OSMOTIC - AIR DEHYDRATION OF CHERRIES AND BLUEBERRIES

By

Liping Yu

A thesis
presented to the University of Manitoba
in partial fulfillment of the
requirements for the degree of

MASTER OF SCIENCE

Department of Biosystems Engineering
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Winnipeg, Manitoba

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OSMOTIC-AIR DEHYDRATION OF CHERRIES AND BLUEBERRIES

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LIPING YU

A Thesis/Practicum submitted to the Faculty of Graduate Studies of The University of Manitoba in partial fulfillment of the requirements of the degree of

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ABSTRACT

Osmotic dehydration (OD) is a process of water removal from a high-moisture solid food by osmotic pressure created by a surrounding liquid medium. Air dehydration (AD) or air drying of solid foods involves vaporization of water and removal of the vapor with a stream of air. Major problems associated with air dehydration are poor product quality; considerable shrinkage caused by collapsed cells following the loss of water; poor rehydration of dried product; and unfavorable changes in color, texture, flavor, and nutritive value of dried products. Osmotic dehydration alone or in combination with air drying could improve texture and prevent much of the heat damage to color and flavor commonly associated with air drying.

Four skin treatments: steam blanching, hot water dipping, and ethyl myristate and lipase treatments were used to improve the mass transfer during osmotic dehydration of cherries and blueberries. Steaming (137.9 KPa) for 30 s was found a simple and effective method for improving the mass transfer during osmotic dehydration of waxy fruits.

Mass transfer phenomenon during air drying was investigated at 40, 60, and 80 °C in a lab oven dryer which has a fixed air flow velocity of 2.0 m/s. Osmotic pretreatment decreased moisture content of fresh cherries from 3.4 to 2.0 g/g DM and blueberries from 5.3 to 4.9 g/g DM, respectively. Initial drying rate in fresh samples directly subjected to air drying was higher than that of samples subjected to osmotic dehydration pretreatment, however, the two rates of drying gradually merged. There was no constant rate period observed at air drying temperatures of 60 and 80 °C. Steam blanching prior to osmotic
dehydration increased the subsequent air drying rate significantly. Drying rates were significantly higher at the higher air temperatures of 60 and 80 °C.

Anthocyanin contents decreased during the air drying and the decrease was higher for blueberries than for cherries. Osmotic pre-drying reduced the loss of anthocyanins at high drying temperatures. Osmotic dehydration pretreatment reduced shrinkage of cherries and blueberries during subsequent air drying as a result of solids gain during the pretreatment. Product shrinkage was linearly related to its water content. Because of sugar uptake during osmotic pretreatment in dried fruits, water uptakes during rehydration of osmotically dehydrated fruits were lower than that of directly air dried samples.

Recovery of anthocyanins was investigated using Amberlite® XAD-2, XAD-4, CG-50, XAD7HP, XAD16HP, and Duolite® XAD765 resins. Amberlite® XAD16HP was the most effective anthocyanins adsorbent-desorbent product tested. Adsorption of anthocyanins to resins was found inversely proportional to the sucrose concentration of the solution. Optimum conditions for recovery of anthocyanins from sucrose solution (after osmotic dehydration) were as follows: resin immersed for 30 min in sucrose solution which contained anthocyanin pigments, then washed with water containing 2.5 % acetic acid and eluted by 90 % ethanol + 5 % acetic acid. The resin was re-equilibrated with aqueous 2.5 % acetic acid.

Computer models were developed using finite elements to predict moisture content during osmotic dehydration and air drying of cherries and blueberries. The developed osmotic dehydration models can be used to analyze transient diffusion in 2-D.
axisymmetric bodies with different boundary conditions. The air drying models have the potential of explaining complicated coupled phenomena of heat and mass transfers in biomaterials with shrinkage, different geometries, and varying boundary conditions.
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LIST OF SYMBOLS

A  area of the triangular element

$A_{\lambda_{\text{max}}}, A_{700\text{nm}}$  Absorbances of total, at wavelength of maximum absorption, and 
at 700nm, respectively

a, b  constants

A, B, C  constants

$a_w$  water activity

$b_i, b_{ij}$  regression coefficients, $i, j = 1, 2, 3$

C  concentration of anthocyanins, mg/L

c  specific heat, J·kg$^{-1}$·K$^{-1}$

D  effective diffusion coefficient, m$^2$/s

dm  the mass of an element, g

$E_a$  Energy of activation for diffusion, J/mol

EMC  equilibrium moisture content, g H$_2$O/100 g DM

ERH  equilibrium relative humidity, %

F  dilution factor

h  surface heat transfer coefficient, W·m$^{-2}$·K$^{-1}$

$h_m$  surface mass transfer coefficient, m/s

$k'$  thermal conductivity, W·m$^{-1}$·K$^{-1}$

L  latent heat of vaporization of water, J/kg

$L$  the path length, most spectrophotometer cells $L=1$ cm

$L_{ij}$  the length of the side with convection heating boundary condition, m
M  moisture content on dry basis, g H₂O/100 g DM
M  moisture content of sample at time t on wet basis, %
M₀  initial moisture content of sample on wet basis, %
MR  mass reduction, %
MW  molecular weights
P  mean relative percent error
R  gas constant, 8.314 J·mol⁻¹·K⁻¹
R  radius, for cherries R = r₁ - r₂, r₁ = average cherry radius, r₂ = average stone radius, for blueberries R = average blueberry radius, m
r, z  radial and vertical coordinate directions
r₀, z₀  initial radial and vertical coordinate value
Rᵢ, Rⱼ, Rₖ  r direction coordinates at nodes i, j, k
S  surface with convection boundary
SEM  standard error of moisture content
SG  solids gain of sample on an initial dry matter basis, g/g DM
T  temperature, °C
T, Tᵢ, Tᵢ₊₁  temperature, K
{T}  nodal temperature, K
T₀  ambient temperature, K
T₀  initial temperature, K
t  time, s
V  body volume, m³
$V_o$ initial body volume, m$^3$

$W$ mass of sample at time $t$, g

$WC$ water content of sample on a dry matter basis, g/g DM

$WL$ water loss of sample on an initial dry matter basis, g/g DM

$WL_{eq}$ equilibrium water loss on an initial dry matter basis, g/g DM

$W_o$ initial mass of sample, g

$X, X_i, X_{i+1}$ moisture content, g H$_2$O/g DM

$dX/dt$ air drying rate, g/g DM$\cdot$h$^{-1}$

$X_a$ ambient moisture content, g H$_2$O/g DM

$X_o$ initial moisture content, g H$_2$O/g DM

{$X$} nodal moisture content, g H$_2$O/g DM

$X_i, X_j$ the independent variables:

$X_1$ = temperature (°C), $X_2$ = concentration of solution (°Brix), and $X_3$ = time (h).

$Y$ the dependent variable

$Z_i, Z_j, Z_k$ z direction coordinates at nodes $i, j, k$

$\rho$ density, kg/m$^3$

$\varepsilon$ molar absorbance
1. INTRODUCTION

1.1 Osmotic and air dehydration

1.1.1 Osmotic dehydration

Osmotic dehydration (OD) is a process of water removal from a high moisture food by osmotic pressure created by a surrounding liquid medium (Lerici et al. 1985). The process involves two major simultaneous flows: (i) water in the food material flows out to the surrounding solution; and (ii) the solute of the solution diffuses into the food material. The flux of water leaving the food is much larger than the counter-current flux of solute because of the semi-permeable nature of the food material cell structure. There are also some food constituents such as organic acids, minerals, and pigments that leach out of the food material. The extent of leaching is usually negligible; however, it may affect the composition of some products (Raoult-Wack 1994).

Because of the moderate operating conditions, osmotic dehydration prevents much of the heat damage to color and flavor commonly associated with conventional drying (Ponting et al. 1966; Collignan et al. 1992). It also helps preserve heat-labile food components such as pigments during drying and storage (Kim 1990, cited by Torreggiani 1993). A high concentration of sugar surrounding food pieces prevents discoloration of the food by enzyme oxidative browning. Therefore a good color can be obtained in the dried products without chemical treatment such as use of sulfur dioxide to preserve color in raisins. Shrinkage of food material due to dehydration is also small (Collignan et al.)
1992). Because water is removed without a phase change during osmotic dehydration, substantial energy savings can be achieved when compared to conventional dehydration processes. This is another major advantage of OD.

In fact, osmotic dehydration is now generally considered as a pretreatment to be introduced to any conventional fruit and vegetable processing operations to improve product quality and reduce energy consumption. Maltini and Torreggiani (1981) used OD to obtain high water-activity fruit products ($a_w$ 0.94-0.97) which, after vacuum packaging and pasteurization, remained stable for many months at ambient temperature. This process causes minimal change to the sensory and physical-chemical characteristics of fruits.

Despite the work as reviewed in OD (section 2.3.1), industrial development of osmotic dehydration processes has been hindered by the poor understanding and control of the two major mass transfer (water loss and solid gain) phenomena.

1.1.2 Air dehydration

Air dehydration (AD) or air drying of solid foods involves vaporization of water and removal of the vapor with a stream of air. This is a process which involves simultaneous heat and mass transfer. Heat is transported to the drying food material by convection, conduction, and radiation from the surroundings. Mass transfer from the drying material occurs by diffusion and convection via the local environment in the dryer to the surroundings. Because air drying is simple and well-developed and in many cases,
most economical, most vegetables and fruits are still air dried even though other drying techniques are available.

Major problems with air dehydration are deteriorative effects on product quality: considerable shrinkage caused by collapsed plant cells as a result of water loss, poor rehydration of the dried product, and unfavorable changes in color, texture, flavor and nutritive value caused by heat and oxygen during drying. Other works (Mazza 1983; Lerici et al. 1985; Sankat et al. 1996) have used osmotic dehydration as a pretreatment prior to air drying to improve the quality of air dried products.

1.2 Objectives

The objectives of the research presented in this thesis were:

1. To obtain data to establish the moisture sorption isotherms for cherries and blueberries and to evaluate the suitability of five commonly used three-parameter equations for the description of the equilibrium moisture content data of cherries and blueberries.

2. To investigate the effect of the wax layer of cherry skin on osmotic dehydration, and methods for improving water movement through this barrier.

3. To study the influence of osmotic dehydration on the subsequent rate of air drying and resulting quality of dried cherry and blueberry products.

4. To determine the loss of cherry and blueberry anthocyanins during osmotic dehydration.
5. To investigate the possibility of recovering these pigments from osmotic dehydration media.

6. To use finite element analysis to establish mass transfer models for both osmotic and air dehydration of cherries and blueberries.
2. LITERATURE REVIEW

2.1 Problem Definition

Osmotic dehydration is a process often used as a pre-dehydration step in commercial practice but is finding more and more applications as an independent process to produce ready-to-use and semi-processed products. This literature review summarizes research work on: 1) osmotic dehydration of fruits and vegetables, and its effect on air drying behavior of food, 2) anthocyanin determination and methods for recovering the pigment from various plant sources, and 3) numerical modeling of mass transfer phenomenon in solids and simultaneous heat and mass transfer during air drying of solid foods.

2.2 Moisture sorption

Sorption isotherms show the dependence of water activity on water content of food at definite temperatures and pressures. Dried foods are microbiologically stable when their water activity ($a_w$) is reduced below 0.6 (Hayes 1987). Equilibrium moisture content (EMC) - equilibrium relative humidity (ERH) relationships are necessary to optimize storage, handling, and processing of foods. The static method of using saturated salt solution to maintain constant vapor pressure has been commonly used to obtain equilibrium moisture content data at constant temperature (Jayas and Mazza 1993; Maroulis et al. 1988; Saravacos et al. 1986). The relationship between $a_w$ and moisture content is described by a moisture sorption isotherm. The five equations commonly used
to describe the moisture sorption isotherm of biological materials are modified Henderson, modified Chung-Pfost, modified Halsey, modified Oswin, and modified GAB equations (Jayas and Mazza 1993). Among them the GAB equation has been applied successfully to predict the \( a_w \) values for fruits (Lim et al. 1995; Maroulis et al. 1988; Saravacos et al. 1986; Tsami et al. 1990), vegetables (Zhang et al. 1996), grains (Jayas and Mazza 1993), and oilseeds (Mazza and Jayas 1991).

### 2.3 Heat and mass transfer

#### 2.3.1 Osmotic dehydration

Since osmotic dehydration can only remove part of the natural water of fruits and vegetables, most studies have focused on rapid and effective removal of desired amount of water. The process variables studied include pretreatment of the biological material, dehydration temperature (Beristain et al. 1990), composition and concentration of the solution (Lerici et al. 1985; Palou et al. 1993; Parjoko et al. 1996), solute molecular weight (Saurel et al. 1994), and processing time (Beristain et al. 1990; Saurel et al. 1994).

Much research has been conducted on various fruits and vegetables, such as apples, peaches, apricots, pineapples, potatoes (Saurel et al. 1994; Maltini et al. 1993; Beristain et al. 1990; Parjoko et al. 1996; Islam and Flink 1982; Kowalska and Lenart 1994; Yang et al. 1987; Yang and LeMaguer 1992; Palou et al. 1993; Rastogi and Raghavarao 1995). Most of these studies have been concerned with the kinetics of dehydration and mass transfer phenomena during the osmotic dehydration process, and
dehydration models have been developed based on Fick's second law to fit experimental data (Beristain et al. 1990; Azuara et al. 1992).

Saurel et al. (1994) used response surface methodology to estimate the effects of solute concentration, temperature, solute molecular weight, and processing time on mass transfer during osmotic dehydration of apples. A polynomial model was used for statistical analysis. It was shown that solute molecular weight was the dominating factor influencing solute uptake. Increasing solute molecular weight increased water loss except for processes utilizing low temperature (T < 50 °C) and short processing time (t < 30 min). Lenart and Lewicki (1990) stated that temperature strongly affects equilibrium water content and solids gain by the carrot tissue. Increasing osmotic temperature resulted in the increase of both water removal and solids gain.

2.3.2 Skin treatment of fruits

A major barrier responsible for the low rate of dehydration of whole fruit is believed to be the skin. Many researchers (Riva and Masi 1990; Ponting and McBean 1970; Raev et al. 1984) have investigated methods for improving mass transfer through fruit skin. The methods usually required weakening or destruction of the waxy layer on the skin.

Tanchev et al. (1983) studied the composition of wax layer of some fruits. They found that the wax of fresh fruit consisted of 69.3 % triterpenoids and 30.7 % petroleum ether soluble substances. The amount of wax on ripe cherries of Lambert cultivar was
13.91 g/m². After blanching in water for 30 s, 2% ethyl oleate, 2% ethyl oleate + 3% K₂CO₃, and immersed in pure ethyl oleate for 5 min, the amount of wax decreased to 11.12, 8.9, 7.4, and 8.48 g/m², respectively. The ratio of triterpenoids to petroleum ether soluble substances changed from 69.3/30.7 to 66.9/33.1, 10.1/89.9, 7.5/92.5 and 25.1/74.9, respectively. After blanching in 2% ethyl oleate solution, the ratio was 1.6/98.4 hard-to-soft wax in the petroleum ether soluble fraction of fruit as compared to 18.0/82.0 prior to blanching.

