldman 2001). Radiation is generally assumed to be negligible as compared to convective and conductive heat transfer in the process of forced air convective drying.

Convection

Convection or convective heat (q_{conv}) transfer occurs with a flow of a medium, most commonly air, over a body of different temperature (Mujumdar 2006). Other media can be used including inert gases (e.g. N_2), direct combustion gases, or superheated steam.

The rate of convective heat transfer $\left(\frac{dq_{conv}}{d\tau}\right)$ as shown by Eqn. 2.4, is based on three main characteristics of the drying system: 1) difference between t_s and t_{air}, 2) heat transfer coefficient of the medium (h), and 3) surface area (A) of the body exposed to the medium (Singh and Heldman 2001).

$$\frac{dq_{conv}}{d\tau} = h\,A\left(t_{air} - t_s\right) \tag{2.4}$$

Conduction

Conduction dryers, otherwise referred to as indirect dryers employ the transfer of heat (q_{cond}) from a heated surface in contact with a body at lower temperature (Mujumdar 2006). The rate of conductive heat transfer $\left(\frac{dq_{cond}}{d\tau}\right)$ (Eqn. 2.5) is based on characteristics of the two surfaces in contact: the difference in temperature with respect to distance in the direction of heat flow $\left(\frac{dt}{dx}\right)$, the thermal conductivity of the material (λ), and A:

$$\frac{dq_{cond}}{d\tau} = \lambda\,A\frac{dt}{dx} \tag{2.5}$$

Steady state heat transfer

Regardless of the mode of heat transfer, it is important to understand the conditions that are present within the process (e.g. steady or unsteady state) (Singh and Heldman 2001). Steady state conditions are those for which time has no influence on the

temperature distribution within a product. However, a temperature gradient may exist between different locations within the product itself. Although, not common in practice, steady state conditions are fairly simple to analyse mathematically using Eqns. 2.3-2.5. If appropriate, steady state conditions may be assumed to determine useful information for specific applications.

Unsteady state heat transfer

Unsteady state or transient heat transfer involves a temperature change with respect to both time and location (Singh and Heldman 2001). This type of heat transfer tends to be the most dominant one in practical drying situations. Based on Fourier's law (Eqn. 2.5), the partial differential equation Eqn. 2.6, is the governing equation for a one-dimensional unsteady state case:

$$\frac{\partial t}{\partial \tau} = \frac{\lambda}{\rho Cp\, r^z} \frac{\partial}{\partial r}\left(r^z \frac{\partial t}{\partial r} \right) \tag{2.6}$$

where ρ = density of the body, r = distance from a centre location in the body, and z = geometrical shape coefficient (0 for a slab, 1 for a cylinder, and 2 for a sphere). If the rate of heat transfer at the surface is due to convection then Eqn. 2.7 is valid:

$$\lambda \left. \frac{\partial t}{\partial r} \right|_{r=R} = h\,(t_{air} - t_s) \tag{2.7}$$

The solution for Eqn. 2.6 in conjunction with Eqn. 2.7 involves the use of advanced mathematics and due to complexity, is possible only for objects of simplified geometry as implied by the coefficient z.

External versus internal resistance. In a transient heat transfer analysis, the relative importance of heat transfer at the surface and interior needs to be considered. Once a solid is immersed in a fluid, the heat transfer from the fluid to the centre of the solid will be confronted by two resistances: the convective resistance in the fluid layer surrounding the solid and conductive resistance inside the solid (Singh and Heldman 2001). The ratio of the internal resistance to heat transfer in the solid to the external resistance to heat transfer in the fluid is defined as the Biot number (Bi) (Eqn. 2.8):

$$\mathrm{Bi} = \frac{h\, d_c}{\lambda} \tag{2.8}$$

where d_c = characteristic dimension.

Lumped system analysis, Bi < 0.1. For a Biot number < 0.1, there is negligible internal resistance to heat transfer; the temperature is nearly uniform (i.e. lumped) within the product (Singh and Heldman 2001). The heat transfer is relatively instantaneous therefore no temperature gradients develop. Beginning with a heat balance as shown by Eqn. 2.9, separation of the variables followed by integration, results in the mathematical expression Eqn. 2.10:

$$\rho\, Cp\, V \frac{dt}{d\tau} = h\, A \left(t_{air} - t\right) \tag{2.9}$$

$$\frac{t_{air} - t}{t_{air} - t_i} = \exp\left[-\left(\frac{h\, A}{\rho\, Cp\, V}\right)\tau\right] \tag{2.10}$$

where t_i = the temperature at the beginning of the drying period; V = volume of the body, and τ = elapsed time of drying.

Finite internal and surface resistance to heat transfer, 0.1 < Bi < 40. For a Biot number between 0.1 and 40, finite internal and external resistance to heat transfer will occur (Singh and Heldman 2001). The solution for this situation is an infinite series containing trigonometric and transcendental functions, as provided in Eqns. 2.11 and 2.12 for a sphere and infinite slab;

Sphere:

$$t = t_{air} + (t_{air} - t_i)\,\frac{2}{\pi}\left(\frac{d_c}{r}\right)\sum_{n=1}^{\infty}\frac{(-1)^{n+1}}{n}\exp\left(\frac{-n^2\,\pi^2\,\alpha\,\tau}{d_c^2}\right)\sin\left(\frac{n\,\pi\,r}{d_c}\right) \qquad (2.11)$$

where α = thermal diffusivity of a body;

Infinite plate:

$$t = t_{air} + (t_i - t_{air})\sum_{n=0}^{\infty}\frac{\left[2(-1)^n\right]\exp\left(\frac{-\lambda_n^2\,\alpha\,\tau}{d_c^2}\right)}{\lambda_n}\cos\left(\frac{\lambda_n\,x}{d_c}\right) \qquad (2.12)$$

where x = variable distance in the x direction from the centre and λ_n = eigenvalue roots. The solutions can be programmed into a spreadsheet for use on a computer or can be solved using temperature time charts that have been developed specifically for geometric shapes [infinite slab, infinite cylinder (solution not provided in this review), and sphere] by Heisler (1944) as cited by Singh and Heldman (2001). The charts are based on three terms: $\frac{t_{air} - t}{t_{air} - t_i}$; $\frac{\lambda}{h\,d_c}$, and $\mathrm{Fo} = \frac{\alpha\,\tau}{d_c^2}$, where Fo is the Fourier number.

Negligible surface resistance to heat transfer, Bi > 40. At a Biot number > 40, there is negligible surface resistance to heat transfer (Singh and Heldman 2001). As with the previous, Heisler charts can be used for the solution, however, the value $\frac{\lambda}{h\,d_c} = 0$.

2.5.3 Analysis for falling rate of drying (diffusion models)

Drying during the falling rate period(s) is mainly controlled by the mechanism of moisture (e.g. liquid or vapour) diffusion (Akpinar 2006; Pabis et al. 1989). Fick's second law in terms of moisture content as provided in Eqn. 2.13 is employed to describe the drying of many food products:

$$\frac{\partial M}{\partial \tau} = \nabla \left(D_m \nabla M \right) \tag{2.13}$$

where, D_m = mass diffusion coefficient (dependent on moisture content or temperature). In a homogeneous, isotropic material in which the resistance to moisture flow is uniformly distributed, D_m is assumed to be constant and volume shrinkage is negligible, Fick's second law can be derived as Eqn. 2.14:

$$\frac{\partial M}{\partial \tau} = D_m \nabla^2 M \tag{2.14}$$

where $\nabla^2 M$ is the Laplacian operator. The Laplacian operators ($\nabla^2 M$) for simple geometries such as an infinite plate and sphere are equivalent to $\frac{\partial^2 M}{\partial x^2}$ and $\frac{\partial^2 M}{\partial r^2} + \frac{2}{r}\frac{\partial M}{\partial r}$, respectively (Pabis et al. 1998).

Analytical solutions

Analytical solutions for the falling rate of drying considering mainly internal resistance to moisture transfer have been developed (Akpinar 2006). Individual food products, slices, or thin layers of particles can be represented geometrically (e.g. infinite plate, sphere, cylinder, parallelpiped). Crank (1975) developed solutions in terms of

moisture ratio (*MR*) (Eqn. 2.15) for each of these shapes and those for an infinite plate and sphere (with boundary conditions of the first kind) are provided by Eqns. 2.16 and 2.17, respectively (Pabis et al. 1998).

$$MR(\tau) = \frac{M(\tau) - M_e}{M_0 - M_e} \tag{2.15}$$

$$MR(\tau) = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left(\frac{-\pi^2 (2n+1)^2 D_m \tau}{4s^2} \right) \tag{2.16}$$

$$MR(\tau) = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp\left(\frac{-n^2 D_m \pi^2 \tau}{R^2} \right) \tag{2.17}$$

The solution for the infinite plate (i.e. thickness is very small compared to its length and width) is valid for the following conditions: plate thickness of $2s$; uniformly distributed M_o; the plate is dried from both sides by a constant airflow at t_{air} and relative humidity (*RH*); properties of the plate are constant, and moisture movement is perpendicular to the surface. The solution for the sphere is valid for the same conditions as the infinite plate except the sphere is of radius (R) and the moisture flow is along its radius.

Simplifications of the infinite exponential series represented by Eqns. 2.16 and 2.17, include using only the first term or the first several terms and are suitable for long drying times (Jayas et al. 1999). The application of the first term simplification for an infinite plate (Eqn. 2.18) was reported for thin layer drying modelling of olive press cake or sludge (Akgun and Doymaz 2005; Celma et al. 2007; Doymaz et al. 2004), flax fibre (Ghazanfari et al. 2006a; 2006b), and organic apple slices (Sacilik and Elicin 2006). The

similarly reduced equation for a sphere (Eqn. 2.19) was reported for grains such as wheat (Becker and Sallans 1955; Hustrulid 1963; Sinicio et al. 1995).

By plotting the natural logarithm of *MR* versus time for the infinite plate, the intercept and slope of the straight line are represented by $\ln\left(\frac{8}{\pi^2}\right)$ and $-\left(\frac{\pi^2 D_m}{4 s^2}\right)$, respectively (Akgun and Doymaz 2005; Celma et al. 2007; Doymaz et al. 2004). This method is commonly applied to determine D_m, which can subsequently be used in the determination of activation energy for a specific product.

$$MR(\tau)=\frac{8}{\pi^2}\exp\left(\frac{-\pi^2 D_m \tau}{4 s^2}\right) \tag{2.18}$$

$$MR(\tau)=\frac{6}{\pi^2}\exp\left(\frac{-\pi^2 D_m \tau}{R^2}\right) \tag{2.19}$$

The major assumption of both the analytical solutions and their simplified versions is that the object is homogeneous in characteristics including moisture content (Pabis et al. 1998). This is not true for the situation in which a thin layer of one material containing moisture is dried on a particle of an inert material which contains no moisture. Therefore, neither one of these is directly suitable for the analysis of drying a thin layer on a spherical particle.

Semi-empirical and empirical solutions

Many semi-empirical and empirical models have been developed for specific commodities and drying systems. Semi-empirical and empirical solutions consider the external resistance to moisture transfer between products and air (Akgun and Doymaz 2005). These models offer a compromise between theory and ease of use. The equations and coefficients are generally only valid for the conditions (i.e. temperature, relative humidity, or air velocity) and moisture content range for which they were developed, require reduced evaluation times compared to theoretical models and do not need assumptions of geometry, mass diffusion coefficient, and conductivity.

Thin layer drying models. Olive pomace, press cake, and bagasse are products that are non-homogeneous and consist of pieces of pit and pulp of different size, shape, and ratios. To determine the model that provides good representation and repeatability of drying curves in the thin layer drying analysis of these products, Agkun and Doymaz (2005), Celma et al. (2007), and Doymaz et al. (2004) analysed several models. Models included the Lewis (Eqn. 2.20) (Lewis 1921; Bruce 1985), Page (Eqn. 2.21) (Page 1949), modified Page (Eqn. 2.22) (Overhults et al. 1973), Henderson and Pabis (Eqn. 2.23) (Henderson and Pabis 1961), modified Henderson and Pabis (Eqn. 2.24) (Karathanos 1999), logarithmic (Eqn. 2.25) (Yagcioglu et al. 1999; Yaldiz et al. 2001), two-term (Eqn. 2.26) (Henderson 1974), two-term exponential (Eqn. 2.27) (Sharaf-Elden et al. 1980), the diffusion approach (Eqn. 2.28) (Kassem 1998), Verma et al. (Eqn. 2.29) (Verma et al. 1985), Midilli-Kucuk (Eqn. 2.30) (Midilli et al. 2002), and Wang and Singh (Eqn. 2.31) (Wang and Singh 1978):

$$MR(\tau) = \exp(-k\,\tau) \tag{2.20}$$

$$MR(\tau) = \exp(-k\,\tau^{n}) \tag{2.21}$$

$$MR(\tau) = \exp[-(k\,\tau)^{n}] \tag{2.22}$$

$$MR(\tau) = a\exp(-k\,\tau) \tag{2.23}$$

$$MR(\tau) = a\exp(-k\,\tau) + b\exp(-g\,\tau) + c\exp(-f\,\tau) \tag{2.24}$$

$$MR(\tau) = a\exp(-k\,\tau) + c \tag{2.25}$$

$$MR(\tau) = a\exp(-k_{o}\,\tau) + b\exp(-k_{1}\,\tau) \tag{2.26}$$

$$MR(\tau) = a\exp(-k\,\tau) + (1-a)\exp(-k\,a\,\tau) \tag{2.27}$$

$$MR(\tau) = a\exp(-k\,\tau) + (1-a)\exp(-k\,b\,\tau) \tag{2.28}$$

$$MR(\tau) = a\exp(-k\,\tau) + (1-a)\exp(-g\,\tau) \tag{2.29}$$

$$MR(\tau) = a\exp(-k\,\tau^{n}) + b\,\tau \tag{2.30}$$

$$MR(\tau) = 1 + a\,\tau + b\,\tau^{2} \tag{2.31}$$

The coefficients a, b, c, f, g, k, k_0, k_1, and n for Eqns. 2.20 to 2.31 are calculated from experimental data. While the majority of the models are based on modification of the simplified analytical solutions to the Fick's Law of diffusion, others have been based on Newton's Law of cooling or on the experimental relationship between moisture content and time. The Wang and Singh model is representative of the latter method, a purely empirical derivation (Akgun and Doymaz 2005).

Assuming that there is a similarity between the cooling and drying of an element, a model analogous to Newton's law of cooling can be used to determine drying rate (Eqn.

2.32) (Lewis 1921). Through the integration from initial time (τ_o) to τ and M_o to M, the Lewis model (Eqn. 2.20) was developed. Modifications to Lewis' model resulted in the development of Page's and the modified Page's models, as discussed by Jayas et al. (1991).

$$\frac{dM}{d\tau} = -k\left(M - M_e\right) \tag{2.32}$$

Review of best fit for thin layer drying models. Akgun and Doymaz (2005) investigated seven (Lewis, Page, modified Page, Henderson and Pabis, logarithmic, Wang and Singh, and diffusion) of the models for olive cake dried at temperatures between 50 and 110°C. All models provided a good fit as represented by the coefficient of determination (r^2) > 0.97, however, a certain amount of under- and over-prediction occurred at different stages of drying. The logarithmic model provided the best overall fit at all temperatures [i.e. high r^2, low mean root square error (*RMSE*), and low residuals].

Celma et al. (2007) validated all the models represented by Eqns. 2.20 to 2.31, at temperatures of 20, 40, and 80°C, for olive sludge with M_o > 65%. Similar to Akgun and Doymaz (2005) all models provided a good fit, however, the best fit was achieved by Midilli (r^2 = 0.9954 to 0.9985, *RMSE* = 0.00815 to 0.01454, and residuals between -0.00397 to 0.00005). The logarithmic model ranked third best for all temperatures, whereas the Lewis and Page models provided better fits with an increase in temperature. Doymaz et al. (2004) determined that the Page model provided a better fit than the Lewis model for olive cake with M_o = 44.78% wb dried at temperatures from 80 to 110°C. The

Page model provided good prediction of moisture ratio (r^2 = 0.996 to 0.998, *RMSE* = 0.00844 to 0.01457) for a thin layer of flax fiber dried at temperatures of 30 to 100°C (Ghazanfari et al. 2006b).

Kudra and Efremov (2003) developed a semi-empirical approach to determine the drying kinetics of fluidized particulate materials as provided in Eqn. 2.33:

$$MR(\tau) = \frac{1}{1+(\tau/\sigma)^m} \tag{2.33}$$

where σ = characteristic time that is constant for given process conditions and m = dimensionless factor which reflects the effect of the convective airflow. For wheat dried in a spouted bed at 70°C, air velocity of 14.7 m/s, M_o = 0.339, and M_e = 0.05, σ and m were found to be 36 min and 1.25, respectively. This method was determined to be applicable to materials with predominantly internal resistance to mass transfer and dryers with active hydrodynamic regimes.

2.5.4 Applications for thin layer drying on an inert particle

The analysis of thin layer drying on an inert particle has been conducted using two main approaches: theoretical and simplified. In this application, "theoretical" represents solutions based on basic drying theory that are general in nature. The "simplified" approach incorporates theoretical drying basics with semi-empirical or empirical models applicable to actual drying situations or specific products. The latter is believed to be more suitable for the prediction of drying data in pilot and industrial type dryers (Kemp and Oakley 2002). It is important, however, that work continues in both areas of modelling to provide opportunity for more precise models. The selection of the

modelling approach depends on knowledge of product characteristics, the complexity of the drying configuration, and the resources available.

Theoretical

Mikhailov and Özişik (1984) presented a unified approach using partial differential equations based on Fourier's and Fick's laws, to solve heat and mass diffusion problems. The solution developed for a composite sphere with generalized boundary and initial conditions was meant to be applicable to many cases without being limited to specific products. It allows for the determination of temperature with respect to distance along the radius and time. Due to the complexity of the solution, its derivation and presentation is beyond the scope of this review.

The validity of solutions based on fundamental drying theories cannot be proven until experimentally validated under a wide range of conditions on a small to large scale (Kemp and Oakley 2002). Konovalov et al. (2003) reported on the development, solution, and validation of a unified mathematical model incorporating a semi-empirical temperature-moisture function based on convective drying of a thin layer. While this model did provide accurate results, sophisticated mathematical programming is required as with the solution proposed by Mikhailov and Özişik (1984). The validation accuracy of the model is discussed in Section 5.3.3.

Simplified

Guu (2003) presented a thorough discussion on drying process energy balances. In drying processes, both sensible and latent heat transfer are involved with moisture removal. Sensible heat is the heat needed to change the temperature and is normally related to the specific heat of a substance. Latent heat is the heat needed to change the phase of a substance (e.g. liquid to vapour). The proportion of latent heat is significantly larger than that of sensible heat and is responsible for most of the energy costs for drying.

Leontieva et al. (2002) analysed the heat and mass transfer for an aqueous solution of finely dispersed solid sprayed over the surface of an inert ceramic sphere. The mass balances proposed take into consideration the dynamics that would occur within a dryer, including the change in mass of product (e.g. water and solids) due to drying as well as product exchange. The heat balances included the heat energy (i.e. convective) transferred from the drying air and the portions of which required to raise the temperature of the inert particle and product (i.e. sensible) and to evaporate the moisture from the product (i.e. latent). While their proposed model included a series of equations to be solved simultaneously, it was based on a simplified, gradientless approach to transport phenomena. The equations representing change in product temperature and inert particle temperature are provided by Eqns. 2.34-2.35:

$$\frac{dt}{d\tau} = \frac{1}{Cp\ \rho s}\left[h\left(t_{air} - t\right) - \frac{\lambda\left(t - t_{ip}\right)}{s} - \frac{dm_{H_2O}}{d\tau}\frac{h_{fg}}{A_{ip}} \right] \tag{2.34}$$

where A_{ip} = surface area of the inert particle; dm_{H_2O} = difference in mass of water; h_{fg} = latent heat of vaporization; s = thickness of product layer, and t_{ip} = temperature of the inert particle;

$$\frac{dt_{ip}}{d\tau} = \frac{\lambda\, d_{ip}}{Cp_{ip}\, \rho_{ip}\, V_{ip}}\left(t - t_{ip}\right) \tag{2.35}$$

where dt_{ip}= difference in temperature of the inert particle; d_{ip} = diameter of the inert particle; Cp_{ip} = specific heat capacity of the inert particle; ρ_{ip} = density of the inert particle, and V_{ip}= volume of the inert particle.

Validation of the model was achieved at two levels, based on: 1) heat and mass transfer kinetics on a single inert particle and 2) parameters of dry material and air exhaust from an industrial dryer (Leontieva et al. 2002). The validation accuracy of the model is discussed in Section 5.3.3. The authors recommended that improvements to the model should include provision for correction factors accounting for interaction of inert particles in the spouted bed and the effect of the mixing regime.

As part of a model for the drying process in a spout-fluid bed, Costa et al. (2001), developed a mass and energy balance for inert particles with a suspension layer. Similar to Leontieva et al. (2002), the balance equation represents a dynamic system (i.e. solids and air exchange) as would occur within a drying situation. Due to the small size of the inert particles, temperature gradients within the particles were neglected. Although this approach has been applied to individual particles or sections of the dryer, it is also valid for an entire drying system (Costa et al. 2001; Leontieva et al. 2002).

Grbavcic et al. (2004) used an overall energy balance to predict the performance of drying of slurries in a fluidized bed of inert particles. The energy balance is based on ingoing and outgoing air, product, and moisture as well as overall losses in the system. The validation accuracy of the model is discussed in Section 5.3.3. Divergences between the predicted and experimental were attributed to possible unaccounted losses in the system.

2.5.5 Suggestions for future research on drying with inert particles

The development and testing of thin layer models for the application of drying granular products in conventional systems have been thoroughly investigated. More knowledge, however, is required for the application of models to fluidized and spouted bed drying systems. These systems are more complicated due to the dynamics of the fluidization of the particles. The inflow and outflow stages of the particles also require consideration. The addition of inert particles adds further complexity due to the many stages involved: delivery of the product; coating of the inert particle; drying of the material on the inert particle; cracking of the material, and finally entrainment and collection of dried particles in the outgoing air. Since these alternative drying methods are being pursued for products such as fruits, vegetables, and other specialty plant products of interest to the functional food and nutraceutical industries, it is also important to understand the drying kinetics and characteristics of these products.

3 INFLUENCE OF HARVEST TIME ON QUALITY OF OIL-BASED COMPOUNDS IN SEA BUCKTHORN (*H. rhamnoides* L. ssp. *sinensis*) SEED AND FRUIT

3.1 Experimental determination of characteristics and bioactive compounds

3.1.1 Harvest and post harvest handling

Sea buckthorn berries ssp. *sinensis* were manually harvested from five year old shrubs at St. Claude, Manitoba, Canada, during the 2003-2004 harvest year. Undamaged berries from 20 shrubs were collected in 200 g lots to form a representative sample from the orchard. The three harvest periods included early maturity (September), maturity (early November), and post-maturity (January). The first severe frost (-24.2°C) occurring on November 7, was selected as the point at which berry development may have ceased.

Bagged berries were frozen in a single layer in -40°C freezer storage, within 4 h of being harvested. Once completely frozen (i.e. a minimum of 24 h in freezer storage) the berries were mixed to form one homogeneous pool for each harvest period. The berries were bagged and kept frozen until required. Berries were thawed at 4°C for 5 h prior to testing. The literature reported that quick freezing and controlled thawing resulted in less ice crystal formation and physical change to berry structure (Feng et al. 1999; Reid 1997).

3.1.2 Temperature monitoring

The temperature was recorded at 15 min intervals from September 4, 2003 to January 25, 2004, using a temperature data logger (model 01-0192, ACR JR-1000 Series, ACR Systems, Inc., Surrey, Canada). The data logger was suspended in an instrument shelter located in a row of shrubs in the orchard.

3.1.3 Berry and seed sizing

Berry sizing was represented by the mass of a randomly selected batch of 100 thawed berries (g% berries), in triplicate (Tang and Tigerstedt 2001). The seed size was measured as the mass of 100 seeds (g% seeds) after air drying at room temperature (25°C) for 2 weeks, in triplicate.

3.1.4 Moisture content

Moisture content in mass percentage [% mass/mass (w/w)] on a wet basis (wb) was determined by the standard vacuum oven method according to the American Organization of Analytical Chemists (AOAC) official method 920.151 (AOAC 2002a).

3.1.5 Seed content in berries

The seed content was the mass of seeds in a sample of berries [% (w/w) wb] (Yang et al. 2001). This evaluation was performed on nine 56 g fruit samples per harvest time.

3.1.6 Colour factors

Colour measurements were conducted on berries from each harvest time, in triplicate, with a Minolta Chroma Meter (model CR-410, Minolta Co. Ltd., Osaka, Japan). The Commission Internationale d'Eclairage (CIE) laboratory colour system was followed (Francis 2003). The CIELab scale measured the degrees of lightness (L^*) and hue [red or green (+/-a^*) and yellow or blue (+/-b^*)] in a sample. The unit was calibrated for white on a scale in which black is represented by $L^* = 0$ and white by $L^* = 100$.

3.1.7 Sample preparation for bioactive compound determination

The seeds, pulp, peel, and juice each contained different levels of bioactive compounds. To simplify the process, the fresh berries were separated into two fractions: seed and fruit (pulp, peel, and juice), according to AOAC method 920.149 (AOAC 2002a). This preparation was performed in triplicate with three sub-samples to yield nine 15 g fruit and three 10 g ground seed fraction samples per harvest period. A triple extraction based on the Folch method (Folch et al. 1957) using a chloroform:methanol solution [1:2 volume:volume (v:v)] was employed for the fruit and seed fractions. Extracted oil samples were diluted and stored in hexane, yielding concentrations of 100 and 500 mg/mL hexane for the fruit and seed fractions, respectively.

Total carotenoids

The determination of total carotenoids was based on a method proposed by Gao et al. (2000). Solutions of fruit and seed fraction oil in hexane, 0.5 and 10 mg/mL hexane,

respectively, were prepared to achieve an absorbance within 0.2 and 0.8. Total carotenoids were measured at 460 nm using a Spectronic spectrophotometer (model 3000 ARRAY, Milton Roy, Ivyland, PA). Quantification of amounts of carotenoids was based on calibration with a β-carotene standard (type II synthetic, Sigma-Aldrich Canada Ltd., Oakville, Canada). Total carotenoids were expressed in mg/100 g oil, β-carotene equivalents.

Fatty acids

Fatty acid composition determination was conducted through analysis of fatty acid methyl esters (FAMEs) prepared according to the American Oil Chemists Society method Ce 1-62 (AOCS 2000). FAMEs were analysed with a gas chromatograph (GC) (model 17AAF, Shimadzu Corporation, Kyoto, Japan) equipped with a programmed split/splitless injector and flame ionization detector (FID). A fused silica capillary column DB-23 (L = 30 m, *i.d.* = 0.25 mm, d_f = 0.25 μm; J & W Scientific, Folsom, CA) was used. The linear velocity of the carrier gas, hydrogen, was 0.5 m/s, with a split valve ratio 1:80. The column temperature program included maintaining 155°C for 2 min, increasing at a rate of 2°C/min to 215°C then holding for 1 min. FAMEs were identified by comparison with retention data of a standard mixture 461 (NuChek Prep, Elysian, MN). The fatty acid composition was expressed as % (w/w) of the total fatty acids.

Tocopherols and tocotrienols

Tocopherol and tocotrienol levels were determined following International Standards Organization procedure 9936 (ISO 2004). These compounds were analysed using

normal-phase high performance liquid chromatography with a Shimadzu 10AD apparatus, a Shimadzu SIL-10A auto injector, and RF-10AXL fluorescence detector (Shimadzu Corporation, Kyoto, Japan). The excitation and emission wavelengths were set at 290 and 335 nm, respectively. A 5 μm silica column (L = 250 mm, *i.d.* = 3.2 mm, d_f = 5 μm; Phenomenex, Torrance, CA) was used for separation with 5% methyl *tert*-butyl ether (MTBE) in hexane as the mobile phase. The oil-hexane solution injection volume was 10 μL at a flow rate of 0.8 mL/min for a 25 min run time. The identification of individual tocol isomer peaks was performed by comparison with retention data of standards (catalog number MT1072, MT1071, MT1073, and MT1790; MJSBiolynx, Brockville, Canada). Quantification of tocol isomers was correlated to an external calibration. Individual isomers were expressed as % (w/w) of the total and in mg/100 g of oil.

Phytosterols

Sterols were analysed in saponified oil samples following a method proposed by Yang et al. (2001). Samples containing the internal standard 5α-cholestane (Sigma-Aldrich Canada Ltd., Oakville, Canada) dissolved in MTBE were saponified at room temperature with 2 mL of 1 N methanolic potassium hydroxide (KOH) solution for 18 h. Water (2 mL), was added to the saponified samples and the unsaponified portion was triple extracted with hexane. Upon removal of all water and solvent through nitrogen evaporation, the residue was dissolved in 1 mL iso-octane and analysed for composition.

Sterols were analysed using a Shimadzu GC (model 17AAF, Shimadzu Corporation, Kyoto, Japan) with a DB-5 capillary column (L = 30 m, *i.d.* = 0.25 mm, d_f= 0.25 μm; Restek, Bellefonte, PA). The column temperature program included maintaining 60°C for 1 min, increasing at a rate of 40°C/min to 240°C, holding for 1 min, increasing at a rate of 2°C/min to a final temperature of 300°C, and holding for 2 min. Hydrogen was the carrier gas (2.2 mL/min) with the injector and FID temperatures set at 275 and 320°C, respectively. Sterols were identified by comparison with retention data for standards of campesterol (Sigma-Aldrich Canada Ltd., Oakville, Canada), β-sitosterol (SRL, Milan, Italy), stigmasterol (SRL, Milan, Italy), cholesterol (Chemservice, Inc., West Chester, PA), and 5α-cholestane and quantified using the internal standard. Individual sterols were expressed as % (w/w) of total sterols and in mg/100 g of oil.

3.1.8 Statistical analysis

Statistical analysis was conducted using JMP IN Statistical Discovery Software (SAS Institute, Inc., Cary, NC, 2001). Simple statistical parameters [mean, standard deviation (*SD*), and coefficient of variation (*CV*)] were estimated for each of the physical characteristics and bioactive compounds studied. Group differences on these traits were established with one way analysis of variance (ANOVA) for equal variances and using non-parametric methods (Wilcoxon/Kruskal-Wallis) for unequal variances. Significant differences among means detected by ANOVA were compared using the Tukey-Kramer Honestly Significant Difference (HSD) test with a probability of $p = 0.05$.

3.2 Characteristics and bioactive analysis with respect to harvest time

3.2.1 Temperature

The three harvest periods occurred during: September 4 to 8, 2003; November 9 to 12, 2003, and January 18 to 20, 2004. The mean dry bulb ambient temperatures for September 4, 2003 to January 25, 2004 ranged from 11.7 to -16.3°C with temperatures ranging from 34.0 to -35.2°C (Table 3.1).

Table 3.1 Mean, maximum, and minimum temperatures (°C) for the 2003-04 harvest period

	Month				
	September	October	November	December	January
Mean	11.7	6.2	-7.0	-8.6	-16.3
Maximum	34.0	27.3	9.2	4.6	-5.4
Minimum	-3.4	-10.0	-24.2	-30.8	-35.2

September berries were firm, easily removed from the branches, and at various stages of ripening. Berry development and ripening continued throughout the mild fall until early November. The November harvest was conducted at temperatures of ~-5.0°C, which slowed the collection of intact whole berries. Freeze and thaw effects on the berry structure, ripeness, and lack of abscission layer caused tearing of the peel of many berries. Berries that remained on the shrubs for 10 additional weeks through winter conditions were exposed to freeze and thaw cycles, temperature fluctuations, precipitation, wind, and sunlight. The January harvested berries were collected at temperatures below -20°C, resulting in easy removal of the frozen berries.

3.2.2 Berry and seed sizing

The effect of harvest time on berry size was significant ($p < 0.05$) (Table 3.2). The berry size for September (15.6 g%) was the lowest ($p < 0.05$) because of berry immaturity. Berries increased in size as ripening progressed from September to November (19.4 g%), with an insignificant decrease to 17.9 g% in January ($p > 0.05$). Differences in seed sizes were insignificant ($p > 0.05$), possibly because of early seed development and protection from the elements by the berry structure. Berry sizes were lower than the 21.8 to 34.2 g% reported by Tang and Tigerstedt (2001). As with berry sizes, Tang and Tigerstedt reported consistently larger seed sizes of 1.4 and 1.5 g% as compared to 1.0 g% (Table 3.2).

Variations between studies throughout this discussion may have been attributed to differences in geographical location, climate, environment, harvest period, and berry maturity. The experimental results and sample calculations for Sections 3.2.2 to 3.2.4 are presented in Appendix A.1.

3.2.3 Moisture content

Moisture contents for September, November, and January were: 77.8, 75.8, and 75.8% wb, respectively (Table 3.2). The differences between harvest times were insignificant ($p > 0.05$) possibly because of the large variation ($SD = 2.1\%$) in berries encountered for November. Values compared well in magnitude with the 74% measured by Ma and Cui (1987), however, were lower than those reported by Tang and Tigerstedt

(2001) of 82.2 to 87.5%. A larger size of the latter berries may have contributed to the higher moisture content.

Table 3.2 Characteristics of sea buckthorn berries for different harvest times

Characteristic	n[a]	Harvest month [b], [c]		
		September	November	January
Berry size (g % berries)[d]	3	$15.6^{a} \pm 1.0$	$19.4^{b} \pm 0.6$	$17.9^{b} \pm 0.9$
Seed size (g % seeds)[e]	3	1.00 ± 0.03	0.97 ± 0.01	0.99 ± 0.02
Moisture content (% wb)[f]	2	77.8 ± 0.0	75.8 ± 2.1	75.8 ± 0.1
Seed content in berries (% w/w)[g]	9	$7.0^{a} \pm 0.3$	$5.9^{b} \pm 0.1$	$6.6^{c} \pm 0.3$

[a] n = number of samples.
[b] Means along a row with like letters are not significantly different at $p = 0.05$.
[c] $\pm$ *SD*, *SD* = standard deviation.
[d] g % berries = mass of 100 berries
[e] g % seeds = mass of 100 seeds
[f] wb = wet basis
[g] w/w = mass/mass

3.2.4 Seed content in berries

Seed contents varied significantly ($p < 0.05$) between harvest times (Table 3.2). The seed content was highest at 7.0% for the September berries, with a decrease to 5.9% by November and an increase to 6.6% by January ($p < 0.05$). The seed content trend across harvest times was opposite to that for the berry size. This relationship was confirmed by the increasing berry and consistent seed sizes. The seed contents were within the ranges of 3.9 to 9.0% and 3.6 to 8.4%, reported by Yang and Kallio (2001) and Yang et al. (2001), respectively.

3.2.5 Colour factors

The effect of harvest time on CIELab factor a^* was significant ($p < 0.05$) (Table 3.3). Positive colour factors (a^* and b^*) for each harvest period confirmed red and yellow values in the fruit, respectively. Visually, a portion of the September berries had a green colouring represented by lower values of a^*. A 42.3% increase in a^* occurred between September and November. Factor a^* (+ 20.2) was significantly higher ($p < 0.05$) for November, possibly because of the processes involved with ripening: disappearance of chlorophylls, major biosynthesis of carotenoids, and esterification of xanthophylls with fatty acids (Mínguez-Mosquera et al. 2002). A 13.9% decrease in a^* occurred between November and January. Lightness factor, L^*, was highest in January samples at 47.2 and was significantly different ($p < 0.05$) from September samples at 45.2. The lightening of the fruit and decrease of a^* may have been due to carotenoid degradation (Erickson 1997). The experimental results and sample calculations are presented in Appendix A.2.

3.2.6 Total carotenoids

Fruit fraction

Means of total carotenoids in the fruit fraction for the three harvest periods were significantly different ($p < 0.05$) (Table 3.4). The values ranged from a low in September of 498.1 mg/100 g oil to a high in November of 817.8 mg/100 g oil. A 24.6% decrease in carotenoids occurred from November to January resulting in 616.8 mg/100 g oil. Redness, a^*, and carotenoid content followed the same trend. The 64.2% increase from September to November was comparable to the 62% increase for whole berries (ssp.

botanitjetskaja, *trofimovskaja*, and *aromatnaja*) obtained by Gao et al. (2000). The total carotenoids compared well with the 500 to 1000 mg/100 g oil range (variety not specified) reported by Xin et al. (1995).

Table 3.3 Colour analysis of sea buckthorn berries for different harvest times

CIELab factor	n[a]	Harvest month[b], [c]		
		September	November	January
Lightness, L^*	3	$45.2^{a} \pm 0.8$	$45.5^{a,b} \pm 1.0$	$47.2^{b} \pm 0.5$
(+)red/(-)green, a^*	3	$+14.2^{a} \pm 0.5$	$+20.2^{b} \pm 0.4$	$+17.4^{c} \pm 0.5$
(+)yellow/(-)blue, b^*	3	$+35.7 \pm 1.6$	$+36.7 \pm 3.1$	$+39.7 \pm 0.7$

[a] n = number of samples.
[b] Means along a row with like letters are not significantly different at $p = 0.05$.
[c] $\pm SD$, SD = standard deviation.

Table 3.4 Total carotenoids in sea buckthorn fruit and seed fractions for different harvest times, expressed in mg/100 g of oil

Fraction	n[a]	Harvest month[b], [c]		
		September	November	January
Fruit	9	$498.1^{a} \pm 37.4$	$817.8^{b} \pm 77.5$	$616.8^{c} \pm 35.4$
Seed	3	24.4 ± 1.6	25.6 ± 4.2	27.6 ± 3.5

[a] n = number of samples.
[b] Means along a row with like letters are not significantly different at $p = 0.05$.
[c] $\pm SD$, SD = standard deviation.

Seed fraction

Total carotenoid levels in the seed fraction did not significantly change between harvest times (Table 3.4). For seeds, the total carotenoid levels of 24.4, 25.6, and 27.6 mg/100 g oil fell into the lower range of 20 to 85 mg/100 g oil measured by Xin et al. (1995). The experimental results and sample calculations for both fruit and seed fractions are presented in Appendix A.3.

3.2.7 Fatty acids

Fruit fraction

Three major fatty acids, palmitic, palmitoleic, and oleic accounted for approximately 32.2, 26.5, and 18.7% of the total fatty acids in the fruit fraction, respectively (Table 3.5 and Fig. 3.1). Yang and Kallio (2001) obtained similar results with palmitoleic and oleic acids accounting for 27.2 and 17.1% of the total fatty acids, respectively. The compound which accounted for 8.1%, was identified by other researchers as vaccenic (Yang and Kallio 2001; Yang and Kallio 2002b) or *cis*-vaccenic acid (Kallio et al. 2002b; Pintea et al. 2001).

The fatty acid profile remained relatively stable between harvest times in the fruit fraction. Significant changes included a 1% increase ($p < 0.05$) in oleic acid from November to January, while minor ($\leq 0.6\%$) differences ($p < 0.05$) occurred for α-linolenic between all harvest times (Table 3.5). Zadernowski et al. (1997) reported that once berries (variety not known) turned a yellow orange (mid-September), palmitic, palmitoleic, linoleic, and α-linolenic acids were fully synthesized and remained relatively constant for the remainder of the maturation period. Another study, in which berries were collected at two week intervals, reported up to 12% variation in levels of oleic and palmitoleic acids in whole berry oil in mid-October (Yang and Kallio 2002b). Linoleic and α-linolenic acids were also reported to be negatively correlated (Yang and Kallio 2001).

Table 3.5 Fatty acids composition in sea buckthorn fruit and seed fractions for different harvest times, expressed in mass percentage [% mass/mass (w/w)] of oil

Fatty acid[a]	Retention time, min[b]	Harvest month[c],[d]		
		September	November	January
		Fruit fraction (n = 9)[e]		
C16:0	7.89	32.1 ± 0.2	32.2 ± 0.6	32.2 ± 1.1
C16:1*n*7	8.10	26.2 ± 0.5	26.8 ± 0.5	26.5 ± 0.8
C18:0	10.16	1.4^{a} ± 0.1	1.2^{b} ± 0.0	1.3^{a} ± 0.2
C18:1*n*9	10.37	$18.8^{a,b}$ ± 0.6	18.2^{a} ± 0.5	19.2^{b} ± 1.0
Unknown	10.46	8.1 ± 0.2	8.0 ± 0.2	8.1 ± 0.4
C18:2*n*6	10.85	6.9 ± 0.3	6.8 ± 0.3	6.6 ± 0.3
C18:3*n*3	11.52	2.6^{a} ± 0.3	2.3^{b} ± 0.1	2.0^{c} ± 0.1
		Seed fraction (n = 3)		
C16:0	7.89	8.6 ± 0.0	8.5 ± 0.2	8.4 ± 0.2
C16:1*n*7	8.09	0.8 ± 0.0	0.6 ± 0.1	0.7 ± 0.1
C18:0	10.16	2.3 ± 0.0	2.3 ± 0.1	2.3 ± 0.2
C18:1*n*9	10.37	19.3 ± 0.2	19.0 ± 0.7	19.9 ± 0.6
Unknown	10.46	2.3 ± 0.0	2.3 ± 0.1	2.3 ± 0.0
C18:2*n*6	10.86	36.2^{a} ± 0.0	36.6^{b} ± 0.1	$36.4^{a,b}$ ± 0.1
C18:3*n*3	11.53	28.9 ± 0.3	28.9 ± 0.8	28.2 ± 1.1

[a] C16:0 = palmitic acid. C16:1*n*7 = palmitoleic acid. C18:0 = stearic acid. C18:1*n*9 = oleic acid. C18:2*n*6 = linoleic acid. C18:3*n*3 = α-linolenic acid.
[b] Retention times correlate with profiles provided in Figs. 3.1 and 3.2.
[c] Means along a row with like letters are not significantly different at $p = 0.05$.
[d] ±*SD*, *SD* = standard deviation.
[e] n = number of samples.

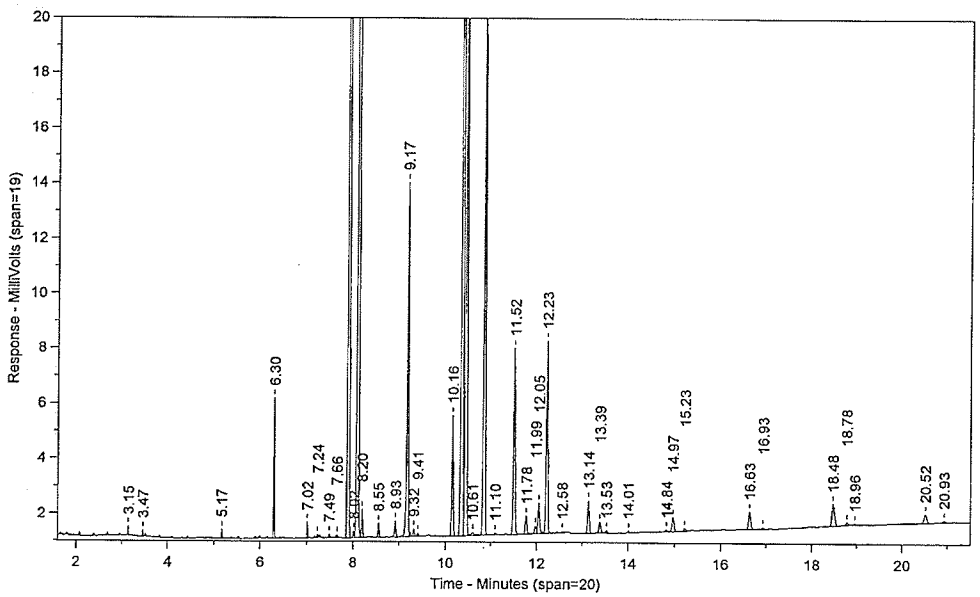


Figure 3.1 Fatty acid profile for sea buckthorn fruit fraction oil (November harvest). Major fatty acids with respective retention times: C16:0, 7.89 min; C16:1*n*7, 8.10 min; C18:0, 10.16 min; C18:1*n*9, 10.37 min; C18:2*n*6, 10.85 min, and C18:3*n*3, 11.52 min.

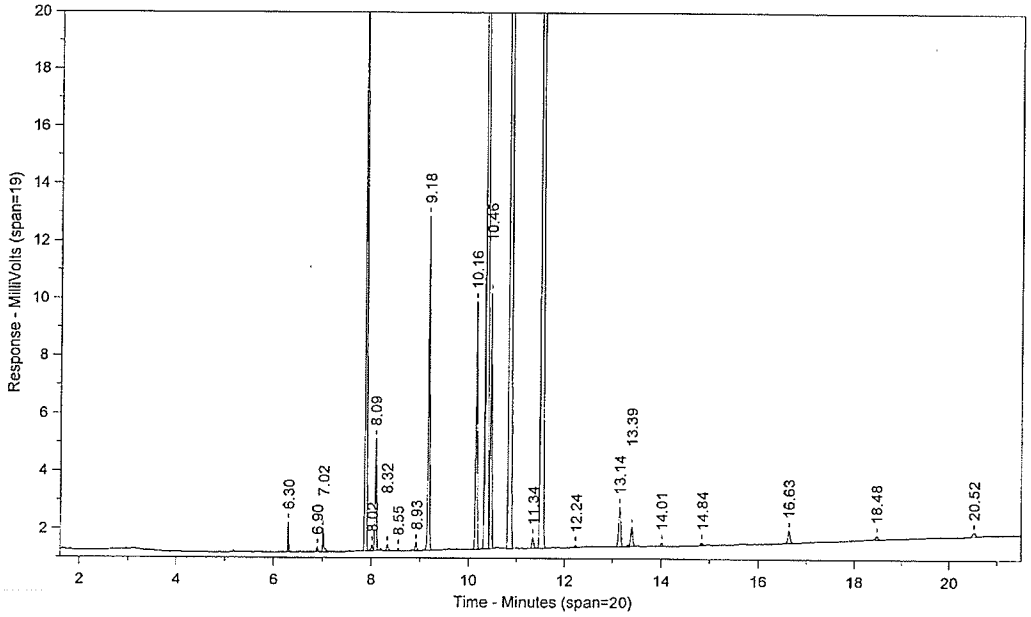


Figure 3.2 Fatty acid profile for sea buckthorn seed fraction oil (November harvest). Major fatty acids with respective retention times: C16:0, 7.89 min; C16:1*n*7, 8.09 min; C18:0, 10.16 min; C18:1*n*9, 10.37 min; C18:2*n*6, 10.86 min, and C18:3*n*3, 11.53 min.

Seed fraction

Three major fatty acids, linoleic, α–linolenic, and oleic accounted for approximately 36.4, 28.7, and 19.4% of the total fatty acids in seed fraction oil, respectively (Table 3.5 and Fig. 3.2). In sharp contrast to the fruit fraction, the seed fraction contained approximately 8.5% palmitic and 0.7% palmitoleic acid. Harvest time had a negligible effect ($p > 0.05$) on fatty acid concentration in the seed fraction. Seed fatty acid proportions were in close agreement with the results reported by Yang and Kallio (2001). However, as with whole berries (Yang and Kallio 2002b), variations in oleic acid content in the seed fraction were reported (Yang and Kallio 2001). The experimental results and sample calculations for both fruit and seed fractions are presented in Appendix A.4.

3.2.8 Tocopherols and tocotrienols

Total tocol concentrations ranged from 485 to 343 mg/100 g oil and 262 to 217 mg/100 g oil for fruit and seed fractions, respectively (Table 3.6). A significant loss of 20.2% ($p < 0.05$) of total tocols in the fruit fraction occurred between November and January. Kallio et al. (2002a) reported the range of tocols in ssp. *sinensis* fruit fraction, 400 to 700 mg/100 g oil, was 2 to 3 times higher than ssp. *rhamnoides* and *mongolica*. They also reported total tocol contents of 100 to 300mg/100 g oil in *sinensis* seeds.

A major proportion (91.4%) of fruit fraction tocols was comprised of α-T (79.3%), β-T3 (8.8%), and δ-T (3.3%). Significant changes ($p < 0.05$) in proportions included: a 1.4% decrease from September to November (α-T), a 1.1% decrease from September to January (δ-T), and a 0.8% increase from September to November (β-T3). Similar

proportions were reported by Kallio et al. (2002a) with changes in α-T up to 10% within a period from August to November.

Fruit fraction

September fruit fraction had significantly higher ($p < 0.05$) levels of α-T, with concentration (mg/100 g oil) losses of 12.9 and 19.5% between September and November and November and January, respectively (Table 3.6 and Fig. 3.3). Significant losses ($p < 0.05$) were also noted from September to January for γ-T, δ-T, and β-T3. The consistent decreasing trend from September to January did not occur for minor isomers, β-T and α-T3, which had slightly higher levels in November ($p < 0.05$). Maximum amounts of α-T, γ-T, and δ-T were also reported for the oil of whole berries (cv. *nadbaltycka*) harvested in September (Zadernowski et al. 2003). The berries were of an olive-yellow colour denoting under-ripeness as in this study.

Seed fraction

The isomers, α-T, β-T, γ-T, δ-T, γ-T3, and plastochromanol 8 (P-8) were detected in the seeds. A major proportion (94.0%) of the seed fraction oil tocols was comprised of α-T (62.9%), γ-T (26.0%), and β-T (5.1%) (Table 3.6 and Fig. 3.4). Effects of harvest time were insignificant ($p > 0.05$) for isomer concentration in seeds. Lower proportions of α-T (30-50%) were reported by Kallio et al. (2002a) with changes in α-T and γ-T approaching 20% within a period from August to November. The experimental results and sample calculations for both fruit and seed fractions are presented in Appendix A.5.

Table 3.6 Major tocopherol and tocotrienol concentrations in sea buckthorn fruit and seed fractions for different harvest times, expressed in mg/100 g of oil

Tocol [a]	Retention time [b], min	Harvest month (2003-2004) [c], [d]		
		September	November	January
		Fruit fraction (n=9) [e]		
α-T	6.83	$388^{a} \pm 42$	$338^{b} \pm 43$	$272^{c} \pm 27$
β-T	8.87	$10^{a} \pm 1$	$12^{b} \pm 2$	$10^{a} \pm 1$
γ-T	10.84	$9^{a} \pm 2$	$4^{b} \pm 1$	$2^{c} \pm 1$
δ-T	15.62	$18^{a} \pm 4$	$15^{a} \pm 2$	$9^{b} \pm 2$
α-T3	7.85	$9^{a} \pm 2$	$12^{b} \pm 2$	$10^{a,b} \pm 2$
β-T3	10.16	$40^{a} \pm 5$	$39^{a} \pm 4$	$31^{b} \pm 5$
γ-T3	12.77	10 ± 2	9 ± 2	8 ± 1
δ-T3	18.56	1 ± 0	1 ± 0	1 ± 0
Total		$485^{a} \pm 53$	$430^{a} \pm 53$	$343^{b} \pm 34$
		Seed fraction (n=3)		
α-T	6.82	158 ± 5	139 ± 28	156 ± 4
β-T	8.85	12 ± 0	11 ± 2	14 ± 3
γ-T	10.81	68 ± 1	57 ± 11	63 ± 2
δ-T	15.65	10 ± 2	7 ± 2	7 ± 0
P-8	10.13	3 ± 0	3 ± 0	3 ± 0
γ-T3	18.56	11 ± 19	n/d [f]	n/d
Total		262 ± 19	217 ± 42	243 ± 5

[a] α-T = α-tocopherol. β-T = β-tocopherol. γ-T = γ-tocopherol. δ-T = δ-tocopherol. α-T3 = α-tocotrienol. β-T3 = β-tocotrienol. γ-T3 = γ-tocotrienol. δ-T3 = δ-tocotrienol. P-8 = Plastochromanol-8.

[b] Retention times correlate with profiles provided in Figs. 3.3 and 3.4.

[c] Means along a row with like letters are not significantly different at $p = 0.05$.

[d] $\pm SD$, SD = standard deviation.

[e] n = number of samples.

[f] n/d = not detected.

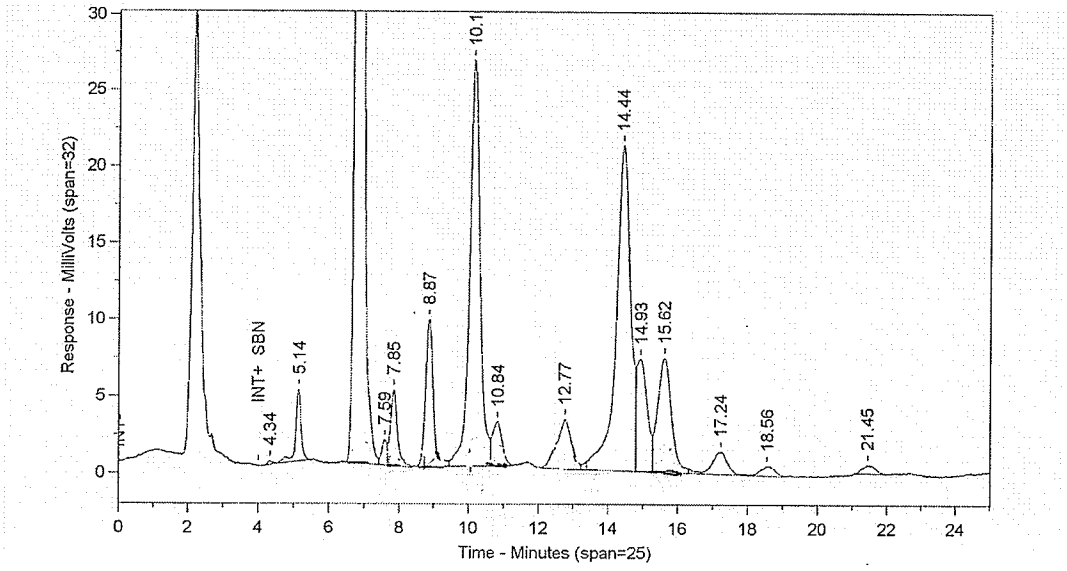


Figure 3.3 Tocol profile for sea buckthorn fruit fraction oil (November harvest). Major tocols with respective retention times: α-tocopherol, 6.83 min; β-tocopherol, 8.87 min; γ-tocopherol, 10.84 min; δ-tocopherol, 15.62 min; α-tocotrienol, 7.85 min; β-tocotrienol, 10.16 min; γ-tocotrienol, 12.77 min, and δ-tocotrienol, 18.56 min.

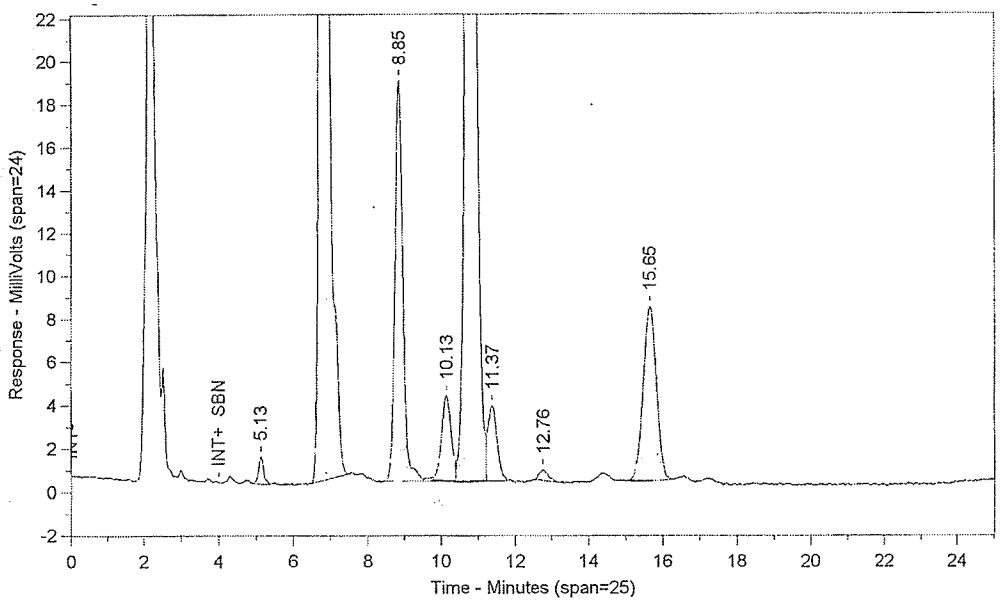


Figure 3.4 Tocol profile for sea buckthorn seed fraction oil (November harvest). Major tocols with respective retention times: α-tocopherol, 6.82 min; β-tocopherol, 8.85 min; γ-tocopherol, 10.81 min; δ-tocopherol, 15.65 min; plastochromanol (P-8), 10.13 min, and γ-tocotrienol, 18.56 min.

3.2.9 Phytosterols

Over 20 phytosterols and terpenes have been identified in the oils of sea buckthorn fruit and seed fractions (Li et al. 2007; Yang et al. 2001). Phytosterols identified in this study included cholesterol, campesterol, stigmasterol, and β-sitosterol (Figs. 3.5 and 3.6). Other peaks were detected but were not identifiable due to limited standards and incomparable spectra presented in other studies. A major proportion of the identified phytosterols in the fruit and seed fraction was comprised of β-sitosterol.

Fruit fraction

Concentrations of β-sitosterol in the fruit fraction were significantly ($p < 0.05$) higher in September (928 mg/100 g oil) with a 25.3% decrease to a low of 693 mg/100 g oil in November (Table 3.7 and Fig. 3.5). Similarly, a decrease was reported in levels of β-sitosterol from August to November paired with a slight increase in campesterol (Yang et al. 2001). Reported values of fruit fraction phytosterol concentration ranged from 1030 to 2870 mg/100 g oil (ssp. *sinensis* and *rhamnoides*) and 771 mg/100 g oil (ssp. *sinensis*) (Yaonian et al. 1995). The content of β-sitosterol ranged from 61 to 83% (ssp. *sinensis* and *rhamnoides*) (Yang et al. 2001) and 85% (ssp. *sinensis*) (Yaonian et al. 1995).

Seed fraction

Concentrations of β-sitosterol in the seed fraction did not vary significantly ($p > 0.05$) from September (521 mg/100 g oil) to January (567 mg/100 g oil)) (Table 3.7 and Fig. 3.6). No significant effects ($p > 0.05$) of harvest time occurred for total sterols and β-

sitosterol in the seed fraction as similarly reported by Yang et al. (2001) for a harvest time between August and November. Reported values of total phytosterol concentration ranged from 1240 to 2300 mg/100 g oil (ssp. *sinensis* and *rhamnoides*) (Yang et al. 2001) and from 1022 to 1298 mg/100 g oil (ssp. *sinensis*) (Yaonian et al. 1995). The content of β-sitosterol ranged from 57 to 76% (ssp. *sinensis* and *rhamnoides*) (Yang et al. 2001) and 74% (ssp. *sinensis*) (Yaonian et al. 1995). Contrary to this study, total phytosterol concentrations have been reported as being higher in the seed versus the fruit fraction (Li et al. 2007; Yang et al. 2001; Yaonian et al. 1995). The experimental results and sample calculations for both the fruit and seed fractions are presented in Appendix A.6.

Table 3.7 Phytosterol concentrations in sea buckthorn fruit and seed fractions, expressed in mg/100 g of oil

Phytosterol	Retention time [a], min	Harvest month (2003-2004) [b],[c]		
		September	November	January
		Fruit fraction (n=9) [d]		
Cholesterol	16.10	4 ± 6	6 ± 4	2 ± 3
Campesterol	18.10	19^{a} ± 4	14^{b} ± 2	$16^{a,b}$ ± 2
Stigmasterol	18.79	7 ± 4	4 ± 3	8 ± 9
β-sitosterol	19.89	928^{a} ± 196	693^{b} ± 105	723^{b} ± 106
		Seed fraction (n=3)		
Cholesterol	16.10	2 ± 2	n/d	3 ± 2
Campesterol	18.10	13 ± 1	12 ± 2	14 ± 1
Stigmasterol	18.79	n/d [e]	n/d	1 ± 1
β-sitosterol	19.89	521 ± 8	528 ± 52	567 ± 27

[a] Retention times correlate with profiles provided in Figs. 3.5 and 3.6.
[b] Means along a row with like letters are not significantly different at $p = 0.05$.
[c] ±*SD*, *SD* = standard deviation.
[d] n = number of samples.
[e] n/d = not detected.

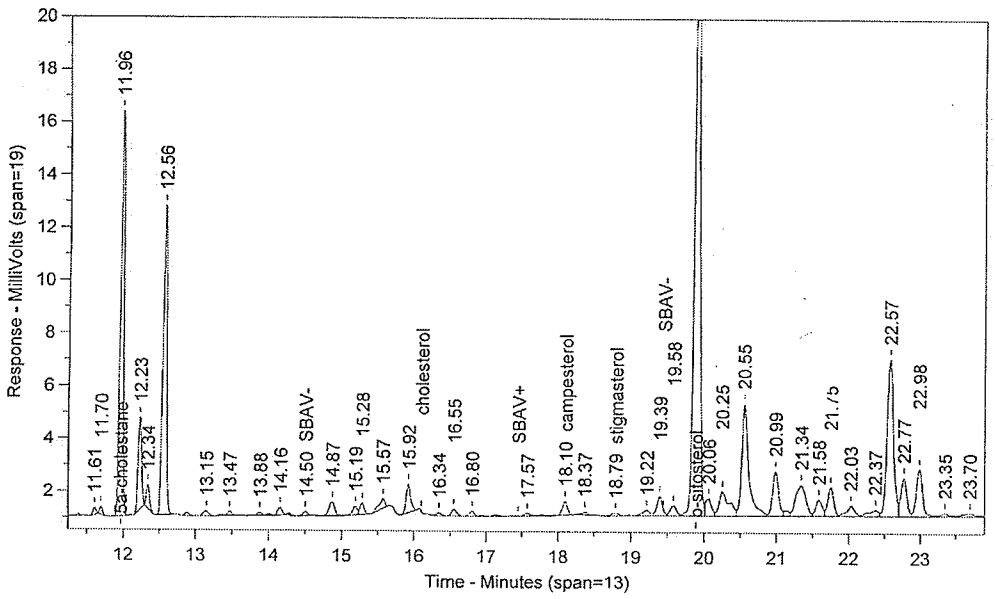


Figure 3.5 Phytosterol profile for sea buckthorn fruit fraction oil (November harvest). Major sterols with respective retention times: cholesterol, 16.10 min; campesterol, 18.10 min; stigmasterol, 18.79 min, and β-sitosterol, 19.89 min.

