EFFECTS OF CANOLA PRETREATMENTS ON SORPTION HYSTERESIS AND RHEOLOGICAL BEHAVIOR OF SINGLE KERNELS.

BY

JERZY BIELEWICZ

A thesis
presented to the University of Manitoba
in partial fulfilment of the requirements for the
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ABSTRACT

Equilibrium moisture content (EMC) and creep and recovery tests were conducted for canola seeds (Brassica napus L., cv. Westar). The effects of sample age, origin and pretreatment (rewetting/drying cycles, irradiation and long-term stress) on EMC properties and viscoelasto-plastic behaviour were investigated. Equilibrium moisture content hysteresis was studied by equilibrating rewetted and dried samples together in an air-tight environment at constant temperature of 25^{0}C. The measured EMC values compared well with the published data. The viscoelastic properties of canola were evaluated using rheological models.

A significant difference in viscoelasto-plastic properties, greater than that which can be explained by the EMC loop between adsorption and desorption isotherms was found. The actual difference in average ratio of elasticity between adsorption and desorption samples was approximately 6 times greater than the difference in ratio of elasticity corresponding to the moisture content loop (0.4% db) for the canola kernels equilibrated at 67% relative humidity (RH).

Adsorption and desorption isotherms obtained for three different samples of different age and origin differed substantially. The difference in EMC between two adsorption samples harvested at different locations was approximately 1.3% db at 23% RH. A discrepancy in instantaneous loading deformation of 13% was attributed to the difference in age and

growing history of two canola samples at 8.5% db MC.

The rewetting/drying pretreatment resulted in a significant mold development. The adsorption and desorption isotherms (EMC - ERH curves) shifted down as a result of the pretreatment. The hysteresis loop widened for low and intermediate moisture contents. The EMC hysteresis loop widened 2.5 times as a result of the pretreatment for samples equilibrated with air at approximately 25% RH. The pretreatment increased instantaneous loading deformation by 35% over the predicted value due to the EMC difference alone for samples equilibrated at 42% RH.

The EMC behaviour was not affected by irradiation or long-term stress. Long-term stress, however, had significant effects on the viscoelastic properties of the canola. Instantaneous loading deformation increased approximately 22% as a result of this pretreatment for samples equilibrated at 58% RH.

The viscoelasto-plastic behaviour of the canola kernels during creep and recovery tests was explained using a rheological model consisting of a plastic component in series with a viscoelastic component.

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LIST OF SYMBOLS

a = water activity, decimal

d = kernel diameter, m

D = contact area diameter, m

δ = error, %

Δ = error

E_0 = modulus of elasticity of a first elastic element in Burgers model, Pa

E_r = modulus of elasticity of a second elastic element in Burgers model, Pa

$\varepsilon(t)$ = strain mm/mm

ε_c = strain dissipated during the entire creep,

ε_D = dissipated strain, mm/mm

ε_L = strain dissipated during instantaneous loading, mm/mm

F = load, N

φ = number of homogeneous phases

$\Phi(\sigma)$ = stress function

G = Gibbs function, J

$J(\sigma,t)$ = creep compliance - strain related to the unit stress, 1/Pa

l = kernel deformation, m

MC = moisture content, % dry basis

m = moisture content, decimal, dry basis

m_0 = thickness of monolayer, m

μ = chemical potential, J/mol

μ_0 = reference chemical potential, J/mol

n = number of moles

P = pressure, Pa

r = number of components

R = universal gas constant, 8.314 J/mol·K

RH = relative humidity, % or decimal

Q = heat of sorption, J/mol

S = entropy, J/K

σ_0 = contact load stress, Pa

t = time, s

T = temperature, K

T_r = retardation time (η/E_r), s

η = viscosity coefficient corresponding to a retarded elastic deformation in Burgers model, Pa·s

η_v = viscosity coefficient of a second viscous element in Burgers model corresponding to Newtonian flow, Pa·s

V = volume, m^3

1. INTRODUCTION

The world production of rapeseed has been rapidly increasing over the years. Estimated 4 Mt (million tonnes) of rapeseed was harvested worldwide in 1960. The number increased to 7 Mt in 1970 and reached 22 Mt in 1989 (FATUS, 1990). The 1989 annual production of rapeseed in Canada was 3 Mt. Canada was the world's largest exporter and third largest producer (after India and China) of rapeseed in 1989 (FAO, 1990). Together with wheat and barley, rapeseed constitutes three most important crops and the base for agricultural development and diversification in Canada.

New varieties of rapeseed are produced by altering its genetic structure. Canola is a generic term referring to varieties of rapeseed with low concentration of erucic acid and glucosinolates. Canola is a source of vegetable oil for human consumption.

The risk of quality deterioration during storage and transportation of canola has to be minimized for economic reasons. The knowledge of physical properties of canola, such as equilibrium moisture content and rheological properties, is of primary importance to maintain its high quality over a long time and in crushing industry during oil extraction process. At high moisture levels there may be a loss of functional properties such as colour, aroma, texture, appearance or nutrients due to microorganism growth (Labuza, 1974). The mechanical properties of agricultural material are also influenced by its moisture content (Multon et al., 1981).

These properties play a major role in evaluating the fitness of agricultural product for post-harvest processing (Szelef and Mohsenin, 1969).

Canola, as a biological material, changes its properties depending on age, growing history and pre-treatment. There is a lack of data on the influence of pre-treatments, such as rewetting/drying cycles, irradiation, growing history or long-term mechanical stress on equilibrium moisture content and mechanical properties of canola.

2. LITERATURE REVIEW

2.1 Equilibrium moisture content - relative humidity relationship

Equilibrium moisture content is the moisture content at thermodynamic equilibrium, when the rate of water adsorption by the material equals to the rate of water desorption from the material. Therefore, in the equilibrium state moisture content of the material does not change (Labuza, 1984). The theoretical aspects and a practical approach to the subject of the equilibrium moisture content in agricultural materials are given in this section.

2.1.1 Equilibrium in heterogeneous system

The theory of equilibrium, it is a basic concept in thermodynamics and one cannot underestimate its importance in the various processes and phenomena analysis. In food

processing and grain storage, the theory of equilibrium applies to all problems related to long-term exposure of foods and grains to air with a constant relative humidity.

Gibbs' equation for heterogeneous system consisting of two or more phases (Hatsopoulos and Keenan, 1965; Hsieh, 1975) is given by the following formula:

$$dG=-SdT+VdP+\sum_{\alpha=1}^{\varphi}\left(\sum_{i=1}^{r}\mu_{i,\alpha}\ dn_{i,\alpha}\right) \qquad (1)$$

where:

G = Gibbs' function, J

S = entropy, J/K

T = temperature, K

V = volume of a whole system consisting of φ phases, m^3

P = pressure, Pa

φ = number of homogeneous phases

r = number of components

μ = chemical potential of component i in phase α J/mol

n = number of moles of component i.

At constant temperature and pressure (thermal and mechanical equilibrium) the Gibbs' function becomes:

$$(dG)_{T,P}=0 \qquad (2)$$

Under the assumption that no chemical reaction occurs (chemical equilibrium) a following constraint equation may be

written:

$$\sum_{\alpha=1}^{\varphi} n_{i,\alpha} = constant \qquad (3)$$

Finally, the solution of Eq.1, under the constraints expressed in Eq. 2 and Eq. 3, is (detailed mathematical analysis given by Hsieh, 1975):

$$\mu_{i,1} = \mu_{i,2} = \ldots = \mu_{i,\varphi} \qquad (4)$$

In the equilibrium state, the chemical potentials of a particular component have to be equal in all the phases, because:

$$\mu = \mu_0 + RT\ln(a) \qquad (5)$$

where:

a = water activity or equilibrium relative humidity, decimal

μ_0 = reference chemical potential, J/mol

R = universal gas constant, 8.314 J/molxK

At normal conditions, the activity of the water in air equals to the relative humidity of the air (Labuza, 1984). Therefore, with respect to activities the following expression may be written:

$$a_{i,1} = a_{i,2} = \ldots = a_{i,\varphi} = RH \qquad (6)$$

where:

RH = relative humidity (decimal)

In the equilibrium state, the activity of water has to be the same in all the phases.

2.1.2 Temperature and pressure effect

In the above section, three assumptions were made. The assumption of constant temperature resulted in thermal equilibrium. The assumption of constant pressure assured mechanical equilibrium. The assumption of no chemical reaction occurring brought in chemical equilibrium.

The effect of temperature on water activity may be described with the Clausius Clapeyron, empirical equation (Labuza, 1984):

$$\ln\frac{a_2}{a_1} = \frac{Q}{R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right) \qquad (7)$$

where:

a_1 = water activity (relative humidity) at temperature T_1

a_2 = water activity (relative humidity) at temperature T_2

Q = heat of sorption, J/mol

The heat of sorption, Q, is a function of moisture content and for high moisture contents is equal to zero. For example, for Sinton wheat, at 10% db moisture content, the

heat of sorption is equal to 7690 J/mole (Labuza, 1984). Based on Eq. 7 the relative change in water activity, for wheat at 10% db moisture content due to its temperature change from 20 to 30^{0}C, would be approximately 11%. Therefore, in experiments concerning the equilibrium moisture content of agricultural materials, the temperature fluctuation should be kept within the smallest possible limits. Otherwise significant error may result. The influence of pressure on the water activity may be analyzed with the following equation (Labuza, 1984):

$$\ln\frac{a_2}{a_1} = \frac{V}{R\,T}(P_2-P_1) \qquad (8)$$

where:

V = molar volume of liquid, m^3/mol,

P_1 = initial pressure, Pa,

P_2 = final pressure, Pa.

a_1 = water activity at pressure P_1

a_2 = water activity at pressure P_2

At 30^{0}C, the molar volume of water is 0.00058 m^3/mol (Labuza, 1984). Substituting numerical values in Eq. 8 results:

$$\ln\frac{a_2}{a_1} = 9\cdot10^{-13}(p_2-p_1) \qquad (9)$$

The small value of constant on the right side of Eq. 9 implies that pressure fluctuations throughout the experiment

would have negligible effect on the final water activity of agricultural materials.

2.1.3 Moisture sorption hysteresis

Water activity is a function of state. Therefore, according to thermodynamics there should not be two different states of water within a material at a given water activity (Labuza, 1984). Nevertheless almost all biological materials exhibit a significant hysteresis loop. Due to hysteresis phenomena, much lower water activity is required to obtain a given moisture content by desorption than by adsorption. In nature, the hysteresis loop has a practical application of preventing the rapid changes, such as a sudden loss of water (Kapsalis, 1981).

Labuza (1984) gave three reasons for the hysteresis loop: (1) supersaturation of some solutes during drying process, followed by rapid crystallization. As a result, some water is trapped in crystalline structures and is not released during drying process, (2) The diameter of capillaries decreases during the drying process and water may be physically trapped and as a consequence not released during desorption. (3) The surface tension of water and wetting angle in capillaries differ for adsorption and desorption processes.

Four different types of hysteresis were classified by Everett (1967) and are shown in Fig. 2.1.

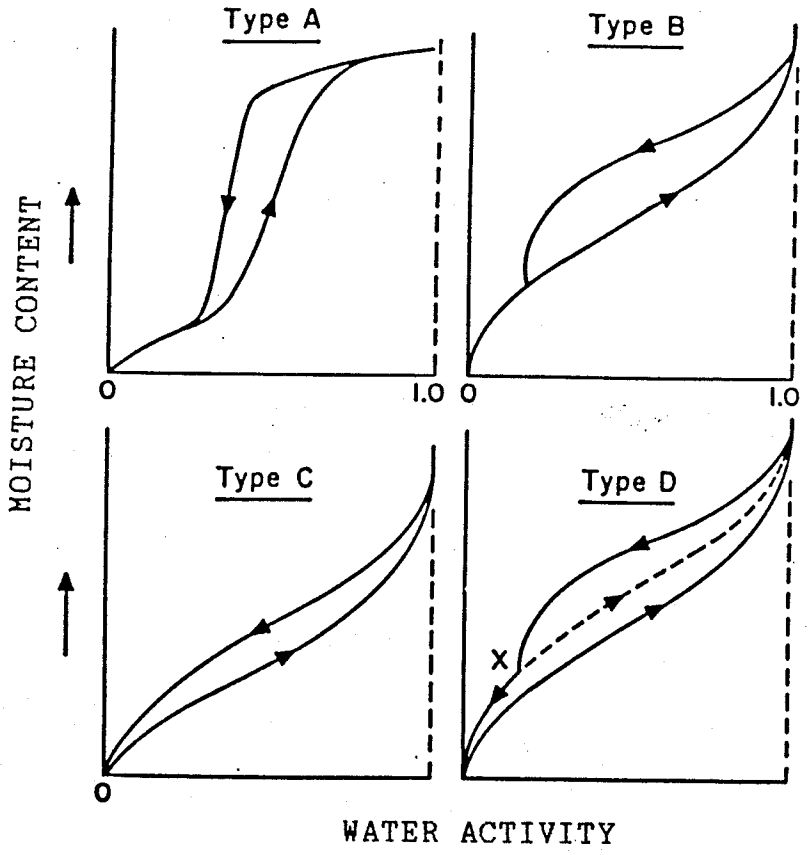


Fig. 2.1 Classification of main types of sorption hysteresis (Everett, 1967).

Type A occurs when the loop extends over a limited range of water activities. In type B, the loop extends from water activity equal to unity to a given closure point. In type C, hysteresis extends over the entire range of water activities. Type D is a combination of type B and C.

The size of hysteresis loop depends on temperature. It may decrease with decreasing temperature reach a minimum and then increase again (Amberg et al., 1957). Also, the rates of

adsorption and desorption process influence the size of a hysteresis loop. In the case when drying is carried out very slowly, the supersaturation of the solutes may be avoided and hysteresis loop narrows (Rao, 1939).

2.1.4 Methods for obtaining sorption isotherms

Methodology for obtaining sorption isotherms was characterized by exceptional diversity of apparatuses and methods (Gal, 1981). As it has been indicated by Gal (1981) the experimental data on equilibrium moisture content (EMC) should be accompanied by detailed description of the material, experimental procedures and apparatus. He stressed that the two basic parameters which should be precisely maintained at constant values, while taking EMC data, are temperature and water vapour pressure (relative humidity) in the space around the sample. He proposed that the temperature should be kept constant within $\pm$ 0.2^{0}C for routine work and $\pm$ 0.02^{0}C for reference purposes. He also indicated that the methods to maintain constant water vapour pressure by generating and controlling the vapour content within the space around the sample had only moderate accuracy. On the other hand the use of saturated and unsaturated salts solutions to maintain a constant vapour pressure produces the accuracy of approximately $\pm$ 0.5% in relative humidity.

Cenkowski et al. (1989) used ERH and EMC methods to obtain moisture content data for lentils. The equilibrium

relative humidity (ERH) method was based on equilibrating a small mass of air with a relatively large mass of grain in an air-tight system. The EMC method was based on equilibrating a thin layer of grain with the air of constant relative humidity. The result obtained with both methods were combined and used to estimate the constants of the modified Henderson and the Chung-Pfost equations.

Osborn et al. (1989) used the ERH method (described in their study as a closed-loop dew point method), to obtain moisture content data for soybeans. They indicated that the ERH method required less time for the small mass of air to reach equilibrium with the grain than for the grain to reach equilibrium with the air when using the saturated salt method.

Mazza and Jayas (1990) used a static method (no air movement, only diffusion), with standard solutions (saturated salt method), to obtain the equilibrium moisture content data for sunflower seeds, hulls and kernels. The sample under investigation was placed into a glass desiccator containing the salt solutions in order to maintain a constant vapour pressure.

Gal (1981) described the method of mixing various quantities of dry and moist lots of the same substance in order to obtain different final water activities (air relative humidity). He indicated that the obtained isotherm points lay inside the hysteresis loop in this case.

Multon et al. (1981) investigated the effect of moisture

content hysteresis on the mechanical properties of wheat kernels. One half of the initial lot of grain was dried, under vacuum without heating, to 5% db moisture content while the other half was wetted, by adding predetermined quantities of water, to 33% db moisture content. Gentian Violet was added to the water as a grain colorant. In order to obtain a hysteresis loop for different relative humidities predetermined quantities of dry and wet grain were mixed and placed into the sealed containers. After the equilibrium was reached, moist and dry samples were hand-separated and the moisture contents were measured.

In the EMC and ERH methods either moisture content of the sample or relative humidity of the air are quasi-constant during each experiment. In the methods described by Gal (1981) and Multon et al. (1981) both moisture content of the sample and relative humidity of the air are changing during the experiment. The changes in relative humidity are due to different rates of adsorption and desorption processes.

2.1.5 Mathematical models

The data obtained in experiments on equilibrium moisture content are usually fitted to one of the available mathematical models for predicting the moisture contents at any given relative humidity.

Van den Berg (1985a) stated 5 requirements for the isotherm mathematical models: (1) the experimental data should

be described by a mathematical model for a specific application as drying or storing, (2) the mathematical model should have a simple form, with a limited number of parameters, (3) the parameters should have a thermodynamic basis, (4) the temperature dependence should be reflected in the model and (5) the hysteresis phenomena should be reflected in the model. It was concluded by Van den Berg that there is no single mathematical model satisfying all the above requirements for describing sorption phenomena in biological materials.

Chirife and Iglesias (1978) evaluated 23 different isotherm equations and concluded that each model was successful in predicting the moisture contents for a specific food or grain and for given ranges of relative humidity and temperature.

Boquet et al. (1978; 1979) evaluated 39 different experimental isotherms and concluded that no single model fitted satisfactorily the experimental data for all the materials under investigation.

The Modified - Henderson and Chung - Pfost equation (Appendix B, Eq. 7 and 8) and the parameters for various materials were adopted as ASAE Standard D254.4, Moisture Relationship of Grains (ASAE, 1987).

Caurie (1981) tested his equation (Appendix B, Eq. 9) using published sorption data for a number of selected protein materials and wheat. Caurie (1981) concluded, that values of

monolayer (the value of moisture content at which all the sorption sides are occupied) are higher when predicted with his equation compared to those predicted with the BET (derived by Brunauer, Elmett, Teller) equation. It was indicated by Caurie that various workers in the field considered the value of monolayer derived from BET equation as underestimated.

Flood and White (1984) applied several known equations to their experimental data for popcorn. Equations presented by Iglesias and Chirife (1976) (Appendix B, Eq. 10) provided the best fit to their experimental data.

Jayas at el., (1987) estimated constants of the Modified - Henderson and Chung - Pfost equations for canola meal. They concluded that the Modified - Henderson equation described the experimental data for canola meal best for humidity from 20 to 80%.

Cenkowski et al. (1989) estimated the constants of Modified - Henderson and Chung - Pfost equations for lentils. It was concluded that the Modified - Henderson equation gave the best fit to the experimental data. Neither of the equations accurately estimated moisture contents for relative humidity from 80 to 90%.

In order to get the best mathematical description of the equilibrium moisture content - relative humidity relation, the experimental data should be analyzed, using several available equations and the constants in the equations should be determined statistically. The residual plots and sum of

squared residuals should be used to choose the mathematical model which gives the best fit to the experimental data.

2.3 Effects of the pre-treatments on physical properties of agricultural materials

Mohsenin (1986) indicated that the most important factors influencing the mechanical properties of biological materials are: moisture content, age, stage of ripening and temperature.

Hubbard et al. (1957) proved that hysteresis effects in wheat decrease as a result of repeated rewetting/drying cycles. Chung and Pfost (1967) explained that rewetting/drying cycles caused cracks which decrease the availability of sorptive sites inside an absorbent. After 3 consecutive rewetting/drying cycles the chemical and physical structure of wheat becomes stable and the hysteresis loop does not decrease further (Chung and Pfost, 1967). Multon et al. (1981) linked the moisture sorption hysteresis to different mechanical behaviour of wheat depending whether the wheat sample was equilibrated with the air through adsorption or desorption process.

In such countries as Japan and USSR grain and other agricultural materials are irradiated prior to the storage (Wilson, 1985). Exposure of agricultural materials to ionizing radiation kills microorganism while causing minimal chemical changes. A dose of 25 kilogray (kGy) (1 kGy - unit of energy absorbed from radiation by the material through

which the radiation passes, equivalent to 1 kJ) kills all the microorganisms within the irradiated grain sample (IAEA, 1985). At low irradiation doses (up to 10 kGy), the irradiation does not cause significant loss in nutritional quality of the grains (Graham, 1980; Murray, 1983). Futhermore, the losses in vitamin content are similar to those caused by heat processing (Graham, 1980).

2.4 Mechanical properties of biological materials

2.4.1 Viscoelasticity

According to Morrow and Mohsenin (1966) the majority of biological materials exhibit viscoelastic behaviour. The term viscoelastic refers to a whole spectrum of mechanical behaviour on the part of the material. According to Lockett (1972) on one side there are classical viscous fluids, e.g.: air or water, while on the other side are elastic solids as rubber under moderate loading. Viscoelastic solids exhibit flow in addition to their elastic properties. The flow behaviour of the viscoelastic solids, referred to the gradual elongation of the sample under the constant load, is called creep.

One of the methods used for describing the behavior of the viscoelastic biological materials is based on the rheological models consisting of a combination of springs and dashpots.

2.4.2 Rheological models for biological materials

Although the assumption of homogeneity and isotropic behaviour is violated with agricultural materials, their mechanical behaviour can be approximated using the rheological models. Zoerb and Hall (1960) used a 2-element Maxwell model to describe the behaviour of pea beans during stress relaxation. Morrow and Mohsenin (1966) indicated that rheological behaviour of McIntosh apples may be represented by 3-element Kelvin and Maxwell models.

4-element Burgers model

The rheological model to represent instantaneous compression, creep, elastic rebound and recovery of the viscoelastic, biological material is a 4-element Burgers model, shown in Fig. 2.2.

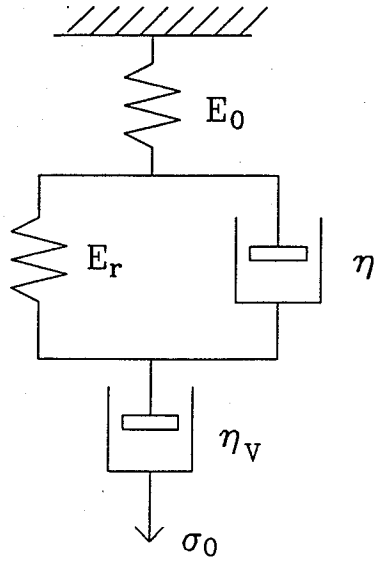


Fig. 2.2 4-element Burgers model (Mohsenin, 1986).

Burgers model consists of a Kelvin model connected in

series to a spring and a dashpot (Mohsenin, 1986). In the creep and recovery tests the load is suddenly applied, held constant for a given time, t_1, and then suddenly removed. A typical creep and recovery curve is shown in Fig. 2.3.

