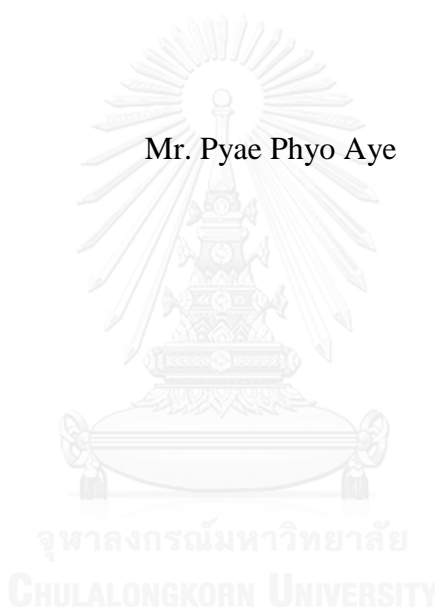


COMPARING EXPRESSION BEHAVIOR OF FROZEN/THAWED FRUITS
CONTAINING SOLUBLE AND INSOLUBLE FIBERS

Mr. Pyae Phyo Aye



บทคัดย่อและแฟ้มข้อมูลฉบับเต็มของวิทยานิพนธ์ตั้งแต่ปีการศึกษา 2554 ที่ให้บริการในคลังปัญญาจุฬาฯ (CUIR)
เป็นแฟ้มข้อมูลของนิสิตเจ้าของวิทยานิพนธ์ ที่ส่งผ่านทางบัณฑิตวิทยาลัย

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A Thesis Submitted in Partial Fulfillment of the Requirements
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Department of Food Technology
Faculty of Science
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การเปรียบเทียบพฤติกรรมการกอดอัดไล่น้ำของผลไม้ที่มีเส้นใยที่ละลายน้ำได้
และที่ไม่ละลายน้ำซึ่งผ่านการแช่เยือกแข็งและคลายสภาพเยือกแข็ง



วิทยานิพนธ์นี้เป็นส่วนหนึ่งของการศึกษาตามหลักสูตรปริญญาวิทยาศาสตรมหาบัณฑิต
สาขาวิชาวิทยาศาสตร์และเทคโนโลยีทางอาหาร ภาควิชาเทคโนโลยีทางอาหาร
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pp.

The effect of freezing and thawing pre-treatments and storage time on expression of apple and pineapple were studied using compression permeability cell. In this study, expression experiments of pineapple (representative of fruit rich in insoluble fiber) and apple (representative of fruit rich in soluble fiber) were done at three levels of pre-treatments, which were control (fresh fruit), slow freezing, and cryogenic freezing. For the slow freezing pre-treatments prepared samples were frozen at -18°C in the freezer for 24 hours, while cryogenic freezing pre-treatment was achieved by freezing with liquid nitrogen until the temperature of fruit decreased to -90°C . Both of freezing pre-treatment methods gave more expression efficiency than those obtained from control pre-treatment method. However, slow freezing pre-treated samples gave higher de-liquoring efficiency than those obtained from cryogenic freezing pre-treatment samples in the initial period of expression. Slow freezing pre-treatment method was chosen as an experiment condition for determination of storage effect. It was found that Terzaghi-Voight combined model fitted better than Terzaghi's model but there were slight differences between 3, 6, and 9 weeks. The fitting of normalized cake thickness (ratio of the cake thickness at any expression time to initial cake thickness) versus time was also done. It was clearly found out that the empirical equation can evaluate the changes of cake thickness during expression time ($r^2 \geq 0.99$). Soluble solid, pH and sugar acid ratio were significantly higher in juices obtained from frozen samples than those obtained from control samples for both fruits, except reversible observed was found out in soluble solid of pineapple ($p \leq 0.05$). There is no significant difference for Vitamin C in juices obtained from neither frozen samples nor control sample. ($p > 0.05$). With longer storage time, sugar acid ratio was clearly found out higher in both apple and pineapple ($p \leq 0.05$).