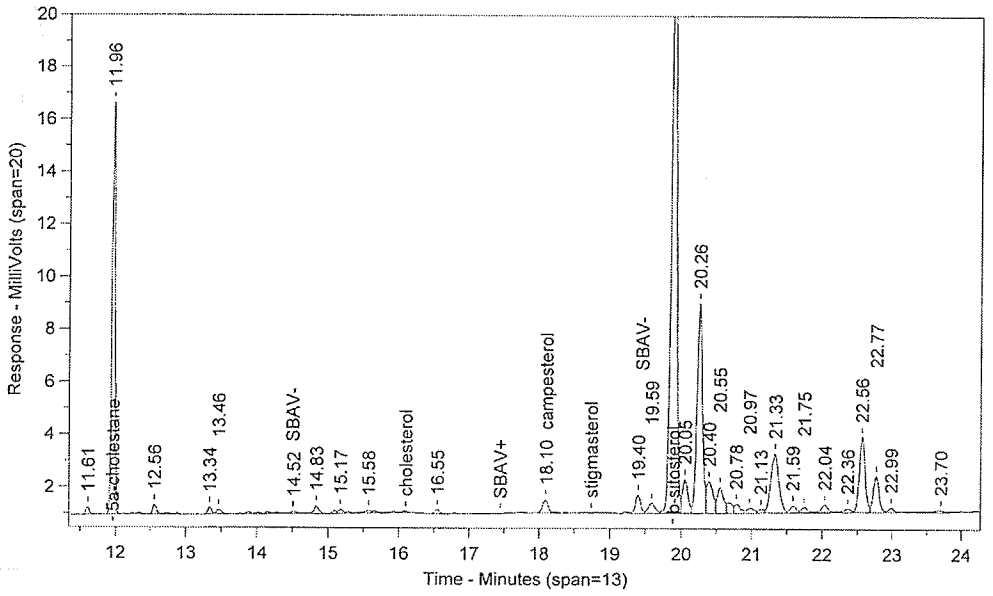


Figure 3.6 Phytosterol profile for sea buckthorn seed fraction oil (November harvest). Major sterols with respective retention times: cholesterol, 16.10 min; campesterol, 18.10 min; stigmasterol, 18.79 min, and β-sitosterol, 19.89 min.

3.3 Conclusions on the influence of harvest time on quality

The influence of harvest time on quality of sea buckthorn berries was investigated. Whole berries were evaluated for colour, size, seed content, and moisture content, whereas the individual fractions, fruit (included pulp, peel, and juice) and seed were evaluated for bioactive content (i.e. total carotenoids, fatty acids, tocols, and sterols). Changes that occurred with respect to harvest time varied depending on the physical characteristic and oil compound. The individual mechanisms by which these changes occurred are complex and were not identified in this study. However, these mechanisms could have been divided into two main groups; those related to (1) fruit development and ripening and (2) degradation due to post-maturity and climatic conditions.

Early maturity and maturity

Varying rates of biosynthesis and metabolic pathways may have attributed to the differences in trends and levels between individual compounds during the fruit development and ripening stage (from September to November). The increase (15.6 to 19.4 g%) in berry size between September and November reflected the growth and development that occurred during the ripening stage. The size increase was due only to the development of the fruit fraction as supported by the significant 1.1 % decrease in seed content and consistent seed size.

Concentrations of major compounds, such as α-T (388 mg/100 g oil) and β-sitosterol (928 mg/100 g oil) for the fruit fraction were at their highest in under-ripe berries with losses of 12.9 and 25.3%, respectively, incurred during the ripening stage.

Concentrations of these compounds as well as total carotenoids in seed oil did not change. Biosynthesis of carotenoids in the fruit fraction during the ripening stage was represented by a 64.2 % increase in total carotenoid concentration and a 42.3 % increase in colour factor a^* (redness). The fatty acid compositions for both the fruit and seed fractions remained relatively consistent between September and November with only slight differences (< 0.4%) occurring for C18:0 and C18:3*n*3 (fruit fraction) and C18:2*n*6 (seed fraction). The stability in seed oil bioactive content is representative of seed development occurring at an early stage of berry development.

Post-maturity

Extended exposure to temperature fluctuations including freeze and thaw conditions and environmental conditions such as precipitation, light, wind, and inherent pro-oxidants would have occurred between November and January. This combined with the cessation of chemical and physical reactions related to fruit development was associated with significant losses in total tocols (20.2%) and individual isomers (i.e. α-T, β-T, γ-T, δ-T, β-T3), total carotenoids (24.6%), and colour [e.g. a^* (13.9%)] in the fruit fraction oil. The antioxidant activity of tocols and carotenoids in the berry may have contributed both to their respective losses as well as the relative stability of the fatty acid profile. A slight gain (≤ 1%) occurred in the proportion of C18:1*n*9 in the fruit fraction Based on this study, harvesting during post-maturity resulted in overall lower quality of fruit fraction oil. Compound degradation was limited to the fruit fraction because no significant changes occurred in the seed fraction oil.

4 THE INFLUENCE OF DRYING ON LEVELS OF BIOACTIVE COMPOUNDS IN PULP AND PEEL OIL OF SEA BUCKTHORN (*H. rhamnoides* L. ssp. *sinensis*) BERRIES

4.1 Experimental determination of characteristics and bioactive compounds

4.1.1 Harvest and post harvest handling

Sea buckthorn berries ssp. *sinensis* were manually harvested from four year old shrubs at St. Claude, Manitoba, Canada, during February 2003. Undamaged berries were collected from 75 shrubs in 1.3 kg lots to form one representative sample from the orchard. Harvested frozen, the berries were cleaned of debris and mixed to form a homogeneous pool of berries. The berries were bagged and kept frozen at -25°C until required. Berries were thawed according to Section 3.1.1.

4.1.2 Fruit preparation

The berries were crushed using a tomato press (model Master, Rigamonti Pietro & Figli, Vercurago, Italy) which yielded juice and press cake (seed and pulp and peel) fractions. Only the press cake fraction was used for the remainder of this research. Two types of samples were prepared from the press cake: a fresh and a drying sample. For fresh sample preparation, seeds were removed manually from 28 g of press cake using forceps. For the drying sample, a total volume of 240 mL of press cake was spread upon

two non-stick drying sheets (model Teflex TF14, Excalibur Products, Sacramento, CA) in six square 0.016 m^2 areas with 2.5 mm thickness. The drying sheets were supported upon a fine wire mesh pan.

4.1.3 Drying trials

Convective drying of the fruit laden sheets was performed within an environmental chamber (model IH-400U, Yamato Scientific America, Inc., Orangeburg, NY). Six chamber temperature and relative humidity (RH) combinations: 50°C at 30.6 and 58.7% RH, 60°C at 24.4 and 57.0% RH, and 70°C at 20.8 and 57.0% RH, were applied in triplicate, resulting in a total of 18 drying trials. Relative humidity was monitored using a humidity sensor (model HIH-4000, Honeywell, Freeport, IL) suspended within the chamber. Air flow measured in preliminary drying trials for each of the drying conditions using a digital anemometer (model HHF300A, Omega Engineering, Inc., Stamford, CT), averaged 1 m/s at 0.25 m from the base of the chamber interior.

One 40 mL fruit section was spread on a non-stick sheet on a wire mesh tray suspended from a scale (model Adventurer Pro, Ohaus Corporation, Pine Brook, NJ), providing ±0.001 g accuracy, located on the roof of the environmental chamber. Drying time for each trial was selected based on the suspended sample achieving an approximate moisture content of 7.0% wb. Estimation of the final moisture content was based on pre-trials run at the same conditions. Drying data were recorded at 15 min intervals using a data acquisition system (model HP 3421A, Hewlett-Packard Company, Houston, TX).

4.1.4 Moisture content and water activity

Moisture content determination as decribed in Section 3.1.4 was conducted in duplicate according to AOAC's Official Methods 920.151 and 934.06 for the fresh and dried press cake, respectively (AOAC 2002a). Water activity (a_w) was determined for the fresh and dried seeded press cake and seeds using an a_w centre (Novasina, Pfaeffikon, Switzerland). Calibration and determination was conducted according to the AOAC's Official Method of Analysis 978.18 (AOAC 2002b).

The seed removal process of the dried press cake sections was conducted using a blender (model LR47897, Osterizer, Sunbeam Corporation, Delray Beach, FL) and a seed shaker (model RX-812) with a standard series of sieves (W.S. Tyler Company, Mentor, OH). The dried press cake was gently threshed in the blender at the lowest setting for short term intervals (1 to 3 s) repeatedly for 10 to 15 cycles. Seed damage was minimized by covering the blades of the blender with lab/food grade tubing, (*i.d.* = 32 mm, *o.d.* = 64 mm, Nalgene, Rochester, NY). Seeds were separated from the dried threshed press cake using 203 mm *o.d.* USA standard test sieves number: 6 (3.36 mm), 8 (2.36 mm), 10 (2.00 mm), 12 (1.70 mm), 14 (1.40 mm), and 16 (1.18 mm). The final pulp and peel sample consisted of the seedless contents from all sieve pans except number 10 [6 % (w/w) of total] due to the large volume of seeds collected in that sieve. The final pulp and peel sample was ground in a mill (model 6389-33, Oster, Sunbeam Products, Inc., Boca Raton, FL) for 10 s, to produce particles of uniform size, ~1 mm.

4.1.5 Colour factors

Colour measurements were conducted on fresh and dried samples, in triplicate, as per section 3.1.6. The difference in colour (ΔE) between the dried and fresh samples was determined using Eqn. 4.1 (Koyuncu et al. 2003):

$$\Delta E = \sqrt{(L^*_o - L^*)^2 + (a^*_o - a^*)^2 + (b^*_o - b^*)^2} \qquad (4.1)$$

where subscript "*o*" represents the fresh sample prepared from the press cake.

4.1.6 Sample preparation for bioactive compound determination

Bioactive compound determination was conducted for fresh and dried pulp and peel samples. The bioactive compounds analysed included: total carotenoids, fatty acids, tocols, and phytosterols. Detailed methods of oil extraction and bioactive compound analysis are described in Section 3.1.7. The only changes made to these methods include the replacement of filters or programming for the fatty acid and phytosterol analysis. A fused silica capillary column DB-225 (L = 30 m, *i.d.* = 0.25 mm, d_f = 0.25 μm; J & W Scientific, Folsom, CA) was used with a split ratio of 1:20 for the fatty acid analysis. The column temperature program included maintaining 155°C for 2 min, increasing at a rate of 4°C/min to 215°C then holding for 4 min. A ZB-1 capillary column (L = 30 m, *i.d.* = 0.25 mm, d_f= 0.25 μm; Phenomenex, Torrance, CA) with a split ratio of 1:20 was used for phytosterol analysis.

4.1.7 Statistical analysis

Statistical analysis was conducted using JMP IN Statistical Discovery Software according to Section 3.1.8.

4.2 Characteristics and bioactive compounds of fresh and dried pulp and peel

4.2.1 Drying behaviour of press cake

The times associated with drying at the lower relative humidity level (i.e. 30.6, 24.4, and 20.8% RH) from a moisture content of 56.4 to approximately 7.0% ranged from 11.4 h at 50°C to 4.0 h at 70°C (Table 4.1). At a chamber relative humidity of approximately 57.0 to 58.7%, drying times increased to 24.1 h, 19.0 h, and 10.8 h at 50, 60, and 70°C, respectively. During the drying process the press cake would have been dried from an $a_w > 0.925$ to 0.390-0.471. Refer to Appendix B.1 for a_w, moisture content, and drying time data. At higher relative humidity, drying rate was reduced resulting in longer drying times, causing the material to pass through the intermediate a_w zone, 0.5-0.8 for an extended period. Several reactions (e.g. lipid oxidation, non-enzymatic browning, and enzyme activity) can occur within this range of a_w (Van Den Berg 1986).

4.2.2 Fresh seeded sea buckthorn pulp and peel characteristics

The three main constituents in fresh press cake included water (56.4%), pulp and peel solids (35.4%), and seeds (8.2%). Prior to oil extraction, the seeded pulp and peel had a moisture content of 43.3%, a_w of 0.925, and oil content of 4.8%. The CIELab colour factors, L^*_o, a^*_o, and b^*_o, were 50.8, 13.1, and 24.5, respectively, representative of a

bright orange colour (Table 4.2). Total carotenoids in the pulp and peel were 920.3 mg/100 g oil (Fig. 4.1). Palmitic, palmitoleic, and oleic acids claimed the three highest proportions of fatty acids at 22.9, 22.5, and 12.9% (Table 4.3). Total tocols amounted to 707 mg/100 g oil with 76% attributed to α-tocopherol (Table 4.4). All tocol isomers were detected except for δ-tocotrienol. The major phytosterol was β-sitosterol at 1370 mg/100 g oil (Table 4.5).

Table 4.1 Drying and material conditions for dried sea buckthorn berry press cake

Temperature	Relative humidity [a]	Drying time[a]	M (final)[a][c]	a_w (final)[a][d]
°C	%	h	% (w/w) wb[e]	
50	30.6±3.7	11.4±1.4	8.0±1.1	0.463±0.05
50	58.7±1.1	24.1±0.1	6.8±0.9	0.428±0.02
60[b]	24.4±1.4[b]	6.4±1.5[b]	6.7±0.4[b]	0.390±0.01[b]
60	57.0±0.1	19.0±0.1	6.4±1.4	0.410±0.02
70	20.8±1.0	4.0±0.2	6.0±1.4	0.407±0.05
70	57.0±0.1	10.8±4.4	7.9±0.4	0.471±0.03

[a] ±*SD* (n = 3) unless otherwise noted, *SD* = standard deviation.
[b] ±*SD* (n = 2).
[c] M = moisture content.
[d] a_w = water activity.
[e] % (w/w) wb = % mass/mass (w/w) wet basis (wb).

4.2.3 Colour

Lightness and hue

Visually the dried pulp and peel appeared darker and less intense than the bright orange of the fresh pulp. The darkening appearance was generally reflected by a reduction in lightness, redness, and yellowness based on CIELab factors L^*, a^*, and b^*, respectively (Table 4.2). Factors L^*, a^*, and b^* were significantly lower than fresh pulp

and peel for drying parameters, 50°C-30.6% RH (a^* only), 50°C-58.7% RH, 60°C-57% RH, 70°C-20.8% RH (a^* and L^* only), and 70°C-57% RH ($p < 0.05$). The colour values of L^*, a^*, and b^* at 60°C-24.4% RH were the closest to fresh of the six temperature-relative humidity combinations. Refer to Appendix B.2 for colour analysis data.

Colour change

Minimal colour change occurred at the low relative humidity level for each drying temperature as compared to the higher relative humidity level. Colour change value was significant for drying parameters 50°C-58.7% RH (ΔE = 7.0), 60°C-57.0% RH (ΔE = 13.5), 70°C-20.8% RH (ΔE = 4.7), and 70°C-57.0% RH (ΔE = 14.9) ($p < 0.05$) (Fig. 4.2). Drying parameter combinations ranked in order from least to greatest ΔE were: 60°C-24.4% RH, 50°C-30.6% RH, 70°C-20.8% RH, 50°C-58.7% RH, 60°C-57.0% RH, and 70°C-57.0% RH.

Table 4.2 CIELab colour factors in fresh and dried sea buckthorn pulp and peel

Factor[a]	Treatment [b], [c]						
	Fresh[d]	Dried					
		50°C		60°C		70°C	
		30.6%	58.7%	24.4%[d]	57.0%	20.8%	57.0%
L^*	50.8^{a}	$47.9^{a,b}$	45.9^{b}	$49.2^{a,b}$	41.9^{c}	47.2^{b}	41.5^{c}
	±1.6	±1.5	±0.2	±0.3	±1.1	±1.4	±0.3
a^*	13.1^{a}	12.4^{b}	10.5^{c}	$12.7^{a,b}$	9.3^{d}	12.3^{b}	9.5^{d}
	±0.0	±0.1	±0.1	±0.0	±0.1	±0.3	±0.0
b^*	$24.5^{a,b}$	$23.2^{a,b,c}$	20.4^{c}	25.6^{a}	15.2^{d}	$21.9^{b,c}$	13.5^{d}
	±0.4	±2.1	±0.7	±0.7	±0.6	±0.8	±0.5

[a] L^* = lightness. a^* = (+)red/(-)green. b^* = (+)yellow/(-)blue.
[b] Means along a row with like letters are not significantly different at $p = 0.05$.
[c] ± *SD* (n = 3) unless otherwise noted, *SD* = standard deviation.
[d] ±*SD* (n=2).

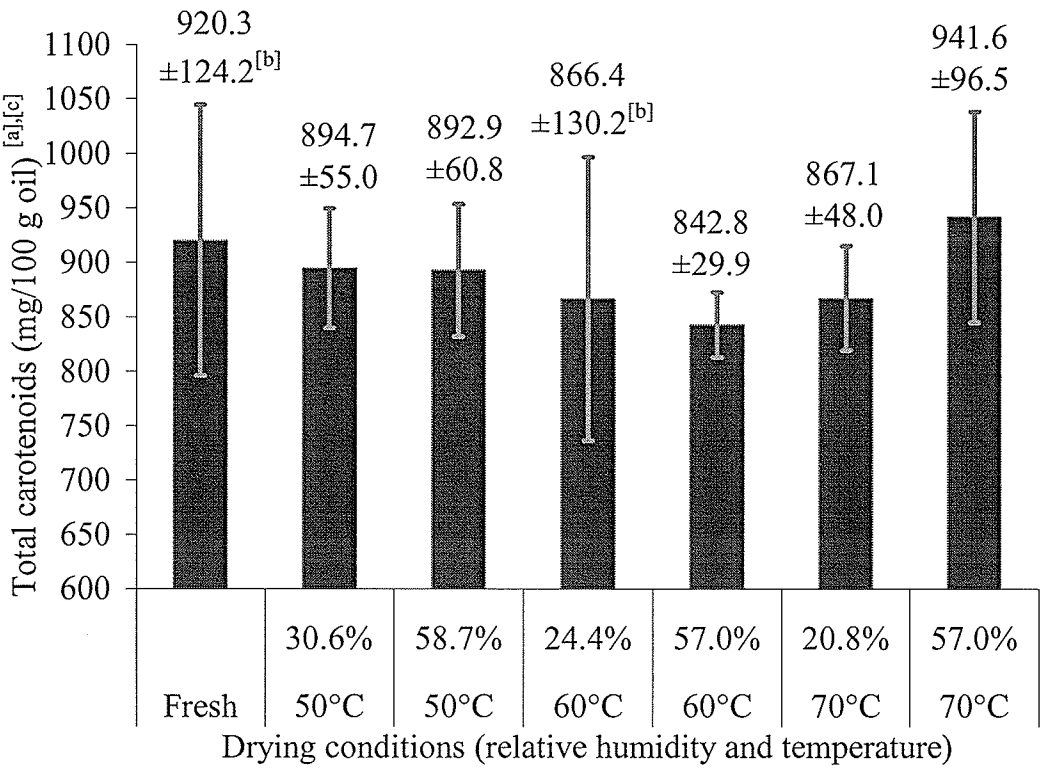


Figure 4.1 Total carotenoids for fresh and dried sea buckthorn pulp and peel
[a] ±*SD* (n=3) unless otherwise noted, *SD* = standard deviation.
[b] ±*SD* (n=2).
[c] Means with like letters are not significantly different at $p = 0.05$.

Effects of drying parameters

For press cake dried at 50°C, 58.7% RH had significantly different ($p < 0.05$) values for a^* (Table 4.2) and ΔE (Fig. 4.2) than the 30.6% RH dried samples. For 60 and 70°C temperature levels, 57.0% RH dried samples had significantly different ($p < 0.05$) values of L^*, a^*, b^* (Table 4.2) and ΔE (Fig. 4.2), than the samples dried at 24.4 and 20.8% RH, respectively. These results were reflected in the extreme darkening of the samples dried at the high relative humidity levels. No comparable research was available for the effect of relative humidity on product colour, however, temperature effects were studied for rosehips and mango pulp powder (Da Cunha et al. 2006; Koyuncu et al. 2003).

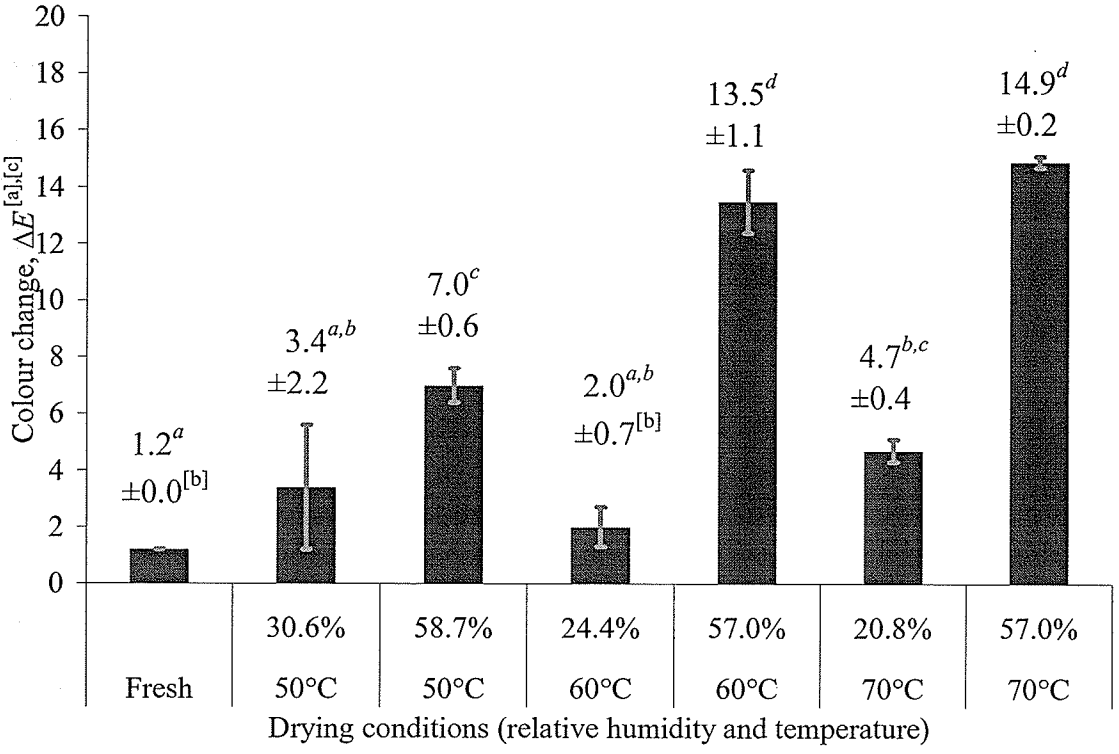


Figure 4.2 Colour change ΔE, between fresh and dried sea buckthorn pulp and peel.
[a] Means along a row with like letters are not significantly different at $p = 0.05$.
[b] $\pm SD$ (n = 3), SD = standard deviation.
[c] $\pm SD$ (n = 2).

Koyuncu et al. (2003) determined that temperatures ≥ 60°C, reduced the drying rate and browning of wild rosehips. Drying temperature of 70°C (20 h - drying time) resulted in smallest ΔE, Δa, and Δb values based on the Hunter colour scale while 60°C (35 h – drying time) yielded highest L values. Temperatures of 30 to 50°C (> 500 to 90 h – drying time) resulted in extensive browning, lowest L value, and highest decreases in a value with 40°C having the largest ΔE. The benefits related to a reduction in drying time for 70°C may have outweighed the potential disadvantages related to a 10°C increase from the 60°C trials. Similar to rosehips (Koyuncu et al. 2003), 70°C was determined as

the optimum drying temperature for colour retention in mango pulp powder (Da Cunha et al. 2006).

Colour degradation of fruits and vegetables during drying may be due to pigment loss or browning, attributed to both enzymatic and non-enzymatic browning reactions (Krokida et al. 2001). Enzymes are deactivated at temperatures ranging from 60 to > 100°C and activity drops as a_w approaches 0.2 (Van Den Berg 1986). The potential of non-enzymatic browning, caused by 1) caramelization of reducing sugars and vitamin C or 2) Maillard browning, a reaction between amines (e.g. amino acids and proteins) and carbonyls (e.g. sugars and flavours) also decreases as a_w approaches 0.2 (Bell 2001; Pokorný and Schmidt 2003). The combination of the moderately high temperature with reduced drying times provided by 60°C-24.4% RH, may have minimized oxidation and both enzymatic and non-enzymatic browning in the sea buckthorn berry pulp and peel (deMan 1999; Nursten 1986). The 10°C difference in optimum temperature for sea buckthorn and the other products (e.g. rosehips and mango pulp powder) may have been due in part to differences in product composition and drying behaviour.

4.2.4 Total carotenoids

Comparison of treatments

No significant difference in total carotenoid levels occurred between dried and fresh pulp and peel oils nor between the oils of dried samples ($p > 0.05$) (Fig. 4.1). Refer to Appendix B.3 for carotenoid analysis data. The extremely darkened samples at 70°C-57% RH had carotenoid concentration levels closest to that of fresh. Upon oil extraction,

dark compounds present in the dried pulp and peel were removed in water soluble solvents leaving rich coloured oil, similar to fresh pulp and peel oil. This colour richness in the oil was reflected in the carotenoid results. Similar to this research, Márkus et al. (1999) reported favourable carotenoid retention in browned (i.e. caramelized) red peppers.

Effects of drying parameters

Regier et al. (2005) reported that carrot slices dried convectively retained lycopene for drying air temperatures between 50 and 90°C at 8% RH and airflow of 4 m/s. βeta-carotene loss (20% to 90°C) occurred for temperatures above 70°C, therefore decreasing total carotenoids. Since temperatures above 70°C were not investigated in this research on sea buckthorn pulp and peel, it is not known if significant losses would have occurred at higher temperatures.

The slight, yet insignificant increase ($p > 0.05$) in total carotenoids in sea buckthorn pulp and peel for 70°C-57.0% RH, may have been due to structural changes caused by extended drying time at higher temperatures. Regier et al. (2005) attributed an increase in carotenoid levels in carrot slices to enhanced extractability caused by structural changes due to thermal treatment. Da Cunha et al. (2006) also reported that temperatures > 70°C resulted in the highest carotenoid levels in mango pulp and peel. Although Gutiérrez et al. (2008), did not determine carotenoid content, oil content was reportedly higher in the convectively dried pulp and peel than in freeze dried samples. The higher

oil content was attributed to cellular structure changes in the convectively dried samples resulting in oil extractions two-fold higher than in freeze dried samples.

4.2.5 Fatty Acids

Comparison of treatments

Fatty acid composition of dried pulp and peel oils was not significantly different than that of the fresh pulp and peel oil ($p > 0.05$) (Table 4.3). Refer to Appendix B.4 for fatty acid sample data. The only significance that occurred among means was that the proportion of palmitoleic acid was greatest for 60°C-57.0% RH at 26.1% and was significantly different ($p < 0.05$) from 20.7% at 70°C-20.8% RH. The main factor that can affect oil stability is oxidation, the rate of which can be influenced by temperature, light, pro-oxidants (e.g. enzymes, minerals, and metals), and a_w (deMan 1999). At an a_w of ~0.2 to 0.3, oxidation is reduced to a minimum whereas at lower (< 0.2) and intermediate a_w's (0.5-0.8) the oxidation rate increases as a result of increased activity of catalysts. Polyunsaturated fatty acids tend to be more susceptible to oxidation than saturated fatty acids (Litwinienko and Kasprzycka-Guttman 2000). Although palmitoleic acid proportions were lower at 70°C, proportions of other polyunsaturated fatty acids, oleic, linoleic, and α-linolenic did not decrease. The lack of major differences in the fatty acid profile between the oils of dried and fresh sea buckthorn pulp and peel signified relative stability during the drying process (Tatum and Chow 2000).

Table 4.3 Fatty acids composition for fresh and dried sea buckthorn berry pulp and peel oil, expressed in mass percentage [% mass/mass (w/w)] of oil

Fatty Acid[a]	Treatment [b], [c]						
	Fresh[d]	Dried					
		50°C		60°C		70°C	
		30.6%	58.7%	24.4%[d]	57.0%	20.8%	57.0%
C16:0	22.9	24.8	25.6	24.9	26.7	22.3	22.5
	±3.6	±2.4	±1.6	±2.0	±0.8	±1.7	±1.7
C16:1*n*7	$22.5^{a,b}$	$24.2^{a,b}$	$24.9^{a,b}$	$24.5^{a,b}$	26.1^{a}	20.7^{b}	$21.1^{a,b}$
	±4.1	±2.0	±1.1	±2.4	±0.9	±1.5	±1.0
C18:0	0.9	0.9	1.0	0.8	1.0	1.0	1.0
	±0.1	±0.1	±0.1	±0.1	±0.0	±0.1	±0.2
C18:1*n*9	12.9	13.6	14.1	13.1	14.0	13.8	13.9
	±1.8	±1.1	±0.8	±1.2	±0.3	±1.9	±1.6
Unknown	7.1	7.2	7.4	7.1	7.4	6.8	6.9
	±1.1	±0.7	±0.2	±1.1	±0.2	±0.7	±0.3
C18:2*n*6	8.0	9.5	10.2	8.7	10.3	11.0	11.1
	±0.9	±0.7	±1.6	±0.7	±0.6	±0.9	±2.5
C18:3*n*3	5.2	5.9	6.5	4.9	5.8	7.5	7.8
	±0.5	±0.5	±1.5	±0.6	±0.8	±0.6	±2.1

[a] C16:0 = palmitic acid. C16:1*n*7 = palmitoleic acid. C18:0 = stearic acid. C18:1*n*9 = oleic acid. C18:2*n*6 = linoleic acid. C18:3*n*3 = α-linolenic acid.
[b] Means along a row with like letters are not significantly different at $p = 0.05$.
[c] ±*SD* (n = 3) unless otherwise noted, *SD* = standard deviation.
[d] ±*SD* (n = 2).

Effects of drying parameters

Gutiérrez et al. (2008) reported that although peroxide values for convectively dried (50°C) samples indicated that some oxidation did occur, lipid composition was not significantly different between convectively dried and freeze dried sea buckthorn berries. Chan et al. (1997) reported that oven dried seaweed samples (60°C, 15 h – drying time) had significantly lower levels of polyunsaturated fatty acids than freeze dried samples, attributing the loss in nutritive value to oxidation. The difference in composition between seaweed and sea buckthorn pulp and peel (e.g. level of antioxidants) may have influenced

stability. The presence of antioxidants such as vitamin C, tocols, and carotenoids in the sea buckthorn pulp and peel may have reduced the rate of oxidation (Cohen et al. 2000).

4.2.6 Tocopherols and tocotrienols

Comparison in treatments

Total levels of tocols ranged from 737 mg/100 g oil (50°C–58.7% RH) to 473 mg/100 g oil (70°C–20.8% RH) (Table 4.4). Refer to Appendix B.5 for tocol sample data. Significant differences ($p < 0.05$) occurred between drying parameter combination 70°C-20.8% RH and 50°C trials (e.g. 30.6 and 58.7% RH) for α-tocopherol, β-tocopherol, and total tocols. The drying parameter combination 70°C-20.8% RH was also significantly lower ($p < 0.05$) than the fresh pulp and peel, 50°C trials, and 70°C-57% RH for β-tocotrienol.

Effect of drying parameters

The 50°C temperature yielded best results in tocol concentrations, signifying that degradation can occur to tocols due to temperatures > 50°C. This statement was valid in a comparison of samples dried at lower relative humidity levels (i.e. 30.6, 24.4, and 20.8% RH). Interestingly, however, mean tocol values were higher for the samples dried at 57.0 or 58.7% RH for each temperature level especially 60 and 70°C. These samples also exhibited a higher level of darkening, as previously discussed. Similarly, Márkus et al. (1999) reported increased levels of tocopherols in browned (e.g. caramelized) dried

red pepper. The specific cause of the possible retention or regeneration of tocols is not known.

Table 4.4 Major tocol concentrations in fresh and dried sea buckthorn berry pulp and peel oil, expressed in mg/100 g oil

Tocol[a]	Treatment[b], [c]						
	Fresh[d]	Dried					
		50°C		60°C		70°C	
		30.6%	58.7%	24.4%[d]	57.0%	20.8%	57.0%
α-T	$537^{a,b}$	530^{a}	547^{a}	$401^{a,b}$	$463^{a,b}$	352^{b}	$519^{a,b}$
	±11	±16	±80	±55	±90	±23	±66
β-T	$25^{a,b}$	26^{a}	26^{a}	$18^{a,b}$	$22^{a,b}$	16^{b}	$25^{a,b}$
	±0	±2	±5	±3	±4	±1	±4
γ-T	30	32	36	24	32	22	34
	±5	±5	±5	±2	±5	±1	±9
δ-T	66	75	77	65	66	52	68
	±3	±5	±8	±20	±15	±4	±11
α-T3	4	7	6	3	2	2	3
	±0	±3	±1	±1	±2	±1	±3
β-T3	35^{a}	34^{a}	34^{a}	$29^{a,b}$	$29^{a,b}$	24^{b}	34^{a}
	±3	±1	±4	±1	±4	±2	±4
γ-T3	10	10	12	7	8	5	9
	±5	±2	±5	±3	±3	±0	±3
δ-T3	n/d [e]	n/d	n/d	n/d	n/d	n/d	n/d
Total	$707^{a,b}$	713^{a}	737^{a}	$548^{a,b}$	$622^{a,b}$	473^{b}	$692^{a,b}$
	±10	±31	±105	±84	±123	±31	±97

[a] α-T = α-tocopherol. β-T = β-tocopherol. γ-T = γ-tocopherol. δ-T = δ-tocopherol. α-T3 = α-tocotrienol. β-T3 = β-tocotrienol. γ-T3 = γ-tocotrienol. δ-T3 = δ-tocotrienol.
[b] Means along a row with like letters are not significantly different at $p = 0.05$.
[c] ±*SD* (n=3), *SD* = standard deviation.
[d] ±*SD* (n=2).
[e] n/d = not detected.

4.2.7 Phytosterols

No significant difference in phytosterol concentration was determined between the oils of the fresh and dried pulp and peel, nor amongst the oils of the dried pulp and peel

($p > 0.05$) (Table 4.5). Refer to Appendix B.6 for phytosterol sample data. The lack of difference in phytosterol concentration between fresh and dried samples could have been due to coefficients of variation up to 18.2% or to the inherent heat stability of phytosterols (Shahidi 2004). There was no comparative research available on effects of drying on phytosterol quality.

Table 4.5 Phytosterol concentrations in fresh and dried sea buckthorn pulp and peel oil, expressed in mg/100 g of oil

Phytosterol	Treatment [a], [b]						
	Fresh[c]	Dried					
		50°C		60°C		70°C	
		30.6%	58.7%	24.4%[c]	57.0%	20.8%	57.0%
Cholesterol	33 ±10	30 ±7	27 ±5	23 ±2	24 ±2	31 ±6	30 ±2
Campesterol	27 ±1	23 ±1	26 ±2	25 ±5	27 ±5	23 ±5	23 ±1
Stigmasterol	n/d[d]	n/d	n/d	n/d	n/d	1 ±2	n/d
β-sitosterol	1370 ±20	1260 ±50	1300 ±100	1190 ±140	1240 ±100	1270 ±230	1250 ±10

[a] Means along a row with like letters are not significantly different at $p = 0.05$.
[b] ±*SD* (n = 3) unless otherwise noted, *SD* = standard deviation.
[c] ±*SD* (n = 2).
[d] n/d = not detected.

4.3 Conclusions on the influence of drying conditions on quality

An evaluation of different drying conditions on sea buckthorn berry pulp and peel resulted in varied effects on quality evaluation parameters (i.e. colour and levels of total carotenoids, fatty acids, tocols, and phytosterols). Temperature and relative humidity each had a significant effect on one or more of the evaluated parameters. Since samples were dried to the same moisture content, drying time increased considerably with a

decrease in temperature or an increase in humidity level or both. The resulting decrease in drying rate and subsequent extended exposure time contributed to the effects of the drying conditions.

The best colour retention compared to fresh pulp and peel was achieved at a temperature and relative humidity combination of 60°C-24.4% RH. A higher degree of colour change due to darkening occurred for all other drying treatments including the milder 50°C temperature level. The shorter drying time and 10°C increase in temperature would have reduced the possibility of enzymatic and non-enzymatic browning. Although colour change in yellow to red food products is commonly attributed to carotenoid loss, carotenoid levels remained relatively stable and the browned portion of the pulp and peel was water soluble and not related to the oil.

At the lower relative humidity levels of 30.6 to 20.8% RH, losses in concentration of α-tocopherol, β-tocopherol, and total tocols occurred at temperatures greater than 50°C. However, relatively high concentrations of these compounds occurred at the 57 to 58.7% relative humidity levels and coincided with a higher level of darkening. The reactions that occurred during the browning of the pulp and peel may have contributed to the retention or regeneration of the tocols. The slightly lower levels of palmitoleic acid in the fatty acid profile at 70°C could be an indication that oxidation occurred. However, the lack of change in other polyunsaturated fatty acids and the remaining portion of the profile indicated stability. Phytosterol levels also appeared to remain stable between different drying treatments.

5 MODEL FOR THIN LAYER DRYING ON AN INERT SPHERE

5.1 Experimental determination of drying kinetics

5.1.1 Harvest and post harvest handling

Sea buckthorn berries (*H. rhamnoides* L. ssp. *sinensis*) were harvested and stored according to Section 4.1.1. Prior to testing, a sample of 250 mL berries was thawed in a thin layer for 5 h at 4°C.

5.1.2 Press cake preparation

The whole berry sample was crushed using a tomato press (Master, Rigamonti Pietro & Figli, Vercurago, Italy) yielding juice and press cake (seed and pulp and peel) fractions. Only the press cake was used for the remainder of this research.

5.1.3 Equipment and materials

The approach used to determine the drying kinetics of material on an inert particle was based on single element drying (Pabis et al. 1998). An inert sphere, ten times the size used in industry, provided enough material for mass change and moisture content determination. A volume of 40 mL of press cake, equivalent to a 2.5 mm layer, was evenly applied to the inside surfaces of two halves of a 63.5 mm diameter strainer (Norco Pro, Everett, WA). The strainer was hand compressed over the surface of a pre-heated

polytetrafluoroethylene (PTFE) sphere, 50.8 mm in diameter, (Applied Plastic Technology Inc., Bristol, RI) and gently removed leaving the press cake on the surface of the sphere. The press cake laden inert sphere was suspended from a scale (Adventurer Pro, Ohaus Corporation, Pine Brook, NJ), providing ±0.001 g accuracy, located on the top of the environmental chamber (IH-400U, Yamato Scientific America Inc., Orangeburg, NY) as depicted in Fig. 5.1.

5.1.4 Press cake drying trials

The trials performed in triplicate were conducted at the drying temperatures and accompanying relative humidities summarized in Table 5.1. As identified in Fig. 5.2, mass of the press cake and inert sphere system (m_{sys}), t_{air}, temperature of the press cake (t_{pc}), temperature at the geometric centre of the inert sphere ($t_{ip\text{-}c}$), temperature of the inert sphere inside surface ($t_{ip\text{-}s}$), and chamber air *RH* were recorded at 15 min intervals using a data acquisition system (HP 3421A, Hewlett-Packard Company, Houston, TX). Temperature (± 0.5°C) was measured using T-type thermocouples AWG30 (Omega Engineering, Inc., Stamford, CT). Relative humidity (± 3.0%) was monitored by wet and dry bulb temperature thermocouples built into the chamber and a separate humidity sensor (HIH-4000, Honeywell, Freeport, IL) suspended within the chamber. In pre-trials, air flow was measured using a digital anemometer (HHF300A, Omega Engineering, Inc., Stamford, CT) and averaged 1 m/s at the location of the sphere.

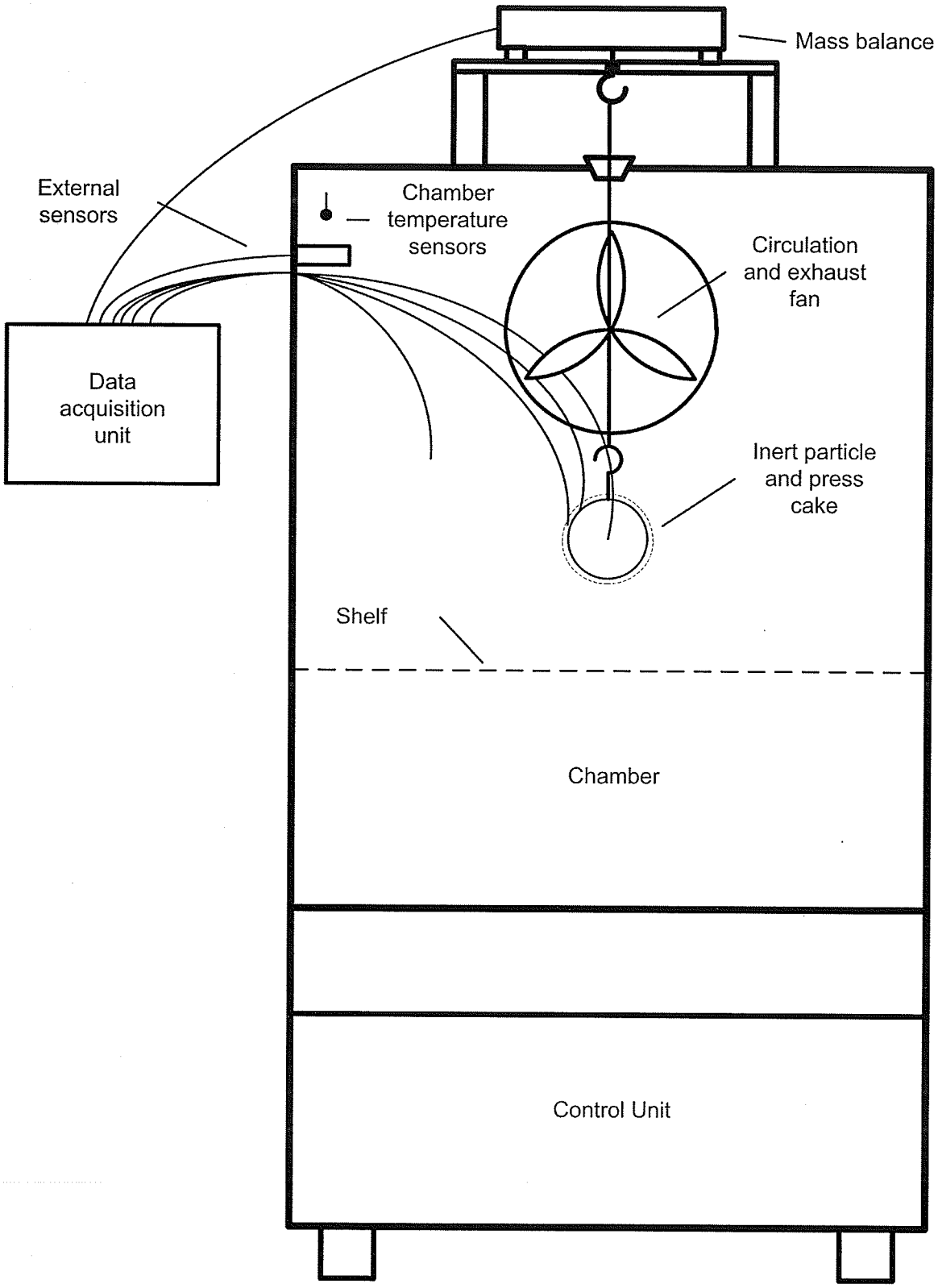


Figure 5.1 Schematic of experimental drying apparatus.

Table 5.1 Environmental chamber drying settings

Temperature °C	Relative humidity %		
50	57	62	70
60	45	57	70
70	36	45	57
80	36	45	57

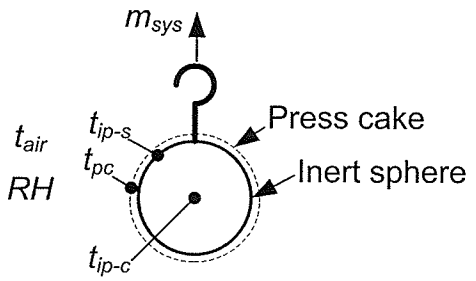


Figure 5.2 Schematic of inert sphere and press cake system. System includes hook and thermocouples (not shown). Measured and recorded parameters include RH = relative humidity; m_{sys} = mass of inert sphere and press cake system (includes hook and attached thermocouples); t_{air} = temperature of the drying air in the chamber; t_{ip-c} = temperature at the geometric centre of the inert sphere; t_{ip-s} = temperature of the inside surface of the sphere, and t_{pc} = temperature of the press cake layer.

The trials were concluded once the suspended system mass differed by less than ± 0.01g in a 30 min time interval. Another 40 mL sample was dried in a 9.8 mm x 9.8 mm x 2.5 mm layer on a flat teflon and glass nonstick sheet (Teflex TF14, Excalibur Products, Sacramento, CA), placed on a wire mesh tray in the chamber for the duration of two drying cycles to determine the equilibrium moisture content for every drying condition. Three drying trials used to validate the model were conducted for temperature and relative humidity settings of 55°C-55% RH; 65°C-45% RH, and 75°C-43% RH.

5.2 Development of the temperature prediction model

5.2.1 Semi-empirical analysis of the drying process

The approach taken for the determination of press cake temperature throughout the drying process of a thin layer of material on an inert sphere included the analysis of the energy balance across the press cake and inert sphere system (Fig. 5.3). Equation 5.1, provided a balance between the incoming energy (q_{in}) and the energy expended as both latent (q_{latent}) and sensible heat ($q_{sensible}$) by the press cake and inert sphere system with no energy generation.

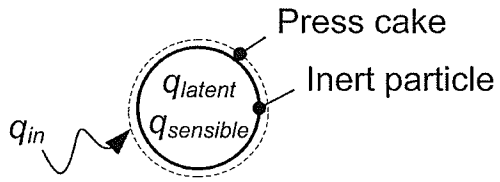


Figure 5.3 Schematic of heat and mass transfer for the sea buckthorn press cake and inert sphere system. The parameters q_{in} = incoming heat energy; q_{latent} = expended latent heat energy (press cake only), and $q_{sensible}$ = expended sensible heat energy (inert sphere and press cake).

$$q_{in} = q_{latent} + q_{sensible} \tag{5.1}$$

Due to the heat and mass transfer processes changing with time, the steady state thermal energy equations were solved for incremental time intervals ($\Delta\tau$) (Eqn. 5.2):

$$\Delta\tau = \tau_i - \tau_{i-1} \tag{5.2}$$

where subscripts (i-1) and (i) represented the beginning and end of each interval, respectively, creating a step process.

An expanded form of Eqn. 5.1 is presented by Eqn. 5.3a:

$$h\,A\,(t_{air} - t_{pc(i-1)})\,\Delta\tau = \left(\Delta m_{H_2O(i)}\,h_{fg}\right) + \left[m_{pc(i)}\,Cp_{pc(i)}\left(t_{pc(i)} - t_{pc(i-1)}\right) + m_{ip}\,Cp_{ip}\left(t_{ip(i)} - t_{ip(i-1)}\right)\right] \quad (5.3a)$$

where $Cp_{pc(i)}$ = the specific heat capacity of the press cake at time i; $\Delta m_{H_2O(i)}$ = the moisture loss from the press cake for time interval $\Delta\tau$; m_{ip} = the mass of the inert sphere; $m_{pc(i)}$ = the mass of the press cake at time i; $t_{ip(i)}$ = the temperature of the inert sphere at time i; $t_{ip(i-1)}$ = the temperature of the inert sphere at time i-1; $t_{pc(i)}$ = the temperature of the press cake at time i, and $t_{pc(i-1)}$ = the temperature of the press cake at time i-1.

To simplify the model it was assumed that the inert sphere temperature, t_{ip} (representative of both $t_{ip\text{-}c}$ and $t_{ip\text{-}s}$), and t_{pc}, were approximately the same after an initial warm-up period (Eqns. 5.3b-c):

$$t_{ip(i)} \cong t_{pc(i)} \quad (5.3b)$$

$$t_{ip(i-1)} \cong t_{pc(i-1)} \quad (5.3c)$$

$$t_{pc(i)} = \frac{h\,A\,(t_{air} - t_{pc(i-1)})\,\Delta\tau - \Delta m_{H_2O(i)}\,h_{fg} + t_{pc(i-1)}\left(m_{pc(i)}\,Cp_{pc(i)} + m_{ip}\,Cp_{ip}\right)}{m_{pc(i)}\,Cp_{pc(i)} + m_{ip}\,Cp_{ip}} \quad (5.4)$$

Equation 5.4, a rearrangement of Eqns. (5.3a, b, and c), was developed to predict $t_{pc(i)}$ at every time interval. The solution for Eqn. 5.4 required the determination of several parameters including heat transfer coefficient, h, $Cp_{pc(i)}$, $m_{pc(i)}$, and $\Delta m_{H_2O(i)}$. The values of surface area, A, latent heat of vaporization, h_{fg}, m_{ip}, and Cp_{ip} were assumed to remain constant throughout the trials.

5.2.2 Model analysis and validation

A program developed using Matlab® (The Mathworks, Inc., Natick, MA, 2004) with an iterative time interval of 120 s for the 55°C trial and 60 s for the 65 and 75°C trials was employed to generate temperature prediction data (Appendix C.1). Statistical analysis was conducted using JMP IN Statistical Discovery Software. The statistical parameters, coefficient of determination, r^2, root mean square error, *RMSE* (Eqn. 5.5), and residuals (Eqn. 5.6) were used to determine the fit of the predicted to the experimental data:

$$RMSE = \left[\frac{1}{N} \sum_{i=1}^{N} \left(t_{pc-\exp(i)} - t_{pc-pred(i)} \right)^2 \right]^{1/2} \tag{5.5}$$

$$residuals = \sum_{i=1}^{N} \left(t_{pc-\exp(i)} - t_{pc-pred(i)} \right) \tag{5.6}$$

where $t_{pc\text{-}exp(i)}$ = experimental temperature of the press cake at time i; $t_{pc\text{-}pred(i)}$ = predicted temperature of the press cake at time i, and N = number of observations. High values for r^2, low values of *RMSE*, and non-patterned residuals centered around zero were used to represent a good fit. This assessment of fit was used for all models unless otherwise noted.

5.2.3 Determination of model parameters

Heat transfer coefficient, *h*

The governing dimensionless correlations in the estimation of heat transfer coefficient, h, include the Nusselt number (Nu) (Eqn. 5.7), Reynolds number (Re) (Eqn. 5.8), and Prandtl number (Pr) (Eqn. 5.9) (Fryer et al. 1997; Singh and Heldman 2001):

$$\mathrm{Nu} = \frac{h\,d}{\lambda_{air}} \tag{5.7}$$

$$\mathrm{Re} = \frac{\rho_{air}\,u\,d}{\mu} \tag{5.8}$$

$$\mathrm{Pr} = \frac{\mu\,Cp_{air}}{\lambda_{air}} \tag{5.9}$$

where Cp_{air} = specific heat capacity of the air; d = diameter of a body; λ_{air} = thermal conductivity of air; μ = viscosity of the air; ρ_{air} = density of the air, and u = velocity of the air. A commonly employed correlation for estimation of convective heat transfer coefficient used in conjunction with Eqns. 5.7 to 5.9 for gas flow past a single sphere is provided in Eqn. 5.10 (Brodkey and Hershey 1998; Saravacos and Maroulis 2001; Singh and Heldman 2001). This correlation is applicable for conditions in which 1<Re<70 000 and 0.6<Pr<400 (Singh and Heldman 2001).

$$\mathrm{Nu} = 2.0 + 0.6\,\mathrm{Re}^{\frac{1}{2}}\,\mathrm{Pr}^{\frac{1}{3}} \tag{5.10}$$

Fryer et al. (1997) also proposed a correlation (Eqn. 5.11) that was specifically developed for foods containing two phases:

$$\mathrm{Nu} = 2.0 + 0.7\,\mathrm{Re}^{\frac{1}{2}}\,\mathrm{Pr}^{\frac{1}{3}} \tag{5.11}$$

Transient press cake mass, $m_{pc(i)}$

Formulation of a model to predict transient press cake mass, $m_{pc(i)}$, was facilitated using terms for transient moisture content ($M_{(i)}$) (based on Eqn. 2.1), transient moisture ratio ($MR_{(i)}$) (based on Eqn. 2.15), and dried press cake mass ($m_{pc(f)}$) (Eqn. 5.12):

$$m_{pc(f)} = \frac{m_{pc(o)}}{M_o + 1} \tag{5.12}$$

where $m_{pc(o)}$ = initial mass of the press cake. Solving for $m_{pc(i)}$ using Eqns. 2.1, 2.15, and 5.12, resulted in the development of the $m_{pc(i)}$ model, Eqn. 5.13. Refer to Appendix C.2 for complete derivation of Eqns. 5.12-5.13.

$$m_{pc(i)} = \frac{\left[MR_{(i)}\left(M_o - M_e\right) + M_e + 1\right]}{M_o + 1} m_{pc(o)} \tag{5.13}$$

Determination of transient moisture ratio, $MR_{(i)}$. Many empirical models and their respective coefficients have been developed for the determination of time dependent moisture ratio for specific products. The Lewis (Eqn. 2.20), Page (Eqn. 2.21), Henderson and Pabis (Eqn. 2.23), and logarithmic (Eqn. 2.25) models were selected based on their simplicity and their acceptability for the modelling of olive press cake, a material similar in composition to sea buckthorn press cake (Akgun and Doymaz 2005; Celma et al. 2007; Doymaz et al. 2004).

The experimental data accumulated from the drying trials listed in Table 5.1 were analysed to determine moisture ratio as expressed in Eqn. 2.15. The coefficients *a, c, k,* and *n* for the Lewis, Page, Henderson and Pabis, and logarithmic models were determined for each experimental trial using non-linear regression analysis and then expressed in terms of the variables [temperature (°C) and relative humidity in decimal (dec.) form], using linear regression techniques.

Determination of equilibrium moisture content, M_e. Equilibrium moisture content prediction models recommended by ASAE Standard D245.5 (ASABE 2006) that include effects of both temperature and relative humidity are the modified Henderson (Eqn. 5.14)

(Thompson et al. 1968), modified Chung-Pfost (Eqn. 5.15) (Pfost et al. 1976), modified Halsey (Eqn. 5.16) (Iglesias and Chirife 1976), and modified Oswin (Eqn. 5.17) (Chen and Morey 1989):

$$M_e = \left[\frac{-\ln(1-RH)}{E(t_{air}+C)}\right]^{\frac{1}{B}} \tag{5.14}$$

$$M_e = -\frac{1}{B}\ln\left[\frac{-(t_{air}+C)}{E}\ln RH\right] \tag{5.15}$$

$$M_e = \left[\frac{-\exp(E+B\,t_{air})}{\ln RH}\right]^{\frac{1}{C}} \tag{5.16}$$

$$M_e = \left(\frac{RH}{1-RH}\right)^{\frac{1}{C}}(E+B\,t_{air}) \tag{5.17}$$

The constants B, C, and E were determined using a non-linear regression analysis based on results accrued from the trials listed in Table 5.1.

Initial moisture content, M_o, and initial press cake mass, $m_{pc(o)}$. Initial values for moisture content, M_o, and press cake mass, $m_{pc(o)}$, were determined experimentally from the validation trial data.

Determination of moisture loss, $\Delta m_{H_2O(i)}$

Assuming that all mass change is due to a loss of moisture, a determination of the moisture removed for a given time interval ($\Delta m_{H_2O(i)}$) (Eqn. 5.18) is based on press cake mass (Eqn. 5.13):

$$\Delta m_{H_2O(i)} = m_{pc(i-1)} - m_{pc(i)} \tag{5.18}$$

where $m_{pc(i-1)}$ = mass of the press cake at time i-1.

Determination of the specific heat capacity of sea buckthorn press cake, $Cp_{pc(i)}$

The transient specific heat capacity of press cake, $Cp_{pc(i)}$, (Eqn. 5.19) was estimated using the empirical equation proposed by Charm (1978) (Singh and Heldman 2001):

$$Cp_{pc(i)} = 2.093X^{m}_{fat(i)} + 1.256X^{m}_{sol(i)} + 4.187X^{m}_{H_2O(i)} \tag{5.19}$$

where $X^{m}_{fat(i)}$ = mass fraction of fat component at time i; $X^{m}_{sol(i)}$ = mass fraction of solids component at time i, and $X^{m}_{H_2O(i)}$ = mass fraction of water component at time i. The transient mass fractions of fat, solids, and water and total mass of the press cake portions, $m_{pc\text{-}tot(i)}$, are presented in Eqns. 5.20-5.23, respectively:

$$X^{m}_{fat(i)} = \frac{m_{pc-fat}}{m_{pc-tot(i)}} \tag{5.20}$$

$$X^{m}_{sol(i)} = \frac{m_{pc-sol}}{m_{pc-tot(i)}} \tag{5.21}$$

$$X^{m}_{H_2O(i)} = \frac{m_{pc-H_2O(i)}}{m_{pc-tot(i)}} \tag{5.22}$$

$$m_{pc-tot(i)} = m_{pc-fat} + m_{pc-sol} + m_{pc-H_2O(i)} \tag{5.23}$$

where $m_{pc\text{-}fat}$ = mass of the fat portion of the press cake; $m_{pc\text{-}sol}$ = mass of the solids portion of the press cake, and $m_{pc-H_2O(i)}$ = mass of the water portion of the press cake at time i. Although $m_{pc\text{-}fat}$ and $m_{pc\text{-}sol}$ remained constant, $m_{pc-H_2O(i)}$ and subsequently $m_{pc\text{-}tot(i)}$ decreased throughout the drying process.

5.3 Model parameter determination and validation

5.3.1 Preliminary selection or development of key parameters

Validation of the accuracy of the temperature prediction model (Eqn. 5.4) required the preliminary selection or development of best fit correlations or models for key parameters h, $m_{pc(i)}$, and $Cp_{pc(i)}$.

Determination of heat transfer coefficient, *h*

Values for heat transfer coefficient, h, determined using Eqns. 5.10 and 5.11 were 15.7 to 15.8 and 18.2 W/(m^2·K), respectively. The data used in the calculations are provided in Appendix C.3. Because the air temperatures used in this research ranged from 50 to 80°C, the values for h were lower than the 20 to 80 W/(m^2·K) reported for heat transfer coefficients in baking ovens (Saravacos and Maroulis 2001).

Determination of transient mass of the press cake, $m_{pc(i)}$

Model and coefficient selection for moisture ratio, $MR_{(i)}$. The coefficients for the evaluated prediction models (Lewis, Page, Henderson and Pabis, and logarithmic) are provided by Eqns. 5.24 – 5.31:

Lewis:

$$k = 0.02562t_{air} - 0.03696RH\,t_{air} + 1.9326RH - 1.2124 \qquad (5.24)$$

Page:

$$k = 0.01908t_{air} - 0.03178RH\,t_{air} + 1.5852RH - 0.8284 \qquad (5.25)$$

$$n = 0.01190 t_{air} + 0.001462 RH\, t_{air} + 0.1657 RH + 0.2475 \quad (5.26)$$

Henderson and Pabis:

$$a = -0.00223 t_{air} + 0.01166 RH\, t_{air} - 0.7873 RH + 1.2633 \quad (5.27)$$

$$k = 0.02957 t_{air} - 0.04158 RH\, t_{air} + 2.1818 RH - 1.4187 \quad (5.28)$$

logarithmic:

$$a = 0.0043 t_{air} + 0.002298 RH\, t_{air} - 0.1141 RH + 0.8106 \quad (5.29)$$

$$k = 0.01739 t_{air} - 0.02494 RH\, t_{air} + 0.9936 RH - 0.5785 \quad (5.30)$$

$$c = -0.006174 t_{air} + 0.003758 RH\, t_{air} - 0.4257 RH + 0.4768 \quad (5.31)$$

These coefficients are applicable for a start time of $\tau = 1.33$ h and an $MR \geq 0.039$, the period during which steady state conditions (i.e. t_{air} and RH) and the first falling rate were achieved for the trials. While all models showed patterned residuals at some point during the drying period, consistently high values for r^2 and low values of $RMSE$ were achieved (Table 5.2). The Lewis and Page models were selected as the best fit for the validation trials, however, the Page model provided a smaller range of residuals for the validation trial of 75°C-43% RH. Model fitting and assessment summaries are provided in Appendix C.4.

Table 5.2 Statistical analysis of moisture ratio, *MR*, model fit for validation trials

Model	t_{air}[a] °C	RH[b] dec.	N[c]	r^2[d]	$RMSE$[e]
Lewis	55	0.55	41	0.9999	0.0119
	65	0.45	25	0.9999	0.0112
	75	0.43	15	0.9989	0.0260
Page	55	0.55	41	0.9998	0.0117
	65	0.45	25	0.9997	0.0158
	75	0.43	15	0.9998	0.0110
Henderson and Pabis	55	0.55	41	0.9998	0.0174
	65	0.45	25	0.9985	0.0149
	75	0.43	15	0.9990	0.0120
logarithmic	55	0.55	41	0.9969	0.0152
	65	0.45	25	0.9985	0.0204
	75	0.43	15	0.9989	0.0089

[a] t_{air} = temperature of the chamber air.
[b] RH = relative humidity of the chamber air, decimal basis (dec.).
[c] N = number of observations.
[d] r^2 = coefficient of determination.
[e] $RMSE$ = root mean square error.

Model and coefficient selection for equilibrium moisture content, M_e. The coefficients for the evaluated equilibrium moisture content models, modified Henderson, modified Chung-Pfost, modified Halsey, and modified Oswin are summarized in Table 5.3. Refer to Table C.4.3 (Appendix C.4) for data used in the determination of coefficients *B*, *C*, and *E*. The modified Chung-Pfost model provided best fit conditions based on a high r^2, low value for *RMSE* (Table 5.4), and randomized residuals. This model also provided a good fit for the prediction of moisture content for products such as mungbean (Chowdhury et al. 2005) and rough rice (Basunia and Abe 2000; San Martin et al. 2001).

Table 5.3 Equilibium moisture content, M_e, coefficients for validation trials

Model[a]	Coefficient values		
	E	B	C
Modified Henderson $M_e = \left[\frac{-\ln(1-RH)}{E(t_{air}+C)}\right]^{\frac{1}{B}}$	0.0621	0.7404	35.9872
Modified Chung-Pfost $M_e = -\frac{1}{B}\ln\left[\frac{-(t_{air}+C)}{E}\ln RH\right]$	113.5669	13.2336	12.6304
Modified Halsey $M_e = \left[\frac{-\exp(E+B\,t_{air})}{\ln RH}\right]^{\frac{1}{C}}$	-2.1330	-0.0131	0.8791
Modified Oswin $M_e = \left(\frac{RH}{1-RH}\right)^{\frac{1}{C}}(E+B\,t_{air})$	0.1037	-0.0008	1.1417

[a] Model nomenclature: M_e = equilibrium moisture content of the press cake; t_{air} = dry bulb temperature of drying air in chamber; RH = relative humidity on a decimal basis (dec.), and B, C, and E = coefficients.