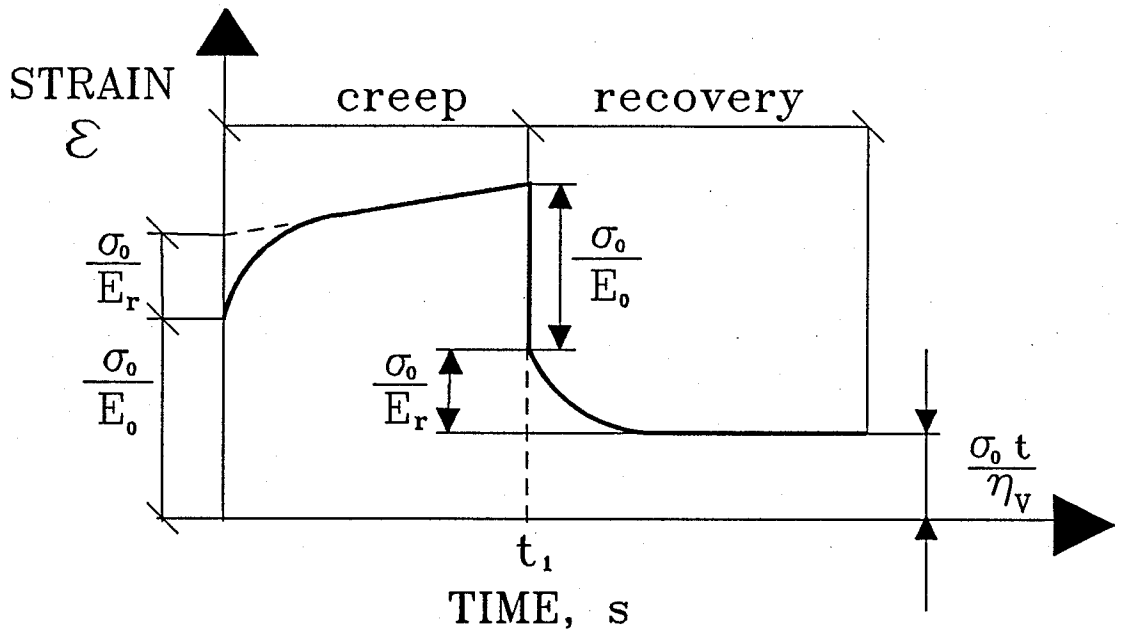


Fig. 2.3 Typical creep and recovery curve in a viscoelastic material (Cenkowski et al., 1990).

The mathematical description of the Burgers model was given by Morrow (1965) in the following form for the creep part:

$$\varepsilon(t) = \frac{\sigma_0}{E_o} + \frac{\sigma_0}{E_r}\left(1 - e^{-\frac{t}{T_r}}\right) + \frac{\sigma_0 t}{\eta_V} \qquad (10)$$

and for the recovery part:

$$\varepsilon(t) = \frac{\sigma_0}{E_r}\left(e^{\frac{t_1}{T_r}} - 1\right) e^{-\frac{t}{T_r}} + \frac{\sigma_0 t_1}{\eta_V} \qquad (11)$$

where:

$\varepsilon(t)$ = strain mm/mm

η = viscosity coefficient corresponding to retarded elastic deformation, Pa·s

η_V = viscosity coefficient of second viscous element corresponding to Newtonian flow, Pa·s

σ_o = contact load stress, Pa

E_o = instantaneous elastic modulus, Pa

E_r = modulus of elasticity of a second elastic element, Pa

T_r = retardation time (η/E_r), s

t = time, s

t_1 = creep time, s

The moduli of elasticity and viscosity coefficients corresponding to the elements of the Burgers model may be derived, based on the graphical description in Fig. 2.3, and then incorporated into the Eq. 10 and 11.

Using this approach Cenkowski et al. (1990) determined the apparent moduli of elasticity and viscosity coefficients representing the elements of the Burgers model for single canola kernels at approximately 8% wb moisture content.

5-element model

The biological yield point was defined as the point on the stress-deformation curve at which the stress decreases or remains constant with increasing deformation (Mohsenin, 1986).

At this point, according to Mohsenin, the initial cell rapture in a small volume of cellular structure occurs. In ASAE Standards (1987) bioyield point is defined, based on the force-deformation curve, as the point where the increase in deformation results in a decrease or no change in force.

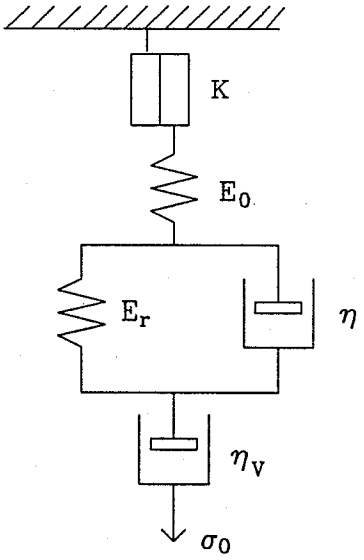


Fig. 2.4 5-element model (Bilanski and Graham, 1983).

Pitt (1982) defined tissue failure as a sequence of individual cell raptures, or a sequence of intercellular bond failures. In the first case, the expected cause of tissue failure is the normal stress while in the second case the shear stress will typically cause the .

Tissue failure results in irreversible deformation of the sample subjected to the stress greater than its critical value of yielding stress. Drake (1971) and Peleg (1983) defined the so called fracture elements to introduce such irreversibility and discontinuity phenomena to rheological models representing biological materials. Peleg (1983) indicated that the

dissipation element may be activated by a critical strain as well as critical stress or either of them depending which critical value was reached first.

Bilanski and Graham (1983) indicated that the rheological behaviour of forage wafers with its yielding characteristic may be represented with the five element model, shown in Fig. 2.4.

The dissipation element K of the five element model represents, according to Bilanski and Graham, instantaneous, irreversible deformation due to the yielding of the material. The instantaneous material response was expressed by:

$$\gamma(\sigma) = \gamma_0 + b(1 - e^{-\tau\sigma}) \qquad (12)$$

where:

γ = deformation, m

σ = stress, Pa

τ, γ_0, b = constants for a given material

The creep behaviour of the material was expressed in the following form:

$$J(\sigma, t) = \Phi(\sigma)(1 + at - e^{-kt}) \qquad (13)$$

where:

$J(\sigma,t)$ = creep compliance - strain related to the unit stress, 1/Pa

$\Phi(\sigma)$ = stress function

t = time, s

a, k = constants for a given material

Creep compliance depends on the magnitude of the applied stress, which indicates that the authors assumed nonlinear viscoelasticity of the investigated material.

3. OBJECTIVES

The specific objectives of the present studies were:

1. To determine the effect of age and growing history on: the equilibrium moisture content - equilibrium relative humidity (EMC - RH) relationships, hysteresis loop and viscoelasto-plastic properties of canola kernels.
2. To determine the effects of irradiation, long-term stress and rewetting/drying cycles on: the equilibrium moisture content - equilibrium relative humidity relationship and hysteresis loop.
3. To relate and quantify the effect of changes in equilibrium moisture content due to the pre-treatments on resulting changes in viscoelasto-plastic properties of single canola kernels.
4. To investigate the character and magnitude of hysteresis loop between adsorption and desorption isotherms for canola.
5. To compare the viscoelasto-plastic properties of the canola kernels equilibrated with the same relative humidity air through adsorption and desorption process (mechanical hysteresis).
6. To evaluate the Burgers model for the creep and recovery

tests performed on single canola kernels.

7. To develop a rheological model for explaining the mechanical behaviour of a single canola kernel during creep and recovery tests.

4. DESCRIPTION OF EQUIPMENT

4.1 Equilibrium moisture content apparatus

A schematic drawing of the experimental unit used in equilibrium moisture content experiments is shown in Fig. 4.1.

The unit consisted of an equilibrium moisture content (EMC) apparatus, heat exchanger and Maxima air pump (Hagen Inc., Montreal,PQ.). The heat exchanger comprised a copper tubing (400 mm - length, I.D. = 5 mm) and a water tank. The elements of the experimental unit were connected with flexible plastic tubing (I.D. = 5 mm).

Six independent experimental units were constructed. Each equilibrium moisture content apparatus consisted of: 10 plastic rings (2) (10 mm thick and I.D. = 95 mm), top (1) and bottom (4) pipe section. A metal mesh (8) was installed in each ring to support a sample (10). In the top section, a bulk polymer resistance humidity sensor RH-2 (9) (General Eastern, Inc., Watertown, MA) was installed.

The humidity sensors mounted in all six EMC experimental apparatuses were calibrated prior to the experiments and then calibration was checked again after a series of experiments. A Hygro-M1 dew point humidity sensor (General Eastern

Instruments Inc., Watertown, Ma.) was used as a standard to calibrate the RH-2 humidity sensors.

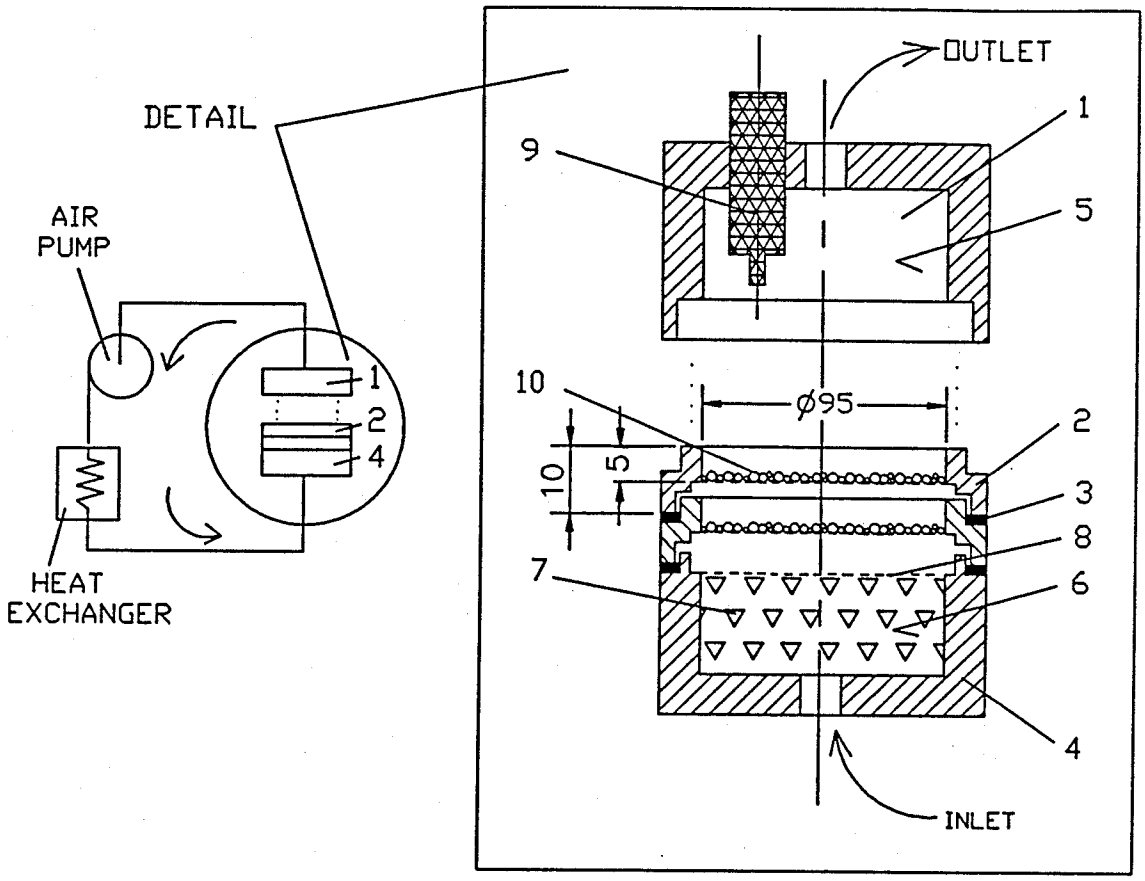


Fig. 4.1 Equilibrium moisture content apparatus. 1 - top pipe section, 2 - plastic ring, 3 - rubber gasket, 4 - bottom pipe section, 5,6 - thermocouples, 7 - glass beads, 8 - metal mesh, 9 - humidity sensor, 10 - sample. All dimensions are mm.

The bottom section of the EMC apparatus was filled with a bed of glass beads (7) (1 mm diameter) in order to temper the incoming air. The temperature of incoming and outgoing air was measured using T-type thermocouple (5,6) mounted in the top and bottom sections of the EMC apparatus. The EMC experimental units were placed in a walk-in temperature

controlled chamber.

4.2 Temperature controlled chamber

A temperature controlled chamber was designed and built to maintain constant temperature (± 0.1 ^{0}C) throughout the experiments. The chamber was constructed of wood and thermally insulated with two layers of styrofoam (each 50 mm thick).

Heat generated inside the chamber by the Maxima air pumps during the experiments was removed via a heat exchanger to a water/ethylene glycol solution and dissipated in the KR-30 compressor (Haake, Inc., Germany) outside of the chamber. The glycol solution was then circulated throughout a R-20 circulator (Haake, Inc., Germany) and back to the heat exchanger inside the chamber. The heating elements of the circulator were controlled electronically in an on-off mode. The temperature feedback was derived from the LM-335 precision temperature sensors (National Semiconductor Corp., Santa Clara, CA) mounted on the wall of the chamber and on the wall of the heat exchanger inside the chamber.

4.3 Spring apparatus

A schematic drawing of the experimental unit used in mechanical properties tests is shown in Fig. 4.2.

The unit consisted of a Chatillon Universal Testing Machine (John Chatillon & Sons Inc., NY) and a spring

apparatus (Bielewicz, 1990).

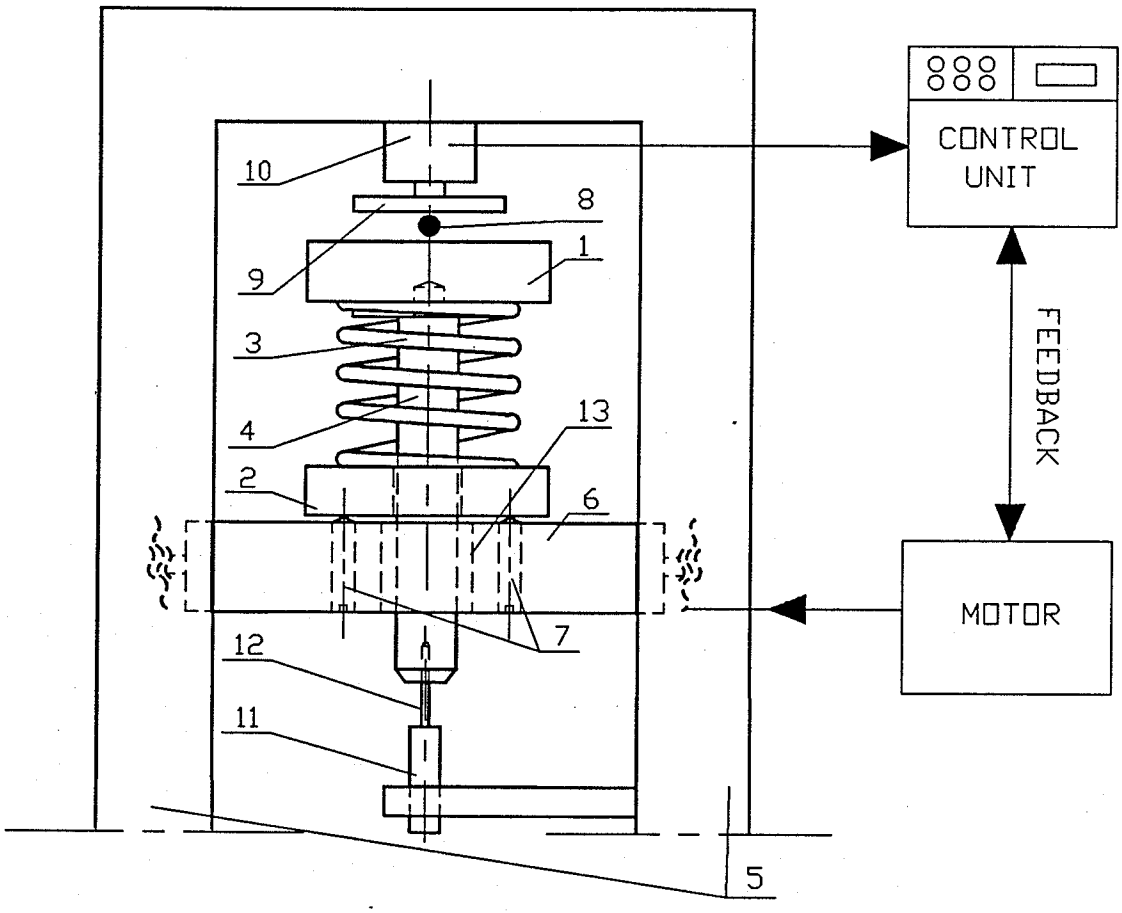


Fig. 4.2 Mechanical properties experimental unit. 1 - rigid metal plate, 2 - adjustable metal plate, 3 - spring, 4 - pilot rod, 5 - frame of the universal testing machine, 6 -movable bar of the universal testing machine, 7 - adjustment screws, 8 - kernel, 9 - metal plate of the universal testing machine, 10 - load cell, 11 - LVDT sensor, 12 - rod of LVDT sensor, 13 - opening in the movable bar of the universal testing machine.

The main components of the spring apparatus were: two round plates (1,2), a guide rod (4) and a spring (3). The movable top plate (1) was supported by the spring. The top plate was mounted upon a rod (4) with the rod extending in a vertical direction. The other end of the rod slid freely in

the guide hole of the bottom plate (2). The clearance between the rod and the opening in the bottom plate was less than 0.01 mm. The main purpose of the rod was to ensure that the top plate (1) maintained its horizontal position while moving in the vertical direction. A rod (12) of the linear voltage displacement transducer (LVDT) (11) was attached to the bottom end of the apparatus rod (4). The spring was mounted in series with a sample (8). The spring was carefully machined so that it rested directly against the surfaces of the plates while the axis of the spring remained directly vertical so that the spring did not provide any twisting action on the top plate (1) which could cause binding of the rod (4) in its guide hole.The whole apparatus, except the displacement transducer (11), was mounted on the movable bar (6) of the Chatillon Universal Testing Machine and adjusted with screws (7) until all three plates (1,2,9) were parallel. The displacement transducer (11) was attached to the main support frame (15) of the Chatillon Universal Testing Machine.

The deformation of the sample (8) was measured with an AC-AC 271-0000 and DC-DC 242-000 linear voltage displacement transducer (11) (Trans-Tek Inc., Elliggton, Con.), while the deformation of the spring (3) was measured with a DC-DC 242-000 LVDT (Trans-Tek Inc., Ellington, Con.). Also, the force acting on the load cell (10) and the displacement of the movable bar (6) were measured by the built-in Chatillon instrumentation. The displacement transducers were connected

to a Hewlett Packard HP 341A data acquisition system. An IBM personal computer was used for data logging.

The diameter of the sample (single kernel) was measured with a DC-DC 351-000 LVDT (Trans-Tek Inc., Ellington, Con.).

All the displacement transducers were calibrated with clearance gages prior to the experiments and the calibration was checked after the experiments.

5. MATERIALS

5.1 Samples of canola

Canola kernels Brassica napus L., cv. Westar were used in the experiments. Three different canola crops (grown at different locations and in different years) were used in the experiments.

The X-sample, 1989 crop, at about 7.1% moisture content, db, was purchased from a local supplier in August, 1990. The average diameter of the kernels was 1.76 mm with a standard deviation of 0.15 mm.

The Y-sample was harvested in 1989 at the Glenlea Experimental Station, Faculty of Agriculture (Glenlea, Manitoba). Plants were threshed with a Vogel thresher, cleaned using a blower and then further cleaned and separated, with a centrifuge spiral separator (Cleland Mfg. Cop., Minneapolis, MN), into two fractions. The Y-sample was then stored for nine months at room temperature at approximately 8% wb moisture content. Examination of the kernels, prior to the

experiments, under magnifying glass showed that some of the kernels were cracked. The outside surface of most of the kernels was wrinkled. The average diameter of the kernels was 1.95 mm with a standard deviation of 0.32 mm.

The Z-sample was also harvested at the Glenlea Farm in August, 1990. Plants were threshed with a Vogel thresher, cleaned using a blower and further cleaned with a specific gravity separator (Kipp Kelly Inc., Winnipeg, MB). The average size of the kernel was 1.70 mm with a standard deviation of 0.20 mm.

5.2 Pre-treatments

Three different X-samples of canola were subjected to three different pre-treatments: rewetting/drying cycles, irradiation and dead load application. For convenience, the sample subjected to long-term stress was named S-sample, the sample subjected to irradiation was named I-sample and the sample subjected to the rewetting/drying cycles was named C-sample.

5.2.1 Rewetting/drying cycles

A schematic diagram of the rewetting/drying procedure is shown in Fig. 5.1.A canola sample of approximately 4 kg was subjected to three consecutive rewetting/drying cycles. The sample of 7.1% db initial moisture content was first moistened to 20% db moisture content by sprinkling with a predetermined

quantity of distilled water.

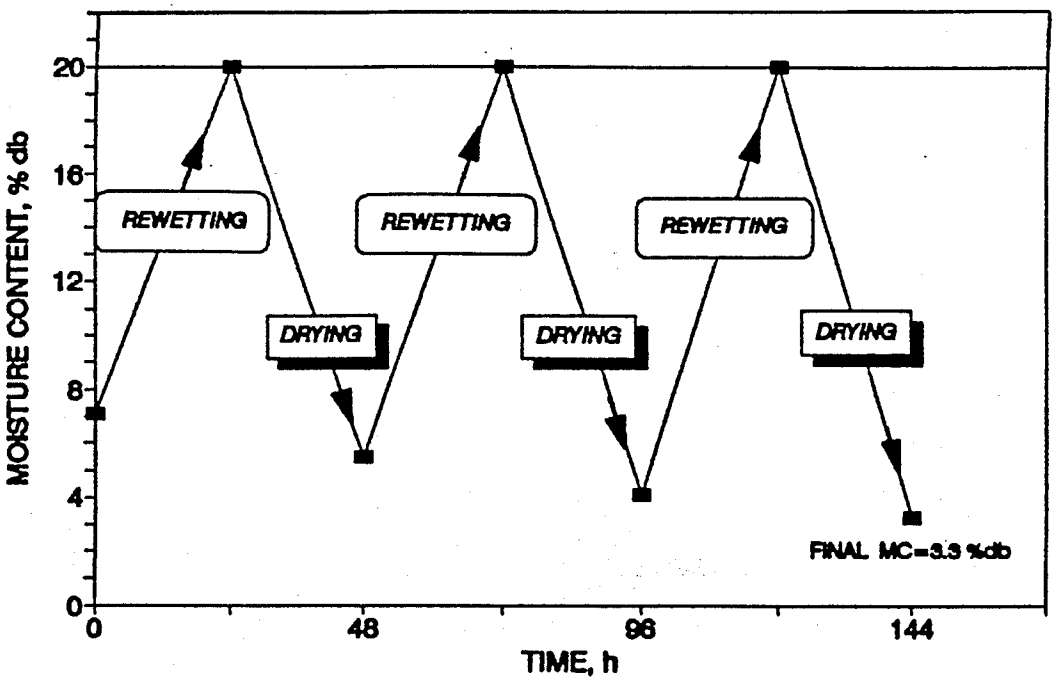


Fig. 5.1 Schematic diagram of the rewetting/drying procedure.