Ponting and McBean (1970) concluded that the most effective dipping materials for increasing drying rate by air were the ethyl esters of fatty acids in the C₁₀ - C₁₈ range. Ethyl oleate was the most convenient to handle and effective alone or in a mixture with potassium or sodium carbonate or other alkali. Oleyl alcohol was also found effective. The optimum concentration in the dip appears to be about 1% for cherries (Ponting and McBean 1970). Dipping times from 10 s to 3 min resulted in approximately the same drying rate. Dipping in a cold aqueous emulsion of ethyl oleate increased the drying rate two-fold or more over that of the water-dipped control. McBean et al. (1971) indicated that it was a general practice to dip prunes in a boiling 0.1 - 0.3% sodium hydroxide solution before drying. This treatment removes about half the total wax, leaving residual wax in an unorganized form and producing small cracks or "checks" in the cuticle. Aguilera and Stanley (1990) stated that the skin of grapes offered considerable external resistance to mass transfer during drying and significantly higher drying rates were obtained if the skin was removed. Treatment with petroleum ether dissolved the waxy layer while NaOH induced microscopic cracks in the cuticle. They listed the moisture
diffusion coefficients (D) during air-drying of grapes at 50 °C using different pretreatments (dipping in NaOH, dipping in ethyloleate, peeling the skin), and found pretreatments could increase the D value by 4-20 times when moisture content was high and by 2-5 times when moisture content was low.

Szymczak and Plocharski (1994) tried to loosen the mass transfer barrier in prunes by using liquefying enzymes and sodium hydroxide to remove wax layer and soften fruit tissue structure. However, it was found that the pectolytic enzymes usually used for treating fruit mash for higher juice yield were not effective for increasing mass transfer in osmotic dehydration.

2.3.3 Effects of osmotic dehydration on air drying

Generally, osmotic dehydration process itself does not produce stable products. Mostly, it is used as a pre-dehydration step for air drying in commercial practice.

2.3.3.1 Drying curves and rates

Mazza (1983) reported that as the concentration of sucrose for dipping carrot slices increased from 5 to 60 %; the subsequent air drying rate of carrots decreased substantially. It was concluded that the lower rates of moisture transport can be attributed to (1) sucrose that crystallizes during air drying lowers the diffusivity of water vapor and impairs heat transfer within the product and (2) the vapor pressure of water in the product is depressed due to dissolved sugar. Biswal et al. (1993) showed that there was no evidence of a constant drying rate for all samples and the mean air drying rate of
osmotically dehydrated sweet potato is much lower than that of the fresh ones. Sankat et al. (1996) found that untreated (fresh, 15 °Brix soluble solids) or lightly osmosed (26 °Brix soluble solids) banana slices had higher drying rates compared to those with the higher sugar contents (34 or 39 °Brix soluble solids). They also stated that air drying of both fresh and osmosed banana slabs occurred in the falling rate period. If a constant rate period does exist, it is very short and insignificant. Lenart and Cerkowniak (1996) found that osmotic dehydration of apples to moisture contents of 7.5, 5, and 3 g H₂O/g dry matter decreased the convection drying rate by 5-10, 35-40, and 55-60 % respectively, compared to the raw apple drying rate. An increase in the temperature of osmotic dehydration from 30 to 80 °C increased the convection drying rate by 20-30 % (Lenart and Cerkowniak 1996).

Collignan et al. (1992) stated that no significant difference was noticed between drying of fresh and osmotic dehydrated fruits in terms of drying periods and energy consumption per unit of evaporated water. But when energy consumption is considered in terms of output weight, less energy is required (2.4 to 7.1 times) for osmotic dehydrated fruit than for drying fresh fruit.

2.3.3.2 Moisture diffusion coefficient

Islam and Flink (1982) showed that relative diffusion coefficients of water for potato slices dried at air temperature of 52 - 68 °C in the first falling rate period were as follows: no osmosis > 60% sucrose > 45% sucrose / 15% salt. They suggested that uptake of sugar and salt increased internal resistance to moisture movement. Sankat et al.
reported that the apparent moisture diffusivity at 60 °C ranged from $3.48 \times 10^{-10}$ m$^2$/s for fresh banana slabs to $8.8 \times 10^{-10}$ m$^2$/s for dried (39 °Brix) slabs. The moisture diffusivity was significantly lowered with reduced moisture content during drying as well as with increased sugar level.

2.4 Product quality

Osmotic dehydration improved the tasting qualities of the end product as a result of increased sugar content and decreased acid content during the process. Using high temperature fluidized bed (HTFB) dehydration of osmotically dehydrated Rabbiteye blueberries, fruits with a soft and raisin-like texture were obtained by Kim and Toledo (1987). Sankat et al. (1996) reported that osmotically dehydrated banana slabs followed by air drying were softer and more chewable than slabs air dried without osmotic dehydration as pretreatment. Also, osmosed slabs maintained a predominantly pleasing yellow/orange color during the air drying process, compared to the untreated slabs which became light to dark brown very rapidly.

2.4.1 Water activity

Mazza (1983) showed that although sucrose treated carrot contain more moisture than the untreated material after air drying, its $a_w$ is lower; therefore higher moisture containing product is as stable as the untreated material with less moisture but the same $a_w$. Ertekin and Cakaloz (1996) studied the influence of osmosis on drying behavior and
product quality of peas. They found that at a water activity of 0.45, air dried samples had a moisture content of 5.2 kg H₂O/100 kg dry matter (DM), while samples osmosed with sucrose/citrate mixture had 5.6 kg H₂O/100 kg DM. They stated that drying would be completed at a higher final moisture content for osmosed samples.

2.4.2 Shrinkage

A major problem in air drying of solid foods is the considerable shrinkage caused by the collapse of cell structure which follows the loss of water. Two types of shrinkage are usually observed in the case of food materials: isotropic shrinkage (uniform shrinkage in all dimensions) and anisotropic shrinkage (non-uniform shrinkage). For most fruits and vegetables, the shrinkage is isotropic (Rahman 1995). Many researchers have shown that fruit shrinkage and water content during drying has a linear relationship (Lozano et al. 1980; 1983; Sjöholm and Gekas 1995). Osmotic dehydration prior to air drying results in reduced shrinkage and improved texture (Ertekin and Cakaloz 1996; Sankat et al. 1996; Kim and Toledo 1987; Lenart 1994). Kim and Toledo (1987) reported that 124 osmosed and tunnel dried blueberries were required to fill a 100 mL cylinder as compared to 215 direct tunnel dried blueberries.

2.4.3 Rehydration

Mazza (1983) showed that at room temperature, the rehydration ratio of air dried carrots decreased by over 50 % as sucrose concentration was increased from 5 to 60 % in
the osmotic dehydration step. Similar results were reported by other researchers (Ponting et al. 1966; Kim and Toledo 1987). Ponting et al. (1966) and Mazza (1983) concluded that reduced rehydration of osmotically dried fruits and vegetables was largely caused by the sugar coating on the fruits and vegetables. Ponting et al. (1966) also pointed out that less rehydration would be an advantage for dried fruits as snack products. Because of the low hygroscopicity of osmotically dried products, they can be exposed for several hours without becoming sticky.

2.5 Numerical modeling

Understanding and controlling dehydration processes is important in establishing improved design guidelines for drying systems and requires an accurate description of the dehydration mechanism. Considerable theoretical and experimental work has been done to describe the dehydration characteristics of fruits and vegetables. Theoretical computation is generally classified into two types: analytical and numerical. Analytical methods are applicable to solving problems in simple systems such as linear governing equations, simple geometry and boundary conditions, and constant physical parameter. Algorithms that use only arithmetic operations and certain logical operations are called numerical methods. Numerical methods are used for obtaining information from mathematical formulations of physical problems. Often these mathematical statements are not solvable by analytical methods. But numerical techniques do not always yield exact results. The error introduced in approximating the solution of a mathematical
problem by a numerical method is usually termed the truncation error of the method (Raghavan 1994).

Among numerical methods available for solving a differential equation are the finite difference and the finite element analysis. Finite difference method is based on approximation by difference of a derivative at a point. Solution by the finite difference method often result in long computational time. Additionally, the material properties are difficult to vary from node to node (Lomauro and Bakshi 1985). Finite element technique provides the flexibility and versatility necessary for the analysis of continuum problem where material behavior, configuration, and boundary and loading conditions are complex.

2.5.1 Osmotic dehydration

Yao and LeMaguer (1994), using finite element method, solved a 1-dimension model for the flow of solutes and water in a shrinkage material made of cells embedded in a continuous cell wall matrix. Yao and LeMaguer (1996) also developed a conceptual model to represent the cellular structure of a tissue consisting of individual cells embedded in a continuous cell wall matrix. A 1-dimensional transient mathematical model that incorporated diffusion, bulk-flow, trans-membrane flux and shrinkage of the matrix was obtained.
2.5.2 Air drying

Simulation studies of food drying depend basically on the analysis of heat and moisture changes within the food. Husain et al. (1973) gave the governing equations for the non-linear coupled heat and moisture diffusion model. Due to the small magnitude of thermogradient coefficient, the thermal diffusion term in the mass transfer equation could be neglected (Husain et al. 1973).

Haghighi and Segerlind (1988) used the finite element formulation to obtain numerical solutions to the simultaneous moisture and heat diffusion equations describing the moisture removal and heat intake process for an isotropic sphere. They used this model to fit the drying process of soybean kernels.

Misra and Young (1980) simulated moisture diffusion and shrinkage in soybeans using finite element method. In this model, they used an equation to describe the effect of shrinkage and water content on mass diffusivity. Rosselló et al. (1997) developed two diffusion models (separation of variable method and finite difference method) for finite cylindrical shaped bodies which take into account that mass transfer can have a nonisotropic nature. In the finite difference model, a volume change (shrinkage) was considered. These models were applied to the simulation of drying curves of green beans.
2.6 Anthocyanin determination and recovery

Red or blue color is an important indicator of quality for fruits such as cherries and blueberries. Color change in dehydration media or fruits indicates loss or degradation of color compounds, mainly anthocyanins. Therefore, color loss during osmotic dehydration should be kept to a minimum during processing of the fruit. On the other hand, leaching of anthocyanins into the dehydration media would result in a rich source of pigments, and the leached anthocyanins could be recovered from the dehydration media and used as a natural color in food formulations.

2.6.1 Anthocyanin determination

Gao and Mazza (1995) developed a simple HPLC method for characterization and quantitation of anthocyanins in sweet cherries. Sondheimer and Kertesz (1948) determined anthocyanin content in strawberries and strawberry products using absorbances at 500 nm at pH 3.4 and 2.0. Fuleki and Francis (1968a) used the absorbance at 535 nm for the determination of total anthocyanin in cranberries in an alcoholic solvent system.

Wrolstad (1976) applied the pH differential method originally developed by Fuleki and Francis (1968b) to determine the anthocyanin pigment content in fruit products using a spectrophotometer. The quantitative determination of anthocyanin content by this method is based on the fact that at pH 1.0, anthocyanins exist predominantly in the highly colored flavylium cation form while at pH 4.5 they are
essentially in the colorless carbinol form (Mazza and Miniati 1993). The difference in absorbance at the wavelength of maximum absorption between the two pHs is proportional to anthocyanin content.

2.6.2 Anthocyanin recovery

Methods applied to the recovery and purification of anthocyanins are based primarily on two principles: (i) adsorption - desorption of anthocyanin pigments on selective adsorbents and (ii) permeation of the extracted pigments through ultrafiltration / hyperfiltration membranes. Peri and Bonini (1976) used a polyamide (Nylonplast) and an insoluble polyvinylpyrrolidone resin (Polycarb AT or PVP) to recover anthocyanins from wine distillation waste. Wrolstad and Putnam (1969) used polyvinylpyrrolidone to isolate anthocyanin pigments from strawberries. Saquet-Barel et al. (1982) studied the adsorption of anthocyanins on formophenolic resins (Duolite® S37, S761 and A5), polyacrylic porous polymers (Duolite® ES861) and PVP. Adsorption of anthocyanins on a formophenolic resin in a continuous process retained about 25 mg of pigments per gram of resin. Using methanol as eluant, a good quality concentrated anthocyanin could be obtained. Shrikhande (1984) patented a process utilizing Amberlite® CG-50 to recover anthocyanins from grape wastes. Chiriboga and Francis (1970) set up a system to recover anthocyanin from cranberry pomace by an Amberlite® CG-50. They found that the adsorptive capacity of the resin is affected by the concentration of the pigment solution: with a less concentrated solution, the resin will adsorb less pigment before it is "saturated". They also reported that this resin is remarkably stable; after 175 cycles, the
resin showed no efficiency loss. Wang and Kao (1987) stated that the degradation index of anthocyanin from red wine pomaces is greater when extracted with acidic methanol than with acidic ethanol or acidic ethanol containing SO₂. Anthocyanin degradation was relatively low upon heating at 65 °C for 120 min, but only 53-66 % anthocyanin remained after heating at 100 °C for 120 min. Chung et al. (1986) concentrated Perilla anthocyanins (cyanidin-3, 5-diglucosides acylated with coumaric acid or caffeic acid) extracted with 10% citric acid by ultrafiltration. Recovery of anthocyanin was over 60% by ultrafiltration at a volume concentration ratio of 4.
3. MATERIALS AND METHODS

3.1 Materials

Cherries: Fresh sweet cherries, *Prunus avium* L. (Rosaceae), cultivar ‘Bing’, 24 mm in diameter on average, with a mean soluble solids of 20.8 °Brix and moisture content (mc) of 78.5 % wet basis, were picked from a research orchard at the Pacific Agri-Food Research Centre, Agriculture and Agri-Food Canada, Summerland, BC.

Sour cherries, *Prunus cerasus* L. (Rosaceae), cultivars ‘Montmorency’ and ‘Erdi Botermo’, were picked at full maturity from a commercial orchard near Kelowna, BC. ‘Montmorency’ cherries were 18 mm in diameter on average, with a mean mc of 84.9 % and soluble solids of 14.2 °Brix; ‘Erdi Botermo’ cherries were 20 mm in diameter on average, with mean mc of 77.9 %, and 22.4 °Brix mean soluble solids.

Fresh cherries were immediately sorted after picking (with stems) and stored at 2 °C until used (within one month).

Blueberries: Highbush blueberries, *Vaccinium Corymbosum* L., cultivar ‘Bluecrops’, 14 mm in diameter on average, with a mean soluble solids of 13.2 °Brix and a moisture content of 84.1 % wet basis, were purchased from South Alder Farm, Aldergrove, BC. The blueberries were packaged in boxes and wrapped with a PD 955 perforated film with 20 μm holes. They were kept in a chamber with RH = 64 % (inside package RH = 99 %), \( O_2 = 18 \% \), \( CO_2 = 1 \% \), at 1 °C until used within 3 weeks.

Sucrose: Commercial food grade sucrose was purchased from a local market and used to prepare sucrose solution as the osmotic dehydration medium.
3.2 Methods

3.2.1 Analytical methods

Moisture and total solids contents of the fresh and treated fruits were measured in triplicate by using the vacuum oven method (AOAC 1990).

Soluble solids of the samples were determined in triplicate with a digital refractometer (Model ABBE MARK II, Reichert Scientific Instruments, Buffalo, NY).

pH of sucrose solutions before and after dehydration was measured with a pH meter (Model pH M82, Bach-Simpson Limited, London, ON).