Table 5.4 Equilibrium moisture content data for validation trials expressed in [mass/mass (w/w)] on a dry basis (db)

t_{air}[a]	RH[b]	$M_{e\text{-}exp}$[c]	$M_{e\text{-}pred}$ for models 1-4[d]			
°C	dec.		1	2	3	4
55	0.55	0.0846	0.0711	0.0780	0.0699	0.0714
65	0.45	0.0562	0.0418	0.0458	0.0433	0.0436
75	0.43	0.0427	0.0338	0.0324	0.0350	0.0344
			Statistical results between $M_{e\text{-}exp}$ and $M_{e\text{-}pred}$			
r^2[e]			0.987	0.999	0.992	0.993
$RMSE$[f]			0.0125	0.0066	0.0086	0.0082

[a] t_{air} = dry bulb temperature of air in drying chamber.
[b] RH = relative humidity of air in drying chamber, decimal (dec.)
[c] $M_{e\text{-}exp}$ = equilibrium moisture content of press cake.
[d] $M_{e\text{-}pred}$ = equilibrium moisture content of press cake, predicted by models 1-4, where E, B, and C are predetermined coefficients.
Model 1 – Modified Henderson; Model 2 – Modified Chung-Pfost; Model 3 – Modified Halsey, and Model 4 – Modified Oswin.
[e] r^2 = coefficient of determination.
[f] $RMSE$ = residual mean squared error.

Validation and analysis of transient mass of the press cake, $m_{pc(i)}$. The values for initial moisture content, M_o, and press cake mass, $m_{pc(o)}$, used in the determination of transient mass of the press cake, $m_{pc(i)}$, are provided in Table 5.5. Both the Lewis and Page based models provided a good fit for all validation trials (Figs. 5.4 and 5.6) determined by high values of r^2 (>0.998) and low values of $RMSE$ (<0.486x10^{-3} kg) (Table 5.6). In all cases, the predicted data were within 3.6 to 0.5% of the experimental data.

Referring to residuals (Figs. 5.5 and 5.7), the Lewis based model slightly over-predicted the values for 55°C, under-predicted the values for 65°C, and resulted in a sinusoidal shaped curve for 75°C with residuals ranging from -0.524x10^{-3} to 0.970x10^{-3} kg. The Page based model also slightly over-predicted the values for 55°C and under-

predicted the values for 65°C. The residual pattern was less distinct at 75°C for the Page predicted data and resulted in residuals ranging from -0.171×10^{-3} to 0.309×10^{-3} kg. The Page based model may be more applicable than the Lewis model for the 75°C temperature trial. Guan et al. (2005) reported that although non-randomized residuals were determined for a portion of the drying period for sea buckthorn leaves (dried at temperatures between 50-100°C), eventually the prediction model did converge toward the experimental data resulting in a good estimate of the final moisture content.

Table 5.5 Initial conditions for the temperature prediction model

	Validation trial[a]		
	V1	V2	V3
$m_{pc(o)}$ (kg)[b]	37.630×10^{-3}	35.816×10^{-3}	33.027×10^{-3}
M_o db[c]	1.337	1.314	1.414
$m_{pc\text{-}fat}$ (kg)[d]	1.407×10^{-3}	1.356×10^{-3}	1.199×10^{-3}
$m_{pc\text{-}sol}$ (kg)[e]	14.695×10^{-3}	14.123×10^{-3}	12.484×10^{-3}
$m_{pc-H_2O(o)}$ (kg)[f]	21.528×10^{-3}	20.336×10^{-3}	19.344×10^{-3}
$Cp_{pc(o)}$[J/(kg·K)][g]	2964	2952	3003
$t_{pc(o)}$ (°C)[h]	39.084	38.531	44.426

[a] Validation trial: V1 = 55°C - 55% RH; V2 = 65°C - 45% RH; V3 = 75°C - 43% RH.
[b] $m_{pc(o)}$ = Initial press cake mass.
[c] M_o db= Initial press cake moisture content, dry basis (db).
[d] $m_{pc\text{-}fat}$ = Mass of press cake fat component.
[e] $m_{pc\text{-}sol}$ = Mass of press cake solids component.
[f] $m_{pc-H_2O(o)}$ = Initial mass of press cake water component.
[g] $Cp_{pc(o)}$ = Initial specific heat capacity of press cake.
[h] $t_{pc(o)}$ = Initial press cake temperature.

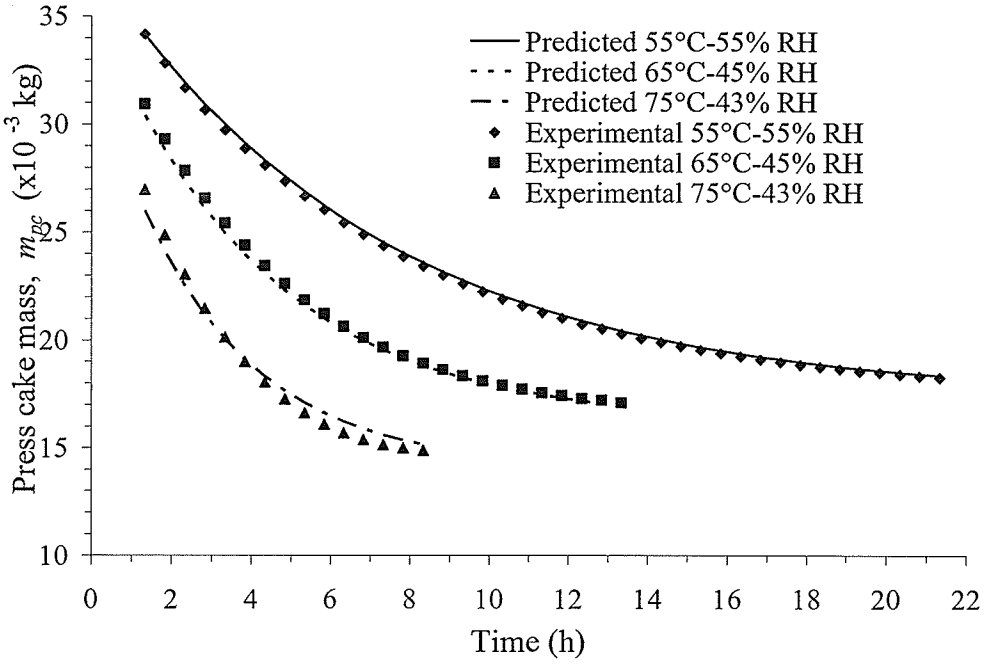


Figure 5.4 Press cake mass based on the Lewis model.

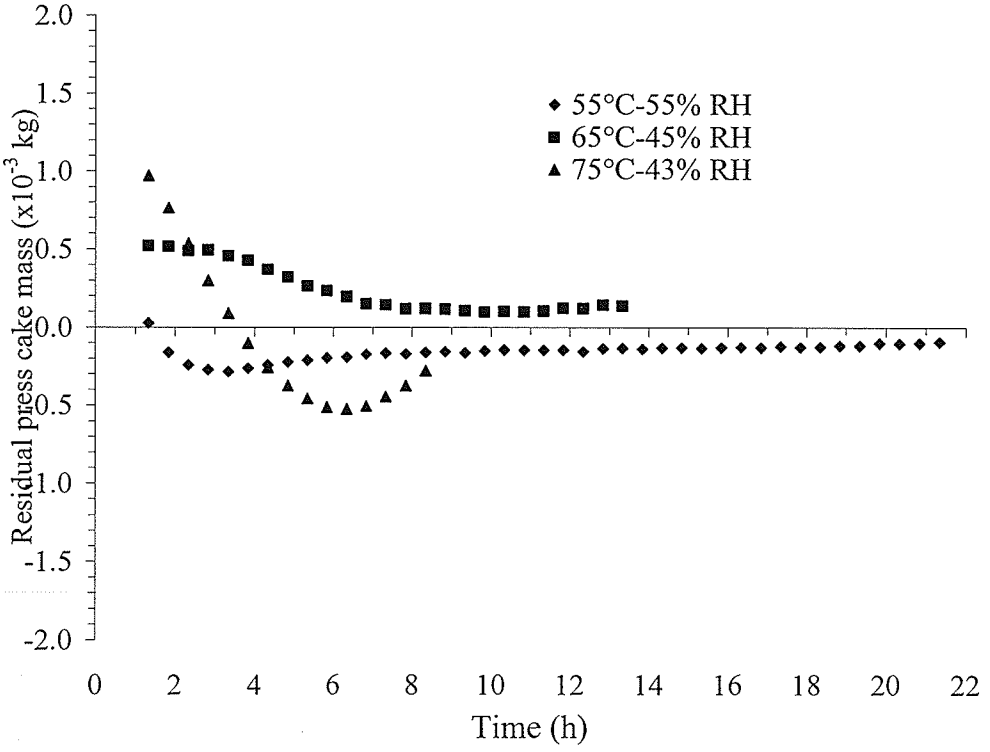


Figure 5.5 Residuals of press cake mass based on the Lewis model.

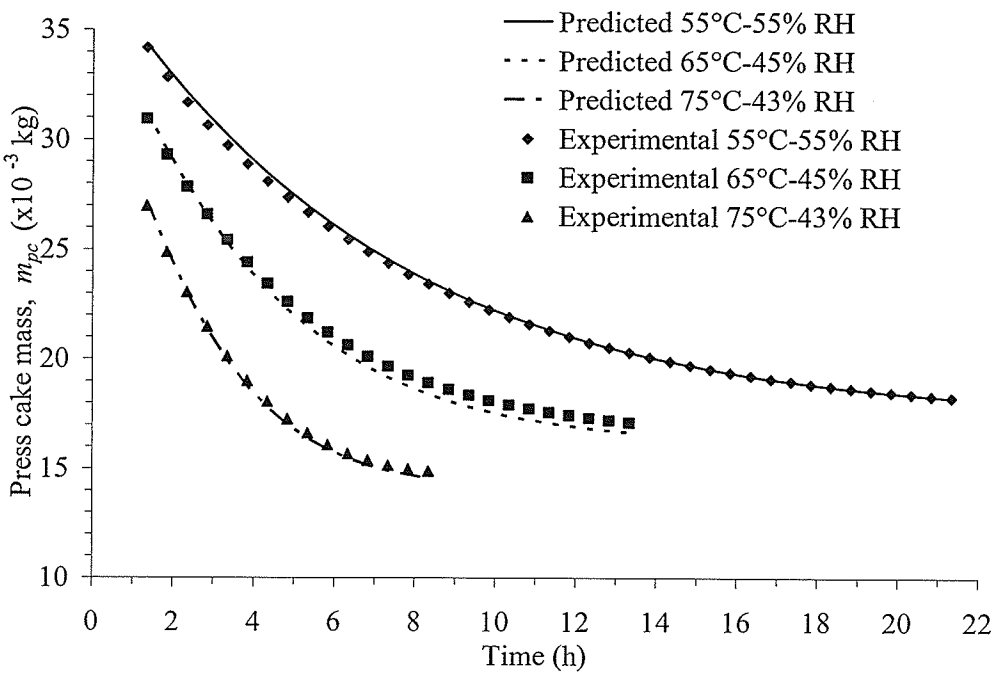


Figure 5.6 Press cake mass based on the Page model.

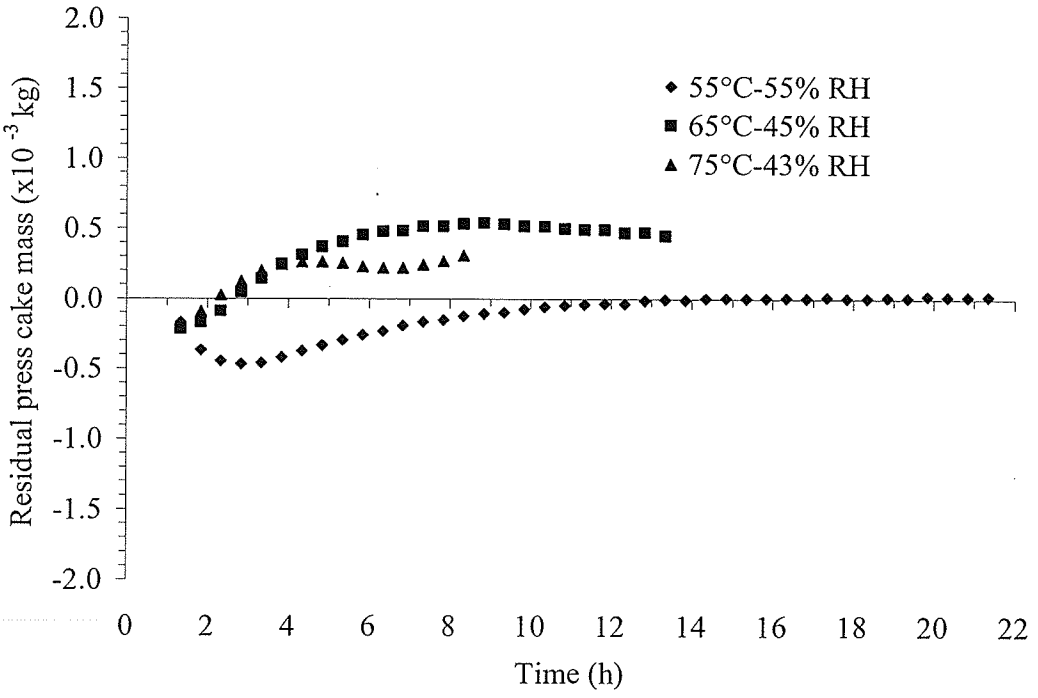


Figure 5.7 Residuals of press cake mass based on the Page model.

Table 5.6 Fit of press cake mass, $m_{pc(i)}$, model

Validation trial	Model basis	r^2[a]	$RMSE$[b] x10^{-3} kg
55°C - 55% RH	Lewis	0.9999	0.166
55°C - 55% RH	Page	0.9998	0.195
65°C - 45% RH	Lewis	0.9999	0.284
65°C - 45% RH	Page	0.9997	0.427
75°C - 43% RH	Lewis	0.9989	0.486
75°C - 43% RH	Page	0.9998	0.221

[a] r^2 = coefficient of determination.
[b] $RMSE$ = residual mean squared error.

Table 5.7 Constants for the temperature prediction model

Parameter	Value
Inert sphere/press cake surface area, A (x10^{-3}m^2)	9.78
Latent heat of vaporization, h_{fg} (x10^3 J/kg)[a]	2258
Specific heat of inert sphere, Cp_{ip} [J/(kg·K)][b]	1400
Mass of inert sphere, m_{ip} (kg)	0.148

[a] Incropera and DeWitt 1985.
[b] Dupont 1999.

5.3.2 Transient press cake temperature prediction model, $t_{pc(i)}$

Initial conditions and constants used in the temperature prediction model are summarized in Tables 5.5 and 5.7, respectively. The determination of specific heat capacity of the press cake, $Cp_{pc(i)}$, is provided in Appendix C.5. A 15.5% difference in heat transfer coefficient, h, (between values determined by Eqns. 5.10 and 5.11), resulted in a 1.4 to 3.0°C difference in press cake temperature occurring at τ = 1.33 h with temperatures eventually converging toward the experimental data. This finding provided an indication that the value of h has an influence on the solution for press cake temperature. Since Eqn. 5.11 provided results closer to the experimental data,

h = 18.2 W/(m^2·K) was used in the remainder of this discussion. Refer to Appendix C.6 for press cake temperature results using the h value determined by Eqn. 5.10.

A gradual convergence of the predicted to the experimental press cake temperatures occurred for all validation trial conditions, however, the convergence was more apparent at lower temperatures of 55 and 65°C (Figs. 5.8 and 5.10). Both the Lewis and Page based models provided a good fit for the two lower temperature validation trials based on high values of r^2 (> 0.984) and relatively low values of *RMSE* (< 1.7°C) (Table 5.8). The 75°C temperature trial was not as well predicted according to *RMSE* values of 3.1 and 4.1°C, for the Lewis and Page based models, respectively. Both the Lewis and Page based models resulted in a general under-prediction of values for all validation trials, except for the 65°C trial between 11 and 13.3 h (Figs. 5.9 and 5.11).

Residuals ranged from (3.1 to 0.1°C), (4.3 to -0.4°C), and (6.7 to 0.3°C) for the 55, 65, and 75°C trials, respectively, for the Lewis based model. Similarly, for the Page model, residuals ranged from (2.9 to 0.1°C), (3.5 to -0.6°C), and (6.4 to ~0°C) for the 55, 65, and 75°C trials, respectively. Predicted temperatures using the Lewis based model were within ±5.0% of the experimental temperatures by 3.1, 2.3, and 3.3 h for the 55, 65, and 75°C trials, respectively. The Page based model resulted in slightly later drying times of 3.3, 3.0, and 4.5 h at which the predicted and experimental temperatures were within ±5.0% for the 55, 65, and 75°C trials, respectively. Residual patterns were determined not to be affected by the initial press cake temperature.

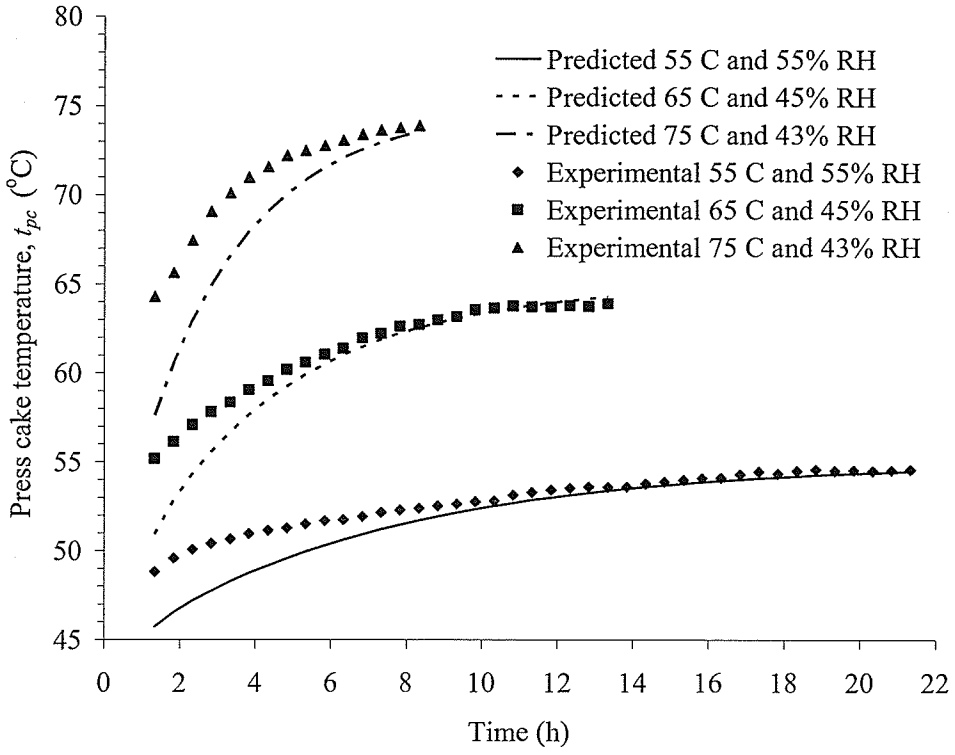


Figure 5.8 Press cake temperature based on the Lewis model.

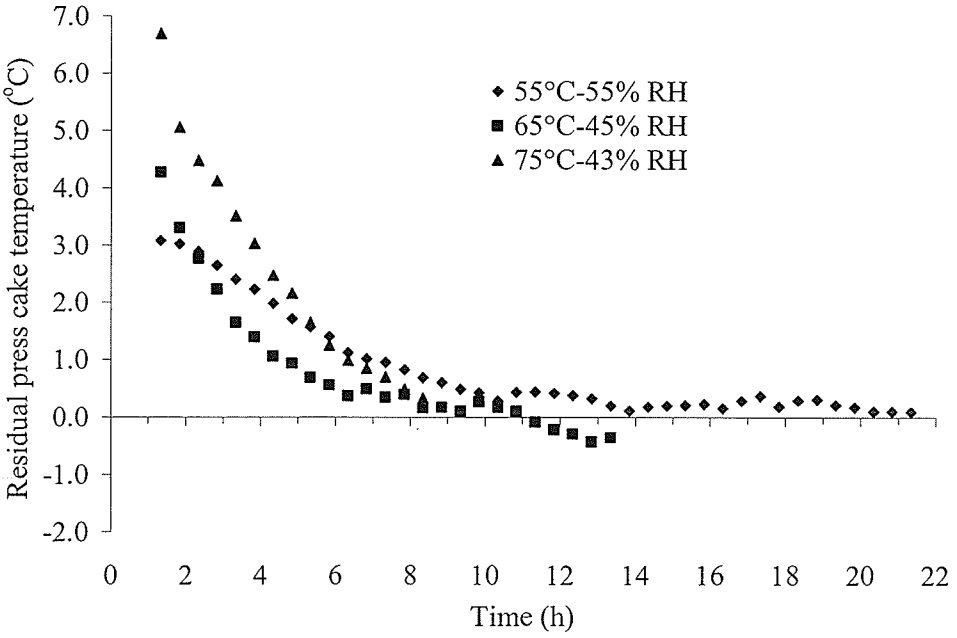


Figure 5.9 Residuals of press cake temperature based on the Lewis model.

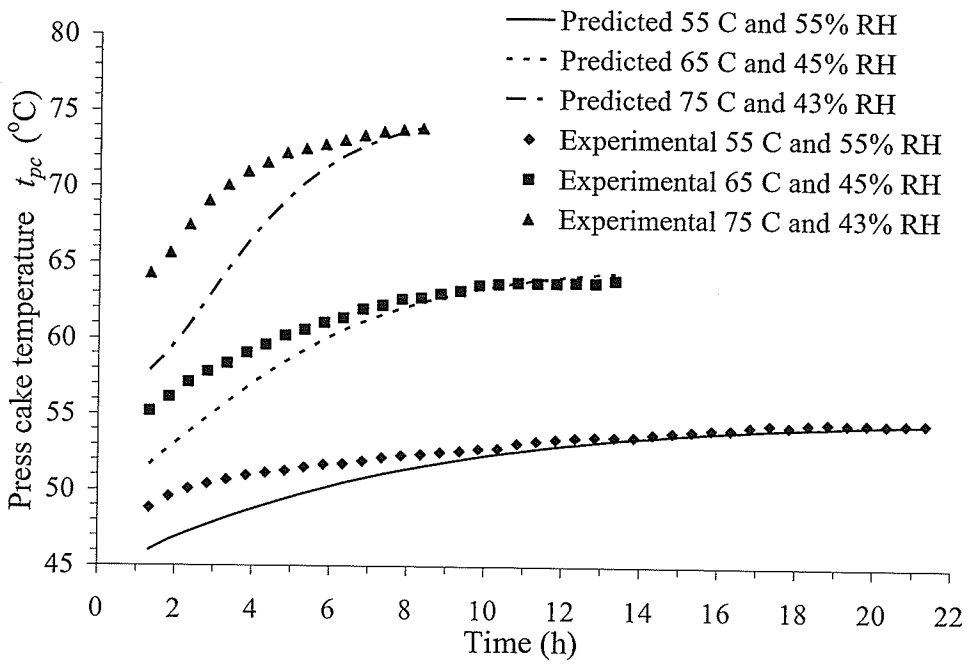


Figure 5.10 Press cake temperature based on the Page model.

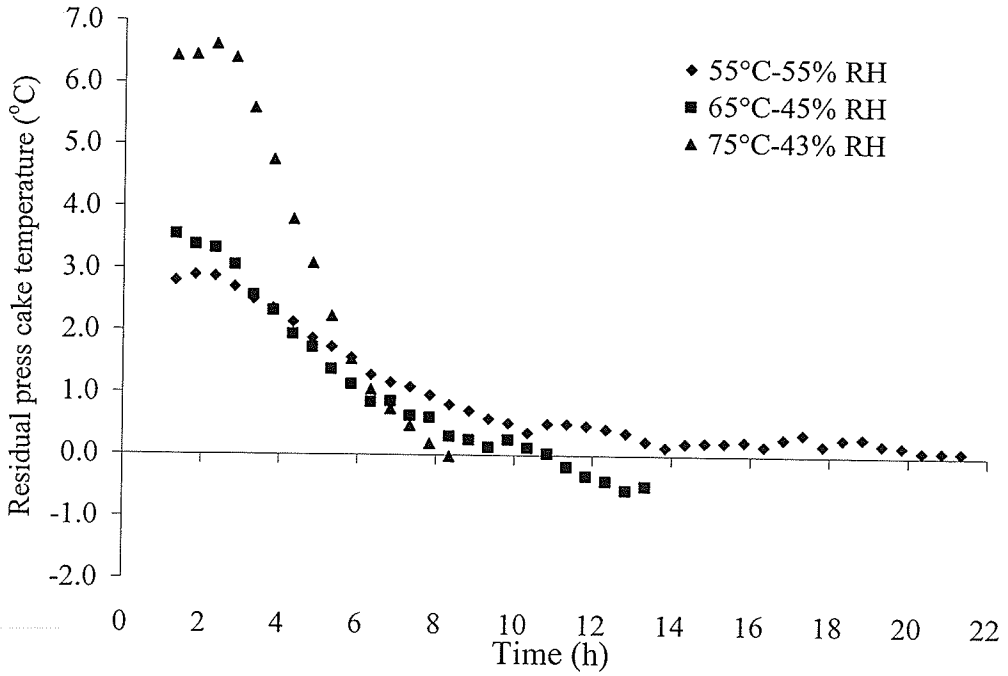


Figure 5.11 Residuals of press cake temperature based on the Page model.

Table 5.8 Fit of the temperature prediction model

Validation trial	Model basis	h[a] $W/(m^2 \cdot K)$	r^2[b]	$RMSE$[c] °C
55°C - 55% RH	Lewis	18.2	0.9850	1.2
55°C - 55% RH	Page	18.2	0.9875	1.3
65°C - 45% RH	Lewis	18.2	0.9929	1.4
65°C - 45% RH	Page	18.2	0.9971	1.7
75°C - 43% RH	Lewis	18.2	0.9943	3.1
75°C - 43% RH	Page	18.2	0.9574	4.1

[a] h = heat transfer coefficient.
[b] r^2 = coefficient of determination.
[c] $RMSE$ = residual mean squared error.

5.3.3 Applicability of the temperature prediction model

The accuracy achieved at the stage during which the press cake temperature approached that of the drying air indicated potential for the application of the proposed model to the prediction of thin layer drying on an inert sphere. During later drying stages, the heat transferred to the product is no longer being expended as sensible heat but only as latent heat (Pabis et al. 1998). This is also when product degradation can occur, therefore prediction of the onset of this stage is useful in quality control of the drying process.

Suitability of this model for the prediction of product temperature at earlier drying stages may have been influenced by the assumptions and models employed. The assumption that the press cake temperature was equal to that of the inert sphere soon after drying began was applicable to the situation for which this model was developed (i.e. inert sphere size 4 to 8 mm in diameter). In the experimental situation, the 50.8 mm diameter inert sphere was determined to have a Biot number (Eqn. 2.8) equal to 1.59-1.85

(Appendix C.3) which is within 0.1 to 40, the range in which a temperature differential is possible between the surface and the centre of the inert sphere. The temperature difference between the press cake and the centre of the inert sphere for each validation trial is provided in Fig. 5.12. Based on the temperature differentials being within 1°C between the start (τ = 1.3 h) and finish time for both the 55 and 65°C trials, the assumption used in the model would be valid. However, for the 75°C trial the temperature differential varied between 1.2 and 3.5°C and was not within 1°C until ~7.1 h. While inert sphere temperature may have had partial impact on the experimental temperature data, especially at higher drying temperatures (75°C), other parameters such as heat transfer coefficient may have influenced the final results. Due to the good fit of the predicted press cake mass values, the models involved with the determination of mass were deemed to not have significant influence on the predicted temperatures.

Visually, drying temperature prediction was most accurate towards the later part of each analysis period. However, differences between predicted and experimental temperature of the press cake did not exceed ~10% at any time for the 75°C trial and were less than 8 and 7% for the 65 and 55°C trials. The simplified approach to drying modelling used in this research also achieved comparable accuracy in other applications (Grbavcic et al. 2004; Leontieva et al. 2002).

Prediction accuracies of 7.5 and 8.5% were reported for the analysis of moisture content of aqueous suspensions dried on an inert particle in lab-scale and industrial trials, respectively (Leontieva et al. 2002). The performance of a fluidized bed of inert particles for the drying of a variety of slurries was predicted with mean absolute deviations

between experimental and calculated values of 5.8% while 85% of the data was within ±10% (Grbavcic et al. 2004). Although, the model developed by Konovalov et al. (2003) was more theoretically based, similar prediction accuracies were reported. The model was validated for several products (i.e. meat processing sludge, heavy corn steep water, gelatin, starch, sugar, salt, combined latex emulsion, P-salt, gamma acid, dispersing and bleaching agents). Differences between simulated and experimental results for temperature, moisture content, and drying time did not exceed 10%.

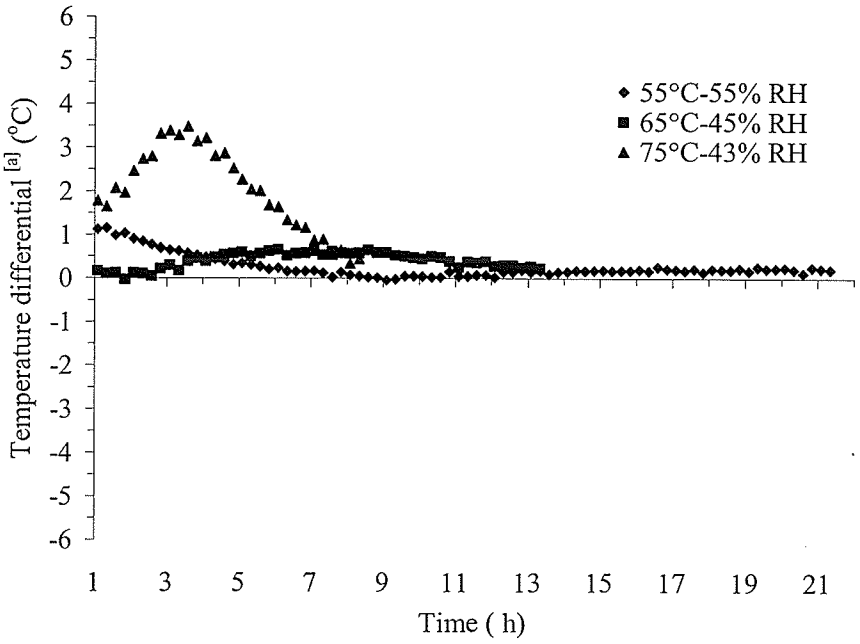


Figure 5.12 Experimental temperature differential between the press cake and centre of inert sphere at different drying conditions.
[a] Temperature differential = $t_{pc} - t_{ip\text{-}c}$; t_{pc} = temperature of the press cake, and $t_{ip\text{-}c}$ = temperature at the geometric centre of the inert sphere.

5.4 Conclusions of model analysis

A semi-empirical model for the temperature prediction of a thin layer of material dried on an inert sphere was developed, solved, and validated. The model was developed based on simple heat and mass balance equations. Solving the model required the selection of models and equations for the determination of moisture ratio, equilibrium moisture content, heat transfer coefficient, and specific heat capacity. Once fully developed the model was validated using trials conducted for temperature and relative humidity settings of 55°C-55% RH, 65°C-45% RH, and 75°C-43% RH.

The determination of moisture ratio was based on the Lewis and Page models and the equilibrium moisture content on the modified Chung-Pfost model. These equations embedded within the model allowed for the accurate determination of press cake mass. The Lewis and Page based models provided a good fit (r^2>0.999 and $RMSE$<0.427x10^{-3} kg) of experimental data at both 55 and 65°C, however, the Page model provided the better fit at 75°C, due to less patterning of the residuals. The Lewis and Page models both slightly over-predicted the results for 55°C and under-predicted the results for 65°C.

Two equations were employed for the determination of heat transfer coefficient and the 15.5% difference between the final values resulted in 1.4 to 3.0°C difference in press cake temperature at the start of the analysis period. Although press cake temperatures were under-predicted and did eventually converge toward the experimental temperatures for both calculated coefficients, heat transfer coefficient was noted to have an influence on the solution for press cake temperature. A gradual convergence toward experimental

values occurred at all temperature and relative humidity conditions, however, was most apparent at the lower temperatures of 55 and 65°C. While predicted temperatures did not exceed ±10% of experimental values, all values were within 5% by 3.1, 2.3, and 3.3 h for 55, 65, and 75°C (Lewis model) and 3.3, 3.0, and 4.5 h for 55, 65, and 75°C (Page model).

6 CONCLUSIONS

The general conclusions for this thesis work were summarized as follows:

No significant difference occurred between harvest times for the parameters (i.e. seed size and bioactive content) measured for the seed fraction of sea buckthorn berries. This may be an indication that the development of the seed (including oil composition) occurred early in the fruit development stage, prior to early maturity as tested in this research. Therefore for optimum seed quality, berries can be harvested at any point during the evaluated period (early to post-maturity). Conversely, harvest time did have a significant effect on the fruit fraction of the berries. Major compounds, α-tocopherol and β-sitosterol at levels of 388 and 928 mg/100 g oil were highest at early maturity, signifying early synthesis. Due to complete fruit development and ripening, highest levels of measured parameters: berry size (19.4 g%), redness (+20.2), and total carotenoids (817.8 mg/100 g oil), occurred at maturity. Harvest at post-maturity resulted in an overall lower quality of fruit fraction oil due to losses ranging from 13.9% (a^*) to 20.2% (total tocols mainly attributed to α-tocopherol), and 24.6% for total carotenoids.

Time of drying of press cake to an approximate content of 7% wb ranged from 24.1h at 50°C–58.7% RH to 4.0 h at 70°C–20.8% RH. As with harvest time, drying conditions also exhibited varying effects on different quality parameters. In the case of lower temperatures or higher humidities or both, decreased drying rates and increased exposure time occurred, contributing to the effect of the drying conditions. The sample dried at 60°C-24.4% RH had a drying time of 6.4 h and the best colour retention (ΔE = 2.0) as

compared to fresh pulp and peel. The improvement of colour retention at this level compared with the lower temperature condition of 50°C-30.6% RH may have been due to the deactivation of enzymes or a 5 h decrease in drying time or both. Increasing the relative humidity to 57.0–58.7% RH resulted in significant darkening (ΔE = 7.0 to 14.9) of the pulp and peel. This darkening did not coincide with a loss of carotenoids but instead with improved retention or regeneration of α-tocopherol, β-tocopherol, and total tocols at temperatures > 50°C. The fatty acid profile remained quite stable between different drying conditions with only a slight decrease in palmitoleic acid levels at 70°C. Concentration of sterols also remained stable, although as with tocols, large variations (< 18.2%) between individual samples may have influenced the results.

To enable the analysis of an unsteady state condition using the proposed temperature prediction model, an iterative time step process was employed. This process allowed for the determination of the transient mass of the press cake and dependent parameters, transient specific heat capacity of the press cake, and the loss in moisture during each time interval. The Lewis or Page model (moisture ratio) in conjunction with the Chung-Pfost model (equilibrium moisture content) were used to provide an accurate prediction ($0.9989 < r^2 < 0.9999$) of transient mass of the press cake. The values for the transient temperature of the press cake were not as accurately predicted at early stages of the drying period resulting in differences of up to ±10, 8, and 7% for the 75, 65, and 55°C trials. As drying progressed beyond 3.1-4.5 h within the first falling rate period, predicted values improved to within 5% of the experimental values. Discrepancies in temperature prediction may be attributed to other parameters such as heat transfer coefficient, the value of which was determined to have an influence on the solution.

7 RECOMMENDATIONS FOR FUTURE WORK

Several recommendations can be offered for each of the phases investigated in this research either for the improvement of accuracy, for the applicability to other products or situations, and for the expansion of research scope.

Within phase I (Chapter 3), an investigation into the berry quality at smaller time intervals between early maturity and full maturity would assist in the determination of the optimal harvest time. This research conducted for several locations within the Prairies (or within Canada) as well as over a series of years would assist in evaluating the effect of location and climatic conditions on the quality of fruit. This quality research in conjunction with further work in non-winter harvest techniques would contribute greatly to the sea buckthorn industry.

Due to interesting results within phase II (Chapter 4), several recommendations can be made. For products exhibiting variability, such as sea buckthorn berry pulp and peel, number of replicates should be investigated and optimized to ensure good accuracy of results. Accurate determination of number of replicates prior to experimentation is important due to the cost and time involved with the preparation and bioactive testing of specialty products. Extra tests (for the determination of oxidation, Maillard browning, non-enzymatic and enzymatic reactions, and antioxidant capacity) could provide insight as to why certain compounds were preserved and others degraded. This knowledge accompanied by further experimentation with a greater range of drying temperatures and humidities can provide knowledge on how specific compounds can be optimized or

enhanced using the drying process. This is especially useful when a processing system is aimed at the extraction of a specific compound.

Within phase III (Chapter 5), since many of the parameters used in the model were based on recommended values or equations and models, improvements in model accuracy may be possible through the accurate assessment of product and fluid properties. These properties include specific heat of the product as well as the inert sphere, equilibrium moisture content of the product, heat transfer coefficient of the drying fluid, and the latent heat of vaporization. A sensitivity analysis would also indicate which factors should be further investigated to reduce the influence of uncertainty on the analysis. An example in this research includes the specific heat capacity of the press cake that was assumed to only vary with moisture content, however, several equations are available for the determination of specific heat based on temperature. This requires knowledge of the solids composition (e.g. protein, ash... etc.) of the product.

The model based on the 50.8 mm diameter inert sphere should be validated using a series of smaller inert spheres, to determine if inert sphere size had an effect on the accuracy of the model. This model should also be investigated for application of other products such as liquids, slurries, and pastes. Finally, if improved model accuracy is achieved on an individual sphere basis, validation trials could be conducted in an actual spouted bed or fluidized bed drying system with inert particles.

8 REFERENCES

Akgun, N.A. and I. Doymaz. 2005. Modelling of olive cake thin-layer drying process. *Journal of Food Engineering* 68:455-461.

Akpinar, E.K. 2006. Determination of suitable thin layer drying curve model for some vegetables and fruits. *Journal of Food Engineering* 73:75-84.

AOAC. 2002a. Fruits and fruit products. In *Official Methods of Analysis of AOAC International*, 17th ed., 1st rev., vol. 2, ed. W. Horwitz, 37.1.01-37.1.14. Gaithersburg, MD: Association of Official Analytical Chemists.

AOAC. 2002b. Vegetable products, processed. In *Official Methods of Analysis of AOAC International*, 17th ed., 1st rev., vol. 2, ed. W. Horwitz, 42.1.01-42.3.03. Gaithersburg, MD: Association of Official Analytical Chemists.

AOCS. 2000. Standard Ce 1-62. In *Official and Tentative Methods of the American Oil Chemists Society*, Champaign, IL: American Oil Chemists Society.

Arimboor, R., V.V. Venugopalan, K. Sarinkumar, C. Arumughan, and R.C. Sawhney. 2006. Integrated processing of fresh Indian sea buckthorn (*Hippophae rhamnoides*) berries and chemical evaluation of products. *Journal of the Science of Food and Agriculture* 86:2345-2353.

ASABE. 2006. ASAE D245.5 Oct1995 (R2001). In *American Society of Agricultural and Biological Engineers*: *Standards 2006*. St. Joseph, MI: American Society of Agricultural and Biological Engineers.

Barry, C.S. and J.J. Giovannoni. 2007. Ethylene and fruit ripening. *Journal of Plant Growth Regulation* 26:143-159.

Basunia, M.A. and T. Abe. 2000. Moisture desorption isotherms of medium-grain rough rice. *Journal of Stored Products Research* 37:205-219.

Becker, H.A. and H.R. Sallans. 1955. A study of internal moisture movement in the drying of wheat kernel. *Cereal Chemistry* 32:212-226. [Cited by Jayas et al. 1999]

Bell, L.N. 2001. Stability testing of nutraceuticals and functional foods. In *Handbook of Nutraceuticals and Functional Foods*, ed. R.E.C. Wildman, 501-516. Boca Raton, FL: CRC Press LLC.

Benali, M. 2004. Thermal drying of foods: Loss of nutritive content and spoilage issues. In *Dehydration of Products of Biological Origin*, ed. A.S. Mujumdar, 137-152. Enfield, NH: Science Publishers, Inc.

Benali, M. and T. Kudra. 2002. Advanced processing technologies for slurries: Preforming/drying/post-treatment. Paper number 2002-015/2002-01-15 National Resources Canada, Canmet Energy Technology Centre – Varennes <<http://cetc-varennes.nrcan.gc.ca/fichier.php/codectec/En/2002-015/2002-015_e.pdf>>. Accessed 2005/05/06.

Beveridge, T.H.J. 2003a. Post harvest handling and storage. In *Sea Buckthorn (Hippophae rhamnoides L.): Production and Utilization*, eds. T.S.C. Li and T.H.J. Beveridge, 69-77. Ottawa, ON: NRC Research Press.

Beveridge, T.H.J. 2003b. Chemical composition and some physical properties. In *Sea Buckthorn (Hippophae rhamnoides L.): Production and Utilization*, eds. T.S.C. Li and T.H.J. Beveridge, 79-88. Ottawa, ON: NRC Research Press.

Beveridge, T.H.J. 2003c. Processing and products. In *Sea Buckthorn (Hippophae rhamnoides L.): Production and Utilization*, eds. T.S.C. Li and T.H.J. Beveridge, 89-99. Ottawa, ON: NRC Research Press.

Beveridge, T.H.J. 2002. Opalescent and cloudy fruit juices: Formation and element stability. *Critical Reviews in Food Science and Nutrition* 42(4):317-337.

Beveridge, T., J.E. Harrison, and J. Drover. 2002. Processing effects on the composition of Sea Buckthorn juice from *Hippophae rhamnoides* L. cv. Indian Summer. *Journal of Agricultural Food Chemistry* 50:113-115.

Beveridge, T.H.J., T.S.C. Li, B.D. Oomah, and A. Smith. 1999. Sea buckthorn products: Manufacture and composition. *Journal of Agricultural Food Chemistry* 47:3480-3488.

Brodkey, R.S. and H.C. Hershey. 1998. *Transport Phenomena*: *A Unified Approach*. New York: NY: McGraw-Hill, Inc.

Bruce, D.M. 1985. Exposed-layer barley drying: Three models fitted to new data up to 150°C. *Journal of Agricultural Engineering Research* 32:337-347. [Cited by Jayas et al. 1991]

Cassini, A.S., L.D.F. Marczak, and C.P.Z. Noreña. 2006. Drying characteristics of textured soy protein. *International Journal of Food Science Technology* 41:1047-1053.

Celma, A.R., S. Rojas, F. Lopez, I. Montero, and T. Miranda. 2007. Thin-layer drying behavior of sludge of olive oil extraction. *Journal of Food Engineering* 80:1261-1271.

Cenkowski, S., R. Yakimishen, R. Przybylski, and W.E. Muir. 2006. Quality of extracted sea buckthorn seed and pulp oil. *Canadian Biosystems Engineering* 48:3.9-3.16.

Chan, J.C.C., P.C.K. Cheung, and P.O. Ang Jr. 1997. Comparative studies on the effect of three drying methods on the nutritional composition of seaweed *Sargassum hemiphyllum* (Turn.) C. Ag. *Journal of Agricultural Food Chemistry* 45:3056-3059.

Chen, C. and R.V. Morey. 1989. Comparison of four EMC/ERH equations. *Transactions of the American Society of Agricultural Engineers* 32:983-990. [Cited by Pabis et al. 1998; Sinicio et al. 1995]

Chowdhury, M.M.I, M.D. Huda, M.A. Hossain, and M.S. Hassan. 2006. Moisture sorption isotherms for mungbean (*Vigna radiata* L.). *Journal of Food Engineering* 74(4):462-467.

Cohen, M., R.S. Robinson, and H.N. Bhagavan. 2000. Antioxidants in dietary lipids. In *Fatty Acids in Foods and Their Health Implications*, 2nd ed., ed. C.K. Chow, 439-450. New York, NY: Marcel Dekker, Inc.

Costa Jr., E.F., M. Cardoso, and M.L. Passos. 2001. Simulation of drying suspensions in spout-fluid beds of inert particles. *Drying Technology* 19(8):1975-2001.

Crank, J. 1975. *The Mathematics of Diffusion*, 2nd ed. Oxford, UK: Clarendon Press.

Da Cunha, R.L., A.G. De La Cruz, and F.C. Menegalli. 2006. Effects of operating conditions on the quality of mango pulp dried in a spout fluidized bed. *Drying Technology* 24(4):423-432.

Delgado-Vargas, F. and O. Paredes-López. 2003. *Natural Colourants for Food and Nutraceutical Uses*. Boca Raton, FL: CRC Press LLC.

deMan, J.M. 2000. Chemical and Physical properties of fatty acids. In *Fatty Acids in Foods and Their Health Implications*, 2nd ed., ed. C.K. Chow, 17-46. New York, NY: Marcel Dekker, Inc.

deMan, J.M. 1999. *Principles of Food Chemistry,* 3rd ed.. Gaithersburg, MD: Aspen Publishers.

Dobson, G. 2002. Analysis of fatty acids in functional foods with emphasis on ω3 fatty acids and conjugated linoleic acid. In *Methods of Analysis for Functional Foods and Nutraceuticals*, ed. W.J. Hurst, 65-99. Boca Raton, FL: CRC Press LLC.

Doymaz, I., O. Gorel, and N.A. Akgun. 2004. Drying characteristics of the solid by-product of olive oil extraction. *Biosystems Engineering* 88(2):213-219.

DuPont.1999. *Teflon PTFE 850-A: Fluoropolymer Resin.* Product Information, DuPont Fluoroproducts <<http://www2.dupont.com/Teflon_Industrial/en_US/assets/downloads/h48634.pdf>>. Accessed 2006/09/20.

Eccleston, C., B. Yang, R. Tahvonen, H. Kallio, G.H. Rimbach, and A.M. Minihane. 2002. Effects of an antioxidant-rich juice (sea buckthorn) on risk factors for coronary heart disease in humans. *Journal of Nutritional Biochemistry* 13:346-354.

Eitenmiller, R. and J. Lee. 2004. *Vitamin E: Food Chemistry, Composition, and Analysis.* New York, NY: Marcel Dekker, Inc.

Erickson, M.C. 1997. Lipid oxidation: Flavor and nutritional quality deterioration in frozen foods. In *Quality in Frozen Food*, eds. M.C. Erickson and Y.-C. Hung, 141-173. New York, NY: Chapman & Hall.

Feng, H., J. Tang, D.S. Mattinson, and J.K. Fellman. 1999. Microwave and spouted bed drying of frozen blueberries: The effect of drying and pretreatment methods on physical properties and retention of flavour volatiles. *Journal of Food Processing and Preservation* 23:463-479.

Folch, J., M. Lees, and G.H.S. Stanley. 1957. A simple method for the isolation and purification of lipids from animal tissues. *Journal of Biological Chemistry* 226:497-509. [Cited by Dobson 2002]

Francis, F.J. 2003. Colour analysis. In *Food Analysis*, 3rd ed., ed. S.S. Nielsen, 529-541. New York, NY: Kluwer Academic/ Plenum Publishers.

Freire, F., A. Figueiredo, and P. Ferrao. 2001. Modelling high temperature, thin layer, drying kinetics of olive bagasse. *Journal of Agricultural Engineering Research* 78(4):397-406.

Fryer, P.J., D.L. Pyle, and C.D. Rielly. 1997. *Chemical Engineering for the Food Industry*. Cornwall, UK: Chapman and Hall.

Gao, X., M. Ohlander, N. Jeppsson, L. Bjork, and V. Trajkovski. 2000. Changes in antioxidants effects and their relationship to phytonutrients in fruits of sea buckthorn (*Hippophae rhamnoides* L.) during maturation. *Journal of Agricultural Food Chemistry* 48(5):1485-1490.

Gaetke, R. and E. Triquart. 1992. Pruning machine for mechanized harvest of sea buckthorn. *Gartenbau Mag.* 1:57-58. [Cited by Beveridge 2003a]

Ghazanfari, A., S. Emami, L.G. Tabil, and S. Panigrahi. 2006a. Thin-layer drying of flax fiber: I. Analysis of modeling using Fick's second law of diffusion. *Drying Technology* 24(12):1631-1635.

Ghazanfari, A., S. Emami, L.G. Tabil, and S. Panigrahi. 2006b. Thin-layer drying of flax fiber: II. Modeling drying process using semi-theoretical and empirical models. *Drying Technology* 24:1637-1642.

Gibson, G.R., and R. Fuller. 1998. The role of probiotics and prebiotics in the functional food concept. In *Functional Foods: The Consumer, the Products and the Evidence*, eds. M.J. Sadler and M. Saltmarsh, 3-4. Cambridge, UK: Cambridge University Press.

Göğüs, F. and M. Maskan. 2006. Air drying characteristics of solid waste (pomace) of olive oil processing. *Journal of Food Engineering* 72(4):378-382.

Grabowski, S., M. Marcotte, M. Poirier, and T. Kudra. 2002. Drying characteristics of osmotically pretreated cranberries: Energy and quality aspects. *Drying Technology* 20(10):1989-2004.

Grabowski, S., M. Marcotte, D. Quan, A.R. Taherian, M.R. Zareifard, M. Poirier, and T. Kudra. 2007. Kinetics and quality aspects of Canadian blueberries and cranberries dried by osmo-convective method. *Drying Technology* 25(2):367-374.

Grabowski, S., A.S. Mujumdar, H.S. Ramaswamy, and C. Strumillo. 1997. Evaluation of fluidized versus spouted bed drying of baker's yeast. *Drying Technology* 15(2):625-634.

Grbavcic, Z.B., Z.L. Arsenijevic, and R.V. Garic-Grulovic. 2004. Drying of slurries in fluidized bed of inert particles. *Drying Technology* 22(8):1793-1812.

Guan, T.Y., S. Cenkowski, and A. Hydamaka. 2005. Effect of drying on the nutraceutical quality of sea buckthorn (*Hippophae rhamnoides* L. ssp *sinensis*) leaves. *Journal of Food Science* 70(9):E514-E518.

Gutiérrez, L.-F., C. Ratti, and K. Belkacemi. 2008. Effects of drying method on the extraction yields and quality of oils from quebec sea buckthorn (*Hippophaë rhamnoides* L.) seeds and pulp. *Food Chemistry* 106:896-904.

Guu, Y.-K. 2003. Dehydration process energy balances. *Encyclopedia of Agricultural, Food, and Biological Engineering* 1(1):176-179.

Hakkinen, S.H., S.O. Karenlampi, H.M. Mukkanen, and A.R. Torronen. 2000. Influence of domestic processing and storage on flavonol contents in berries. *Journal of Agricultural Food Chemistry* 48:2960-2965.

Harrison, J.E. and T.H.J. Beveridge. 2002. Fruit structure of *Hippophae rhamnoides* cv. Indian Summer (sea buckthorn). *Canadian Journal of Botany* 80:399-409.

Health Canada. 1998. *Nutraceuticals/Functional Foods and Health Claims on Foods.* Policy paper, Therapeutic Products Programme and the Food Directorate from the Health Protection Branch. <http://www.hc-sc.gc.ca/fn-an/alt_formats/hpfb-dgpsa/pdf/label-etiquet/nutra-funct_foods-nutra-fonct_aliment_e.pdf>. Accessed 2007/03/04.

Heilscher, K. and S. Lorber. 1996. Cold working process for obtaining clear juice, sediment and oil from sea buckthorn berries and their use. GFR Patent 5665361, Sept 9, 1997. [Cited by Beveridge et al. 1999]

Henderson, S.M. 1974. Progress in developing the thin-layer drying equation. *Transactions of the American Society of Agricultural Engineering* 17:1167-1168,1172. [Cited by Jayas et al. 1991]

Henderson, S.M. and S. Pabis. 1961. Grain drying theory I: Temperature effect on drying coefficient. *Journal of Agricultural Engineering Research* 6:169-174. [Cited by Jayas et al. 1991]

Hlinak, A.J. and A. Saleki-Gerhardt. 2000. An evaluation of fluid bed drying of aqueous granulations. *Pharmaceutical Development and Technology* 5(1):11-17.

Hustrulid, A. 1963. Comparative drying rates of naturally moist, re-moistened and forzen wheat. *Transactions of the American Society of Agricultural Engineers* 5:64-67. [Cited by Jayas et al. 1991]

Iglesias, H.A. and J. Chirife. 1976. Prediction of effect of temperature on water sorption isotherms of food materials. *Journal of Food Technology* 11:109-116. [Cited by Pabis et al. 1998; Sinicio et al. 1995]

Incropera, F.P. and D.P. DeWitt. 1985. *Fundamentals of Heat and Mass Transfer*, 2nd ed. New York, NY: John Wiley and Sons, Inc.

ISO. 2004. *Procedure 9936: Determination of Tocopherols and Tocotrienols Content – Method of High Performance Liquid Chromatography*. Geneva, Switzerland: International Standards Organization.

Jalili, T., R.E.C. Wildman, and D.M. Medeiros. 2001. Dietary fiber and coronary heart disease. In *Handbook of Nutraceuticals and Functional Foods*, ed. R.E.C. Wildman, 281-293. Boca Raton, FL: CRC Press LLC.

Jayas, D.S., S. Cenkowski, S. Pabis, and W.E. Muir. 1991. Review of thin-layer drying and wetting equations. *Drying Technology* 9(3):551-558.

Jiang, Y.D., Y.C. Zhou, C.F. Bi, J.M. Li, J.X. Yang, Z.D. Yu, Z.Y. Hu, and S.X. Zhao. 1993. A clinical investigation of effects of sea buckthorn seed oil on hyperlipeamia. *Hippophae* 6:23-24. [Cited by Yang and Kallio 2002a]

Jiang, Z. and G. Li. 1987. Research on the active anti-ulcer component of sea buckthorn oil. *Academic Journal of the Second Military Medical University of China* 8:119. [In Chinese: Cited by Yang and Kallio 2002a]

Johansson, A.K., H. Korte, B. Yang, J.C. Stanley, and H.P. Kallio. 2000. Sea buckthorn berry oil inhibits platelet aggregation. *Journal of Nutritional Biochemistry* 11:491-495.

Kallio, H., B. Yang, and P. Peippo. 2002a. Effects of different origins and harvesting time on vitamin C, tocopherols, and tocotrienols in sea buckthorn (*Hippophae rhamnoides*) berries. *Journal of Agricultural Food Chemistry* 50:6136-6142.

Kallio, H., B. Yang, P. Peippo, R. Tahvonen, and R. Pan. 2002b. Triacylglycerols, glycerophospholipids, tocopherols, and tocotrienols in berries and seeds of two subspecies (ssp. *sinensis and mongolica*) of sea buckthorn (*Hippophae rhamnoides*). *Journal of Agricultural Food Chemistry* 50:3004-3009.

Kallio, H., B. Yang, B. Wang, H. Wang, J. Wang, J. Song, H. Meng, and H. Zhao. 2001. Animal experiments on the anti-inflammatory and analgesic effects of sea buckthorn (*Hippophae rhamnoides* L.) oils. In *Biologically-Active Phytochemicals*, eds. W. Pfannhauser, R. Fenwick, and S. Khokhar, 69-73. Cambridge, UK: The Royal Chemistry Society. [Cited by Yang and Kallio 2002a]

Karanthanos, V.T. 1999. Determination of water content of dried fruits by drying kinetics. *Journal of Food Engineering* 39:337-344. [Cited by Celma et al. 2007]

Kassem, A.S. 1998. Comparative studies on thin layer drying models for wheat. In *Proceedings of the 13th International Congress on Agricultural Engineering,* 6:2-6. Rabat, Morroco [Cited by Celma et al. 2007]

Kemp, I.C., B.C. Fyhr, S. Laurent, M.A. Roques, C.E. Groenewold, E. Tsotsas, A.A. Sereno, C.B. Bonazzi, J.-J. Bimbenet, and M. Kind. 2001. Methods for processing experimental drying kinetics data. *Drying Technology* 19(1): 15-34.

Kemp, I.C. and D.E. Oakley. 2002. Modeling of particulate drying in theory and practice. *Drying Technology* 20(9): 1699-1750.

Kołakowska, A. 2003. Lipid oxidation in food systems. In *Chemical and Functional Properties of Food Lipids*, eds. Z.E. Sikorski and A. Kołakowska, 133-166. Boca Raton, FL:CRC Press LLC.

Kołakowska, A. and Z.E. Sikorski. 2003. The role of lipids in food quality. In *Chemical and Functional Properties of Food Lipids*, eds. Z.E. Sikorski and A. Kołakowska, 1-8. Boca Raton, FL:CRC Press LLC.

Konovalov, V.I., N.Z. Gatapova, and T. Kudra. 2003. Drying of liquid dispersions: A unified approach to kinetics and modeling. *Drying Technology* 21(6):1029-1047.

Koyuncu, T., I. Tosun, and N.S. Ustun. 2003. Drying kinetics and colour retention of dehydrated rosehips – Technical note. *Drying Technology* 21(7):1369-1381.

Krokida, M.K., Z.B. Maroulis, and G.D. Saravacos. 2001. The effects of the method of drying on the colour of dehydrated products. *International Journal of Food Science Technology* 36:53-59.

Kudra, T. and G.I. Efremov. 2003. A quasi-stationary approach to drying kinetics of fluidized particulate materials. *Drying Technology* 21(6):1077-1090.

Kudritskaya, S.E., L.M. Zagorodskaya, and E.E. Shishkina. 1989. Carotenoids of the sea buckthorn, variety Obil'naya. *Chemistry of Natural Compounds* 25:724-725. [Cited by Oomah 2003]

Kundu, K.M. 2004. Drying of oilseeds. In *Dehydration of Products of Biological Origin*, ed. A.S. Mujumdar, 275-295. Enfield, NH: Science Publishers, Inc.

Kutsakova, V.E. 2004. Drying of liquid and pasty products in a modified spouted bed of inert particles. *Drying Technology* 22(10):2343-2350.

Laos, K., T. Lõugas, A. Mändmets, and R. Vokk. 2007. Encapsulation of β-carotene from sea buckthorn (*Hippophaë rhamnoides* L.) juice in furcellan beads. *Innovative Food Science and Emerging Technologies* 8:395-398.

Lebedeva, L.D., N.E. Akmolova, K.H. Haydarov, and M.B. Ismailova. 1992. Comparison of the effects of sea buckthorn oil, β-sitosterol oleate, β-sitosterol palmitate on the healing process of burns. In *Advances in the Biochemical and Pharmacological Research on Sea Buckthorn*, 147-148. Scientific communication, Wugong Agricultural Research Center, Shaanxi Province, China: Wugong Publishing House. [Cited by Yang and Kallio 2002a]

Leontieva, A.I., K.V. Bryankin, V.I. Konovalov, and N.P. Utrobin. 2002. Heat and mass transfer during drying of a liquid film from the surface of a single inert particle. *Drying Technology* 20(4-5):729-747.

Lewis, W.K. 1921. The rate of drying of solid materials. *Industrial Engineering Chemistry* 13:427. [Cited by Jayas et al. 1991]

Li, F. and T. Guo. 1989. Application of *Hippophae rhamnoides* L. in Tibetan medicine. In *Proceedings of the International Symposium on Sea-Buckthorn (H. rhamnoides* L.), 409-412. Xi'an, China. [Cited by Schroeder and Yao 1995]

Li, T.S.C. 2003a. Introduction. In *Sea Buckthorn (Hippophae rhamnoides L.): Production and Utilization*, eds. T.S.C. Li and T.H.J. Beveridge, 1-5. Ottawa, ON: NRC Research Press.

Li, T.S.C. 2003b. Taxonomy, natural distribution and botany. In *Sea Buckthorn (Hippophae rhamnoides L.): Production and Utilization*, eds. T.S.C. Li and T.H.J. Beveridge, 7-11. Ottawa, ON: NRC Research Press.

Li, T.S.C. 2002. Product development of sea buckthorn. In *Trends in New Crops and New Uses*, eds. J. Janick and A. Whipkey, 393-398. Alexandria, VA: ASHS Press.

Li, T.S.C. 1999. Sea buckthorn: New crop opportunity. In *Perpectives on New Crops and New Uses*, ed. J. Janick, 335-337. Alexandria, VA: ASHS Press.

Li, T.S.C., T.H.J. Beveridge, and J.C.G. Drover. 2007. Phytosterol content of sea buckthorn (*Hippophae rhamnoides* L.) seed oil: Extraction and identification. *Food Chemistry* 101:1633-1639.

Li, T.S.C. and L.C.H. Wang. 1998. Physiological components and health effects of Ginseng, Echinacea, and Sea Buckthorn. In *Functional Foods: Biochemical & Processing Aspects*, ed. G. Mazza, 329-355. Lancaster, PA: Technomic Publishing Company, Inc.

Li, Z.R. and S.Z. Tan. 1993. A clinical observation on the effects of oral supplementation of sea buckthorn oil on patients with malignant tumor under chemotherapy. *Hippophae* 6:41-42. [Cited by Yang and Kallio 2002a]

Litwinienko, G. and T. Kasprzycka-Guttman. 2000. Study on the autoxidation kinetic of fat components by differential scanning calorimetry. 2. Unsaturated fatty acids and their esters. *Industrial and Engineering Chemistry Research* 39:13-17.

Liu, J. and Z. Liu. 1989. Research of processing technology for sea buckthorn concentrated juice. *Proceedings of International Symposium on Sea Buckthorn (H. rhamnoides* L.), 314-317. Xi'an, China. [Cited by Beveridge et al. 1999]

Lobatcheva, I.I., W. Letchamo, L. Huszar, S.A. Murchortov, N.N. Malkova, and E.I. Pantaleeva. 2002. Evaluation of Siberian sea buckthorn cultivars in Washington State. In *Trends in New Crops and New Uses*, eds. J. Janick and A. Whipkey, 402-404. Alexandria, VA: ASHS Press.