The rewetted sample was then kept in a sealed plastic bag. Throughout the first hour the rewetted sample was gently mixed at room temperature to ensure uniform moisture distribution. The rewetted sample was then stored at 10^0C for a further 23 h for equilibration. The canola sample was finally spread on two trays in a 40 mm thick bed and dried in an oven (Labline Inc., Chicago, Il) at 50^0C. The sample was mixed in the oven every hour for the initial 4 hours. After 24 h the sample was taken from the oven and examined. A

strong fermentation-like odour was noted. Then the moisture content of the sample was determined and the sample was remoistened to 20% moisture content, db. Two additional rewetting/drying cycles were performed.

A careful visual examination of the sample after a third drying cycle revealed substantial mold development. Prior to testing the core of mold (stuck together canola kernels) was removed from the sample.

5.2.2 Irradiation

A 5 kg sample of canola was transported to the Whiteshell Nuclear Research Establishment at Pinawa, Manitoba. An AECL I-10/1, 10 Mev - 1 kW prototype industrial linear accelerator was used to irradiate the canola sample. A roller conveyor delivered the sample to the target room. The sample moved through the accelerator where it was subjected to the electron beam penetrating the sample from the top . The sample was exposed to a dose of radiation equal to approximately 10 kGy.

5.2.3 Constant pressure application

A 5 kg sample of canola was subjected to long term-stress. A constant stress on the sample was obtained by applying a dead load to the canola bed.

A cylinder (I.D. = 95 mm) was filled to approximately 300 mm level with the canola kernels. A weight of 200 N was applied to a pressure plate. The pressure of approximately

30 kPa exerted on the canola sample was equivalent to the static pressure caused by the weight of 3 m high canola bed. The canola sample was left under the load at room temperature for a period of three weeks.

6. METHODS

The equilibrium moisture content tests and mechanical properties tests were performed simultaneously. The experimental material acquired from the EMC tests was subjected to the mechanical properties tests.

6.1 Equilibrium moisture content tests

All three different samples (X, Y, Z) of canola and three different subsamples of the X-sample subjected to different pre-treatments were used as the experimental material for the EMC tests.

Preliminary tests indicated that the heat generated by the Maxima air pump (Fig. 4.1) caused a temperature gradient across the layers of canola kernels inside the EMC apparatus of approximately 0.6^0C. Therefore, the temperature within a working area of the temperature control chamber was kept constant at 24.6^0C to maintained average temperature of the sample at approximately 25^0C.

6.1.1 Sample preparation

A 4 kg canola sample, at approximately 7.1% db moisture

content, was divided into two subsamples. The first subsample was rewetted to approximately 14% db moisture content, by adding predetermined quantities of distilled water. The rewetted subsample was kept in sealed plastic bag. Throughout the first hour the rewetted subsample was gently mixed at room temperature to ensure uniform moisture distribution. The sample was then stored, at 10^0C, for at least another 23 h.

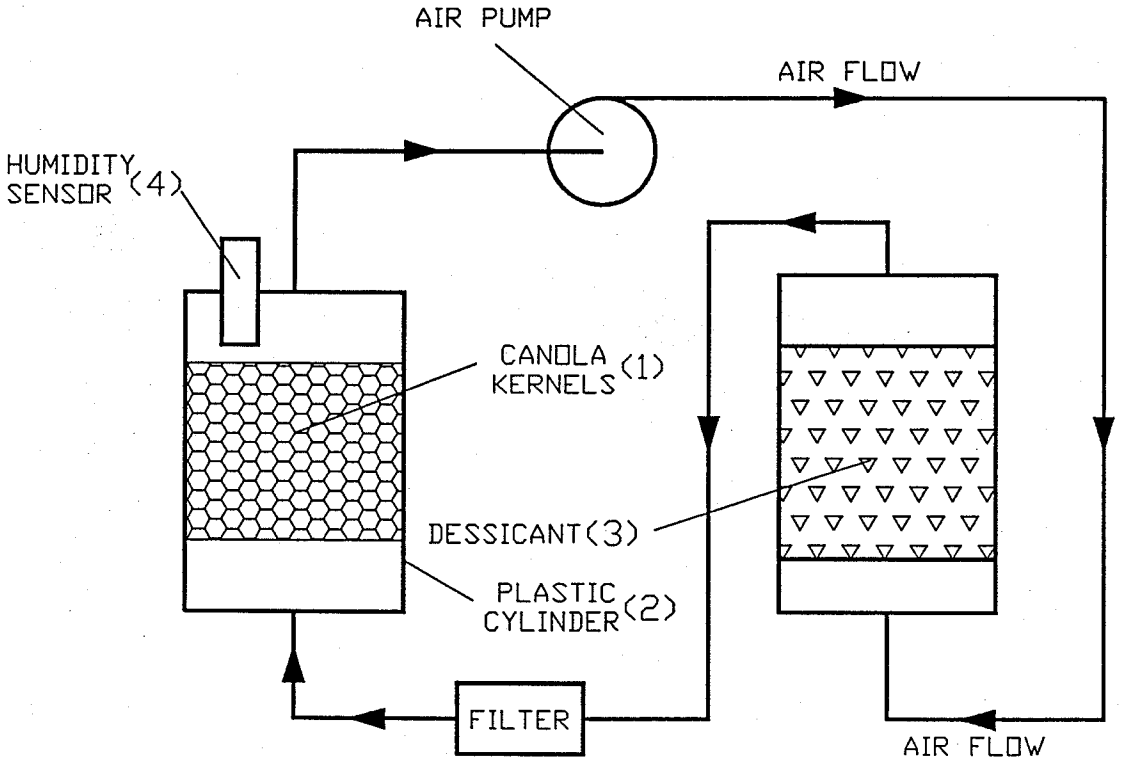


Fig. 6.1 Experimental unit used in the preparation of low moisture content sample.

The second subsample was dried in an experimental unit (Fig. 6.1) to approximately 3.3-4.5% db moisture content.

The sample (1) was placed into a plastic cylinder (2). Dry air was forced through the sample. Water absorbed by the air while it passed through the canola bed was transferred and desorbed from the air to a desiccant (3). Air was circulating in a closed loop. The whole unit was air-tight. The desiccant (3) had to be replaced once during the drying process in order to achieve the required level of canola moisture content. The final moisture content of the canola was predicted based on the reading of a humidity sensor (4) and the results of previous experiments. The drying process lasted approximately 24 h. The dried subsample was then placed in a plastic bag and stored at room temperature for several hours.

6.1.2 Test procedure

Prior to the experiments, both dry and moist subsamples were placed in the sealed plastic bags in the control chamber at 24.6 ^{0}C and left there for a period of 2 hours. Six EMC units were used in the EMC experiments. Each of 10 intermediate section (rings) of the EMC apparatus (Fig. 4.1) was loaded with approximately 15 g of canola, creating a single kernel thick layers.

Different levels of final relative humidity at each EMC test were achieved by loading the EMC apparatuses with different ratios of the moist subsample mass to the dry subsample mass. An experimental layout would typically

look as follow: section #1,3,5,7,9 were loaded with dry canola kernels (3.5-4.5% db) while section #2,4,6,8,10 were loaded with canola kernels of 14% db moisture content. A section containing a dry subsample was immediately followed with a section containing a moist subsample. Such an arrangement increased the section-to-section moisture gradient, therefore accelerated the moisture adsorption to the dry subsample and moisture desorption from the moist subsample.

The task of loading the EMC apparatus was performed with maximum possible efficiency and speed to minimize the moisture loss by the kernels at high moisture content and the moisture increase in the kernels at low moisture content. A predetermined quantity of dry and moist subsample, required to load a single EMC apparatus, were separated from the rest of the samples, kept in the sealed bags inside the chamber. The EMC apparatus was then loaded according to layout which was previously planned. The time required to load a single EMC apparatus with 10 intermediate sections was no longer than 5 min. The average room temperature, where the samples were loaded into the EMC apparatus, was approximately 24^0C with maximum deviation of $\pm 2^0$C.

A series of preliminary experiments were conducted in order to estimate the time required for the dry and moist subsample to reach an equilibrium. It was determined that

after 17 to 20 h (depending on the dry to moist mass ratio), the relative humidity sensed by the R-2 humidity sensor (Fig. 4.1) did not change. It was further noticed that an increase in the experimental time from 24 h to 48 h did not change the final moisture content of initially dry and moist subsamples, nor changed the hysteresis loop between the subsample equilibrated through adsorption process and the subsample equilibrated through desorption process. Had the equilibrium been not reached, the hysteresis loop would have narrowed with the time. In the case of no hysteresis, both points on the adsorption and desorption curve would have finally overlapped each other in the equilibrium state.

If relative humidity sensed by the R-2 humidity sensor (Fig. 4.1) did not change through 4 consecutive hours the intermediate sections of the EMC apparatus containing canola samples were removed from the EMC apparatus and unloaded. All the canola originally taken from the moist subsample and coming from different sections of the EMC apparatus was mixed together and placed into a plastic bag. The same procedure was followed with the canola originated from the dry subsample. The time required to unload a single apparatus was no longer than 2 min.

Moisture content determinations were done with the convection oven method, by drying 15 g samples for 4 h at 130^0C, as outlined in ASAE Standard S352.1 (ASAE, 1987).

In the experimental design, the EMC tests were to

provide the canola samples for mechanical properties evaluation. This required the inclusion of the EMC apparatus section loaded with X-nontreated sample as a reference. Based on comparison between pretreated and reference samples the impact of different pre-treatments on the mechanical properties was determined.

6.2 Mechanical properties test

The principal goal of the mechanical properties experiments was to compare viscoelastic properties of single canola kernels previously subjected to different pre-treatments with the properties of reference kernels after both samples were equilibrated with air in exactly the same conditions. The kernels previously subjected to the EMC tests were used as an experimental material in the mechanical properties experiments.

As a result of the investigation a rheological model to represent viscoelasto-plastic behaviour of a single canola kernel subjected to the creep and recovery test was proposed.

6.2.1 Selection of working settings

Cenkowski et al. (1990) investigated the mechanical properties of the canola kernels by compressing a single kernel in series with a spring at constant rate of 1.7 mm/s. In these studies the same upward velocity of the movable bar

(6) (Fig. 4.3) was chosen. The kernel was compressed with a constant rate of loading of approximately 2 N/s. The load was released with the same rate in the unloading process for all the mechanical experiments.

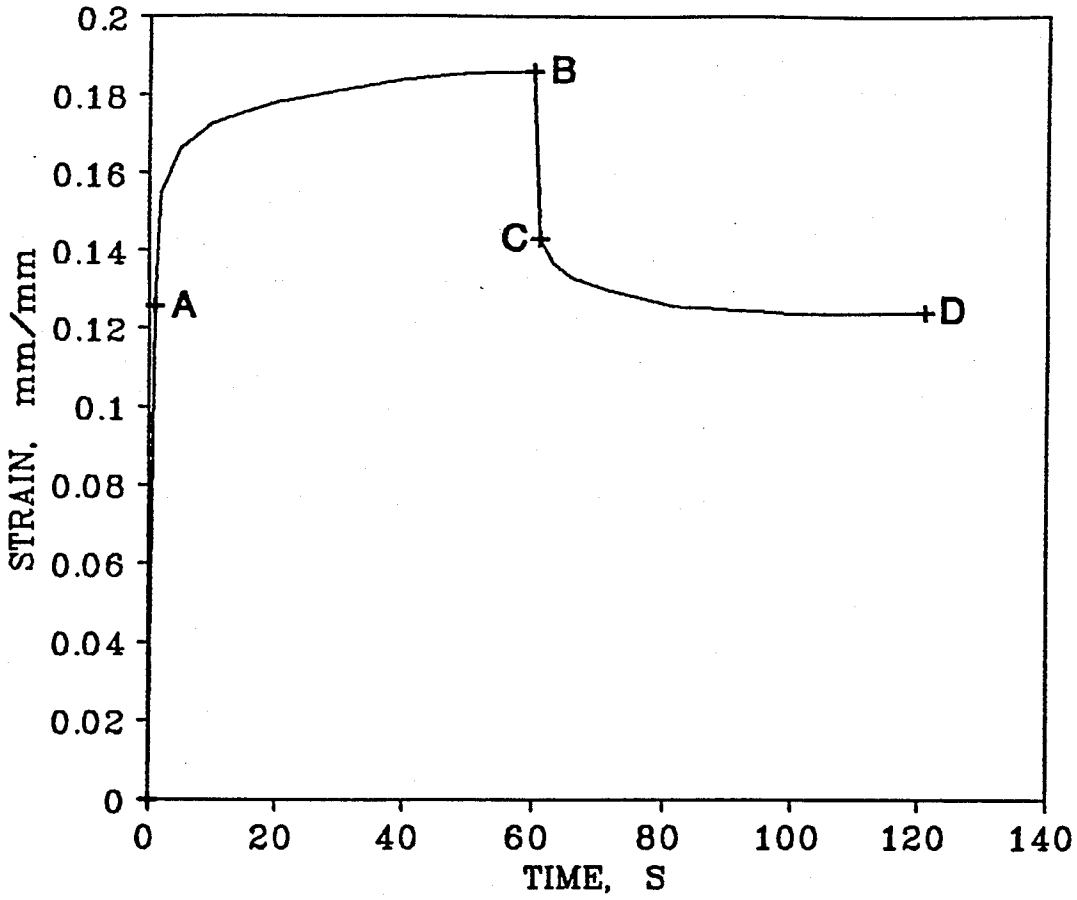


Fig. 6.2 Typical creep and recovery test for the kernel at 20.5% db moisture content.
Origins to A - loading, AB - creeping period, BC - unloading, CD - recovery period.

Preliminary creep and recovery tests on the single canola kernels at high moisture content (above 8% db moisture content) showed that strain caused by loading the

kernel was much greater than strain corresponding to unloading process. The same tests also revealed that creep strain was much greater than recovery strain for the kernels at high moisture content. A typical creep and recovery test for the kernel at 20.5% moisture content is shown in Fig. 6.2. In this test loading strain was approximately 2.9 times greater than the strain corresponding to the unloading process. The creep strain was approximately 2.9 times greater than recovery strain. Creep and recovery, loading and unloading strains were approximately of the same magnitude in the second creep and recovery test performed on the same kernel. The creep and recovery characteristics obtained in the first creep and recovery test could not be explained with a simple Burgers model, discussed in Section 2.4.2. In order to eliminate plastic, irreversible deformation contributing to instantaneous loading and creep deformations each creep and recovery test was preceded by 30 loading/unloading cycles. This method of eliminating plasticity of the biological material was described by several authors (Shpolyanskaya, 1952, Davison et al., 1975, Cenkowski et al., 1990). A typical loading/unloading cycles test performed using the spring apparatus, is shown in Fig. 6.3. As result of the initial loading/unloading cycles elastic rebounce (A, Fig. 6.3) of the kernel was equal to the deformation caused by loading during further loading/unloading cycles. Preliminary tests with canola

kernels at 13 and 20% moisture content were performed to select upper force limit.

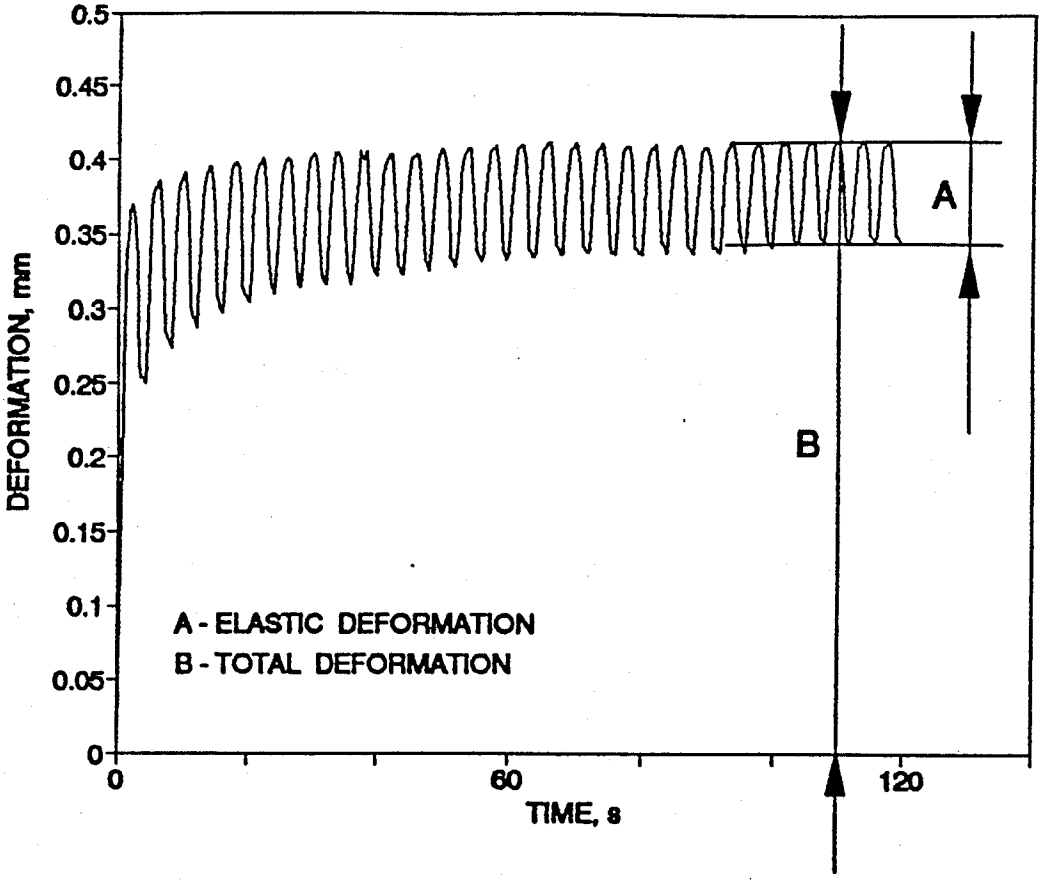


Fig. 6.3 Typical loading/unloading cycles for canola kernel at 20.5% db moisture content (Appendix A, Table A8).

The results of these tests revealed that plastic deformation (B-A, Fig. 6.3) in each consecutive cycle in cycle test increased when the maximum force acting on the kernel at the end of each cycle, was greater than 2.2 N for the kernels at high moisture content. Further testing

showed that the plastic deformation, for the kernels at high moisture content, was eliminated after approximately 25 cycles so that the loading/unloading characteristic levelled-off with the maximum force acting on the kernel of approximately 2 N. Therefore, all the creep and recovery tests were preceded with 30 loading/unloading cycles.

Further preliminary tests showed that, with the upper force limit set at 1.85 N and the lower at 0.16 N, maximum average force at the end of loading process was 2 N with maximum deviation of $\pm$ 0.01 N and minimum average force at the end of unloading cycle was 0.01 N with maximum deviation of $\pm$ 0.01 N.

In the creep and recovery tests a 60 s creep time was sufficient for the slope of a tangent to the creep curve not to change during at least the last 10 s of the creep part of the experiment. A 60 s recovery time was sufficient for the kernel to recover.

6.2.2 Sample preparation

In order to avoid a change in canola moisture content, due to the exposure of kernels to ambient air during mechanical testing, the canola sample, stored in the plastic bag immediately after EMC tests, was divided into several small subsamples. Up to four kernels from each subsample were used for mechanical tests. Each kernel was chosen from a small portion of canola kernels separated from the rest of

the subsample. The subsamples were stored in the plastic bags inside a temperature control chamber at 24.6^0C.

The positioning of a single canola kernel during a loading period is shown in Fig. 6.4.

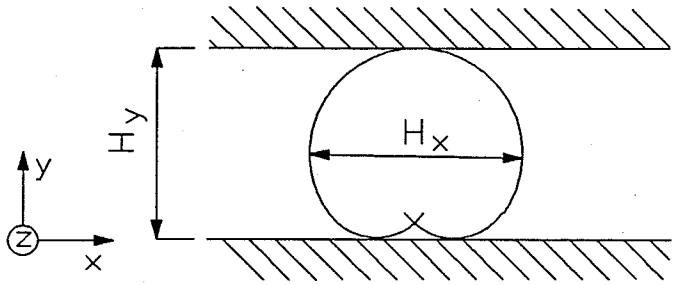


Fig. 6.4 Loading position of a single canola kernel.

Preliminary tests demonstrated that kernels placed in the crease side down position were most stable and therefore most likely not to exhibit a side movement during the rheological tests. Only kernels with a dark outside surface were selected for mechanical properties tests. Kernels of a spherical shape and approximately of the same size were chosen for mechanical properties experiments.
Preliminary tests showed that kernel diameter in Y direction (H_Y) (Fig. 6.4) could be well expressed as arithmetic average of kernel diameters in X (H_X) and Z directions (H_Z). The diameter in Y direction (H_Y) of each kernel was measured.

A single kernel (Fig. 6.5), chosen for the experiments,

was positioned in the centre of the top plate of the spring apparatus (Fig.6.6).

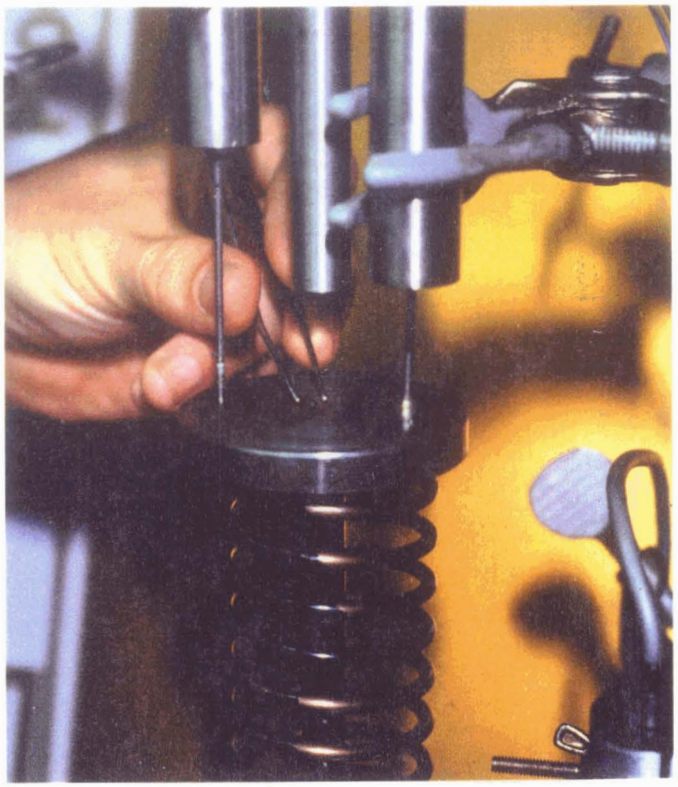


Fig. 6.5 **"A single kernel, chosen for the experiments, was positioned in the centre of the top plate of spring apparatus."**

The upper force limit was set through the keyboard of the control unit at 0.04 N. The movable bar of 'Chatillon' universal testing machine (6) was moved upward with a velocity of 5 mm/min (minimum velocity of the bar) until the kernel made contact with the load cell plate (9). Force displayed on the screen of the control unit, at the moment of the initial contact when the movable bar of testing machine halted, was not greater than 0.04 N.