Total anthocyanin content of sucrose solution or extracts were measured using the pH differential method (Wrolstad 1976) with a spectrophotometer (Model DU 640, Beckman Instruments, Inc., Fullerton, CA).

According to Lambert-Beer’s Law, the concentration of anthocyanin could be determined by the following equations:

\[
C = \frac{A}{eL} \times 10^3 \times MW \times F
\]

\[
A = (A_{\lambda_{\text{max}}} - A_{700\text{nm}})_{pH1.0} - (A_{\lambda_{\text{max}}} - A_{700\text{nm}})_{pH4.5}
\]

where:

\( C = \) concentration of anthocyanins (mg/L),

\( A = \) Absorbances of pigment which is measured with a spectrophotometer,

\( e = \) molar absorbance,
L = the path length, most spectrophotometer cells L = 1 cm.

F = dilution factor,

MW = molecular weights, and

$A_{\lambda_{\text{max}}}, A_{700\text{nm}}$ = absorbance of pigment at wavelength of maximum absorption, and at 700 nm, respectively,

To determine the anthocyanin content of materials with complex anthocyanin compositions, different standards (Wrolstad 1976) were used to reflect the compositional difference. Total anthocyanin content of cherries was expressed as cyanidin-3-glucoside (MW = 445.2, ε = 26900, $\lambda_{\text{max}}$ = 510 nm). The anthocyanin content of blueberries was calculated as malvidine-3-glucoside (MW = 493.5, ε = 28000, $\lambda_{\text{max}}$ = 520 nm).

3.2.2 Osmotic dehydration

Osmotic dehydration of fruit was carried out in covered containers. A sucrose solution was first warmed to the designated temperature and cherries or blueberries (fruits : solution = 1:20, w/w) were immersed in the solution. The system was continuously shaken in a temperature-controlled shaker (100 rpm, model 3597-PR, Environ Shaker, Melrose Park, IL). At the end of operation, samples were removed from the solutions, quickly rinsed with distilled water, gently blotted with a paper towel to remove surface solution, and weighed. Water content (WC, g/g DM), solids gain (SG, g/g DM), and water loss (WL, g/g DM) were calculated as follows:
\[ WC = \frac{W \cdot M}{W \cdot (1 - M)} \]  

(3)

\[ SG = \frac{W \cdot (1 - M)}{W_o \cdot (1 - M_o)} - 1 \]  

(4)

\[ WL = \frac{W_o \cdot M_o - W \cdot M}{W_o \cdot (1 - M_o)} \]  

(5)

where:

\( M = \) moisture content of sample at time \( t \) on wet basis (\%),

\( M_o = \) initial moisture content of sample on wet basis (\%),

\( W = \) mass of sample at time \( t \) (g),

\( W_o = \) initial mass of sample (g),

\( WC = \) water content of sample on a dry matter basis (g/g DM),

\( SG = \) solids gain of sample on an initial dry matter basis (g/g DM), and

\( WL = \) water loss of sample on an initial dry matter basis (g/g DM).

Response-surface methodology (RSM) was used to study the main effects of the process variables on mass transfer during osmotic dehydration of fresh cherries and to find the optimum operation conditions. The experimental design adopted was a central composite design with three factors and five levels (Haaland 1989). The actual factor values, chosen from preliminary tests, and the corresponding coded values (-1.68, -1, 0, 1, 1.68) are given in Table 3.1. Water content, solids gain, water loss, and mass reduction (MR %) of the dehydrated samples were the dependent variables. The complete design
consisted of 18 experimental runs with four replications of the center point. Experimental runs were carried out in random order.

Table 3.1 Response surface central composite design with three factors and five levels

<table>
<thead>
<tr>
<th>Run No</th>
<th>Temperature (X₁) (ºC)</th>
<th>Concentration (X₂) (ºBrix)</th>
<th>Time (X₃) (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>25 (-1)</td>
<td>55 (-1)</td>
<td>1.5 (-1)</td>
</tr>
<tr>
<td>2</td>
<td>25 (-1)</td>
<td>55 (-1)</td>
<td>4.5 (+1)</td>
</tr>
<tr>
<td>3</td>
<td>25 (-1)</td>
<td>65 (+1)</td>
<td>1.5 (-1)</td>
</tr>
<tr>
<td>4</td>
<td>25 (-1)</td>
<td>65 (+1)</td>
<td>4.5 (+1)</td>
</tr>
<tr>
<td>5</td>
<td>45 (+1)</td>
<td>55 (-1)</td>
<td>1.5 (-1)</td>
</tr>
<tr>
<td>6</td>
<td>45 (+1)</td>
<td>55 (-1)</td>
<td>4.5 (+1)</td>
</tr>
<tr>
<td>7</td>
<td>45 (+1)</td>
<td>65 (+1)</td>
<td>1.5 (-1)</td>
</tr>
<tr>
<td>8</td>
<td>45 (+1)</td>
<td>65 (+1)</td>
<td>4.5 (+1)</td>
</tr>
<tr>
<td>9</td>
<td>18 (-1.68)</td>
<td>60 (0)</td>
<td>3.0 (0)</td>
</tr>
<tr>
<td>10</td>
<td>52 (+1.68)</td>
<td>60 (0)</td>
<td>3.0 (0)</td>
</tr>
<tr>
<td>11</td>
<td>35 (0)</td>
<td>52 (-1.68)</td>
<td>3.0 (0)</td>
</tr>
<tr>
<td>12</td>
<td>35 (0)</td>
<td>68 (+1.68)</td>
<td>3.0 (0)</td>
</tr>
<tr>
<td>13</td>
<td>35 (0)</td>
<td>60 (0)</td>
<td>0.5 (-1.68)</td>
</tr>
<tr>
<td>14</td>
<td>35 (0)</td>
<td>60 (0)</td>
<td>5.5 (+1.68)</td>
</tr>
<tr>
<td>15</td>
<td>35 (0)</td>
<td>60 (0)</td>
<td>3.0 (0)</td>
</tr>
<tr>
<td>16</td>
<td>35 (0)</td>
<td>60 (0)</td>
<td>3.0 (0)</td>
</tr>
<tr>
<td>17</td>
<td>35 (0)</td>
<td>60 (0)</td>
<td>3.0 (0)</td>
</tr>
<tr>
<td>18</td>
<td>35 (0)</td>
<td>60 (0)</td>
<td>3.0 (0)</td>
</tr>
</tbody>
</table>

The response surface regression (PROC RSREG) of SAS (SAS 1990a) was used to analyze the experimental results to fit a quadratic polynomial model (Haaland 1989; Saurel et al. 1994) as follows:
\[ Y = b_0 + \sum_{i=1}^{3} b_i X_i + \sum_{i=1}^{3} \sum_{j=1}^{3} b_{ij} X_i X_j \]  

(6)

where:

- \( Y \): the dependent variable.
- \( b_0, b_i, b_{ij} \): regression coefficients, \( i, j = 1, 2, 3 \), and
- \( X_i, X_j \): the independent variables: \( X_1 = \) temperature (°C), \( X_2 = \) concentration of solution (°Brix), and \( X_3 = \) time (h).

Three-dimensional surfaces were drawn using the Statistical Graphics procedure (PROC G3D) of SAS (SAS 1990b).

The water diffusion coefficient \( D \) during osmotic dehydration was determined by the following equation:

\[
\frac{WL}{WL_\infty} = 1 - \sum_{\pi=1}^{6} \frac{6}{(\pi^2)} \exp\left[-(\pi^2) \left(\frac{Dt}{R^2}\right)\right]
\]

(7)

where:

- \( WL \): water loss of sample on an initial dry matter basis (g/g DM),
- \( WL_\infty \): equilibrium water loss on an initial dry matter basis (g/g DM),
- \( D \): effective diffusion coefficient (m²/s),
- \( t \): time (s), and
- \( R \): radius, for cherries \( R = r_2 - r_1, r_1 = \) average stone radius, \( r_2 = \) average cherry radius, for blueberries \( R = \) average blueberry radius (m).
3.2.3 Skin treatment

To study the influence of skin treatments on osmotic dehydration and final air-dried product quality, four skin treatments were carried out on whole fresh cherries:

- Blanching with steam (137.9 KPa) for 30 s.
- Solvents treatment: 1 % ethyl myristate in 5 % ethanol solution (fruit : solution = 1:5) at 30 °C for 1 min, followed by washing with distilled water for 20 s.
- Hot water dipping at 90 °C for 30 s.
- Lipase (Palatase® M 1000 L, Novo Nordisk, Denmark) 24000 Lipase unit (LU) in 200 mL, (fruit : solution = 1:5) at 37 °C for 40 s, followed by washing with distilled water for 20 s.

Three treatments were carried out on fresh blueberries:

- Blanching with steam (137.9 KPa) for 30 s.
- Solvents treatment: 1 % ethyl myristate in 5 % ethanol solution (fruit : solution = 1:5) at 30 °C for 1 min, followed by washing with distilled water for 20 s.
- Lipase (Palatase® M 1000 L, Novo Nordisk, Denmark) 24000 LU in 200 mL, (fruit : solution = 1:5) at 30 °C for 40 s, followed by washing with distilled water for 20 s.

Processing and quality variables that were measured included: water content, moisture diffusion coefficients, and anthocyanin leaching during osmotic dehydration.
3.2.4 Air drying

The effect of osmotic dehydration treatment on subsequent air drying was studied using fresh and osmotically dehydrated blueberries and cherries. The cherries were subjected to treatments a, b, and c as listed below:

a) pitting -- air drying
b) pitting -- osmotic dehydration -- air drying
c) steaming -- pitting -- osmotic dehydration -- air drying

Blueberries were treated as follows:
d) air drying
e) osmotic dehydration -- air drying
f) steaming -- osmotic dehydration -- air drying

Air drying was carried in a lab oven dryer (Precision Scientific Co., Chicago, IL) at 40, 60, and 80 °C. The air flow velocity was 2.0 m/s. The air temperatures were measured with thermocouples connected with a thermometer (Model: DP702, Omega Engineering, Inc., Stamford, CT) and the air flow velocity was measured with a hot wire anemometer (Datametrics 100VT airflow meter, Edwards High Vacuum International, Wilmington, MA).

Drying curves were determined by weighing the subsamples (25-30 g initial weight) manually and returned to oven at 30 min intervals.

Product shrinkages after various times of drying were determined by displacement of toluene (Mazza and LeMaguer 1980; Viollaz et al. 1975). The ratio of fruit to toluene was about 5 g (1 % of total sample) to 100 ml.
Rehydration rates of dried samples were measured in distilled water at 25 °C. Dehydrated cherries or blueberries (5 g) were added to 150 mL distilled water in a 250 mL beaker, mixed thoroughly and allowed to rehydrate for various lengths of time (Mazza and LeMaguer 1980).

The anthocyanin content of fruits before and after osmotic dehydration or air drying were determined by the pH differential method using a spectrophotometer. The content was expressed in mg/100g fresh basis (see section 3.2.1).

3.2.5 Moisture sorption isotherms

The equilibrium moisture content of freeze dried fruits, osmotically dehydrated followed by freeze dried fruits, and air dried fruits (kept in a low relative humidity room with RH = 21-22 % at 22 °C) were determined by placing subsamples in desiccators containing saturated salt solutions which gave different constant relative humidities in the range 0.11 < \( a_w \) < 0.96. The desiccators were kept at constant temperatures of 10, 25, or 40 °C until equilibrium was reached. Small amounts of crystalline thymol were placed in the desiccators which had RH > 60% to prevent microbial spoilage of the fruits (Saravacos et al. 1986, Zhang et al. 1996).

Five equations were used for the analysis of the equilibrium moisture data (Table 3.2).
Table 3.2 EMC-ERH relationships used to analyze EMC-ERH data of cherries and blueberries*

<table>
<thead>
<tr>
<th>Relationship</th>
<th>Equation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Modified Henderson (1952) equation (Thompson et al. 1968)</td>
<td>( 1 - a_w = \exp\left[-A(T+C)M^B\right] )</td>
</tr>
<tr>
<td>Modified Chung-Pfost (1967) equation (Pfost et al. 1976)</td>
<td>( a_w = \exp\left[-\frac{A}{T+C}\exp(-BM/100)\right] )</td>
</tr>
<tr>
<td>Modified Halsey (1948) equation (Iglesias and Chirife 1976)</td>
<td>( a_w = \exp\left[-\exp(A+BT)M^{-C}\right] )</td>
</tr>
<tr>
<td>Modified Oswin (1946) equation (Chen and Morey 1989)</td>
<td>( a_w = \frac{I}{\left[(A+BT)/M\right]^C + I} )</td>
</tr>
<tr>
<td>Modified GAB (Guggenheim 1966; Anderson 1946; de Boer 1953) equation (Jayas and Mazza 1993)</td>
<td>( M = \frac{A(C/T)Ba_w}{(1-Ba_w)(1-Ba_w + (C/T)Ba_w)} )</td>
</tr>
</tbody>
</table>

* A, B, C are constants, M is percent water content on dry basis, \( a_w \) is water activity, and T is temperature, °C.

### 3.2.5 Anthocyanin recovery

When the amount of anthocyanin in the sucrose solution reached a level above 25 mg/L as a result of leaching during repeated osmotic dehydration, the anthocyanin was recovered by adsorption - desorption using food grade resins.

The resin was first poured in the anthocyanin-containing sucrose solution for the resin to adsorb the anthocyanins. Subsequently, the mixture was centrifuged (Stainless Steel Juicer, ACME Juicer MFG. Co, Sierra Madre, CA) to release the sucrose solution. The resin with anthocyanins was rinsed with distilled water containing 2.5 % acetic acid (pH about 2.5) to further remove any adhered sucrose solution. The recovered sugar solution coming off the Juicer was monitored with an ABBE MARK II digital refractometer for sugar concentration and reused for osmotic dehydration. Finally, the
pigments adsorbed on the resin were eluted with ethanol (95 %) containing 5 % acetic acid (pH about 3.0). The regenerated resin was re-equilibrated with aqueous 2.5 % acetic acid and reused. The ethanol was recovered by evaporation from the solution with a thin film rotary evaporator (Büchi, Laboratoriums - Technik AG, Switzerland) and the concentrated pigments were freeze dried and collected as dry powder.

The adsorption characteristics of anthocyanin pigments on resins was studied using Amberlite® XAD-2, XAD-4, CG-50, XAD7HP, XAD16HP, and Duolite® XAD765 resins (Rohm & Haas, Philadelphia, PA). Resins (10 g) were immersed in 15 mL of sucrose solution for saturation with anthocyanins for various lengths of time (30 s, 1, 5, 10, 15, 20, 25, and 30 min), the mixture (resin and solution) was immediately poured onto a Büchner funnel under vacuum. The concentration of pigment in the filtered solution was measured.

The ratios of 1:1.5, 1:2, 1:5, 1:10, and 1:20 (resin : solution) were used to determine the optimum ratio of resin to solution for adsorption.

The effect of sucrose in solution on anthocyanin adsorption was determined by dissolving 18.15 mg/L pigment in 15 mL sucrose solutions of 2, 5, 10, 20, 30, 40, 50, and 60 °Brix, respectively. The level of adsorption was measured using Amberlite® CG-50 as the test resin.