Lu, R. 1992. *Sea buckthorn: A multipurpose plant species for fragile mountains.* International Centre for Integrated Mountain Development. Katmandu, Nepal. [Cited by Li 2003a]

Ma, Z. and Y. Cui. 1987. Studies on the chemical composition of Chinese seabuckthorn. *Wuhan Botanical Research* 5(4):397-403. [In Chinese: Cited by Tang and Tigerstedt 2001]

Mann, D.D., D.S. Petkau, T.G. Crowe, and W.R. Schroeder. 2001. Removal of sea buckthorn (*Hippophae rhamnoides* L.) berries by shaking. *Canadian Biosystems Engineering* 43:2.23-2.28.

Márkus, F., H.G. Daood, J. Kapitány, and P.A. Biacs. 1999. Change in the carotenoid and antioxidant content of spice red pepper (Paprika) as a function of ripening and some technological factors. *Journal of Agricultural Food Chemistry* 47:100-107.

Marmo, L. 2007. Low temperature drying of pomace in spout and spout-fluid beds. *Journal of Food Engineering* 79:1179-1190.

Mathur, K.B. and N. Epstein. 1974. *Spouted Beds.* New York, NY: Academic Press.

Midilli, A., H. Kucuk, and Z.A. Yapar. (2002). New model for single layer drying. *Drying Technology* 20(7): 1503-1513.

Mikhailov, M.D. and M.N. Özişik. 1984. *Unified Analysis and Solutions of Heat and Mass Diffusion*. New York, NY: John Wiley and Sons, Inc.

Mínguez-Mosquera, M.I., D. Hornero-Méndez, and A. Pèrez-Gálvez. 2002. Carotenoids and provitamin A in functional foods. In *Methods of Analysis for Functional Foods and Nutraceuticals*, ed. J. Hurst, 101-157. Boca Raton, FL: CRC Press LLC.

Mironov, V.A. 1989. Chemical composition of *Hippophae rhamnoides* of different populations of the USSR. In *Proceedings of the First International Symposium on Sea Buckthorn*, October 19-23, Xi'an, China 67-70. [Cited by Beveridge et al. 1999].

Mironov, V.A., T.N. Guseva-Donskaya, and Y.Y. Dubrovina. 1989. Chemical composition and biological activity of extracts from sea buckthorn fruit components. *Zh. Org. Farm. Khim.* 23:1357-1364. [In Russian: Cited by Oomah 2003; Yang and Kallio 2002a].

Mironov, V.A., G.S. Vasil'ev, V.S. Matrosov, T.M. Filippova, V.A. Zamurenko, V.V. Mishchenko, V.G. Maironovskii, I.I. Kas'yanenko, and L.M. Maksimova. 1983. Physiologically active alcohols from sea buckthorn fruit. *Khimiko-Farm-atsevticheskii Zhurnal* 17:1242-1247. [Cited by Yang and Kallio 2002a]

Mujumdar, A.S. 2006. Principles, classification, and selection of dryers. In *Handbook of Industrial Drying*, 3rd ed., ed. A.S. Mujumdar, 4-31. New York, NY:CRC Press LLC.

Neish, G. 2003. Sea buckthorn shows promise as alternative crop. Agriculture and Agri-food Canada. <http://res2.agr.gc.ca/research-recherche/ann-dir/1999-2001/2x7x2_e.html>. Accessed 2003/02/27.

Nindo, C.I., J.R. Powers, and J. Tang. 2007. Influence of refractance window evaporation on quality of juices from small fruits. *LWT – Food Science and Technology* 40:1000-1007.

Normén, L., J. Frohlich, and E. Trautwein. 2004. Role of plant sterols in cholesterol lowering. In *Phytosterols as Functional Food Components and Nutraceuticals*, ed. P.C. Dutta, 243-315. New York, NY: Marcel Dekker, Inc.

NRCC. 2002. *Canadian Seabuckthorn Technology Mission to Russia and Germany – Final Report*. National Research Council Canada/Industrial Research Assistance Program, Newfoundland & Labrador and Nunavut Region.

Nursten, H.E. 1986. Maillard browning reactions in dried foods. In *Concentration and Drying of Foods*, ed. D. MacCarthy, 53-68. New York, NY: Elsevier Applied Science Publishers.

Oliviera, W.P., R.F. Bott, and C.R.F. Souza. 2006. Manufacture of standardized dried extracts from medicinal Brazilian plants. *Drying Technology* 24(4):523-533.

Oomah, B.D. 2003. Sea buckthorn lipids. In *Sea Buckthorn (Hippophae rhamnoides L.): Production and Utilization*, eds. T.S.C. Li and T.H.J. Beveridge, 51-68. Ottawa, ON: NRC Research Press.

Oomah, B.D. and G. Mazza. 2000. Functional foods. In *The Wiley Encyclopedia of Food Science and Technology*, 2nd ed., vol. 2, ed. F.J. Francis, 1176-1182. New York, NY: John Wiley and Sons, Inc.

Orsat, V. and G.S.V. Raghavan. 2007. Dehydration technologies to retain bioactive components. In *Functional Food Ingredients and Nutraceuticals: Processing Technologies*, ed. J. Shi, 173-191. Boca Raton, FL: CRC/Taylor and Francis.

Overhults, D.G., H.E. White, H.E. Hamilton, and I.J. Ross. 1973. Drying soybeans with heated air. *Transactions of the American Society of Agricultural Engineers* 16:112-113. [Cited by Jayas et al. 1991]

Pabis, S., D.S. Jayas, and S. Cenkowski. 1998. *Grain Drying: Theory and Practice.* New York, NY: John Wiley and Sons, Inc.

Page, G.E. 1949. Factors influencing the maximum rates of air drying shelled corn in thin layers. M.Sc. Thesis, Purdue University, West Lafayette, IN. [Cited by Jayas et al. 1991]

Packer, L. and S.U. Weber. 2001. The role of vitamin E in the emerging field of nutraceuticals. In *Nutraceuticals in Health and Disease Prevention*, ed. K. Krämer, 27-46. New York, NY: Marcel Dekker, Inc.

Pallai, E., T. Szentmarjay, E. Szijjártó. 2001. Effect of partial processes of drying on inert particles on product quality. *Drying Technology* 19(8):2019-2032.

Palozza, P. 2004. Evidence for pro-oxidant effects of carotenoids in vitro and in vivo: Implications in health and disease. In *Carotenoids in Health and Disease*, ed. N.I. Krinsky, S.T. Mayne, and H. Sies, 129-149. New York, NY: Marcel Dekker, Inc.

Parker, R. 2000. Phytochemicals: Carotenoids. In *Encyclopedia of Food Science and Technology*, 2nd ed., vol. 3, ed. F.J. Francis, 1909-1915. New York, NY: John Wiley and Sons, Inc.

Partanen, R., H. Yoshii, H. Kallio, B. Yang, and P. Forssell. 2002. Encapsulation of sea buckthorn kernel oil in modified starches. *JOACS* 79(3):219-223.

Perera, C.O. 2005. Selected quality attributes of dried foods. *Drying Technology* 23(4): 717-730.

Pfost, H.B., S.G. Maurer, D.S. Chung, and G.A. Milliken. 1976. Summarizing and reporting equilibrium moisture data for grains. *American Society of Agricultural Engineers.* Paper no. 76-3520. [Cited by Pabis et al. 1998; Sinicio et al. 1995]

Piironen, V. and A.-M. Lampi. 2004. Occurrence and levels of phytosterols in foods. In *Phytosterols and Functional Food Components and Nutraceuticals*, ed. P.C. Dutta, 1-32. New York, NY: Marcel Dekker, Inc.

Pintea, A., A. Marpeau, M. Faye, C. Socaciu, and M. Gleizes. 2001. Polar lipid and fatty acid distribution in carotenolipoprotein complexes extracted from seabuckthorn fruits. *Phytochemical Analysis* 12:293-298.

Pokorný, J. and Š. Schmidt. 2003. The impact of food processing in phytochemicals: The case of antioxidants. In *Phytochemical Functional Foods*, eds. I. Johnson and G. Williamson, 298-314. Boca Raton, FL: CRC Press.

Rahman, M. S. 2005. Dried food properties: Challenges ahead. *Drying Technology* 23:695-715.

Ramaswamy, H.S. and F. Nsonzi. 1998. Convective-air drying kinetics of osmotically pre-treated blueberries. *Drying Technology* 16(3-5):743-759.

Ratti, C. 2001. Hot air and freeze-drying of high-value foods: A review. *Journal of Food Engineering* 49:311-319.

Regier, M., E. Mayer-Miebach, D. Behsnilian, E. Neff, and H.P. Schuchmann. 2005. Influences of drying and storage of lycopene-rich carrots on the carotenoid content. *Drying Technology* 23:989-998.

Reid, D.S. 1997. Overview of physical/chemical aspects of freezing. In *Quality in Frozen Food*, eds. M.C. Erickson and Y.-C. Hung, 10-28. New York, NY: Chapman and Hall.

Ren, L.F., J.P. Yang, H.X. Zhang, C.J. Zhong, and R.X. Su. 1992. Observations on the anti-mutagenic and anti-immunosuppressive effects of sea buckthorn seed oil. *Hippophae* 5:23-26. [Cited by Yang and Kallio 2002a]

Rongsen, A. 1992. *Seabuckthorn a multi-purpose plant species for fragile mountains.* ICIMOD Occasional Paper No. 20, Khathmandou, Nepal. [Cited by Schroeder and Yao 1995]

Roos, Y.H. 2004. Phase and state transitions in dehydration of biomaterials and foods. In *Dehydration of Products of Biological Origin*, ed. A.S. Mujumdar, 3-22. Enfield, NH: Science Publishers, Inc.

Rösch, D., A. Krumbein, and L.W. Kroh. 2004. Antioxidant gallocatechins, dimeric and trimeric proanthocyanidins from sea buckthorn (*Hippophae rhamnoides*) pomace. *European Food Research Technology* 219:605-613.

Sablani, S.S. 2006. Drying of fruits and vegetables: Retention of nutritional/functional quality. *Drying Technology* 24(2):123-135.

Sacilik, K. and A.K. Elicin. 2006. The thin layer drying characteristics of organic apple slices. *Journal of Food Engineering* 73: 281-289.

San Martin, M.B., J.I. Mate, T. Fernandez, and P. Virseda. 2001. Modelling adsorption equilibrium moisture characteristics of rough rice. *Drying Technology* 19(3): 681-690.

Saravacos, G.D. and Z.B. Maroulis. 2001. *Transport Properties of Foods*. New York, NY: Marcel Dekker, Inc.

Schapiro, D.C. 1989. Biochemical studies on some hopeful forms and species of sea buckthorn in USSR. In *Proceedings of the First International Symposium on Sea Buckthorn*, 64-66. Xi'an, China. [Cited by Oomah 2003]

Schreiner, M., S. Huyskens-Keil, A. Krumbein, I. Schonhof, and M. Linke. 2000. Environmental effects on product quality. In *Fruit & Vegetable Quality*: *An Integrated View*, eds. R.L. Shewfelt and B. Brückner, 85-95. Lancaster, PA: Technomic Publishing Company, Inc.

Schroeder, W.R. and Y. Yao. 1995. *Sea buckthorn: a promising multipurpose crop for Saskatchewan*. Indian Head, SK: Prairie Farm Rehabilitation Administration, Agriculture Canada.

Shahidi, F. 2004. Quality characteristics of edible oils. In *Quality of Fresh and Processed Foods*, eds. F. Shahidi, A.M. Spanier, C.-T. Ho, and T. Braggins, 239-249. New York, NY: Kluwer Academic / Plenum Publishers.

Shahidi, F. and M. Naczk. 2004. *Phenolics in Food and Nutraceuticals*. Boca Raton, FL: CRC Press LLC.

Shahidi, F. and S.P.J.N. Senanayake. 2006. Nutraceutical and specialty lipids. In *Nutraceutical and Specialty Lipids and their Co-Products*, ed. F. Shahidi, 1-25. New York, NY: CRC Press/ Taylor and Francis.

Sharaf-Elden, Y.I., J.L. Blaisdell, and M.Y. Hamdy. 1980. A model for ear corn drying. *Transactions of the American Society of Agricultural Engineers* 23:1261-1265,1271. [Cited by Jayas et al. 1991]

Shi, J., M. Le Maguer, and M. Bryan. 2002. Lycopene from tomatoes. In *Functional Foods: Biochemical and Processing Aspects*, vol 2, ed. J. Shi, 135-167. New York, NY: CRC Press LLC.

Sinicio, R., W.E. Muir, D.S. Jayas, and S. Cenkowski. 1995. Thin-layer drying and wetting of wheat. *Postharvest Biology and Technology* 5:261-275.

Singh, R.P. and D.R. Heldman. 2001. *Introduction to Food Engineering*, 3rd ed., London, UK: Academic Press, Inc.

Small, E., and P.M. Catling. 1999. *Canadian Medicinal Crops*. Ottawa, ON: NRC Research Press.

Sokhansanj, S. and D.S. Jayas. 2006. Drying of foodstuffs. In *Handbook of Industrial Drying*, 3rd ed., ed. A.S. Mujumdar, 522-546. New York, NY:CRC Press LLC.

Sokolovskii, A.A., P.B. Bagleev, N.M. Kondrat'eva, V.V. Zarutskii, and A.S. Ginzburg. 1976. Development of an effective method for drying sea buckthorn fruit pulp in a vortex dryer. *Pharmaceutical Chemistry Journal* 10(2):212-214.

Somogyi, L.P. 2000. Fruit dehydration. In *The Wiley Encyclopedia of Food Science and Technology*, 2nd ed., vol. 2, ed. F.J. Francis, 1142-1148. New York, NY: John Wiley and Sons, Inc.

St. George, S.D. and S. Cenkowski. 2008. The influence of drying on levels of bioactive compounds in pulp and peel oil of sea buckthorn (*Hippophae rhamnoides* L. ssp. *sinensis*) berries. *Journal of Food Processing and Preservation.* Accepted for publication April, 2008.

St. George, S.D. and S. Cenkowski. 2007. Influence of harvest time on the quality of oil-based compounds in sea buckthorn (*Hippophae rhamnoides* L. ssp. *sinensis*) seed and fruit. *Journal of Agricultural and Food Chemistry* 55:8054-8061.

Tadesse, T., E.W. Hewett, M.A. Nichols, and K.J. Fisher. 2002. Changes in physicochemical attributes of sweet pepper cv. Domino during fruit growth and development. *Scientia Horticulturae* 93:91-103.

Tang, X and P.M.A. Tigerstedt. 2001. Variation of physical and chemical characters within an elite sea buckthorn (*Hippophae rhamnoides* L.) breeding population. *Scientia Horticulturae* 88(3):203-214.

Tatum, V. and C.K. Chow. 2000. Effects of processing and storage on fatty acids in edible oils. In *Fatty Acids in Foods and Their Health Implications,* 2nd ed., ed. C.K. Chow, 411-425. New York, NY: Marcel Dekker, Inc.

Thompson, T.L., R.M. Peart, and G.H. Foster. 1968. Mathematical simulation of corn drying – a new model. *Transactions of the American Society of Agricultural Engineers* 11:582-586. [Cited by Pabis et al. 1998; Sinicio et al. 1995]

Tomaino, R. and E.A. Decker. 2000. Phytochemicals: Antioxidants. In *Encyclopedia of Food Science and Technology*, 2nd ed., vol. 3, ed. F.J. Francis, 1895-1901. New York, NY: John Wiley and Sons, Inc.

Tong, J., C. Zhang, Z. Zhao, Y. Yang, and K. Tian. 1989. The determination of the physico-chemical constants and sixteen mineral elements in sea buckthorn raw juice. In *Proceedings of the First International Symposium on Sea Buckthorn*, 132-137. Xi'an, China. [Cited by Beveridge 2003b]

Van Arsdel, W.B., M.J. Copley, and A.I. Morgan Jr. 1973. *Food Dehydration: Drying Methods and Phenomena*, 2nd ed., vol.1. Westport, CT: Avi Publishing Company, Inc.

Van Den Berg, C. 1986. Water activity. In *Concentration and Drying of Foods* ed. D. MacCarthy, 11-51. New York, NY: Elsevier Applied Science Publishers.

Vega-Mercado, H., M.M. Góngora-Nieto, and G.V. Barbosa-Cánovas. 2001. Advances in dehydration of foods. *Journal of Food Engineering* 49:271-289.

Verma, L.R., R.A. Bucklin, J.B. Endan, and F.T. Wratten. 1985. Effects of drying air parameters on rice drying models. *Transactions of the American society of Agricultural Engineers* 28(1): 296-301. [Cited by Celma et al. 2007]

Wang, C.Y. and R.P. Singh. 1978. Use of variable equilibrium moisture content in modelling rice drying. Transactions of American Society of Agricultural Engineers 11:668-672. [Cited in Celma et al. 2007]

Wang, X.Q., Q.H. Hu, Y.Z. Liu, C. Zhao, R.F. Wu, X.H. Cui, J.M. Liu, and X.J. Feng. 1989. Studies on effects of sea buckthorn on humoral immune function of experimental animals. *Ningxia Medical Journal* 11:281-282. [In Chinese: Cited by Yang and Kallio 2002a]

Webb, Geoffrey P. 2006. *Dietary Supplements and Functional Foods*. Oxford, UK: Blackwell Publishing Ltd.

WHO. 2004. Global strategy on diet, physical activity and health. <http://www.who.int/dietphysicalactivity/strategy/eb11344/strategy_english_web.pdf>. Accessed 2007/03/04.

Wildman, R.E.C. 2001a. Nutraceuticals: A brief review of historical and teleological aspects. In *Handbook of Nutraceuticals and Functional Foods*, ed. R.E.C. Wildman, 1-12. Boca Raton, FL: CRC Press LLC.

Wildman, R.E.C. 2001b. Classifying nutraceuticals. In *Handbook of Nutraceuticals and Functional Foods*, ed. R.E.C. Wildman, 13-30. Boca Raton, FL: CRC Press LLC.

Wolf, D. and F. Wegert. 1993. Experience gained in the harvesting and utilization of sea buckthorn. In *Cultivation and Utilization of Wild Fruit Crops*, 23-29. Bernard Thalacker Verlag GmbH&Co. [In German: Cited by Beveridge 2003a]

Xin, Y., Y. Li, S. Wu, and X. Sun. 1995. A study of the composition of sea buckthorn oils in China. In *Proceedings of the International Workshop on Sea Buckthorn*, 82-89. Beijing, China. [Cited by Oomah 2003]

Xing, J., B. Yang, Y. Dong, B. Wang, J. Wang, and H.P. Kallio. 2002. Effects of sea buckthorn (*Hippophae rhamnoides* L.) seed and pulp oils on experimental models of gastric ulcer in rats. *Fitoterapia* 73: 644-650.

Yagcioglu, A., A. Degirmencioglu, and F. Cagatay. 1999. Drying characteristics of bay leaves under different drying conditions. In *Proceedings of the Seventh International Congress on Mechanization and Energy in Agriculture*, 565-569. Adana, Turkey. [Cited by Yaldiz et al. 2001]

Yaldiz, O., C. Erketing, and H.I. Uzum. 2001. Mathematical modelling of thin layer solar drying of sultana grapes. *Energy* 26:457-465.

Yang, B., K. Kalimo, L. Mattila, S. Kallio, J. Katajisto, O. Peltola, and H. Kallio. 1999. Effects of dietary supplementation with sea buckthorn (*Hippophae rhamnoides*) seed and pulp oils on atopic dermatitis. *Journal of Nutritional Biochemistry* 10:622-630.

Yang, B., K.O. Kalimo, R.L. Tahvonen, L.M. Mattila, J.K. Katajisto, and H.P. Kallio. 2000. Effect of dietary supplementation with sea buckthorn (*Hippophae rhamnoides*) seed and pulp oils on the fatty acid composition of skin glycerophospholipids of patients with atopic dermatitis. *Journal of Nutritional Biochemistry* 11:338-340.

Yang, B. and H. Kallio. 2002a. Composition and physiological effects of sea buckthorn (*Hippophae*) lipids. *Trends in Food Science Technology* 13:160-167.

Yang, B. and H. Kallio. 2002b. Effects of harvesting time on triacylglycerols and glycerophospholipids of sea buckthorn (*Hippophae rhamnoides* L.) berries of different origins. *Journal of Food Composition Analysis* 15:143-157.

Yang, B. and H. Kallio. 2001. Fatty acid composition of lipids in sea buckthorn (*Hippophae rhamnoides* L.) berries of different origins. *Journal of Agricultural Food Chemistry* 49:1939-1947.

Yang, B., R.M. Karlsson, P.H. Oksman, and H.P. Kallio. 2001. Phytosterols in sea buckthorn (*Hippophae rhamnoides* L.) berries: Identification and effects of different origins and harvesting times. *Journal of Agricultural Food Chemistry* 49:5620-5629.

Yang, B., A.-M. Linko, H. Adlercreutz, and H. Kallio. 2006. Secoisolariciresinol and matairesinol of sea buckthorn (*Hippophae rhamnoides* L.) berries of different subspecies and harvesting times. *Journal of Agricultural Food Chemistry* 54:8065-8070.

Yang, J.P., X. Wang, Y.Y. Liu, G.X. Li, L.F. Ren, J.J. Jing, H.X. Zhang, C.J. Zhong, and R.X. Shu. 1989. A preliminary research on the anti-tumor effects of sea buckthorn press residue oil. In *Proceedings of International Symposium on Sea Buckthorn* (*H. rhamnoides* L.), 382-383. Xi'an, China. [Cited by Yang and Kallio 2002a]

Yaonian, X., L. Yonghai, W. Sulin, S. Xiuzhi, and K. Aitztmuller. 1995. A study of the compositions of seabuckthorn oils in China. In *Proceedings of the International Workshop on Sea Buckthorn*, 82-89. Beijing, China. [Cited by Beveridge 2003b]

Zadernowski, R., M. Naczk, and R. Amarowicz. 2003. Tocopherols in sea buckthorn (*Hippophae rhamnoides* L.) berry oil. *JOACS* 80(1):55-58.

Zadernowski, R., H. Nowak-Polakowska, B. Lossow, and J. Nesterowicz. 1997. Sea buckthorn lipids. *Journal of Food Lipids* 4:165-172.

Zhang, P.Z., X.F. Ding, L.N. Mao, D.X. Li, and L.P. Li. 1989a. Anti-cancer effects of sea buckthorn juice and seed oil and their effects on immune function. In *Proceedings of International Symposium on Sea Buckthorn (H. rhamnoides* L.*)*, 373-381. Xi'an China. [Cited by Yang and Kallio 2002a]

Zhang, W., C.-Y. Li, D. Liu, X.-Z. Deng, H. Zhang, C.-Y. Du, and Y.-J. Li. 1989b. Studies on the ripening pattern of sea buckthorn (Hippophae species) fruits. *Acta-Horticulturae-Sinica* 16(4):241-247. [Abstract in English]

Zhang, W.L., Z.F. Zhang, J.J. Fan, S.Y. Yang, Z.M. Li, Z.C. Deng, G.L. Wang, and F.S. Zhang. 1988. Experimental observation and clinical investigation effect of sea buckthorn oil on acute radio dermatitis. *Hippophae* 1:27-30. [Cited by Yang and Kallio 2002a]

Zhang, Y., J. Xu, and N. Yu. 1989c. Research on the centrifuge technology of sea buckthorn juice and oil. Proceedings of International Symposium on Sea Buckthorn (*H. rhamnoides* L.), 129-132. Xi'an, China. [Cited by Beveridge et al. 1999]

Zhao, L., Y. Pan, J. Li, G. Chen, and A.S. Mujumdar. 2004. Drying of a dilute suspension in a revolving flow fluidized bed of inert particles. *Drying Technology* 22(1&2): 363-376.

A – APPENDICES FOR CHAPTER 3

Appendix A.1 Physical characteristics of sea buckthorn berries

Table A.1.1 Berry and seed size data (coordinates with Table 3.2)

Harvest month	Sample	Berry size[a] (g% berries)	Seed size[b] (g% seeds)
September	1	16.425	0.978
	2	15.925	1.014
	3	14.508	0.971
	Mean	15.619	0.987
	SD[c]	0.995	0.023
	CV, %[d]	6.37	2.33
November	1	18.929	0.958
	2	19.240	0.976
	3	20.086	0.987
	Mean	19.418	0.974
	SD	0.599	0.014
	CV, %	3.08	1.44
January	1	17.102	1.005
	2	17.772	0.969
	3	18.919	1.022
	Mean	17.931	0.999
	SD	0.919	0.027
	CV, %	5.13	2.70

[a] Berry size = mass of 100 berries.
[b] Seed size = mass of 100 seeds.
[c] *SD* = standard deviation.
[d] *CV* = coefficient of variation.

Sample calculations for Table A.1.1: [Mean, *SD*, and *CV* for berry size – September]

1) Mean berry size using Eqn. A.1a:

$$\text{Mean} = \frac{\sum_{p=1}^{n} \text{berry size}_{(p)}}{n} \qquad \text{(A.1a)}$$

where berry size$_{(p)}$ = berry size for sample p; p = sample number, and n = number of samples.

$$\text{Mean} = \frac{16.425 + 15.925 + 14.508}{3}$$

3) Standard deviation *SD* of berry size using Eqn. A.1b:

$$SD = \sqrt{\frac{\sum_{p=1}^{n} \left(\text{berry size}_{(p)} - \text{Mean}\right)^2}{n-1}} \tag{A.1b}$$

where *SD* = standard deviation of berry size; Mean = mean berry size; berry size$_{(p)}$ = berry size for sample *p*; *p* = sample number, and *n* = number of samples.

$$SD = \sqrt{\frac{(16.425 - 15.619)^2 + (15.925 - 15.619)^2 + (14.508 - 15.619)^2}{3-1}}$$

4) Coefficient of variation *CV* for berry size using Eqn. A.1c:

$$CV = \frac{SD}{\text{Mean}} \times 100 \tag{A.1c}$$

where *CV* = coefficient of variation for berry size; *SD* = standard deviation for berry size, and Mean = mean value for berry size.

$$CV = \frac{0.995}{15.619} \times 100$$

Table A.1.2 Moisture content, *M*, data (Coordinates with Table 3.2)

	Harvest month		
	September	November	January
Sample 1			
m_i, g[a]	15.1654	15.3120	15.0189
m_f, g[b]	3.3806	3.9229	3.6256
M_1, % (w/w) wb[c]	77.84	74.38	75.86
Sample 2			
m_i, g	15.2561	15.1926	15.3248
m_f, g	3.3869	3.4487	3.7117
M_2, % (w/w) wb	77.80	77.30	75.78
Mean, % (w/w) wb	77.82	75.84	75.82
SD, % (w/w) wb[d]	0.03	2.06	0.06
CV, %[e]	0.04	2.72	0.08

[a] m_i = initial mass of sample.
[b] m_f = final mass of sample.
[c] M_1 = moisture content for sample 1, % mass/mass (w/w) on a wet basis (wb).
[d] *SD* = standard deviation.
[e] *CV* = coefficient of variation.

Sample calculations for Table A.1.2: [M_1 – September (sample1)]

1) Moisture content, *M*, on a wet basis (wb), using Eqn. A.1d:

$$M = \frac{m_i - m_f}{m_i} \times 100 \qquad \text{(A.1d)}$$

where m_i = initial mass of sample; m_f = final mass of sample, and M = moisture content wb.

$$M_1 = \frac{15.1654g - 3.3806g}{15.1654g} \times 100$$

Refer to "Sample calculations for Table A.1.1" in Appendix A.1 for Mean, *SD*, and *CV*.

Table A.1.3 Seed content (Coordinates with Table 3.2)

		Harvest month		
		September	November	January
Sample 1	$m_{berries}$, g[a]	56.00	56.03	56.05
	m_{seeds}, g[b]	3.94	3.23	3.47
	SC, % (w/w)[c]	7.04	5.76	6.19
Sample 2	$m_{berries}$, g	56.01	56.01	56.00
	m_{seeds}, g	3.89	3.34	3.65
	SC, % (w/w)	6.95	5.96	6.52
Sample 3	$m_{berries}$, g	56.03	56.02	56.01
	m_{seeds}, g	4.16	3.47	3.59
	SC, % (w/w)	7.42	6.19	6.41
Sample 4	$m_{berries}$, g	56.00	56.05	56.00
	m_{seeds}, g	3.93	3.26	3.72
	SC, % (w/w)	7.02	5.82	6.64
Sample 5	$m_{berries}$, g	56.06	56.00	56.04
	m_{seeds}, g	3.79	3.36	3.81
	SC, % (w/w)	6.76	6.00	6.80
Sample 6	$m_{berries}$, g	56.06	56.03	56.00
	m_{seeds}, g	3.74	3.37	3.84
	SC, % (w/w)	6.67	6.01	6.86
Sample 7	$m_{berries}$, g	56.06	56.04	56.04
	m_{seeds}, g	3.90	3.33	3.61
	SC, % (w/w)	6.96	5.94	6.44
Sample 8	$m_{berries}$, g	56.03	56.03	56.02
	m_{seeds}, g	3.90	3.27	3.63
	SC, % (w/w)	6.96	5.84	6.50
Sample 9	$m_{berries}$, g	56.03	56.00	56.06
	m_{seeds}, g	4.20	3.24	4.07
	SC, % (w/w)	7.50	5.79	7.26
	Mean, % (w/w)	7.03	5.92	6.62
	SD, % (w/w) [d]	0.27	0.14	0.31
	CV, %[e]	3.84	2.36	4.68

[a] $m_{berries}$ = mass of fresh berry sample.
[b] m_{seeds} = mass of seeds in fresh berry sample.
[c] *SC* = seed content for sample 1, % mass/mass (w/w) of seeds in fresh fruit sample.
[d] *SD* = standard deviation.
[e] *CV* = coefficient of variation.

Sample calculations for Table A.1.3: [*SC* – September (sample 1)]

1) Seed content, *SC*, using Eqn. A.1e:

$$SC = \frac{m_{seed}}{m_{berries}} \times 100 \qquad \text{(A.1e)}$$

where $m_{berries}$ = mass of berry sample; m_{seed} = mass of seeds from original berry sample, and *SC* = seed content (i.e. mass ratio of seeds in fruit sample).

$$SC_1 = \frac{3.94g}{56.00g} \times 100$$

Refer to "Sample calculations for Table A.1.1" in Appendix A.1 for Mean, *SD*, and *CV*.

Appendix A.2 Colour analysis for sea buckthorn berries

Table A.2.1 Colour analysis data (Coordinates with Table 3.3)

Month /sample	Colour factors[a]											
	L^*_{SSp}			a^*_{SSp}			b^*_{SSp}			L^*_{Sp}	a^*_{Sp}	b^*_{Sp}
	SS1[b]	SS2	SS3	SS1	SS2	SS3	SS1	SS2	SS3			
Sept S1[b]	45.50	45.41	45.36	14.79	14.75	14.79	34.04	34.02	33.98	45.42	14.78	34.01
Sept S2	45.92	45.88	45.86	14.03	14.08	14.06	37.27	37.16	37.13	45.89	14.06	37.19
Sept S3	44.50	44.35	44.33	13.68	13.79	13.74	35.87	35.66	35.69	44.39	13.74	35.74
Mean										45.23	14.19	35.65
SD[c]										0.77	0.53	1.59
CV, %[d]										1.70	3.74	4.46
Nov S1	44.67	44.65	44.70	20.64	20.62	20.43	33.63	33.38	33.30	44.67	20.56	33.44
Nov S2	46.73	46.51	46.41	19.58	19.72	19.87	39.28	39.79	39.80	46.55	19.72	39.62
Nov S3	45.19	45.16	45.13	20.27	20.23	20.27	36.97	36.95	36.93	45.16	20.26	36.95
Mean										45.46	20.18	36.67
SD										0.98	0.43	3.10
CV, %										2.16	2.13	8.45
Jan S1	46.84	46.76	46.69	17.76	17.74	17.70	38.88	38.87	38.88	46.76	17.73	38.88
Jan S2	47.20	47.17	47.17	16.85	16.98	16.93	40.00	39.94	39.98	47.18	16.92	39.97
Jan S3	47.71	47.64	47.67	17.62	17.75	17.65	40.00	40.29	40.35	47.67	17.67	40.21
Mean										47.20	17.44	39.69
SD										0.46	0.45	0.71
CV, %										9.75	2.58	1.79

[a] L^*_{Sp}/L^*_{SSp} = lightness; a^*_{Sp}/a$^*_{SSp}$ = hue, (+) red or (-) green; b^*_{Sp}/b^*_{SSp} = hue, (+) yellow or (-) blue for samples and subsamples *p*.
[b] Sept S1 = sample 1 for September (Nov = November, Jan = January); SS1 = subsample 1, 1st evaluation of sample 1.
[c] *SD* = standard deviation.
[d] *CV* = coefficient of variation.

Sample calculations for Table A.2.1: [L^*_{S1}, a^*_{S1}, and b^*_{S1} – Sept S1]

1) Lightness factor, L^*_{Sp}, a^*_{Sp}, and b^*_{Sp} using Eqns. A.2a-c:

$$L^*_{Sp} = \frac{\sum_{p=1}^{n} L^*_{SSp}}{n} \tag{A.2a}$$

$$a^*_{Sp} = \frac{\sum_{p=1}^{n} a^*_{SSp}}{n} \tag{A.2b}$$

$$b_{Sp} = \frac{\sum_{p=1}^{n} b^*_{SSp}}{n} \tag{A.2c}$$

where L^*_{Sp} = lightness for sample p, where p = 1,2,3 and n = 3; a^*_{Sp} = hue, (+)red or (-)green for sample p, where p = 1,2,3 and n = 3; b^*_{Sp} = hue, (+)yellow or (-)blue for sample p, where p = 1,2,3 and n = 3; L^*_{SSp} = lightness for subsample p, where p = 1,2,3 and n = 3; a^*_{SSp} = hue, (+)red or (-)green for subsample p, where p = 1,2,3 and n = 3; b^*_{SSp} = hue, (+)yellow or (-)blue for subsample p, where p = 1,2,3 and n = 3; p = sample number, and n = number of samples.

$$L^*_{S1} = \frac{45.50 + 45.41 + 45.36}{3}$$

$$a^*_{S1} = \frac{14.79 + 14.75 + 14.79}{3}$$

$$b^*_{S1} = \frac{34.04 + 34.02 + 33.98}{3}$$

Refer to "Sample calculations for Table A.1.1" in Appendix A.1 for Mean, *SD*, and *CV*.

Appendix A.3 Carotenoid analysis for sea buckthorn berries

Table A.3.1 Total carotenoid analysis for fruit and seed fractions (Coordinates with Table 3.4)

Month /sample	Y_{carot}[a]	X_{carot}[b] x10^{-3}mg/mL	D_2[c]	$X1_{carot}$[d] x10^{-3}mg/mL	D_1[e]	$X2_{carot}$[f] mg/mL	V_{hex}[g] mL	m_{carot}[h] mg	m_{oil}[i] g	X_{final}[j] mg/100 g
					Fruit fraction					
Sept S1[k]	0.311	1.817	4	7.270	50	0.3635	10	3.635	0.7177	506.4
Sept S2	0.304	1.775	4	7.099	50	0.3550	10	3.550	0.7615	466.2
Sept S3	0.283	1.647	4	6.589	50	0.3294	10	3.294	0.6478	508.5
Sept S4	0.287	1.672	4	6.686	50	0.3343	10	3.343	0.7090	471.5
Sept S5	0.333	1.951	4	7.805	50	0.3902	10	3.902	0.7478	521.8
Sept S6	0.317	1.854	4	7.416	50	0.3708	10	3.708	0.7503	494.2
Sept S7	0.286	1.666	4	6.662	50	0.3331	10	3.331	0.6269	531.2
Sept S8	0.354	2.079	4	8.315	50	0.4158	10	4.158	0.7523	552.7
Sept S9	0.261	1.514	4	6.054	50	0.3027	10	3.027	0.7037	430.3
Mean										498.1
SD[l]										37.4
CV, %[m]										7.51
Nov S1	0.434	2.565	4	10.260	50	0.5130	10	5.130	0.6663	769.9
Nov S2	0.438	2.589	4	10.357	50	0.5179	10	5.179	0.6730	769.4
Nov S3	0.621	3.702	4	14.807	50	0.7403	10	7.403	0.7422	997.6
Nov S4	0.403	2.377	4	9.506	50	0.4753	10	4.753	0.5831	815.3
Nov S5	0.516	3.064	4	12.254	50	0.6127	10	6.127	0.8157	751.2
Nov S6	0.574	3.416	4	13.664	50	0.6832	10	6.832	0.8096	843.9
Nov S7	0.470	2.784	4	11.135	50	0.5568	10	5.568	0.7483	744.1
Nov S8	0.533	3.167	4	12.667	50	0.6334	10	6.334	0.7551	838.8
Nov S9	0.458	2.711	4	10.844	50	0.5422	10	5.422	0.6530	830.3
Mean										817.8
SD										77.5
CV, %										9.48

Table A.3.1 Total carotenoid analysis for fruit and seed fractions (cont'd)

Month /sample	Y_{carot}	X_{carot} x10^{-3}mg/mL	D_2	$X1_{carot}$ x10^{-3}mg/mL	D_1	$X2_{carot}$ mg/mL	V_{hex} mL	m_{carot} mg	m_{oil} g	X_{final} mg/100 g
					Fruit fraction					
Jan S1	0.399	2.3523	4	9.409	50	0.4705	10	4.705	0.7271	647.0
Jan S2	0.450	2.6623	4	10.649	50	0.5325	10	5.325	0.8311	640.6
Jan S3	0.453	2.6805	4	10.722	50	0.5361	10	5.361	0.7940	675.3
Jan S4	0.388	2.2854	4	9.142	50	0.4571	10	4.571	0.7190	635.6
Jan S5	0.404	2.3827	4	9.531	50	0.4765	10	4.765	0.8357	570.3
Jan S6	0.404	2.3827	4	9.531	50	0.4765	10	4.765	0.7857	606.6
Jan S7	0.375	2.2064	4	8.826	50	0.4413	10	4.413	0.7523	586.5
Jan S8	0.459	2.7170	4	10.868	50	0.5434	10	5.434	0.8879	612.0
Jan S9	0.391	2.3037	4	9.215	50	0.4607	10	4.607	0.7984	577.1
Mean										616.8
SD										35.4
CV, %										5.74
					Seed fraction					
Sept S1	0.514	3.0513	1	3.0513	20	0.0610	5	0.305	1.2059	25.3
Sept S2	0.529	3.1425	1	3.1425	20	0.0628	5	0.314	1.2361	25.4
Sept S3	0.433	2.5590	1	2.5590	20	0.0512	5	0.256	1.1380	22.5
Mean										24.4
SD										1.6
CV, %										6.6
Nov S1	0.474	2.8082	1	2.8082	20	0.0562	5	0.281	0.9239	30.4
Nov S2	0.386	2.2733	1	2.2733	20	0.0455	5	0.227	0.9932	22.9
Nov S3	0.393	2.3158	1	2.3158	20	0.0463	5	0.232	0.9912	23.4
Mean										25.6
SD										4.2
CV, %										16.4

Table A.3.1 Total carotenoid analysis for fruit and seed fractions (cont'd)

Month /sample	Y_{carot}	X_{carot} x10^{-3}mg/mL	D_2	$X1_{carot}$ x10^{-3}mg/mL	D_1	$X2_{carot}$ mg/mL	V_{hex} mL	m_{carot} mg	m_{oil} g	X_{final} mg/100 g
					Seed fraction					
Jan S1	0.452	2.6744	1	2.6744	20	0.0535	5	0.267	1.0397	25.7
Jan S2	0.481	2.8507	1	2.8507	20	0.0570	5	0.285	1.1163	25.5
Jan S3	0.462	2.7352	1	2.7352	20	0.0547	5	0.274	0.8667	31.6
Mean										27.6
SD										3.5
CV, %										12.7

[a] Y_{carot} = absorbance value (measured).
[b] X_{carot} = concentration of total carotenoids in hexane solution.
[c] D_2 = dilution ratio 2.
[d] $X1_{carot}$ = concentration of total carotenoids in hexane solution, corrected for dilution 2.
[e] D_1 = dilution ratio 1.
[f] $X2_{carot}$ = concentration of total carotenoids in hexane solution, corrected for dilution 1.
[g] V_{hex} = volume of hexane used in original dilution.
[h] m_{carot} = mass of total carotenoids.
[i] m_{oil} = mass of oil.
[j] X_{final} = concentration of total carotenoids per 100 g oil.
[k] Sept S1 = sample 1 for September harvest month; Nov = November; Jan = January.
[l] *SD* = standard deviation.
[m] *CV* = coefficient of variation.

Sample calculations for Table A.3.1: [Total carotenoids - Sept S1(fruit fraction)]

1) Determine the concentration of total carotenoids using Eqn. A.3a:

$$Y_{carot} = 164.52X_{carot} + 0.012 \qquad \text{(A.3a)}$$

where Y_{carot}= absorbance value (measured) (calibration curve in Fig. A.3.1) and X_{carot} = concentration of total carotenoids in hexane solution.

$$X_{carot} = \frac{(0.311 - 0.012)}{164.52}$$

2) Determine concentration of total carotenoids accounting for 2nd dilution in hexane using Eqn. A.3b:

$$X1_{carot} = X_{carot}\, D_2 \qquad \text{(A.3b)}$$

where $X1_{carot}$ = concentration of total carotenoids in hexane solution corrected for 2nd dilution and D_2= dilution ratio 2 for the fruit fraction,

$$D_2 \text{ (fruit fraction)} = \frac{(1.5\text{mL} + 0.500\text{mL})}{0.500\text{mL}}$$

$$X1_{carot} = 1.817 \times 10^{-3}\,\text{mg/mL} \times 4\text{mL/mL solution}$$

3) Determine concentration of total carotenoids accounting for 1st dilution in hexane using Eqn. A.3c:

$$X2_{carot} = X1_{carot}\, D_1 \qquad \text{(A.3c)}$$

where $X2_{carot}$= concentration of total carotenoids in hexane solution corrected for 1st dilution and D_1 = dilution ratio 1 for fruit fraction,

$$D_1 \text{ (fruit fraction)} = \frac{(0.100\text{mL} + 4.9\text{mL})}{0.100\text{mL}}$$

$$X2_{carot} = 7.270 \times 10^{-3}\,\text{mg/mL} \times 50\text{mL/mL solution}$$

4) Determine mass of total carotenoids in oil sample using Eqn. A.3d:

$$m_{carot} = X2_{carot} V_{hex} \quad \text{(A.3d)}$$

where m_{carot} = mass of total carotenoids in extracted oil sample and V_{hex} = the volume of hexane used in original oil dilution.

$$m_{carot} = 0.3635\text{mg/mL} \times 10\text{mL hexane}$$

5) Determine mass concentration of total carotenoids in 100 g oil using Eqn. A.3e:

$$X_{final} = \frac{m_{carot}}{m_{oil}} \frac{100}{100} \quad \text{(A.3e)}$$

where X_{final} = Concentration of total carotenoids in 100 g fruit fraction oil and m_{oil} = mass of extracted oil.

$$X_{final} = \frac{3.635\text{mg}}{0.7177\text{g}} \times \frac{100}{100}$$

Note: D_2 (seed fraction) = 1,

$$D_1 \text{ (seed fraction)} = \frac{(0.100\text{mL} + 1.9\text{mL})}{0.100\text{mL}}.$$

Refer to "Sample calculations for Table A.1.1" in Appendix A.1 for Mean, *SD*, and *CV*.

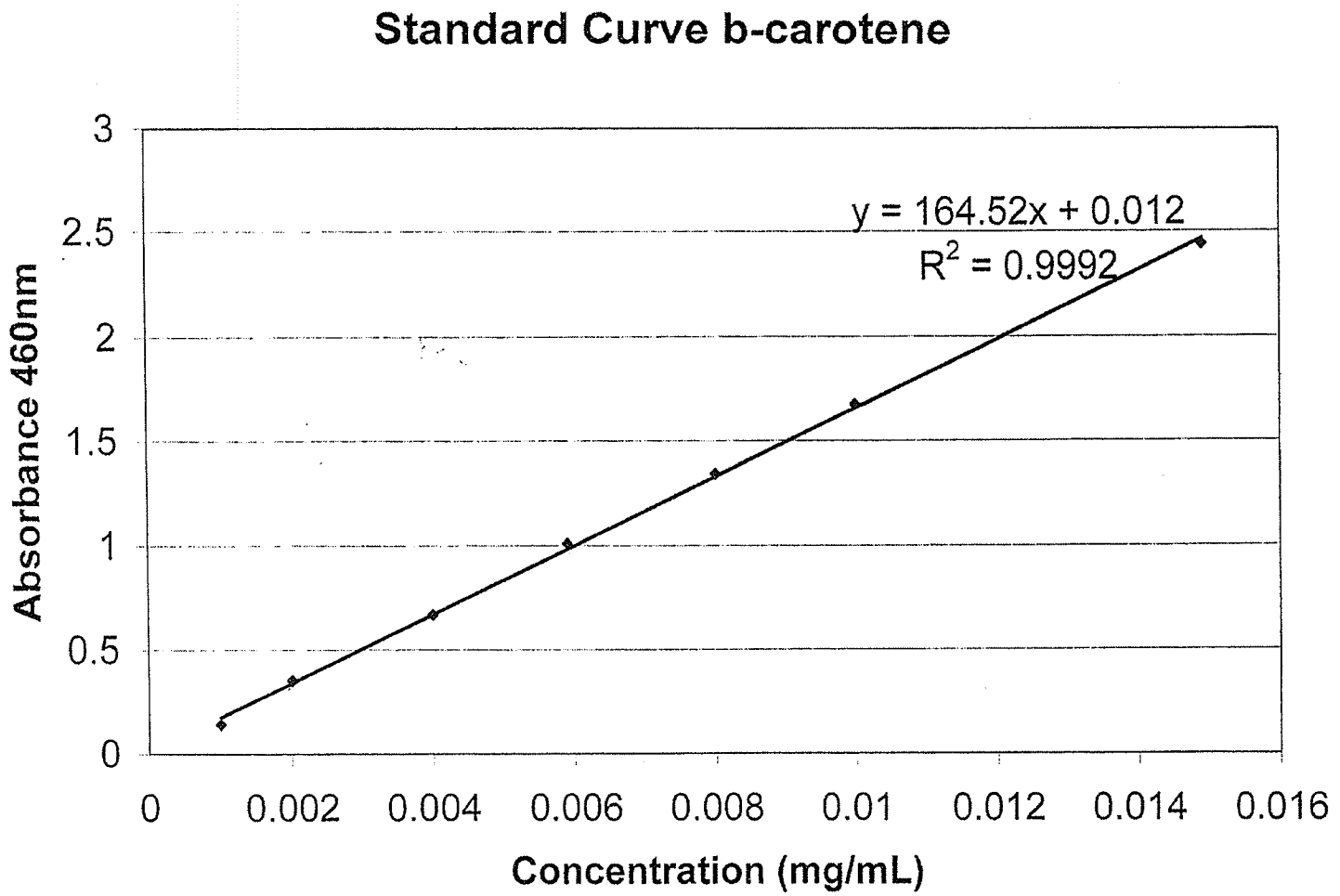


Figure A.3.1 β-carotene calibration curve.

Appendix A.4 Fatty acid analysis for sea buckthorn berries

Table A.4.1 Fatty acid sample analysis for fruit and seed fractions (Coordinates with Tables 3.5 and A.4.2)

Fatty acid[a]	Standard	Fruit fraction (November, sample 7)			Seed fraction (November, sample 1)		
	Retention time, min	Retention time, min	A_{FA}[b]	Amount $\%_{FA}$[c]	Retention time, min	A_{FA}	Amount $\%_{FA}$
C6:0	1.95	n/d[d]	n/d	n/d	n/d	n/d	n/d
C8:0	2.96	3.15	308	0.04	n/d	n/d	n/d
C10:0	4.09	n/d	n/d	n/d	n/d	n/d	n/d
C12:0	5.18	5.17	367	0.05	n/d	n/d	n/d
C14:0	6.30	6.30	5914	0.76	6.30	1259	0.15
C14:1	6.50	n/d	n/d	n/d	n/d	n/d	n/d
C15:0	7.02	7.02	793	0.10	7.02	1379	0.16
C16:0	7.88	7.89	249334	31.94	7.89	72876	8.68
C16:1*n*7	8.09	8.10	201192	25.77	8.09	6376	0.76
C17:0	8.93	8.93	1181	0.15	8.93	574	0.07
C17:1	9.17	9.17	26653	n/a[e]	9.18	22752	n/a
C18:0	10.16	10.16	9519	1.22	10.16	19438	2.32
C18:1*n*9	10.36	10.37	139946	17.93	10.37	156120	18.60
Unknown	n/d	10.46	59992	7.69	10.46	19474	2.32
C18:2*n*6	10.84	10.85	51224	6.56	10.86	306032	36.46
C18:3*n*6	11.09	11.10	181	0.02	n/d	n/d	n/d
C18:3*n*3	11.52	11.52	17287	2.22	11.53	244292	29.10
C20:0	13.14	13.14	3318	0.43	13.14	3905	0.47
C20:1	13.39	13.39	1048	0.13	13.39	1952	0.23
C20:2	14.01	14.01	192	0.02	14.01	364	0.04
C20:3*n*6	14.29	n/d	n/d	n/d	n/d	n/d	n/d
C20:4	14.42	n/d	n/d	n/d	n/d	n/d	n/d
C20:3*n*3	14.83	14.84	214	0.03	14.84	324	0.04
C20:5	15.24	15.23	350	0.05	n/d	n/d	n/d

Table A.4.1 Fatty acid sample analysis for fruit and seed fractions (cont'd)

Fatty acid	Standard	Fruit fraction (November, sample 7)			Seed fraction (November, sample 1)		
	Retention time, min	Retention time, min	A_{FA}	Amount $\%_{FA}$	Retention time, min	A_{FA}	Amount $\%_{FA}$
C22.0	16.64	16.63	2191	0.28	16.63	1371	0.16
C22:1	16.93	16.93	195	0.025	n/d	n/d	n/d
C22:2	17.63	n/d	n/d	n/d	n/d	n/d	n/d
C22:4	18.19	18.48	2844	0.36	18.48	429	0.05
C22:5*n*3	19.10	18.96	214	0.02	n/d	n/d	n/d
C22:6*n*3	19.24	n/d	n/d	n/d	n/d	n/d	n/d
C24:0	20.52	20.52	1497	0.19	20.52	662	0.08
C24:1	20.94	20.93	341	0.04	n/d	n/d	n/d
Total[f]			807271.7			862124.2	
$Total_{adj}A$[g]			780618.7			839372.2	

[a] C6:0 = caproic acid. C8:0 = caprylic acid. C10:0 = capric acid. C12:0 = lauric acid. C14:0 = myristic acid. C14:1 = myristoleic acid. C15:0 = pentadecanoic acid. C16:0 = palmitic acid. C16:1*n*7 = palmitoleic acid. C17:0 = heptadecanoic acid. C17:1 = cis-10-heptadecanoic acid. C18:0 = stearic acid. C18:1*n*9 = oleic acid. C18:2*n*6 = linoleic acid. C18:3*n*6 = γ-linolenic acid. C18:3*n*3 = α-linolenic acid. C20:0 = arachidic acid. C20:1 = eicosenoic acid isomer. C20:2 = eicosadienoic acid isomer. C20:3*n*6 = eicosatrienoic acid isomer. C20:4 = arachidonic acid. C20:3*n*3 = eicosatrienoic acid isomer. C20:5 = eicosapentaenoic acid. C22:0 = behenic acid. C22:1 = erucic acid. C22:2 = docosadienoic acid isomer. C22:4 = docosatetraenoic acid isomer. C22:5*n*3 = docosapentaenoid acid isomer. C22:6*n*3 = docosahexaenoic acid isomer. C24:0 = lignoceric acid. C24:1 = nervonic acid.

[b] A_{FA} = area of fatty acid provided on chromatograms (Figures A.4.2 and A.4.3).

[c] Amount $\%_{FA}$ = percent amount of fatty acid.

[d] n/d = not detected.

[e] n/a = not applicable for calculation (standard).

[f] Total = total area of fatty acids provided on chromatograms (Figures A.4.2 and A.4.3).

[g] $Total_{adj}\ A$ = total area of fatty acids excluding the standard C17:1.

Sample calculations for Table A.4.1: [Palmitoleic acid (C16:0) fruit fraction]

1) Refer to retention times for fatty acids provided on the chromatogram for Standard 461 (Figure A.4.1) as summarized in Table A.4.1.

2) Based on these retention times, identify fatty acids on chromatogram for fruit (Figure A.4.2) and seed (Figure A.4.3) fractions as summarized in Table A.4.1.

3) Record the area for each fatty acid as well as total area. Adjust the total area by subtracting the area for the standard C17:1.

4) Calculate % amount for C16:0 in the fruit and seed fractions using Eqn. A.4a:

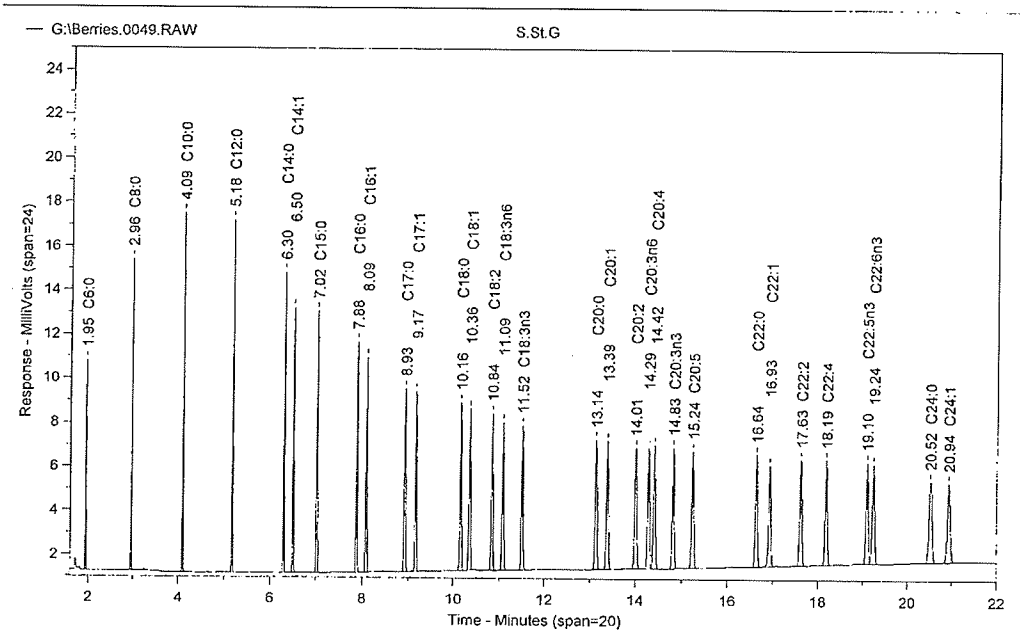
$$\text{Amount }\%_{FA} = \frac{A_{FA}}{\text{Total}_{adj}A} 100 \qquad \text{(A.4a)}$$

where Amount $\%_{FA}$ = percent proportion of individual fatty acid in fatty acid profile; A_{FA} = area associated with individual fatty acid peak on chromatogram, and $\text{Total}_{adj}A$ = total area associated with all fatty acid peaks except standard.

Fruit faction: $$\text{Amount }\%_{C16:0} = \frac{249334}{780618.7} \times 100 = 31.94\%$$

Seed fraction: $$\text{Amount }\%_{C16:0} = \frac{72876}{839372.2} \times 100 = 8.68\%$$

Chrom Perfect Chromatogram Report



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Instrument = Shimadzu
Heading 1 = 1uL injected; 1:80split
Heading 2 = Apr25,2005

Acquisition Port = 4

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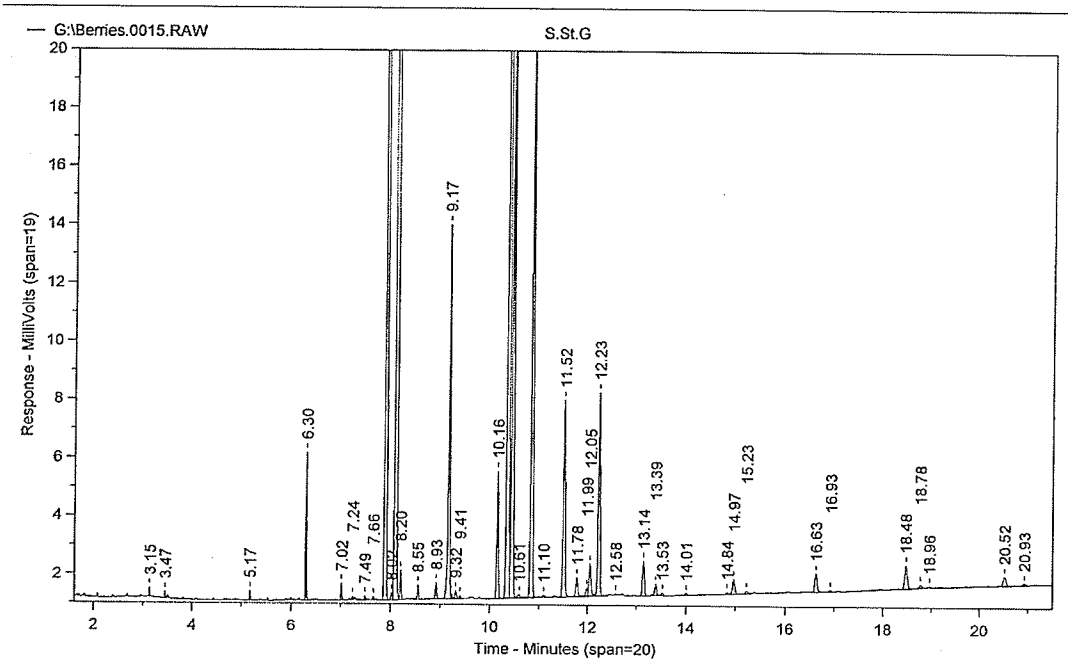
Method File Name = G:Dennis1.MET
Calibration File Name = G:Dennis1.CAL

Date Taken (end) = 4/27/2005 12:38:12 AM
Method Version = 23
Calibration Version = 4

Peak #	Ret. Time	Name	Amount	Amt %	Area	Area %	Type	Width
1	1.95	C6:0	0.00	0.000	9168	1.877	BB	0.01
2	2.96	C8:0	0.00	0.000	13715	2.808	BB	0.01
3	4.09	C10:0	0.00	0.000	15001	3.071	BB	0.01
4	5.18	C12:0	0.00	0.000	15489	3.171	BB	0.02
5	6.30	C14:0	0.00	0.000	15694	3.213	BV	0.02
6	6.50	C14:1	0.00	0.000	15326	3.137	VB	0.02
7	7.02	C15:0	0.00	0.000	15699	3.213	BB	0.02
8	7.88	C16:0	0.00	0.000	16239	3.324	BV	0.02
9	8.09	C16:1	0.00	0.000	16039	3.283	VB	0.03
10	8.93	C17:0	0.00	0.000	15932	3.261	BV	0.03
11	9.17	C17:1	0.00	0.000	16009	3.277	VB	0.03
12	10.16	C18:0	0.00	0.000	16260	3.328	BB	0.03
13	10.36	C18:1	0.00	0.000	16494	3.376	BB	0.04
14	10.84	C18:2	0.00	0.000	16321	3.341	BB	0.04
15	11.09	C18:3n6	0.00	0.000	16155	3.307	BB	0.04
16	11.52	C18:3n3	0.00	0.000	16295	3.335	BB	0.04
17	13.14	C20:0	0.00	0.000	16084	3.292	BV	0.04
18	13.39	C20:1	0.00	0.000	16280	3.332	VB	0.04
19	14.01	C20:2	0.00	0.000	15868	3.248	BB	0.04
20	14.29	C20:3n6	0.00	0.000	16227	3.322	BV	0.05
21	14.42	C20:4	0.00	0.000	16284	3.333	VB	0.05
22	14.83	C20:3n3	0.00	0.000	15951	3.265	BB	0.04
23	15.24	C20:5	0.00	0.000	16275	3.332	BB	0.05
24	16.64	C22:0	0.00	0.000	16205	3.317	BB	0.05

Figure A.4.1 Fatty acid profile chromatogram for Standard 461.

Chrom Perfect Chromatogram Report



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Heading 2 = Apr25,2005

Acquisition Port = 4

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Method File Name = G:Dennis1.MET
Calibration File Name = G:Dennis1.CAL

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Method Version = 23
Calibration Version = 4

Peak #	Ret. Time	Name	Amount	Amt %	Area	Area %	Type	Width
1	3.15	C8:0	0.00	0.000	308	0.038	BB	0.02
2	3.47		0.00	0.000	230	0.028	BB	0.02
3	5.17	C12:0	0.00	0.000	367	0.045	BB	0.02
4	6.30	C14:0	0.00	0.000	5914	0.733	BB	0.02
5	7.02	C15:0	0.00	0.000	793	0.098	BB	0.02
6	7.24		0.00	0.000	187	0.023	BB	0.03
7	7.49		0.00	0.000	218	0.027	BB	0.03
8	7.66		0.00	0.000	173	0.021	BB	0.03
9	7.89	C16:0	0.00	0.000	249334	30.886	BB	0.02
10	8.02		0.00	0.000	418	0.052	BV	0.03
11	8.10	C16:1	0.00	0.000	201192	24.923	VV	0.03
12	8.20		0.00	0.000	1774	0.220	VB	0.03
13	8.55		0.00	0.000	924	0.114	BB	0.03
14	8.93	C17:0	0.00	0.000	1181	0.146	BB	0.03
15	9.17	C17:1	0.00	0.000	26653	3.302	BV	0.03
16	9.32		0.00	0.000	564	0.070	VV	0.03
17	9.41		0.00	0.000	236	0.029	VB	0.03
18	10.16	C18:0	0.00	0.000	9519	1.179	BV	0.03
19	10.37	C18:1	0.00	0.000	139946	17.336	VV	0.03
20	10.46		0.00	0.000	59992	7.431	VV	0.03
21	10.61		0.00	0.000	451	0.056	VV	0.04
22	10.85	C18:2	0.00	0.000	51224	6.345	VV	0.04
23	11.10	C18:3n6	0.00	0.000	181	0.022	VB	0.04
24	11.52	C18:3n3	0.00	0.000	17287	2.141	BV	0.04

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Figure A.4.2 Fatty acid profile chromatogram for the fruit fraction – November (Sample 7).