6.2.3 Test procedure

Creep and recovery tests

Each creep and recovery test was preceded with the loading/unloading cycles. Prior to the loading/unloading cycles, the upper and lower force limits were set through the keyboard of the control unit (Fig. 4.2) at 0.16 and 1.85 N. The upward and downward velocity of movable bar (6) of testing machine was set through the keyboard of the control unit at 100 mm/min (1.7 mm/s). The Chatillon Testing Machine was set to "Automatic mode". The data acquisition system was then initiated and the kernel was subjected to 30 loading/unloading cycles. After 30 cycles, the movable bar (6) of the testing machine was automatically halted. The working mode of testing machine was changed to "Manual". The set force limits as well as an upward and downward velocity of the movable bar were left unchanged. The kernel recovered for a period of approximately 60 s and then the movable bar was moved upward. The bar halted when the force acting on the kernel reached the set maximum value (2 N). The bar stayed in this position for 60 s and then was moved downward and halted when the force acting on the kernel reached the set minimum value (0.01 N). The data was collected for another 60 s.

Contact area tests

In order to calculate moduli of elasticity and

viscosity coefficients defined in Section 2.4.3 in the Burgers model the contact area between the kernel and compression plates had to be evaluated. In the contact area experiments, the canola kernels were subjected to 30 consecutive loading/unloading cycles.

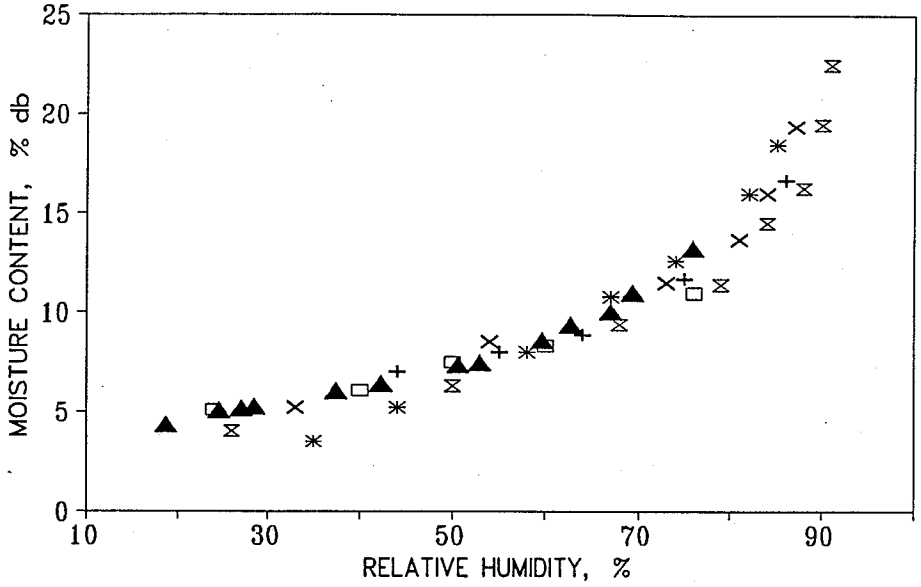
A glass slide, with a thin layer of carbon, was introduced between the load cell plate (9) (Fig. 4.2) and the kernel. The layer of carbon was placed on the glass surface by means of a candle. The area from which the carbon was removed by the kernel during loading/unloading cycles represented the contact area. Diameter of the contact area was measured by first enlarging the image of the contact area by projecting it onto the screen from the glass slide and tracing it with the pen. Calliper was then used to measured diameter of the contact area by projecting its image onto the screen and then by adjusting calliper to the traced image of the contact area. Four replicates were performed at each of the six moisture content levels.

7. RESULTS AND DISCUSSION

7.1 Evaluation of the method used in equilibrium moisture content tests

The equilibrium moisture content data for the canola Brassica napus L., cv. Westar harvested from different fields, in different years and subjected to different pre-treatments, for adsorption and desorption isotherm at 25^0C,

in terms of moisture content versus relative humidity, are given in Appendix A (Tables A1 to A6).



⧖ - Pixton and Henderson (1981) + - Pichler (1957)
✳ - Rao and Pfost (1980) □ - Timber and Hocking (1974)
× - Sokhansajn et al. (1986) ▲ - present work

Fig. 7.1 Comparison of the present experimental data on equilibrium moisture content with the published data.

The experimental data for the X sample are given in Appendix A, Table A1. The averages of the adsorption and desorption moisture contents were calculated for each relative humidity level.

In order to verify the equipment used in EMC test the calculated EMC data obtained in these studies are presented along with the data published by Pixton and Henderson (1981), Pichler (1957), Rao and Pfost (1980), Timbers and Hocking (1974), Sokhansanj et al. (1986) in Fig. 7.1. The

published data were obtained in temperature range of 20^{0}C to 25^{0}C. The experimental data obtained in the present work were found to be in a good agreement with the published data.

7.2 Pre-treatment effects on isotherm shape and hysteresis loop

The comparison of the adsorption and desorption experimental data for X, Y and X, Z samples is shown in Fig. 7.2 and 7.3.

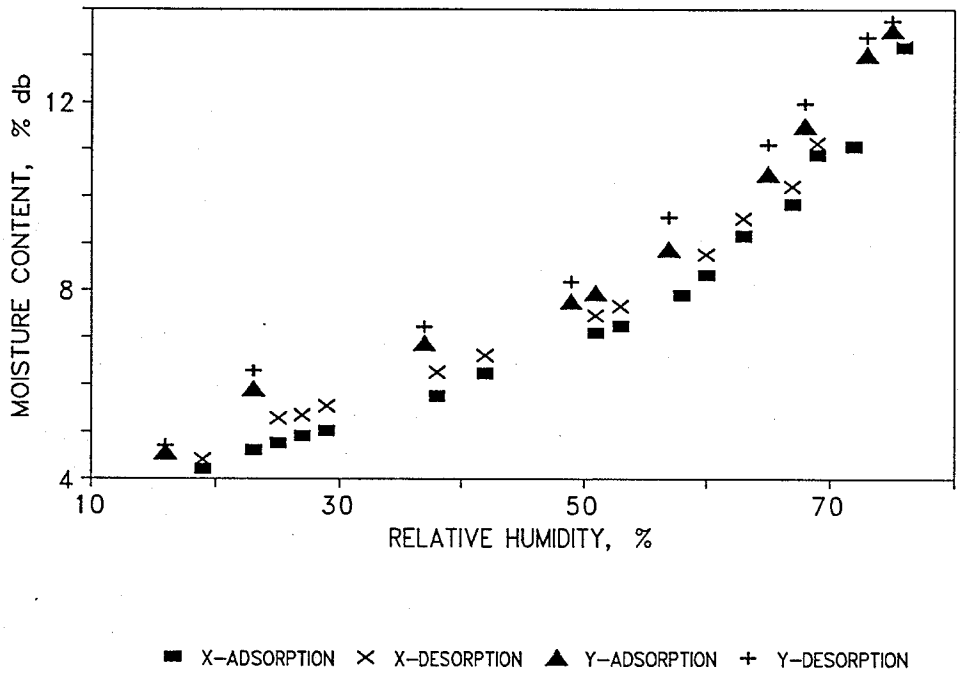


Fig. 7.2 Desorption and adsorption isotherms at 25^{0}C for two different samples of Westar canola.
X - 1989 crop, purchased from the local supplier.
Y - 1989 crop, harvested at Glenlea Farm.

The data points representing the adsorption and desorption processes of the Y-sample lie above the data points representing adsorption and desorption process of the X-sample.

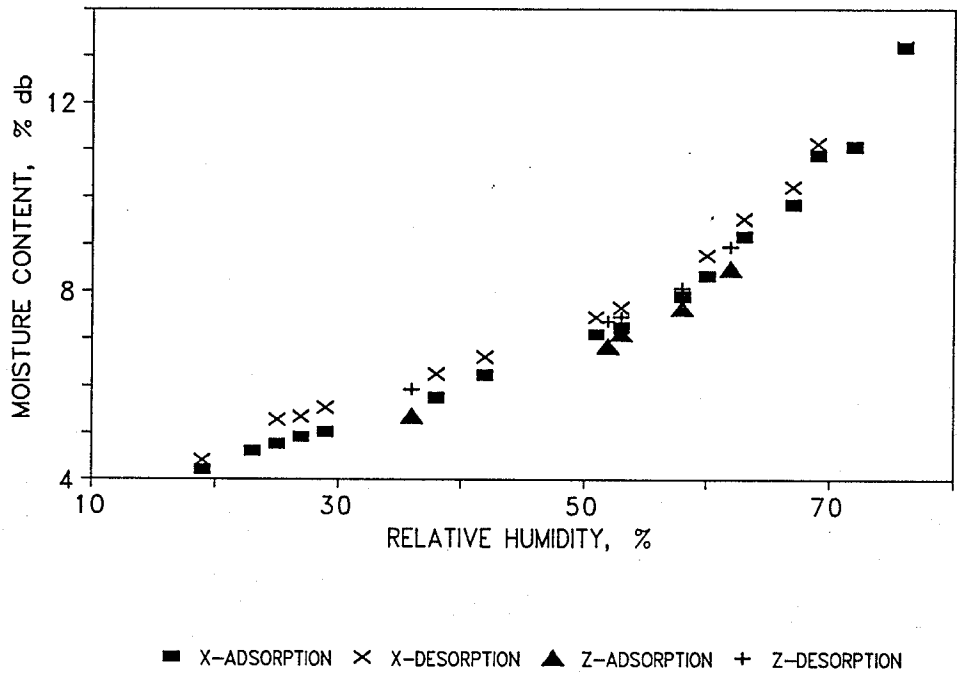


Fig. 7.3 Desorption and adsorption isotherms at 25^{0}C for two different samples of Westar canola.
X - 1989 crop, purchased from the local supplier.
Z - 1990 crop, harvested at Glenlea Farm.

The data points representing Z-sample lie below the X-sample data points. The difference between moisture contents of the X adsorption sample and Y adsorption sample, equilibrated with the air of approximately 23% RH (relative humidity), was approximately 1.3% db (Appendix A, Tables A1 and A2). The difference between moisture contents of X

adsorption sample and Z adsorption sample, equilibrated with the air at 53% RH was approximately 0.1% db (Appendix A, Tables A1 and A3). The hysteresis loop, between adsorption and desorption curves, for X and Z samples was constant at approximately 0.4%, db, for relative humidity ranging from approximately 24% to 67%. The Y-sample hysteresis loop increased in size, for relative humidity in range from approximately 50 to 70% RH, with the maximum of approximately 0.7% db at approximately 53% RH.

The results, shown in Fig. 7.2 and 7.3, indicated that the age of the sample as well as growing history of the sample influenced the equilibrium moisture content - relative humidity relationship for the adsorption and desorption process as well as its hysteresis loop.

The X sample after being subjected to rewetting/drying cycles was named sample C. The adsorption and desorption isotherms at 25^0C for the X-reference and C-samples are shown in Fig. 7.4.

It was pointed earlier the rewetting/drying cycles resulted in a significant mold development. The data points, representing adsorption and desorption samples, shifted down as a result of the pre-treatment. At approximately 23% RH, the moisture content of the C sample was approximately 3.6% db, while the moisture content of the nontreated, control sample was be approximately 4.6% db.

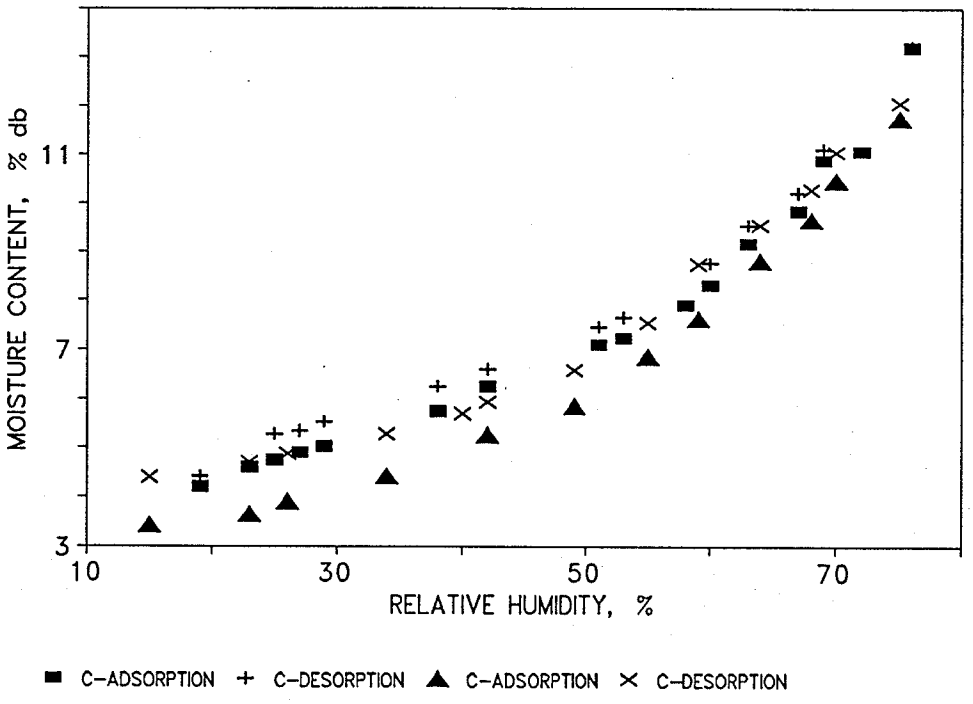


Fig. 7.4 Desorption and adsorption isotherms at 25 ^{0}C of the X-canola sample before and after being subjected to the rewetting/drying cycles.

The hysteresis loop also widened 2.5 times from 0.4 to 1.0% db, for the samples equilibrated with the air at approximately 23% RH, as the result of this pre-treatment.

The results, shown in Fig. 7.4, indicated that the rewetting/drying cycle pre-treatment, resulting in a significant mold development, influenced the equilibrium moisture content - relative humidity relationship as well as the size of the hysteresis loop. Data on equilibrium moisture content of canola should thus be accompanied by evaluation of the extend of microorganism growth within the

sample.

The adsorption and desorption experimental data for X and I sample (X- sample irradiated prior to the EMC tests) are shown in Fig. 7.5 while the experimental results for the X and S-sample (X subjected to the long-term stress prior to the EMC tests) are shown in Fig. 7.6.

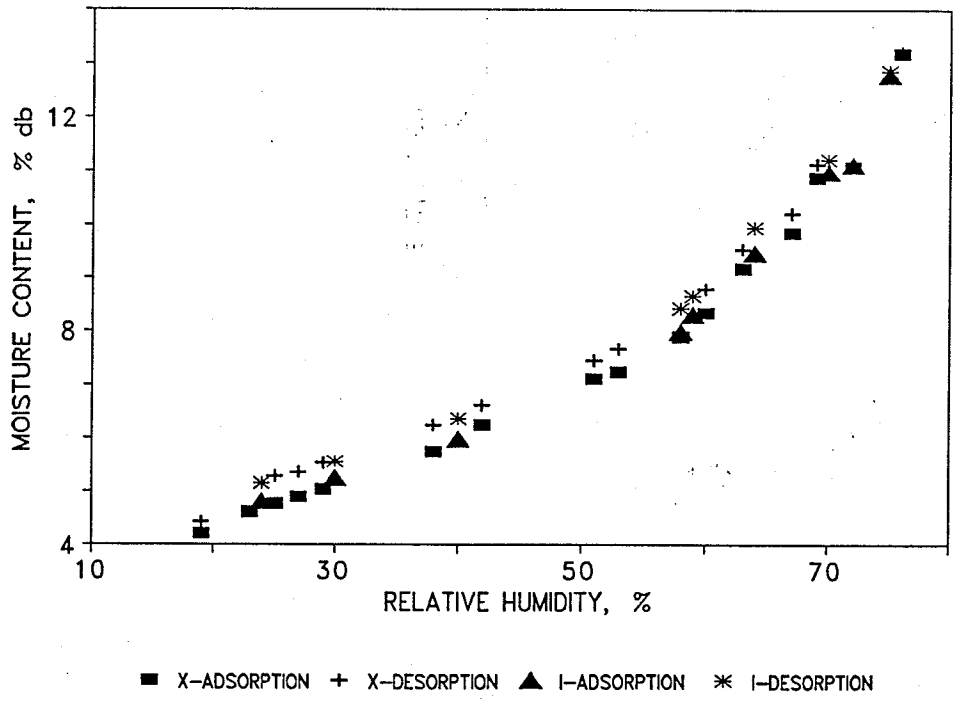


Fig. 7.5 Desorption and adsorption isotherms at 25 ^{0}C for the X canola sample before and after exposure to 10 kGy dose of irradiation.

The result shown in Fig. 7.5 and 7.6 proved that neither pre-treatment (irradiation or long lasting stress prior to the EMC tests) had a substantial effect on the adsorption and desorption isotherm shape and hysteresis

loop, within the investigated range of relative humidities.

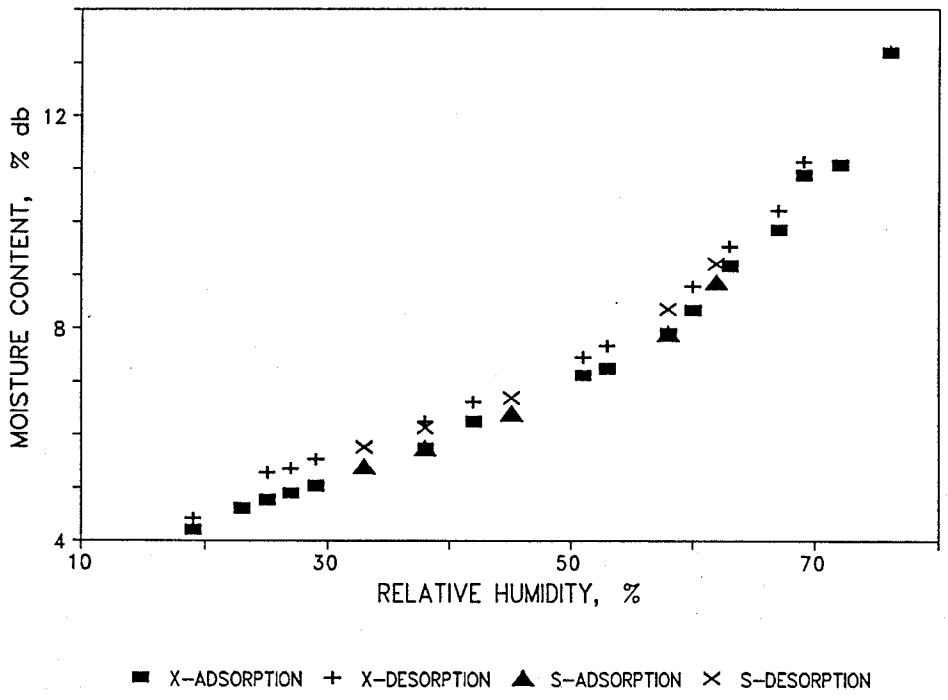


Fig. 7.6 Desorption and adsorption isotherms at 25^{0}C for the X canola sample before and after application of long-term stress.

Kapsalis (1981) stated that in nature a hysteresis loop has a practical implication of preventing the changes which are too rapid, such as loss of water. Mohsenin (1986) indicated that mechanical damage to the kernels results in the accelerated microorganism growth. In the case of X and Z sample, the observed hysteresis loop was found to be constant and equal to approximately 0.4% db, for relative humidity ranging from approximately 25% to 67%. The kernels of X and Z samples were in a comparatively good condition,

most kernels of the Y-sample were damaged. One could suspect a substantial microorganism development within the Y-sample.

A comparison of the hysteresis loops of X, Y, Z, and C samples indicated that the widening of hysteresis loop in the case of Y and C samples was caused by increased microorganism development within these samples.

The experimental data on equilibrium moisture content are usually fitted to one of the available mathematical models. To date the constants of the mathematical models for canola were calculated regardless of whether the canola was equilibrated through adsorption or desorption process. The non-linear regression procedure of SAS program (SAS, 1985) was used to estimate the constants of the Henderson's equation (Appendix B, Eq. 11, given by Henderson, 1952) for the X, Y, Z and C samples distinguishing adsorption and desorption isotherms. The values of constants in Henderson's equation, the standard deviations of the estimates and the sum of squares of residuals are given in Table A7 (Appendix A). Constants K and N in the Henderson's equation varied considerably depending on whether the equilibrium was reached through adsorption or desorption process. The K and N constants, for adsorption process of the C sample (X sample subjected to the rewetting/drying cycles) were approximately 0.049 and 1.397, respectively, while for the desorption process of the same sample were

approximately 0.026 and 1.628, respectively. The values of constants in Henderson's equation varied considerably depending of the pre-treatment history. The K and N constants, for adsorption process of the Y-sample, were 0.0156 and 1.7628 respectively while K and N constants, for the C sample were found to be 0.0488 and 1.3974.

7.3 Data from mechanical experiments

To evaluate the effect of pre-treatment of canola on its viscoelastic properties the single kernels were exposed to creep and recovery tests (Fig. 7.7). Each creep and recovery test was preceded with 30 loading/unloading cycles to eliminate plastic deformation and to establish a constant contact area. McLaughlin (1987) described the phenomena of yielding for the apple tissue which was in contact with compression plates during the compression tests. He indicated that fracture lines, turned brown when exposed to the air, were always perpendicular and nearest to the outside surface of the sample, which was in contact with compression plates. The fracture lines were discontinuous at some points indicating the end of the rapture zone.

The visual examination of the kernel, after loading/unloading cycles, showed that the top and bottom part of the kernels which were in contact with the compression plates, were flattened and did not recover to the original shape.