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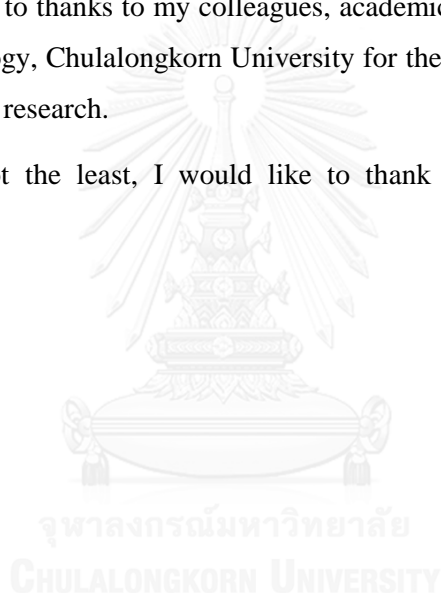
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CHAPTER I

INTRODUCTION

Expression is a separation process extensively used in industry of extracting the soft vegetables and fruits: fruit juice and vegetables juice, pressing of oilseeds and dewatering of fibrous materials (sugar beets). The benefits of expression method over the other methods are higher dewatering rate, higher yield, better quality of filtrate than thermal dewatering method and less energy requirement than thermal dewatering method. The objectives of using expression method are to achieve the high yield and high dry matter content. However, food material consists of various components as complex materials, still not be easy to fit to the simple model such as Terzaghi model or Terzaghi-Voight combined model. There was one attempt tried to express the expression dewatering mechanism of tofu and found that it needed at least three or more series of combination of Terzaghi and Voight elements to fit the dewatering phenomena of tofu material that was not easy to determine the parameters in the model. Many pre-treatment methods such as slicing, grinding, blanching, pulsed electric field and adding enzymes were applied prior to expression in order to gain dewatering efficiency. Freezing and thawing pre-treatment method is one of the alternative ways to reduce filter cake resistance as reported in environmental field for activated sludge dewatering (Lee & Hsu, 1994). As the effects of freezing can rupture cell wall due to moisture migration and growth of ice crystal and slowing down the chemical, biochemical reaction and limiting the microbial growth and it significantly increased the juice yield, so that it should be possible to apply to food industries especially in the fruit juice manufacturing. However, there is no publication on the

study of expression behavior of frozen/thawed fruits and vegetables. Thus, this research is aimed to investigate the expression behavior of frozen/thawed fruits containing soluble and insoluble fiber and investigate the effects of frozen storage time on the yield and properties of obtained juice.



CHAPTER II

LITERATURE REVIEW

2.1 Pineapple fruit description and composition

Pineapple (Hassan, Othman et al.) is believed to be originated from South America and the third most important tropical fruit in the international trade after bananas and citrus. Pineapple is produced for both fresh consumption and processing. Pineapple for fresh consumption is marketed in whole or minimally processed form with a short marketable period (Hassan, Othman et al. 2011). Fresh pineapple is a good source of carbohydrate, fibre, minerals and some vitamins including vitamin A, B1 (thiamine), B2 (riboflavin), B3 (niacin), B6 (pyridoxine), B9 (folate) and C (ascorbic acid). According to the data of the USDA (2015), the nutritional and mineral contents are shown as Table 2.1, however these values are influenced by several factors including varieties, soil, climatic condition, maturity stage, and handling.

Table 2. 1 Composition of nutrient data of pineapple according to the USDA National Nutrient Database for Standard Reference (2015). (Library 2015)

Pineapple		
Nutrient	Unit	1 Value Per 100g
Proximate		
Water	g	86
Energy	kcal	50
Protein	g	0.54
Total lipid (fat)	g	0.12
Carbohydrate, by difference	g	13.12
Fiber, total dietary	g	1.4
Sugars, total	g	9.85
Minerals		
Calcium, Ca	mg	13
Iron, Fe	mg	0.29

Magnesium, Mg	mg	12
Phosphorus, P	mg	8
Potassium, K	mg	109
Sodium, Na	mg	1
Zinc, Zn	mg	0.12
Vitamins		
Vitamin C, total ascorbic acid	mg	47.8
Thiamin	mg	0.079
Riboflavin	mg	0.032
Niacin	mg	0.5
Vitamin B-6	mg	0.112
Folate, DFE	µg	18
Vitamin B-12	µg	0
Vitamin A, RAE	µg	3
Vitamin A, IU	IU	58
Vitamin E (alpha-tocopherol)	mg	0.02
Vitamin D (D2+D3)	µg	0
Vitamin D	IU	0
Vitamin K (phylloquinone)	µg	0.7
Lipids		
Fatty acids, total saturated	g	0.009
Fatty acids, total monounsaturated	g	0.013
Fatty acids, total polyunsaturated	g	0.04
Cholesterol	mg	0
Other		
Caffeine	mg	0

Source: USDA National Nutrient Database for Standard Reference Release 27.

2.2 Apple fruit description and composition

Delicious and crunchy, apple fruit is one of the most popular and favorite fruits among the health conscious, fitness lovers who firmly believe in the concept of “health is wealth.” Today, apple (*Malus domestica*) is being cultivated in many parts of the world including the US as an important commercial crop. It is known to be good source for soluble and insoluble fiber and vitamin C and B complex and others

nutritional components. The composition of nutrient data of apple was shown in table

2.2.