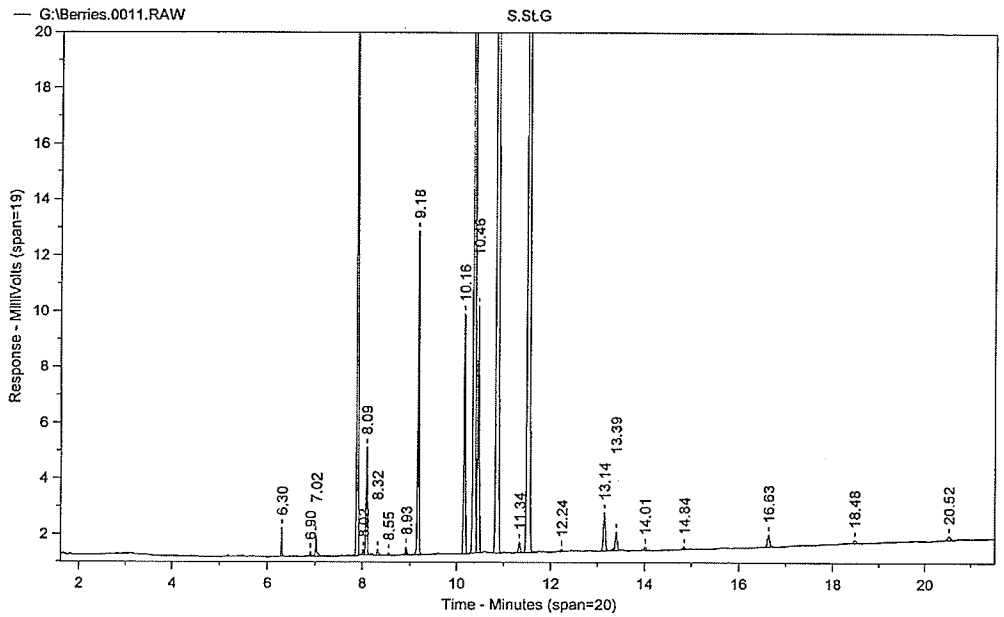
Chrom Perfect Chromatogram Report

Peak #	Ret. Time	Name	Amount	Amt %	Area	Area %	Type	Width
25	11.78		0.00	0.000	1814	0.225	VV	0.04
26	11.99		0.00	0.000	705	0.087	VV	0.04
27	12.05		0.00	0.000	3087	0.382	VV	0.04
28	12.23		0.00	0.000	17886	2.216	VB	0.04
29	12.58		0.00	0.000	113	0.014	BB	0.04
30	13.14	C20:0	0.00	0.000	3318	0.411	BB	0.04
31	13.39	C20:1	0.00	0.000	1048	0.130	BB	0.04
32	13.53		0.00	0.000	274	0.034	BB	0.05
33	14.01	C20:2	0.00	0.000	192	0.024	BB	0.05
34	14.84	C20:3n3	0.00	0.000	214	0.027	BV	0.05
35	14.97		0.00	0.000	1590	0.197	VB	0.05
36	15.23	C20:5	0.00	0.000	350	0.043	BB	0.06
37	16.63	C22:0	0.00	0.000	2191	0.271	BB	0.05
38	16.93	C22:1	0.00	0.000	195	0.024	BB	0.05
39	18.48	C22:4	0.00	0.000	2844	0.352	BB	0.05
40	18.78		0.00	0.000	332	0.041	BV	0.05
41	18.96	C22:5n3	0.00	0.000	214	0.027	VB	0.07
42	20.52	C24:0	1.50	81.450	1497	0.185	BB	0.07
43	20.93	C24:1	0.34	18.550	341	0.042	BB	0.07

Total Area = 807271.7 Total Height = 441347.6 Total Amount = 1.838449

Figure A.4.2 Fatty acid profile chromatogram for the fruit fraction – November (Sample 7). (cont'd)

Chrom Perfect Chromatogram Report



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Heading 2 = Apr25,2005

Acquisition Port = 4

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Method File Name = G:Dennis1.MET
Calibration File Name = G:Dennis1.CAL

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Method Version = 23
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Peak #	Ret. Time	Name	Amount	Amt %	Area	Area %	Type	Width
1	6.30	C14:0	0.00	0.000	1259	0.146	BB	0.02
2	6.90		0.00	0.000	241	0.028	BB	0.02
3	7.02	C15:0	0.00	0.000	1379	0.160	BB	0.02
4	7.89	C16:0	0.00	0.000	72876	8.453	BV	0.02
5	8.02		0.00	0.000	420	0.049	VV	0.03
6	8.09	C16:1	0.00	0.000	6376	0.740	VB	0.03
7	8.32		0.00	0.000	488	0.057	BB	0.03
8	8.55		0.00	0.000	208	0.024	BB	0.03
9	8.93	C17:0	0.00	0.000	574	0.067	BV	0.03
10	9.18	C17:1	0.00	0.000	22752	2.639	VB	0.03
11	10.16	C18:0	0.00	0.000	19438	2.255	BB	0.04
12	10.37	C18:1	0.00	0.000	156120	18.109	BV	0.04
13	10.46		0.00	0.000	19474	2.259	VB	0.03
14	10.86	C18:2	0.00	0.000	306032	35.497	BB	0.04
15	11.34		0.00	0.000	973	0.113	BV	0.04
16	11.53	C18:3n3	0.00	0.000	244292	28.336	VB	0.04
17	12.24		0.00	0.000	213	0.025	BB	0.04
18	13.14	C20:0	0.00	0.000	3905	0.453	BV	0.04
19	13.39	C20:1	0.00	0.000	1952	0.226	VB	0.04
20	14.01	C20:2	0.00	0.000	364	0.042	BB	0.05
21	14.84	C20:3n3	0.00	0.000	324	0.038	BB	0.05
22	16.63	C22:0	0.00	0.000	1371	0.159	BB	0.05
23	18.48	C22:4	0.00	0.000	429	0.050	BB	0.05
24	20.52	C24:0	0.66	100.000	662	0.077	BB	0.08

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Figure A.4.3 Fatty acid profile chromatogram for the seed fraction – November (Sample 1).

Total Area = 862124.2 Total Height = 389306 Total Amount = 0.6622307

Figure A.4.3 Fatty acid profile chromatogram for the seed fraction – November (Sample 1). (cont'd)

Table A.4.2 Major fatty acids of fruit and seed fractions
(Coordinates with Tables 3.5 and A.4.1)

Month /sample	Fatty acid amount, % (w/w)[a]						
	C16:0	C16:1*n*7	C18:0	C18:1*n*9	Unknown	C18:2*n*6	C18:3*n*3
				Fruit fraction			
September							
Sample1	32.43	26.58	1.37	18.97	8.24	6.55	2.27
Sample 2	31.92	25.07	1.49	20.25	8.29	6.77	2.40
Sample 3	32.04	26.02	1.36	18.87	8.05	7.08	2.65
Sample 4	32.48	26.43	1.38	18.60	7.93	6.65	2.43
Sample 5	31.67	26.26	1.33	18.31	8.14	7.17	2.93
Sample 6	32.16	25.82	1.36	18.40	7.91	7.28	2.90
Sample 7	32.12	26.78	1.32	18.47	8.20	6.63	2.40
Sample 8	32.05	26.23	1.40	18.99	8.29	6.80	2.29
Sample 9	32.05	26.28	1.33	18.36	8.00	7.15	2.83
Mean	32.10	26.16	1.37	18.80	8.12	6.90	2.57
SD[b]	0.24	0.50	0.05	0.60	0.15	0.28	0.26
CV, %[c]	7.48	1.91	3.65	3.19	1.85	4.06	10.1
November							
Sample 1	32.34	26.84	1.19	17.7	7.99	7.08	2.51
Sample 2	32.66	26.87	1.18	17.51	8.01	7.13	2.36
Sample 3	32.41	26.86	1.21	17.80	8.10	7.12	2.34
Sample 4	32.52	26.94	1.17	18.22	8.01	6.78	2.25
Sample 5	33.03	26.82	1.20	18.45	7.89	6.56	2.08
Sample 6	32.29	26.62	1.19	18.12	8.20	7.00	2.21
Sample 7	31.94	25.77	1.22	17.93	7.69	6.56	2.21
Sample 8	31.99	27.13	1.19	18.67	8.03	6.57	2.20
Sample 9	30.89	27.52	1.26	19.09	8.44	6.58	2.20
Mean	32.23	26.82	1.20	18.17	8.04	6.82	2.26
SD	0.60	0.46	0.03	0.50	0.21	0.26	0.12
CV, %	1.86	1.72	2.50	2.75	2.61	3.81	5.31
January							
Sample1	31.54	28.02	1.24	18.93	8.22	6.43	2.02
Sample 2	32.76	27.00	1.2	18.37	7.92	6.63	2.04
Sample 3	32.69	26.72	1.22	18.24	7.86	6.72	2.08
Sample 4	32.25	26.30	1.28	18.70	8.02	6.71	2.07
Sample 5	32.72	25.95	1.34	19.44	8.03	6.72	2.13
Sample 6	32.47	26.15	1.29	18.66	8.12	7.10	2.17
Sample 7	32.92	26.66	1.31	19.80	7.85	6.21	1.84
Sample 8	32.92	26.51	1.31	19.75	7.85	6.30	1.83
Sample 9	29.58	25.33	1.71	21.36	9.06	6.91	1.97
Mean	32.21	26.52	1.32	19.25	8.10	6.64	2.02
SD	1.07	0.75	0.15	0.97	0.38	0.28	0.12
CV, %	3.32	2.83	11.36	5.04	4.69	4.22	5.94

Table A.4.2 Major fatty acids of fruit and seed fractions (cont'd)

Month /sample	Fatty acid amount % w/w						
	C16:0	C16:1*n*7	C18:0	C18:*1n*9	Unknown	C18:2*n*6	C18:3*n*3
				Seed fraction			
September							
Sample1	8.59	0.79	2.35	19.52	2.35	36.18	28.56
Sample 2	8.55	0.75	2.28	19.21	2.28	36.15	29.09
Sample 3	8.56	0.80	2.28	19.18	2.27	36.16	29.06
Mean	8.57	0.78	2.30	19.30	2.30	36.16	28.90
SD	0.02	0.03	0.04	0.19	0.04	0.02	0.30
CV, %	0.23	3.85	1.74	9.84	1.74	0.06	1.04
November							
Sample 1	8.68	0.76	2.32	18.60	2.32	36.46	29.10
Sample 2	8.51	0.57	2.48	19.80	2.30	36.64	28.01
Sample 3	8.35	0.59	2.21	18.64	2.22	36.69	29.52
Mean	8.51	0.64	2.34	19.01	2.28	36.60	28.88
SD	0.17	0.10	0.14	0.68	0.05	0.12	0.78
CV, %	2.00	15.63	5.98	3.58	2.19	0.33	2.70
January							
Sample 1	8.61	0.66	2.50	20.63	2.29	36.56	27.01
Sample 2	8.24	0.85	2.21	19.46	2.21	36.31	28.95
Sample 3	8.31	0.65	2.24	19.67	2.24	36.36	28.73
Mean	8.39	0.72	2.32	19.92	2.25	36.41	28.23
SD	0.19	0.11	0.16	0.63	0.04	0.13	1.06
CV, %	2.26	15.28	6.90	3.16	1.78	0.36	3.75

[a] C16:0 = palmitic acid. C16:1*n*7 = palmitoleic acid. C18:0 = stearic acid. C18:1*n*9 = oleic acid. C18:2*n*6 = linoleic acid. C18:3*n*3 = α-linolenic acid; % mass/mass (w/w).
[b] *SD* = standard deviation.
[c] *CV* = coefficient of variation.

Refer to "Sample calculations for Table A.1.1" in Appendix A.1 for Mean, *SD*, and *CV*.

Appendix A.5 Tocol analysis for sea buckthorn berries

Table A.5.1 Tocol concentration analysis for fruit and seed fractions
(Coordinates with Tables 3.6 and A.5.2)

	Tocols[a]							
	α – T	α – T3	β – T	β – T3[b] or P-8	γ – T	γ – T3	δ – T	δ – T3
	Fruit fraction (November, sample 7)							
Retention time, min	6.83	7.85	8.87	10.16	10.84	12.77	15.62	18.56
Y_{tocol}[c]	2624946	68790	141148	552810	54786	96114	204417	16823
$X1_{tocol}$[d] ng/10μL	66.231	1.736	2.288	8.960	1.118	1.961	3.170	0.261
C_{oil}[e] mg/mL	2.88	2.88	2.88	2.88	2.88	2.88	2.88	2.88
$X2_{tocol}$[f] mg/100g	230.0	6.0	7.9	31.1	3.9	6.8	11.0	0.9
	Seed fraction (November, sample 1)							
Retention time, min	6.82	n/d[g]	8.85	10.13	10.81	n/d	15.65	n/d
Y_{tocol}	2139235	n/d	279521	70700	1173974	n/d	198960	n/d
$X1_{tocol}$ ng/10μL	53.976	n/d	4.530	1.146	23.947	n/d	3.085	n/d
C_{oil} mg/mL	3.48	3.48	3.48	3.48	3.48	3.48	3.48	3.48
$X2_{tocol}$ mg/100g	155.1	n/d	13.0	3.3	68.8	n/d	8.9	n/d

[a] α-T = α-tocopherol. β-T = β-tocopherol. γ-T = γ-tocopherol. δ-T = δ-tocopherol. α-T3 = α-tocotrienol. β-T3 = β-tocotrienol. γ-T3 = γ-tocotrienol. δ-T3 = δ-tocotrienol. P-8 = Plastochromanol-8.
[b] β-T3 = β-T3 is present in fruit oil; P-8 = P-8 is present in seed oil.
[c] Y_{tocol} = area of individual tocol.
[d] $X1_{tocol}$ = concentration of tocol in prepared hexane solution.
[e] C_{oil} = concentration of oil in hexane solution.
[f] $X2_{tocol}$ = concentration of tocol in oil.
[g] n/d = not detected.

Sample calculations for Table A.5.1: [α-tocopherol concentration (fruit fraction)]

1) Identify individual tocols shown in Figures A.5.3 (fruit fraction) and A.5.4 (seed fraction) based on calibration chromatograms and data (Figures A.5.1a to i; Figures A.5.2a - e).

2) Record area "Y_{tocol}" for individual identified tocols in Table A.5.1.

3) Calculate amount of α-tocopherol (α-T) in hexane solution using Eqn. A.5a:

$$Y_{\alpha\text{-}T} = 39633.3X_{\alpha\text{-}T} + 0 \qquad \text{(A.5a)}$$

where $Y_{\alpha\text{-}T}$ = area of tocol on chromatogram and $X_{\alpha\text{-}T}$ = amount of tocol in 10 μL hexane.

Note: Eqn. (A.5a) also applies to calculation of α-tocotrienol.

$$X1_{\alpha\text{-}T} = \frac{2624946}{39633.3}$$

4) Convert amount of α-tocopherol in prepared hexane solution to oil basis using Eqn. A.5b:

$$X2_{tocol} = \frac{X1_{tocol}\,\dfrac{10^3\,\mu L}{mL}}{C_{oil}} \times \frac{mg \times 10^3\,mg}{10^6\,ng \times g} \times \frac{100}{100} \qquad \text{(A.5b)}$$

where $X2_{tocol}$ = concentration of tocol in 100 g oil and C_{oil} = concentration of oil in hexane solution.

$$X2_{\alpha\text{-}T} = \frac{66.23ng}{10\mu L\_hexane\_sol'n} \times \frac{1000\mu L}{mL} \times \frac{mL\ hexane\ sol'n}{2.88mg\ oil} \times \frac{mg \times 1000mg}{10^6\,ng \times g} \times \frac{100}{100}$$

5) To calculate β-tocopherol and tocotrienol, γ-tocopherol and –tocotrienol, and δ-tocopherol and tocotrienol use equations A.5c, A.5d, and A.5e, respectively:

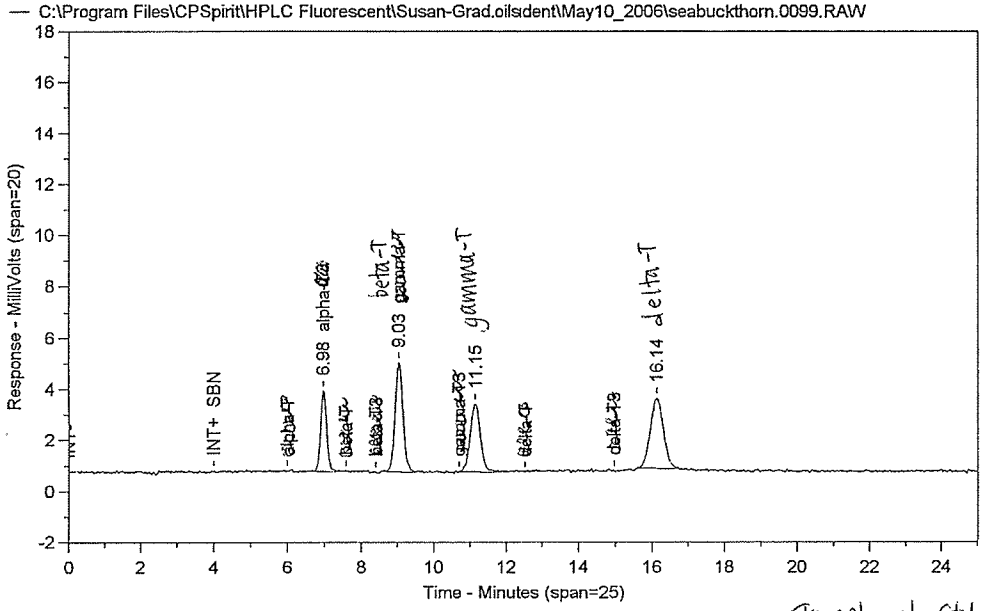
(β-T and β-T3) $$Y = 61698.06X + 0$$ (A.5c)

(γ-T and γ-T3) $$Y = 49023.2X + 0$$ (A.5d)

(δ-T and δ-T3) $$Y = 64487.47X + 0$$ (A.5e)

Chrom Perfect Chromatogram Report

— C:\Program Files\CPSpirit\HPLC Fluorescent\Susan-Grad.oilsdent\May10_2006\seabuckthorn.0099.RAW



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Heading 1 = Tocopherol Analysis -Susan -seabuckthorn
Heading 2 = May10,2006; 10uL inj; 0.8mL/min; 25 min. run time

Sample Identification: Tocopherol Std
Wt (mg) = _____ 50,000X
mg/mL = _____ 1µL

1mg

Today's Date = 5/13/06 Today's Time = 11:22:21 AM
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Sample Name = oils

Method File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol.MET
Method Description = Tocopherol Analysis - column: Phenomenex 250x3.2mm 5micron; 5u silicon
Calibration File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol_T3.CAL
Run Time = 25

Peak #	Ret. Time	Peak Name	Amount	Amt %	Area	Area %	
1	6.98	alpha-T3	1.810	39.532	36528	16.30	alpha-T
2	9.03	gamma-T	2.768	60.468	65062	29.03	beta-T
3	11.15		0.000	0.000	53184	23.73	gamma-T
4	16.14		0.000	0.000	69360	30.95	delta-T

Total Area = 224133.7 Total Amount = 4.577673

Checked by _____ Date _____

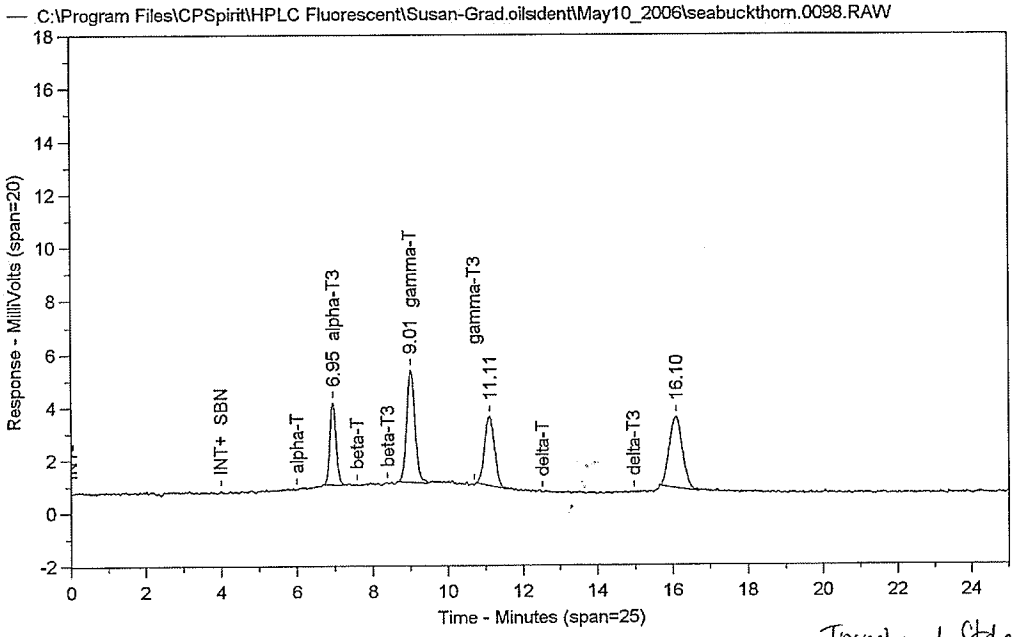
ug/g or ppm
alpha-T = _____
beta-T = _____
P-8 = _____
gamma-T = _____
delta-T = _____

Printed on 5/13/06 11:22:22 AM Page 1 of 1

Figure A.5.1a Calibration chromatogram (50,000 x 1µL – run 1).

Chrom Perfect Chromatogram Report

— C:\Program Files\CPSpirit\HPLC Fluorescent\Susan-Grad.oilsdent\May10_2006\seabuckthorn.0098.RAW



Instrument = HPLC Fluorescent
Heading 1 = Tocopherol Analysis -Susan -seabuckthorn
Heading 2 = May10,2006; 10uL inj; 0.8mL/min; 25 min. run time

Sample Identification: Tocopherol Stds.
Wt. (mg) = _____ 50,000x
mg/mL = _____ 1µl

Today's Date = 5/13/06 Today's Time = 11:22:32 AM
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Sample Name = oils

Method File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol.MET
Method Description = Tocopherol Analysis - column: Phenomenex 250x3.2mm 5micron; 5u silicon
Calibration File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol_T3.CAL
Run Time = 25

Peak #	Ret. Time	Peak Name	Amount	Amt %	Area	Area %	
1	6.95	alpha-T3	1.717	38.319	34662	16.26	alpha-T
2	9.01	gamma-T	2.764	61.681	64972	30.47	beta-T
3	11.11		0.000	0.000	48966	22.97	gamma-T
4	16.10		0.000	0.000	64608	30.30	delta-T

Total Area = 213209 Total Amount = 4.481432

Checked by _____ Date _____

ug/g or ppm
alpha-T = _____
beta-T = _____
P-8 = _____
gamma-T = _____
delta-T = _____

Printed on 5/13/06 11:22:33 AM Page 1 of 1

Figure A.5.1b Calibration chromatogram (50,000 x 1µL – run 2).

Chrom Perfect Chromatogram Report

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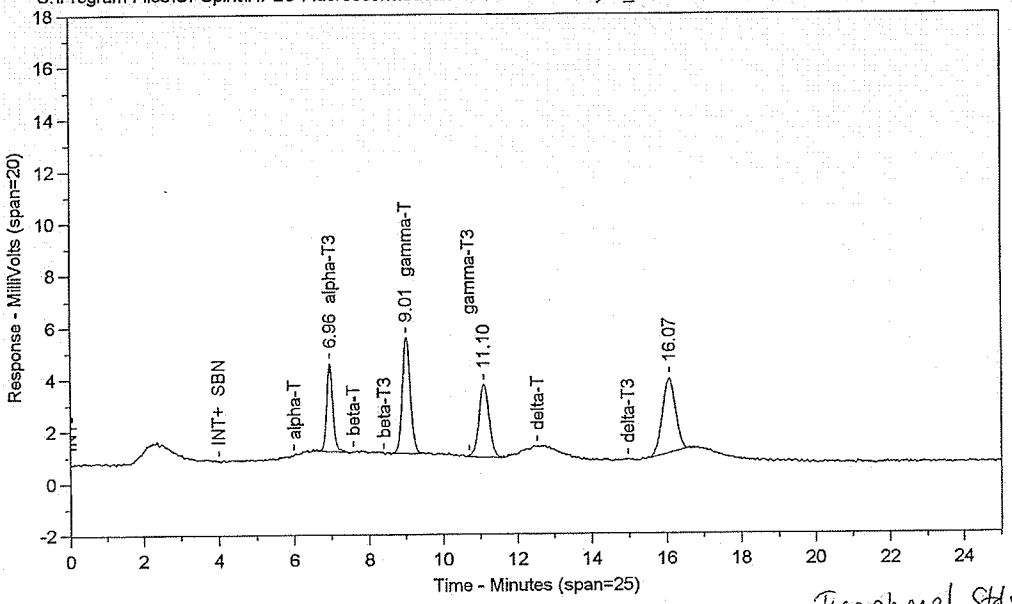

Instrument = HPLC Fluorescent
Heading 1 = Tocopherol Analysis -Susan -seabuckthorn
Heading 2 = May10,2006; 10uL inj; 0.8mL/min; 25 min. run time

Sample Identification: Tocopherol Stds.
Wt. (mg) = _____ 50,000x
mg/mL = _____ 1 µL

Today's Date = 5/13/06 Today's Time = 11:22:54 AM
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Sample Name = oils

Method File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol.MET
Method Description = Tocopherol Analysis - column: Phenomenex 250x3.2mm 5micron; 5u silicon
Calibration File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol_T3.CAL
Run Time = 25

Peak #	Ret. Time	Peak Name	Amount	Amt %	Area	Area %	
1	6.96	alpha-T3	1.958	40.434	39528	16.99	alpha-T
2	9.01	gamma-T	2.885	59.566	67811	29.15	beta-T
3	11.10		0.000	0.000	54658	23.49	gamma-T
4	16.07		0.000	0.000	70669	30.37	delta-T

Total Area = 232667.1 Total Amount = 4.843285

Checked by _____ Date _____

ug/g or ppm
alpha-T = _____
beta-T = _____
P-8 = _____
gamma-T = _____
delta-T = _____

Printed on 5/13/06 11:22:55 AM Page 1 of 1

Figure A.5.1c Calibration chromatogram (50,000 x 1 µL – run 3).

Chrom Perfect Chromatogram Report

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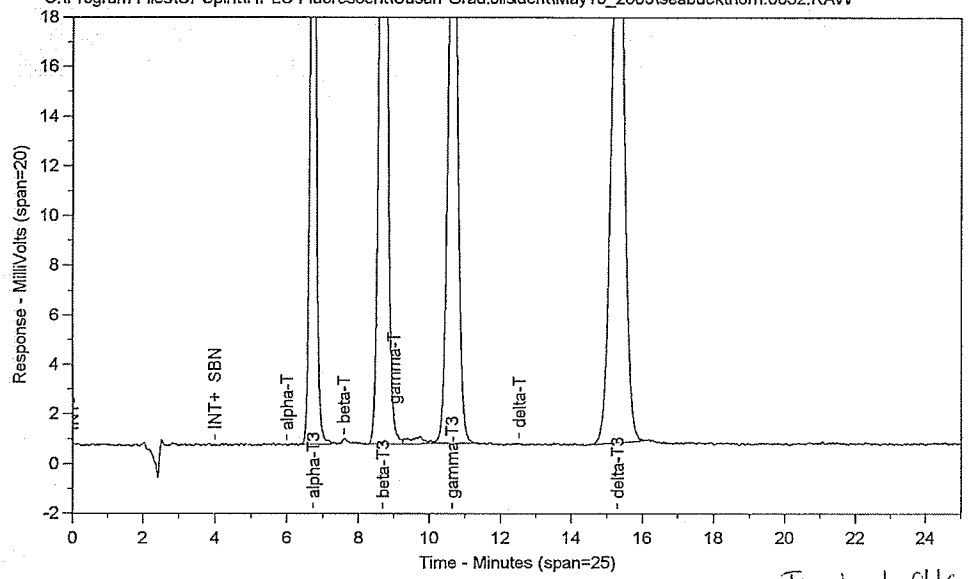


Instrument = HPLC Fluorescent
Heading 1 = Tocopherol Analysis -Susan -seabuckthorn
Heading 2 = May10,2006; 10uL inj; 0.8mL/min; 25 min. run time

Sample Identification: Tocopherol Stds
Wt (mg) = _____ 50,000X
mg/mL = _____ 10µl

Today's Date = 5/15/06 Today's Time = 10:34:50 AM
Raw File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Susan-Grad.Student\May10_2006\seabuckthorn.0032.RAW
Sample Name = oils

10ng

Method File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol.MET
Method Description = Tocopherol Analysis - column: Phenomenex 250x3.2mm 5micron; 5u silicon
Calibration File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol_T3.CAL
Run Time = 25

Peak #	Ret. Time	Peak Name	Amount	Amt %	Area	Area %	
1	6.72	alpha-T3	17.479	22.134	352807	17.51	alpha-T
2	8.69	beta-T3	21.903	27.737	581100	28.83	beta-T
3	10.65	gamma-T3	19.524	24.724	458905	22.77	gamma-T
4	15.31	delta-T3	20.062	25.405	622603	30.89	delta-T

Total Area = 2015415 Total Amount = 78.96758

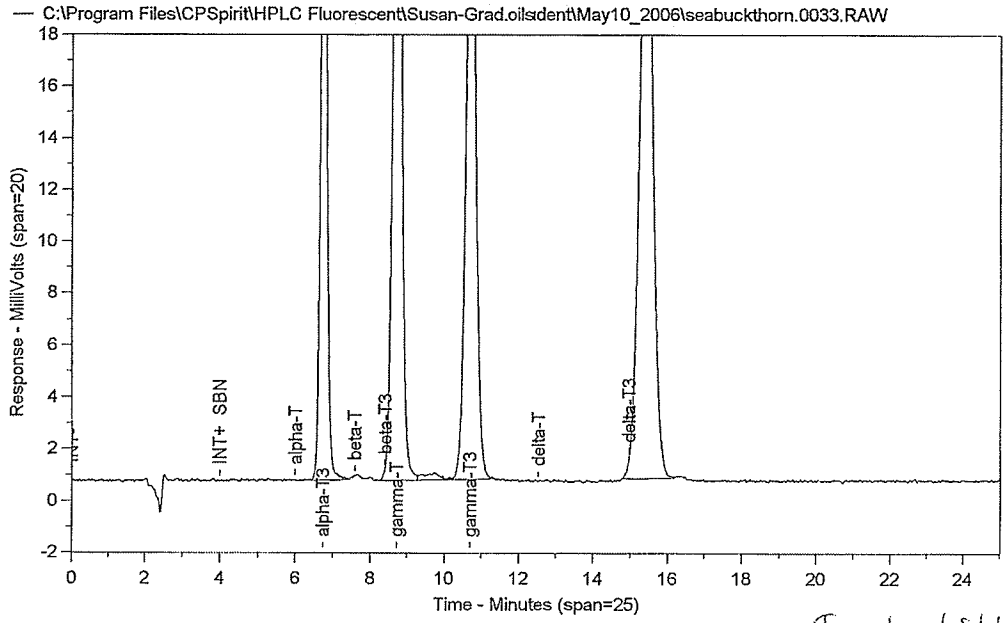
Checked by _____ Date _____

ug/g or ppm
alpha-T = _____
beta-T = _____
P-8 = _____
gamma-T = _____
delta-T = _____

Printed on 5/15/06 10:34:51 AM Page 1 of 1

Figure A.5.1d Calibration chromatogram (50,000 x 10 µL – run 1).

Chrom Perfect Chromatogram Report



Instrument = HPLC Fluorescent
Heading 1 = Tocopherol Analysis -Susan -seabuckthorn
Heading 2 = May10,2006; 10uL inj; 0.8mL/min; 25 min. run time

Sample Identification: Tocopherol Stds.
Wt. (mg) = _____ 50,000x
mg/mL = _____ 10µl

Today's Date = 5/15/06 Today's Time = 10:34:59 AM
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Sample Name = oils

Method File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol.MET
Method Description = Tocopherol Analysis - column: Phenomenex 250x3.2mm 5micron; 5u silicon
Calibration File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol_T3.CAL
Run Time = 25

Peak #	Ret. Time	Peak Name	Amount	Amt %	Area	Area %	
1	6.75	alpha-T3	17.584	28.311	354924	17.56	alpha-T
2	8.72	gamma-T	24.998	40.249	587571	29.07	beta-T
3	10.71	gamma-T3	19.527	31.440	458979	22.70	gamma-T
4	15.43		0.000	0.000	620082	30.67	delta-T

Total Area = 2021556 Total Amount = 62.10839

Checked by _____ Date _____

ug/g or ppm
alpha-T = _____
beta-T = _____
P-8 = _____
gamma-T = _____
delta-T = _____

Printed on 5/15/06 10:35:00 AM Page 1 of 1

Figure A.5.1e Calibration chromatogram (50,000 x 10 µL – run 2).

Chrom Perfect Chromatogram Report

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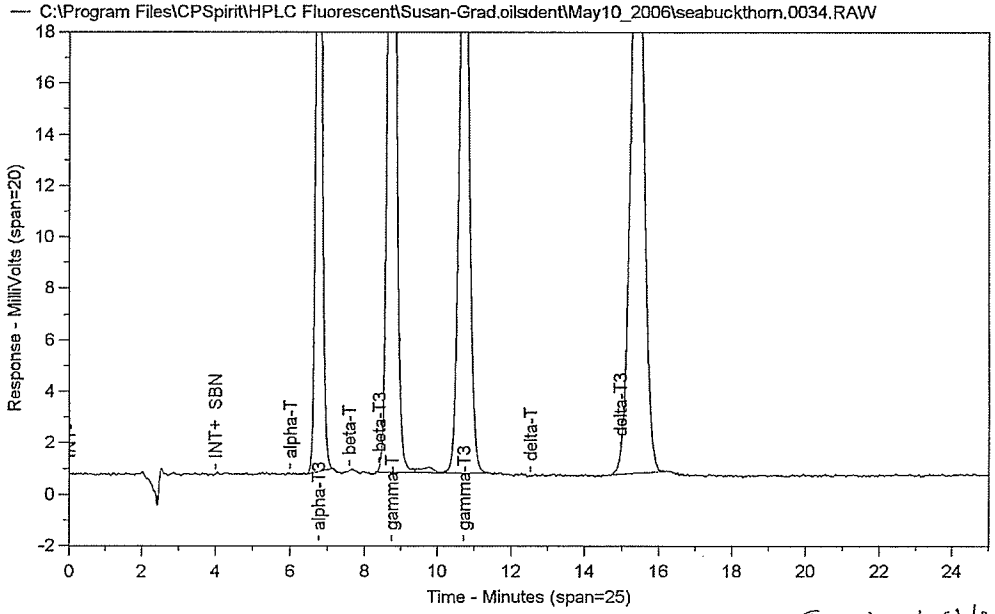

Instrument = HPLC Fluorescent
Heading 1 = Tocopherol Analysis -Susan -seabuckthorn
Heading 2 = May10,2006; 10uL inj; 0.8mL/min; 25 min. run time

Sample Identification: Tocopherol Stds.
Wt. (mg) = _____ 50,000 x
mg/mL = _____ 10 µL

Today's Date = 5/15/06 Today's Time = 10:35:09 AM
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Sample Name = oils

Method File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol.MET
Method Description = Tocopherol Analysis - column: Phenomenex 250x3.2mm 5micron; 5u silicon
Calibration File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol_T3.CAL
Run Time = 25

Peak #	Ret. Time	Peak Name	Amount	Amt %	Area	Area %	
1	6.77	alpha-T3	17.269	28.132	348575	17.41	alpha-T
2	8.74	gamma-T	24.712	40.257	580857	29.02	beta-T
3	10.72	gamma-T3	19.404	31.610	456090	22.78	gamma-T
4	15.42		0.000	0.000	616313	30.79	delta-T

Total Area = 2001835 Total Amount = 61.3853

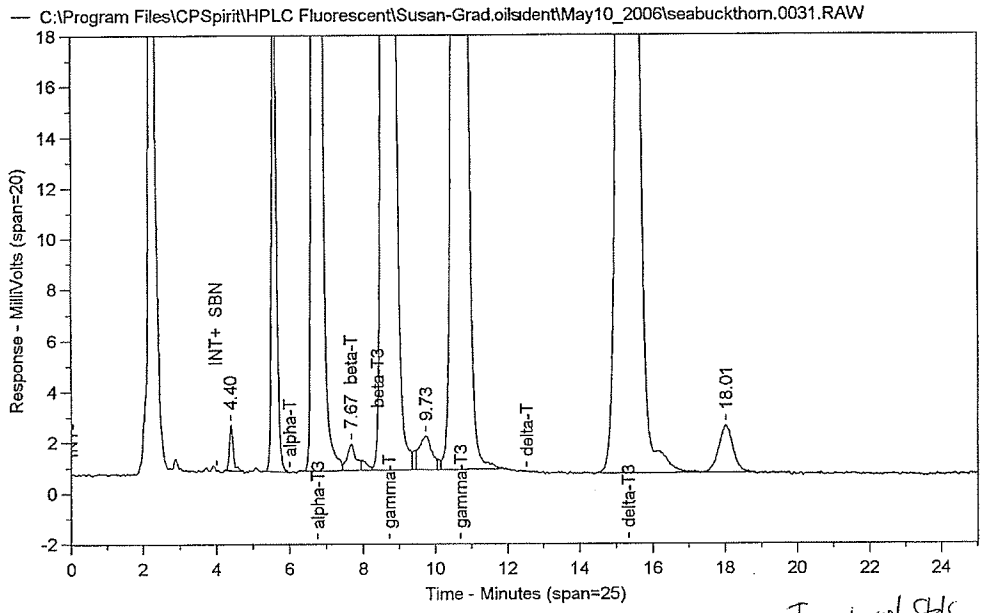
Checked by _____ Date _____

ug/g or ppm
alpha-T = _____
beta-T = _____
P-8 = _____
gamma-T = _____
delta-T = _____

Printed on 5/15/06 10:35:10 AM Page 1 of 1

Figure A.5.1f Calibration chromatogram (50,000 x 10 µL – run 3).

Chrom Perfect Chromatogram Report



Instrument = HPLC Fluorescent
Heading 1 = Tocopherol Analysis -Susan -seabuckthorn
Heading 2 = May10,2006; 10uL inj; 0.8mL/min; 25 min. run time

Sample Identification: Tocopherol Stds.
Wt. (mg) = _____ 10,000 X
mg/mL = _____ 10µL

50mg

Today's Date = 5/15/06 Today's Time = 10:34:38 AM
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Sample Name = oils

Method File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol.MET
Method Description = Tocopherol Analysis - column: Phenomenex 250x3.2mm 5micron; 5u silicon
Calibration File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol_T3.CAL
Run Time = 25

Peak #	Ret. Time	Peak Name	Amount	Amt %	Area	Area %	
1	4.40		0.000	0.000	13278	0.12	
2	5.59		0.000	0.000	178130	1.60	
3	6.76	alpha-T3	99.951	22.578	2017497	18.12	alpha-T
4	7.67	beta-T	0.657	0.149	17442	0.16	
5	8.72	gamma-T	132.388	29.905	3111760	27.95	beta-T
6	9.73		0.000	0.000	31309	0.28	
7	10.69	gamma-T3	105.281	23.782	2474619	22.22	gamma-T
8	15.34	delta-T3	104.410	23.585	3240276	29.10	delta-T
9	18.01		0.000	0.000	50474	0.45	

Total Area = 1.113478E+07 Total Amount = 442.6866

Checked by _____ Date _____

ug/g or ppm
alpha-T = _____

Printed on 5/15/06 10:34:39 AM Page 1 of 2

Figure A.5.1g Calibration chromatogram (10,000 x 10 µL – run 1).

Chrom Perfect Chromatogram Report

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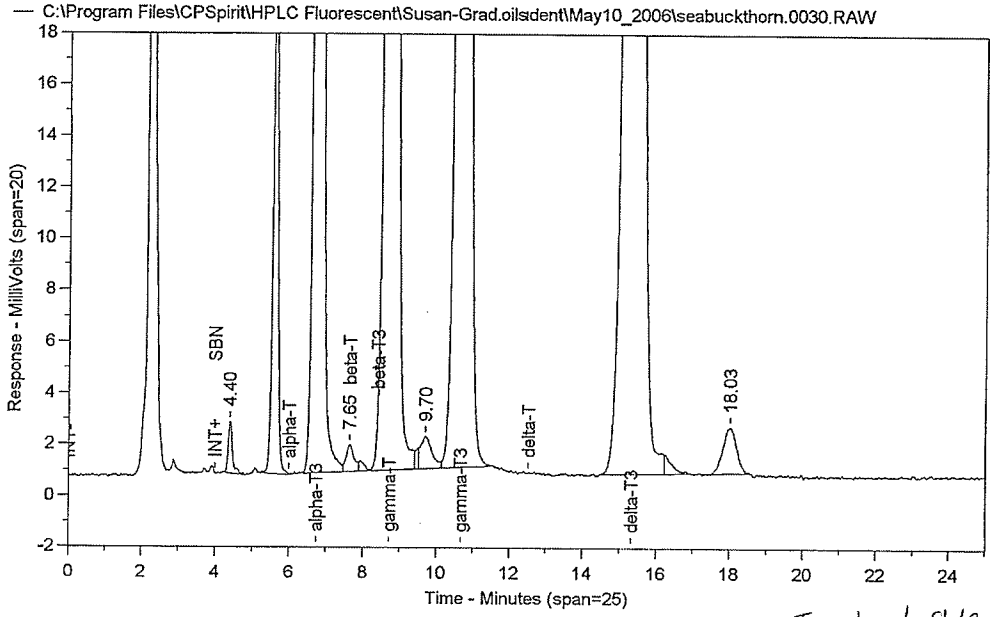

Instrument = HPLC Fluorescent
Heading 1 = Tocopherol Analysis -Susan -seabuckthorn
Heading 2 = May10,2006; 10uL inj; 0.8mL/min; 25 min. run time

Sample Identification: Tocopherol Stds.
Wt (mg) = _____ 10,000X
mg/mL = _____ 10µl

Today's Date = 5/15/06 Today's Time = 10:34:27 AM
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Sample Name = oils

Method File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol.MET
Method Description = Tocopherol Analysis - column: Phenomenex 250x3.2mm 5micron; 5u silicon
Calibration File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol_T3.CAL
Run Time = 25

Peak #	Ret. Time	Peak Name	Amount	Amt %	Area	Area %	
1	4.40		0.000	0.000	16502	0.15	
2	5.59		0.000	0.000	199333	1.77	
3	6.74	alpha-T3	101.179	22.632	2042280	18.14	alpha-T
4	7.65	beta-T	0.590	0.132	15643	0.14	
5	8.71	gamma-T	133.753	29.918	3143852	27.93	beta-T
6	9.70		0.000	0.000	27474	0.24	
7	10.67	gamma-T3	106.110	23.735	2494108	22.16	gamma-T
8	15.33	delta-T3	105.430	23.583	3271944	29.06	delta-T
9	18.03		0.000	0.000	46275	0.41	

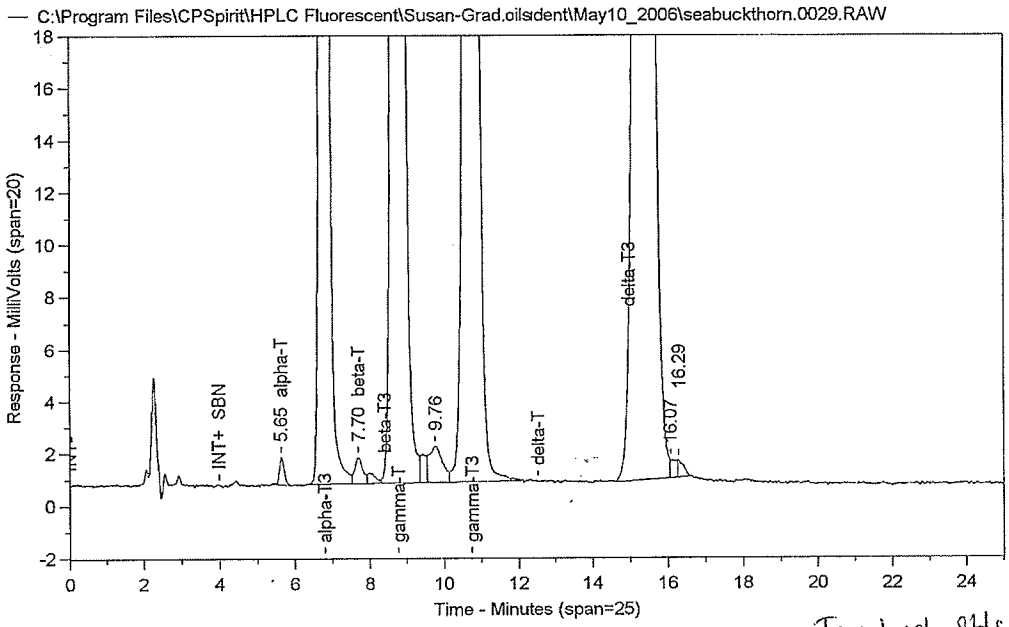
Total Area = 1.125741E+07 Total Amount = 447.0616

Checked by _____ Date _____

ug/g or ppm
alpha-T = _____

Figure A.5.1h Calibration chromatogram (10,000 x 10 µL – run 2).

Chrom Perfect Chromatogram Report



Instrument = HPLC Fluorescent
Heading 1 = Tocopherol Analysis -Susan -seabuckthorn
Heading 2 = May10,2006; 10uL inj; 0.8mL/min; 25 min. run time

Sample Identification: Tocopherol Stds
Wt. (mg) = _____ 10,000x
mg/mL = _____ 10µl

Today's Date = 5/12/06 Today's Time = 3:20:22 PM
Raw File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Susan-Grad.Student\May10_2006\seabuckthorn.0029.RAW
Sample Name = oils

Method File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol.MET
Method Description = Tocopherol Analysis - column: Phenomenex 250x3.2mm 5micron; 5u silicon
Calibration File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol_T3.CAL
Run Time = 25

Peak #	Ret. Time	Peak Name	Amount	Amt %	Area	Area %	
1	5.65	alpha-T	0.454	0.139	9167	0.09	
2	6.80	alpha-T3	94.721	29.014	1911921	18.07	alpha-T
3	7.70	beta-T	0.568	0.174	15076	0.14	
4	8.77	gamma-T	128.446	39.345	3019124	28.53	beta-T
5	9.76		0.000	0.000	33924	0.32	
6	10.74	gamma-T3	102.272	31.327	2403901	22.71	gamma-T
7	15.39		0.000	0.000	3175803	30.01	delta-T
8	16.07		0.000	0.000	7597	0.07	
9	16.29		0.000	0.000	6981	0.07	

Total Area = 1.05835E+07 Total Amount = 326.4617

Checked by _____ Date _____

ug/g or ppm
alpha-T = _____

Printed on 5/12/06 3:20:24 PM

Page 1 of 2

Figure A.5.1i Calibration chromatogram (10,000 x 10 µL – run 3).

	alpha-T	beta-T	gamma-T	delta-T
1ng	36528	65062	53184	69360
	34662	64972	48966	64608
	39528	67811	54658	70669
AVG	36906	65948.33	52269.33	68212.33
STDEV	2454.924	1613.744	2954.18	3189.323
CV%	6.651829	2.446983	5.651841	4.675582

	alpha-T	beta-T	gamma-T	delta-T
10ng	352807	581100	458905	622603
	354924	587571	458979	620082
	348575	580857	456090	616313
AVG	352102	583176	457991.3	619666
STDEV	3232.68	3808.12	1647.019	3165.567
CV%	0.918109	0.652997	0.359618	0.510851

	alpha-T	beta-T	gamma-T	delta-T
50ng	2017497	3111760	2474619	3240276
	2042280	3143852	2494108	3271944
	1911921	3019124	2403901	3175803
AVG	1990566	3091579	2457543	3229341
STDEV	69226.63	64766.76	47466.06	48994.42
CV%	3.477736	2.094941	1.931444	1.517165

	alpha-T	beta-T	gamma-T	delta-T
100ng	3852353	6143649	4823424	6425946
	3818227	5991357	4782713	6330006
	3793311	5965460	4743171	6314995
AVG	3821297	6033489	4783103	6356982
STDEV	29640.48	96276.36	40127.92	60194.04
CV%	0.775665	1.5957	0.838952	0.946897

alpha-T

$y = 39633.3\,X + 0$ (X ↑ amt.)

beta-T

$y = 61698.06\,X + 0$ (X ↑ amt.)

gamma-T | P-8

$y = 49023.2\,X + 0$ (X ↑ amt.)

delta-T

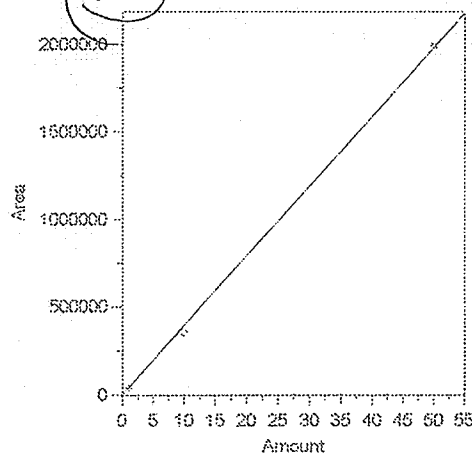
$y = 64487.47\,X + 0$ (X ↑ amt.)

- not included in calibration curve

Figure A.5.2a Tocol calibration data – summary.

Chrom Perfect Calibration File

1 alpha-T



Expected retention time:	5.84 minutes
Search window:	0.4 minutes
No retention time reference component	
Group number:	0
High alarm limit:	0
Low alarm limit:	0
Component constant:	0

Single peak quantification by area

$Y = 39633.3\ X + 0$

Linear fit with equal weighting, forced to origin

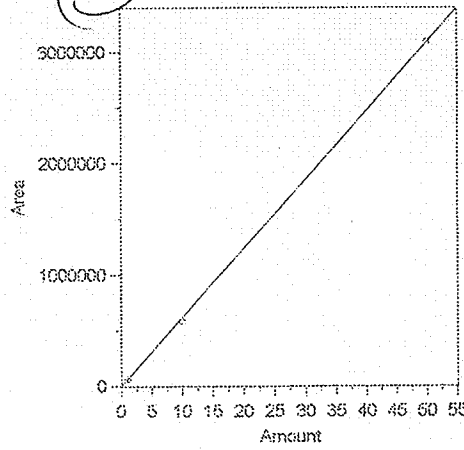
Coefficient of determination:	0.9990714
Average error:	6.164%
Average CF:	37309.17
RSD:	6.237%

Level	Amount	Response	Cal Factor	Error, %	Source	Date and time
1	10	352102	35210.2	-11.160	Manual	5/15/06 3:24:48 PM
2	50	1990566	39811.32	0.449	Manual	5/15/06 3:25:04 PM
3	1	36906	36906	-6.881	Manual	5/15/06 3:27:38 PM

Figure A.5.2b Calibration data – (α-tocopherol and α-tocotrienol).

Chrom Perfect Calibration File

2 beta-T



Expected retention time:	7.44 minutes
Search window:	0.4 minutes
No retention time reference component	
Group number:	0
High alarm limit:	0
Low alarm limit:	0
Component constant:	0

Single peak quantification by area

Y = 61698.06 X + 0

Linear fit with equal weighting, forced to origin

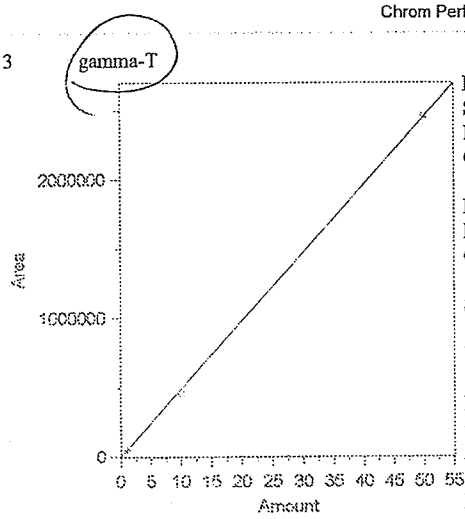
Coefficient of determination:	0.9997699
Average error:	4.195%
Average CF:	62032.5
RSD:	6.157%

Level	Amount	Response	Cal Factor	Error, %	Source	Date and time
1	10	583176	58317.6	-5.479	Manual	5/15/06 3:24:51 PM
2	50	3091579	61831.58	0.216	Manual	5/15/06 3:25:07 PM
3	1	65948.33	65948.33	6.889	Manual	5/15/06 3:27:41 PM

Figure A5.2c Calibration data – (β-tocopherol and β-tocotrienol).

Chrom Perfect Calibration File

3 gamma-T



Expected retention time:	8.93 minutes
Search window:	0.4 minutes
No retention time reference component	
Group number:	0
High alarm limit:	0
Low alarm limit:	0
Component constant:	0

Single peak quantification by area

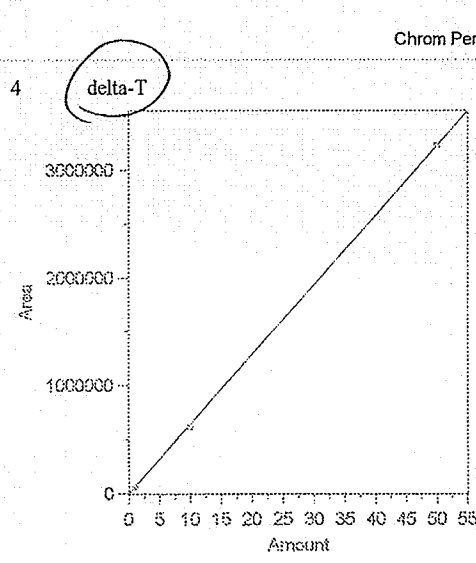
Y = 49023.2 X + 0

Linear fit with equal weighting, forced to origin

Coefficient of determination:	0.9996711
Average error:	4.486%
Average CF:	49073.11
RSD:	6.594%

Level	Amount	Response	Cal Factor	Error, %	Source	Date and time
1	10	457991.3	45799.13	-6.577	Manual	5/15/06 3:24:56 PM
2	50	2457543	49150.86	0.260	Manual	5/15/06 3:25:10 PM
3	1	52269.33	52269.33	6.622	Manual	5/15/06 3:27:44 PM

Figure A.5.2d Calibration data (γ-tocopherol and γ-tocotrienol).



Expected retention time:	12.47 minutes
Search window:	0.4 minutes
No retention time reference component	
Group number:	0
High alarm limit:	0
Low alarm limit:	0
Component constant:	0

Single peak quantification by area

Y = 64487.47 X + 0

Linear fit with equal weighting, forced to origin

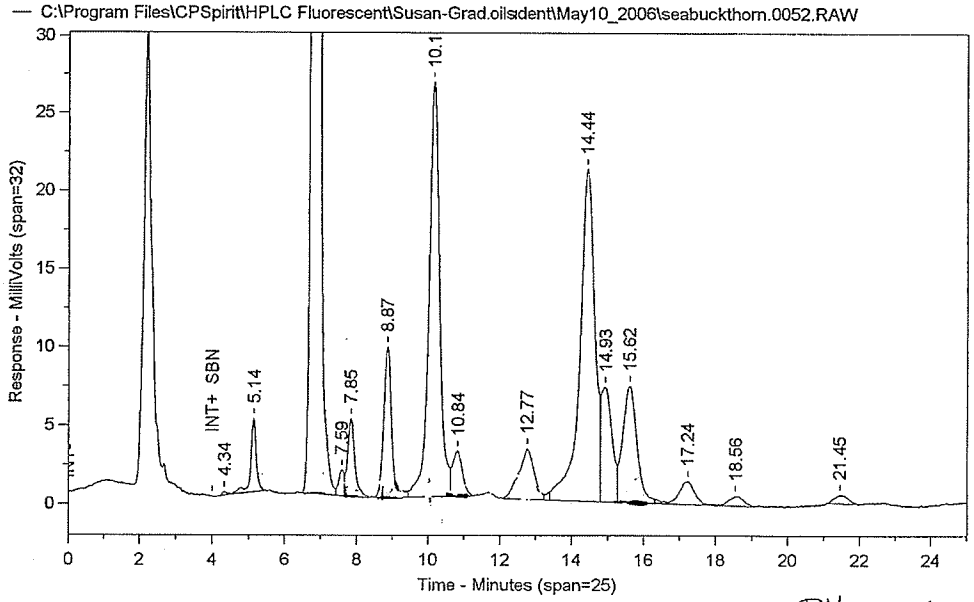
Coefficient of determination:	0.9998818
Average error:	3.280%
Average CF:	64921.92
RSD:	4.831%

Level	Amount	Response	Cal Factor	Error, %	Source	Date and time
1	10	619666	61966.6	-3.909	Manual	5/15/06 3:24:59 PM
2	50	3229341	64586.82	0.154	Manual	5/15/06 3:25:13 PM
3	1	68212.33	68212.33	5.776	Manual	5/15/06 3:27:47 PM

Figure A.5.2e Calibration data (δ-tocopherol and δ-tocotrienol).

Chrom Perfect Chromatogram Report

— C:\Program Files\CPSpirit\HPLC Fluorescent\Susan-Grad.oils\dent\May10_2006\seabuckthorn.0052.RAW



Instrument = HPLC Fluorescent
Heading 1 = Tocopherol Analysis -Susan -seabuckthorn
Heading 2 = May10,2006; 10uL inj; 0.8mL/min; 25 min. run time

Sample Identification: R11 P3-1
Wt. (g) = _____

Today's Date = 5/15/06 Today's Time = 11:05:18 AM
Raw File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Susan-Grad.Student\May10_2006\seabuckthorn.0052.RAW
Sample Name = oils

Method File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol5.MET
Method Description = Tocopherol Analysis - column: Phenomenex 250x3.2mm 5micron; 5u silicon
Calibration File Name =
Run Time = 25

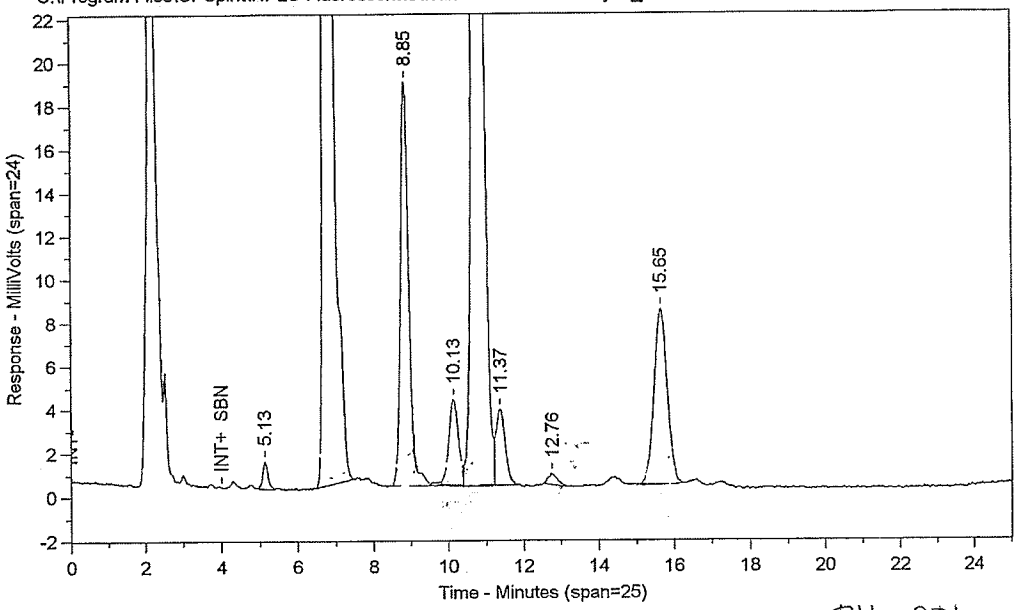
Peak #	Ret. Time	Peak Name	Amount	Amt %	Area	Area %	Group #
1	4.34		0.000	N/A	2403	0.05	[No data]
2	5.14		0.000	N/A	50890	1.09	[No data]
3	6.83	– alpha	0.000	N/A	2624946	56.40	[No data]
4	7.59		0.000	N/A	17444	0.37	[No data]
5	7.85	– alphaT3	0.000	N/A	68790	1.48	[No data]
6	8.87	– beta	0.000	N/A	141148	3.03	[No data]
7	10.16	– betaT3	0.000	N/A	552810	11.88	[No data]
8	10.84	– gamma	0.000	N/A	54786	1.18	[No data]
9	12.77	– gammaT3	0.000	N/A	96114	2.07	[No data]
10	14.44		0.000	N/A	614477	13.20	[No data]
11	14.93		0.000	N/A	152506	3.28	[No data]
12	15.62	– delta	0.000	N/A	204417	4.39	[No data]
13	17.24		0.000	N/A	41889	0.90	[No data]
14	18.56	– deltaT3	0.000	N/A	16823	0.36	[No data]
15	21.45		0.000	N/A	14623	0.31	[No data]

Printed on 5/15/06 11:05:19 AM Page 1 of 2

Figure A.5.3 Tocol chromatogram for the fruit fraction – November, sample 7.

Chrom Perfect Chromatogram Report

— C:\Program Files\CPSpirit\HPLC Fluorescent\Susan-Grad.oilsdent\May10_2006\seabuckthorn.0044.RAW



Instrument = HPLC Fluorescent
Heading 1 = Tocopherol Analysis -Susan -seabuckthorn
Heading 2 = May10,2006; 10uL inj; 0.8mL/min; 25 min. run time

Sample Identification: RII SPI
Wt. (g) = _____

Today's Date = 5/15/06 Today's Time = 11:02:17 AM
Raw File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Susan-Grad.Student\May10_2006\seabuckthorn.0044.RAW
Sample Name = oils

Method File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol5.MET
Method Description = Tocopherol Analysis - column: Phenomenex 250x3.2mm 5micron; 5u silicon
Calibration File Name =
Run Time = 25

Peak #	Ret. Time	Peak Name	Amount	Amt %	Area	Area %	Group #
1	5.13		0.000	N/A	11822	0.30	[No data]
2	6.82	– alpha	0.000	N/A	2139235	54.25	[No data]
3	8.85	– beta	0.000	N/A	279521	7.09	[No data]
4	10.13	– P-8	0.000	N/A	70700	1.79	[No data]
5	10.81	– gamma	0.000	N/A	1173974	29.77	[No data]
6	11.37		0.000	N/A	60682	1.54	[No data]
7	12.76		0.000	N/A	8226	0.21	[No data]
8	15.65	– delta	0.000	N/A	198960	5.05	[No data]

Total Area = 3943119 Total Amount = 0

Checked by _____ Date _____

Printed on 5/15/06 11:02:18 AM

Page 1 of 1

Figure A.5.4 Tocol chromatogram for the seed fraction – November, sample 1.