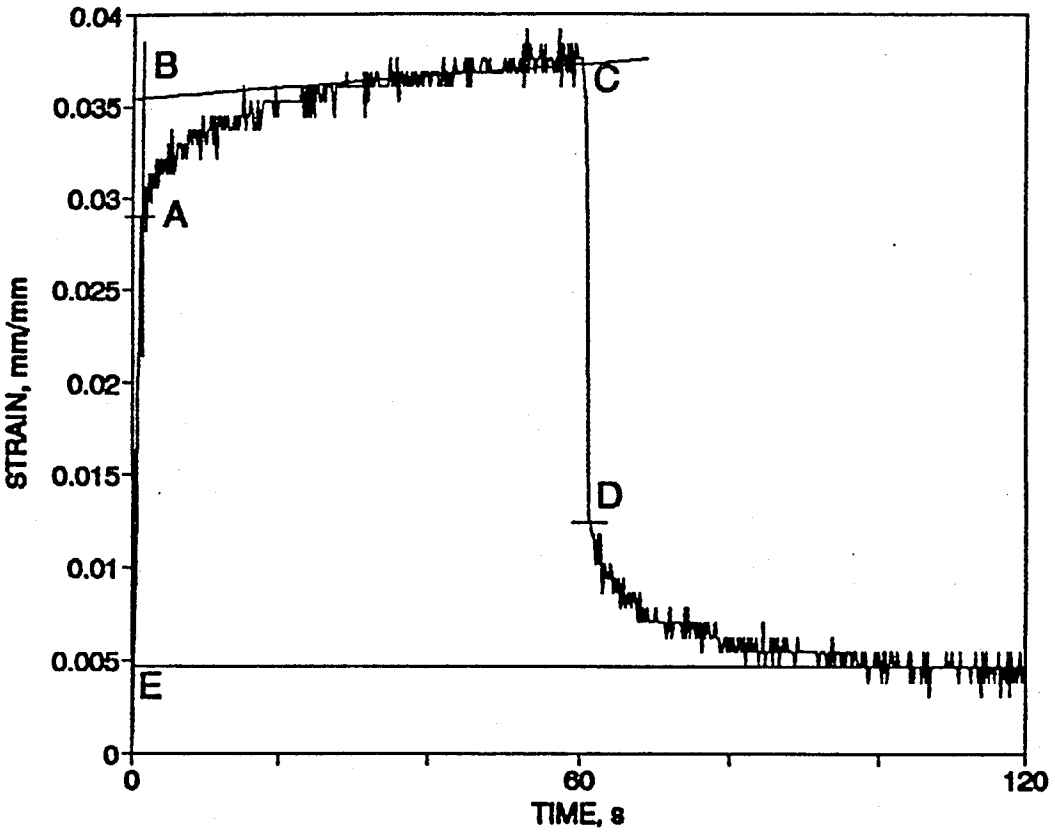


Fig. 7.7 Typical creep and recovery experiment preceded with loading/unloading cycles for canola kernel at 10.2% db moisture content (Appendix A, Table A22, Experiment No. 17).

This was because the contact stress was of a greater magnitude than the yielding stress for canola kernels. The cells crushed as a result of yielding were dislocated into the interior of the kernel. The cell rapture and dislocation were due to near point-loading of the original spherical surfaces. This phenomenon of initial, irreversible plastic deformation and dislocation of the raptured cells into the interior of the kernel before

constant contact area was established, will be referred as a "apparent yielding".

In further analysis it was assumed that the contact area does not change during the creep and recovery tests which were preceded with loading/unloading cycles.

A typical creep and recovery test preceded with 30 loading/unloading cycles is shown in Fig 7.7. Line from origins to A represents instantaneous loading. At point A the creep process starts and lasts until the load is removed at point C. Line CD represents instantaneous unloading with point D corresponding to the beginning of recovery period. The distance between origins and E is equal to the residual strain while the distance between points A and B equals the creep strain.

The results for creep and recovery tests, as well as cycle test, are presented in Appendix A in terms of deformations to avoid any systematic error and to facilitate future discussion on the obtained mechanical properties data. Standard deviation of estimates were calculated with respect to deformations.

7.3.1 Contact area data

Experimental results obtained in the contact area tests for X-sample, in terms of different moisture content levels, contact area diameter, contact area, total and elastic deformation, mean values and standard deviations are

shown in Appendix A (Tables A8).

The contact area between kernel and compression plates was evaluated assuming that the canola kernel was of spherical shape, and that the contact area of the kernel with the top plate equalled the contact area of the kernel with the bottom plate.

The linear regression procedure of the SAS program (SAS, 1985) was used to find the coefficients of a straight line representing a contact area diameter versus moisture content relationship, for moisture content ranging from 4.3 to 11.7%, db. The relation obtained was:

$$DIAMETER = 0.0874 \cdot (MC) - 0.0927 \qquad (14)$$

where:

DIAMETER - contact area diameter, mm

MC - moisture constant, % db.

Experimental data and the regression model are shown in Fig. 7.8.

The correlation coefficient (R Squared, Appendix A, Table A9) was 0.90 for this model.

Average contact area increased with the moisture content from approximately 0.0836 mm^2 at 4.3% to 0.7548 mm^2 at 11.7% db moisture content.

Coefficient of variation decreased with moisture content from approximately 41% at 4.3% db to 14% at 6.7% db to approximately 5% for 8% db moisture content.

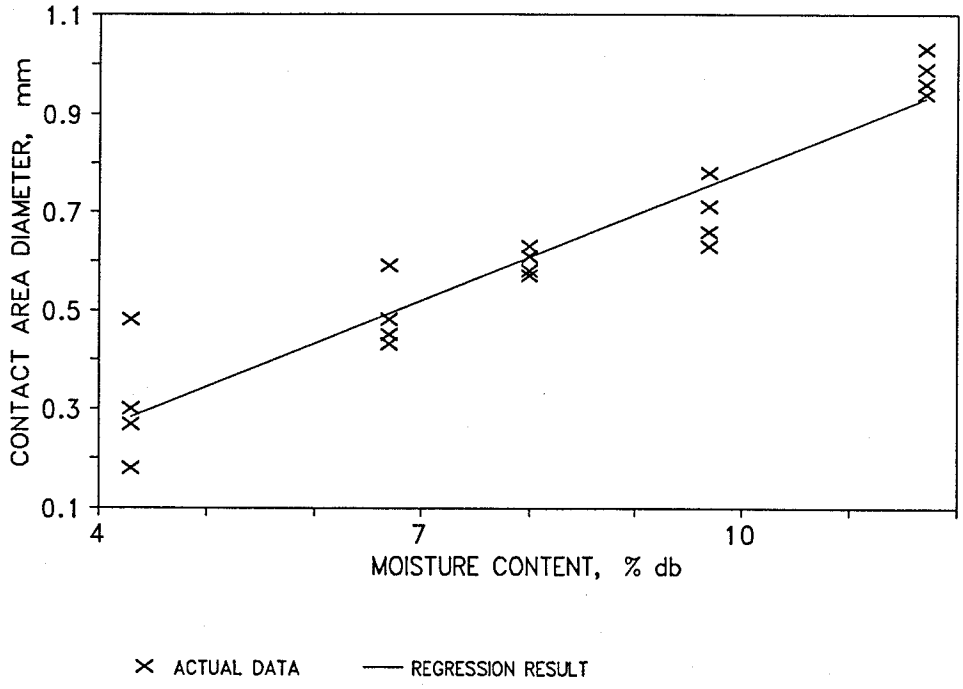


Fig. 7.8 Contact area diameter versus moisture content based on the loading/unloading cycle tests.

It was concluded that for the kernels at low moisture contents the magnitude of the contact stress, depending on contact area, varied substantially from kernel to kernel.

7.3.2 Different pre-treatment data

Three separate X-samples were subjected to different pre-treatments: C-sample to the rewetting/drying cycles, I-sample to the irradiation and S- sample to the long-term stress. The pre-treatment effect on the mechanical properties of canola was studied on the samples equilibrated with the air through the adsorption process. The X-

adsorption sample, equilibrated with the air at exactly the same conditions as the sample under the investigation, was used as a reference. The effect of different pre-treatments were studied at different equilibrium moisture content levels.

Rewetting/drying cycles

The viscoelastic properties of the C-sample were studied for the kernels equilibrated with the air of 23% and 42% relative humidity. The experimental data, for C-adsorption and desorption samples, as well as the reference sample, are given in Appendix A (Tables A9 to A13) in terms of deformations, average values of deformations, standard deviations of deformations, ratio of elasticity (A/B, Fig. 6.3) and kernel diameter.

It was concluded, based on the t-tests (α = 95%) (Appendix A, Table A23), using the instantaneous loading deformation as a criterion, that the rewetting/drying pre-treatment and consequent molding caused a significant change in viscoelastic properties of the canola kernels at both moisture content levels.

Average values of instantaneous loading deformation, for the samples equilibrated at 42.3% relative humidity, were 0.0593 mm with 0.0072 mm standard deviation for the C-adsorption sample (5.3% db moisture content) and 0.0469 mm with 0.0090 mm standard deviation for reference sample (6.2%

db moisture content). The instantaneous loading deformation increased approximately 0.0.0124 mm (26%) as a result of the pre-treatment.

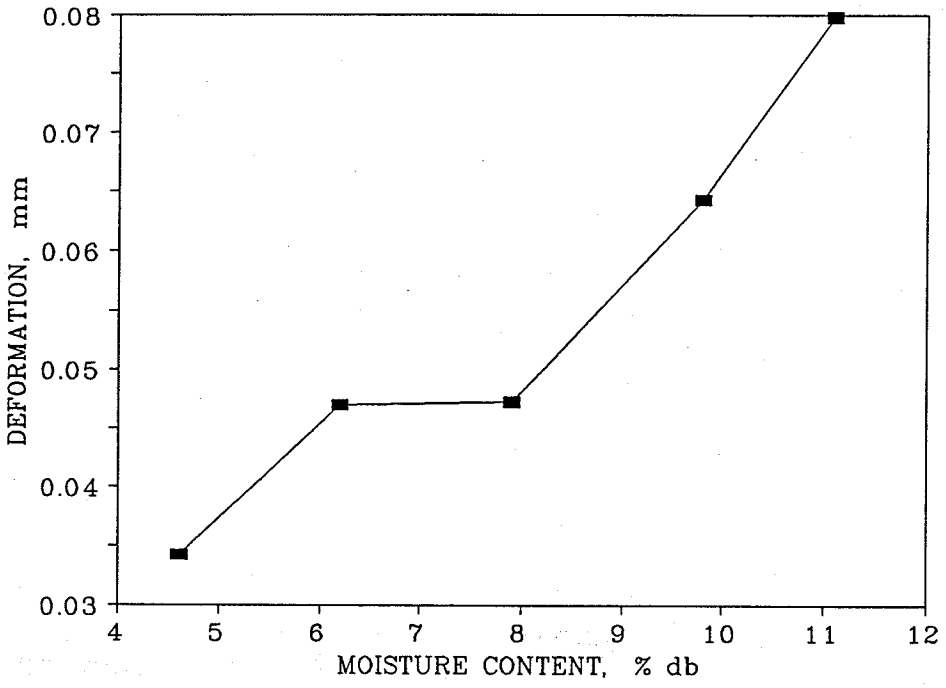


Fig. 7.9 Average instantaneous loading deformation versus moisture content for X adsorption reference sample in creep and recovery test.

Predicted change in instantaneous loading deformation due to the moisture content difference alone of approximately 0.9% db between C and X sample was approximately 0.008 mm (Fig. 7.9). Therefore, the actual change in instantaneous loading deformation of approximately 0.0124 mm between X and C samples, caused by rewetting /drying pre-treatment, was approximately 35% greater than the change predicted by the moisture content difference.

A similar pre-treatment effect was noticed for the samples equilibrated at approximately 23% RH. Average values of a creep deformation, for the samples equilibrated at 23% relative humidity, were found to be 0.0048 mm with 0.0019 mm standard deviation for the C-adsorption sample (3.6% db, moisture content) and 0.0034 mm with 0.0010 mm standard deviation for reference sample (4.6% db moisture content). The creep deformation increased approximately 0.0014 mm (29%) as a result of the pre-treatment.

Irradiation

The data for I-adsorption and reference sample are shown in Appendix A, Tables A18 and A19. The effect of irradiation was studied for the samples equilibrated with air at 72% relative humidity.

No significant effect of irradiation on mechanical properties of the canola kernels was proven, based on the results of two sample t-tests (α = 95%) performed for this sample.

The average value of instantaneous loading deformation for the irradiated, adsorption samples was found to be approximately 0.0763 mm with 0.0153 mm standard deviation, (11.1% db moisture content), and 0.0798 mm with 0.0143 mm standard deviation for reference sample (11.1% db moisture content).

Constant pressure application

The mechanical properties experimental data, for the S and reference sample are given in Tables A14 to A17 (Appendix B). Prior to the EMC experiments two series of mechanical tests (Appendix A, Table A14 and A15) were performed. Their objective was to prove that recovery of canola in bulk which took place in a short time after removing a dead load did not play a significant role during the EMC tests and basic mechanical properties tests. The first series of tests were performed an hour after the pressure on the canola bed was removed. The second series of experiments was performed 7 days after the dead load was removed. A two sample t-test (α = 95%) (Appendix A, Table A23) showed no significant difference in mechanical properties between these two samples.

The mechanical properties experimental data for the S-adsorption and reference sample, equilibrated with the air of approximately 58% RH, is shown in Appendix A, Table A16 and A17.

It was concluded, based on a t-test (α = 95%) (Appendix A, Table A23), with the instantaneous loading as a criterion, that constant pressure application caused a significant change in mechanical properties of canola kernels.

The average value of the instantaneous loading

deformation was approximately 0.0473 mm with approximately 0.0068 mm standard deviation for reference sample and 0.0577 mm with 0.0065 mm standard deviation for sample subjected to a long lasting stress. The instantaneous loading deformation decreased approximately 0.0104 mm (22%) as a result of the pre-treatment.

The average value of creep deformation was found to be 0.009 mm with 0.0013 mm standard deviation for the S-adsorption sample (7.9% moisture content, db), and 0.0102 mm with 0.0008 mm standard deviation for reference sample (7.9% moisture content, db). The creep deformation decreased approximately 0.0013 mm (13%) as a result of the pre-treatment.

Z-sample

The mechanical properties experimental data for the Z-adsorption sample, at 8.5% db moisture content, equilibrated with air of approximately 62% relative humidity, are shown in Appendix A, Table A20.

The average value of creep deformation was approximately 0.0107 mm with 0.0008 mm standard deviation and loading deformation approximately 0.0463 mm with 0.0077 mm standard deviation. The average ratio of elasticity was 0.3746 with 0.0797 standard deviation.

The average value of creep deformation for X-sample at 8.5% db moisture content was 0.011 (Fig. 7.10). The average

value of loading deformation was 0.053 (Fig. 7.8). The average value of ratio of elasticity was approximately 0.46 (Fig. 7.11).

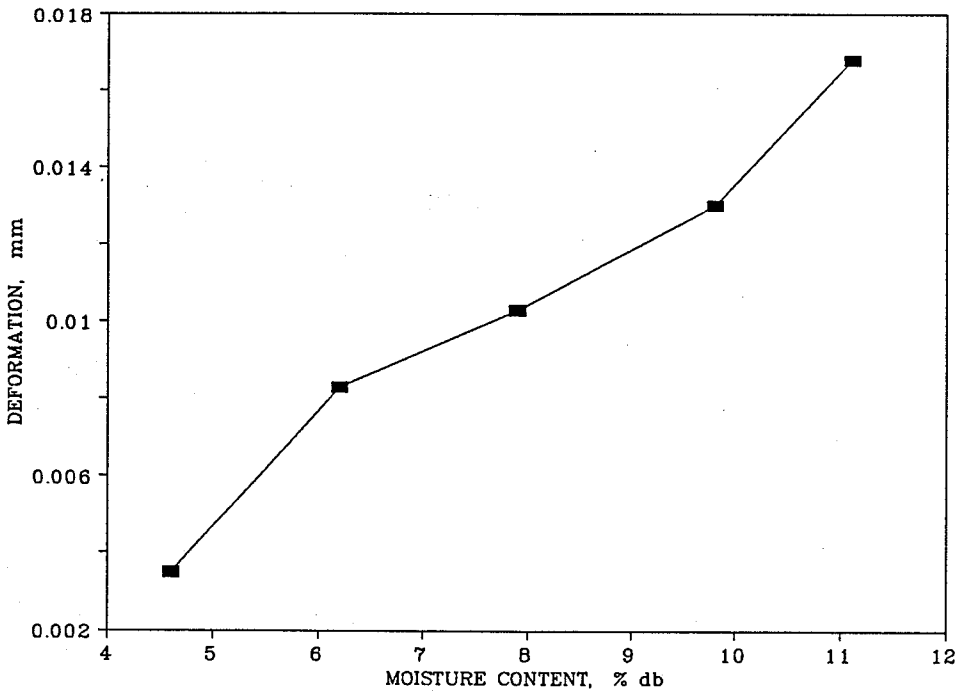


Fig. 7.10 Average creep deformation versus moisture content for X adsorption (reference) sample.

Comparison of the experimental data for Z and X-sample at 8.5% db moisture content indicated that creep deformation for both samples were approximately of the same magnitude. The loading deformation was approximately 13% greater for X sample than for Z-sample. The ratio of elasticity was approximately 19% greater for X-sample than for Z-sample.

Based on the data obtained for both samples it was

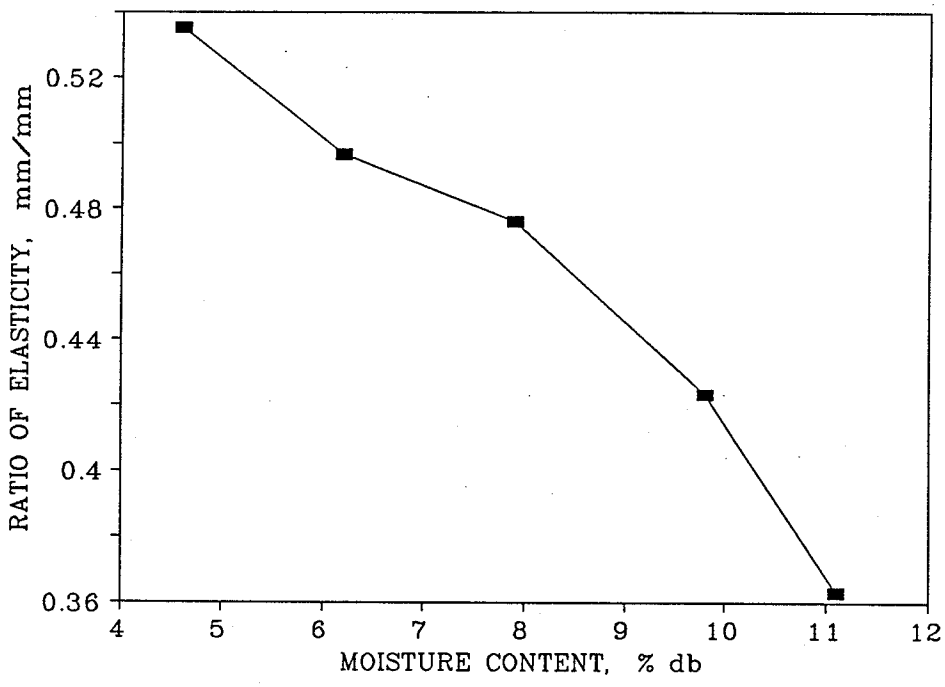


Fig 7.11 Ratio of elasticity versus moisture content for X adsorption reference sample.

concluded that age and growing history of the sample influenced its rheological properties.

7.3.3 Mechanical hysteresis

The mechanical experiments results for the X-adsorption and desorption samples, equilibrated with the air at 67% relative humidity, are shown in Appendix A, Tables A21 and A22. All the average deformations corresponding to the creep and recovery tests were of the same magnitude for both samples. Two sample t-test (Appendix A, Table A23), with the ratio of elasticity as a criterion, indicated that there

was a significant difference in mechanical behaviour between canola kernels coming from the adsorption and desorption samples.

The adsorption and desorption samples equilibrated with the air of approximately 67% relative humidity to approximately 9.8 and 10.2% db moisture content, respectively. Therefore, the moisture content hysteresis loop was approximately 0.4% db. The average ratio of elasticity was approximately 0.4234 mm/mm for the adsorption sample and 0.3 mm/mm for the desorption sample. Therefore, the ratio of elasticity hysteresis loop was approximately 0.1234 mm/mm.

The difference in ratio of elasticity for the adsorption samples at 9.8 and 10.2% db moisture content was approximately 0.02 mm/mm (Fig. 7.11). The actual difference in ratio of elasticity between adsorption and desorption samples of 0.1234 mm/mm was approximately 6 times greater than the difference of 0.02 mm/mm corresponding to the moisture content loop of 0.4% db.

It was concluded, that the water molecules are incorporated into the kernel structure in a different way in the kernels equilibrated with the air through adsorption and in the kernels equilibrated through desorption process. This dissimilarity causes a significant difference in the mechanical properties of the canola kernels equilibrated with the air through the adsorption process and canola

kernels equilibrated through the desorption process.

7.3.4 Burgers model versus viscoelasto - plastic model

The goal of these studies has been to develop a rheological model which could describe the viscoelastic properties of single canola kernel based on the creep and recovery test which is not preceded with loading/unloading cycles. Experimental data, shown in Appendix A, for reference sample (X-adsorption) obtained in creep and recovery experiments, preceded by loading/unloading cycles, were used to calculate the modulus of elasticity corresponding to instantaneous loading and unloading strains describing behaviour of the first elastic element of the Burgers model (Fig. 2.2 and 2.3). Each creep and recovery test was preceded with loading/unloading tests to eliminate the apparent yielding behaviour of the kernel.

The modulus of elasticity was calculated based on the assumption that contact area between the kernel and compression plates did not change after the loading/ unloading cycles and stayed constant during the each creep and recovery experiment. The magnitude of the stress was found by dividing the force acting on the kernel by the contact area calculated from Eq. 16.

The calculated values of the instantaneous loading and unloading modulus of elasticity as a function of moisture content are shown in Fig. 7.12.

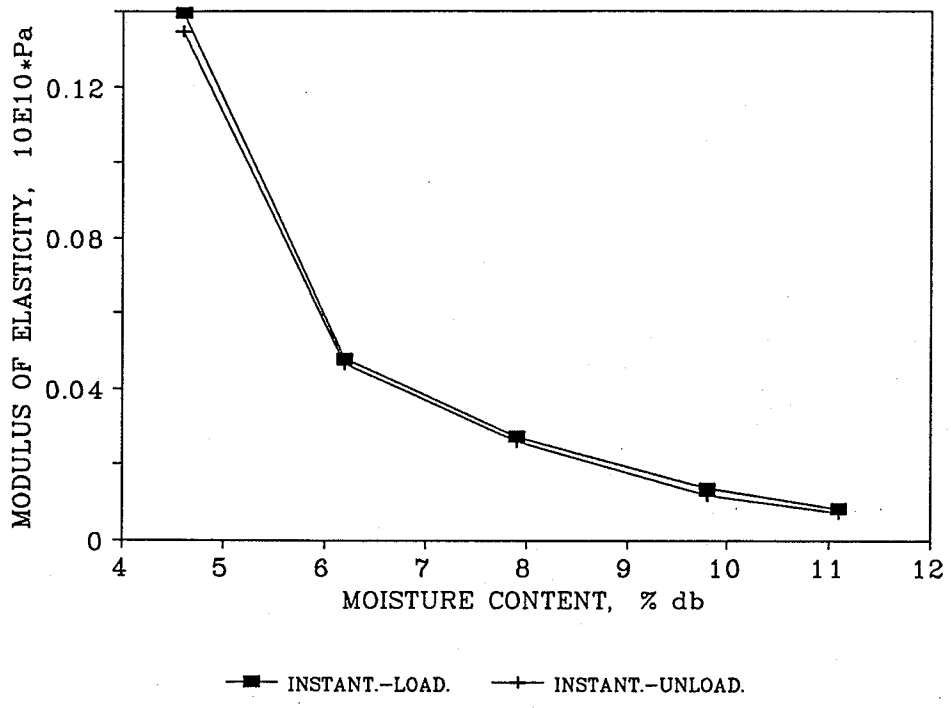


Fig 7.12 Average instantaneous modulus of elasticity versus moisture content for X adsorption (reference) sample.