The effect of pH on adsorption was determined by adjusting the sucrose solution pH to 1, 2, 3, 4, and 5, respectively, and using 10 g Amberlite® XAD-2 immersed in 15 mL sucrose solution to measure the adsorption rate.
The effect of acetic acid concentration in EtOH on pigment desorption from resin was determined using 0.5, 1.0, 2.4, 4.8, 9.1, and 16.7% acetic acid in 95% EtOH to elute the pigment from the Amberlite XAD7HP.

Table 3.3 Response surface central composite design with two factors and five levels

<table>
<thead>
<tr>
<th>Run No</th>
<th>Ethanol concentration (%)</th>
<th>Desorption time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>45 (-1)</td>
<td>5 (-1)</td>
</tr>
<tr>
<td>2</td>
<td>45 (-1)</td>
<td>25 (+1)</td>
</tr>
<tr>
<td>3</td>
<td>85 (+1)</td>
<td>5 (-1)</td>
</tr>
<tr>
<td>4</td>
<td>85 (+1)</td>
<td>25 (+1)</td>
</tr>
<tr>
<td>5</td>
<td>36.8 (-1.41)</td>
<td>15 (0)</td>
</tr>
<tr>
<td>6</td>
<td>93.2 (+1.41)</td>
<td>15 (0)</td>
</tr>
<tr>
<td>7</td>
<td>65 (0)</td>
<td>0.9 (-1.41)</td>
</tr>
<tr>
<td>8</td>
<td>65 (0)</td>
<td>29.1 (+1.41)</td>
</tr>
<tr>
<td>9</td>
<td>65 (0)</td>
<td>15 (0)</td>
</tr>
<tr>
<td>10</td>
<td>65 (0)</td>
<td>15 (0)</td>
</tr>
<tr>
<td>11</td>
<td>65 (0)</td>
<td>15 (0)</td>
</tr>
</tbody>
</table>

To select the best resin and optimal desorption conditions (EtOH concentration and desorption time), a central composite response-surface design with two factors and five levels (Haaland 1989) was used (Table 3.3). The actual factor values and the corresponding coded values (-1.41, -1, 0, 1, 1.41) are given in Table 3.3. The recovery rate of pigments from Amberlite® XAD7HP, XAD16HP, and Duolite® XAD765 were the dependent variables. The complete design consisted of 11 experimental runs with three replications of the center point. Experimental runs were carried out in random order.
3.2.6 Numerical modeling

Finite Element Method (FEM) was used to obtain numerical solutions to the axisymmetric transient mass transfer during osmotic dehydration of whole cherries and blueberries, and to simultaneous mass and heat transfer coupled with a shrinkage during air drying of pitted cherries and whole blueberries.

3.2.6.1 Osmotic dehydration

The mathematical description of the axisymmetric transient mass transfer problem in cylindrical coordinates is given below:

\[
\frac{\partial X}{\partial t} = D \frac{\partial^2 X}{\partial r^2} + \frac{D}{r} \frac{\partial X}{\partial r} + D \frac{\partial^2 X}{\partial z^2}
\]  

(8)

where:

\[X = \text{moisture content (g H}_2\text{O/g DM)},\]
\[t = \text{time (s)},\]
\[D = \text{effective diffusion coefficient (m}^2/\text{s)}, \text{and}\]
\[r, z = \text{radial and vertical coordinate directions.}\]

Initial and boundary conditions for the governing equation (8) are:

1) Initially, the sphere is at uniform moisture content \(t = 0\)

\[X = X_0\]  

(9)

2) for \(t > 0\) and at the surface

\[D\left(\frac{\partial X}{\partial r} + \frac{\partial X}{\partial z}\right) + h_m (X - X_w) = 0\]  

(10)

where:
h_m = surface mass transfer coefficient (m/s), and
X_w = ambient moisture content (g H_2O/g DM).

The functional equivalent of this problem is:

\[ F = \int \frac{1}{2} \left[ D \left( \frac{\partial X}{\partial r} \right)^2 + D \left( \frac{\partial X}{\partial z} \right)^2 + 2 \left( \frac{\partial X}{\partial t} \right) X \right] dv + \int \frac{h_m}{2} (X - X_w)^2 dS \]

where:

V = body volume (m³), and
S = surface with convection boundary (m²).

Following the procedure in lecture notes (Jayas 1995), the solution was obtained.

Define:

\[ \{ \phi \}^T = \begin{bmatrix} \frac{\partial X}{\partial r} & \frac{\partial X}{\partial z} \end{bmatrix} ; \quad [D] = \begin{bmatrix} D_{rr} & 0 \\ 0 & D_{zz} \end{bmatrix} \]

Let:

\[ X^{(e)} = [N^{(e)}] \{ X \} \]

(12)

where:

\[ [N^{(e)}] = \text{the matrix of shape functions, and} \]

\[ \{ X \} = \text{nodal moisture content (g H}_2\text{O/g DM).} \]

F is minimized when:
\[ \frac{\partial F}{\partial \{X\}} = \sum_{e=1}^{E} \frac{\partial F^{(e)}}{\partial \{X\}} = 0 \]  

(13)

For each element:

\[ \left[ C^{(e)} \right] \frac{\partial \{X\}}{\partial t} + \left[ K^{(e)} \right] \{X\} - \{F^{(e)}\} = 0 \]  

(14)

Where: \[ C^{(e)} = \int_V [N]^T [N] dV \]

\[ K^{(e)} = \int_V [B]^T [D] [B] dV + \int_S h_m [N]^T [N] dS \]

\[ F^{(e)} = \int_S h_m X_m [N]^T dS \]

For a linear three-node triangular element:

\[ 2A = R_j Z_k + Z_j R_i + R_k Z_i - R_k Z_j - Z_k R_i - R_j Z_i \]

Let:

\[ r = \frac{R_i + R_j + R_k}{3} \]

where:

\[ R_i, R_j, R_k = r \text{ direction coordinates at nodes } i, j, k, \text{ and} \]

\[ Z_i, Z_j, Z_k = z \text{ direction coordinates at nodes } i, j, k \]

The matrix \([C]\) is:

\[ C^{(e)} = 2\pi \int_A r [N]^T [N] dA \]

\[ C_{ij} = \frac{2\pi A}{60} \left( 1 + \delta_{ij} \right) \left( 3r + R_i + R_j \right) \] where \( \delta_{ij} = \text{Kronecker delta} \)

\[ b_i = Z_j - Z_k ; \quad b_j = Z_k - Z_i ; \quad b_k = Z_i - Z_j \]
The equation (14) could be solved by using Euler’s Backward Difference Techniques:

\[
\begin{bmatrix}
 \frac{a_i}{t} = R_j - R_k; \quad \frac{a_j}{t} = R_k - R_i; \quad \frac{a_k}{t} = R_i - R_j
\end{bmatrix}
\]

\[
\int [B]^T [D] [B] dV = \frac{2\pi r D_{zt}}{4A} \begin{bmatrix}
 b_i^2 & b_i b_j & b_i b_k \\
 b_i b_j & b_j^2 & b_j b_k \\
 b_i b_k & b_j b_k & b_k^2
\end{bmatrix} + \frac{2\pi r D_{zt}}{4A} \begin{bmatrix}
 a_i^2 & a_i a_j & a_i a_k \\
 a_i a_j & a_j^2 & a_j a_k \\
 a_i a_k & a_j a_k & a_k^2
\end{bmatrix}
\]

\[
\int h_m [N]^T [N] dS = \frac{\pi h_m L_{jk}}{6} \begin{bmatrix}
 0 & 0 & 0 \\
 0 & 3R_j + R_k & R_j + R_k \\
 0 & R_j + R_k & R_j + 3R_k
\end{bmatrix}
\]

\[
\int h_m X_{mn} [N]^T dS = \frac{\pi h_m X_{mn} L_{jk}}{3} \begin{bmatrix}
 0 & 0 & 0 \\
 0 & 2 & 1 \\
 0 & 1 & 2
\end{bmatrix} \begin{bmatrix}
 R_i \\
 R_j \\
 R_k
\end{bmatrix}
\]

The equation (14) could be solved by using Euler’s Backward Difference Techniques:

\[
\left( [K] + \frac{[C]}{\Delta t} \right) \{X^n\} = \left[ \frac{[C]}{\Delta t} \right] \{X^i\} + \{F\}
\]

where \( \Delta t \) is the time step.

Finally, assembling the element matrices into global system matrices, and solving the problem gives the vector \( \{X^*\} \).

In order to have a measure of the overall moisture content, rather than the nodal moisture values, the mass average moisture was used (assuming constant density).

\[
\overline{X} = \frac{\int \overline{X(r,z)} dm}{\nu} \quad \text{for every time step}
\]

where \( dm \) is the mass of an element, \( g \).
Fig. 3.1 Flow chart for the Finite Element program to simulate mass transfer during osmotic dehydration.

and using equation (12):
The flow diagram for the FORTRAN program is shown in Fig. 3.1.

Application

The formulation shown above was used to solve the osmotic dehydration of fresh cherries and blueberries and predictions of the model were compared with the experimental data.

The following assumptions for the model were made:

1. The cherries and blueberries were isotropic sphere symmetrical with respect to their center. Two dimensional axisymmetric finite element grids as shown in Fig. 3.2 (a and b) were used.

2. All material parameters of cherries and blueberries were assumed to be constant. The material properties and initial values are listed in Table 3.4.

3. The shrinkage was negligible.

<table>
<thead>
<tr>
<th>Table 3.4 Data for calculation of osmotic dehydration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material properties*</td>
</tr>
<tr>
<td>-----------------------</td>
</tr>
<tr>
<td>D_1 = 1.75 \times 10^{-11} m^2/s</td>
</tr>
<tr>
<td>D_2 = 8.75 \times 10^{-13} m^2/s</td>
</tr>
<tr>
<td>h_m = 1.75 \times 10^{-4} m/s</td>
</tr>
<tr>
<td>Material size</td>
</tr>
<tr>
<td>Initial values</td>
</tr>
<tr>
<td>Solution condition</td>
</tr>
<tr>
<td>Time step</td>
</tr>
</tbody>
</table>

* h_m calculated using equation in Hallström et al. (1988, p90).
Fig. 3.2 The two-dimensional axisymmetric element grid of cherry (a) and blueberry (b) in osmotic dehydration.
3.2.6.2 Air drying

The mathematical description of the axisymmetric simultaneous transient mass and heat transfer problem in cylindrical coordinates is given as:

\[
\frac{\partial X}{\partial t} = D \frac{\partial^2 X}{\partial r^2} + \frac{D}{r} \frac{\partial X}{\partial r} + D \frac{\partial^2 X}{\partial z^2}
\]

\[
\varrho c \frac{\partial T}{\partial t} = k \frac{\partial^2 T}{\partial r^2} + \frac{k'}{r} \frac{\partial T}{\partial r} + k' \frac{\partial^2 T}{\partial z^2} + L \varrho \frac{\partial X}{\partial t}
\]

where:

- \( X \) = moisture content (g H\(_2\)O/g DM),
- \( t \) = time (s),
- \( D \) = effective diffusion coefficient (m\(^2\)/s),
- \( r, z \) = radial and vertical coordinate directions,
- \( T \) = temperature (K),
- \( \varrho \) = density (kg/m\(^3\)),
- \( c \) = specific heat (J*kg\(^{-1}\)•K\(^{-1}\)),
- \( k' \) = thermal conductivity (W*cm\(^{-1}\)•K\(^{-1}\)), and
- \( L \) = latent heat of vaporization of water (J/kg).

Initial and boundary conditions for the governing equations (18) and (19) are:

1) The sphere is at uniform temperature and moisture content initially \((t = 0)\)

\[
T = T_o, \quad X = X_o
\]

(20)

2) for \( t > 0 \) and at the surface
\[
D \left( \frac{\partial X}{\partial r} + \frac{\partial X}{\partial z} \right) + h_m (X - X_w) = 0
\]  

(21)

\[
k' \left( \frac{\partial T}{\partial r} \frac{\partial T}{\partial z} \right) + h (T - T_w) = 0
\]  

(22)

where:

- \( h_m \) = surface mass transfer coefficient (m/s),
- \( X_w \) = ambient moisture content (g H₂O/g DM),
- \( h \) = surface heat transfer coefficient (W·m⁻²·K⁻¹), and
- \( T_w \) = ambient temperature (K).

The procedure used to solve Eq. 8 was also used to solve the equation (18).

The functional equivalent of the equation (19) is:

\[
F = \int \frac{1}{2} \left[ k' \left( \frac{\partial T}{\partial r} \right)^2 + k' \left( \frac{\partial T}{\partial z} \right)^2 - 2Lq \left( \frac{\partial X}{\partial t} \right) + 2qc \left( \frac{\partial T}{\partial t} \right) \right] dV + \int \frac{h}{2} (T - T_w)^2 dS
\]  

(23)

Following the similar procedure as in section 3.2.6.1, for each element:

\[
\left[ C_{ii}^{(e)} \right] \frac{\partial \{ X \}}{\partial t} + \left[ K_{ii}^{(e)} \right] \{ X \} - \{ F_{i}^{(e)} \} = 0
\]  

(24)

\[
\left[ C_{zz}^{(e)} \right] \frac{\partial \{ T \}}{\partial t} + \left[ K_{zz}^{(e)} \right] \{ T \} - \{ F_{z}^{(e)} \} = 0
\]  

(25)

Where:

\[
\left[ C_{ii}^{(e)} \right] = \int y [N]^T [N] dV
\]

\[
\left[ K_{ii}^{(e)} \right] = \int [B]^T [D][B] dV + \int h_m [N]^T [N] dS
\]

39
Solving equation (24) for \( \partial M/\partial t \) and inserting in equation (25):

\[
[C_{22} \frac{\partial T}{\partial t} + [K_2] \{T\} - \{F_2\} + L_0 ([K_1]\{X\} - \{F_1\}) = 0
\]

The equation (24) and (26) could be solved by using Euler's Backward Difference Techniques:

\[
\left( [K_1] + \frac{[C_{11}]}{\Delta t} \right) \{X^n\} = \left( \frac{[C_{11}]}{\Delta t} \right) \{X^r\} + \{F_1\}
\]

\[
\left( [K_2] + \frac{[C_{22}]}{\Delta t} \right) \{T^n\} = \left( \frac{[C_{22}]}{\Delta t} \right) \{T^r\} + \{F_2\} + L_0 ([F_1] - [K_1]\{X^n\})
\]

Finally, assembling the element matrices into global system matrices, the problem can be solved. First solving \( \{X^n\} \) from equation (27), the equation (28) will yield the \( \{T^n\} \). Then using equations (17) and (29) will give the mass average moisture content and temperature.
Air drying with shrinkage

Shrinkage factor $V/V_o$ and water content have the following relationship:

\[
\frac{V}{V_o} = ax + b
\]  

where $a$ and $b$ are the constants; $V$ is the body volume, $m^3$; and $V_o$ is the initial body volume, $m^3$.