Table A.5.2 Tocol concentration in fruit and seed fractions
(Coordinates with Tables 3.6 and A.5.1)

Month/ sample	Tocol concentration mg/100 g oil[a]							
	α – T	α – T3	β - T	β – T3	γ - T	γ – T3	δ - T	δ – T3
	Fruit fraction							
September								
Sample 1	357.6	8.8	8.6	36.1	7.4	9.2	14.7	1.3
Sample 2	402.3	8.1	9.6	39.2	8.0	9.2	18.6	1.5
Sample 3	378.3	10.5	9.7	39.2	9.0	10.2	14.9	1.4
Sample 4	384.3	9.6	10.0	41.9	10.2	8.4	18.9	1.3
Sample 5	424.2	9.6	11.0	44.9	13.0	10.3	26.3	1.6
Sample 6	412.7	10.0	10.3	44.0	9.3	11.0	19.1	1.5
Sample 7	424.2	10.0	10.9	43.1	9.1	11.6	17.5	1.5
Sample 8	415.5	10.3	7.8	41.4	10.0	9.7	16.6	1.6
Sample 9	295.2	4.9	12.5	28.2	7.4	6.3	11.8	0.8
Mean	388.2	9.1	10.0	39.8	9.3	9.5	17.6	1.4
SD[b]	41.6	1.7	1.4	5.1	1.7	1.5	4.0	0.2
CV, %[c]	10.7	18.7	14.0	12.8	18.3	15.8	22.7	14.3
November								
Sample 1	339.8	12.6	12.4	37.5	4.8	6.3	15.3	1.2
Sample 2	345.9	11.9	12.8	38.0	4.1	6.2	16.3	0.8
Sample 3	341.3	13.0	12.8	37.1	3.1	9.8	13.8	0.9
Sample 4	353.9	12.1	11.9	40.3	3.3	8.4	14.8	1.2
Sample 5	338.9	13.1	11.9	40.4	3.5	8.5	14.5	1.5
Sample 6	385.2	11.3	14.4	46.7	5.0	10.9	18.2	1.4
Sample 7	230.0	6.0	7.9	31.1	3.9	6.8	11.0	0.9
Sample 8	354.5	14.5	13.2	42.1	4.1	12.1	15.7	1.3
Sample 9	348.8	12.6	12.8	38.2	3.4	9.0	14.5	1.5
Mean	337.6	11.9	12.2	39.0	3.9	8.7	14.9	1.2
SD	42.8	2.4	1.8	4.2	0.7	0.6	2.0	0.2
CV, %	12.7	20.2	14.8	10.8	17.9	6.9	13.4	16.7
January								
Sample 1	299.0	13.1	10.2	30.3	2.4	7.8	12.8	1.3
Sample 2	238.4	7.1	8.8	25.4	3.2	5.6	9.2	0.7
Sample 3	287.4	10.7	11.2	28.3	2.2	7.3	7.5	0.9
Sample 4	304.3	11.8	11.4	32.4	2.2	9.0	9.2	0.8
Sample 5	275.7	10.8	10.3	42.2	3.2	10.2	11.0	1.7
Sample 6	220.1	7.9	7.7	29.8	2.0	6.3	7.5	0.9
Sample 7	276.6	10.3	9.6	32.3	2.4	8.1	8.8	1.5
Sample 8	266.8	11.6	9.3	30.0	1.9	7.6	8.2	1.2
Sample 9	283.6	10.8	10.5	31.7	2.2	8.7	10.6	1.2
Mean	272.4	10.5	9.9	31.4	2.4	7.8	9.4	1.1
SD	27.5	1.9	1.2	4.6	0.4	1.4	1.8	0.3
CV, %	10.1	18.1	12.1	14.6	16.7	17.9	19.1	27.3

Table A.5.2 Tocol concentration in fruit and seed fractions (cont'd)

Month/ sample	Tocol concentration mg/100 g oil							
	α – T	α – T3	β - T	P-8	γ - T	γ – T3	δ - T	δ – T3
	Seed fraction							
September								
Sample 1	155.0	n/d[d]	12.1	3.1	67.5	32.8	12.3	n/d
Sample 2	154.9	n/d	12.7	3.2	67.7	n/d	8.3	n/d
Sample 3	163.4	n/d	12.6	3.2	68.9	n/d	8.0	n/d
Mean	157.8	n/d	12.5	3.1	68.0	10.9	9.5	n/d
SD	4.8	n/d	0.3	0.1	0.8	18.9	2.4	n/d
CV, %	3.0	n/d	2.4	3.2	1.2		25.3	n/d
November								
Sample 1	155.1	n/d	13.0	3.3	68.8	n/d	8.9	n/d
Sample 2	106.7	n/d	8.5	2.6	45.9	n/d	5.5	n/d
Sample 3	155.9	n/d	11.1	3.1	56.9	n/d	6.6	n/d
Mean	139.2	n/d	10.9	3.0	57.2	n/d	7.0	n/d
SD	28.2	n/d	2.2	0.3	11.4	n/d	1.7	n/d
CV, %	20.2	n/d	20.2	10.0	19.9	n/d	24.3	n/d
January								
Sample 1	154.9	n/d	14.2	2.8	64.1	n/d	7.3	n/d
Sample 2	152.4	n/d	12.0	3.0	63.5	n/d	7.3	n/d
Sample 3	160.0	n/d	17.0	2.7	61.3	n/d	6.9	n/d
Mean	155.8	n/d	14.4	2.8	63.0	n/d	7.2	n/d
SD	3.9	n/d	2.5	0.1	1.5	n/d	0.2	n/d
CV, %	2.5	n/d	17.4	3.6	2.4	n/d	2.8	n/d

[a] α-T = α-tocopherol. β-T = β-tocopherol. γ-T = γ-tocopherol. δ-T = δ-tocopherol. α-T3 = α-tocotrienol. β-T3 = β-tocotrienol. γ-T3 = γ-tocotrienol. δ-T3 = δ-tocotrienol. P-8 = Plastochromanol-8.
[b] *SD* = standard deviation.
[c] *CV* = coefficient of variation.
[d] n/d = not detectable.

Refer to "Sample calculations for Table A.1.1" in Appendix A.1 for Mean, *SD*, and *CV*.

Appendix A.6 Phytosterol analysis for sea buckthorn berries

Table A.6.1 Phytosterol concentration analysis of fruit and seed fractions
(Coordinates with Tables 3.7 and A.6.2)

	Phytosterol				
	5α-cholestane	Cholesterol	Campesterol	Stigmasterol	β-sitosterol
	Standard				
Retention time, min	11.96	16.10	18.10	18.74	19.88
	Fruit fraction (November, sample 7)				
Retention time, min	11.96	n/d[a]	18.10	18.79	19.89
A_{sterol}[b]	48046	n/d	2504	729	124678
$X1_{sterol}$[c] μg	206	n/d	10.74	3.13	534.56
m_{oil}[d] g	0.0835	0.0835	0.0835	0.0835	0.0835
$X2_{sterol}$[e] mg/100 g oil	n/a[f]	n/d	12.858	3.743	640.197
	Seed fraction (November, sample 1)				
Retention time, min	11.96	n/d	18.10	n/d	19.89
A_{sterol}	48662	n/d	2876	n/d	116297
$X1_{sterol}$ μg	206	n/d	12.17	n/d	492.32
m_{oil} g	0.0838	0.0838	0.0838	0.0838	0.0838
$X2_{sterol}$ mg/100 g oil	n/a	n/d	14.529	n/d	587.492

[a] n/d – not detected.
[b] A_{sterol} – area of individual phytosterol on chromatogram.
[c] $X1_{sterol}$ – amount of individual sterol.
[d] m_{oil} – mass of oil.
[e] $X2_{sterol}$ – mass of individual phytosterol per 100 g oil sample.
[f] n/a – not applicable.

Sample calculations for Table A.6.1: [β-sitosterol concentration (fruit fraction)]

1) Identify the retention time for major sterols using Figure A.6.1.

2) Identify the major sterols and associated chromatogram area A_{sterol} in Figures A.6.2 (fruit fraction) and A.6.3 (seed fraction).

3) Determine the amount of individual phytosterols in solution using A.6a:

$$X1_{sterol} = \frac{A_{sterol}}{A_{IS}} X1_{IS} \qquad \text{(A.6a)}$$

where $X1_{sterol}$ = amount of individual phytosterol; A_{sterol} = area of individual phytosterol on chromatogram; A_{IS} = area of internal standard, 5α-cholestane, and

$X1_{IS}$ = amount of internal standard 5α-cholestane = $\frac{2.06\mu g}{\mu L} \times 100\mu L = 206.0\mu g$.

$$X1_{\beta-sitosterol} = \frac{124678}{48046} \times 206\mu g$$

4) Determine the amount of individual phytosterols in oil using Eqn. A.6b:

$$X2_{sterol} = \frac{X1_{sterol}}{m_{oil}} \frac{100}{100} \qquad \text{(A.6b)}$$

where $X2_{sterol}$ = mass concentration of individual phytosterols per 100 g oil sample; $X1_{sterol}$ = amount of individual phytosterol, and m_{oil} = mass of oil.

$$X2_{b-sitosterol} = \frac{534.6\mu g}{0.0835g} \times \frac{mg}{10^3 \mu g} \times \frac{100}{100}$$

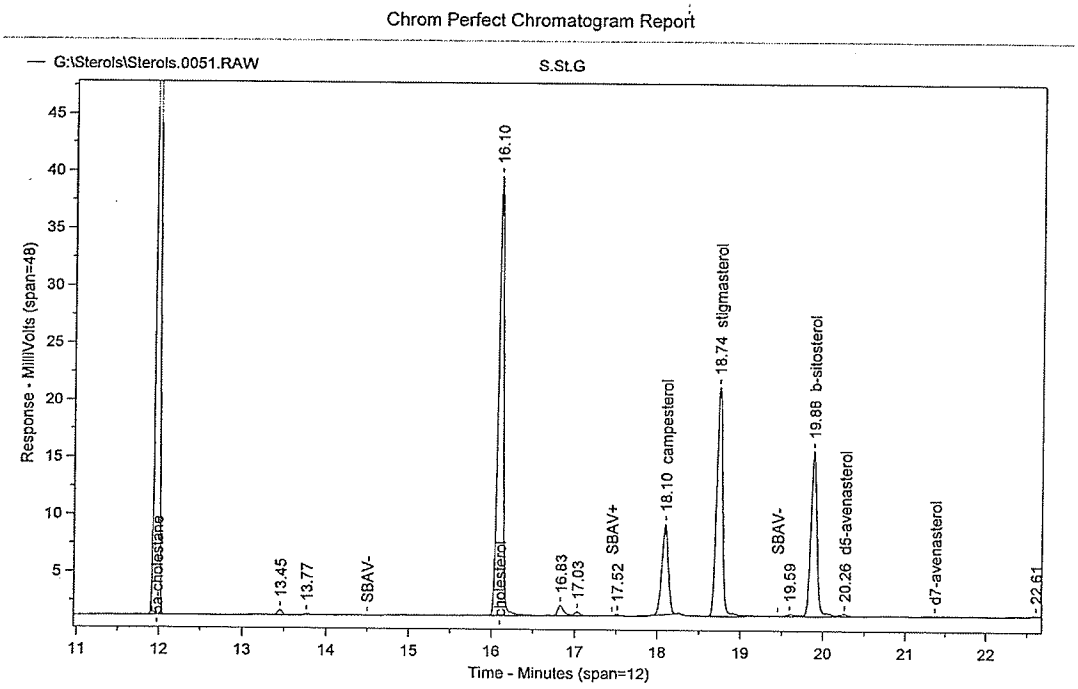
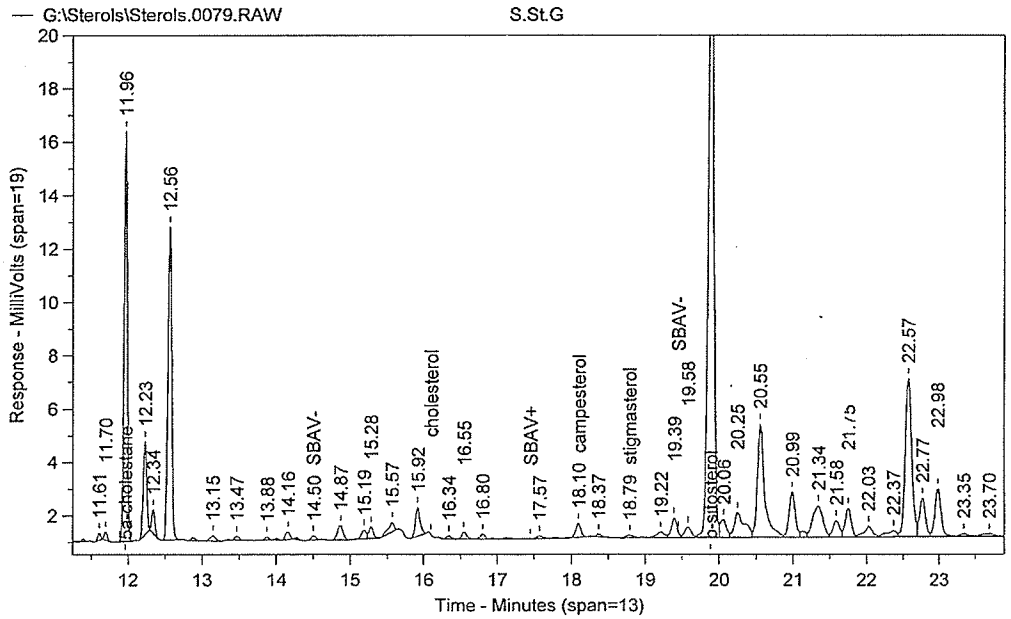


Figure A.6.1 Phytosterol chromatogram for standards.

Chrom Perfect Chromatogram Report



Instrument = Shimadzu
Heading 1 = 1uL injected; 1:40split
Heading 2 = Apr27,2005

Sample Identification: 11 P3-1
Wt. (mg) = _____
mg/mL = _____

Today's Date = 6/3/2005 Today's Time = 4:00:54 PM
Raw File Name = G:\Sterols\Sterols.0079.RAW
Sample Name = S.St.G

Method File Name = G:\Sterols\Sterols.MET
Method Description = Sterol Analysis - column: DB-5; 30m;
Calibration File Name = G:\Sterols\Sterols.CAL
Run Time = 38.5

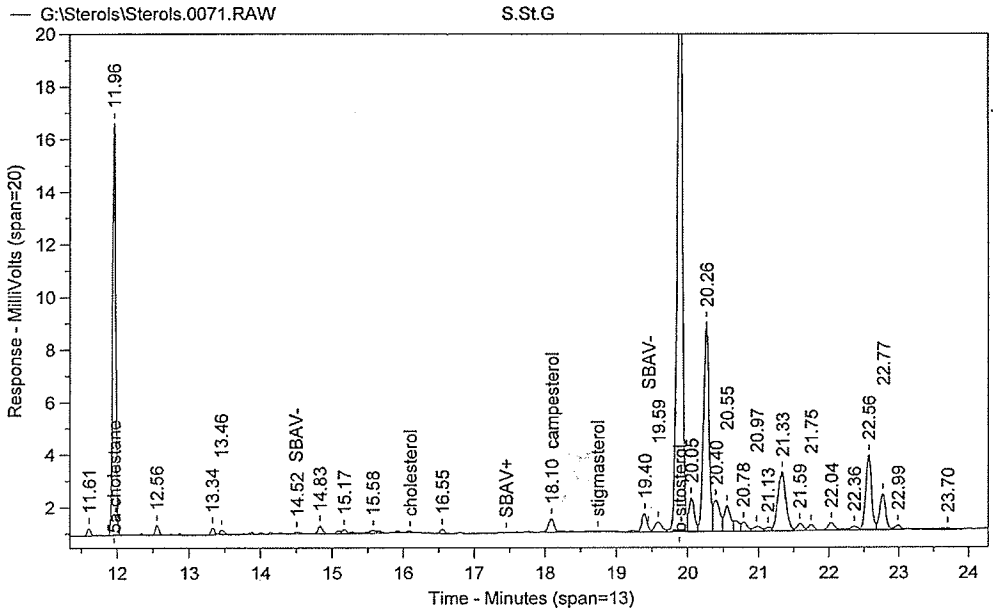
Peak #	Ret. Time	Peak Name	Amount	Amt %	Area	Area %
2	10.34		0.000	0.000	786	0.18
5	11.16		0.000	0.000	3570	0.82
7	11.61		0.000	0.000	754	0.17
8	11.70		0.000	0.000	865	0.20
9	11.96	5a-cholestane	1.000	0.036	48046	10.99
10	12.23		0.000	0.000	11584	2.65
11	12.34		0.000	0.000	2723	0.62
12	12.56		0.000	0.000	42223	9.66
14	13.15		0.000	0.000	971	0.22
16	13.47		0.000	0.000	699	0.16
17	13.88		0.000	0.000	583	0.13
19	14.16		0.000	0.000	1286	0.29
21	14.50		0.000	0.000	731	0.17
22	14.87		0.000	0.000	2957	0.68
23	15.19		0.000	0.000	1564	0.36
24	15.28		0.000	0.000	1871	0.43

Figure A.6.2 Phytosterol chromatogram for the fruit fraction - November, sample 7.

25	15.57		0.000	0.000	2179	0.50
26	15.92		0.000	0.000	4755	1.09
27	16.34		0.000	0.000	539	0.12
28	16.55		0.000	0.000	1298	0.30
29	16.80		0.000	0.000	937	0.21
31	17.57		0.000	0.000	632	0.14
34	18.10	campesterol	48.877	1.757	2504	0.57
35	18.37		0.000	0.000	567	0.13
36	18.79	stigmasterol	8.021	0.288	729	0.17
37	19.22		0.000	0.000	1640	0.38
38	19.39		0.000	0.000	3922	0.90
39	19.58		0.000	0.000	2448	0.56
40	19.89	b-sitosterol	2342.043	84.170	124678	28.51
41	20.06		0.000	0.000	3854	0.88
42	20.25	d5-avenasterol	169.056	6.076	9000	2.06
43	20.55		0.000	0.000	28510	6.52
44	20.99		0.000	0.000	9874	2.26
45	21.34	d7-avenasterol	214.515	7.709	11420	2.61
46	21.58		0.000	0.000	3908	0.89
47	21.75		0.000	0.000	6190	1.42
48	22.03		0.000	0.000	3117	0.71
49	22.37		0.000	0.000	2347	0.54
50	22.57		0.000	0.000	38365	8.77
51	22.77		0.000	0.000	8360	1.91
52	22.98		0.000	0.000	10897	2.49
53	23.35		0.000	0.000	899	0.21
54	23.70		0.000	0.000	1418	0.32
55	24.01		0.000	0.000	734	0.17
56	24.31		0.000	0.000	1162	0.27
57	24.56		0.000	0.000	536	0.12
58	24.75		0.000	0.000	1211	0.28
59	25.07		0.000	0.000	5062	1.16
60	25.23		0.000	0.000	2476	0.57
61	25.58		0.000	0.000	588	0.13
63	26.15		0.000	0.000	804	0.18
64	26.38		0.000	0.000	5046	1.15
65	26.72		0.000	0.000	1579	0.36
66	27.13		0.000	0.000	2265	0.52
67	27.43		0.000	0.000	574	0.13
68	27.57		0.000	0.000	1223	0.28
69	28.19		0.000	0.000	2945	0.67
71	29.97		0.000	0.000	708	0.16
72	30.41		0.000	0.000	941	0.22
73	30.63		0.000	0.000	872	0.20
74	31.59		0.000	0.000	606	0.14
75	32.31		0.000	0.000	1207	0.28
76	32.94		0.000	0.000	777	0.18
77	33.97		0.000	0.000	857	0.20
79	34.77		0.000	0.000	661	0.15
80	35.00		0.000	0.000	540	0.12
81	35.93		0.000	0.000	946	0.22
82	37.25		0.000	0.000	29966	6.85
83	38.35		0.000	0.000	14851	3.40

Figure A.6.2 Phytosterol chromatogram for the fruit fraction – November, sample 7. (cont'd)

Chrom Perfect Chromatogram Report



Instrument = Shimadzu
Heading 1 = 1uL injected; 1:40split
Heading 2 = Apr27,2005

Sample Identification: 11 Sd1
Wt. (mg) = _____
mg/mL = _____

Today's Date = 6/3/2005 Today's Time = 3:59:19 PM
Raw File Name = G:\Sterols\Sterols.0071.RAW
Sample Name = S.St.G

Method File Name = G:\Sterols\Sterols.MET
Method Description = Sterol Analysis - column: DB-5; 30m;
Calibration File Name = G:\Sterols\Sterols.CAL
Run Time = 38.5

Peak #	Ret. Time	Peak Name	Amount	Amt %	Area	Area %
4	11.16		0.000	0.000	684	0.25
5	11.61		0.000	0.000	995	0.37
6	11.96	5a-cholestane	1.000	0.030	48662	17.89
9	12.56		0.000	0.000	1339	0.49
12	13.34		0.000	0.000	885	0.33
13	13.46		0.000	0.000	656	0.24
18	14.52		0.000	0.000	508	0.19
19	14.83		0.000	0.000	1419	0.52
21	15.17		0.000	0.000	728	0.27
23	15.58		0.000	0.000	784	0.29
26	16.55		0.000	0.000	715	0.26
30	18.10	campesterol	55.424	1.669	2876	1.06
33	19.40		0.000	0.000	3663	1.35
34	19.59		0.000	0.000	2597	0.95
35	19.89	b-sitosterol	2156.944	64.970	116297	42.75
36	20.05		0.000	0.000	6838	2.51

Figure A.6.3 Phytosterol chromatogram for the seed fraction – November, sample 1.

Chrom Perfect Chromatogram Report

37	20.26	d5-avenasterol	779.960	23.493	42054	15.46
38	20.40		0.000	0.000	7681	2.82
39	20.55		0.000	0.000	6040	2.22
40	20.78		0.000	0.000	1918	0.71
41	20.97		0.000	0.000	1355	0.50
42	21.13		0.000	0.000	730	0.27
43	21.33	d7-avenasterol	327.603	9.868	17664	6.49
44	21.59		0.000	0.000	1656	0.61
45	21.75		0.000	0.000	1389	0.51
46	22.04		0.000	0.000	2160	0.79
47	22.36		0.000	0.000	1003	0.37
48	22.56		0.000	0.000	16513	6.07
49	22.77		0.000	0.000	7986	2.94
50	22.99		0.000	0.000	987	0.36
51	23.70		0.000	0.000	1366	0.50
53	24.55		0.000	0.000	675	0.25
54	25.06		0.000	0.000	596	0.22
55	25.22		0.000	0.000	760	0.28
56	26.12		0.000	0.000	1524	0.56
58	27.13		0.000	0.000	604	0.22
59	28.21		0.000	0.000	674	0.25
63	33.17		0.000	0.000	731	0.27
64	35.75		0.000	0.000	1062	0.39
65	37.06		0.000	0.000	6399	2.35
66	37.33		0.000	0.000	7509	2.76

Total Area = 272018.6 Total Amount = 3319.931

Checked by _____ Date _____

Printed on 6/3/2005 3:59:21 PM

Figure A.6.3 Phytosterol chromatogram for the seed fraction - November, sample 1. (cont'd)

Table A.6.2 Major phytosterol concentrations in fruit and seed fractions
(Coordinates with Tables 3.7 and A.6.1)

Month /sample	Phytosterol concentration mg/100 g oil			
	Cholesterol	Campesterol	Stigmasterol	β-sitosterol
		Fruit fraction		
September				
Sample 1	n/d[a]	23.71	13.25	1113.68
Sample 2	n/d	23.67	7.82	1078.00
Sample 3	13.76	22.06	4.74	1046.76
Sample 4	n/d	19.80	8.31	955.64
Sample 5	10.12	19.16	4.03	1014.40
Sample 6	9.48	17.22	4.88	825.32
Sample 7	n/d	22.03	14.05	1082.96
Sample 8	n/d	13.29	4.25	654.19
Sample 9	n/d	12.26	5.70	584.37
Mean	3.71	19.24	7.45	928.37
SD[b]	5.68	4.24	3.83	195.87
CV, %[c]		22.0	51.4	21.10
November				
Sample 1	9.26	13.83	n/d	693.91
Sample 2	12.53	17.90	n/d	924.72
Sample 3	3.05	13.98	3.61	677.14
Sample 4	n/d	14.44	6.90	704.00
Sample 5	3.45	13.37	5.23	646.41
Sample 6	5.14	15.88	8.58	770.95
Sample 7	n/d	12.86	3.74	640.20
Sample 8	8.32	11.91	6.27	595.77
Sample 9	7.72	11.86	4.49	579.26
Mean	5.50	14.00	4.31	692.48
SD	3.71	1.92	2.91	104.58
CV, %		13.7		15.10
January				
Sample 1	7.22	14.09	2.35	634.79
Sample 2	n/d	15.58	4.24	663.29
Sample 3	n/d	19.53	n/d	839.85
Sample 4	n/d	15.61	7.74	716.68
Sample 5	n/d	15.52	12.11	683.09
Sample 6	n/d	20.84	27.75	952.11
Sample 7	n/d	14.10	9.17	630.34
Sample 8	6.54	15.14	n/d	674.13
Sample 9	n/d	15.60	5.20	709.74
Mean	1.53	16.22	7.62	722.67
SD	3.04	2.35	8.58	106.23
CV, %		14.5		14.70

Table A.6.2 Major phytosterol concentrations in fruit and seed fractions (cont'd)

Month /sample	Phytosterol concentration mg/100 g oil			
	Cholesterol	Campesterol	Stigmasterol	β-sitosterol
		Seed fraction		
September				
Sample 1	3.12	12.57	n/d	523.36
Sample 2	2.08	12.32	n/d	527.18
Sample 3	n/d	13.28	n/d	511.57
Mean	1.73	12.72	n/d	520.70
SD	1.59	0.50	n/d	8.13
CV, %		3.93	n/d	1.56
November				
Sample 1	n/d	14.53	n/d	587.49
Sample 2	n/d	10.19	n/d	488.30
Sample 3	n/d	12.26	n/d	508.61
Mean	n/d	12.33	n/d	528.13
SD	n/d	2.17	n/d	52.39
CV, %	n/d	17.6	n/d	9.92
January				
Sample 1	4.45	14.58	n/d	596.21
Sample 2	3.04	13.28	2.43	558.87
Sample 3	n/d	13.56	n/d	544.48
Mean	2.50	13.81	0.81	566.52
SD	2.27	0.68	1.41	26.70
CV, %		4.92		4.71

[a] n/d = not detected.
[b] *SD* = standard deviation.
[c] *CV* = coefficient of variation.

Refer to "Sample calculations for Table A.1.1" in Appendix A.1 for Mean, *SD*, and *CV*.

B - APPENDICES FOR CHAPTER 4

Appendix B.1 Moisture analysis for sea buckthorn berry pulp and peel

Table B.1.1 Water activity, a_w, analysis for fresh and dried pulp and peel
(Coordinates with Tables 4.5 and B.1.3)

Sensor		Water activity	
		Fresh pulp and peel (sample 2)	Dried pulp and peel [50°C/30.6% RH (sample 1)]
1	$X1$[a]	0.919	0.410
	$Y1$[b]	0.922	0.411
2	$X2$	0.923	0.413
	$Y2$	0.924	0.412
3	$X3$	0.926	0.421
	$Y3$	0.928	0.420
Mean		0.925	0.415

[a] $X1$ = sensor water activity (a_w) reading.
[b] $Y1$ = adjusted a_w based on calibration.

Sample calculations for Table B.1.1: [Water activity, a_w, of dried pulp and peel]

1) Corrected a_w for sensor 1 using Eqn. B.1:

$$Y1 = 1.0029X1 + 0.0003 \qquad \text{(B.1)}$$

where $Y1$ = corrected a_w based on equipment calibration for sensor 1 (Table B.1.2),

and $X1$ = sensor a_w reading.

$$Y1 = 1.0029 \times 0.410 + 0.0003$$

Table B.1.2 Calibration and linear regression for water activity centre

Calibration salt[a]	Y_{cal}[b]	Sensor a_w		
		$X1$[c]	$X2$	$X3$
Sal – 11	0.113	0.115	0.114	0.114
Sal – 33	0.328	0.323	0.330	0.330
Sal – 53	0.529	0.527	0.529	0.536
Sal – 75	0.753	0.752	0.753	0.744
Sal – 90	0.901	0.898	0.899	0.903
a[d]		1.0029	1.0039	1.0049
b[e]		0.0003	-0.0022	-0.0032
r^2[f]		0.99	0.99	0.99

[a] Calibration salt: Sal – 11 = LiCl. Sal – 33 = $MgCl_2$. Sal – 53 = $Mg(NO_3)_2$. Sal – 75 = NaCl. Sal – 90 = $BaCl_2$.

[b] Y_{cal} = known water activity, a_w of calibration salt at 25°C.

[c] $X1$ = water activity, a_w reading for sensor 1 at 25°C.

[d] a = slope, based on linear relationship ($Y_{cal} = a\,X + b$).

[e] b = Y – intercept, based on linear relationship ($Y_{cal} = a\,X + b$).

[f] r^2 = coefficient of determination.

Linear regression analysis performed using JMP IN Statistical Discovery Software (SAS Institute, Inc., Cary, NC, 2001).

Table B.1.3 Drying parameters and characteristics of pulp and peel
(Coordinates with Tables 4.5 and B.1.1)

Temperature °C	Sample	RH[a] %	Time h	M[b] % (w/w) wb	a_w[c]
			Dried pulp and peel		
	1	26.3	13.0	7.13	0.415
50	2	32.3	10.5	7.69	0.467
	3	33.1	10.6	9.19	0.506
	Mean	30.6	11.4	8.00	0.463
	SD[d]	3.72	1.4	1.07	0.046
	CV, %[e]	12.2	12.3	13.4	9.9
	1	57.8	24.0	7.16	0.437
50	2	58.4	24.2	7.49	0.438
	3	59.9	24.2	5.76	0.409
	Mean	58.7	24.1	6.80	0.428
	SD	1.1	0.1	0.92	0.016
	CV, %	1.9	0.4	13.5	3.7
	1	23.4	7.4	6.43	0.396
60	2	25.4	5.3	6.98	0.383
	3[g]	27.7	6.3	6.93	0.413
	Mean	24.4	6.4	6.71	0.390
	SD	1.4	1.5	0.39	0.009
	CV, %	5.7	23.4	5.8	2.3
	1	57.1	19.1	4.78	0.383
60	2	56.9	19.1	7.06	0.420
	3	57.0	18.9	7.40	0.428
	Mean	57.0	19.0	6.41	0.410
	SD	0.1	0.1	1.42	0.024
	CV, %	.2	.5	22.2	5.9
	1	19.9	4.1	4.42	0.355
70	2	20.7	3.8	6.69	0.429
	3	21.8	4.1	6.87	0.437
	Mean	20.8	4.0	5.99	0.407
	SD	1.0	0.2	1.37	0.045
	CV, %	4.8	5.0	22.9	11.1
	1	57.0	15.9	7.49	0.435
70	2	57.0	8.3	8.34	0.489
	3	57.1	8.3	7.85	0.490
	Mean	57.0	10.8	7.89	0.471
	SD	0.10	4.4	0.43	0.031
	CV, %	0.1	40.7	5.4	6.6

Table B.1.3 Drying parameters and characteristics of pulp and peel (cont'd)

Temperature °C	Sample	*RH* %	Time h	*M* % (w/w) wb	a_w
		Fresh pulp and peel			
n/a[f]	1[g]	n/a	n/a	43.62	0.925
n/a	2	n/a	n/a	40.68	0.925
n/a	3	n/a	n/a	45.92	0.926
n/a	Mean	n/a	n/a	43.30	0.925
n/a	*SD*	n/a	n/a	3.71	0.001
n/a	*CV*	n/a	n/a	8.57	0.11

[a] *RH* = relative humidity.
[b] *M* = moisture content, % mass/mass (w/w) on a wet basis (wb).
[c] a_w = water activity.
[d] *SD* = standard deviation.
[e] *CV* = coefficient of variation.
[f] n/a = not applicable.
[g] Sample data not used for analysis due to outlier.

Refer to "Sample calculations for Table A.1.1" in Appendix A.1 for Mean, *SD*, and *CV*.

Appendix B.2 Colour analysis for sea buckthorn berry pulp and peel

Table B.2.1 Colour analysis data for fresh and dried pulp and peel (Coordinates with Tables 4.2 and B.2.2)

Temp/ RH[a]	Sample	Colour factors[b]											
		L^*_{SSp}			a^*_{SSp}			b^*_{SSp}			L^*_{Sp}	a^*_{Sp}	b^*_{Sp}
		SS1[c]	SS2	SS3	SS1	SS2	SS3	SS1	SS2	SS3			
							Dried						
50°C/ 30.6%	1	48.91	49.45	49.06	12.59	12.37	12.23	24.18	24.45	23.88	49.14	12.40	24.17
	2	48.02	48.24	49.15	12.83	12.48	12.48	24.40	24.32	24.90	48.47	12.60	24.54
	3	45.85	46.36	46.41	12.50	12.21	12.32	20.76	20.93	20.72	46.21	12.34	20.80
	Mean										47.94	12.45	23.17
	SD[d]										1.54	0.14	2.06
	CV,%[e]										3.21	1.12	8.89
50°C/ 58.7%	1	45.41	45.51	46.07	10.53	10.48	10.30	20.12	20.33	20.48	45.66	10.44	20.31
	2	45.49	46.35	46.43	10.92	10.64	10.54	20.91	21.30	21.27	46.09	10.70	21.16
	3	45.64	45.87	45.96	10.55	10.43	10.36	19.50	19.80	19.75	45.82	10.45	19.68
	Mean										45.86	10.53	20.38
	SD										0.22	0.15	0.74
	CV,%										0.48	1.42	3.63
60°C/ 24.4%	1	48.63	49.13	49.12	12.92	12.66	12.54	26.20	26.30	25.83	48.96	12.71	26.11
	2	49.04	49.67	49.61	12.99	12.73	12.54	24.97	25.34	25.02	49.44	12.75	25.11
	3[f]	48.04	48.32	48.76	12.84	12.82	12.60	23.30	23.62	23.64	48.37	12.75	23.52
	Mean										49.20	12.73	25.61
	SD										0.34	0.03	0.71
	CV,%										0.69	0.24	2.77

Table B.2.1 Colour analysis data for fresh and dried pulp and peel (cont'd)

Temp /*RH*	Sample	Colour factors											
		L^*_{SSp}			a^*_{SSp}			b^*_{SSp}			L^*_{Sp}	a^*_{Sp}	b^*_{Sp}
		SS1	SS2	SS3	SS1	SS2	SS3	SS1	SS2	SS3			
							Dried						
60°C/ 57.0%	1	40.14	40.92	41.13	9.40	9.47	9.23	14.10	14.63	14.49	40.73	9.37	14.41
	2	42.70	42.75	43.42	9.38	9.03	9.12	15.36	15.28	15.93	42.96	9.18	15.52
	3	41.51	41.93	42.22	9.61	9.33	9.34	15.44	15.52	15.69	41.89	9.43	15.55
	Mean										41.86	9.33	15.16
	SD										1.12	0.13	0.65
	CV,%										2.68	1.39	4.29
70°C/ 20.8%	1	48.63	49.06	48.89	12.11	11.90	11.92	21.08	21.28	20.49	48.86	11.98	20.95
	2	46.08	46.37	47.01	12.33	12.26	12.26	22.33	22.42	22.82	46.49	12.28	22.52
	3	46.09	46.55	46.49	12.65	12.52	12.45	22.11	22.40	22.14	46.38	12.54	22.22
	Mean										47.24	12.27	21.90
	SD										1.40	0.28	0.83
	CV,%										2.96	2.28	3.79
70°C/ 57.0%	1	41.30	41.56	41.51	9.54	9.56	9.39	13.47	13.63	13.36	41.46	9.50	13.49
	2	41.80	41.70	41.92	9.66	9.53	9.36	13.12	13.00	12.96	41.81	9.52	13.03
	3	40.83	41.02	41.63	9.73	9.50	9.40	14.08	14.01	14.09	41.16	9.54	14.06
	Mean										41.48	9.52	13.53
	SD										0.33	0.02	0.52
	CV,%										0.80	0.21	3.84

Table B.2.1 Colour analysis data for berry pulp and peel (cont'd)

Temp /*RH*	Sample	Colour factors											
		L^*_{SSp}			a^*_{SSp}			b^*_{SSp}			L^*_{Sp}	a^*_{Sp}	b^*_{Sp}
		SS1	SS2	SS3	SS1	SS2	SS3	SS1	SS2	SS3			
n/a	1[f]	49.03	50.40	48.85	14.93	14.13	14.90	25.81	25.71	25.87	49.43	14.65	25.80
	2	51.64	52.04	52.36	13.48	13.24	12.48	24.70	24.62	25.00	52.01	13.07	24.77
	3	49.83	49.93	49.28	12.90	12.82	13.47	24.49	24.61	23.52	49.68	13.06	24.21
	Mean										50.85	13.07	24.49
	SD										1.65	0.01	0.40
	CV,%										3.24	0.05	1.63

[a] Temp/*RH* = drying conditions (temperature and relative humidity).
[b] L^*_{Sp}/L^*_{SSp} = lightness; a^*_{Sp}/a^*_{SSp} = hue, (+) red or (-) green; b^*_{Sp}/b^*_{SSp} = hue, (+) yellow or (-) blue for samples and subsamples *p*.
[c] SS1 = subsample 1, 1st evaluation of sample 1.
[d] *SD* = standard deviation.
[e] *CV* = coefficient of variation.
[f] Sample data not used for analysis due to outlier.

Table B.2.2 Colour change, ΔE, for dried pulp and peel
(Coordinates with Fig. 4.2 and Table B.2.1)

Temp/*RH*[a]	Sample	ΔE_{Sp}[b]
50°C/30.6%	1	1.86
	2	2.42
	3	5.97
	Mean	3.42
	SD[c]	2.23
	CV, %[d]	65.20
50°C/58.7%	1	7.16
	2	6.27
	3	7.43
	Mean	6.95
	SD	0.61
	CV, %	8.78
60°C/24.4%	1	2.51
	2	1.57
	3[e]	2.67
	Mean	2.04
	SD	0.66
	CV,%	32.4
60°C/57.0%	1	14.75
	2	12.56
	3	13.16
	Mean	13.49
	SD	1.13
	CV, %	8.38
70°C/20.8%	1	4.20
	2	4.84
	3	5.04
	Mean	4.69
	SD	0.44
	CV,%	9.38
70°C/57.0%	1	14.89
	2	15.02
	3	14.66
	Mean	14.86
	SD	0.18
	CV,%	1.2

[a] Temp/*RH* = drying conditions (temperature and relative humidity).
[b] ΔE_{Sp} = colour change between dried and fresh pulp and peel.
[c] *SD* = standard deviation.
[d] *CV* = coefficient of variation.
[e] Sample data not used for analysis due to outlier.

Sample calculations for Tables B.2.1-2: [Colour change, ΔE, for 50°C - 30.6% RH, Sample 1]

1) Colour change using Eqn. B.2a

$$\Delta E = \sqrt{(L^*_0 - L^*)^2 + (a^*_0 - a^*)^2 + (b^*_0 - b^*)^2} \qquad \text{(B.2a)}$$

where = mean L^* for fresh sample p, where $p = 1$ to 3; a^*_0 = mean a^* for fresh sample p, where $p = 1$ to 3, and b^*_0 = mean b^* for fresh sample p, where $p = 1$ to 3.

$$\Delta E_{S1} = \sqrt{(50.85 - 49.14)^2 + (13.07 - 12.40)^2 + (24.49 - 24.17)^2}$$

Refer to "Sample calculations for Table A.2.1" in Appendix A.2 for colour factors L^*, a^*, and b^* and "Sample calculations for Table A.1.1" in Appendix A.1 for Mean, *SD*, and *CV*.

Appendix B.3 Carotenoid analysis for sea buckthorn berry pulp and peel

Table B.3.1 Total carotenoid analysis for berry pulp and peel (Coordinates with Fig. 4.1)

Temp /RH[a]	Sample	Y_{carot}[b] mg/mL	X_{carot}[c] x10^{-3} mg/mL	D_2[d]	$X1_{carot}$[e] x10^{-3} mg/mL	D_1[f]	$X2_{carot}$[g] mg/mL	V_{hex}[h] mL	m_{carot}[i] mg	m_{oil}[j] g	X_{final}[k] mg/100 g
					Dried pulp and peel						
50°C /30.6%	1	0.581	3.459	4	13.834	50	0.6917	10	6.917	0.7990	865.8
	2	0.567	3.373	4	13.494	50	0.6747	10	6.747	0.7843	860.1
	3	0.702	4.194	4	16.776	50	0.8388	10	8.388	0.8755	958.1
	Mean										894.7
	SD[l]										55.0
	CV,%[m]										6.15
50°C /58.7%	1	0.602	3.586	4	14.345	50	0.7172	10	7.172	0.8644	829.7
	2	0.616	3.671	4	14.685	50	0.7343	10	7.343	0.7720	951.0
	3	0.630	3.756	4	15.026	50	0.7513	10	7.513	0.8364	898.1
	Mean										892.9
	SD										60.8
	CV,%										6.81
60°C /25.5%	1	0.669	3.993	4	15.974	50	0.7987	12	9.584	0.9990	958.4
	2	0.576	3.428	4	13.713	50	0.6856	10	6.856	0.8854	774.3
	3[o]	0.562	3.343	4	13.372	50	0.6686	11	7.355	0.8475	867.8
	Mean										866.4
	SD										130.2
	CV,%										15.0

Table B.3.1 Total carotenoid analysis for berry pulp and peel (cont'd)

Temp /*RH*	Sample	Y_{carot} mg/mL	X_{carot} x10^{-3} mg/mL	D_2	$X1_{carot}$ x10^{-3} mg/mL	D_1	$X2_{carot}$ mg/mL	V_{hex} mL	m_{carot} mg	m_{oil} g	X_{final} mg/100 g
					Dried pulp and peel						
60°C /57.0%	1	0.603	3.592	4	14.369	50	0.7185	10	7.185	0.8196	876.5
	2	0.602	3.586	4	14.345	50	0.7172	10	7.172	0.8754	819.3
	3	0.596	3.550	4	14.199	50	0.7099	10	7.099	0.8528	832.6
	Mean										842.8
	SD										29.9
	CV,%										3.55
70°C /20.8%	1	0.513	3.045	4	12.181	50	0.6090	10	6.090	0.7502	811.8
	2	0.665	3.969	4	15.876	50	0.7938	10	7.938	0.8842	897.8
	3	0.663	3.957	4	15.828	50	0.7914	10	7.914	0.8874	891.8
	Mean										867.1
	SD										48.0
	CV,%										5.54
70°C /57.0%	1	0.541	3.215	4	12.862	50	0.6431	10	6.431	0.7521	854.9
	2	0.676	4.036	4	16.144	50	0.8072	10	8.072	0.7720	1045.6
	3	0.598	3.562	4	14.248	50	0.7124	10	7.124	0.7707	924.4
	Mean										941.6
	SD										96.5
	CV,%										10.25

Table B.3.1 Total carotenoid analysis for berry pulp and peel (cont'd)

Temp /*RH*	Sample	Y_{carot} mg/mL	X_{carot} x10^{-3} mg/mL	D_2	$X1_{carot}$ x10^{-3} mg/mL	D_1	$X2_{carot}$ mg/mL	V_{hex} mL	m_{carot} mg	m_{oil} g	X_{final} mg/100 g
					Fresh pulp and peel						
n/a[n]	1[o]	0.215	1.234	4	4.936	50	0.2468	11	2.715	0.3340	812.8
	2	0.412	2.431	4	9.725	50	0.4863	10	4.863	0.4823	1008.1
	3	0.333	1.951	4	7.805	50	0.3902	12	4.683	0.5625	832.4
	Mean										920.3
	SD										124.2
	CV,%										13.50

[a] Temp/*RH* = drying conditions (temperature and relative humidity).
[b] Y_{carot} = absorbance value (measured).
[c] X_{carot} = concentration of total carotenoids in hexane solution.
[d] D_2 = dilution ratio 2 (fruit fraction).
[e] $X1_{carot}$ = concentration of total carotenoids in hexane solution, corrected for dilution 2.
[f] D_1 = dilution ratio 1 (fruit fraction).
[g] $X2_{carot}$ = concentration of total carotenoids in hexane solution, corrected for dilution 1.
[h] V_{hex} = volume of hexane used in original dilution.
[i] m_{carot} = mass of total carotenoids.
[j] m_{oil} = mass of oil.
[k] X_{final} = concentration of total carotenoids per 100 g oil.
[l] *SD* = standard deviation.
[m] *CV* = coefficient of variation.
[n] n/a = not applicable.
[o] Sample data not used for analysis due to outlier.

Refer to "Sample calculations for Table A.3.1" in Appendix A.3 for total carotenoids and "Sample calculations for Table A.1.1" in Appendix A.1 for Mean, *SD*, and *CV*.

Appendix B.4 Fatty acid analysis for sea buckthorn berry pulp and peel

Table B.4.1 Fatty acid analysis for berry pulp and peel (Coordinates with Tables 4.3 and B.4.2)

Fatty acid[a]	Standard	Fresh pulp and peel (sample1)			Dried pulp and peel (50°C - 30.6%RH, sample1)		
	Retention time, min	Retention time, min	A_{FA}[b]	Amount $\%_{FA}$[c]	Retention time, min	A_{FA}	Amount $\%_{FA}$
C6:0	0.90	0.94	2127	0.11	n/d[d]	n/d	n/d
C8:0	1.07	1.13	2140	0.01	1.13	1094	0.06
C10:0	1.46	1.17	4440	0.22	1.17	2086	0.12
C12:0	2.33	2.30	1450	0.07	2.30	961	0.05
C14:0	4.00	3.95	18818	0.94	3.95	13766	0.76
C14:1	4.35	n/d	n/d	n/d	n/d	n/d	n/d
C15:0	5.17	5.10	3079	0.15	5.10	2297	0.13
C16:0	6.53	6.49	522355	26.01	6.48	399504	22.17
C16:1*n*7	6.86	6.81	500426	24.92	6.81	396090	21.98
C17:0	8.04	8.16	35609	1.77	8.16	22585	1.25
C17:1	8.40	8.32	92874	n/a[e]	8.32	78749	n/a
C18:0	9.64	9.57	20235	1.01	9.57	15144	0.84
C18:1*n*9	9.91	9.85	279229	13.90	9.84	222629	12.35
Unknown	n/d	9.96	149845	7.46	9.96	115871	6.43
C18:2*n*6	10.53	10.46	195362	9.73	10.46	155690	8.64
C18:3*n*6	10.84	10.75	676	0.03	10.74	712	0.04
C18:3*n*3	11.33	11.26	114151	5.68	11.25	102769	5.70

Table B.4.1 Fatty acid sample analysis for berry pulp and peel (cont'd)

Fatty acid	Standard	Fresh pulp and peel (sample1)			Dried pulp and peel (50°C - 30.6%RH, sample1)		
	Retention time, min	Retention time, min	A_{FA}	Amount $\%_{FA}$	Retention time, min	A_{FA}	Amount $\%_{FA}$
C20:0	12.96	12.88	10827	0.54	12.88	6968	0.39
C20:1	13.25	13.30	1655	0.08	13.30	1342	0.07
C20:2	13.90	13.82	857	0.04	13.81	619	0.03
C20:3*n*6	14.21	n/d	n/d	n/d	14.33	6027	0.33
C20:4	14.37	14.47	16164	0.80	14.47	8317	0.46
C20:3*n*3	14.73	14.74	3359	0.17	14.74	3897	0.22
C20:5	15.19	14.89	2744	0.14	14.89	3416	0.19
C22:0	16.25	16.17	6243	0.31	16.17	36658	2.03
C22:1	16.56	16.59	12709	0.63	16.61	27029	1.50
C22:2	17.23	17.59	13489	0.67	17.59	77439	4.30
C22:4	17.84	17.83	3771	0.19	17.82	23282	1.29
C22:5*n*3	18.82	18.40	1792	0.09	n/d	n/d	n/d

Table B.4.1 Fatty acid sample analysis for berry pulp and peel (cont'd)

Fatty acid	Standard	Fresh pulp and peel (sample1)			Dried pulp and peel (50°C - 30.6%RH, sample1)		
	Retention time, min	Retention time, min	A_{FA}	Amount $\%_{FA}$	Retention time, min	A_{FA}	Amount $\%_{FA}$
C22:6*n*3	19.00	n/d	n/d	n/d	19.19	115181	6.39
C24:0	19.94	19.85	5205	0.26	19.85	3890	0.22
C24:1	20.42	20.33	2085	0.10	20.34	1700	0.09
Total[f]			2101363			1881107	
Total$_{adjA}$[g]			2008489			1802358	

[a] C6:0 = caproic acid. C8:0 = caprylic acid. C10:0 = capric acid. C12:0 = lauric acid. C14:0 = myristic acid. C14:1 = myristoleic acid. C15:0 = pentadecanoic acid. C16:0 = palmitic acid. C16:1*n*7 = palmitoleic acid. C17:0 = heptadecanoic acid. C17:1 = cis-10-heptadecanoic acid. C18:0 = stearic acid. C18:1n9 = oleic acid. C18:2*n*6 = linoleic acid. C18:3*n*6 = γ-linolenic acid. C18:3*n*3 = α-linolenic acid. C20:0 = arachidic acid. C20:1 = eicosenoic acid isomer. C20:2 = eicosadienoic acid isomer. C20:3*n*6 = eicosatrienoic acid isomer. C20:4 = arachidonic acid. C20:3*n*3 = eicosatrienoic acid isomer. C20:5 = eicosapentaenoic acid. C22:0 = behenic acid. C22:1 = erucic acid. C22:2 = docosadienoic acid isomer. C22:4 = docosatetraenoic acid isomer. C22:5*n*3 = docosapentaenoid acid isomer. C22:6*n*3 = docosahexaenoic acid isomer. C24:0 = lignoceric acid. C24:1 = nervonic acid.

[b] A_{FA} = area of individual fatty acids provided on chromatograms (Figures B.4.2 and B.4.3).

[c] Amount $\%_{FA}$ = percent proportion of individual fatty acid in fatty acid profile.

[d] n/d = not detected.

[e] n/a = not applicable.

[f] Total = total area of fatty acids provided on chromatograms (Figures B.4.2 and B.4.3).

[g] Total$_{adjA}$ = total area of fatty acids provided on chromatograms excluding the standard C17:1.

Refer to "Sample calculations for Table A.4.1" in Appendix A.4 for fatty acid proportions in conjunction with Figs. B.4.1-3.

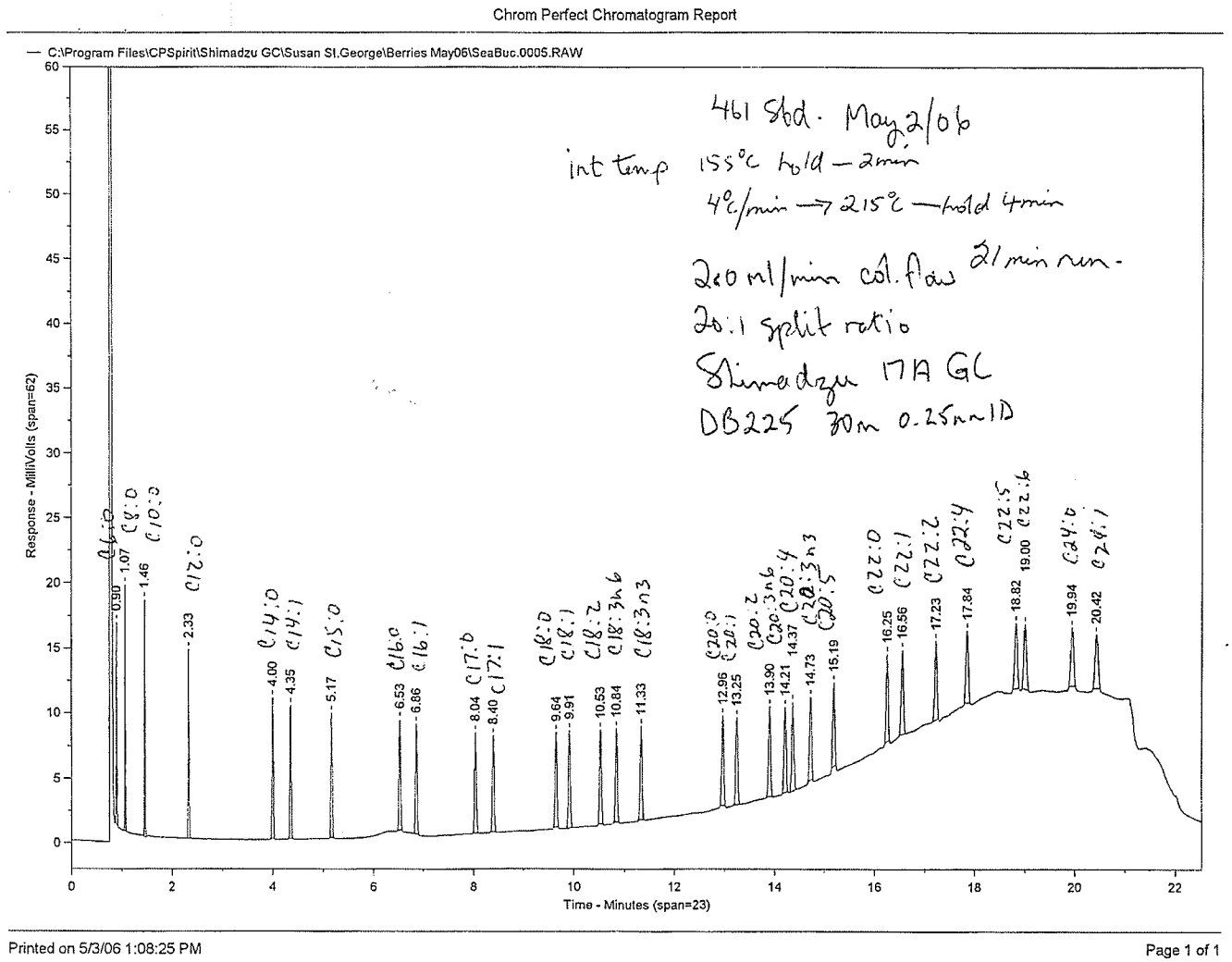


Figure B.4.1 Fatty acid profile chromatogram for Standard 461.

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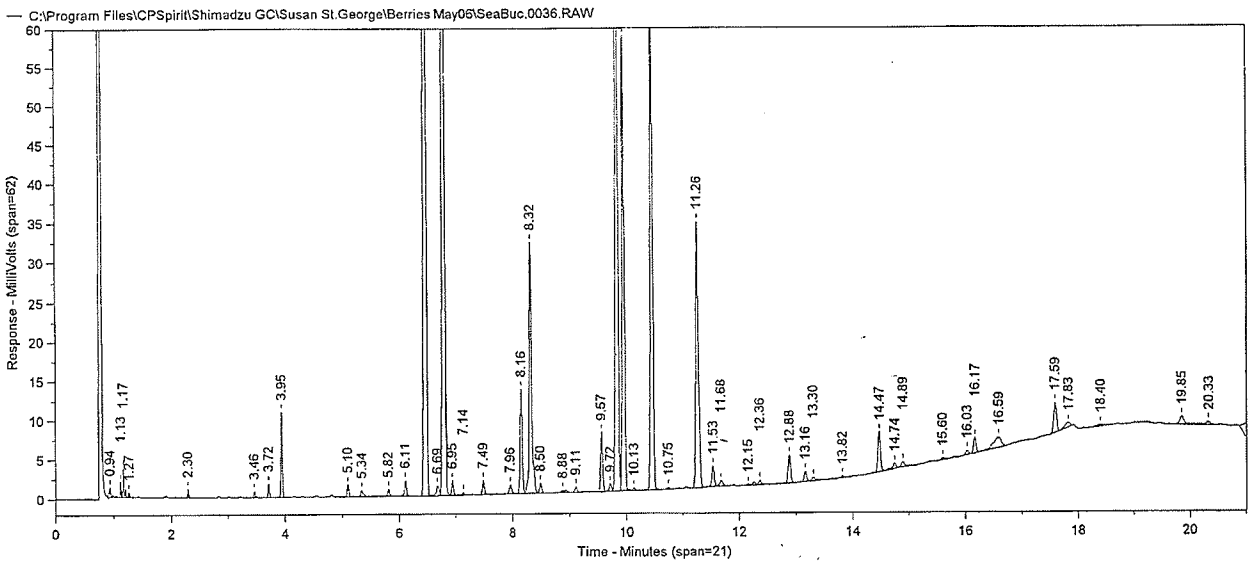

Sample Name = 06Berries

Instrument = Shimadzu
Heading 1 = 1uL inj.; 20:1split DB225 30m x 0.25um
Heading 2 = May2/06 155int. 4C/min.-215C - 4min

Acquisition Port = 4

Raw File Name = C:\Program Files\CPSpirit\Shimadzu GC\Susan St.George\Berries May06\SeaBuc 5/3/2006 1:54:37 PM
Method File Name = C:\Program Files\CPSpirit\Shimadzu GC\Hima.met
Calibration File Name =

Method Version = 5
Calibration Version = 0

Peak #	Ret. Time	Name	Amount	Amt %	Area	Area %	Type	Width
1	0.94		0.00	N/A	2127	0.101	BB	0.03
2	1.13		0.00	N/A	2140	0.102	BV	0.02
3	1.17		0.00	N/A	4440	0.211	VV	0.01
4	1.27		0.00	N/A	786	0.037	VB	0.02
5	2.30		0.00	N/A	1450	0.069	BB	0.02
6	3.46		0.00	N/A	1535	0.073	BV	0.03

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Figure B.4.2 Fatty acid profile chromatogram for fresh pulp and peel (sample 1).

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Peak #	Ret. Time	Name	Amount	Amt %	Area	Area %	Type	Width
7	3.72		0.00	N/A	3019	0.144	VB	0.03
8	3.95		0.00	N/A	18818	0.895	BB	0.03
9	5.10		0.00	N/A	3079	0.147	BB	0.03
10	5.34		0.00	N/A	2316	0.110	BB	0.04
11	5.82		0.00	N/A	2294	0.109	BV	0.04
12	6.11		0.00	N/A	4822	0.229	VB	0.04
13	6.49		0.00	N/A	522355	24.858	BV	0.04
14	6.69		0.00	N/A	4072	0.194	VV	0.05
15	6.81		0.00	N/A	500426	23.814	VV	0.04
16	6.95		0.00	N/A	4338	0.206	VB	0.04
17	7.14		0.00	N/A	775	0.037	BB	0.04
18	7.49		0.00	N/A	4200	0.200	BB	0.04
19	7.96		0.00	N/A	3376	0.161	BV	0.04
20	8.16		0.00	N/A	35609	1.695	VV	0.04
21	8.32		0.00	N/A	92874	4.420	VV	0.04
22	8.50		0.00	N/A	3440	0.164	VB	0.04
23	8.88		0.00	N/A	1590	0.076	BB	0.10
24	9.11		0.00	N/A	1849	0.088	BB	0.04
25	9.57		0.00	N/A	20235	0.963	BV	0.04
26	9.72		0.00	N/A	2793	0.133	VV	0.05
27	9.85		0.00	N/A	279229	13.288	VV	0.04
28	9.96		0.00	N/A	149845	7.131	VV	0.04
29	10.13		0.00	N/A	858	0.041	VV	0.04
30	10.46		0.00	N/A	195362	9.297	VV	0.04
31	10.75		0.00	N/A	676	0.032	VB	0.05
32	11.26		0.00	N/A	114151	5.432	BB	0.05
33	11.53		0.00	N/A	7496	0.357	BV	0.04
34	11.68		0.00	N/A	2782	0.132	VB	0.05
35	12.15		0.00	N/A	626	0.030	BV	0.09
36	12.36		0.00	N/A	1744	0.083	VB	0.05
37	12.88		0.00	N/A	10827	0.515	BB	0.05
38	13.16		0.00	N/A	3724	0.177	BB	0.05
39	13.30		0.00	N/A	1655	0.079	BB	0.06
40	13.82		0.00	N/A	857	0.041	BB	0.07
41	14.47		0.00	N/A	16164	0.769	BB	0.05
42	14.74		0.00	N/A	3359	0.160	BV	0.07
43	14.89		0.00	N/A	2744	0.131	VB	0.06
44	15.60		0.00	N/A	1266	0.060	BB	0.12
45	16.03		0.00	N/A	2060	0.098	BB	0.06
46	16.17		0.00	N/A	6243	0.297	BB	0.05
47	16.59		0.00	N/A	12709	0.605	BB	0.20
48	17.59		0.00	N/A	13489	0.642	BB	0.06
49	17.83		0.00	N/A	3771	0.179	BB	0.14
50	18.40		0.00	N/A	1792	0.085	BB	0.19
51	19.85		0.00	N/A	5205	0.248	BV	0.07
52	20.33		0.00	N/A	2085	0.099	VB	0.08
53	21.01		0.00	N/A	15886	0.756	BB	0.20

Total Area = 2101363 Total Height = 803189.5 Total Amount = 0

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Figure B.4.2 Fatty acid profile chromatogram for fresh pulp and peel (sample 1). (cont'd)

Chrom Perfect Chromatogram Report

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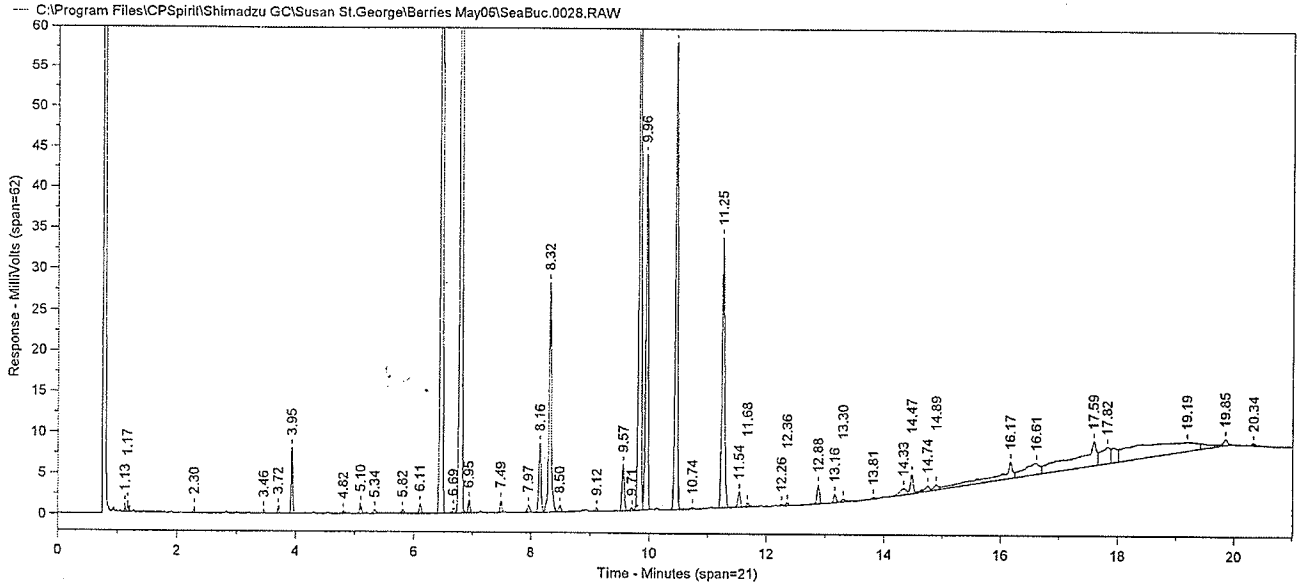

Sample Name = 06Berries

Instrument = Shimadzu
Heading 1 = 1uL inj.: 20:1split DB225 30m x 0.25um
Heading 2 = May2/06 155int. 4C/min.-215C - 4min

Acquisition Port = 4

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Method File Name = C:\Program Files\CPSpirit\Shimadzu GC\Hma.met
Calibration File Name =

Method Version = 5
Calibration Version = 0

Peak #	Ret. Time	Name	Amount	Amt %	Area	Area %	Type	Width
1	1.13		0.00	N/A	1094	0.058	BV	0.02
2	1.17		0.00	N/A	2086	0.111	VB	0.04
3	2.30		0.00	N/A	961	0.051	BB	0.02
4	3.46		0.00	N/A	850	0.045	BB	0.03
5	3.72		0.00	N/A	1562	0.083	BB	0.03
6	3.95		0.00	N/A	13766	0.732	BB	0.03

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Figure B.4.3 Fatty acid profile chromatogram for dried pulp and peel (50°C - 30.6%RH, sample 1).