Table 2. 2 Composition of nutrient data of apple according to the USDA National Nutrient Database for Standard Reference (2015). (Library 2015)

Apple		
Nutrient	Unit	1 Value Per 100g
Proximate		
Water	g	85.56
Energy	kcal	52
Protein	g	0.26
Total lipid (fat)	g	0.17
Carbohydrate, by difference	g	13.81
Fiber, total dietary	g	2.4
Sugars, total	g	10.39
Minerals		
Calcium, Ca	mg	6
Iron, Fe	mg	0.12
Magnesium, Mg	mg	5
Phosphorus, P	mg	11
Potassium, K	mg	107
Sodium, Na	mg	1
Zinc, Zn	mg	0.04
Vitamins		
Vitamin C, total ascorbic acid	mg	4.6
Thiamin	mg	0.017
Riboflavin	mg	0.026
Niacin	mg	0.091
Vitamin B-6	mg	0.041
Folate, DFE	µg	3
Vitamin B-12	µg	0
Vitamin A, RAE	µg	3
Vitamin A, IU	IU	54
Vitamin E (alpha-tocopherol)	mg	0.18
Vitamin D (D2+D3)	µg	0
Vitamin D	IU	0
Vitamin K (phylloquinone)	µg	2.2
Lipids		
Fatty acids, total saturated	g	0.028
Fatty acids, total	g	0.007

monounsaturated		
Fatty acids, total polyunsaturated	g	0.051
Cholesterol	mg	0
Other		
Caffeine	mg	0

Source: USDA National Nutrient Database for Standard Reference Release 27.

2.3 Expression

Expression is a separation process whereby liquid is expelled out from a wet material by applying pressure (Fellow 2000). It is therefore often called pressing or squeezing. The liquid is separated out from a solid-liquid mixture by mechanical pressure through the porous bed. The porous bed is often called a 'cake' in both cases. Mechanical expression is extensively used in industry of extracting the soft vegetables and plant agro food materials: fruit juice and vegetables juice, pressing of oilseeds and dewatering of fibrous materials (sugar beets). The by-products of mechanical expression are solid residues which are either processed into animal feeds or use in agricultural land. The expression process does not include solvent extraction or water leaching of solutes which are mass transfer processes.

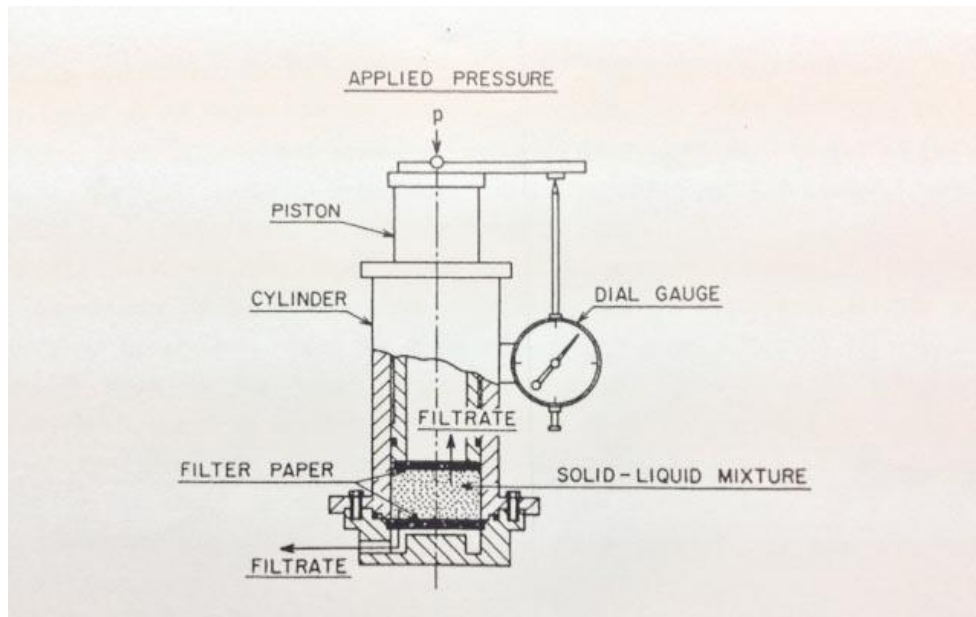


Figure 2. 1 Compression permeability cell
Source:(Shirato 1986)

In order to evaluate the expression mechanism, the expression experiments were done by using this compressing permeability cell as shown in Figure 2.1. It gives the data of applied pressure, filtrate volume, expression time, and thickness of the cake. The solid-liquid mixture to be expressed put into the expression chamber and filtrate is filter through the filter medium by applying the hydraulic pressure to the piston. The thickness of the cake was measured by dial gauge.