Rahman (1995) stated that most fruits and vegetables have an isotropic shrinkage property. It means that there is an uniform shrinkage in all dimensions of the materials. Therefore, we have:

\[
\frac{r}{r_o} = \left( \frac{V}{V_o} \right)^{\frac{1}{3}}, \quad \frac{z}{z_o} = \left( \frac{V}{V_o} \right)^{\frac{1}{3}}
\]

where: $r, z = $ radial and vertical coordinate directions, and $r_o, z_o = $ initial radial and vertical coordinate value.

After each time step, we know $X$ and can calculate the new coordinates $r, z$, moisture content and temperature for the next time step. This procedure was repeated until the moisture content reached the equilibrium moisture content at $a_w < 0.6$.

The flow diagram for a FORTRAN program is shown in Fig. 3.3.
Read initial coordinates and set initial values \((X_i, T_i)\) and boundary conditions.

Set time step \((T_S)\) for each node point \((X_{i+},)\).

Calculate node temperature \((T_{i+})\) using Euler's Backward Difference method.

Calculate volume average water content and temperature of the body.

Calculate water activity \(a_w\).

If \(a_w \leq 0.6\), print results and stop.

If \(a_w > 0.6\), update temperature \(T_{M_{i+1}} = T_{M_i} + T_S\) and calculate new coordinates using shrinkage factor.

Fig. 3.3 Flow chart for the Finite Element program to simulate simultaneous heat and mass transfer during air drying.
Application

The formulation developed above was applied to air drying of fresh cherries and blueberries and the results were compared with the experimental data.

The following assumptions for the model were made:

1. The cherries and blueberries were isotropic spheres symmetrical with respect to their center. Two dimensional axisymmetric finite element grids as shown in Fig. 3.4 were used.

2. All material parameters of the cherries and blueberries were assumed to be constant.

The material properties and initial values are listed in Table 3.5.

### Table 3.5 Data for calculation of air drying

<table>
<thead>
<tr>
<th>Material properties*</th>
<th>Fresh blueberries</th>
<th>Fresh cherries</th>
</tr>
</thead>
<tbody>
<tr>
<td>c = 3785 J<em>kg⁻¹</em>K⁻¹</td>
<td>c = 3689 J<em>kg⁻¹</em>K⁻¹</td>
<td></td>
</tr>
<tr>
<td>Q = 1021.6 kg/m³</td>
<td>Q = 1073.3 kg/m³</td>
<td></td>
</tr>
<tr>
<td>L= 2.75×10⁶ J/kg</td>
<td>L= 2.79×10⁶ J/kg</td>
<td></td>
</tr>
<tr>
<td>Kₘₚ Kₓₓ = 0.528 W<em>m⁻¹</em>K⁻¹</td>
<td>Kₘₚ Kₓₓ = 0.514 W<em>m⁻¹</em>K⁻¹</td>
<td></td>
</tr>
<tr>
<td>h = 47.1 W<em>m⁻²</em>K⁻¹</td>
<td>h₁ = 34.4 W<em>m⁻²</em>K⁻¹</td>
<td></td>
</tr>
<tr>
<td>D₁ = 1.21×10⁻⁹ m³/s</td>
<td>D₁ = 4.08×10⁻¹⁰ m³/s</td>
<td></td>
</tr>
<tr>
<td>D₂ = 6.25×10⁻¹¹ m³/s</td>
<td>D₂ = 1.63×10⁻¹¹ m³/s</td>
<td></td>
</tr>
<tr>
<td>hₘ = 0.0505 m/s</td>
<td>hₘ₁ = 0.037 m/s</td>
<td></td>
</tr>
<tr>
<td>R = 6.7 mm</td>
<td>R₁ = 5.7 mm</td>
<td></td>
</tr>
<tr>
<td>R₂ = 12.0 mm</td>
<td>R₂ = 12.0 mm</td>
<td></td>
</tr>
<tr>
<td>Xₒ = 5.278 g H₂O/g DM</td>
<td>Xₒ = 3.408 g H₂O /g DM</td>
<td></td>
</tr>
<tr>
<td>Tₒ = 20 °C</td>
<td>Tₒ = 20 °C</td>
<td></td>
</tr>
<tr>
<td>Xₑ = 0.026 g H₂O/g dry air</td>
<td>Xₑ = 0.02 g H₂O/g dry air</td>
<td></td>
</tr>
<tr>
<td>V/Vₒ and water content X</td>
<td>0.2862+0.1228+X</td>
<td></td>
</tr>
<tr>
<td>0.178+0.2297+X</td>
<td>0.178+0.2297+X</td>
<td></td>
</tr>
</tbody>
</table>

* c=1.67+2.5Xₚ (Hallström et al. 1988, p6); L from Hayes (1987, p117); K= 0.58Xₚ+0.155Xₚ+0.25Xₚ+0.16Xₚ+0.135Xₚ (Rao and Rizvi 1995, p132), where Xₚ, Xₚ, Xₚ, Xₚ, and Xₚ are the compositions of cherries and blueberries which were from the (Anonymous 1982); h calculated using equation in Rahman (1995, p405); hₚ calculated using equation in Hallström et al. (1988, p90).
Fig. 3.4 The two-dimensional axisymmetric element grid of cherry (a) and blueberry (b) in air drying.
4. RESULTS AND DISCUSSION

4.1 Moisture sorption

4.1.1 Moisture sorption isotherms

The mean equilibrium moisture contents (g H₂O/100 g dry matter) for freeze dried, osmo-freeze dried, and osmo-air dried cherries, at 10, 25, and 40 °C are given in Table 4.1 and the values for blueberries are given in Table 4.2. Moisture sorption isotherms for sweet cherries and blueberries at 40 °C are shown in Fig. 4.1 (a) and (b), respectively. They show the typical relationship of high-sugar foods, with equilibrium moisture contents (EMC) increasing sharply at high water activities (Mazza 1984; Saravcos et al. 1986; Maroulis et al. 1988; Tsami et al. 1990). The standard deviation varied significantly with water activity. Large deviations were found at high water activities because of the difficulty in determining constant equilibrium moisture content. Similar results were reported by Maroulis et al. (1988).

The equilibrium moisture content (EMC) of osmo-freeze dried cherries was slightly higher than that of fresh freeze dried cherries but the difference was small (Tables 4.1 and 4.2). The EMCs of osmo-air dried cherries were greater at 10 and 25 °C and lower at 40 °C than fresh freeze dried or osmo-freeze dried cherries (Tables 4.1 and 4.2).
Table 4.1 Mean equilibrium moisture content (g H₂O/100g dry matter) of cherries equilibrated at relative humidities in the range of 11 to 96 % at 10, 25, and 40 °C

<table>
<thead>
<tr>
<th>ERH* (%)</th>
<th>Freeze dried Mean</th>
<th>Freeze dried S.D.</th>
<th>Osmo-freeze dried Mean</th>
<th>Osmo-freeze dried S.D.</th>
<th>Osmotic-air dried Mean</th>
<th>Osmotic-air dried S.D.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10 °C</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>4.9 b</td>
<td>0.37</td>
<td>6.6 b</td>
<td>0.49</td>
<td>10 a</td>
<td>1.84</td>
</tr>
<tr>
<td>23</td>
<td>5.7 b</td>
<td>0.18</td>
<td>6.3 b</td>
<td>0.20</td>
<td>11 a</td>
<td>1.27</td>
</tr>
<tr>
<td>34</td>
<td>8 b</td>
<td>2.00</td>
<td>6.8 b</td>
<td>0.61</td>
<td>11 a</td>
<td>1.53</td>
</tr>
<tr>
<td>57</td>
<td>14.7 a</td>
<td>0.75</td>
<td>13.7 a</td>
<td>0.21</td>
<td>13 a</td>
<td>2.53</td>
</tr>
<tr>
<td>68</td>
<td>18 b</td>
<td>1.60</td>
<td>29 a</td>
<td>3.74</td>
<td>25 a</td>
<td>1.67</td>
</tr>
<tr>
<td>76</td>
<td>38 a</td>
<td>1.30</td>
<td>40 a</td>
<td>3.46</td>
<td>34 a</td>
<td>4.25</td>
</tr>
<tr>
<td>87</td>
<td>56 a</td>
<td>5.07</td>
<td>57 a</td>
<td>1.56</td>
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<td>5.89</td>
<td>96 a</td>
<td>2.15</td>
</tr>
</tbody>
</table>

* Equilibrium Relative Humidity.
† Standard deviation, n=3.
‡ Means followed by the same letters (a, b, c) for individual equilibrium relative humidity with each temperature are not significantly different (P > 0.05). Duncan's multiple range test.
Table 4.2 Mean equilibrium moisture content (g H₂O/100g dry matter) of blueberries equilibrated at relative humidities in the range of 11 to 96% at 10, 25, and 40 °C

<table>
<thead>
<tr>
<th>ERH* (%)</th>
<th>Freeze dried</th>
<th>Osmo-freeze dried</th>
<th>Osmotic-air dried</th>
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<td>Mean</td>
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<tr>
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<td>0.38</td>
<td>5.4 ab</td>
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<td>0.38</td>
<td>6.8 a</td>
</tr>
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<td>0.34</td>
<td>9.0 a</td>
</tr>
<tr>
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<td>19.2 a</td>
</tr>
<tr>
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<td>0.79</td>
<td>27.4 a</td>
</tr>
<tr>
<td>76</td>
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<td>0.73</td>
<td>33 b</td>
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<td>55 a</td>
</tr>
<tr>
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<td>77 a</td>
<td>2.72</td>
<td>75 a</td>
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<td></td>
</tr>
<tr>
<td>11</td>
<td>3.0 b</td>
<td>0.12</td>
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<td>5.5 a</td>
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<td>32.4 b</td>
</tr>
<tr>
<td>84</td>
<td>48 a</td>
<td>1.77</td>
<td>48 a</td>
</tr>
<tr>
<td>94</td>
<td>73 a</td>
<td>4.62</td>
<td>73 a</td>
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<td>40 °C</td>
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<td>0.22</td>
<td>2.6 a</td>
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<td>4.2 a</td>
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<td>0.40</td>
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</tr>
<tr>
<td>89</td>
<td>70 a</td>
<td>2.25</td>
<td>71 a</td>
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</table>

* Equilibrium Relative Humidity.
† Standard deviation, n=3.
¶ Means followed by the same letters (a, b, c) for individual equilibrium relative humidity with each temperature are not significantly different (P > 0.05). Duncan's multiple range test.

Figures 4.2 and 4.3 showed that at low water activities, the equilibrium moisture contents of the fruits were lower at higher temperatures. These results are in agreement with the general characteristics of food isotherms which show a stronger water binding (increased heat of sorption) at lower temperatures. However, at high water activities,
equilibrium moisture contents were higher at higher temperatures, as usually occurs only in high sugar foods. This will result in a crossing point of moisture sorption isotherms at different temperatures. The crossing points of the isotherms have also been reported for raisins (Saravcos et al. 1986), figs, prunes, and apricots (Maroulis et al. 1988; Tsami et al. 1990). Significantly increased solubility of sugars at higher temperature would result in an increased equilibrium moisture content, off-setting the opposite effect of temperature on the sorption of non-sugar solids.

The equilibrium moisture contents - water activity values for freeze dried blueberries at 25 °C from the present study are compared with data of Lim et al. (1995) in Fig. 4.4. There is an excellent agreement between the two sets of data for the common range of the isotherm.
Fig. 4.1 Moisture sorption isotherms of sweet cherries (a) and blueberries (b) at 40 °C. OD: osmotic dehydration; OD+AD: osmotic dehydration followed by air drying.

Fig. 4.2 Moisture sorption isotherms of freeze dried cherries (a) and osmo-air dried cherries (b) at 10, 25, and 40 °C. The lines represent the best fits. 
- 10 °C; 25 °C; 40 °C, the lines:  25 °C; —— 40 °C.
Fig. 4.3 Moisture sorption isotherms of freeze dried blueberries (a) and osmo-freeze dried blueberries (b) at 10, 25, and 40 °C. The lines represent the best fits. ◆10 °C; ○ 25 °C; △ 40 °C, the lines: --- 10 °C; ----- 25 °C; —— 40 °C.

Fig. 4.4 Comparison of sorption isotherms of freeze dried blueberries with published data (25 °C).
4.1.2 Equilibrium moisture content - equilibrium relative humidity equations

The coefficients of the modified equations of Henderson, Chung-Pfost, Halsey, Oswin, and GAB are given in Tables 4.3 and 4.4 for cherries and blueberries, respectively. Also given in the Tables are the associated mean relative percent error (P), standard error of moisture content (SEM) and results of the residual plots. Among the five equations, the modified Henderson equation predicts the isotherms with the smallest SEM and P value. The SEM values for the Modified Halsey, Oswin, and Chung-Pfost were, however very high. Also, residuals from the Modified Chung-Pfost equation were patterned. The experimental EMC data for the freeze dried cherries and blueberries at 40 °C were compared with the predicted EMC data using the five equations in Fig. 4.5. None of the equations described the EMC data for the entire range of equilibrium relative humidity (ERH). The modified Henderson equation was a good predictor at \( a_w < 0.8 \); for \( a_w < 0.5 \), the modified Oswin and Halsey equations were also good; however, for \( a_w > 0.8 \), the Halsey equation was the best model.
Table 4.3 Coefficients of modified equations of Henderson, modified Chung-Pfost, modified Halsey, modified Oswin, and modified GAB for freeze dried, osmo-freeze dried, osmo-air dried cherries equilibrated at 11 to 96 % relative humidity and at 10, 25, and 40 °C

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<tr>
<th>Coefficient</th>
<th>Equation</th>
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<th>Modified Chung-Pfost</th>
<th>Modified Halsey</th>
<th>Modified Oswin</th>
<th>Modified GAB</th>
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* SEM = $\sqrt{\frac{\sum (\text{Predicted EMC} - \text{Measured EMC})^2}{\text{Degrees of freedom of regression model}}}$

** p = $\frac{100 \sum (\text{Predicted EMC} - \text{Measured EMC})^2}{\text{No. of data point} \cdot \text{Degrees of freedom of regression model}}$
Table 4.4 Coefficients of modified equations of Henderson, modified Chung-Pfost, modified Halsey, modified Oswin, and modified GAB for freeze dried, osmo-freeze dried, osmo-air dried blueberries equilibrated at 11 to 96% relative humidity and at 10, 25, and 40 °C

<table>
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<th>Coefficient</th>
<th>Modified Henderson</th>
<th>Modified Chung-Pfost</th>
<th>Modified Halsey</th>
<th>Modified Oswin</th>
<th>Modified GAB</th>
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</table>

* $SEM = \sqrt{\frac{\sum \text{(Predicted EMC - Measured EMC)}^2}{\text{Degrees of freedom of regression model}}}$

** $p = \frac{100 \sum \text{(Predicted EMC - Measured EMC)}^2}{\text{No. of data point} \times \text{Degrees of freedom of regression model}}$
Fig. 4.5 EMC of freeze dried cherries (a) and blueberries (b) compared with predicted values obtained using modified equations of Henderson, Chung-Pfost, Halsey, Oswin, and GAB at 40 °C.
4.2 Mass transfer

4.2.1 Osmotic dehydration of cherries and blueberries

Optimized conditions for osmotic dehydration of cherries and effects of various skin treatments on osmotic dehydration of cherries and blueberries are discussed below.