Chrom Perfect Chromatogram Report

Peak #	Ret. Time	Name	Amount	Amt %	Area	Area %	Type	Width
7	4.82		0.00	N/A	715	0.038	BB	0.03
8	5.10		0.00	N/A	2297	0.122	BB	0.03
9	5.34		0.00	N/A	1370	0.073	BB	0.04
10	5.62		0.00	N/A	1203	0.064	BV	0.03
11	6.11		0.00	N/A	2615	0.139	VB	0.04
12	6.48		0.00	N/A	399504	21.238	BB	0.04
13	6.69		0.00	N/A	1924	0.102	BV	0.05
14	6.81		0.00	N/A	396090	21.056	VV	0.04
15	6.95		0.00	N/A	3539	0.188	VB	0.03
16	7.49		0.00	N/A	3229	0.172	BB	0.04
17	7.97		0.00	N/A	2699	0.143	BB	0.05
18	8.16		0.00	N/A	22585	1.201	BV	0.04
19	8.32		0.00	N/A	78749	4.186	VV	0.04
20	8.50		0.00	N/A	2036	0.108	VB	0.04
21	9.12		0.00	N/A	1125	0.060	BB	0.04
22	9.57		0.00	N/A	15144	0.805	BB	0.04
23	9.71		0.00	N/A	940	0.050	BV	0.05
24	9.84		0.00	N/A	222629	11.835	VV	0.04
25	9.96		0.00	N/A	115871	6.160	VB	0.04
26	10.46		0.00	N/A	155690	8.277	BV	0.04
27	10.74		0.00	N/A	712	0.038	VB	0.05
28	11.25		0.00	N/A	102769	5.463	BV	0.05
29	11.54		0.00	N/A	5249	0.279	VV	0.04
30	11.68		0.00	N/A	1549	0.082	VB	0.05
31	12.26		0.00	N/A	662	0.035	BV	0.05
32	12.36		0.00	N/A	1062	0.056	VB	0.05
33	12.88		0.00	N/A	6968	0.370	BB	0.05
34	13.16		0.00	N/A	3069	0.163	BB	0.05
35	13.30		0.00	N/A	1342	0.071	BB	0.07
36	13.81		0.00	N/A	619	0.033	BB	0.08
37	14.33		0.00	N/A	6027	0.320	BV	0,15
38	14.47		0.00	N/A	8317	0.442	VV	0.05
39	14.74		0.00	N/A	3897	0.207	VV	0.09
40	14.89		0.00	N/A	3416	0.182	VV	0.07
41	16.17		0.00	N/A	36658	1.949	VV	0.23
42	16.61		0.00	N/A	27029	1.437	VV	0.26
43	17.59		0.00	N/A	77439	4.117	VV	0.12
44	17.82		0.00	N/A	23282	1.238	VV	0.12
45	19.19		0.00	N/A	115181	6.123	VV	1.12
46	19.85		0.00	N/A	3890	0.207	VB	0.07
47	20.34		0.00	N/A	1700	0.090	BB	0.09

Total Area = 1881107 Total Height = 637202.3 Total Amount = 0

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Figure B.4.3 Fatty acid profile chromatogram for dried pulp and peel (50°C - 30.6%RH, sample 1). (cont'd)

Table B.4.2 Major fatty acids of berry pulp and peel (Coordinates with Tables 4.3 and B.4.1)

Temp /*RH*[a]	Sample	Fatty acid amount, % mass/mass (w/w)[b]						
		C16:0	C16:1*n*7	C18:0	C18:1*n*9	unknown	C18:2*n*6	C18:3*n*3
		Dried pulp and peel						
50°C /30.6%	1	22.17	21.98	0.84	12.35	6.43	8.64	5.70
	2	26.83	25.85	0.94	13.97	7.47	9.82	5.56
	3	25.48	24.69	0.95	14.50	7.82	9.93	6.44
	Mean	24.82	24.17	0.91	13.61	7.24	9.46	5.90
	SD[c]	2.40	1.99	0.06	1.12	0.72	0.72	0.47
	CV,%[d]	9.67	8.23	6.59	8.23	9.94	7.61	7.97
50°C /58.7%	1	24.58	23.82	1.05	14.09	7.13	11.9	8.06
	2	24.87	24.75	0.92	13.27	7.39	9.85	6.16
	3	27.45	26.10	0.93	14.91	7.60	8.89	5.21
	Mean	25.63	24.89	0.96	14.09	7.37	10.22	6.47
	SD	1.58	1.14	0.07	0.82	0.24	1.55	1.45
	CV,%	6.16	4.58	7.29	5.82	3.26	15.2	22.4
60°C /25.5%	1	26.30	26.25	0.90	13.93	7.86	9.15	5.38
	2	23.51	22.82	0.78	12.19	6.30	8.20	4.48
	3[f]	24.67	23.38	1.03	14.52	7.60	10.37	6.81
	Mean	24.90	24.54	0.84	13.06	7.08	8.67	4.93
	SD	1.98	2.42	0.09	1.23	1.10	0.67	0.64
	CV,%	7.95	9.86	10.2	9.42	15.5	7.73	13.0
60°C /57.0%	1	27.55	26.57	0.97	13.76	7.44	9.82	5.30
	2	25.87	25.07	0.98	14.27	7.26	10.98	6.81
	3	26.81	26.63	0.94	13.85	7.59	9.96	5.41
	Mean	26.74	26.09	0.96	13.96	7.43	10.25	5.84
	SD	0.84	0.88	0.02	0.27	0.17	0.63	0.84
	CV,%	3.14	3.37	2.08	1.93	2.29	6.15	14.4

Table B.4.2 Major fatty acids of berry pulp and peel (cont'd)

Temp /*RH*	Sample	Fatty acid amount, % (w/w)						
		C16:0	C16:1*n*7	C18:0	C18:1*n*9	unknown	C18:2*n*6	C18:3*n*3
					Dried pulp and peel			
70°C /20.8%	1	22.35	20.87	1.02	14.23	6.96	10.87	7.48
	2	20.53	19.10	0.87	11.70	6.06	10.18	6.93
	3	23.95	22.05	1.09	15.34	7.37	11.89	8.09
	Mean	22.27	20.67	0.99	13.75	6.79	10.98	7.50
	SD	1.71	1.49	0.11	1.87	0.67	0.86	0.58
	CV,%	7.68	7.21	11.1	13.6	9.87	7.83	7.73
70°C /57.0%	1	23.35	21.45	1.13	14.69	6.95	12.86	9.35
	2	20.56	19.99	0.79	12.08	6.51	8.33	5.45
	3	23.67	21.95	1.09	14.98	7.19	12.23	8.64
	Mean	22.53	21.13	1.00	13.92	6.88	11.14	7.81
	SD	1.71	1.01	0.19	1.60	0.34	2.45	2.08
	CV,%	7.59	4.78	19.0	11.5	4.94	22.0	26.6
					Fresh pulp and peel			
n/a[e]	1[f]	26.01	24.92	1.01	13.90	7.46	9.73	5.68
	2	20.33	19.55	0.78	11.59	6.30	7.40	4.89
	3	25.36	25.38	0.97	14.14	7.87	8.68	5.54
	Mean	22.85	22.46	0.88	12.87	7.09	8.04	5.22
	SD	3.56	4.12	0.14	1.81	1.11	0.90	0.46
	CV,%	15.6	18.3	15.9	14.1	15.7	11.2	8.81

[a] Temp/*RH* = drying conditions (temperature and relative humidity).
[b] C16:0 = palmitic acid. C16:1*n*7 = palmitoleic acid. C18:0 = stearic acid. C18:1*n*9 = oleic acid. C18:2*n*6 = linoleic acid. C18:3*n*3 = α-linolenic acid.
[c] *SD* = standard deviation.
[d] *CV* = coefficient of variation.
[e] n/a = not applicable.
[f] Sample data not used for analysis due to outlier.

Refer to Sample calculations for Table A.1.1 in Appendix A.1 for Mean, *SD*, and *CV*.

Appendix B.5 Tocol analysis for sea buckthorn berry pulp and peel

Table B.5.1 Tocol concentration analysis for berry pulp and peel
(Coordinates with Table 4.4 and B.5.2)

	Tocols[a]						
	α – T	α – T3	β – T	β – T3	γ – T	γ – T3	δ – T
	Fresh pulp and peel (sample1)						
Retention time, min	6.81	7.83	8.83	10.12	10.79	12.81	15.55
Y_{tocol}[b]	5217024	42971	421309	542400	466467	104669	1286075
$X1_{tocol}$[c] ng/10μL	131.632	1.084	6.829	8.791	9.515	2.135	19.943
C_{oil}[d] mg/mL	2.93	2.93	2.93	2.93	2.93	2.93	2.93
$X2_{tocol}$[e] mg/100g	449.2	3.7	23.3	30.0	32.5	7.3	68.1
	Dried pulp and peel (50°C - 30.6%RH, sample 1)						
Retention time, min	6.65	7.45	8.58	9.80	10.44	12.29	14.90
Y_{tocol}	6838357	122301	527002	670130	540955	127321	1599077
$X1_{tocol}$ ng/10μL	172.541	3.086	8.542	10.861	11.035	2.597	24.797
C_{oil} mg/mL	3.25	3.25	3.25	3.25	3.25	3.25	3.25
$X2_{tocol}$ mg/100g	530.9	9.5	26.3	33.4	33.9	8.0	76.3

[a] α-T = α-tocopherol. β-T = β-tocopherol. γ-T = γ-tocopherol. δ-T = δ-tocopherol. α-T3 = α-tocotrienol. β-T3 = β-tocotrienol. γ-T3 = γ-tocotrienol. δ-T3 = δ-tocotrienol. P-8 = Plastochromanol-8.
[b] Y_{tocol} = area of individual tocol.
[c] $X1_{tocol}$ = concentration of tocol in prepared hexane solution.
[d] C_{oil} = concentration of oil in hexane solution.
[e] $X2_{tocol}$ = concentration of tocol in oil.

Refer to "Sample calculations for Table A.5.1" in Appendix A.5 for tocol concentration in conjunction with Figs. B.5.1-2.

Chrom Perfect Chromatogram Report

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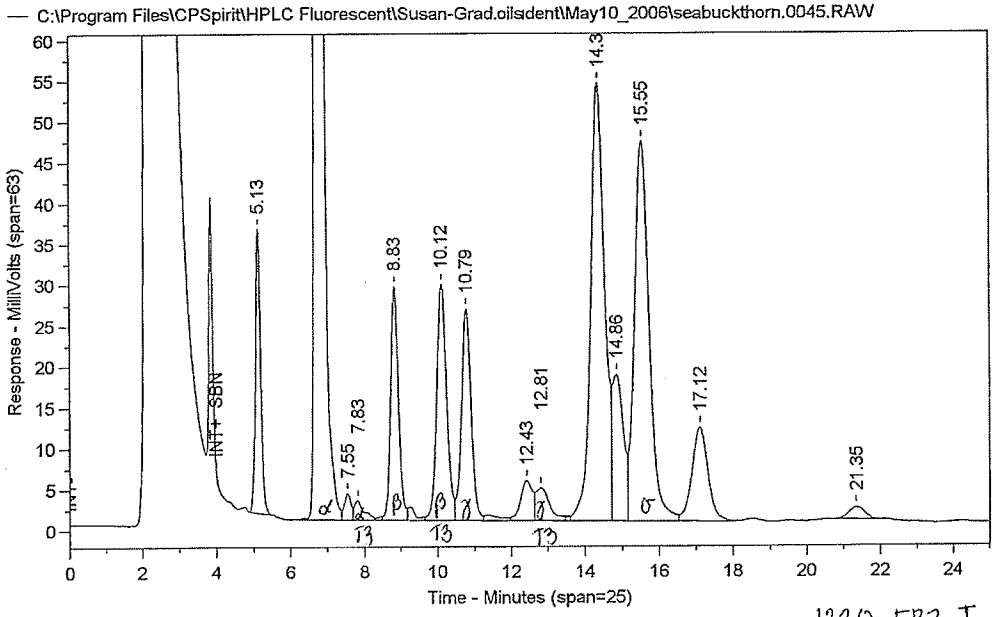

Instrument = HPLC Fluorescent
Heading 1 = Tocopherol Analysis -Susan -seabuckthorn
Heading 2 = May10,2006; 10uL inj; 0.8mL/min; 25 min. run time

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Wt. (g) = _____

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Sample Name = oils

Method File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol5.MET
Method Description = Tocopherol Analysis - column: Phenomenex 250x3.2mm 5micron; 5u silicon
Calibration File Name =
Run Time = 25

Peak #	Ret. Time	Peak Name	Amount	Amt %	Area	Area %	Group #
1	5.13		0.000	N/A	333410	3.11	[No data]
2	6.81	- alpha	0.000	N/A	5217024	48.69	[No data]
3	7.55		0.000	N/A	42195	0.39	[No data]
4	7.83	- alphaT3	0.000	N/A	42971	0.40	[No data]
5	8.83	- beta	0.000	N/A	421309	3.93	[No data]
6	10.12	- beta T3	0.000	N/A	542400	5.06	[No data]
7	10.79	- gamma	0.000	N/A	466467	4.35	[No data]
8	12.43		0.000	N/A	109911	1.03	[No data]
9	12.81	- gammaT3	0.000	N/A	104669	0.98	[No data]
10	14.37		0.000	N/A	1389876	12.97	[No data]
11	14.86		0.000	N/A	367625	3.43	[No data]
12	15.55	- delta	0.000	N/A	1286075	12.00	[No data]
13	17.12		0.000	N/A	346795	3.24	[No data]
14	21.35		0.000	N/A	44083	0.41	[No data]

Total Area = 1.071481E+07 Total Amount = 0

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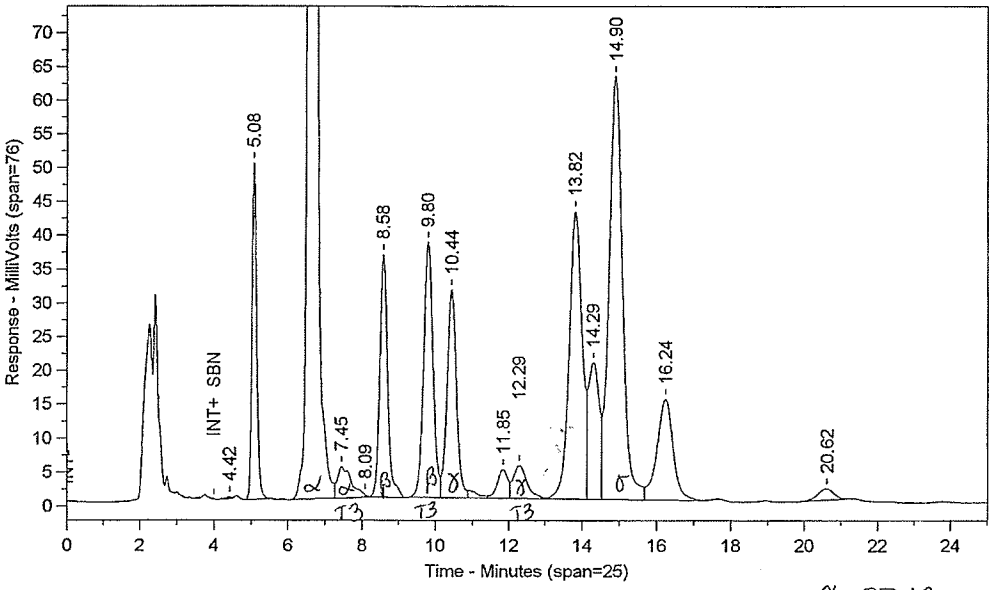
Figure B.5.1 Tocol chromatogram for fresh pulp and peel (sample 1).

Chrom Perfect Chromatogram Report

— C:\Program Files\CPSpirit\HPLC Fluorescent\Susan-Grad.oils\dent\May10_2006\seabuckthorn.0013.RAW



Instrument = HPLC Fluorescent
Heading 1 = Tocopherol Analysis -Susan -seabuckthorn
Heading 2 = May10,2006; 10uL inj; 0.8mL/min; 25 min. run time

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Wt. (g) = _____

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Sample Name = oils

Method File Name = C:\Program Files\CPSpirit\HPLC Fluorescent\Tocopherol5.MET
Method Description = Tocopherol Analysis - column: Phenomenex 250x3.2mm 5micron; 5u silicon
Calibration File Name =
Run Time = 25

Peak #	Ret. Time	Peak Name	Amount	Amt %	Area	Area %	Group #
1	4.42		0.000	N/A	2052	0.02	[No data]
2	5.08		0.000	N/A	430109	3.34	[No data]
3	6.65	- alpha	0.000	N/A	6838357	53.12	[No data]
4	7.45	- alphaT3	0.000	N/A	122301	0.95	[No data]
5	8.09		0.000	N/A	1600	0.01	[No data]
6	8.58	- beta	0.000	N/A	527002	4.09	[No data]
7	9.80	- betaT3	0.000	N/A	670130	5.21	[No data]
8	10.44	- gamma	0.000	N/A	540955	4.20	[No data]
9	11.85		0.000	N/A	83917	0.65	[No data]
10	12.29	- gammaT3	0.000	N/A	127321	0.99	[No data]
11	13.82		0.000	N/A	1016798	7.90	[No data]
12	14.29		0.000	N/A	419604	3.26	[No data]
13	14.90	- delta	0.000	N/A	1599077	12.42	[No data]
14	16.24		0.000	N/A	444605	3.45	[No data]
15	20.62		0.000	N/A	50657	0.39	[No data]

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Figure B.5.2 Tocol chromatogram for dried pulp and peel (50°C - 30.6%RH, sample 1).

Table B.5.2 Tocols of berry pulp and peel (Coordinates with Tables 4.4 and B.5.1)

Temp /*RH*[a]	Sample	Tocols[b]								
		α – T	α – T3	β - T	β – T3	γ – T	γ – T3	δ - T	δ – T3	Total
		Dried pulp and peel								
50°C /30.6%	1	530.9	9.5	26.3	33.4	33.9	8.0	76.3	n/d[c]	718.3
	2	544.9	7.7	27.8	34.8	35.9	11.3	78.0	n/d	740.4
	3	513.3	3.9	23.4	32.7	26.1	11.3	69.4	n/d	680.1
	Mean	529.7	7.0	25.8	33.7	32.0	10.2	74.6	n/d	713.0
	SD[d]	15.8	2.9	2.2	1.1	5.2	1.9	4.5	n/d	30.5
	CV,%[e]	3.0	41.4	8.5	3.3	16.3	18.6	6.0	n/d	4.3
50°C /58.7%	1	622.4	5.7	30.8	37.9	41.1	15.2	81.9	n/d	835.2
	2	555.5	4.8	26.3	34.7	34.7	13.5	80.9	n/d	750.5
	3	463.7	6.8	21.6	29.4	30.9	6.0	67.2	n/d	625.8
	Mean	547.2	5.8	26.3	34.0	35.6	11.6	76.7	n/d	737.2
	SD	79.7	1.0	4.6	4.3	5.2	4.9	8.2	n/d	105.3
	CV,%	14.6	17.2	17.5	12.6	14.6	42.2	10.7	n/d	14.3
60°C /24.4%	1	362.0	2.8	16.1	28.4	22.7	4.8	51.4	n/d	488.2
	2	439.3	3.7	19.9	29.4	26.1	9.4	79.3	n/d	607.1
	3[g]	239.2	0.6	11.0	17.5	12.9	3.7	35.5	n/d	320.4
	Mean	400.7	3.2	18.0	28.9	24.4	7.1	65.4	n/d	547.6
	SD	54.7	0.6	2.7	0.7	2.4	3.3	19.7	n/d	84.1
	CV,%	13.7	18.8	15.0	2.4	9.8	46.5	30.1	n/d	15.4
60°C /57.0%	1	518.5	1.4	24.6	32.0	33.5	10.5	72.9	n/d	693.5
	2	510.7	4.2	24.0	31.2	36.9	9.3	76.5	n/d	692.8
	3	358.6	1.1	17.3	24.7	26.5	4.2	48.3	n/d	480.8
	Mean	462.6	2.2	22.0	29.3	32.3	8.0	65.9	n/d	622.3
	SD	90.1	1.7	4.1	4.0	5.3	3.3	15.3	n/d	122.6
	CV,%	19.5	77.3	18.6	13.7	16.4	41.3	23.2	n/d	19.7

Table B.5.2 Tocols of berry pulp and peel (cont'd)

Temp /*RH*	Sample	Tocols								
		α – T	α – T3	β – T	β – T3	γ – T	γ – T3	δ - T	δ – T3	Total
70°C /20.8%	1	377.4	2.8	16.9	25.3	23.4	5.4	56.8	n/d	508.1
	2	335.1	1.0	15.7	21.6	21.2	5.0	49.3	n/d	448.8
	3	342.8	2.6	16.5	24.8	22.6	4.9	49.2	n/d	463.3
	Mean	351.8	2.1	16.3	23.9	22.4	5.1	51.8	n/d	473.4
	SD	22.5	1.0	0.6	2.0	1.1	0.3	4.4	n/d	30.9
	CV,%	6.4	47.6	3.7	8.4	4.9	5.9	8.5	n/d	6.5
70°C /57.0%	1	557.6	6.2	27.3	35.2	41.4	12.9	74.7	n/d	755.4
	2	443.3	1.1	19.8	29.5	24.1	6.1	55.8	n/d	579.7
	3	556.7	2.8	26.5	36.3	35.9	8.5	73.1	0.6	740.4
	Mean	519.2	3.4	24.6	33.6	33.8	9.2	67.9	0.2	691.8
	SD	65.8	2.6	4.1	3.6	8.8	3.4	10.5	0.3	97.4
	CV,%	12.7	76.5	16.7	10.7	26.0	37.0	15.5	150	14.1
		Fresh pulp and peel								
n/a[f]	1[g]	449.2	3.7	23.3	30.0	32.5	7.3	68.1	n/d	614.0
	2	544.0	3.5	25.5	36.8	33.8	6.9	64.0	n/d	714.4
	3	529.2	3.8	25.4	32.6	26.9	13.7	68.8	n/d	700.4
	Mean	536.6	3.6	25.4	34.7	30.4	10.3	66.4	n/d	707.4
	SD	10.5	0.2	0.0	3.0	4.9	4.9	3.4	n/d	9.9
	CV,%	2.0	5.2	0.3	8.6	16.1	46.6	5.1	n/d	1.4

[a] Temp/*RH* = drying conditions (temperature and relative humidity).
[b] α-T = α-tocopherol. β-T = β-tocopherol. γ-T = γ-tocopherol. δ-T = δ-tocopherol. α-T3 = α-tocotrienol. β-T3 = β-tocotrienol. γ-T3 = γ-tocotrienol. δ-T3 = δ-tocotrienol.
[c] n/d = not detectable.
[d] *SD* = standard deviation.
[e] *CV* = coefficient of variation.
[f] n/a = not applicable.
[g] Sample data not used for analysis due to outlier.

Refer to "Sample calculations for Table A.1.1" in Appendix A.1 for Mean, *SD*, and *CV*.

Appendix B.6 Phytosterol analysis for sea buckthorn berry pulp and peel

Table B.6.1 Phytosterol concentration analysis of pulp and peel
(Coordinates with Tables 4.5 and B.6.2)

	Phytosterol				
	5α-cholestane	Cholesterol	Campesterol	Stigmasterol	β-sitosterol
	Standard				
Retention time, min	15.52	16.13	18.08	18.74	19.87
	Fresh pulp and peel (sample 1)				
Retention time, min	15.53	16.15	18.10	n/d[a]	19.97
A_{sterol} [b]	95233	32804	18525	n/d	952169
$X1_{sterol}$ [c] µg	216	74.403	42.017	n/d	2159.6
m_{oil} [d] g	0.1021	0.1021	0.1021	0.1021	0.1021
$X2_{sterol}$ [e] mg/100 g oil	n/a[f]	72.873	41.153	n/d	2115.2
	Dried pulp and peel (50°C - 30.6%RH, sample 1)				
Retention time, min	15.53	16.16	18.09	n/d	19.94
A_{sterol}	111135	16941	11102	n/d	612680
$X1_{sterol}$ µg	216	32.926	21.578	n/d	1190.8
m_{oil} g	0.0975	0.0975	0.0975	0.0975	0.0975
$X2_{sterol}$ mg/100 g oil	n/a	33.770	22.131	n/d	1221.3

[a] n/d = not detected.
[b] A_{sterol} = area of individual phytosterol on chromatogram.
[c] $X1_{sterol}$ = amount of individual sterol.
[d] m_{oil} = mass of oil.
[e] $X2_{sterol}$ = mass of individual phytosterol per 100 g oil sample.
[f] n/a = not applicable.

Refer to "Sample calculations for Table A.6.1" in Appendix A.6 for phytosterol concentration in conjunction with Figs. B.6.1-3.

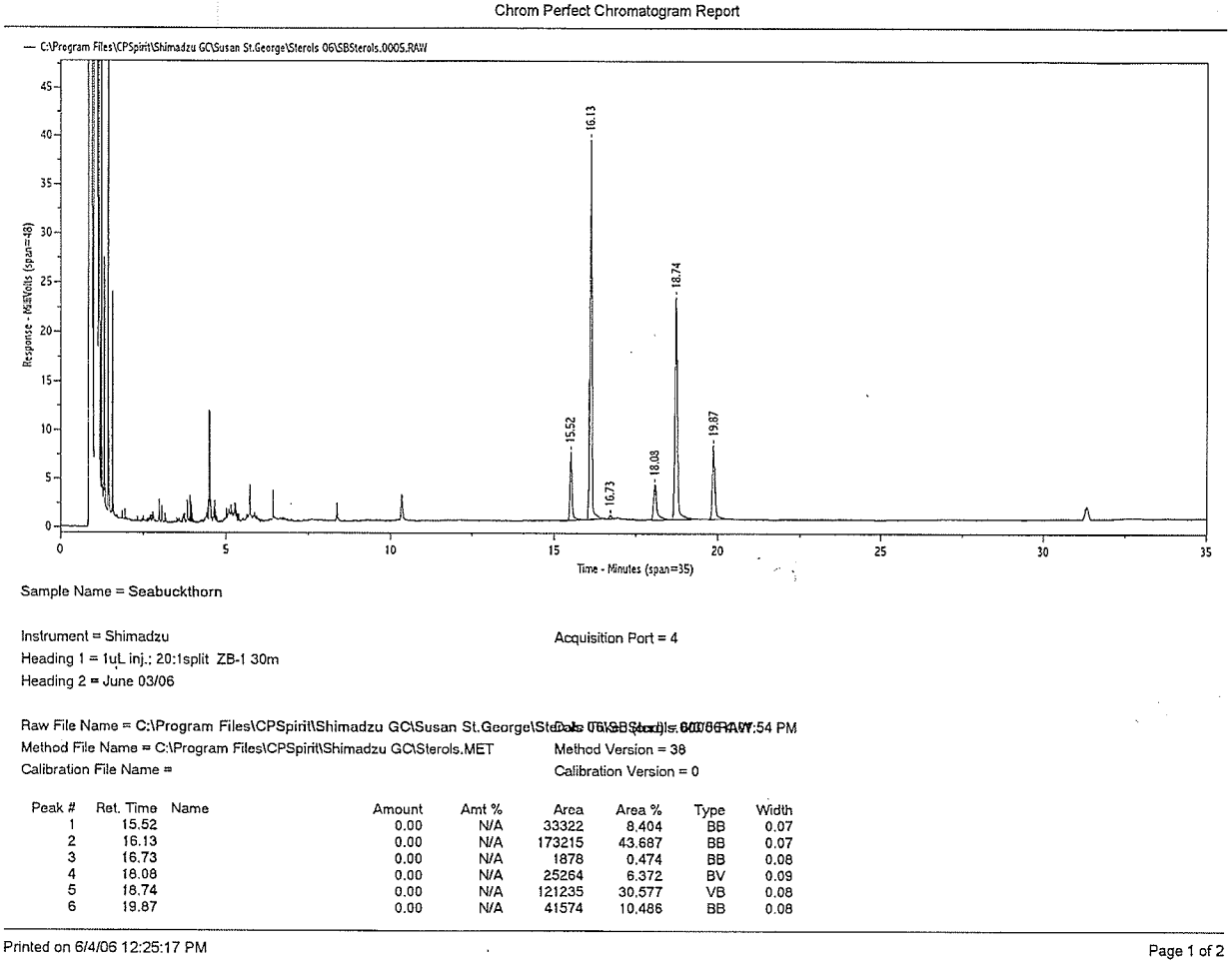

Figure B.6.1 Phytosterol chromatogram for standards. Standards with respective retention times: 5α-cholestane, 15.52min; cholesterol, 16.13min; campesterol, 18.08 min; stigmasterol, 18.74min, and β-sitosterol, 19.87min.

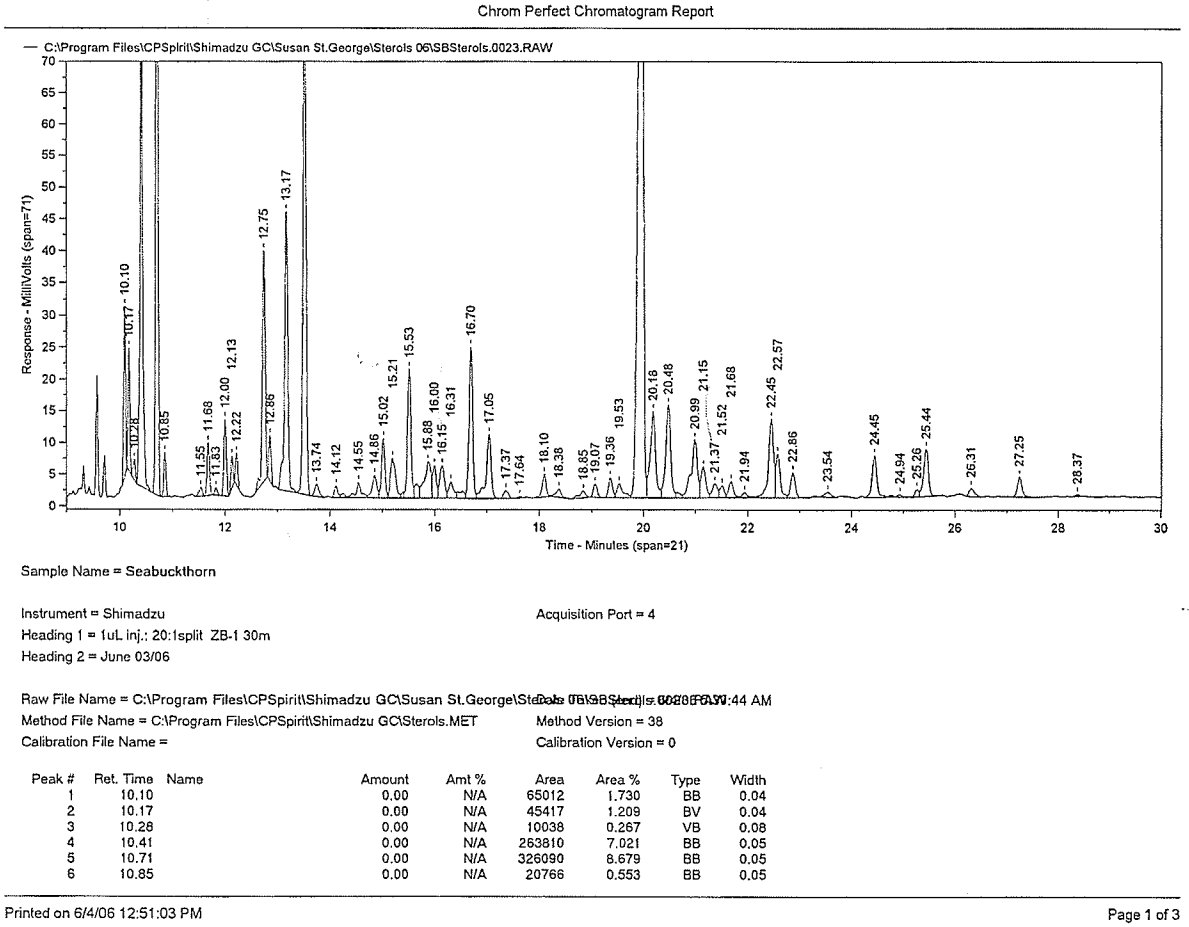

Figure B.6.2 Phytosterol chromatogram for fresh pulp and peel (sample 1). Standards with respective retention times: 5α-cholestane, 15.53 min; cholesterol, 16.15 min; campesterol, 18.10 min, and β-sitosterol, 19.97 min.

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Peak #	Ret. Time	Name	Amount	Amt %	Area	Area %	Type	Width
7	11.55		0.00	N/A	6060	0.161	BB	0.07
8	11.68		0.00	N/A	28086	0.748	BB	0.05
9	11.83		0.00	N/A	4054	0.108	BB	0.05
10	12.00		0.00	N/A	41267	1.098	BB	0.05
11	12.13		0.00	N/A	14554	0.387	BB	0.06
12	12.22		0.00	N/A	15451	0.411	BB	0.06
13	12.75		0.00	N/A	127239	3.387	BB	0.05
14	12.86		0.00	N/A	21658	0.576	BB	0.05
15	13.17		0.00	N/A	190210	5.062	BB	0.06
16	13.52		0.00	N/A	316229	8.417	BB	0.06
17	13.74		0.00	N/A	11178	0.298	BB	0.09
18	14.12		0.00	N/A	8362	0.223	BV	0.07
19	14.55		0.00	N/A	17071	0.454	VV	0.07
20	14.86		0.00	N/A	21779	0.580	VV	0.08
21	15.02		0.00	N/A	42113	1.121	VV	0.06
22	15.21		0.00	N/A	44683	1.189	VV	0.11
23	15.53		0.00	N/A	95233	2.535	VV	0.07
24	15.88		0.00	N/A	50041	1.332	VV	0.11
25	16.00		0.00	N/A	24484	0.652	VV	0.08
26	16.15		0.00	N/A	32804	0.873	VV	0.09
27	16.31		0.00	N/A	16752	0.446	VV	0.07
28	16.70		0.00	N/A	117610	3.130	VV	0.07
29	17.05		0.00	N/A	66800	1.778	VB	0.08
30	17.37		0.00	N/A	8195	0.218	BB	0.11
31	17.64		0.00	N/A	1194	0.032	BB	0.08
32	18.10		0.00	N/A	18525	0.493	BB	0.08
33	18.38		0.00	N/A	8587	0.229	BB	0.12
34	18.85		0.00	N/A	8665	0.231	BB	0.11
35	19.07		0.00	N/A	11343	0.302	BB	0.09
36	19.36		0.00	N/A	16597	0.442	BV	0.08
37	19.53		0.00	N/A	18173	0.484	VV	0.09
38	19.97		0.00	N/A	952169	25.342	VV	0.10
39	20.18		0.00	N/A	99007	2.635	VV	0.09
40	20.48		0.00	N/A	97235	2.588	VV	0.09
41	20.99		0.00	N/A	76669	2.041	VV	0.10
42	21.15		0.00	N/A	32295	0.860	VV	0.11
43	21.37		0.00	N/A	15696	0.418	VV	0.10
44	21.52		0.00	N/A	11021	0.293	VV	0.09
45	21.68		0.00	N/A	16568	0.441	VV	0.10
46	21.94		0.00	N/A	5568	0.148	VV	0.08
47	22.45		0.00	N/A	86006	2.289	VV	0.10
48	22.57		0.00	N/A	40189	1.070	VV	0.10
49	22.86		0.00	N/A	25020	0.666	VB	0.10
50	23.54		0.00	N/A	6738	0.179	BB	0.15
51	24.45		0.00	N/A	42212	1.123	BV	0.09
52	24.94		0.00	N/A	2532	0.067	VB	0.10
53	25.26		0.00	N/A	6289	0.167	BV	0.09
54	25.44		0.00	N/A	50042	1.332	VB	0.10
55	26.31		0.00	N/A	9087	0.242	BB	0.11
56	27.25		0.00	N/A	20467	0.545	BB	0.09
57	28.37		0.00	N/A	1853	0.049	BB	0.10

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Figure B.6.2 Phytosterol chromatogram for fresh pulp and peel (sample 1). Standards with respective retention times: 5α-cholestane, 15.53 min; cholesterol, 16.15 min; campesterol, 18.10 min, and β-sitosterol, 19.97 min. (cont'd).

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Peak #	Ret. Time	Name	Amount	Amt %	Area	Area %	Type	Width
58	31.18		0.00	N/A	5065	0.135	BV	0.12
59	31.54		0.00	N/A	2676	0.071	VV	0.18
60	31.89		0.00	N/A	3131	0.083	VB	0.09
61	32.55		0.00	N/A	3129	0.083	BB	0.12
62	34.22		0.00	N/A	3570	0.095	BB	0.17
63	36.43		0.00	N/A	6886	0.183	BB	0.33

Total Area = 3757247 Total Height = 802379.4 Total Amount = 0

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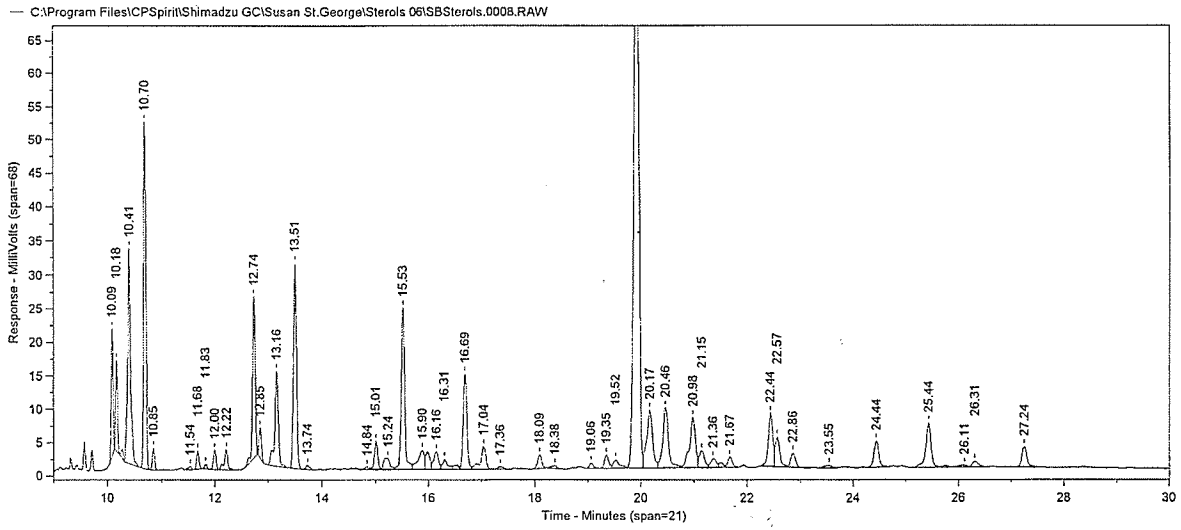
Figure B.6.2 Phytosterol chromatogram for pulp and peel (sample 1). Standards with respective retention times: 5α-cholestane, 15.53 min; cholesterol, 16.15 min; campesterol, 18.10 min, and β-sitosterol, 19.97 min (cont'd).

Chrom Perfect Chromatogram Report



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Instrument = Shimadzu
Heading 1 = 1uL inj.: 20:1split ZB-1 30m
Heading 2 = June 03/06

Acquisition Port = 4

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Method File Name = C:\Program Files\CPSpirit\Shimadzu GC\Sterols.MET
Calibration File Name =

Method Version = 38
Calibration Version = 0

Peak #	Ret. Time	Name	Amount	Amt %	Area	Area %	Type	Width
1	10.09		0.00	N/A	46040	2.228	BB	0.04
2	10.18		0.00	N/A	33972	1.644	BB	0.04
3	10.41		0.00	N/A	119149	5.767	BB	0.05
4	10.70		0.00	N/A	141366	6.842	BB	0.04
5	10.85		0.00	N/A	9883	0.478	BB	0.05
6	11.54		0.00	N/A	2398	0.116	BB	0.06

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Figure B.6.3 Phytosterol chromatogram for dried pulp and peel (50°C - 30.6%RH, sample 1). Sterols with respective retention times: 5α-cholestane, 15.53 min; cholesterol, 16.16 min; campesterol, 18.09 min, and β-sitosterol, 19.94 min.

Chrom Perfect Chromatogram Report

Peak #	Ret. Time	Name	Amount	Amt %	Area	Area %	Type	Width
7	11.68		0.00	N/A	9207	0.446	BB	0.05
8	11.83		0.00	N/A	2205	0.107	BB	0.05
9	12.00		0.00	N/A	9862	0.477	BB	0.05
10	12.22		0.00	N/A	14737	0.713	BB	0.06
11	12.74		0.00	N/A	88794	4.298	BB	0.06
12	12.85		0.00	N/A	13968	0.676	BB	0.06
13	13.16		0.00	N/A	68263	3.304	BB	0.06
14	13.51		0.00	N/A	122556	5.932	BB	0.06
15	13.74		0.00	N/A	3336	0.161	BB	0.09
16	14.84		0.00	N/A	2162	0.105	BV	0.08
17	15.01		0.00	N/A	18811	0.910	VV	0.07
18	15.24		0.00	N/A	14308	0.693	VV	0.12
19	15.53		0.00	N/A	111135	5.379	VV	0.07
20	15.90		0.00	N/A	24979	1.209	VV	0.11
21	16.16		0.00	N/A	16941	0.820	VV	0.08
22	16.31		0.00	N/A	10660	0.516	VV	0.07
23	16.69		0.00	N/A	70168	3.396	VV	0.07
24	17.04		0.00	N/A	23746	1.149	VB	0.08
25	17.36		0.00	N/A	2602	0.126	BB	0.11
26	18.09		0.00	N/A	11102	0.537	BB	0.08
27	18.38		0.00	N/A	3315	0.160	BB	0.16
28	19.06		0.00	N/A	4604	0.223	BV	0.08
29	19.35		0.00	N/A	11328	0.548	VV	0.09
30	19.52		0.00	N/A	10868	0.526	VV	0.10
31	19.94		0.00	N/A	612680	29.655	VV	0.09
32	20.17		0.00	N/A	67508	3.268	VV	0.10
33	20.46		0.00	N/A	66561	3.222	VV	0.09
34	20.98		0.00	N/A	57026	2.760	VV	0.10
35	21.15		0.00	N/A	18264	0.884	VV	0.12
36	21.36		0.00	N/A	10199	0.494	VV	0.10
37	21.67		0.00	N/A	9550	0.462	VB	0.09
38	22.44		0.00	N/A	47435	2.296	BV	0.09
39	22.57		0.00	N/A	25673	1.243	VV	0.10
40	22.86		0.00	N/A	12994	0.629	VB	0.10
41	23.55		0.00	N/A	4022	0.195	BB	0.18
42	24.44		0.00	N/A	25060	1.213	BB	0.10
43	25.44		0.00	N/A	47019	2.276	BV	0.10
44	26.11		0.00	N/A	3333	0.161	VV	0.17
45	26.31		0.00	N/A	7005	0.339	VB	0.12
46	27.24		0.00	N/A	21542	1.043	BB	0.10
47	35.28		0.00	N/A	4484	0.217	BB	0.48
48	36.38		0.00	N/A	3206	0.155	BB	0.31

Total Area = 2066026 Total Height = 432184.1 Total Amount = 0

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Figure B.6.3 Phytosterol chromatogram for dried pulp and peel (50°C - 30.6%RH, sample 1). Sterols with respective retention times: 5α-cholestane, 15.53 min; cholesterol, 16.16 min; campesterol, 18.09 min, and β-sitosterol, 19.94 min (cont'd).

Table B.6.2 Major phytosterol concentrations in pulp and peel
(Coordinates with Tables 4.5 and B.6.1)

Temp /*RH*[a]	Sample	Phytosterol concentration mg/100 g oil			
		Cholesterol	Campesterol	Stigmasterol	β-sitosterol
		Dried pulp and peel			
50°C /30.6%	1	33.77	22.13	n/d[b]	1221.33
	2	34.24	21.86	n/d	1310.82
	3	21.25	24.33	n/d	1242.06
	Mean	29.75	22.78	n/d	1258.07
	SD[c]	7.37	1.36	n/d	46.85
	CV,%[d]	24.8	5.97	n/d	3.72
50°C /58.7%	1	23.89	28.34	n/d	1337.65
	2	23.62	24.46	n/d	1376.47
	3	32.56	25.31	n/d	1182.16
	Mean	26.69	26.04	n/d	1298.76
	SD	5.09	2.04	n/d	102.83
	CV,%	19.1	7.83	n/d	7.92
60°C /24.4%	1	21.77	28.22	n/d	1280.42
	2	25.02	21.44	n/d	1089.85
	3[f]	37.29	25.50	n/d	1436.10
	Mean	23.40	24.83	n/d	1185.14
	SD	2.30	4.80	n/d	134.75
	CV,%	9.8	19.3	n/d	11.4
60°C /57.0%	1	21.04	22.33	n/d	1160.07
	2	24.66	28.18	n/d	1213.94
	3	25.66	31.53	n/d	1343.10
	Mean	23.79	27.34	n/d	1239.04
	SD	2.43	4.66	n/d	94.06
	CV,%	10.2	17.0	n/d	7.59

Table B.6.2 Major phytosterol concentrations in pulp and peel (cont'd)

Temp /*RH*	Sample	Phytosterol concentration mg/ 100 g oil			
		Cholesterol	Campesterol	Stigmasterol	β-sitosterol
			Dried pulp and peel		
70°C /20.8%	1	30.68	19.97	n/d	1191.92
	2	24.97	28.89	n/d	1527.03
	3	36.80	20.98	2.58	1084.72
	Mean	30.82	23.28	0.86	1267.89
	SD	5.92	4.89	1.49	230.73
	CV,%	19.2	21.0		18.20
70°C /57.0%	1	31.34	23.64	n/d	1253.57
	2	31.55	22.58	n/d	1230.98
	3	28.46	22.11	n/d	1251.10
	Mean	30.45	22.78	n/d	1245.21
	SD	1.72	0.79	n/d	12.39
	CV,%	5.65	3.47	n/d	0.995
			Fresh pulp and peel		
n/a[e]	1[f]	72.87	41.15	n/d	2115.22
	2	25.98	26.05	n/d	1377.14
	3	40.55	27.15	n/d	1353.96
	Mean	33.26	26.60	n/d	1365.55
	SD	10.30	0.78	n/d	16.39
	CV,%	31.0	2.9	n/d	1.20

[a] Temp/*RH* = drying conditions (temperature and relative humidity).
[b] n/d = not detected.
[c] *SD* = standard deviation.
[d] *CV* = coefficient of variation.
[e] n/a = not applicable.
[f] Sample data not used for analysis due to outlier.

Refer to "Sample calculations for Table A.1.1" in Appendix A.1 for Mean, *SD*, and *CV*.

C – APPENDICES FOR CHAPTER 5

Appendix C.1 Matlab program for press cake temperature, $t_{pc(i)}$, model

Note: Text following "%" are comments and not part of the program

% Heat and mass transfer analysis for material dried on an inert sphere

% Constants for drying model at 65°C and 45% RH

M_o = 1.314; % Initial moisture content, db

M_e = 0.0458; % Equilibrium moisture content, db

A = 9.78x10^{-3}; % Surface area of press cake/inert sphere system, (m^2)

m_{ip} = 0.148; % Mass of inert sphere, (kg)

Cp_{ip} = 1.400; % Specific heat capacity of inert sphere, (kJ/kg·K)

$F_{pc\text{-}fat}$ = 0.0379; % Multiplication factor for fat component in press cake

$F_{pc\text{-}sol}$ = 0.3943; % Multiplication factor for solids component in press cake

F_{pc-H_2O} = 0.5678; % Multiplication factor for water component in press cake

t_{air} = 65; % Chamber air temperature (°C)

h = 18.2; % Heat transfer coefficient, (W/m^2·K)

L = 2258; % Latent heat of vaporization at 100 Pa (kJ/kg)

$\Delta\tau$ = 0.016667; % Time increment (h)

k = 0.1956; % Page coefficient

n = 1.1382; % Page coefficient

% Initial conditions for drying model @ 65degc, 45% RH

$m_{pc(o)}$ = 0.035816; % Initial mass of press cake (kg)

$m_{pc(i-1)}$ = $m_{pc(o)}$; % Setting initial mass to mass at beginning of next time interval (kg)

$m_{pc\text{-}fat} = F_{pc\text{-}fat} m_{pc(o)}$; % Mass of fat component in press cake (kg)

$m_{pc\text{-}sol} = F_{pc\text{-}sol} m_{pc(o)}$; % Mass of solids component in press cake (kg)

$m_{pc-H_2O} = F_{pc-H_2O} m_{pc(o)}$; % Initial mass of water component in press cake (kg)

$m_{pc\text{-}w(i\text{-}1)} = m_{pc\text{-}w}$; % Setting initial $m_{pc\text{-}w}$ to $m_{pc\text{-}w}$ at beginning of next time interval (kg)

$t_{pc(o)} = 38.531$; % Initial temperature of press cake (°C)

$t_{pc(i\text{-}1)} = t_{pc(o)}$; % Setting initial t_{pc} to t_{pc} at beginning of next time interval (°C)

$i = 0$; % Iteration counter zeroed

C = zeros(490, 5); % Establishment of matrix with 490 rows and 5 columns

% Press cake temperature determination during drying cycle

for τ = (0.033334:0.016667:13.350); % Time iteration (h)

$i = i+1$; % Iteration counter

Term1 = $[hA(t_{air}\text{-}t_{pc(i\text{-}1)})\Delta\tau]\cdot 3.6$; % Convective heat transfer term (kJ)

% Calculations for moisture evaporation term

$MR = \exp(-k\tau^n)$; % Moisture ratio at $\tau_{(i)}$, using Page's model

$$m_{pc} = \frac{\left[MR\left(M_o - M_e\right) + M_e + 1\right] m_{pc(o)}}{M_o + 1}$$; % Mass of press cake at $\tau_{(i)}$ (kg)

$\Delta m_{pc} = m_{pc(i\text{-}1)}\text{-}m_{pc(i)}$; % Moisture loss during time interval (kg)

Term2 = $\Delta m_{pc} L$; % Energy required to evaporate moisture (kJ)

% Calculations for specific heat capacity of press cake

$m_{pc-H_2O} = m_{pc-H_2O(i-1)} - \Delta m_{pc}$; % Water component in press cake at time $\tau_{(i)}$ (kg)

$m_{pc\text{-}tot} = m_{pc\text{-}fat} + m_{pc\text{-}sol} + m_{pc-H_2O}$; % Mass of all components in press cake at $\tau_{(i)}$ (kg)

$$X_{fat}^{m} = \frac{m_{pc-fat}}{m_{pc-tot}}$$; % Mass fraction of fat component in press cake at $\tau_{(i)}$

$X^{m}_{sol} = \frac{m_{pc-sol}}{m_{pc-tot}}$; % Mass fraction of solids component in press cake at $\tau_{(i)}$

$X^{m}_{H_2O} = \frac{m_{pc-H_2O}}{m_{pc-tot}}$; % Mass fraction of water component in press cake at $\tau_{(i)}$

$Cp_{pc} = 2.309\, X^{m}_{fat} + 1.256\, X^{m}_{sol} + 4.187\, X^{m}_{H_2O}$; % Specific heat capacity at $\tau_{(i)}$ (kJ/kg·K)

Term3 = $t_{pc(i-1)}(m_{pc}Cp_{pc}+m_{ip}Cp_{ip})$; % Partial term for energy required to increase temperature of inert sphere and press cake at time $\tau_{(i)}$ (kJ)

Term4 = $(m_{pc}*Cp_{pc}+m_{ip}*Cp_{ip})$; % Partial term for energy required to increase temperature of inert sphere and press cake at time $\tau_{(i)}$ (kJ/K)

% Calculation of press cake temperature

t_{pc} = (Term1-Term2+Term3) / Term4; % Temperature of press cake at $\tau_{(i)}$ (°C)

% Resetting of parameters at $\tau_{(i)}$ to $\tau_{(i-1)}$ for the next time increment

$m_{pc(i-1)} = m_{pc}$; % Mass of press cake (kg)

$m_{pc\text{-}w(i-1)} = m_{pc\text{-}w}$; % Mass of water component in press cake (kg)

$t_{pc\text{-}(i-1)} = t_{pc}$; % Temperature of press cake (°C)

$m_{pc(g)} = m_{pc}*1000$; % Converting mass in (kg) to mass in (g) for tabulation purposes

% Storing the data in matrices

B = [$i\ \tau\ MR\ m_{pc(g)}\ t_{pc}$]; % Storing the data from time $\tau_{(i)}$ to matrix B

C(i,:) = B; % A copy of matrix B

end % End of iteration loop

Appendix C.2 Derivation of Eqns. 5.12 and 5.13

C.2.1 Derivation of final press cake mass, $m_{pc(f)}$ (Eqn. 5.12) (on the basis that mass of the complete sample at end of drying trial is equivalent to the final sample in moisture content determination).

1) Using ratio as shown in Eqn. C.2a

$$\frac{m_{mc(f)}}{m_{mc(f1)}} = \frac{m_{pc(f)}}{m_{pc(f1)}} \qquad \text{(C.2a)}$$

where $m_{mc(f)}$ = final mass of sample used in moisture content determination; $m_{mc(fi)}$ = initial mass of sample used in moisture content determination, and $m_{pc(f1)}$ = mass of complete sample at end of the drying trial.

2) Rearranging Eqn. C.2a in terms of $m_{pc(f)}$:

$$m_{pc(f)} = \left(\frac{m_{mc(f)}}{m_{mc(f1)}} \right) m_{pc(f1)}$$

Using the data from the moisture content determination test and the mass of complete sample at end of the drying trial, $m_{pc(f)}$ can be determined.

3) The moisture contents M_o and M_i can be determined using Eqn. 2.1, where $m_{pc(f)} = m_f$ and $m_{pc(o)} = m_i$. Since initial conditions for the validation trials are used in the model, $m_{pc(f)}$ (Eqn. 5.12) can be stated in terms of M_o:

$$m_{pc(f)} = \frac{m_{pc(o)}}{M_o + 1}$$

C.2.2 Derivation of press cake mass at time *i*, $m_{pc(i)}$ (Eqn. 5.13)

Incorporating Eqns. 2.1 and 5.12 into Eqn. 2.15 results in an equation in terms of $m_{pc(i)}$:

1) State Eqn. 2.15 in terms of $M_{(i)}$:

$$M_{(i)} = MR_{(i)}\left(M_o - M_e\right) + M_e$$

2) Replace $M_{(i)}$ by the terms in Eqn. 2.1:

$$\frac{m_{pc(i)} - m_{pc(f)}}{m_{pc(f)}} = MR_{(i)}\left(M_o - M_e\right) + M_e$$

where $m_{pc(i,)}$ and $m_{pc(f)}$ are represented by m_i and m_f, respectively, in Eqn.2.1.

3) Replace $m_{pc(f)}$ by terms in Eqn. 5.12:

$$m_{pc(i)} = \left[\left(\frac{m_{pc(o)}}{M_o + 1}\right)\left(MR_{(i)}\left(M_o - M_e\right) + M_e\right)\right] + \left(\frac{m_{pc(o)}}{M_o + 1}\right)$$

$$m_{pc(i)} = \frac{m_{pc(o)}}{M_o + 1}\left[MR_{(i)}\left(M_o - M_e\right) + M_e + 1\right]$$

Appendix C.3 Determination of heat transfer coefficient, *h*

Table C.3.1 Heat transfer coefficient, *h*, for a sphere (*d* = 0.0558 m)

t_{air}[a] K	λ_{air}[b] $\frac{W}{m \cdot K}$	ρ_{air}[c] $\frac{kg}{m^3}$	μ[d] $\times 10^{-5}$ $\frac{kg}{m \cdot s}$	Cp_{air}[e] $\frac{J}{kg \cdot K}$	Re[f]	Pr[g]	h_1[h] $\frac{W}{m^2 \cdot K}$	h_2[i] $\frac{W}{m^2 \cdot K}$
300.00	0.0263	1.1386	1.846	1007	3442	0.707	15.7	18.2
323.15	0.0280	1.0631	1.955	1008	3034	0.704	15.8	18.2
328.15	0.0284	1.0468	1.979	1008	2952	0.702	15.8	18.2
333.15	0.0288	1.0305	2.002	1008	2872	0.701	15.8	18.2
338.15	0.0291	1.0142	2.026	1009	2793	0.702	15.7	18.2
343.15	0.0295	0.9978	2.050	1009	2716	0.701	15.7	18.2
348.15	0.0299	0.9815	2.073	1009	2642	0.699	15.7	18.2
350.00	0.0300	0.9755	2.082	1009	2614	0.700	15.7	18.2
353.15	0.0302	0.9678	2.096	1009	2576	0.700	15.7	18.2
400.00	0.0338	0.8540	2.301	1014	2071	0.690	15.8	18.3

[a] t_{air} = temperature of drying chamber air.
[b] λ_{air} = thermal conductivity of air (Incropera and DeWitt 1995).
[c] ρ_{air} = density of the air (Incropera and DeWitt 1995), corrected for P = 100 Pa.
[d] μ = viscosity of air (Incropera and De Witt 1995).
[e] Cp_{air} = specific heat capacity of air (Incropera and DeWitt 1995).
[f] $\mathrm{Re} = \frac{\rho_{air} u d}{\mu}$; Reynolds Number where u = 1 m/s is the air velocity and d = diameter of sphere.
[g] $\mathrm{Pr} = \frac{\mu Cp_{air}}{\lambda_{air}}$; Pr = Prandtl's Number.
[h] $h_1 = \frac{\lambda_{air}}{d}\left[2.0 + 0.6(\mathrm{Re})^{\frac{1}{2}} \times (\mathrm{Pr})^{\frac{1}{3}}\right]$, heat transfer coefficient for flow over a sphere.
[i] $h_2 = \frac{\lambda_{air}}{d}\left[2.0 + 0.7(\mathrm{Re})^{\frac{1}{2}} \times (\mathrm{Pr})^{\frac{1}{3}}\right]$, heat transfer coefficient for flow over a sphere.