Apparent moduli of elasticity representing instantaneous loading and unloading decreased with the moisture content. All the other elements of the Burgers model may be calculated applying the same methodology.

The above approach of eliminating first plasticity and then evaluating viscoelastic properties of the biological material reflects viscoelasto-plastic behaviour of the single canola kernel. The viscoelastic properties of the single canola kernel may be well represented by a simple Burgers model only when creep and recovery test is preceded

with the loading/unloading cycles. In such case, the strains corresponding to the elements of the Burgers model should be related not to the initial size of the kernel but to the height of the "barrel" which results because of the precycling. Therefore precycling of the kernel prior to the creep and recovery test influences in a obvious way the viscoelastic properties of the canola kernels represented by the Burgers model.

An example of the creep and recovery test, which was not preceded with the loading/unloading cycles, is given in Section 6.2.1. The differences between creep and recovery, loading and unloading strains were caused by apparent yielding and resulting in irreversible plastic deformation, which occurred during instantaneous loading and creep period of the creep and recovery test.

The process of instantaneous compression, creep, instantaneous response to unloading and recovery of the single canola kernel during the creep and recovery test, which was not preceded with the loading/unloading cycles, is represented by a viscoelasto - plastic model (Fig. 7.13).

This model consists of a plastic component and a viscoelastic component. The viscoelastic component was represented by the Burgers model described in Chapter 2 section 2.4.6. The plastic component of the model was defined as:

$$\varepsilon_D(t) = \varepsilon_L + \varepsilon_c(1-e^{-kt}) \qquad (15)$$

where:

ε_D = dissipated strain, mm/mm

ε_L = strain dissipated during instantaneous loading, mm/mm

ε_C = strain dissipated during the entire creep, mm/mm

t = time, s

k = empirical constant

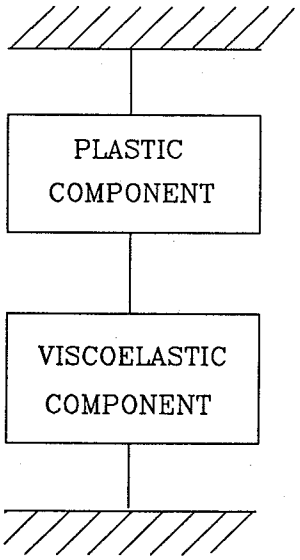


Fig. 7.13 Viscoelasto - plastic model.

The graphical representation of the viscoelasto-plastic model is shown in Fig. 7.14.

The magnitude of the irreversible deformation and, therefore, of the dissipated strain, defined in Eq. 15, caused due to the apparent yielding, depend on the magnitude of the applied load. If the contact stress is such that

apparent yielding does not occur the plastic component behaves as a rigid body.

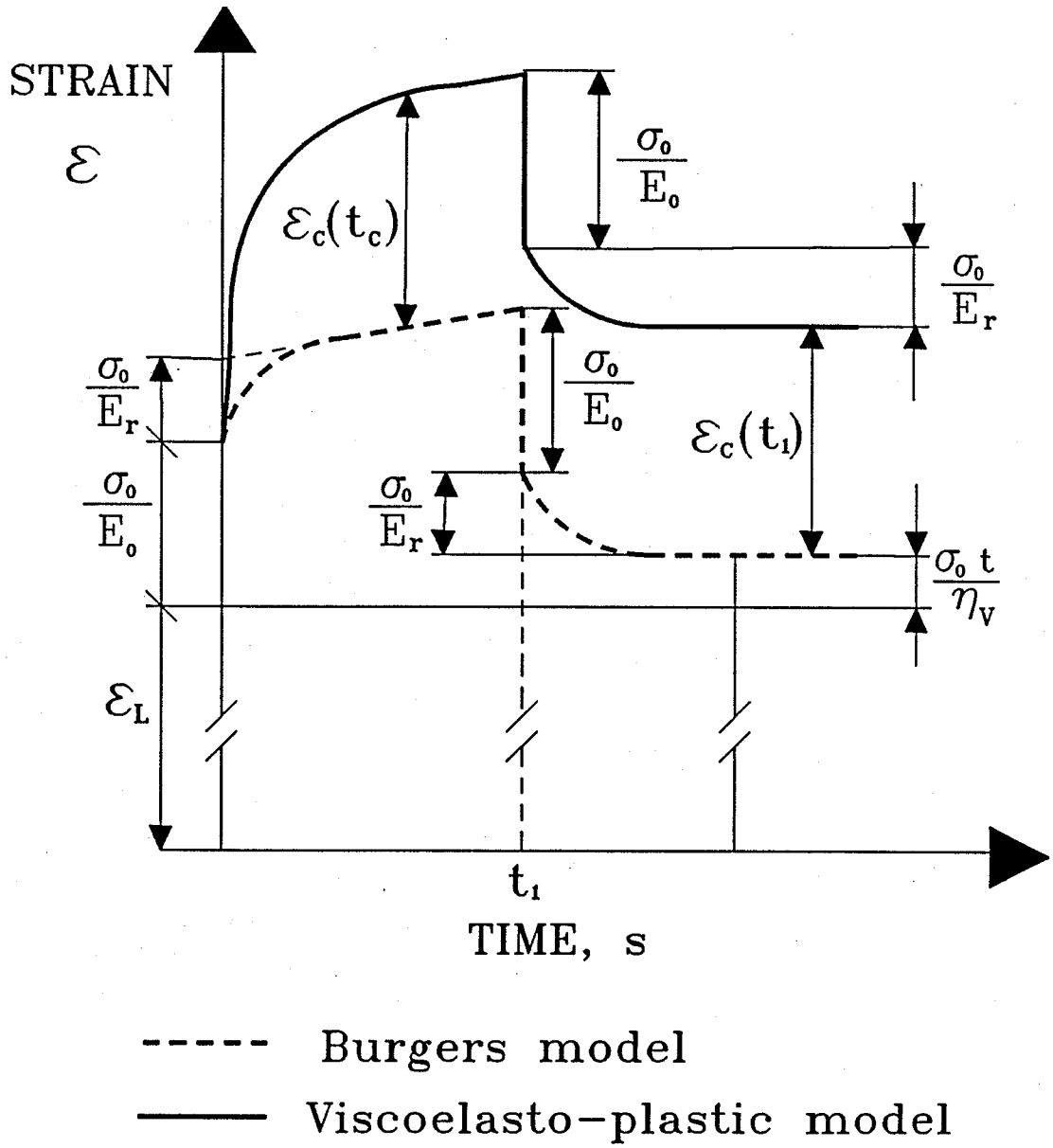


Fig. 7.14 Graphical representation of the viscoelasto-plastic model.

The total instantaneous strain of the material, represented by the viscoelasto-plastic model, consists of instantaneous elastic strain of element E_0 of the Burgers

model and instantaneous irreversible strain (ε_L) of the plastic component. The total creep strain of the material, represented by the viscoelasto-plastic model, consists of the strain due to the first viscous element (η) interaction with the second elastic element (E_r) (Burgers model), strain of the second viscous element (η_v) (Burgers model) and strain due to the plastic component (ε_c). Because plastic component behaves like a rigid body during the recovery period the recovery period, it is represented by a simple Burgers model.

The plastic component in the viscoelasto-plastic model is connected in series with four other elements representing the Burgers model. Therefore, mathematical representation of the viscoelasto-plastic model is a superposition of the solution given in Eq. 15 and the solutions given in Eq. 10 for creep and in Eq. 11 for recovery. The solution representing creep behaviour is:

$$\varepsilon(t) = \frac{\sigma_0}{E_0} + \varepsilon_D + \frac{\sigma_0}{E_r}\left(1 - e^{-\frac{t}{T_r}}\right) + \frac{\sigma_0 t}{\eta_v} \qquad (16)$$

and recovery behaviour is:

$$\varepsilon(t) = \frac{\sigma_0 t_1}{\eta_v} + \varepsilon_D(t_1) + \frac{\sigma_0}{E_r}\left(e^{\frac{t_1}{T_r}} - 1\right) e^{-\frac{t}{T_r}} \qquad (17)$$

where:

$\varepsilon(t)$ = strain mm/mm

$\varepsilon_D(t_1)$ = strain dissipated during instantaneous

loading and entire creep, mm/mm

η = viscosity coefficient corresponding to retarded elastic deformation, Pa·s

η_v = viscosity coefficient of second viscous element corresponding to Newtonian flow, Pa·s

σ_0 = contact load stress, Pa

E_0 = instantaneous elastic modulus, Pa

E_r = modulus of elasticity of a second elastic element, Pa

T_r = retardation time (η/E_r), s

The constants in Eq. 16 and 17 representing plastic component as well as elements of the Burgers model component were calculated, as an example, for the experimental data given in Appendix A, Table A24 for the test shown in Fig. 6.2. In this experiment creep deformation was 2.9 times greater than recovery deformation and instantaneous loading deformation was 2.9 times greater than instantaneous unloading deformation. This was because creep and recovery test was not preceded with the loading/unloading cycles.

Retardation time was calculated, by differentiating Eq. 11 (or Eq. 17) over the time and substituting numerical values (Appendix A, Table A24):

$$\frac{d\varepsilon(t)}{dt} = \frac{\sigma_0}{E_r T_r}\left(e^{\frac{t_1}{T_r}} - 1\right) e^{\frac{-t_1}{T_r}} = tg\beta \qquad (18)$$

$$\frac{0.018}{T_r}\left(e^{\frac{60}{T_r}}-1\right)e^{-\frac{60}{T_r}} = 0.00325 \qquad (19)$$

where:

$tg\beta$ = rate of strain at the end of the creep period, 1/s

as 5.5 s.

The magnitude of σ_0/η_v was calculated, based on the assumption that creep behaviour at time approaching 60 s is represented by a straight line which means that at this time apparent yielding and retarded elastic deformation approached asymptotically their maximum values. Therefore, differentiating Eq. 10 (or 16) over the time and substituting numerical values (Appendix A, Table B24):

$$\frac{d\varepsilon(t)}{dt} = \frac{\sigma_0}{E_r T_r}\, e^{-\frac{t}{T_r}} + \frac{\sigma_0}{\eta_v} = tg\alpha \qquad (20)$$

$$\frac{\sigma_0}{\eta_v} = tg\alpha = 0.000066 \qquad (21)$$

where:

$tg\alpha$ = rate of strain at the beginning of the recovery period, 1/s

Finally, the Burgers model component for the creep period is:

$$\varepsilon(t) = 0.043 + 0.018\left(1-e^{-\frac{t}{5.5}}\right) + 6.6x10^{-5}t \qquad (22)$$

and for the recovery period:

$$\varepsilon(t) = 0.004 + 984.062\, e^{-\frac{t}{5.5}} \qquad (23)$$

To calculate constant k in Eq. 15, line representing

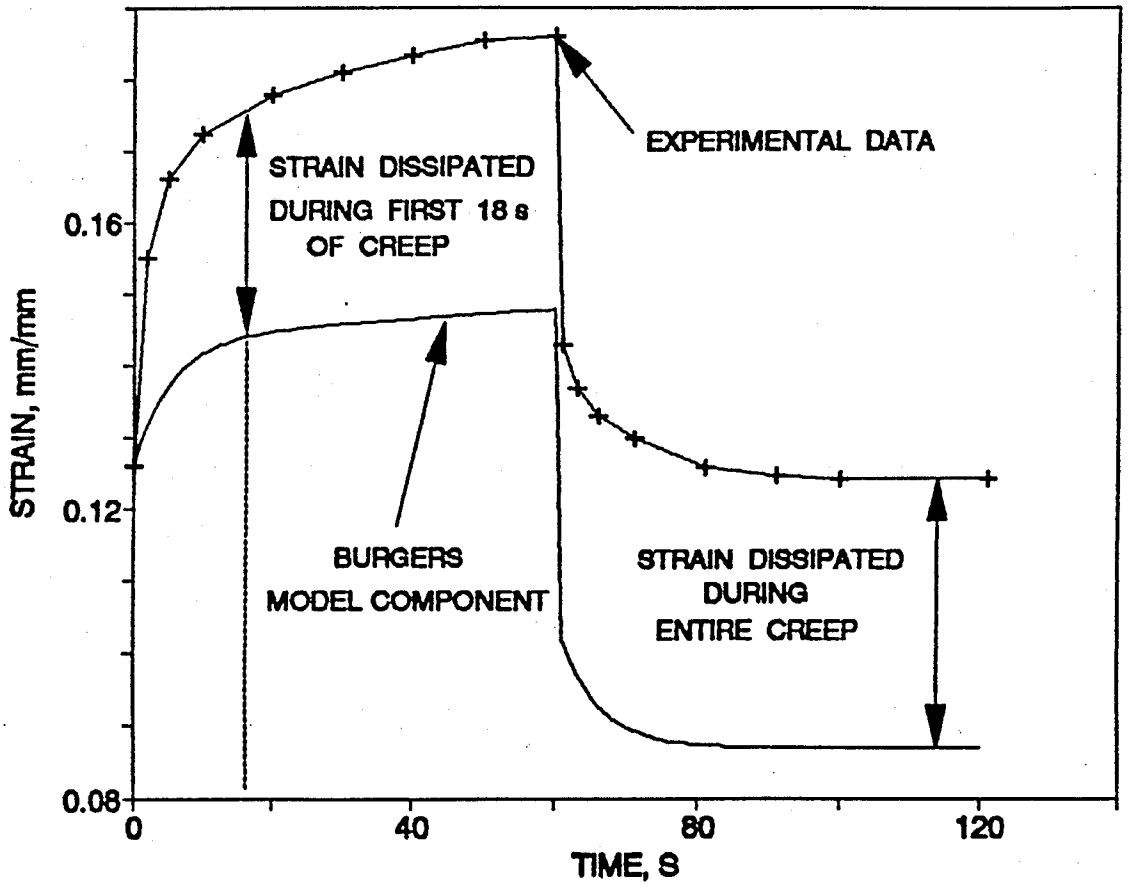


Fig. 7.15 Evaluation of the strain dissipated during creep period.

the Burgers model component in viscoelasto-plastic model was shifted upward by the value corresponding to the strain which was dissipated during instantaneous loading (Fig.

7.15). The differences between the curves, representing experimental data and the Burgers model component of the viscoelasto-plastic model, were measured over the time. The strain dissipated over the time, during instantaneous loading and creep is shown in Fig. 7.16.

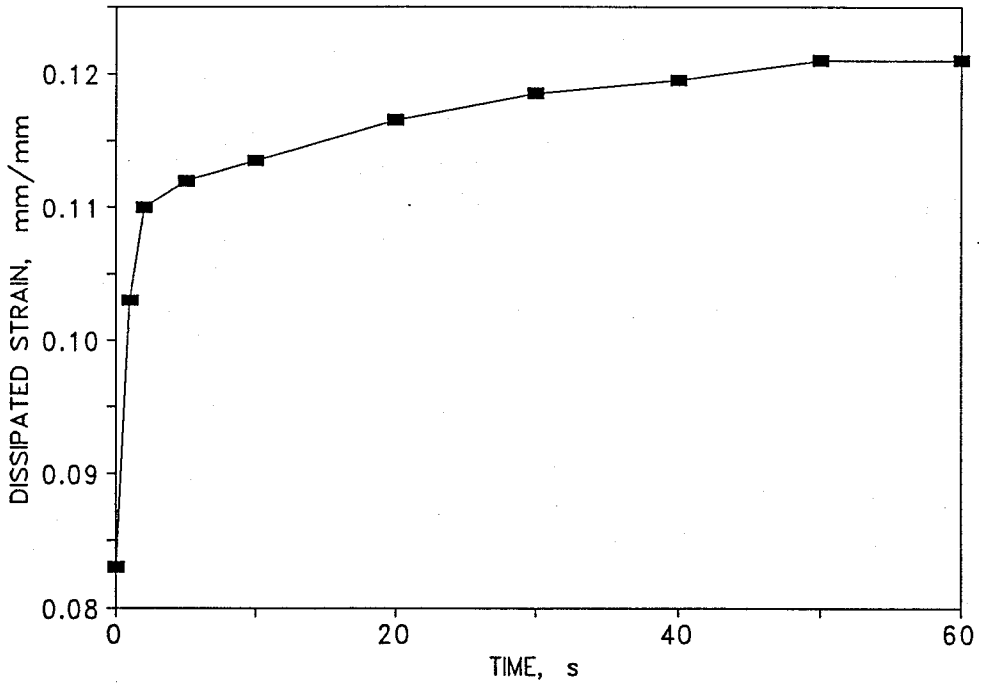


Fig. 7.16 Strain dissipated during instantaneous loading and creep period.

The nonlinear regression procedure of the SAS program was used to calculate constant k in Eq. 15.

Mathematical representation of the plastic component in the viscoelasto-plastic model is:

$$\varepsilon_D(t) = 0.083 + 0.038\,(1 - e^{-0.224t}) \qquad (24)$$

Therefore, the mathematical solution of the viscoelasto-plastic model for the creep period is:

$$\varepsilon(t) = 0.126 + 0.038\,(1-e^{-0.224t}) + 0.018\,(1-e^{-\frac{t}{5.5}}) + 6.6x10^{-5}t \tag{25}$$

And for the recovery period:

$$\varepsilon(t) = 0.125 + 984.062\,e^{-\frac{t}{5.4}} \tag{26}$$

The experimental data and curve corresponding to the mathematical solutions of the viscoelasto-plastic model, expressed in Eq. 25 and 26, are shown on the same graph in Fig. 7.18.

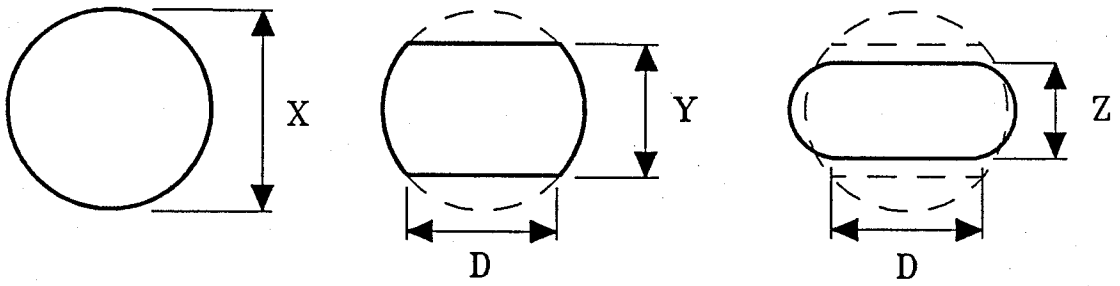


Fig. 7.17 Kernel deformation during creep and recovery test.

Contact area was found based on the drawing in Fig. 7.17. The kernel was considered to be a sphere which was trimmed at both ends at a depth equal to the half of irreversible plastic deformation caused by apparent yielding (excluding the deformation due to the second viscous element). Contact area was approximately 0.5436 mm^2.

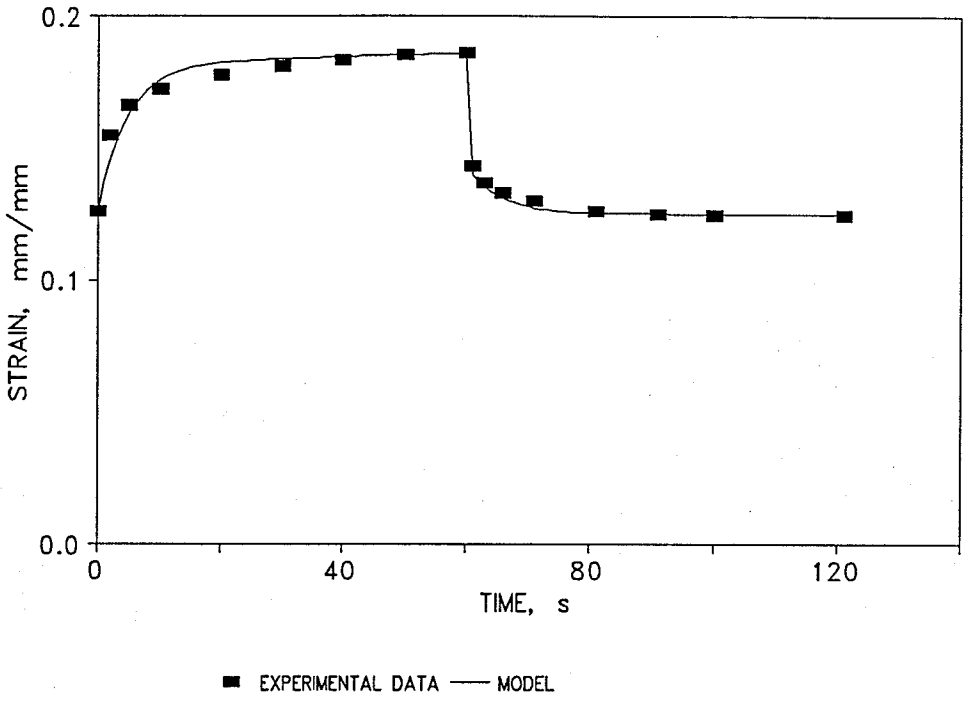


Fig. 7.18 Experimental data and curve corresponding to mathematical solution of viscoelasto-plastic model for canola kernel at 20.5% db moisture content.

Apparent moduli of elasticity were found based on the graphical description in Fig. 7.14 and experimental data shown in Appendix A, Table A24. The magnitude of the contact stress was $\sigma_0 = 2/(0.5436 \times 10^{-6}) = 3.68 \times 10^6$ Pa. Instantaneous modulus of elasticity was approximately 8.56×10^7 Pa. Modulus of elasticity of the second elastic element was approximately 2.04×10^8 Pa. Viscosity coefficient corresponding to the first viscous element was approximately 1.12×10^9 Pa·s. Viscosity coefficient corresponding to the second viscous element was approximately 5.52×10^{10} Pa·s.