4.2.1.1 Optimized conditions for osmotic dehydration of cherries

The regression results for 'Bing' sweet cherries, and 'Montmorency' and 'Erdi Botermo' sour cherries are summarized in Tables 4.5, 4.6, and 4.7, respectively. The models for water content of cherries, water losses, and mass reductions fit well to the experimental data ($R^2 > 0.85$, Tables 4.5, 4.6, and 4.7).

The models of 'Montmorency' cherries gave the best results according to the total regression coefficient ($R^2 > 0.93$) and the relationships were linear. The relatively small size (averaging 18 mm in diameter) of this cherry cultivar in comparison to 'Bing' (averaging 24 mm in diameter) and 'Erdi Botermo' cherries (averaging 20 mm in diameter) increased the surface area to mass ratio and shortened the distance for diffusion of water and sucrose. This physical characteristic probably resulted in increased mass transfer during osmotic dehydration of 'Montmorency' cherries. Its high acidity may also have had a positive effect on osmotic dehydration. The pH change of sugar solutions during dehydration was 0.73 pH unit (from about 4.3 - 3.5 to about 3.5 - 3.15) on average. In contrast, the pH change of sugar solutions during dehydration of 'Bing' and 'Erdi Botermo' were 0.48 pH unit (from about 4.95 - 4.65 to 4.45 - 4.12) and 0.58 (from
about 4.5 - 4.1 to 3.9 - 3.4) on average, respectively. Lewicki and Mazur (1994) indicated that lower pH of dehydration solution increases water flux and diffusion coefficient.

The results of this study generally show that mass transfer increased with increasing dehydration temperature, time, and concentration of sugar solution. However, the effect of solution concentration was not as large as the influences of time and temperature (Figs. 4.6 and 4.7). These results are in agreement with those of Saurel et al. (1994) who found that water loss is influenced mainly by processing time, and to a lesser extent by temperature and concentration of the dehydration solution. The data in Tables 4.5, 4.6, and 4.7 show that an increase in temperature had no significant influence on solids gain. Raoult-Wack et al. (1991) also found that in model gels, an increase in temperature produces an increase in the rate of water loss, but has no influence on solids gain.

The data for solids gain by ‘Bing’ cherries during dehydration did not adequately fit the polynomial model although water loss data were fitted very well (Table 4.5). This suggests that the solids gain of cherries was also affected by factors not investigated in this study. Similar results were obtained for solids gain in ‘Erdi Botermo’ cherries. Therefore, they were only used to observe trends.

The water loss was relatively low. For ‘Montmorency’ cherries dehydrated at 45 °C, 65 °Brix for 4.5 h, the moisture content was reduced from 84.9 to 69.1 % (wb). Reductions of moisture of ‘Bing’ and ‘Erdi Botermo’ cherries were even less pronounced. For ‘Bing’ cherries the moisture content decreased from 78.5 to 70.2 % and
for ‘Erdi Botermo’ from 77.9 to 68.3 %. A major hindrance for a higher moisture loss was the relatively small surface area available for moisture transfer of the pitted cherries as compared to non-skin cherries. Giangiacomo et al. (1987) reported that in addition to structure and compactness of the tissues, surface exchange area has a major influence on osmotic dehydration rates of pitted cherries and cut peaches. These authors found that after 6 h of osmotic dehydrated in 70 °Brix, 50 % (w/w) corn syrup/sucrose solution, the dry matter content of peaches increased by 94.6 % as compared to 53.3 % for cherries. Experiments carried out in our laboratory have shown that whole ‘Bing’ cherries subjected to osmotic dehydration at 45 °C and 65 °Brix for 5 h lost only 1 % moisture (data not shown).

The favorable conditions for each dependent variable are summarized in Tables 4.5, 4.6, and 4.7. There were differences among the cultivars, but the trends were similar. Overall, optimum concentration of sugar solution for osmotic dehydration of cherries were from 60 to 68 °Brix. The optimum temperature ranged from 40 to 50 °C, and the dehydration time was about 5 h.
Table 4.5 Regression analysis for osmotic dehydration of 'Bing' sweet cherries.

<table>
<thead>
<tr>
<th></th>
<th>Water content (WC g/g DM)</th>
<th>Solids gain (SG g/g DM)</th>
<th>Water loss (WL g/g DM)</th>
<th>Mass reduction (MR %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Linear</td>
<td>0.69***</td>
<td>0.34</td>
<td>0.82***</td>
<td>0.926***</td>
</tr>
<tr>
<td>Quadratic</td>
<td>0.16*</td>
<td>0.20</td>
<td>0.11**</td>
<td>0.018***</td>
</tr>
<tr>
<td>Interactions</td>
<td>0.04</td>
<td>0.06</td>
<td>0.04*</td>
<td>0.051***</td>
</tr>
<tr>
<td>Total regression</td>
<td>0.89***</td>
<td>0.60</td>
<td>0.97***</td>
<td>0.995***</td>
</tr>
</tbody>
</table>

Parameter

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<th>b_3</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>5.51</td>
<td>-0.76</td>
<td>-0.024</td>
<td>0.10</td>
<td>-1.1x10^{-3}***</td>
<td>-0.55x10^{-3}</td>
<td>-5.2x10^{-3}</td>
<td>0.67x10^{-3}</td>
<td>0.88x10^{-3}</td>
<td>0.11x10^{-3}</td>
</tr>
</tbody>
</table>

Lack of fit

| Lack of fit | 0.70 | 0.73 | 0.61 | 0.13 |

Favorable condition

<table>
<thead>
<tr>
<th>Conc. (°Brix)</th>
<th>Temp. (°C)</th>
<th>Time (h)</th>
</tr>
</thead>
<tbody>
<tr>
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<td>M</td>
<td>M - H</td>
</tr>
<tr>
<td>M</td>
<td>L or H</td>
<td>H</td>
</tr>
<tr>
<td>M</td>
<td>M</td>
<td>H</td>
</tr>
<tr>
<td>M - H</td>
<td>H</td>
<td>H</td>
</tr>
</tbody>
</table>

* 0.05 < p < 0.1, possibly significant; ** 0.01 < p < 0.05, significant; *** p < 0.01, highly significant.

* On sample dry matter basis; * On initial dry matter basis; * On fresh sample basis.

* L, M, and H indicate relatively low, medium, and high condition in the experimental range, respectively.
Table 4.6 Regression analysis for osmotic dehydration of 'Montmorency' sour cherries.

<table>
<thead>
<tr>
<th></th>
<th>Water content (WC g/g DM)&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Solids gain (SG g/g DM)&lt;sup&gt;b&lt;/sup&gt;</th>
<th>Water loss (WL g/g DM)&lt;sup&gt;b&lt;/sup&gt;</th>
<th>Mass reduction (MR %)&lt;sup&gt;c&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>R&lt;sup&gt;2&lt;/sup&gt;</strong></td>
<td>0.93***</td>
<td>0.89***</td>
<td>0.92***</td>
<td>0.88***</td>
</tr>
<tr>
<td>Linear</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Quadratic</td>
<td>0.03</td>
<td>0.03</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>Interactions</td>
<td>0.003</td>
<td>0.01</td>
<td>0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>Total regression</td>
<td>0.97***</td>
<td>0.94***</td>
<td>0.95***</td>
<td>0.93***</td>
</tr>
</tbody>
</table>

**Parameter**

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<th>b&lt;sub&gt;2&lt;/sub&gt;</th>
<th>b&lt;sub&gt;3&lt;/sub&gt;</th>
<th>b&lt;sub&gt;11&lt;/sub&gt;</th>
<th>b&lt;sub&gt;22&lt;/sub&gt;</th>
<th>b&lt;sub&gt;33&lt;/sub&gt;</th>
<th>b&lt;sub&gt;12&lt;/sub&gt;</th>
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<th>b&lt;sub&gt;23&lt;/sub&gt;</th>
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<tr>
<td></td>
<td>15.77</td>
<td>-4.05</td>
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<td>-0.097</td>
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</tr>
<tr>
<td></td>
<td>-0.25</td>
<td>0.10</td>
<td>-0.063</td>
<td>-2.21</td>
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<td></td>
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<td></td>
</tr>
<tr>
<td></td>
<td>-0.77</td>
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<td>-0.15</td>
<td>-4.68</td>
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<tr>
<td></td>
<td>0.71x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>-8.80x10&lt;sup&gt;-4&lt;/sup&gt;</td>
<td>-0.55x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>-7.2x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.7x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>-0.69x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>0.55x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>0.017</td>
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</tr>
<tr>
<td></td>
<td>0.057**</td>
<td>-0.011*</td>
<td>-8.2x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>0.023</td>
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<tr>
<td></td>
<td>0.43x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>-0.29x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>0.30x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>6.5x10&lt;sup&gt;-3&lt;/sup&gt;</td>
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<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>-2.9x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>0.40x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>5.0x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>0.061</td>
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<td></td>
</tr>
<tr>
<td></td>
<td>1.8x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>-1.9x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>4.3x10&lt;sup&gt;-3&lt;/sup&gt;</td>
<td>0.077</td>
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</tbody>
</table>

Lack of fit | 0.35 | 0.18 | 0.47 | 0.53 |

**Favorable condition**<sup>d</sup>

<table>
<thead>
<tr>
<th>Conc. (°Brix)</th>
<th>M - H</th>
<th>M</th>
<th>M - H</th>
<th>M - H</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temp. (°C)</td>
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<td>M - H</td>
<td>H</td>
<td>H</td>
</tr>
<tr>
<td>Time (h)</td>
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<td>H</td>
<td>H</td>
<td>H</td>
</tr>
</tbody>
</table>

* 0.05 < p < 0.1, possibly significant; ** 0.01 < p < 0.05, significant; *** p < 0.01, highly significant.

<sup>a</sup> On sample dry matter basis; <sup>b</sup> On initial dry matter basis; <sup>c</sup> On fresh sample basis.

<sup>d</sup> L, M, and H indicate relatively low, medium, and high condition in the experimental range, respectively.
Table 4.7 Regression analysis for osmotic dehydration of ‘Erdi Botermo’ sour cherries.

<table>
<thead>
<tr>
<th>R²</th>
<th>Water content (WC g/g DM)</th>
<th>Solids gain (SG g/g DM)</th>
<th>Water loss (WL g/g DM)</th>
<th>Mass reduction (MR %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Linear</td>
<td>0.64***</td>
<td>0.43**</td>
<td>0.77***</td>
<td>0.82***</td>
</tr>
<tr>
<td>Quadratic</td>
<td>0.16</td>
<td>0.27*</td>
<td>0.09</td>
<td>0.06</td>
</tr>
<tr>
<td>Interactions</td>
<td>0.05</td>
<td>0.06</td>
<td>0.04</td>
<td>0.03</td>
</tr>
<tr>
<td>Total regression</td>
<td>0.85***</td>
<td>0.77*</td>
<td>0.90***</td>
<td>0.91***</td>
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</table>

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<th>$b_{11}$</th>
<th>$b_{21}$</th>
<th>$b_{31}$</th>
<th>$b_{12}$</th>
<th>$b_{22}$</th>
<th>$b_{32}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Linear</td>
<td>-7.66</td>
<td>-0.081</td>
<td>0.42</td>
<td>0.043</td>
<td>0.74×10⁻³</td>
<td>-0.38×10⁻³</td>
<td>0.021</td>
<td>0.68×10⁻³</td>
<td>-6.3×10⁻³</td>
<td>-1.8×10⁻³</td>
</tr>
<tr>
<td>Quadratic</td>
<td>2.89*</td>
<td>8.0×10⁻¹</td>
<td>-0.10*</td>
<td>0.010</td>
<td>-87.0×10⁻⁶</td>
<td>0.93×10⁻³**</td>
<td>-3.7×10⁻⁴</td>
<td>-0.10×10⁻³</td>
<td>1.0×10⁻¹</td>
<td>-1.5×10⁻³</td>
</tr>
<tr>
<td>Interactions</td>
<td>4.88</td>
<td>0.065</td>
<td>-0.20</td>
<td>-0.11</td>
<td>-0.59×10⁻³</td>
<td>1.8×10⁻¹</td>
<td>-0.011</td>
<td>-0.41×10⁻¹</td>
<td>4.0×10⁻³</td>
<td>2.8×10⁻³</td>
</tr>
</tbody>
</table>

| Lack of fit | 0.24 | 0.60 | 0.14 | 0.16 |

Favorable condition:

<table>
<thead>
<tr>
<th></th>
<th>Conc. (°Brix)</th>
<th>Temp. (°C)</th>
<th>Time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concentration</td>
<td>H</td>
<td>L - M</td>
<td>M - H</td>
</tr>
<tr>
<td>Temperature</td>
<td>M</td>
<td>M - H</td>
<td>M - H</td>
</tr>
<tr>
<td>Time</td>
<td>M - H</td>
<td>H</td>
<td>H</td>
</tr>
</tbody>
</table>

* 0.05 < p < 0.1, possibly significant; ** 0.01 < p < 0.05, significant; *** p < 0.01, highly significant.

*On sample dry matter basis; *On initial dry matter basis; *On fresh sample basis.

L, M, and H indicate relatively low, medium, and high condition in the experimental range, respectively.
Fig. 4.6 Response surface of water loss for pitted 'Bing' sweet cherries (a), 'Montmorency' (b), and 'Erdi Botermo' (c) sour cherries at 3 h of dehydration time.
Fig. 4.7 Response surface of water loss for pitted 'Bing' sweet cherries (a), 'Montmorency' (b), and 'Erdi Botermo' (c) sour cherries at 35 °C.
4.2.1.2 Effect of skin treatment on osmotic dehydration

The process of osmotic dehydration was conducted at 45 °C in 65 °Brix sucrose solution for 5 h after various skin treatments.

Among the four skin treatments applied to cherries and blueberries, steam blanching, ethyl myristate treatment, and hot water dipping had a similar effect on water removal. The lipase treatment had little effect (Fig. 4.8). However, the hot water treatment damaged the cherry surface layer, causing small cracks to appear in the skin tissue. During dehydration, red color leached out and the fruits became soft, apparently because of cell damage. For the ethyl myristate treatment, it was difficult to remove the solvent from the fruit surface after treatment. The solvent residue on the fruit surface would subsequently contaminate the osmotic solution. That would in turn cause a potential problem for reuse of the osmotic solution after dehydration.

The moisture diffusion coefficients of cherries and blueberries during osmotic dehydration are listed in Table 4.8. The moisture diffusion coefficients are comparable to values published for similar systems. Ertekin and Cakaloz (1996) found that the apparent diffusion coefficient of water in osmotic dehydration of pea was in the range 9.10×10⁻¹¹ to 7.63×10⁻¹⁰ m²/s depending on the composition of osmotic solution. Hough et al. (1993) reported that the diffusivity at 45 °C for osmotic dehydration of apple slices using 55 % glucose syrup was 1.9×10⁻¹⁰ m²/s. The moisture diffusivity values for both steam blanched cherries and blueberries were about 3.6 times higher than that of the fresh fruit (Table 4.8). The diffusivity values of the lipase treated samples were only 1.8 times higher than that of the fresh fruit. These results show that steaming (137.9 KPa) cherries
and blueberries for 30 s is a simple and effective method for improving the mass transfer during the osmotic dehydration of waxy fruits.