Sample calculation of Biot number: [65°C – 45% RH; h_1 = 15.7 W/(m^2·K) and h_2 = 18.2 W/(m^2·K)]

Based on Eqn. 2.8 the Biot number for the inert sphere of d = 0.0508 m (does not include press cake layer) and with λ = 0.25 W/(m·K) for h_1 and h_2 are 1.59 and 1.85, respectively, with calculations as follows:

h_1 :

$$\text{Bi} = \frac{15.7 \times 0.0254}{0.25}$$

h_2 :

$$\text{Bi} = \frac{18.2 \times 0.0254}{0.25}$$

Appendix C.4 Determination of moisture ratio, *MR*, and model coefficients

Table C.4.1 Data for sample trial (60°C - 45%RH, sample 1)

τ[a]	t_{air}[b]	RH[c]	m_{sys}[d]	$t_{air\text{-}s}$[e]	t_{amb}[f]	$t_{ip\text{-}s}$[g]	t_{pc}[h]	$t_{ip\text{-}c}$[i]
h	°C	%	x10^{-3} kg	°C	°C	°C	°C	°C
0	54.0	21	0.000	38.923	21.604	35.716	34.301	55.352
0.02	53.9	31	181.318	60.206	21.614	36.650	35.286	54.150
0.03	59.2	53	181.467	63.113	21.935	40.535	39.214	52.839
0.05	60.9	57	181.629	58.794	21.590	43.905	42.867	51.423
0.07	59.8	62	181.716	60.489	21.649	46.070	45.295	50.142
0.08	60.2	61	181.767	59.757	21.540	47.431	46.892	49.068
0.33	60.0	51	181.412	60.412	21.703	49.572	49.293	49.045
0.58	60.2	47	180.334	60.206	21.343	49.666	49.279	49.629
0.83	60.0	45	179.281	59.878	21.385	50.126	49.972	50.019
1.08	59.9	45	178.344	60.404	21.483	50.732	50.810	50.554
1.33	59.9	45	177.514	60.268	21.286	51.266	51.578	51.183
1.58	60.0	46	176.820	60.216	21.404	51.714	52.164	51.652
1.83	59.8	45	176.121	60.556	21.524	52.187	52.614	52.197
2.08	59.9	45	175.468	60.478	21.414	52.628	53.022	52.674
2.33	60.1	45	174.857	60.011	21.592	52.994	53.352	53.049
2.58	60.1	45	174.284	59.919	21.356	53.374	53.639	53.398
2.83	60.0	45	173.744	60.102	21.281	53.700	53.894	53.722
3.08	60.1	45	173.235	59.989	21.492	53.990	54.093	53.990
3.33	59.9	45	172.752	60.057	21.567	54.301	54.278	54.301
3.58	60.0	45	172.299	60.309	21.567	54.591	54.508	54.546
3.83	60.0	45	171.872	60.380	21.620	54.860	54.698	54.802
4.08	60.0	45	171.470	59.991	21.718	55.103	54.849	55.047
4.33	60.1	45	171.087	59.835	21.352	55.338	55.024	55.269
4.58	60.0	45	170.726	60.064	21.106	55.556	55.139	55.476
4.83	60.0	46	170.444	60.298	21.407	55.688	55.297	55.596
5.08	60.1	45	170.118	60.174	21.396	55.887	55.426	55.809
5.33	60.1	45	169.811	59.767	21.377	56.145	55.592	56.021
5.58	60.0	45	169.523	60.182	21.579	56.357	55.712	56.223
5.83	60.3	45	169.252	59.922	21.372	56.533	55.840	56.408
6.08	60.1	45	168.999	60.326	21.439	56.756	55.995	56.622
6.33	60.0	45	168.762	60.244	21.399	56.903	56.120	56.802
6.58	60.1	45	168.539	60.213	21.404	57.045	56.240	56.945
6.83	59.9	45	168.327	60.318	21.527	57.207	56.401	57.096
7.08	60.0	46	168.129	59.992	21.497	57.364	56.535	57.230

Table C.4.1 Data for sample trial (60°C - 45%RH, sample 1) (cont'd)

τ	t_{air}	RH	m_{sys}	$t_{air\text{-}s}$	t_{amb}	$t_{ip\text{-}s}$	t_{pc}	$t_{ip\text{-}c}$
h	°C	%	x10^{-3} kg	°C	°C	°C	°C	°C
7.33	60.3	45	167.948	59.984	21.329	57.460	56.632	57.358
7.58	60.1	45	167.773	60.432	21.429	57.576	56.747	57.465
7.83	60.1	45	167.608	60.299	21.335	57.662	56.844	57.584
8.08	60.1	45	167.456	60.528	21.424	57.800	56.972	57.732
8.33	59.8	45	167.313	60.171	21.665	57.910	57.106	57.832
8.58	59.9	45	167.177	60.208	21.483	58.016	57.212	57.938
8.83	60.0	45	167.051	60.638	21.542	58.118	57.290	58.040
9.08	60.1	46	166.932	60.446	21.345	58.210	57.395	58.098
9.33	60.0	45	166.823	60.208	21.276	58.260	57.479	58.181
9.58	60.1	45	166.720	59.910	21.385	58.384	57.593	58.274
9.83	60.0	45	166.622	60.290	21.414	58.466	57.676	58.370
10.08	60.0	45	166.532	59.947	21.473	58.512	57.732	58.402
10.33	59.9	46	166.450	60.162	21.335	58.604	57.824	58.494
10.58	59.9	45	166.371	60.208	21.409	58.682	57.892	58.548
10.83	60.0	45	166.299	60.048	20.940	58.682	57.915	58.586
11.08	60.0	45	166.232	60.242	21.002	58.832	58.064	58.722
11.33	60.0	45	166.169	60.233	21.140	58.858	58.102	58.749
11.58	60.1	44	166.113	59.693	21.116	58.868	58.156	58.801
11.83	60.2	45	166.061	60.244	21.069	58.994	58.274	58.829
12.08	59.9	45	166.013	60.181	20.860	58.998	58.310	58.888
12.33	60.1	45	165.966	59.938	20.920	59.053	58.365	58.930
12.58	60.0	45	165.922	60.651	20.925	59.118	58.476	59.017
12.83	60.0	45	165.885	60.204	20.826	59.157	58.554	59.047
13.08	60.0	45	165.848	60.045	21.012	59.183	58.610	59.106
13.33	60.0	45	165.815	59.991	20.745	59.198	58.616	59.087
13.58	60.0	45	165.783	60.320	20.938	59.254	58.702	59.129
13.83	60.0	45	165.757	60.549	20.754	59.243	58.739	59.166
14.08	60.0	45	165.729	60.343	20.814	59.276	58.818	59.198
14.33	60.1	45	165.707	60.393	20.735	59.271	58.821	59.194
14.58	60.0	45	165.683	59.903	20.897	59.248	58.835	59.161
14.83	60.0	45	165.662	60.032	20.749	59.308	58.917	59.207
15.08	60.0	45	165.657	60.064	20.735	59.203	58.835	59.216
15.33	59.9	45	165.658	60.446	20.631	59.175	58.877	59.039
15.58	60.1	45	165.627	60.102	20.678	59.242	58.943	59.086
15.83	59.9	45	165.606	60.208	20.842	59.255	58.980	59.168
16.08	60.0	45	165.589	59.992	20.772	59.283	59.039	59.228

Table C.4.1 Data for sample trial (60°C - 45%RH, sample 1) (cont'd)

τ	t_{air}	RH	m_{sys}	$t_{air\text{-}s}$	t_{amb}	$t_{ip\text{-}s}$	t_{pc}	$t_{ip\text{-}c}$
h	°C	%	x10^{-3} kg	°C	°C	°C	°C	°C
16.33	60.0	45	165.575	60.268	20.757	59.338	59.086	59.251
16.58	59.9	45	165.563	60.326	20.599	59.329	59.122	59.288
16.83	60.2	45	165.550	60.144	20.673	59.351	59.168	59.288
17.33	60.0	45	165.557	60.254	20.742	58.866	59.132	59.099
17.58	60.0	45	165.587	59.906	20.752	58.874	59.104	58.971
17.83	60.1	45	165.562	60.354	20.579	59.012	59.095	58.994
18.08	60.0	45	165.539	60.263	20.678	59.081	59.141	59.063
18.33	60.1	44	165.521	60.139	20.644	59.187	59.177	59.168
18.58	59.8	45	165.509	60.487	20.812	59.228	59.228	59.209
18.83	60.1	45	165.496	60.107	20.871	59.329	59.305	59.265
19.08	60.1	45	165.492	60.130	20.807	59.338	59.305	59.319
19.33	60.0	45	165.484	60.290	20.658	59.338	59.315	59.319
19.58	59.9	46	165.476	60.404	20.461	59.315	59.283	59.297
19.83	60.0	45	165.469	59.778	20.402	59.351	59.297	59.333
20.08	60.1	45	165.463	60.171	20.529	59.356	59.333	59.338
20.33	59.9	45	165.460	60.244	20.485	59.361	59.361	59.365
20.58	60.0	45	165.455	60.116	20.584	59.384	59.361	59.374
20.83	60.0	45	165.450	60.034	20.629	59.401	59.379	59.370
21.08	60.0	44	165.446	60.286	20.570	59.347	59.324	59.407
21.33	60.1	45	165.441	60.428	20.658	59.384	59.361	59.387
21.58	60.1	45	165.440	60.052	20.564	59.398	59.387	59.401
21.83	60.0	45	165.435	59.780	20.666	59.391	59.391	59.404
22.08	60.1	45	165.432	59.887	20.989	59.393	59.370	59.361
22.33	60.1	43	165.430	60.326	20.762	59.365	59.343	59.379
22.58	60.0	45	165.428	60.099	20.723	59.374	59.365	59.356
22.83	60.0	45	165.424	59.997	20.812	59.365	59.333	59.333
23.08	59.9	45	165.420	60.216	20.693	59.347	59.347	59.370

[a] τ = drying time.
[b] t_{air} = temperature inside environmental chamber (chamber sensor).
[c] RH = relative humidity (chamber sensor).
[d] m_{sys}= mass of inert sphere and press cake system
(i.e inert sphere, hook, thermocouples, and press cake).
[e] $t_{air\text{-}s}$ = temperature inside environmental chamber (independent thermocouple).
[f] t_{amb} = ambient temperature outside environmental chamber.
[g] $t_{ip\text{-}s}$ = temperature on inside surface of inert sphere.
[h] t_{pc} = temperature of press cake on surface of the inert sphere.
[i] $t_{ip\text{-}c}$ = temperature at the geometric centre of the inert sphere.

Table C.4.2 Moisture ratio for sample trial (60°C - 45%RH, sample 1)

τ[a]	t_{air}[b]	RH[c]	m_{sys}[d]	m_{pc}[e]	M[f]	MR[g]	lnMR[h]
h	°C	%	x10^{-3} kg	x10^{-3} kg	w/w db		
0	54.0	21	0.000				
0.02	53.9	31	181.318	29.087	1.337	1.000	0.000
0.03	59.2	53	181.467	29.236	1.349	1.009	0.009
0.05	60.9	57	181.629	29.398	1.362	1.019	0.019
0.07	59.8	62	181.716	29.485	1.369	1.025	0.025
0.08	60.2	61	181.767	29.536	1.373	1.028	0.028
0.33	60.0	51	181.412	29.181	1.344	1.006	0.006
0.58	60.2	47	180.334	28.103	1.258	0.939	-0.063
0.83	60.0	45	179.281	27.050	1.173	0.873	-0.136
1.08	59.9	45	178.344	26.113	1.098	0.814	-0.205
1.33	59.9	45	177.514	25.283	1.031	0.762	-0.271
1.58	60.0	46	176.820	24.589	0.975	0.719	-0.330
1.83	59.8	45	176.121	23.89	0.919	0.675	-0.392
2.08	59.9	45	175.468	23.237	0.867	0.635	-0.455
2.33	60.1	45	174.857	22.626	0.818	0.596	-0.517
2.58	60.1	45	174.284	22.053	0.772	0.561	-0.579
2.83	60.0	45	173.744	21.513	0.728	0.527	-0.641
3.08	60.1	45	173.235	21.004	0.687	0.495	-0.703
3.33	59.9	45	172.752	20.521	0.649	0.465	-0.766
3.58	60.0	45	172.299	20.068	0.612	0.437	-0.829
3.83	60.0	45	171.872	19.641	0.578	0.410	-0.892
4.08	60.0	45	171.470	19.239	0.546	0.385	-0.955
4.33	60.1	45	171.087	18.856	0.515	0.361	-1.019
4.58	60.0	45	170.726	18.495	0.486	0.338	-1.083
4.83	60.0	46	170.444	18.213	0.463	0.321	-1.137
5.08	60.1	45	170.118	17.887	0.437	0.300	-1.202
5.33	60.1	45	169.811	17.580	0.412	0.281	-1.268
5.58	60.0	45	169.523	17.292	0.389	0.263	-1.334
5.83	60.3	45	169.252	17.021	0.367	0.246	-1.401
6.08	60.1	45	168.999	16.768	0.347	0.231	-1.467
6.33	60.0	45	168.762	16.531	0.328	0.216	-1.534
6.58	60.1	45	168.539	16.308	0.310	0.202	-1.600
6.83	59.9	45	168.327	16.096	0.293	0.189	-1.668
7.08	60.0	46	168.129	15.898	0.277	0.176	-1.736
7.33	60.3	45	167.948	15.717	0.263	0.165	-1.802

Table C.4.2 Moisture ratio for sample trial (60°C- 45%RH, sample 1) (cont'd)

τ	t_{air}	RH	m_{sys}	m_{pc}	M	MR	lnMR
h	°C	%	x10^{-3} kg	x10^{-3} kg	w/w db		
7.58	60.1	45	167.773	15.542	0.249	0.154	-1.871
7.83	60.1	45	167.608	15.377	0.235	0.144	-1.940
8.08	60.1	45	167.456	15.225	0.223	0.134	-2.008
8.33	59.8	45	167.313	15.082	0.212	0.125	-2.077
8.58	59.9	45	167.177	14.946	0.201	0.117	-2.148
8.83	60.0	45	167.051	14.820	0.191	0.109	-2.217
9.08	60.1	46	166.932	14.701	0.181	0.101	-2.288
9.33	60.0	45	166.823	14.592	0.172	0.095	-2.357
9.58	60.1	45	166.720	14.489	0.164	0.088	-2.428
9.83	60.0	45	166.622	14.391	0.156	0.082	-2.500
10.08	60.0	45	166.532	14.301	0.149	0.076	-2.571
10.33	59.9	46	166.450	14.219	0.142	0.071	-2.640
10.58	59.9	45	166.371	14.140	0.136	0.066	-2.712
10.83	60.0	45	166.299	14.068	0.130	0.062	-2.782
11.08	60.0	45	166.232	14.001	0.125	0.058	-2.852
11.33	60.0	45	166.169	13.938	0.120	0.054	-2.922
11.58	60.1	44	166.113	13.882	0.115	0.050	-2.989
11.83	60.2	45	166.061	13.830	0.111	0.047	-3.056
12.08	59.9	45	166.013	13.782	0.107	0.044	-3.122
12.33	60.1	45	165.966	13.735	0.103	0.041	-3.191
12.58	60.0	45	165.922	13.691	0.100	0.038	-3.260
12.83	60.0	45	165.885	13.654	0.097	0.036	-3.322
13.08	60.0	45	165.848	13.617	0.094	0.034	-3.388
13.33	60.0	45	165.815	13.584	0.091	0.032	-3.451
13.58	60.0	45	165.783	13.552	0.089	0.030	-3.516
13.83	60.0	45	165.757	13.526	0.087	0.028	-3.573
14.08	60.0	45	165.729	13.498	0.084	0.026	-3.637
14.33	60.1	45	165.707	13.476	0.083	0.025	-3.691
14.58	60.0	45	165.683	13.452	0.081	0.023	-3.753
14.83	60.0	45	165.662	13.431	0.079	0.022	-3.810
15.08	60.0	45	165.657	13.426	0.079	0.022	-3.824
15.33	59.9	45	165.658	13.427	0.079	0.022	-3.821
15.58	60.1	45	165.627	13.396	0.076	0.020	-3.914
15.83	59.9	45	165.606	13.375	0.074	0.019	-3.982
16.08	60.0	45	165.589	13.358	0.073	0.018	-4.041
16.33	60.0	45	165.575	13.344	0.072	0.017	-4.092
16.58	59.9	45	165.563	13.332	0.071	0.016	-4.137

Table C.4.2 Moisture ratio for sample trial (60°C - 45%RH, sample 1) (cont'd)

τ	t_{air}	RH	m_{sys}	m_{pc}	M	MR	lnMR
h	°C	%	x10^{-3} kg	x10^{-3} kg	w/w db		
16.83	60.2	45	165.550	13.319	0.070	0.015	-4.190
17.08	60.1	45	165.578	13.347	0.072	0.017	-4.080
17.33	60.0	45	165.557	13.326	0.071	0.016	-4.161
17.58	60.0	45	165.587	13.356	0.073	0.017	-4.048
17.83	60.1	45	165.562	13.331	0.071	0.016	-4.141
18.08	60.0	45	165.539	13.308	0.069	0.014	-4.236
18.33	60.1	44	165.521	13.290	0.068	0.013	-4.317
18.58	59.8	45	165.509	13.278	0.067	0.013	-4.375
18.83	60.1	45	165.496	13.265	0.066	0.012	-4.441
19.08	60.1	45	165.492	13.261	0.065	0.012	-4.463
19.33	60.0	45	165.484	13.253	0.065	0.011	-4.507
19.58	59.9	46	165.476	13.245	0.064	0.011	-4.554
19.83	60.0	45	165.469	13.238	0.063	0.010	-4.596
20.08	60.1	45	165.463	13.232	0.063	0.010	-4.634
20.33	59.9	45	165.460	13.229	0.063	0.010	-4.653
20.58	60.0	45	165.455	13.224	0.062	0.009	-4.687
20.83	60.0	45	165.450	13.219	0.062	0.009	-4.721
21.08	60.0	44	165.446	13.215	0.062	0.009	-4.750
21.33	60.1	45	165.441	13.210	0.061	0.008	-4.786
21.58	60.1	45	165.440	13.209	0.061	0.008	-4.794
21.83	60.0	45	165.435	13.204	0.061	0.008	-4.832
22.08	60.1	45	165.432	13.201	0.061	0.008	-4.856
22.33	60.1	43	165.430	13.199	0.060	0.008	-4.872
22.58	60.0	45	165.428	13.197	0.060	0.008	-4.889
22.83	60.0	45	165.424	13.193	0.060	0.007	-4.922
23.08	59.9	45	165.420	13.189	0.060	0.007	-4.957

[a] τ = drying time.
[b] t_{air} = temperature inside environmental chamber (chamber sensor).
[c] RH = relative humidity (chamber sensor).
[d] m_{sys}= mass of inert sphere and press cake system
(i.e inert sphere, hook, thermocouples, and press cake).
[e] m_{pc}= mass of press cake.
[f] M db = moisture content, mass/mass (w/w) on a dry basis (db) at time τ.
[g] MR = moisture ratio at time τ.
[h] ln MR = natural logarithm of MR at time τ.

Sample calculations for Table C.4.2: ($\tau = 0.33$ h)

1) Mass of press cake, m_{pc}:

$$m_{pc} = m_{sys} - m_{ip-sys} \tag{C.4a}$$

where $m_{ip\text{-}sys} = 152.231\text{x}10^{-3}$ kg [initial mass of inert sphere system (includes hook and thermocouples) prior to addition of press cake] and m_{sys} = mass of press cake and inert sphere system (includes hook and thermocouples).

2) Moisture contents M_o and $M_{\tau=0.33\text{h}}$, were calculated based on Eqn. 2.1 using the parameters as described in Appendix C.2.2, where $m_{pc(o)} = 29.087\text{x}10^{-3}$ kg ; $m_{pc(\tau=0.33\text{h})} = 29.181\text{x}10^{-3}$ kg ; $m_{pc(f)} = 12.448\text{x}10^{-3}$kg :

$$M_o = \frac{29.087 - 12.448}{12.448}$$

$$M_{\tau=0.33h} = \frac{29.181 - 12.448}{12.448}$$

3) Moisture ratio $MR_{\tau=0.33\text{h}}$ was calculated based on Eqn. 2.15

$$MR_{\tau=0.33h} = \frac{1.344 - 0.0505}{1.337 - 0.0505}$$

where $M_o = 1.337$, $M_{\tau=0.33\text{h}} = 1.344$, $M_e = 0.0505$ is the equilibrium moisture content as per Table C.4.3.

4) The falling rates were identified based on plots of lnMR versus τ as shown in Fig. C.4.1.

Note: For all drying conditions based on 3 samples each, a time of 13.3 h was determined as being suitable for the analysis of the first falling rate period.

Table C.4.3 Equilibrium moisture content, M_e, for experimental trials

Trial #	Temperature °C	Relative humidity (dec.)[b]	M_e[a] w/w
1	70	0.36	0.0185
2	80	0.36	0.0258
3	60	0.45	0.0505
4	70	0.45	0.0427
5	80	0.45	0.0395
6	50	0.57	0.0895
7	60	0.57	0.0534
8	70	0.57	0.0739
9	80	0.57	0.0409
10	50	0.62	0.1016
11	50	0.70	0.1256
12	60	0.70	0.1261

[a] M_e = Equilibrium moisture content, mean of duplicate samples.
[b] dec. = decimal form.

Note: M_e was calculated using Eqn. 2.1 based on drying trials described in Section 5.1.4.

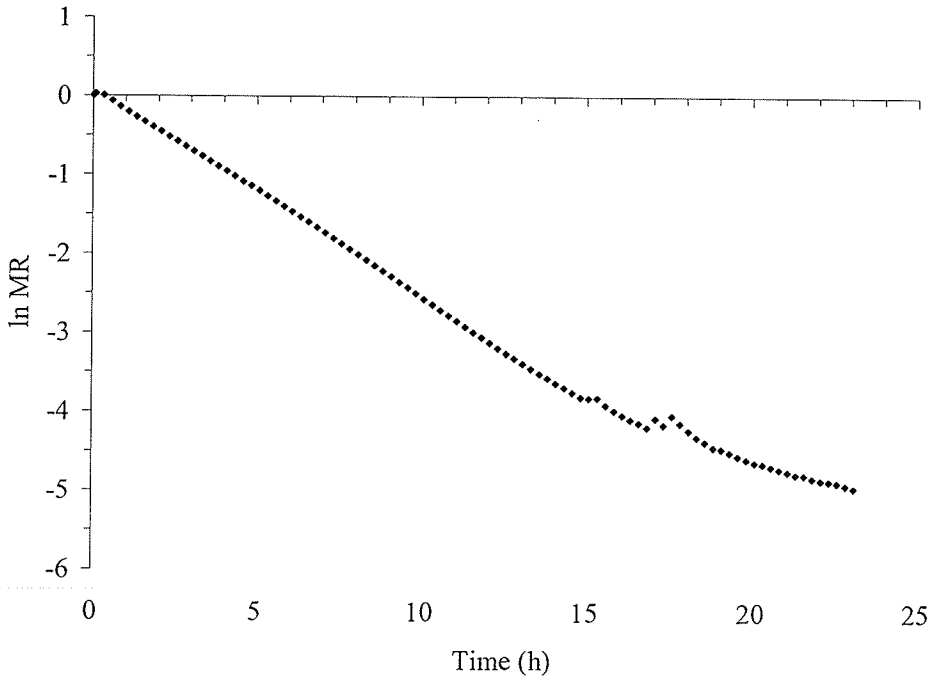

Figure C.4.1 Plot of ln*MR* for sample trial (60°C - 45%RH, sample 1).

Determination of moisture ratio model fit.

1) A summary of data from the experimental trials that was used for the determination of model coefficients is summarized in Table C.4.4.
2) The data from individual trials was fit using nonlinear regression analysis (Table C.4.5).
3) The coefficients shown in Table C.4.6 were fit across different temperatures using linear regression analysis.
4) The final coefficients based on a second linear regression analysis are provided in Eqns. 5.24-5.31.
5) The fit of predicted to experimental values for moisture ratio and the residuals for all validation trials are provided in Figures C.4.2-C4.7.

Table C.4.4 Summary of model fitting data and constraints

RH[a]	Temperature	Final moisture ratio, MR	Time		Number of data points
			Initial	Final	
dec.	°C		h	h	#
0.36	70	0.038	0.02	10.58	43
	70	0.038	0.58	9.83	39
	70	0.039	0.58	9.33	36
	80	0.039	0.02	6.33	26
	80	0.036	0.33	6.58	26
	80	0.036	0.08	5.58	23
0.45	60	0.038	0.58	12.58	49
	60	0.039	0.58	12.83	50
	60	0.039	0.83	13.33	51
	70	0.040	0.02	9.83	40
	70	0.039	0.02	9.33	38
	70	0.037	0.33	8.08	32
	80	0.039	0.33	8.08	31
	80	0.038	0.02	6.58	27
	80	0.037	0.33	6.08	24
0.57	50	0.039	1.08	25.83	100
	50	0.039	1.08	23.58	91
	50	0.039	1.33	27.08	104
	60	0.039	0.33	12.83	51
	60	0.038	0.83	14.58	55
	60	0.040	0.33	12.83	49
	70	0.038	0.33	13.08	52
	70	0.038	0.38	12.83	51
	70	0.036	0.33	11.83	47
*	80	0.077	0.33	11.08	44
*	80	0.074	0.33	9.08	36
	80	0.037	0.33	11.83	47
0.62	50	0.038	0.33	23.83	95
**	50	0.057	0.33	24.33	97
**	50	0.10	0.33	24.08	96
0.70	50	0.042	0.33	53.08	212
	50	0.038	0.58	35.33	140
	50	0.039	0.33	32.33	129
**	60	0.089	0.33	24.08	96
**	60	0.135	0.58	23.58	83
	60	0.038	0.35	21.10	84

[a] RH = relative humidity on a decimal basis (dec.); * = data removed (1st falling rate ended), and ** = insufficient data (trial was ended at ~24 h which didn't allow sufficient time to achieve MR = ~0.039).

Table C.4.5 Model coefficients based on nonlinear fitting analysis

RH[a] dec.	t_{air}[b] °C	Rep[c] #	Model coefficients							
			Lewis[d]	Page[e]		Henderson and Pabis[f]		logarithmic[g]		
			k	k	n	a	k	a	k	c
0.36	70	1	0.30282	0.24488	1.15650	1.07350	0.32479	1.08354	0.30585	-0.02156
0.36	70	2	0.32672	0.23913	1.24576	1.18979	0.38519	1.18948	0.38385	-0.00107
0.36	70	3	0.34825	0.30892	1.09761	1.08770	0.37794	1.08964	0.38832	0.00775
0.36	80	1	0.43375	0.30872	1.35380	1.12018	0.48392	1.18254	0.38852	-0.09745
0.36	80	2	0.43763	0.34427	1.24795	1.15189	0.50249	1.15922	0.45312	-0.03703
0.36	80	3	0.50214	0.36873	1.37786	1.12293	0.56074	1.18445	0.45173	-0.09564
0.45	60	1	0.23673	0.19731	1.11402	1.08350	0.25645	1.08503	0.24683	-0.01247
0.45	60	2	0.22674	0.18435	1.12640	1.08451	0.24587	1.08913	0.22913	-0.02389
0.45	60	3	0.21588	0.18024	1.10693	1.08322	0.23363	1.08361	0.22375	-0.01389
0.45	70	1	0.27970	0.20864	1.20865	1.09025	0.30502	1.12354	0.26500	-0.05755
0.45	70	2	0.28946	0.20617	1.24831	1.09895	0.31803	1.14645	0.26472	-0.07818
0.45	70	3	0.31426	0.21103	1.31475	1.17319	0.36858	1.20710	0.30397	-0.08058
0.45	80	1	0.35098	0.25737	1.26413	1.16528	0.40810	1.17145	0.37914	-0.02678
0.45	80	2	0.40405	0.28305	1.34815	1.12429	0.45296	1.19038	0.36135	-0.10207
0.45	80	3	0.42048	0.30854	1.31583	1.17384	0.49267	1.20522	0.40111	-0.08443
0.57	50	1	0.12996	0.12636	1.01261	1.02026	0.13260	1.02097	0.13810	0.01147
0.57	50	2	0.13564	0.12495	1.03779	1.03869	0.14089	1.03899	0.14479	0.00799
0.57	50	3	0.11965	0.11621	1.01262	1.01412	0.12132	1.01432	0.12199	0.00155
0.57	60	1	0.22972	0.18313	1.13985	1.08873	0.25028	1.09530	0.23610	-0.02049
0.57	60	2	0.21178	0.18329	1.08394	1.07043	0.22647	1.07028	0.22486	-0.00215
0.57	60	3	0.22802	0.18916	1.11507	1.08043	0.24672	1.08285	0.24109	-0.00795
0.57	70	1	0.20201	0.13040	1.25449	1.12759	0.22793	1.16207	0.19098	-0.07252
0.57	70	2	0.22891	0.16126	1.21736	1.13454	0.25952	1.14105	0.24414	-0.02200
0.57	70	3	0.23144	0.14471	1.29689	1.17202	0.27087	1.18769	0.24231	-0.04349
0.57	80	1	0.22525	0.15748	1.22701	1.13608	0.25702	1.14388	0.24503	-0.01881
0.57	80	2	0.26798	0.18370	1.27101	1.16195	0.31280	1.17223	0.29348	-0.02567

Table C.4.5 Model coefficients based on nonlinear fitting analysis (cont'd)

RH dec.	t_{air} °C	Rep #	Model coefficients							
			Lewis	Page		Henderson and Pabis		logarithmic		
			k	*k*	*n*	*a*	*k*	*a*	*k*	*c*
0.57	80	3	0.25812	0.16093	1.31739	1.18267	0.30360	1.18903	0.28217	-0.02657
0.62	50	1	0.11254	0.10992	1.01013	1.00788	0.11348	1.01291	0.11021	-0.01049
0.62	50	2	0.10790	0.13064	0.91811	0.97243	0.10459	0.94732	0.12518	0.05896
0.62	50	3	0.13104	0.12686	1.01471	1.01572	0.13317	1.01578	0.13310	-0.00018
0.70	50	1	0.08562	0.11474	0.88764	1.01089	0.08665	1.02763	0.11622	0.06858
0.70	50	2	0.10201	0.10025	1.00705	1.02778	0.10492	1.02595	0.11358	0.02259
0.70	50	3	0.10633	0.11428	0.97030	1.00380	0.10675	0.99922	0.11452	0.02070
0.70	60	1	0.09656	0.07938	1.08135	1.05405	0.10230	1.06214	0.09876	-0.01427
0.70	60	2	0.08277	0.06822	1.07800	1.04813	0.08746	1.05528	0.08530	-0.01111
0.70	60	3	0.12971	0.08661	1.18624	1.10849	0.14404	1.12320	0.13183	-0.03392

[a] *RH* = relative humidity of drying chamber air on a decimal basis (dec.).
[b] t_{air} = temperature of drying chamber air.
[c] Rep = replicate.
[d] Lewis = Lewis model [$MR = \exp(-k\,\tau)$]; *MR* = moisture ratio; *k* = coefficient, and τ = time (h).
[e] Page = Page model [$MR = \exp(-k\,\tau^{n})$]; *MR* = moisture ratio; *k,n* = coefficients, and τ = time (h).
[f] Henderson and Pabis = Henderson and Pabis model [$MR = a\exp(-k\,\tau)$];*MR* = moisture ratio; *a,k* = coefficients, and τ = time (h).
[g] logarithmic = logarithmic model [$MR = a\exp(-k\,\tau)+c$]; *MR* = moisture ratio; *a,k,c* = coefficients, and τ = time (h).

Table C.4.6 Model coefficients based on linear regression analysis

Model parameter	Relative humidity, dec.[g] 0.36	0.45	0.57	0.70
Lewis[a]				
$a(k)$[b]	0.01319	0.00827	0.00364	0.00050
$b(k)$[c]	-0.59744	-0.27460	-0.03071	0.07285
Page[d]				
$a(k)$	0.00763	0.00478	0.00095	-0.00317
$b(k)$	-0.26953	-0.10860	0.09348	0.26819
$a(n)$	0.01599	0.00968	0.00896	0.01602
$b(n)$	0.04723	0.54991	0.58331	0.15400
Henderson and Pabis[e]				
$a(a)$	0.00147	0.00354	0.00472	0.00561
$b(a)$	1.01431	0.87213	0.79518	0.73382
$a(k)$	0.01531	0.01039	0.00490	0.00118
$b(k)$	-0.7089	-0.37838	-0.08948	0.04031
logarithm[f]				
$a(a)$	0.00545	0.00515	0.00512	0.00626
$b(a)$	0.73927	0.78383	0.77731	0.70457
$a(k)$	0.00718	0.00736	0.00408	-0.00095
$b(k)$	-0.14314	-0.21832	-0.04785	0.16216
$a(c)$	-0.00717	-0.00272	-0.00128	-0.00571
$b(c)$	0.49727	0.13689	0.06489	0.32257

[a] Lewis = Lewis model [$MR = \exp(-k\,\tau)$]; MR = moisture ratio; k = coefficient, and τ = time (h).

[b] $a(k)$ = parameter a, the slope in the linear equation ($y = a\,x + b$); where $y = k$ in the model, x= temperature, and b=intercept.

[c] $b(k)$ = parameter b, the intercept in the linear equation ($y = a\,x + b$); where $y = k$ in the model, a = slope, x = temperature.

[d] Page = Page model [$MR = \exp(-k\,\tau^n)$]; MR = moisture ratio; k,n = coefficients; τ = time (h).

[e] Henderson and Pabis = Henderson and Pabis model [$MR = a\exp(-k\,\tau)$];MR = moisture ratio; a,k = coefficients; τ = time (h).

[f] logarithmic = logarithmic model [$MR = a\exp(-k\,\tau) + c$]; MR = moisture ratio; a,k,c = coefficients; τ = time (h).

[g] dec. = decimal basis

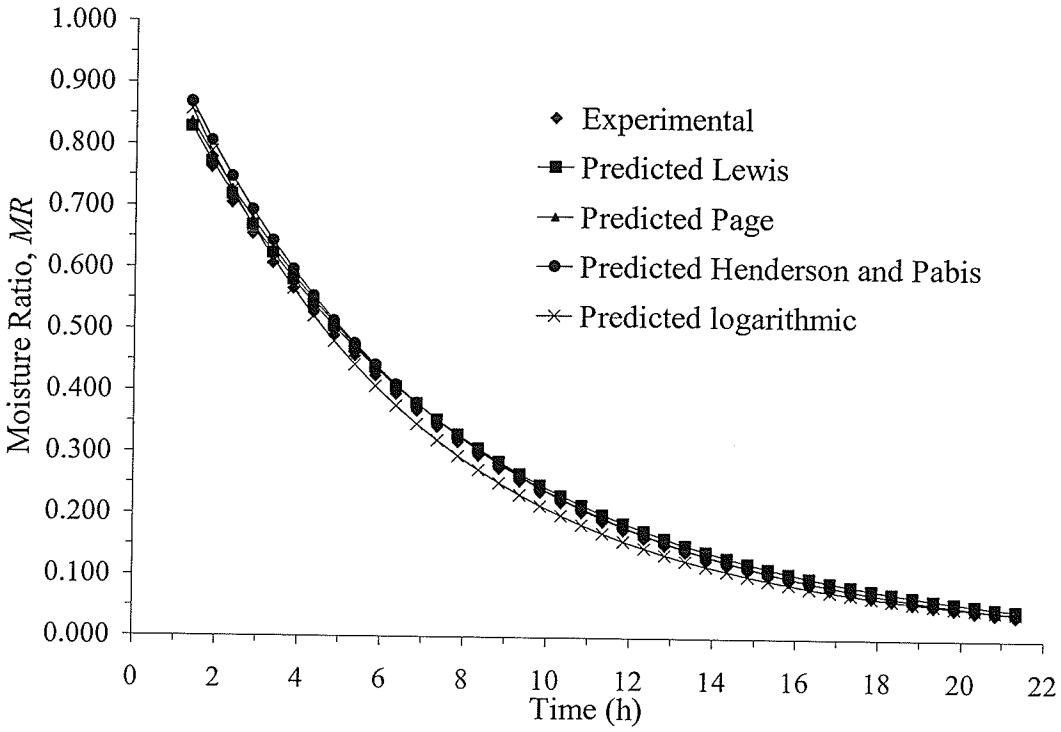

Figure C.4.2 Moisture ratio for models validated at 55°C - 55% RH.

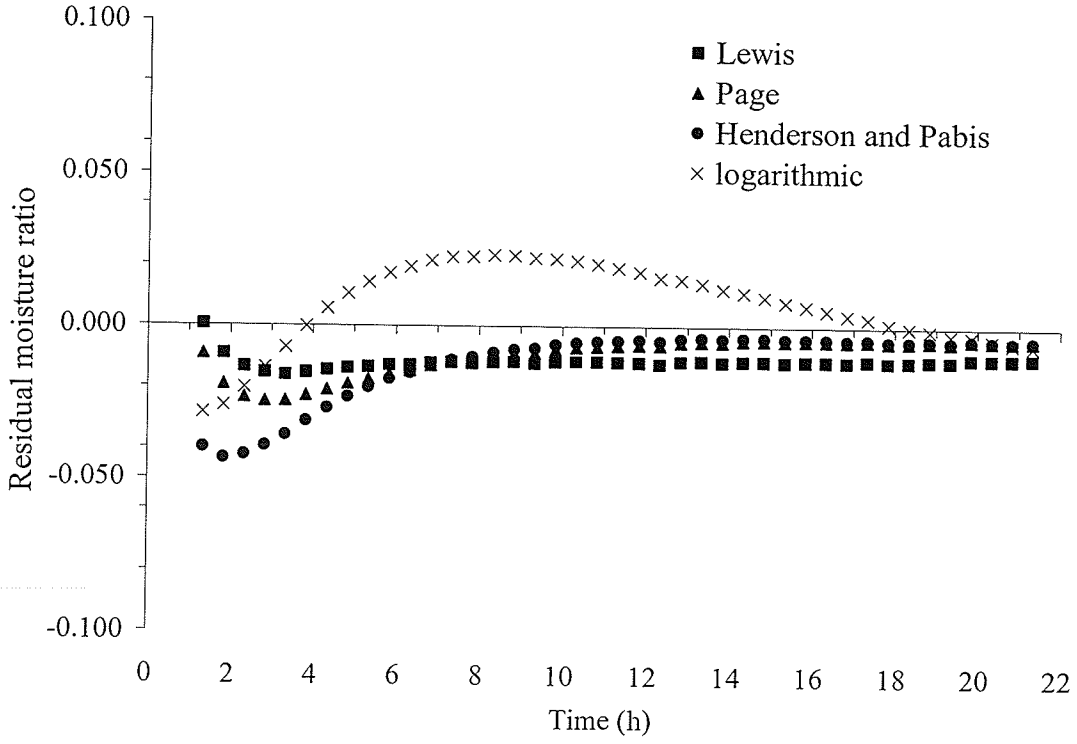

Figure C.4.3 Residual moisture ratio for models at 55°C - 55% RH.

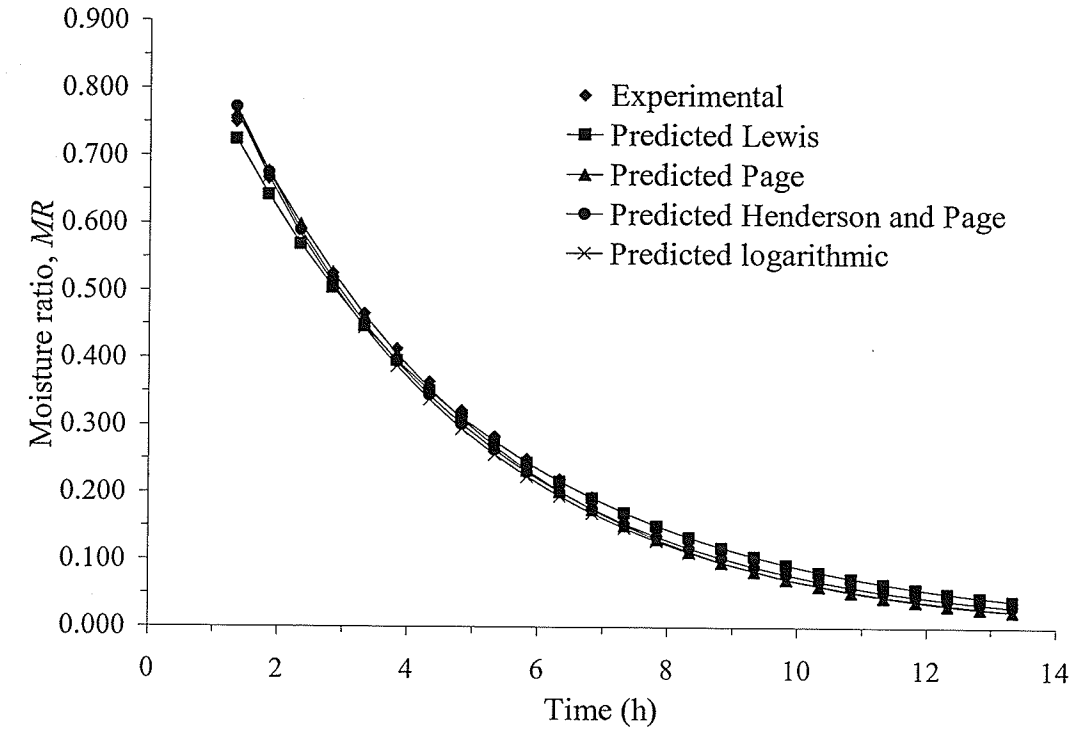

Figure C.4.4 Moisture ratio for models validated at 65°C - 45% RH.

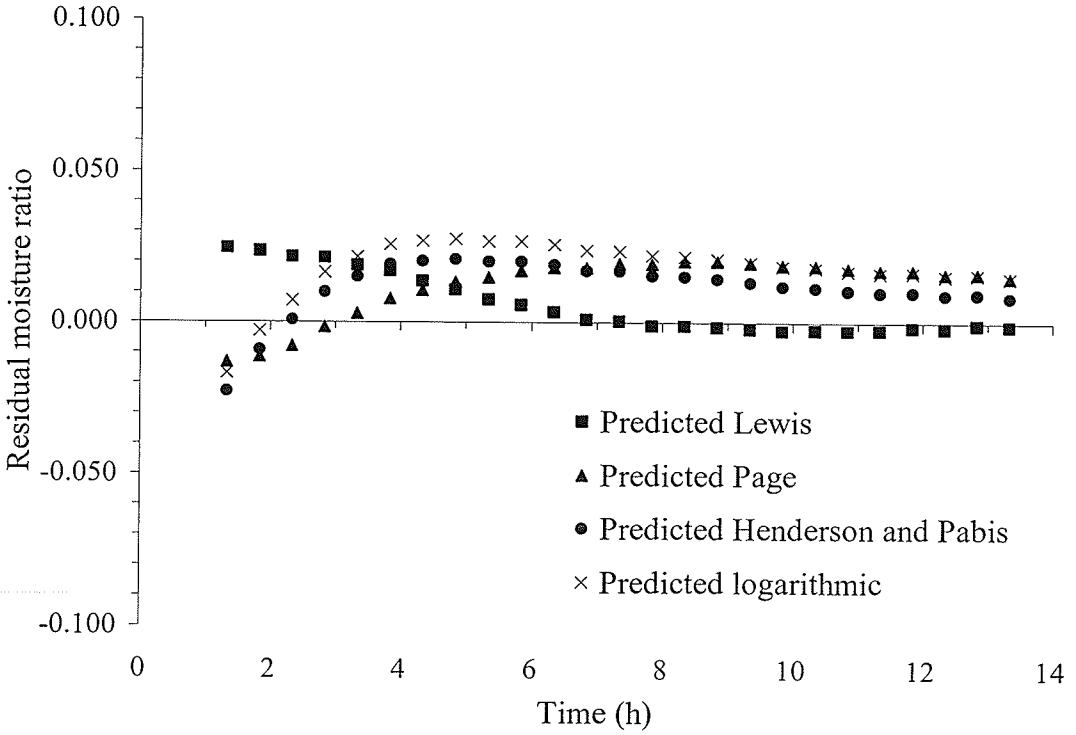

Figure C.4.5 Residual moisture ratio for models at 65°C - 45% RH.

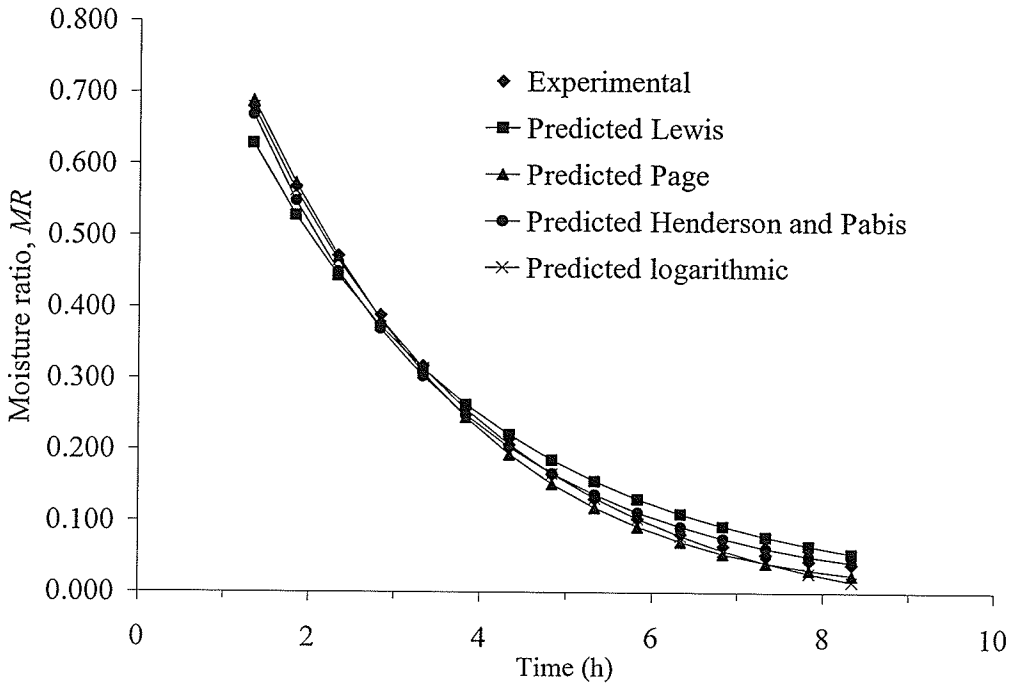

Figure C.4.6 Moisture ratio for models validated at 75°C - 43% RH.

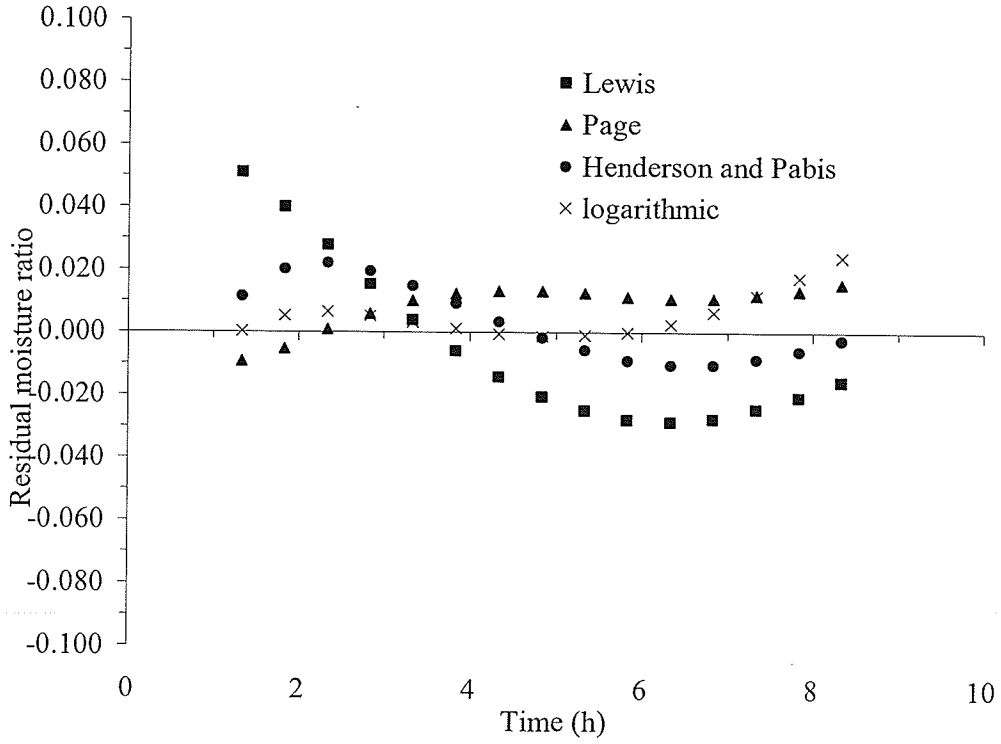

Figure C.4.7 Residual moisture ratio for 75°C - 43% RH.

Appendix C.5 Determination of specific heat capacity of press cake, $Cp_{pc(i)}$

Determination of press cake composition: (validation trial, 65°C - 45% RH)

1) The solution to Eqns. 5.19-23 requires the determination of the mass portions of the fat, $m_{pc\text{-}fat}$, water m_{pc-H_2O}, solids, $m_{pc\text{-}sol}$, and total components, $m_{pc\text{-}tot}$, of press cake. Determination of these parameters is based on an analysis of fresh berries at M = 75.82% wb. Referring to Table C.5.1 (Tables C.5.2-3 for 55°C - 55% RH and 75°C - 43% RH), the measured data is as follows: $m_{berries}$ = mass of fresh berries; m_{seeds} = mass of seed portion of fresh berries; $m_{seed\ oil}$ = mass of oil in three combined seed samples, and $m_{pulp/peel\ oil}$ = mass of pulp and peel oil in the pulp and peel portion of dried press cake.

2) The values for $m_{pulp/\text{peel}}$, m_{pc-H_2O}, and m_{pc} are calculated from the parameters mentioned above:

$$m_{pulp/peel} = m_{berries} - \left(m_{berries-H_2O} + m_{seeds}\right) \tag{C.5a}$$

where $m_{pulp/peel}$ = the mass of the pulp and peel in the fresh berry and press cake samples and $m_{berries-H_2O}$ = the mass of moisture in fresh berries based on an approximate moisture content of 75.82% wb;

$$m_{pc-H_2O} = \frac{\left(m_{pulp/peel} + m_{seeds}\right)}{\left(\frac{1}{M_{pc}} - 1\right)} \tag{C.5b}$$

where m_{pc-H_2O} = the mass of the water in the press cake based on 56.78% wb moisture content, and M_{pc} = the moisture content wb of the press cake in decimal form.

$$m_{pc} = \left(m_{pulp/peel} + m_{seeds} + m_{pc-H_2O}\right) \tag{C.5c}$$

where m_{pc} = equivalent mass of the press cake based on fresh berry components.

3) Based on the results in Table C.5.1, average ratios for the following terms can be determined: $\frac{m_{pulp/peel}}{m_{pc}} = 0.3138$, $\frac{m_{seeds}}{m_{pc}} = 0.1184$, $\frac{m_{H_2O}}{m_{pc}} = 0.5678$,

$\frac{m_{pulp/peel\,oil}}{m_{pulp/peel}} = 0.0806$, and $\frac{m_{seed\,oil}}{m_{seeds}} = 0.1062$.

4) Using the ratios in the previous step, factors can be developed to enable the determination of the initial mass of the separate components (i.e. fat, solids, and water) in the press cake of mass $m_{pc(o)}$:

$$F_{pc-fat} = \frac{m_{pulp/peel\,oil}}{m_{pulp/peel}} \frac{m_{pulp/peel}}{m_{pc}} + \frac{m_{seed\,oil}}{m_{seeds}} \frac{m_{seeds}}{m_{pc}} \tag{C.5d}$$

$$m_{pc-fat} = F_{pc-fat}\, m_{pc(o)} \tag{C.5e}$$

$$F_{pc-sol} = \left(1 - \frac{m_{pulp/peel\,oil}}{m_{pulp/peel}}\right) \frac{m_{pulp/peel}}{m_{pc}} + \left(1 - \frac{m_{seed\,oil}}{m_{seeds}}\right) \frac{m_{seeds}}{m_{pc}} \tag{C.5f}$$

$$m_{pc-sol} = F_{pc-sol}\, m_{pc(o)} \tag{C.5g}$$

$$F_{pc-H_2O} = \frac{m_{H_2O}}{m_{pc}} \tag{C.5h}$$

$$m_{pc-H_2O} = F_{pc-H_2O}\, m_{pc(o)} \tag{C.5i}$$

where $F_{pc\text{-}fat}$ = factor for the determination of the fat portion in the press cake; $F_{pc\text{-}sol}$ = factor for the determination of the solids portion in the press cake, and

F_{pc-H_2O} = factor for the determination of the water portion in the press cake. The values for $m_{pc\text{-}fat}$, $m_{pc\text{-}sol}$, *and* m_{pc-H_2O} are provided in Table 5.5. It was assumed that no losses of solids and fat occur, therefore $m_{pc\text{-}fat}$ and $m_{pc\text{-}sol}$ do not change during the drying period.

Table C.5.1 Press cake components for fresh berry samples 65°C-45%RH

$m_{berries}$[a] x10^{-3} kg	m_{pc}[b] x10^{-3} kg	m_{pc-H_2O}[c] x10^{-3} kg	m_{seeds}[d] x10^{-3} kg	$m_{seed\ oil}$[e] x10^{-3} kg	$m_{pulp/peel}$[f] x10^{-3} kg	$m_{pulp/peel\ oil}$[g] x10^{-3} kg
56.05	31.35	17.80	3.47		10.08	0.8215
56.00	31.33	17.79	3.65	1.1063	9.89	0.8416
56.01	31.33	17.79	3.59		9.95	0.7244
56.00	31.33	17.79	3.72		9.82	0.8514
56.04	31.35	17.80	3.81	1.2780	9.74	0.7753
56.00	31.33	17.79	3.84		9.70	0.7479
56.04	31.35	17.80	3.61		9.94	0.7932
56.02	31.35	17.80	3.64	1.1660	9.91	0.8433
56.06	31.38	17.82	4.07		9.49	0.7374

[a] $m_{berries}$ = mass of fresh berry sample at M = 75.82% wet basis (wb).
[b] m_{pc} = equivalent press cake mass ($m_{berries}$ reduced to M = 56.78% wb).
[c] m_{pc-H_2O} = initial mass of water in press cake.
[d] m_{seeds} = mass of seeds in fresh berry sample.
[e] $m_{seed\ oil}$ = mass of seed oil in three seed samples of fresh berries.
[f] $m_{pulp/peel}$ = mass of pulp and peel in the fresh berry sample.
[g] $m_{pulp/peel\ oil}$ = mass of pulp and peel oil in pulp and peel.

Table C.5.2 Press cake components for fresh berry samples 55°C-55%RH

$m_{berries}$[a] x10^{-3} kg	m_{pc}[b] x10^{-3} kg	m_{pc-H_2O}[c] x10^{-3} kg	m_{seeds}[d] x10^{-3} kg	$m_{seed\ oil}$[e] x10^{-3} kg	$m_{pulp/peel}$[f] x10^{-3} kg	$m_{pulp/peel\ oil}$[g] x10^{-3} kg
56.05	31.67	18.12	3.47		10.08	0.8215
56.00	31.64	18.10	3.65	1.1063	9.89	0.8416
56.01	31.64	18.10	3.59		9.95	0.7244
56.00	31.64	18.10	3.72		9.82	0.8514
56.04	31.67	18.12	3.81	1.2780	9.74	0.7753
56.00	31.64	18.10	3.84		9.70	0.7479
56.04	31.67	18.12	3.61		9.94	0.7932
56.02	31.67	18.12	3.64	1.1660	9.91	0.8433
56.06	31.69	18.13	4.07		9.49	0.7374

[a] $m_{berries}$ = mass of fresh berry sample at M = 75.82% wet basis (wb).
[b] m_{pc} = equivalent press cake mass ($m_{berries}$ reduced to M = 57.21% wb).
[c] m_{pc-H_2O} = initial mass of water in press cake.
[d] m_{seeds} = mass of seeds in fresh berry sample.
[e] $m_{seed\ oil}$ = mass of seed oil in three seed samples of fresh berries.
[f] $m_{pulp/peel}$ = mass of pulp and peel in the fresh berry sample.
[g] $m_{pulp/peel\ oil}$ = mass of pulp and peel oil in pulp and peel.

Table C.5.3 Press cake components for fresh berry samples 75°C-43%RH

$m_{berries}$[a] x10^{-3} kg	m_{pc}[b] x10^{-3} kg	m_{pc-H_2O}[c] x10^{-3} kg	m_{seeds}[d] x10^{-3} kg	$m_{seed\ oil}$[e] x10^{-3} kg	$m_{pulp/peel}$[f] x10^{-3} kg	$m_{pulp/peel\ oil}$[g] x10^{-3} kg
56.05	32.71	19.16	3.47		10.08	0.8215
56.00	32.68	19.14	3.65	1.1063	9.89	0.8416
56.01	32.68	19.14	3.59		9.95	0.7244
56.00	32.68	19.14	3.72		9.82	0.8514
56.04	32.71	19.16	3.81	1.2780	9.74	0.7753
56.00	32.68	19.14	3.84		9.70	0.7479
56.04	32.71	19.16	3.61		9.94	0.7932
56.02	32.71	19.16	3.64	1.1660	9.91	0.8433
56.06	32.73	19.17	4.07		9.49	0.7374

[a] $m_{berries}$ = mass of fresh berry sample at M = 75.82% wet basis (wb).
[b] m_{pc} = equivalent press cake mass ($m_{berries}$ reduced to M = 58.57% wb).
[c] m_{pc-H_2O} = initial mass of water in press cake.
[d] m_{seeds} = mass of seeds in fresh berry sample.
[e] $m_{seed\ oil}$ = mass of seed oil in three seed samples of fresh berries.
[f] $m_{pulp/peel}$ = mass of pulp and peel in the fresh berry sample.
[g] $m_{pulp/peel\ oil}$ = mass of pulp and peel oil in pulp and peel.

Appendix C.6 Press cake temperature model (heat transfer coefficient, h, using Eqn. 5.10)

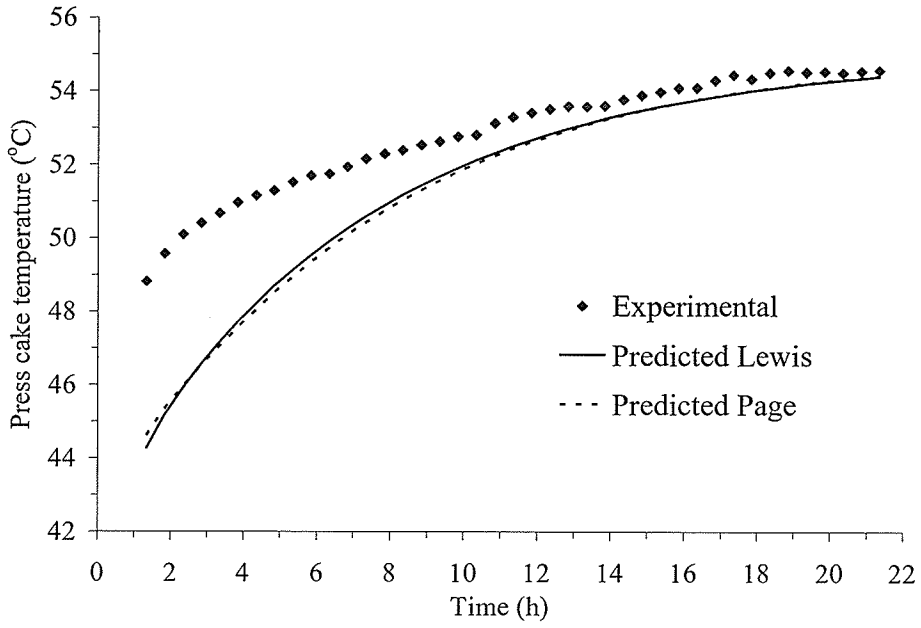

Figure C.6.1 Press cake temperature for 55°C - 55% RH trial : h=15.8 W/(m^2·K).

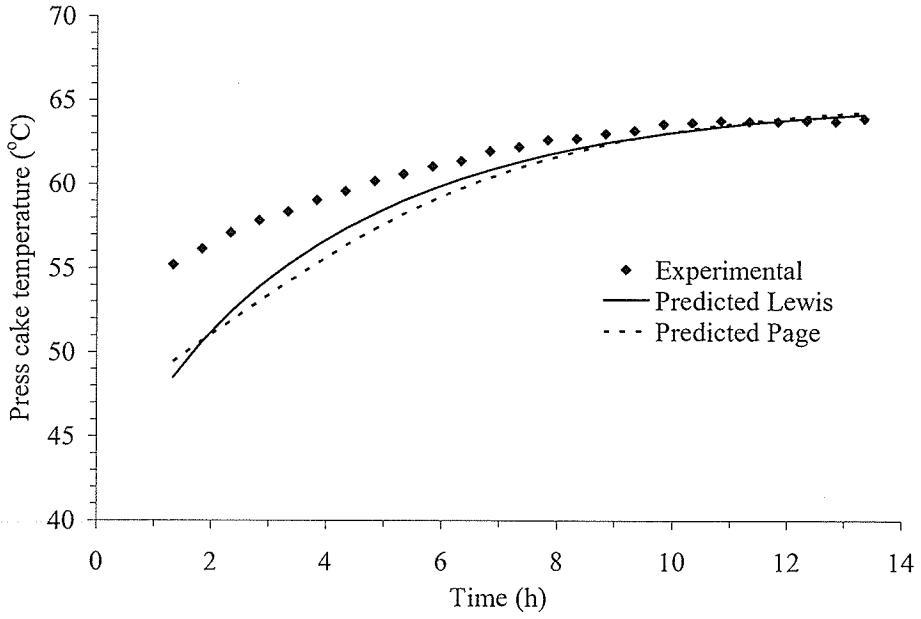

Figure C.6.2 Press cake temperature for 65°C - 45% RH trial : h=15.7 W/(m^2·K).

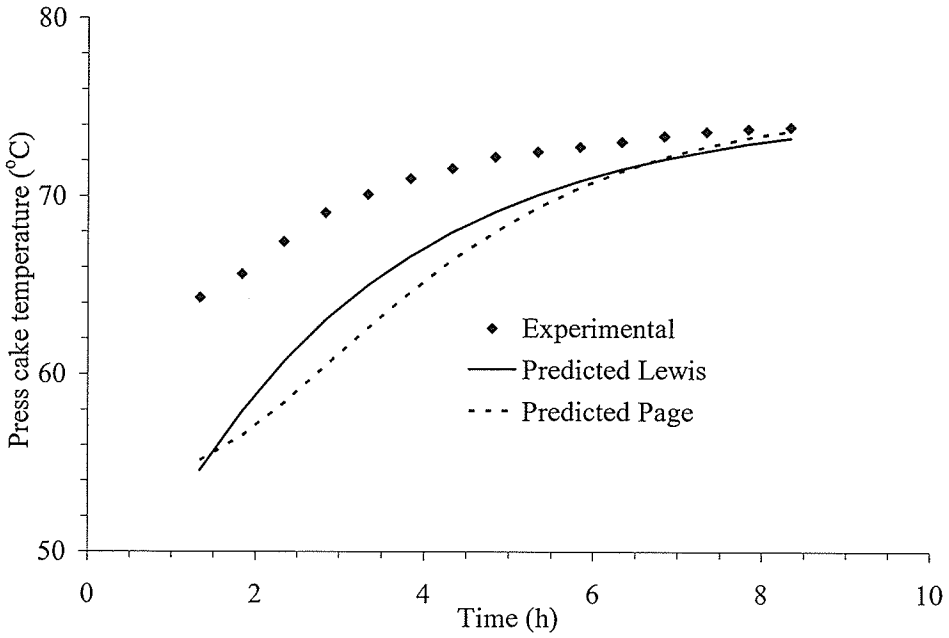

Figure C.6.3 Press cake temperature for 75°C - 43% RH trial: h=15.7 W/(m^2·K).