7.4 Error analysis

The RH-2 relative humidity sensors, according to manufacturers specification, have ±2% accuracy of relative humidity measurement. The RH-2 sensors were calibrated using the H-1 dew point sensor as a reference. The maximum deviation from the expected value was approximately 1.2% from the mean relative humidity.

The oven method was used to measure the moisture content of the canola sample. The maximum deviation from the mean value for the repeated moisture content measurements was no greater than ±0.06% moisture content, db.

The nonlinearity of the 242-0000 LVDT sensor, used to measure the deformation of the kernel, was, according to the manufacturers, within 0.5% for ± 0.25 mm range which is equivalent to 0.0025 mm. Nonlinearity of the sensor, related to the output fluctuations shown in Fig. 7.4 was approximately 0.0044 mm. Resolution of the LVDT sensors, according to manufacturers, is infinite. Accuracy of the measurements, derived from the average interpolated value (Fig. 7.4) of the sensor output, was proven, by the means of the calibration using the clearance gages, to be less than 0.001 for all the LVDT sensors used in the experiments.

Cenkowski et al, 1990, indicated a semi-dead load as a factor contributing to the error in modulus of elasticity evaluation. The load decreases during the creep period

because the kernel deforms as result of the pressure, which causes a spring relaxation (Fig. 4.2). This influences elastic rebounce of the kernel during the unloading process. Maximum average creep deformation was approximately 0.017 mm (Appendix A, Tab A18), which caused approximately 1.5% force decrease during the creep period. The average strain corresponding to the instantaneous modulus of elasticity for the same series of experiments (Appendix B, Table B10) was approximately 0.075 mm. Contact area diameter were measured within $\pm$ 0.01 mm accuracy.

The accumulated error in the instantaneous modulus of elasticity for the canola at 11.1% db moisture content was calculated according to the formulas:

$$\delta = \frac{\Delta E_0}{E_0} 100\% \tag{27}$$

where:

$$E_0 = \frac{4Fl}{\pi D^2 d} \tag{28}$$

and:

$$\Delta E_0 = \sqrt{\left(\frac{\partial E_0}{\partial l}\Delta l\right)^2 + \left(\frac{\partial E_0}{\partial F}\Delta F\right)^2 + \left(\frac{\partial E_0}{\partial d}\Delta d\right)^2 + \left(\frac{\partial E_0}{\partial D}\Delta D\right)^2} \tag{29}$$

where:

E_0= instantaneous modulus of elasticity, Pa

δ = error, %

F = load, N

l = kernel deformation, m

D = contact area diameter, m

d = kernel diameter, m

Δ = error,

The accumulated error in the instantaneous modulus of elasticity (8×10^7 Pa) was approximately 4.7%. The major contributions to the error were by the kernel deformation and contact area diameter measurements.

8. CONCLUSIONS

Based on the result of these studies following specific conclusions can be drawn:

1. The age and growing history of canola have significant effects on the EMC - ERH relationships for adsorption and desorption processes, hysteresis loop between adsorption and desorption isotherms and viscoelasto-plastic properties of the canola. The difference in EMC between two adsorption samples harvested at different locations was approximately 1.3% db at 23% RH. A discrepancy in instantaneous loading deformation of 13% was attributed to the difference in age and growing history of two canola samples at 8.5% db MC.

2. The rewetting/drying cycle pretreatment, resulting in a significant mold development, influences the EMC - ERH relationship, the size of the hysteresis loop and viscoelasto-plastic properties of the canola kernels. The EMC hysteresis loop widened 2.5 times as a result of the pretreatment for the samples equilibrated with air at approximately 25% RH. The pretreatment caused a change in instantaneous loading deformation 35% greater than that predicted due to the EMC difference alone for samples equilibrated at 42% RH.

3. The long-term stress on the canola sample does not have a significant effect on the adsorption and desorption isotherms.

4. The long-term stress on the canola sample causes a change in viscoelasto-plastic properties of the canola kernels. The instantaneous loading deformation increased approximately 22% as a result of the pretreatment for the samples equilibrated with air at 58% RH.

5. EMC behaviour and viscoelasto-plastic properties of the canola are not affected by irradiation pretreatment.

6. Viscoelastic properties of canola differ for adsorption

and desorption sample. Difference in viscoelasto-plastic properties of canola is greater than that can be explained by the EMC loop between adsorption and desorption isotherms. The actual difference in average ratio of elasticity between adsorption and desorption sample was approximately 6 times greater than the difference corresponding to the moisture content loop alone for the canola kernels equilibrated at 67% RH.

7. Viscoelastic properties of a single canola kernel may be evaluated using a 4-element Burgers model only in the case when the plasticity of kernel is eliminated prior to the creep and recovery tests.

8. Viscoelasto-plastic behaviour of a canola kernel during creep and recovery test may be explained using rheological model consisting of a plastic component in series with a viscoelastic component.

9. SUGGESTIONS FOR FUTURE RESEARCH

1. The viscoelasto-plastic model should be evaluated for different loads. Nonlinearity (depending on load) of viscoelasto-plastic properties of canola should be assumed because of the shape of the sample. The relationship between strain and stress should be expressed in terms of

the creep compliance (strain per unit stress). Plastic strain should be expressed as a function of stress.

2. The phenomena of mechanical hysteresis should be studied in greater details. Canola equilibrated with the air through desorption process exhibits much greater plastic deformation under the load than canola equilibrated through the adsorption process. This may be beneficial in oil extraction process.

3. The long-term stress on the canola kernels at intermediate moisture contents analogous to that caused by canola bed during the storage should be simulated using the spring apparatus. The time and the magnitude of the stress required for the kernel to rapture should be evaluated. The contribution of the plastic component and the second viscous element of the Burgers component in the viscoelasto-plastic model should be considered in this case as a factor of primary importance.

4. The effect of temperature and temperature fluctuations on viscoelasto-plastic properties of the single canola kernels should be studied using the spring apparatus. The mechanical behaviour of the kernel should be expressed using viscoelasto-plastic model.

5. The effect of molding and microorganism growth on EMC - ERH relationship and viscoelasto-plastic properties of the canola should be studied in greater details.

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APPENDIX A

TABLE A1. EQUILIBRIUM MOISTURE CONTENT DATA FOR X-SAMPLE, 1989 CROP, PURCHASED FROM THE LOCAL SUPPLIER.

RELATIVE HUMIDITY %	ADSORPTION MC % db	DESORPTION MC % db
19	4.2	4.4
23	4.6	NA *
25	4.8	5.3
27	4.9	5.4
29	5.0	5.5
38	5.8	6.3
42	6.2	6.6
51	7.1	7.5
53	7.2	7.7
58	7.9	NA
60	8.3	8.8
63	9.2	9.5
67	9.8	10.2
69	10.9	11.1
72	11.1	NA
76	13.2	13.2
77	CONDENSATION	
79		
88		
90		

TABLE A2. EQUILIBRIUM MOISTURE CONTENT DATA FOR Y-SAMPLE, 1989 CROP, HARVESTED AT GLENLEA EXPERIMENTAL STATION.

RELATIVE HUMIDITY %	ADSORPTION MC % db	DESORPTION MC % db
16	4.6	4.7
23	5.9	6.3
37	6.9	7.2
49	7.8	8.2
51	8.0	NA
57	8.9	9.6
65	10.5	11.1
68	11.5	12.0
73	13.0	13.4
75	13.8	13.8
77	CONDENSATION	
79		
85		
90		

* - not available.

TABLE A3 EQUILIBRIUM MOISTURE CONTENT DATA FOR Z-SAMPLE, 1990 CROP, HARVESTED AT GLENLEA EXPERIMENTAL STATION.

RELATIVE HUMIDITY %	ADSORPTION MC % db	DESORPTION MC % db
36	5.4	5.9
52	6.9	7.4
53	7.1	7.5
58	7.7	8.1
62	8.5	8.9

TABLE A4 EQUILIBRIUM MOISTURE CONTENT DATA FOR X-SAMPLE AFTER BEING SUBJECTED TO THE REWETTING/DRYING CYCLES.

RELATIVE HUMIDITY %	ADSORPTION MC % db	DESORPTION MC % db
15	3.4	4.4
23	3.6	4.7
26	3.9	4.9
34	4.4	5.3
40	NA	5.7
42	5.3	5.9
49	5.8	6.6
55	6.9	7.6
59	7.6	8.8
64	8.8	9.5
68	9.6	10.3
70	10.5	11.1
75	11.8	12.1

TABLE A5 EQUILIBRIUM MOISTURE CONTENT DATA FOR X-SAMPLE AFTER BEING SUBJECTED TO THE 10 kGy IRRADIATION.

RELATIVE HUMIDITY %	ADSORPTION MC % db	DESORPTION MC % db
24	4.8	5.2
30	5.2	5.6
40	6.0	6.4
58	8.0	8.4
59	8.3	8.6
64	9.5	9.9
70	11.0	11.2
72	11.1	NA
75	12.8	12.9

TABLE A6 EQUILIBRIUM MOISTURE CONTENT DATA FOR X-SAMPLE AFTER BEING SUBJECTED TO THE LONG-TERM STRESS.

RELATIVE HUMIDITY %	ADSORPTION MC % db	DESORPTION MC % db
33	5.4	5.8
38	5.7	6.1
45	6.4	6.7
58	7.9	8.4
62	8.9	9.2

TABLE A7 CONSTANTS OF THE HENDERSON EQUATION SPECIFIC FOR X, Y, Z AND C, HARVESTED AT DIFFERENT LOCATION, YEARS AND WITH DIFFERENT PREATRETMENT HISTORY.

SAMPLE		K		N		SUM OF SQU
		MEAN	STD. DEV.	MEAN	STD. DEV.	OF RESIDUA
X	ADSORP.	0.025165	0.004993	1.647161	0.095678	0.019029
	DESORP.	0.018195	0.004234	1.760063	0.110332	0.013598
Y	ADSORP.	0.015639	0.005697	1.762825	0.161649	0.016681
	DESORP.	0.011513	0.003919	1.853065	0.146243	0.010369
Z	ADSORP.	0.023826	0.006331	1.750685	0.134231	0.000621
	DESORP.	0.014664	0.0047	1.937898	0.157637	0.000698
C	ADSORP.	0.048842	0.009672	1.39739	0.099224	0.015688
	DESORP.	0.025984	0.007128	1.627848	0.132309	0.024054

TABLE A8 CONTACT AREA DIAMETER, ELASTIC, AND TOTAL DEFORMATION VERSUS MOISTURE CONTENT FOR APPLIED LOAD OF 2N.

MOISTURE CONTENT % db	CONTACT AREA DIAMETER mm	ELASTIC DEFORMATION mm	TOTAL DEFORMATION mm	KERNAL DIAMETER mm	CONTACT AREA mm^2
4.3	0.48	0.030	0.041	1.721	0.1809
	0.27	0.025	0.041	1.750	0.0572
	0.18	0.027	0.035	1.726	0.0254
	0.3	0.028	0.039	1.721	0.0707
MEAN	0.308	0.0275	0.039	1.7295	0.0836
STD. DEV.	0.126	0.0021	0.0028	0.014	0.0676
6.7	0.45	0.038	0.066	1.723	0.159
	0.48	0.040	0.072	1.699	0.1809
	0.59	0.041	0.075	1.769	0.2733
	0.43	0.042	0.084	1.742	0.1451
MEAN	0.490	0.0403	0.0743	1.7333	0.1896
STD. DEV.	0.071	0.0017	0.0075	0.030	0.0577
8	0.57	0.045	0.100	1.754	0.255
	0.58	0.042	0.069	1.714	0.2641
	0.63	0.041	0.077	1.716	0.3116
	0.61	0.071	0.107	1.759	0.2921
MEAN	0.600	0.0498	0.0883	1.7358	0.2807
STD. DEV.	0.028	0.0143	0.0161	0.024	0.0260
9.7	0.71	0.082	0.131	1.711	0.3960
	0.66	0.051	0.108	1.771	0.3419
	0.78	0.069	0.131	1.714	0.4776
	0.63	0.044	0.086	1.702	0.3116
MEAN	0.695	0.0615	0.1140	1.7245	0.3818
STD. DEV.	0.066	0.0173	0.0216	0.031	0.0728
11.7	0.99	0.080	0.493	1.747	0.7694
	0.96	0.073	0.393	1.706	0.7235
	1.03	0.087	0.326	1.750	0.8328
	0.94	0.078	0.400	1.735	0.6936
MEAN	0.9800	0.0795	0.4030	1.7345	0.7548
STD. DEV.	0.039	0.0058	0.0686	0.020	0.0606
20.5	1.02	0.053	0.353	1.699	0.8167
	1.01	0.107	0.439	1.711	0.8008
	0.89	0.060	0.402	1.769	0.6218
	0.93	0.067	0.409	1.774	0.7085
MEAN	0.9625	0.0718	0.4008	1.7383	0.7370
STD. DEV.	0.0629	0.0242	0.0356	0.0388	0.0904

TABLE A9 CREEP/RECOVERY AND LOADING/UNLOADING EXPERIMENTAL DATA FOR X ADSORPTION (REFERENCE) SAMPLE AT 4.6 % db MOISTURE CONTENT.

		LOADING/UNLOADING TEST			CREEP AND RECOVERY TEST					
EXPERIM. NO.	DIAMETER mm	ELASTIC DEFORMATION mm	TOTAL DEFORMATION mm	RATIO OF ELASTICITY	CREEP DEFORMATION mm	LOADING DEFORMATION mm	UNLOADING DEFORMATION mm	RECOVERY DEFORMATION mm	RESIDUAL DEFORMATION mm	SLOPE AT TIME = 0
1	1.735	0.043	0.072	0.588	0.003	0.049	0.043	0.005	0.005	1.1E-04
2	1.663	0.019	0.039	0.489	0.003	0.022	0.021	0.003	0.003	8.0E-05
3	1.754	0.023	0.040	0.585	0.003	0.031	0.029	0.004	0.005	7.0E-05
4	1.687	0.030	0.062	0.486	0.003	0.031	0.034	0.002	0.004	4.0E-05
5	1.689	0.018	0.035	0.507	0.003	0.020	0.019	0.004	0.002	7.0E-05
6	1.793	0.032	0.065	0.501	0.004	0.041	0.039	0.006	0.003	7.0E-05
7	1.718	0.034	0.058	0.588	0.003	0.037	0.036	0.003	0.004	5.0E-05
8	1.711	0.042	0.058	0.715	0.006	0.043	0.037	0.003	0.001	1.0E-04
9	1.716	0.036	0.064	0.567	0.003	0.040	0.040	0.005	0.001	7.0E-05
10	1.701	0.022	0.062	0.359	0.002	0.028	0.030	0.002	0.002	5.0E-05
11	1.786	0.036	0.063	0.568	0.003	0.041	0.044	0.002	0.001	7.0E-05
12	1.771	0.024	0.050	0.483	0.004	0.028	0.028	0.004	0.001	1.3E-04
13	1.738	0.029	0.057	0.519	0.004	0.035	0.034	0.003	0.003	9.0E-05
AVERAGE	1.7280	0.0298	0.0558	0.5350	0.0034	0.0343	0.0334	0.0035	0.0027	7.6923E-05
STD. DEV	0.0397	0.0083	0.0114	0.0832	0.0010	0.0086	0.0078	0.0013	0.0015	2.5293E-05

TABLE A10 CREEP/RECOVERY AND LOADING/UNLOADING EXPERIMENTAL DATA FOR C ADSORPTION SAMPLE AT 3.6 % db MOISTURE CONTENT.

		LOADING/UNLOADING TEST			CREEP AND RECOVERY TEST				
EXPERIM NO.	DIAMETER mm	ELASTIC DEFORMATION mm	TOTAL DEFORMATION mm	RATIO OF ELASTICITY	CREEP DEFORMATION mm	LOADING DEFORMATION mm	UNLOADING DEFORMATION mm	RECOVERY DEFORMATION mm	RESIDUAL DEFORMATION mm
1	1.689	0.063	0.089	0.707	0.005	0.072	0.070	0.007	0.002
2	1.783	0.067	0.133	0.502	0.006	0.079	0.077	0.009	0.002
3	1.752	0.044	0.073	0.608	0.006	0.051	0.051	0.006	0.003
4	1.750	0.051	0.080	0.636	0.002	0.057	0.055	0.005	0.001
5	1.728	0.046	0.069	0.673	0.003	0.050	0.047	0.004	0.003
6	1.694	0.093	0.136	0.681	0.007	0.098	0.094	0.006	0.008
7	1.754	0.071	0.108	0.661	0.003	0.083	0.077	0.008	0.004
8	1.687	0.054	0.085	0.630	0.006	0.063	0.060	0.007	0.003
9	1.750	0.055	0.074	0.737	0.008	0.058	0.062	0.004	0.002
10	1.759	0.051	0.089	0.576	0.004	0.061	0.057	0.007	0.003
11	1.735	0.054	0.078	0.699	0.006	0.060	0.059	0.006	0.003
12	1.701	0.029	0.046	0.615	0.003	0.037	0.034	0.003	0.004
13	1.767	0.051	0.072	0.710	0.003	0.055	0.056	0.003	0.000
AVERAGE	1.7346	0.0561	0.0871	0.6488	0.0048	0.0634	0.0615	0.0058	0.0029
STD. DEV	0.032	0.0153	0.0253	0.0641	0.0019	0.0160	0.0152	0.0019	0.0019

TABLE A11 CREEP/RECOVERY AND LOADING/UNLOADING EXPERIMENTAL DATA FOR C DESORPTION SAMPLE AT 4.7 % db MOISTURE CONTENT

		LOADING/UNLOADING TEST			CREEP AND RECOVERY TEST				
EXPERIM. NO.	DIAMETER mm	ELASTIC DEFORMATION mm	TOTAL DEFORMATION mm	RATIO OF ELASTICITY	CREEP DEFORMATION mm	LOADING DEFORMATION mm	UNLOADING DEFORMATION mm	RECOVERY DEFORMATION mm	RESIDUAL DEFORMATION mm
1	1.764	0.033	0.068	0.481	0.004	0.041	0.040	0.007	0.003
2	1.767	0.034	0.072	0.471	0.003	0.027	0.026	0.003	0.005
3	1.788	0.046	0.071	0.637	0.005	0.057	0.053	0.007	0.004
4	1.663	0.052	0.091	0.567	0.005	0.057	0.055	0.008	0.008
5	1.774	0.056	0.107	0.524	0.008	0.066	0.063	0.010	0.005
6	1.704	0.038	0.079	0.484	0.005	0.046	0.040	0.006	0.006
7	1.663	0.035	0.073	0.477	0.004	0.077	0.068	0.009	0.007
8	1.759	0.030	0.051	0.588	0.004	0.036	0.039	0.003	0.001
9	1.675	0.041	0.075	0.548	0.006	0.052	0.054	0.008	0.003
10	1.711	0.059	0.092	0.641	0.006	0.066	0.062	0.006	0.007
11	1.706	0.023	0.057	0.393	0.004	0.031	0.031	0.005	0.002
12	1.663	0.025	0.054	0.465	0.003	0.030	0.027	0.007	0.003
13	1.675	0.024	0.047	0.509	0.004	0.025	0.027	0.005	0.001
AVERAGE	1.7163	0.0382	0.0721	0.5219	0.0047	0.0470	0.0450	0.0065	0.0042
STD. DEV	0.0478	0.0120	0.0175	0.0720	0.0014	0.0169	0.0149	0.0021	0.0023

TABLE A12 CREEP/RECOVERY AND LOADING/UNLOADING EXPERIMENTAL DATA FOR X ADSORPTION (REFERENCE) SAMPLE AT 6.2 % db MOISTURE CONTENT.

		LOADING/UNLOADING TEST			CREEP AND RECOVERY TEST					
EXPERIM. NO.	DIAMETER mm	ELASTIC DEFORMATION mm	TOTAL DEFORMATION mm	RATIO OF ELASTICITY	CREEP DEFORMATION mm	LOADING DEFORMATION mm	UNLOADING DEFORMATION mm	RECOVERY DEFORMATION mm	RESIDUAL DEFORMATION mm	SLOPE AT TIME = 0
1	1.675	0.046	0.087	0.532	0.007	0.054	0.052	0.007	0.006	0.00043
2	1.747	0.040	0.090	0.450	0.011	0.043	0.046	0.008	0.003	0.00040
3	1.769	0.042	0.088	0.478	0.009	0.052	0.048	0.007	0.011	0.00033
4	1.759	0.032	0.065	0.493	0.007	0.038	0.039	0.007	0.003	0.00027
5	1.711	0.039	0.091	0.432	0.007	0.043	0.042	0.008	0.005	0.00041
6	1.740	0.038	0.072	0.527	0.007	0.051	0.042	0.008	0.002	0.00032
7	1.699	0.037	0.089	0.418	0.011	0.047	0.047	0.009	0.008	0.00045
8	1.663	0.051	0.116	0.437	0.010	0.058	0.055	0.006	0.011	0.00049
9	1.783	0.037	0.079	0.468	0.009	0.043	0.041	0.008	0.006	0.00048
10	1.735	0.058	0.090	0.642	0.008	0.063	0.063	0.010	0.002	0.00045
11	1.740	0.028	0.045	0.623	0.007	0.032	0.032	0.001	0.002	0.00037
12	1.747	0.031	0.066	0.462	0.008	0.039	0.040	0.007	0.005	0.00031
AVERAGE	1.7308	0.0399	0.0815	0.4968	0.0084	0.0469	0.0456	0.0072	0.0053	3.92E-04
STD. DEV.	0.0368	0.0085	0.0178	0.0725	0.0016	0.0090	0.0082	0.0022	0.0033	7.29E-05

TABLE A13 CREEP/RECOVERY AND LOADING/UNLOADING EXPERIMENTAL DATA FOR C ADSORPTION SAMPLE AT 5.3 % db MOISTURE CONTENT.

		LOADING/UNLOADING TEST			CREEP AND RECOVERY TEST				
EXPERIM. NO.	DIAMETER mm	ELASTIC DEFORMATION mm	TOTAL DEFORMATION mm	RATIO OF ELASTICITY	CREEP DEFORMATION mm	LOADING DEFORMATION mm	UNLOADING DEFORMATION mm	RECOVERY DEFORMATION mm	RESIDUAL DEFORMATION mm
1	1.783	0.049	0.098	0.500	0.008	0.059	0.057	0.008	0.007
2	1.663	0.041	0.090	0.460	0.007	0.051	0.051	0.008	0.005
3	1.764	0.058	0.093	0.621	0.011	0.062	0.065	0.008	0.005
4	1.680	0.060	0.086	0.698	0.009	0.067	0.063	0.007	0.009
5	1.699	0.052	0.077	0.670	0.012	0.053	0.053	0.008	0.009
6	1.709	0.052	0.104	0.500	0.010	0.057	0.057	0.008	0.006
7	1.716	0.050	0.083	0.597	0.007	0.057	0.059	0.005	0.006
8	1.759	0.050	0.095	0.527	0.008	0.056	0.054	0.010	0.004
9	1.706	0.061	0.106	0.572	0.009	0.054	0.062	0.007	0.005
10	1.788	0.047	0.099	0.478	0.010	0.053	0.051	0.011	0.003
11	1.781	0.067	0.102	0.658	0.007	0.074	0.068	0.008	0.008
12	1.747	0.065	0.089	0.732	0.010	0.069	0.073	0.007	0.003
AVERAGE	1.733	0.0543	0.0935	0.5844	0.0090	0.0593	0.0594	0.0079	0.0058
STD. DEV.	0.043	0.0078	0.0089	0.0922	0.0017	0.0072	0.0070	0.0015	0.0021

TABLE A14. CREEP/RECOVERY AND LOADING/UNLOADING EXPERIMENTAL DATA FOR S SAMPLE AT 7.1 db MOISTURE CONTENT AN HOUR AFTER THE CONSTANT PRESSURE WAS REMOVED.