Table 4.8 Moisture diffusion coefficients of cherries and blueberries during osmotic dehydration in 65 °Brix sucrose at 45 °C.

<table>
<thead>
<tr>
<th>Skin treatment</th>
<th>Diffusivity (m²/s)</th>
<th>Cherries (whole)</th>
<th>Blueberries</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fresh</td>
<td>2.19x10⁻¹¹</td>
<td>1.75x10⁻¹¹</td>
<td></td>
</tr>
<tr>
<td>Lipase</td>
<td>4.04x10⁻¹¹</td>
<td>3.35x10⁻¹¹</td>
<td></td>
</tr>
<tr>
<td>Ethyl myristate</td>
<td>7.54x10⁻¹¹</td>
<td>6.03x10⁻¹¹</td>
<td></td>
</tr>
<tr>
<td>Steam blanched</td>
<td>8.39x10⁻¹¹</td>
<td>6.31x10⁻¹¹</td>
<td></td>
</tr>
<tr>
<td>Hot water</td>
<td>9.63x10⁻¹¹</td>
<td>--</td>
<td></td>
</tr>
</tbody>
</table>

-- not measured.

Fig. 4.8 Effect of skin treatment on moisture content of whole cherries (a) and blueberries (b) osmotically dehydrated in 65 °Brix sucrose at 45 °C. The lines represent the best fits.
4.2.2 Air drying

In this section the influence of osmotic dehydration pretreatment on subsequent air drying rate is discussed.

4.2.2.1 Drying curves and rates

The air drying curves of cherries and blueberries at 80 °C are shown in Figs. 4.9 and 4.10, respectively. The differences in initial moisture content reflected the varying degrees of water loss in the osmotic pretreatments. Osmotic pretreatment decreased moisture content of pitted fresh cherries from 3.4 to 2.0 g H₂O/g DM and whole blueberries from 5.3 to 4.9 g H₂O/g DM, respectively. Initially, the moisture content in fresh samples directly subjected to air drying (AD) dropped more rapidly than that of the samples subjected to osmotic dehydration pretreatment.

The drying rates are shown in Figs. 4.11 and 4.12, respectively. No constant rate period was observed at air drying temperatures of 60 and 80 °C. The steam blanching of cherries prior to osmotic dehydration increased the rate of air drying (Fig. 4.11). Initial drying rates in fresh samples (AD) were higher than that of the samples subjected to osmotic pretreatment (OD+AD), but the drying rates gradually approached each other with drying. Sankat et al. (1996) obtained similar result for banana slices. The drying behaviour of blueberries, however, was different from that of cherries. At 80 °C, all blueberry samples, irrespective of the treatment, showed a drying rate and a pattern similar to that reported for other food products (Mazza and LeMaguer 1980; Mazza 1984; Roman et al. 1979). At 60 and 40 °C, the rate of drying was substantially lower.
than that of 80 °C and, contrary to expectation, the osmotically dehydrated samples had a higher drying rate than the fresh sample.

The strong influence of drying air temperature is further illustrated in Fig. 4.13, which clearly shows that drying rate at 80 °C was significantly higher than that of drying at 40 and 60 °C.

![Graph showing drying curves](image_url)

Fig. 4.9 Air drying curves of fresh and osmotically dehydrated pitted cherries at 80 °C. The lines represent the best fits. AD: air drying; OD+AD: osmotic dehydration followed by air drying; S+OD+AD: steam blanched followed by osmotic dehydration and air drying.
Fig. 4.10 Air drying curves of fresh and osmotically dehydrated blueberries at 80 °C. The lines represent the best fits. AD: air drying; OD+AD: osmotic dehydration followed by air drying; S+OD+AD: steam blanched followed by osmotic dehydration and air drying.

Fig. 4.11 Drying rate curves of pitted cherries. The lines represent the best fits. AD: air drying; OD+AD: osmotic dehydration followed by air drying; S+OD+AD: steam blanched followed by osmotic dehydration and air drying.
Fig. 4.12 Drying rate curves of blueberries. The lines represent the best fits. 
AD: air drying; OD+AD: osmotic dehydration followed by air drying; S+OD+AD: steam blanched followed by osmotic dehydration and air drying.

Fig. 4.13 Effect of air drying temperature on drying rate of fresh pitted cherries (a) and blueberries (b). The lines represent the best fits.
4.2.2.2 Moisture diffusion coefficient

Table 4.9 show that steam blanched samples had higher moisture diffusivity values in air drying, compared to those of non blanched samples. But, with increasing drying air temperature, the differences become relatively small. It also shows (Table 4.9) that the osmotic pretreatment did not change the D values for cherries significantly; but for blueberries, a higher D value was obtained for osmotically pretreated sample at lower temperatures of 40 and 60 °C. However, at the higher temperature (80 °C), the trends for blueberries were reversed, and the result became consistent with the results of Mazza (1983) and Sankat et al. (1996), who found a lower rate of moisture transport in foodstuffs subjected to osmotic treatment. At low temperature air drying of blueberries, the sugar on the skin surface is not dried immediately and serves as a barrier to water diffusion. On the contrary, it acts as a drive force to draw water from the interior fruit. This may be the cause for the increased D value at 40 °C for air drying of osmotically treated blueberries.

Table 4.9 Moisture diffusion coefficients of pitted cherries and blueberries during air drying at 40, 60, and 80 °C and 2.0 m/s air flow rate.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Diffusivity (m²/s)</th>
<th>Cherries (pitted)</th>
<th>Blueberries</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Air dried</td>
<td>Osmo-air dried</td>
<td>Steamed - osmo-air dried</td>
</tr>
<tr>
<td>40</td>
<td>0.90×10⁻¹⁰</td>
<td>0.79×10⁻¹⁰</td>
<td>1.08×10⁻¹⁰</td>
</tr>
<tr>
<td>60</td>
<td>1.88×10⁻¹⁰</td>
<td>1.69×10⁻¹⁰</td>
<td>2.20×10⁻¹⁰</td>
</tr>
<tr>
<td>80</td>
<td>4.08×10⁻¹⁰</td>
<td>4.08×10⁻¹⁰</td>
<td>4.64×10⁻¹⁰</td>
</tr>
</tbody>
</table>
The natural logarithm of D values obtained at 40, 60, and 80 °C for both fruits were plotted against the inverse of absolute temperature (1/T) and the results are presented in Fig. 4.14 (a) and (b) for cherries and blueberries, respectively. Energy of activation (E<sub>a</sub>) for diffusion was estimated using an Arrhenius type equation:

\[ D = Ae^{-E_a/RT} \]  

(31)

The slope of the straight line is equal to E<sub>a</sub>/R. The values of E<sub>a</sub> for cherries and blueberries are listed in Table 4.10. The steam blanching pretreatment reduced the E<sub>a</sub> value for both cherries and blueberries. Osmotic dehydration treatment slightly increased E<sub>a</sub> for cherries but decreased E<sub>a</sub> for blueberries. Water movement out of blueberries appears to be limited by the skin. The fruit skin is a compact fibrous material coated by a waxy layer and does not allow fast mass transfer through it. This is confirmed by the much higher E<sub>a</sub> values for blueberries as listed in Table 4.10.

Fig. 4.14 Effect of temperature on the diffusivity of moisture through cherries (a) and blueberries (b) during air drying. The lines represent the best fits. AD: air drying; OD+AD: osmotic dehydration followed by air drying; S+OD+AD: steam blanched followed by osmotic dehydration and air drying.
Table 4.10 Energy of activation for diffusion of water through cherries and blueberries.

<table>
<thead>
<tr>
<th></th>
<th>Activation energy for diffusion (J/mol)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Air dried Osmo-air dried Steamed - osmo-air dried</td>
</tr>
<tr>
<td>Cherries (pitted)</td>
<td>34640 37662 33533</td>
</tr>
<tr>
<td>Blueberries (whole)</td>
<td>90049 75140 74521</td>
</tr>
</tbody>
</table>

4.3 Product quality

4.3.1 Anthocyanin Pigments

The value of total anthocyanin content of whole cherries and blueberries subjected to osmotic dehydration following various skin pretreatments are given in Table 4.11. All samples lost anthocyanin with drying. The hot water treatment (90 °C, 30 s) process caused a large anthocyanins loss during the pretreatment and subsequent osmotic dehydration operation. The steaming treatment also caused some degree of anthocyanins loss, especially in blueberries. Because most of the anthocyanins in blueberries are in the skin layer, skin damage caused by steaming greatly increased the loss of anthocyanins during osmotic dehydration (Table 4.11). For the fresh and steam blanched blueberries, the loss was most pronounced during the first hour of dehydration. Steam blanching caused the approximate same magnitude of anthocyanins loss in fresh cherries, however, the loss of anthocyanin during dehydration was relatively low (from 62.6 to 50.1 mg/100 g). The ethyl myristate pretreatment had the least effect on anthocyanin loss of fruits during osmotic dehydration.

The retentions of anthocyanins as percent of the initial amount in air drying of sweet cherries and blueberries at 60 °C are shown in Figs. 4.15 and 4.16, respectively.
For cherries, osmotic pre-drying reduced the loss of anthocyanins at the high drying temperature. However, osmotic dehydration of blueberries did not show this phenomena. The anthocyanins level were reduced during air drying, and the rate of loss was more rapid for blueberries than for cherries.

Table 4.11 Effect of skin treatments on anthocyanins content (mg/100g fresh) in cherries and blueberries during osmotic dehydration

<table>
<thead>
<tr>
<th>Dehydration time (h)</th>
<th>Fresh</th>
<th>Steam blanched</th>
<th>Lipase</th>
<th>Ethyl myristate</th>
<th>Hot water</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cherries</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0</td>
<td>91.26</td>
<td>62.60</td>
<td>87.52</td>
<td>82.13</td>
<td>59.20</td>
</tr>
<tr>
<td>1</td>
<td>84.04</td>
<td>57.37</td>
<td>66.97</td>
<td>74.33</td>
<td>43.86</td>
</tr>
<tr>
<td>3</td>
<td>72.13</td>
<td>53.24</td>
<td>64.52</td>
<td>73.13</td>
<td>40.31</td>
</tr>
<tr>
<td>5</td>
<td>58.45</td>
<td>50.08</td>
<td>52.08</td>
<td>55.86</td>
<td>29.83</td>
</tr>
<tr>
<td>Blueberries</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0</td>
<td>90.24</td>
<td>88.44</td>
<td>103.86</td>
<td>81.60</td>
<td>--</td>
</tr>
<tr>
<td>1</td>
<td>81.83</td>
<td>66.53</td>
<td>96.66</td>
<td>81.11</td>
<td>--</td>
</tr>
<tr>
<td>3</td>
<td>79.45</td>
<td>62.11</td>
<td>87.35</td>
<td>80.01</td>
<td>--</td>
</tr>
<tr>
<td>5</td>
<td>77.19</td>
<td>56.04</td>
<td>73.07</td>
<td>79.43</td>
<td>--</td>
</tr>
</tbody>
</table>

-- not measured.

Fig. 4.15 Changes in concentration of anthocyanin in air drying of sweet cherries at 60 °C. The lines represent the best fits. AD: air drying; OD+AD: osmotic dehydration followed by air drying; S+OD+AD: steam blanched followed by osmotic dehydration and air drying.
Fig. 4.16 Changes in concentration of anthocyanin in air drying of blueberries at 60 °C. The lines represent the best fits. AD: air drying; OD+AD: osmotic dehydration followed by air drying; S+OD+AD: steam blanched followed by osmotic dehydration and air drying.

Fig. 4.17 Retention of anthocyanins as percent of initial amount in air drying of fresh cherries (a) and blueberries (b) at 80 °C as a function of water content. The line represents the best fit.
Figure 4.17 shows the general trend that with decreasing water content during air
drying, the anthocyanin content also decreased.

From Fig. 4.18, it can be seen that there was a large difference in anthocyanin
retention by fruits that were air dried at 40 and 60 °C. These results are consistent with
that of Raynal et al. (1989), who found that plum anthocyanins decreased very rapidly
during air drying, and more marked by at high temperatures.

![Graph showing effect of temperature on anthocyanin content during air drying](image)

**Fig. 4.18** Effect of temperature on anthocyanin content during air drying of
fresh sweet cherries (a) and blueberries (b). ◆ 80 °C; ▲ 60 °C; × 40 °C.

4.3.2 Shrinkage

Osmotic dehydration reduced the extent of shrinkage in cherries and blueberries
during air drying (Fig. 4.19), apparently because of the solids gained during this
pretreatment. Kim and Toledo (1987) stated that osmotic dehydration prevented
shrinkage of blueberries. They also found that the osmotically pre-dried blueberries had a larger diameter than that of non osmotically dehydrated samples.

![Graph showing shrinkage of cherries and blueberries](image)

**Fig. 4.19** Shrinkage of cherries (a) and blueberries (b) during air drying. AD: air drying; OD+AD: osmotic dehydration followed by air drying; S+OD+AD: steam blanched followed by osmotic dehydration and air drying.

The relationship between shrinkage and water content of fruits was linear (Table 4.12). Sjöholm and Gekas (1995) reported that the volume shrinkage factor with water content for Mutsu apples was: \( S_v = 0.114 + 0.642 X \) with \( R^2 = 0.95 \). A similar result was also reported with Granny Smith apples (Lozano et al. 1980).

**Table 4.12** Relationships of volume shrinkage factor \( S_v \) (\( V/V_0 \)) with water content \( X \) during air drying.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Cherries</th>
<th>Blueberries</th>
</tr>
</thead>
<tbody>
<tr>
<td>AD</td>
<td>( S_v = 0.178 + 0.2297 X ), ( R^2 = 0.986 )</td>
<td>( S_v = 0.286 + 0.1228 X ), ( R^2 = 0.987 )</td>
</tr>
<tr>
<td>OD+AD</td>
<td>( S_v = 0.178 + 0.2432 X ), ( R^2 = 0.981 )</td>
<td>( S_v = 0.298 + 0.1268 X ), ( R^2 = 0.982 )</td>
</tr>
<tr>
<td>S+OD+AD</td>
<td>( S_v = 0.168 + 0.2303 X ), ( R^2 = 0.987 )</td>
<td>( S_v = 0.173 + 0.1454 X ), ( R^2 = 0.990 )</td>
</tr>
</tbody>
</table>
4.3.3 Rehydration

The rehydration curves of cherries and blueberries vs. rehydration time at 25 °C are shown in Figs. 4.20 and 4.21, respectively. Rehydration of osmotically dehydrated fruits was less than that of untreated samples. This is because of the sugar on the dehydrated fruits, which does not rehydrate as readily as the fruit tissue. This would be an advantage for dried cherries and blueberries if they are used in such products as snack foods or breakfast cereal mixtures. With low hygroscopicity of the osmotically dried products, they can be exposed in the open air for several hours without becoming sticky.