EXPERM. NO.	KERNEL DIAMETER mm	ELASTIC DEFORMATION mm	TOTAL DEFORMATION mm	RATIO OF ELASTICITY	CREEP DEFORMATION mm	LOADING DEFORMATION mm	UNLOADING DEFORMATION mm	RECOVERY DEFORMATION mm	RESIDUAL DEFORMATION mm
1	1.762	0.027	0.081	0.339	0.009	0.030	0.028	0.005	0.007
2	1.735	0.033	0.079	0.415	0.007	0.037	0.035	0.006	0.007
3	1.675	0.032	0.054	0.593	0.006	0.035	0.033	0.005	0.006
4	1.670	0.029	0.055	0.518	0.007	0.042	0.032	0.011	0.010
5	1.728	0.029	0.072	0.407	0.006	0.033	0.030	0.007	0.008
6	1.689	0.041	0.111	0.371	0.011	0.045	0.043	0.010	0.009
7	1.783	0.042	0.106	0.396	0.007	0.044	0.047	0.007	0.008
8	1.706	0.041	0.079	0.525	0.007	0.045	0.031	0.009	0.013
9	1.786	0.032	0.058	0.552	0.008	0.033	0.033	0.007	0.008
AVERAGE	1.7260	0.0340	0.0772	0.4573	0.0076	0.0382	0.0347	0.0074	0.0084
STD. DEV.	0.0444	0.0058	0.0207	0.0903	0.0016	0.0058	0.0063	0.0021	0.0021

TABLE A15. CREEP/RECOVERY AND LOADING/UNLOADING EXPERIMENTAL DATA FOR S SAMPLE AT 7.1 db MOISTURE CONTENT 7 DAYS AFTER THE CONSTANT PRESSURE WAS REMOVED.

EXPERM. NO.	KERNAL DIAMETER mm	ELASTIC DEFORMATION mm	TOTAL DEFORMATION mm	RATIO OF ELASTICITY	CREEP DEFORMATION mm	LOADING DEFORMATION mm	UNLOADING DEFORMATION mm	RECOVERY DEFORMATION mm	RESIDUAL DEFORMATION mm
1	1.783	0.037	0.093	0.399	0.007	0.042	0.040	0.007	0.006
2	1.716	0.046	0.097	0.475	0.008	0.053	0.053	0.005	0.007
3	1.783	0.029	0.075	0.392	0.008	0.032	0.034	0.004	0.005
4	1.735	0.038	0.088	0.429	0.006	0.043	0.043	0.004	0.006
5	1.716	0.045	0.064	0.704	0.005	0.045	0.047	0.003	0.005
6	1.716	0.023	0.036	0.642	0.005	0.030	0.027	0.006	0.005
7	1.706	0.035	0.059	0.592	0.008	0.039	0.038	0.006	0.006
8	1.735	0.027	0.052	0.515	0.003	0.029	0.027	0.007	0.005
9	1.747	0.027	0.052	0.529	0.005	0.029	0.027	0.007	0.006
AVERAGE	1.7374	0.0341	0.0684	0.5197	0.0061	0.0380	0.0373	0.0054	0.0057
STD. DEV.	0.0288	0.0082	0.0211	0.1092	0.0018	0.0085	0.0094	0.0015	0.0007

TABLE A16 CREEP/RECOVERY AND LOADINNG/UNLOADING EXPERIMENTAL DATA FOR X ADSORPTION (REFERENCE) SAMPLE AT 7.9 % db MOISTURE CONTENT.

EXPERM. NO.	KERNEL DIAMETER mm	ELASTIC DEFORMATION mm	TOTAL DEFORMATION mm	RATIO OF ELASTICITY	CREEP DEFORMATION mm	LOADING DEFORMATION mm	UNLOADING DEFORMATION mm	RECOVERY DEFORMATION mm	RESIDUAL DEFORMATION mm	SLOPE AT TIME=1s
1	1.689	0.044	0.087	0.505	0.010	0.045	0.044	0.010	0.005	0.0011
2	1.779	0.033	0.072	0.454	0.009	0.041	0.035	0.009	0.007	0.0012
3	1.738	0.032	0.073	0.436	0.011	0.041	0.038	0.012	0.005	0.0014
4	1.755	0.044	0.101	0.437	0.010	0.050	0.051	0.007	0.003	0.0005
5	1.714	0.042	0.088	0.474	0.010	0.048	0.045	0.010	0.005	0.0010
6	1.706	0.057	0.105	0.548	0.011	0.059	0.058	0.009	0.005	0.0011
AVERAGE	1.7300	0.0420	0.0877	0.4757	0.0102	0.0473	0.0452	0.0095	0.0050	0.0011
STD. DEV.	0.0332	0.0091	0.0137	0.0439	0.0008	0.0068	0.0084	0.0016	0.0013	0.0003

TABLE A17 CREEP/RECOVERY AND LOADINNG/UNLOADING EXPERIMENTAL DATA FOR S ADSORPTION SAMPLE AT 8.0 % db MOISTURE CONTENT.

EXPERM. NO.	KERNEL DIAMETER mm	ELASTIC DEFORMATION mm	TOTAL DEFORMATION mm	RATIO OF ELASTICITY	CREEP DEFORMATION mm	LOADING DEFORMATION mm	UNLOADING DEFORMATION mm	RECOVERY DEFORMATION mm	RESIDUAL DEFORMATION mm
1	1.673	0.051	0.101	0.503	0.009	0.056	0.054	0.011	0.004
2	1.694	0.063	0.106	0.598	0.009	0.069	0.067	0.013	0.001
3	1.791	0.051	0.097	0.531	0.007	0.058	0.052	0.014	0.003
4	1.735	0.044	0.102	0.431	0.011	0.049	0.045	0.011	0.005
5	1.718	0.052	0.101	0.509	0.009	0.058	0.053	0.012	0.002
6	1.687	0.049	0.102	0.477	0.009	0.056	0.050	0.014	0.004
AVERAGE	1.7163	0.0517	0.1015	0.5082	0.0090	0.0577	0.0535	0.0125	0.0032
STD. DEV.	0.0428	0.0063	0.0029	0.0558	0.0013	0.0065	0.0073	0.0014	0.0015

TABLE A18. CREEP/RECOVERY AND LOADING/UNLOADING EXPERIMENTAL DATA FOR X ADSORPTION (REFERENCE) SAMPLE AT 11.1 % db MOISTURE CONTENT.

		LOADING/UNLOADING TEST			CREEP AND RECOVERY TEST					
EXPERIM. NO.	DIAMETER mm	ELASTIC DEFORMATION mm	TOTAL DEFORMATION mm	RATIO OF ELASTICITY	CREEP DEFORMATION mm	LOADING DEFORMATION mm	UNLOADING DEFORMATION mm	RECOVERY DEFORMATION mm	RESIDUAL DEFORMATION mm	SLOPE AT TIME = 1s
1	1.694	0.066	0.181	0.365	0.016	0.080	0.068	0.019	0.015	0.0029
2	1.771	0.075	0.192	0.389	0.018	0.089	0.075	0.019	0.021	0.0041
3	1.711	0.063	0.158	0.398	0.017	0.080	0.075	0.020	0.006	0.0016
4	1.796	0.077	0.209	0.367	0.017	0.090	0.074	0.022	0.020	0.0019
5	1.783	0.077	0.192	0.400	0.019	0.088	0.074	0.021	0.022	0.0017
6	1.663	0.040	0.154	0.260	0.013	0.052	0.049	0.017	0.006	0.0013
AVERAGE	1.7363	0.0663	0.1810	0.3632	0.0167	0.0798	0.0692	0.0197	0.0150	0.0023
STD. DEV.	0.0543	0.0142	0.0214	0.0527	0.0021	0.0143	0.0102	0.0018	0.0074	0.0011

TABLE A19. CREEP/RECOVERY AND LOADING/UNLOADING EXPERIMENTAL DATA FOR I ADSORPTION SAMPLE AT 11.1 % db MOISTURE CONTENT.

		LOADING/UNLOADING TEST			CREEP AND RECOVERY TEST				
EXPERIM. NO.	DIAMETER mm	ELASTIC DEFORMATION mm	TOTAL DEFORMATION mm	RATIO OF ELASTICITY	CREEP DEFORMATION mm	LOADING DEFORMATION mm	UNLOADING DEFORMATION mm	RECOVERY DEFORMATION mm	RESIDUAL DEFORMATION mm
1	1.788	0.067	0.172	0.391	0.012	0.077	0.072	0.015	0.006
2	1.687	0.073	0.188	0.388	0.018	0.087	0.074	0.020	0.017
3	1.699	0.063	0.183	0.347	0.017	0.080	0.069	0.022	0.015
4	1.747	0.040	0.190	0.212	0.017	0.046	0.041	0.017	0.012
5	1.759	0.067	0.188	0.358	0.016	0.085	0.073	0.018	0.014
6	1.723	0.069	0.165	0.419	0.014	0.083	0.073	0.017	0.015
AVERAGE	1.7338	0.0632	0.1810	0.3525	0.0157	0.0763	0.0670	0.0182	0.0132
STD. DEV.	0.0381	0.0118	0.0102	0.0734	0.0023	0.0153	0.0129	0.0025	0.0039

TABLE A20. CREEP/RECOVERY AND LOADING/UNLOADING EXPERIMENTAL DATA FOR Z ADSORPTION SAMPLE AT 8.5 % db MOISTURE CONTENT.

		LOADING/UNLOADING TEST			CREEP AND RECOVERY TEST				
EXPERIM. NO.	DIAMETER mm	ELASTIC DEFORMATION mm	TOTAL DEFORMATION mm	RATIO OF ELASTICITY	CREEP DEFORMATION mm	LOADING DEFORMATION mm	UNLOADING DEFORMATION mm	RECOVERY DEFORMATION mm	RESIDUAL DEFORMATION mm
1	1.704	0.050	0.126	0.397	0.011	0.057	0.051	0.016	0.007
2	1.730	0.053	0.098	0.537	0.011	0.054	0.055	0.012	0.002
3	1.723	0.034	0.113	0.300	0.011	0.041	0.034	0.014	0.007
4	1.776	0.044	0.119	0.371	0.009	0.049	0.048	0.009	0.004
5	1.711	0.038	0.121	0.313	0.011	0.046	0.045	0.011	0.005
6	1.735	0.036	0.109	0.331	0.011	0.042	0.036	0.011	0.009
7	1.762	0.031	0.084	0.373	0.011	0.035	0.031	0.013	0.005
AVERAGE	1.7344	0.0409	0.1100	0.3746	0.0107	0.0463	0.0429	0.0123	0.0056
STD. DEV.	0.0262	0.0083	0.0147	0.0797	0.0008	0.0077	0.0092	0.0023	0.0023

TABLE A21. CREEP/RECOVERY AND LOADING/UNLOADING EXPERIMENTAL DATA FOR X ADSORPTION SAMPLE AT 9.8 % db MOISTURE CONTENT.

		LOADING/UNLOADING TEST			CREEP AND RECOVERY TEST					
EXPERIM. NO.	KERNEL DIAMETER mm	ELASTIC DEFORMATION mm	TOTAL DEFORMATION mm	RATIO OF ELASTICITY	CREEP DEFORMATION mm	LOADING DEFORMATION mm	UNLOADING DEFORMATION mm	RECOVERY DEFORMATION mm	RESIDUAL DEFORMATION mm	SLOPE AT T = 1s
1	1.699	0.059	0.150	0.396	0.015	0.075	0.063	0.021	0.009	0.0016
2	1.769	0.057	0.151	0.379	0.016	0.069	0.063	0.016	0.011	0.0011
3	1.740	0.065	0.144	0.454	0.015	0.077	0.065	0.021	0.011	0.0014
4	1.779	0.065	0.133	0.490	0.013	0.073	0.063	0.021	0.007	0.0017
5	1.689	0.064	0.134	0.475	0.014	0.073	0.064	0.021	0.007	0.0016
6	1.771	0.057	0.148	0.383	0.013	0.067	0.059	0.019	0.007	0.0015
7	1.779	0.055	0.133	0.410	0.014	0.061	0.045	0.018	0.017	0.0015
8	1.697	0.043	0.067	0.640	0.007	0.051	0.045	0.011	0.006	0.0018
9	1.723	0.069	0.172	0.403	0.015	0.081	0.069	0.019	0.016	0.0009
10	1.752	0.044	0.159	0.277	0.013	0.055	0.046	0.018	0.007	0.0018
11	1.755	0.055	0.116	0.477	0.012	0.063	0.052	0.019	0.008	0.0015
12	1.735	0.051	0.125	0.406	0.013	0.057	0.053	0.016	0.005	0.0014
13	1.740	0.051	0.118	0.429	0.013	0.068	0.058	0.017	0.011	0.0015
14	1.742	0.055	0.135	0.406	0.011	0.067	0.060	0.013	0.012	0.0022
15	1.735	0.049	0.133	0.367	0.013	0.072	0.057	0.019	0.013	0.0012
16	1.738	0.053	0.133	0.400	0.015	0.061	0.052	0.018	0.011	0.0021
17	1.720	0.045	0.112	0.399	0.011	0.049	0.043	0.013	0.007	0.0013
18	1.750	0.055	0.130	0.422	0.013	0.063	0.055	0.020	0.008	0.0015
19	1.726	0.050	0.118	0.424	0.010	0.059	0.049	0.019	0.003	0.0016
20	1.711	0.043	0.101	0.431	0.013	0.047	0.043	0.012	0.009	0.0011
AVERAGE	1.7375	0.0543	0.1306	0.4234	0.0130	0.0644	0.0552	0.0176	0.0093	0.0015
STD. DEV.	0.0263	0.0076	0.0226	0.0685	0.0021	0.0095	0.0081	0.0031	0.0035	0.0003

TABLE A22. CREEP/RECOVERY AND LOADING/UNLOADING EXPERIMENTAL DATA FOR X DESORPTION SAMPLE AT 10.2 % db MOISTURE CONTENT

		LOADING/UNLOADING TEST			CREEP AND RECOVERY TEST				
EXPERIM. NO.	KERNEL DIAMETER mm	ELASTIC DEFORMATION mm	TOTAL DEFORMATION mm	RATIO OF ELASTICITY	CREEP DEFORMATION mm	LOADING DEFORMATION mm	UNLOADING DEFORMATION mm	RECOVERY DEFORMATION mm	RESIDUAL DEFORMATION mm
1	1.767	0.050	0.196	0.255	0.015	0.049	0.048	0.023	0.010
2	1.740	0.059	0.152	0.386	0.012	0.069	0.059	0.019	0.010
3	1.699	0.056	0.191	0.293	0.013	0.065	0.053	0.015	0.013
4	1.769	0.053	0.146	0.364	0.013	0.061	0.055	0.014	0.009
5	1.740	0.052	0.186	0.279	0.013	0.063	0.054	0.021	0.007
6	1.771	0.049	0.203	0.243	0.013	0.060	0.049	0.015	0.018
7	1.769	0.049	0.189	0.258	0.013	0.059	0.045	0.014	0.013
8	1.745	0.050	0.202	0.247	0.014	0.057	0.042	0.021	0.014
9	1.723	0.051	0.201	0.252	0.013	0.057	0.048	0.016	0.013
10	1.747	0.057	0.189	0.299	0.013	0.067	0.055	0.016	0.014
11	1.723	0.061	0.240	0.253	0.015	0.063	0.058	0.015	0.010
12	1.733	0.055	0.204	0.271	0.015	0.064	0.051	0.019	0.016
13	1.716	0.063	0.167	0.378	0.013	0.067	0.062	0.013	0.009
14	1.711	0.051	0.142	0.362	0.009	0.062	0.053	0.018	0.009
15	1.742	0.051	0.151	0.339	0.010	0.058	0.051	0.015	0.007
16	1.735	0.057	0.201	0.285	0.013	0.062	0.057	0.010	0.013
17	1.699	0.053	0.151	0.348	0.011	0.062	0.051	0.020	0.011
18	1.742	0.053	0.206	0.259	0.011	0.067	0.053	0.018	0.015
19	1.735	0.057	0.161	0.355	0.012	0.063	0.052	0.016	0.013
20	1.769	0.055	0.200	0.273	0.016	0.066	0.056	0.019	0.013
AVERAGE	1.7388	0.0541	0.1839	0.3000	0.0129	0.0621	0.0526	0.0169	0.0119
STD. DEV.	0.0226	0.0040	0.0262	0.0495	0.0017	0.0046	0.0048	0.0032	0.0029

TABLE A23. T-TEST RESULTS PERFORMED TO EVALUATE THE EFFECT OF PRETREATMENTS AND EQUILIBRIUM MOISTURE CONTENT HYSTERESIS ON MECHANICAL PROPERTIES OF CANOLA

T-TEST #	SAMPLE	MC % db	RH %	CRITERION	DF	p-value	| T |	t	FISHER's LSD	CONCLUSSION AT 95 % CONFIDENCE LEVEL
1	C-ADSORP. X-ADSORP.	5.26 6.24	42.3	INSTANTANEOUS LOADING DEFORMATION	22	0.012	3.708	2.074	0.0069	Mean values of loading deformation are not equal for these samples
2	C-ADSORP. X-ADSORP.	3.63 4.6	22.7	INSTANTANEOUS LOADING DEFORMATION	24	0.0001	5.778	2.064	0.0104	Mean values of loading deformation are not equal for these samples
3	C-ADSORP. C-DESORP.	3.63 4.71	22.7	INSTANTANEOUS LOADING DEFORMATION	24	0.0182	2.534	2.064	0.0134	Mean values of loading deformation are not equal for these samples
4	X-ADSORP. X-DESORP.	9.84 10.2	67	INSTANTANEOUS LOADING DEFORMATION	38	0.318	1.013	2.025	-	Mean values of loading deformation are equal for these samples
5	X-ADSORP. X-DESORP.	9.84 10.2	67	RATIO OF ELASTICITY	38	0.0001	6.529	2.025	0.0383	Mean values of ratios of elasticity are not equal for these samples
6	S-ADSORP. X-ADSORP.	7.9 7.9	57.6	INSTANTANEOUS LOADING DEFORMATION	10	0.191	2.791	2.228	0.0084	Mean values of loading deformation are not equal for these samples
7	I-ADSORP. X-ADSORP.	11.1 11.1	71.5	INSTANTANEOUS LOADING DEFORMATION	10	0.683	0.42	2.228	-	Mean values of loading deformation are equal for these samples
8	S-0 DAYS S-7 DAYS	7.1 7.1	AMB.	INSTANTANEOUS LOADING DEFORMATION	16	0.937	0.08	2.21	-	Mean values of loading deformation are equal for these samples

TABLE A24. EXPERIMENTAL DATA FOR 2 CONSECUTIVE CREEP AND RECOVERY TESTS NOT PRECEDED WITH LOADING/UNLOADING CYCLES, PERFORMED ON THE CANOLA KERNEL AT 20.5 % db MOISTURE CONTENT.

EXPERIM. NO.	CREEP STRAIN mm/mm	RECOVERY STRAIN mm/mm	TANGENT AT t = 61s	TANGENT AT t = 62s	LOADING STRAIN mm/mm	UNLOADING STRAIN mm/mm	DISSIPATED STRAIN mm/mm	STRAIN AT t = 61s mm/mm	STRAIN AT t = 122s mm/mm
1	0.053	0.018	6.6E-05	-0.00325	0.126	0.043	0.083	0.187	0.125
2	0.016	0.017	5.5E-05	-0.00241	0.047	0.043	0.005	0.193	0.136

APPENDIX B

1. BET ISOTHERM EQUATION (Braunauer et al., 1939).

$$\frac{a}{(1-a)\,m} = \frac{1}{m_0 C} + \frac{C-1}{m_0 C}$$

Where:

a = water activity,

m = moisture content (dry basis, decimal) at water activity a and temperature T, kg/kg

m_0 = monolayer moisture content, kg/kg

C = empirical constant.

2. GAB ISOTHERM EQUATION (Van den Berg, 1985b).

$$\frac{m}{m_0} = \frac{CKa}{(1-Ka)\,(1-Ka+CKa)}$$

Where:

C, K, = empirical constants.

m_0 = monolayer moisture content, kg/kg.

3. HAILWOOD AND HORROBIN's EQUATION (Pohorecki and Wronski, 1977).

$$\frac{a}{m} = B + Ca + Da^2$$

Where:

B, C, D = empirical constants.

4. SMITH ISOTHERM EQUATION (Labuza, 1984).

$$\log(1-a) = Bm + C$$

Where:

B, C = empirical constants.

5. LANGMUIR's EQUATION (Pohorecki and Wronski, 1977).

$$\frac{m}{A} = \frac{Ka}{\frac{1}{P_s} + Ka}$$

Where:

A, K = empirical constants,

p_s = saturated vapour pressure, Pa.

6. SIP's EQUATION (Pohorecki and Wronski, 1977).

$$\frac{m}{A} = \left[\frac{Ka}{1+(K-1)a}\right]^B$$

Where:

A, B, K = empirical constants.

7. MODIFIED HENDERSON's EQUATION (ASAE, 1987).

$$m=\left[-\log\frac{(1-a)}{K(T+C)}\right]^{\frac{1}{n}}$$

Where:

K, C, n = empirical constants.

8. CHUNG AND PFOST's EQUATION (1967).

$$m=E-F\log\left[-(T-C)\log(a)\right]$$

Where:

E, F, C = empirical constants.

9. CAURIE's EQUATION (1981)

$$\ln\frac{1}{m}=-\ln(Cm_0)+\frac{2C}{m_0\ln\frac{1-a}{a}}$$

Where:

C = empirical constant

m_0 = monolayer moisture, kg/kg.

10. MODIFIED - HALSEY EQUATION (Iglesias and Chirife 1976)

$$a=\exp\left[-\exp(A+BT)\,m^{-c}\right]$$

Where:

A, B, c = empirical constants

T = temperature.

11. HENDERSON's EQUATION (1952)

$$1 - a = e^{-km^n}$$

Where:

k,n = empirical constants