![Graph showing rehydration curves](image)

**Fig. 4.20** Rehydration of air dried cherries at 25 °C (dried at 80 °C). The lines represent the best fits. AD: air drying; OD+AD: osmotic dehydration followed by air drying; S+OD+AD: steam blanched followed by osmotic dehydration and air drying.
4.4 Numerical modeling

4.4.1 Osmotic dehydration

Based on the results for mass average water content, drying curves were obtained and then compared with the experimental data of fresh whole cherries and blueberries in osmotic dehydration process, as shown in Fig 4.22. A good agreement between the simulated and experimental results for blueberries was obtained. However, for cherries, the model over-predicted the mass average water content during the final stage of osmotic dehydration. The variations could be attributed largely to the assumption of the constant moisture diffusivity. Zhang et al. (1984) pointed out that mass diffusivity is a function of
concentration; and an error in prediction of moisture can result if diffusivity is assumed to be constant. Also the assumptions of fruits being represented by sphere and the neglect of shrinkage during the osmotic dehydration process are other possible sources of error.

Fig. 4.22 Mass average water content of cherries (a) and blueberries (b) during osmotic dehydration predicted by the finite element method compared with experimental results.

4.4.2 Air drying

Figure 4.23 shows the experimental versus model predictions of drying curves for cherries and blueberries. The models under-predicted the experimental data during the first 1.5 h of drying whereas the blueberry model over-predicted the mass average water content during the last stage of drying (after 2 h). As discussed before, the variations could also be attributed to the assumption of the constant moisture diffusivity.
Water content profiles of cherries and blueberries predicted by models at different radii are plotted in Fig. 4.24. The nodes located on the skin reached equilibrium much faster than the internal nodes. Fig. 4.25 shows the mass average temperature of cherries and blueberries as a function of drying time. There is a very sharp increase in simulated mass average temperature during the first hour of drying. The mass average temperature approached the equilibrium temperature (drying air temperature) in 1-2 h. Haghighi and Segerlind (1988) stated that the sharp increase of the mass average temperature in the first hour of drying suggests high magnitude of temperature gradients, and therefore, high levels of thermal stress during that period. Damage to the fruits, due to high temperature gradients, occurs during the first hour of drying.
In general, the model has the potential of explaining the complicated phenomena of transient heat and mass transfer in biomaterials with different geometries and boundary conditions.

Fig. 4.24 Water content distribution of cherries (a) and blueberries (b) predicted by the finite element models at different radii during air drying ($z = 0$).

Fig. 4.25 Temperature distribution of cherries (a) and blueberries (b) predicted by the finite element models at different radii during air drying ($z = 0$).
4.5 Recovery of pigments

Effects of sucrose concentration and pH on the adsorption of anthocyanins on resins were investigated using Amberlite® CG-50 and Amberlite® XAD-2, respectively. Anthocyanins adsorption decreased with increasing concentration of sucrose. However the effect is relative small (Fig. 4.26). Because sucrose molecules (MW = 342) are much bigger and move slower than water molecules (MW = 18), sugar molecules may block the adsorption-desorption process of anthocyanins. With increasing concentration of sugar, blocking effect increased. The increased viscosity of the solution with increasing concentration of sucrose also reduced the diffusivity of the anthocyanins.

![Graph showing anthocyanin adsorption on Amberlite CG-50 in sucrose solution](image)

**Fig. 4.26** Anthocyanin adsorption on Amberlite® CG-50 in sucrose solution (resin : solution = 10 g : 15 mL), anthocyanin concentration of solution is 25 mg/mL. The line represents the best fit.
Fig. 4.27 Effect of pH on anthocyanin adsorption on Amberlite®XAD-2 in 65 °Brix sucrose solution (resin : solution = 10 g : 15 mL), anthocyanin concentration of solution is 25 mg/mL. The line represents the best fit.

Figure 4.27 shows the effect of pH on the adsorption of anthocyanins to Amberlite®XAD-2. Generally, the level of adsorption was stable over the range of pH 1 to 5. However, adsorption at pH 2 was slightly higher than that at other pHs. It is not clear why anthocyanins are more strongly adsorbed to the resin at pH 2.

Based on the literature, the weakly acidic ion exchange resin Amberlite®CG-50 is the most commonly used adsorbent for recovery or concentration and purification of anthocyanins from fruits such as cranberry pomace (Fuleki and Francis 1968a; Chiriboga and Francis 1970) and sweet cherries (Gao and Mazza 1995). Recently, the non-ionic polystyrene Amberlite®XAD resins were used for separation of phenolics (Stefano and Guidoni 1990) and flavonoids from plant extracts (Tomás-Barberán et al. 1992). Tomás-Barberán et al. (1992) found that Amberlite®XAD-2 was the most suitable for the recovery and fractionation of flavonoids from plant extracts and foods rich in sugars and
other polar compounds such as honey and fruit jams. In this study, the Amberlite® CG-50, XAD-2, XAD-4 as well as three other resins (Amberlite® XAD7HP, XAD16HP, and Duolite® XAD765) were selected to recover anthocyanins from the osmotic media (sucrose solution) for dehydration of cherries. Duolite® XAD765 is an ion exchange adsorbent which has been specifically designed to remove organic impurities from solution by both adsorption and ion exchange (Anonymous 1996a). Amberlite® XAD7HP and XAD16HP are polymeric adsorbents.

The anthocyanin adsorption curves on different resins are shown in Fig. 4.28. Among the resins, Amberlite® XAD16HP and XAD7HP were the most effective: the rate of anthocyanins adsorption onto these two resins reached 98 and 96 %, respectively. The length of time required to reach equilibrium was about 10 to 15 min. For Duolite® XAD765, adsorption rate reached 85 % at 25 to 30 min.

![Anthocyanin adsorption curves](image)

Fig. 4.28 Anthocyanin adsorption curves on resins in 65 °Brix sucrose solution as a function of adsorption time (resin : solution = 10 g : 15 mL); anthocyanin concentration of solution is 25 mg/mL. The lines represent the best fits.
Fig. 4.29 Anthocyanin adsorption on resins in 65 °Brix sucrose solution as a function of ratio of resin to solution, anthocyanin concentration of solution is 25 mg/mL. The lines represent the best fits curves.

The optimum ratio of resin to solution (65 °Brix, anthocyanin concentration of 25 mg/mL) for Amberlite® XAD16HP and XAD7HP was about 0.5 to 0.67 (Fig 4.29). The ratio for other resins was greater than 0.67.

From the results shown above, it can be seen that Duolite® XAD765, Amberlite® XAD16HP and XAD7HP resins were the three most effective resins for adsorbing anthocyanins from the sucrose solution.

Acidified methanol and acidified ethanol are commonly used to elute anthocyanins from adsorbents in column chromatography or solid phase extraction. If the concentration of acetic acid in ethanol was ≤ 5 %, the desorption rate from Amberlite® XAD7HP was greater than 90 % (Fig. 4.30). However, the effectiveness of the eluant
decreased with higher level of acetic acid, probably due to decreased hydrophobicity of the eluant by increased content of acetic acid.

![Graph showing the effect of acetic acid concentration in eluant on anthocyanin desorption from Amberlite® XAD7HP (D_o = desorption at 95% EtOH). The line represents the best fit.](image)

Fig. 4.30 Effect of acetic acid concentration in eluant on anthocyanin desorption from Amberlite® XAD7HP (D_o = desorption at 95% EtOH). The line represents the best fit.

Response surface methodology was used to find the optimum desorption condition and the best resin to recover anthocyanins from sucrose solution. The results of the RSM are listed in Table 4.13 and Fig. 4.31. Among the three resins, Amberlite® XAD16HP gave the best result; with 85.6% ethanol as eluant and 17.6 min of desorption time, the recovery was 68.1. Duolite® XAD765 had the lowest recovery rate (21.3%) among the three resins. In addition to its performance, Amberlite® XAD16HP meets the requirements of FDA 21 CFR 173.65 for removal of organic substances from aqueous foods except carbonated beverages (Anonymous 1996b). Therefore, this resin could be used for recovery of anthocyanins as a food colorant.
Table 4.13 Response surface regression analysis for recovery of anthocyanins from sucrose solution using different resins

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Amberlite® XAD7HP</th>
<th>Amberlite® XAD16HP</th>
<th>Duolite® XAD765</th>
</tr>
</thead>
<tbody>
<tr>
<td>R²</td>
<td>0.851***</td>
<td>0.837***</td>
<td>0.682**</td>
</tr>
<tr>
<td>Linear</td>
<td>0.039</td>
<td>0.008</td>
<td>0.015</td>
</tr>
<tr>
<td>Quadratic</td>
<td>0.898**</td>
<td>0.988***</td>
<td>0.847**</td>
</tr>
<tr>
<td>Interactions</td>
<td></td>
<td></td>
<td>0.015</td>
</tr>
<tr>
<td>Total regression</td>
<td></td>
<td></td>
<td>0.847**</td>
</tr>
<tr>
<td>Parameter</td>
<td></td>
<td></td>
<td>0.015</td>
</tr>
<tr>
<td>b₀</td>
<td>-47.737</td>
<td>-40.415***</td>
<td>-10.428</td>
</tr>
<tr>
<td>b₁</td>
<td>1.946*</td>
<td>2.558***</td>
<td>0.605</td>
</tr>
<tr>
<td>b₂</td>
<td>1.671</td>
<td>-0.112</td>
<td>0.440</td>
</tr>
<tr>
<td>b₁₁</td>
<td>-8.24×10⁻¹</td>
<td>-0.016***</td>
<td>-4.0×10⁻¹</td>
</tr>
<tr>
<td>b₂₂</td>
<td>-0.026</td>
<td>-0.013</td>
<td>-0.016</td>
</tr>
<tr>
<td>b₁₂</td>
<td>-0.010</td>
<td>6.50×10⁻³</td>
<td>3.75×10⁻³</td>
</tr>
<tr>
<td>Lack of fit</td>
<td>0.11</td>
<td>0.74</td>
<td>0.20</td>
</tr>
<tr>
<td>Favorable condition</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ethanol Conc. (%)</td>
<td>95.0</td>
<td>85.6</td>
<td>87.1</td>
</tr>
<tr>
<td>Desorption Time (min)</td>
<td>13.6</td>
<td>17.6</td>
<td>24.4</td>
</tr>
<tr>
<td>Recovery rate (%)</td>
<td>67.6</td>
<td>68.1</td>
<td>21.3</td>
</tr>
</tbody>
</table>

* 0.05 < p < 0.1, possibly significant; ** 0.01 < p < 0.05, significant; *** p < 0.01, highly significant.

Based on these results, optimum conditions for recovery of anthocyanins from the media of osmotic dehydration of cherries are as follows: Amberlite® XAD16HP resin with a ratio of 2:3 immersed in sucrose solution for 30 min for saturation with anthocyanin. The sugar solution devoid of anthocyanins can be recovered by applying the mixture onto a juicer type centrifuge. The anthocyanin pigments can be eluted from resin with spraying 90% ethanol + 5% acetic acid solution. Ethanol is evaporated by a thin film rotary evaporator and the concentrated pigments are freeze dried and collected as dry powder. The resin is re-equilibrated with aqueous 2.5% acetic acid and reused.
Fig. 4.31 Response surface of anthocyanins recovery from (a) Amberlite® XAD7HP, (b) Amberlite® XAD16HP, and (c) Duolite® XAD765.
5. CONCLUSIONS

1. The EMC - ERH of cherries and blueberries showed a relationship typical of high-sugar foods; EMC increased sharply at high ERH. At constant relative humidities, equilibrium moisture content of fruits decreased as temperature increased at low water activities, but increased with temperature at high water activities. These properties result in a crossing point of moisture sorption isotherms at different temperatures.

2. Among the five equations studied, the modified Henderson equation predicted isotherms with the smallest SEM and P values. None of the equations could describe the EMC data for the entire range of ERH. The modified Henderson equation was found to be good at $a_w < 0.8$. For $a_w < 0.5$, the modified Oswin and Halsey equations were also good. However, the Halsey equation was the best one for $a_w > 0.8$.

3. For optimum dehydration of cherries, optimum concentration of sugar solution was 60 to 68 °Brix, optimum temperature fell into the range of 40 - 50 °C, and dehydration time was about 5 h.

4. Osmotic pretreatment decreased moisture content of fresh cherries from 3.5 to 2.1 g/g DM and of blueberries from 5.4 to 5.0 g/g DM, respectively. Steaming (137.9
KPa) cherries and blueberries for 30 s was a simple and effective method for improving mass transfer during osmotic dehydration of waxy fruits. Moisture diffusivity values for both steam blanched cherries and blueberries were > 3.6 times greater than those of the respective fresh fruit.

5. During air drying, the initial drying rate in fresh samples subjected to air drying were higher than that of the samples subjected to osmotic dehydration pretreatment. But, both drying rates gradually approached each other with drying process. There was no constant rate period observed at air drying temperatures of 60 and 80 °C. Steam blanching prior to osmotic dehydration increased subsequent air drying rate. Drying rates were significantly higher at higher air temperatures of 60 and 80 °C.

6. Osmotic pre-drying reduced the loss of anthocyanins at high drying temperatures. Total anthocyanin content decreased during air drying, and the decrease was more rapid for blueberries than for cherries. Osmotic dehydration reduced shrinkage of cherries and blueberries during subsequent air drying as a result of solids gain during the pretreatment. Drying shrinkage was linearly related to water content. Because of sugar uptake during osmotic pretreatment in fruit, rehydration of the osmotically dehydrated fruit was lower than that of directly air dried samples.
7. Numerical models were developed using finite elements to predict the moisture content of cherries and blueberries during osmotic dehydration and air drying. The developed osmotic dehydration models can be used to analyze diffusion in 2-D axisymmetric transient body with different boundary conditions. The air drying models have the potential of explaining complicated couple phenomena of 2-D axisymmetric transient heat and mass transfers in biological materials with shrinkage, different geometries, and varying boundary conditions.

8. Amberlite® XAD16HP resin was the most effective resin for recovering anthocyanins from the high sugar media used in osmotic dehydration of cherries. Optimum process and conditions for recovery of anthocyanins from sucrose solution were: resin immersed in anthocyanin containing sucrose solution for 30 min, then washed with water containing 2.5 % acetic acid and eluted with 90 % ethanol + 5 % acetic acid. The resin was re-equilibrated with aqueous 2.5 % acetic acid and reused. Pigments can be recovered by evaporating ethanol under vacuum followed by freeze or spray drying.
REFERENCES


Wrolstad, R.E. 1976. Color and pigment analyses in fruit products. *Station Bulletin 624*, Agricultural Experiment Station, Oregon State University, Corvallis, OR.


Fig. a.1 EMC of freeze dried cherries compared with predicted values obtained using modified equations of Henderson, Chung-Pfost, Halsey, Oswin, and GAB.
Fig. 3.2 EMC of osmo-freeze dried cherries compared with predicted values obtained using modified equations of Henderson, Chung-Pfost, Halsey, Oswin, and GAB.
Fig. a.3 EMC of osmo-air dried cherries compared with predicted values obtained using modified equations of Henderson, Chung-Pfost, Halsey, Oswin, and GAB.
Fig. a.4 EMC of freeze dried blueberries compared with predicted values obtained using modified equations of Henderson, Chung-Pfost, Halsey, Oswin, and GAB.
Fig. a.5 EMC of osmo-freeze dried blueberries compared with predicted values obtained using modified equations of Henderson, Chung-Pfost, Halsey, Oswin, and GAB.
Fig. a.6 EMC of osmo-air dried blueberries compared with predicted values obtained using modified equations of Henderson, Chung-Pfost, Halsey, Oswin, and GAB.