The sensitivity of flow resistance and liquid content to contact pressure is denoted compressibility. It is the key factor in determining the basic behavior of the deliquoring phenomenon. High compressibility means that flow resistance and liquid content are very sensitive to changes in contact pressure.

2.4 Factors affecting expression

2.4.1 Compressibility of the cake

Compressibility of the cake is the response of the cake to applied pressure which depends on its mechanical properties such as rigidity, pliability, hardness. The relationship between the applied pressure and reduction in volume can be modeled as Equation (2.1).

$$\log \frac{v - v_{\infty}}{v_0 - v_{\infty}} = -k_k P \quad (2.1)$$

Where:

V = volume of compressed cake in equilibrium with pressure P

V_0 = volume of cake before compression

V_{∞} = volume of cake compressed by an infinitely high pressure

P = Pressure, Pa

k_k = compressibility constant, Pa^{-1}

Equation (2.1) may be used to evaluate approximately the maximum yield of juice that can be expressed by applying pressure P . Experimentation is needed to validate the model and to determine the compressibility constant (Berk 2013).

2.4.2 Viscosity of the Liquid

Both the rate of liquid release and final yield are negatively affected by high liquid viscosity. For example, the juice of white grapes expressed by applying moderate pressure is considered as of higher quality for the production of white wine and expression of “first press” is used to indicate premium quality. Using multistage expression is used to obtain products with different quality grades (Berk 2013).

2.4.3 Pre-treatment to the cake before expression

During the expression of food materials, pretreatments of food materials before expression also play an important role. The liquid subjected to expression (cellular juice, oil) stays initially inside the cellular plant tissue. This intracellular liquid can be expressed just after the cell rupture. To induce the cell rupture and enhance the extraction yield, solid-liquid expression is generally combined with many kinds of pre-treatments. There are varieties of mechanical (grinding, cutting), thermal (heating, freezing-thawing) and chemical and enzymatic treatments and other treatment which is pulse electric field: PEF are usually given to the materials before expression. Combinations of above pre-treatments are also used to in expression of materials to get more yield and shorten the expression time. But the cell breaking phenomena in a fresh tissue depends not only on the applied pressure but also on the structural tissue characteristics (cell size and shape, thickness of the cell wall and the middle lamella, osmotic pressure inside a cell, etc. (Zhuw 2003)).

The most common mechanical pre-treatment is comminution or crushing to fruits, vegetables, olives, sugar cane etc. and grinding is applied to oilseeds. Comminution destroy the cellular tissue, cut fibers, releases liquid and increases the compressibility of the solid. In the other way, extensive comminution may increase the amount of fines that can block the passage of liquid flow through the cake and impair the rate of juice releases. Moreover, fines are not desirable if the product is not to be cloudy. Extensive comminution can also cause the extraction of undesirable, less soluble substances such as bitter compounds into the liquid. It is therefore essential to optimize the range of comminution depending on the characteristics of the food

materials. The fruit pieces and particles should be of the proper size (not too small and not too large).

Thermal pre-treatment may be applied for a variety reasons. The objectives of thermal pre-treatment are: to plasticize the mass, to denature the proteins and free the oil from emulsions, reduce the viscosity of the oil and to remove the moisture if necessary.

A short pulsed electric treatment (microsecond or millisecond duration) is sufficient to induce a cell membrane electroporation and enhance the yield (Parniakov, Lebovka et al. 2015). Pulsed electric field produces a current through the biological tissue by using pulse generator and may result in damage of membrane. The food material is placed in the pulsed electric field treatment cell between two electrodes. As a result, a number of different phenomena, such as intracellular liquid release, diffusion of solutes, membrane resealing process, develop inside the cellular structure after their treatment. Specific effects like electro-osmotic flow and electrolysis phenomena can also be important (Grimi 2010).

Effect of freezing on cell structure is the osmotic pressure difference between intracellular and extracellular fluid, then causing moisture migration which lead to cell lysis (Hui 2006). Moisture migration can be caused by osmotic differences and thermal gradients. The size of ice crystal formation depends on the rate of freezing. Slower freezing rate causing the formation of bigger ice crystal while faster rate freezing causing smaller ice crystal. Formation of ice crystal cause the expansion of volume which may cause cell rupture as shown in Figure 2.2. Freezing pre-treatment have another good effects on fruits which are slowing down the chemical,

biochemical reaction and physiological changes, limiting the microbial growth which resulting in nutritional and sensory characteristic improvement of final juice (Fennema 1975).

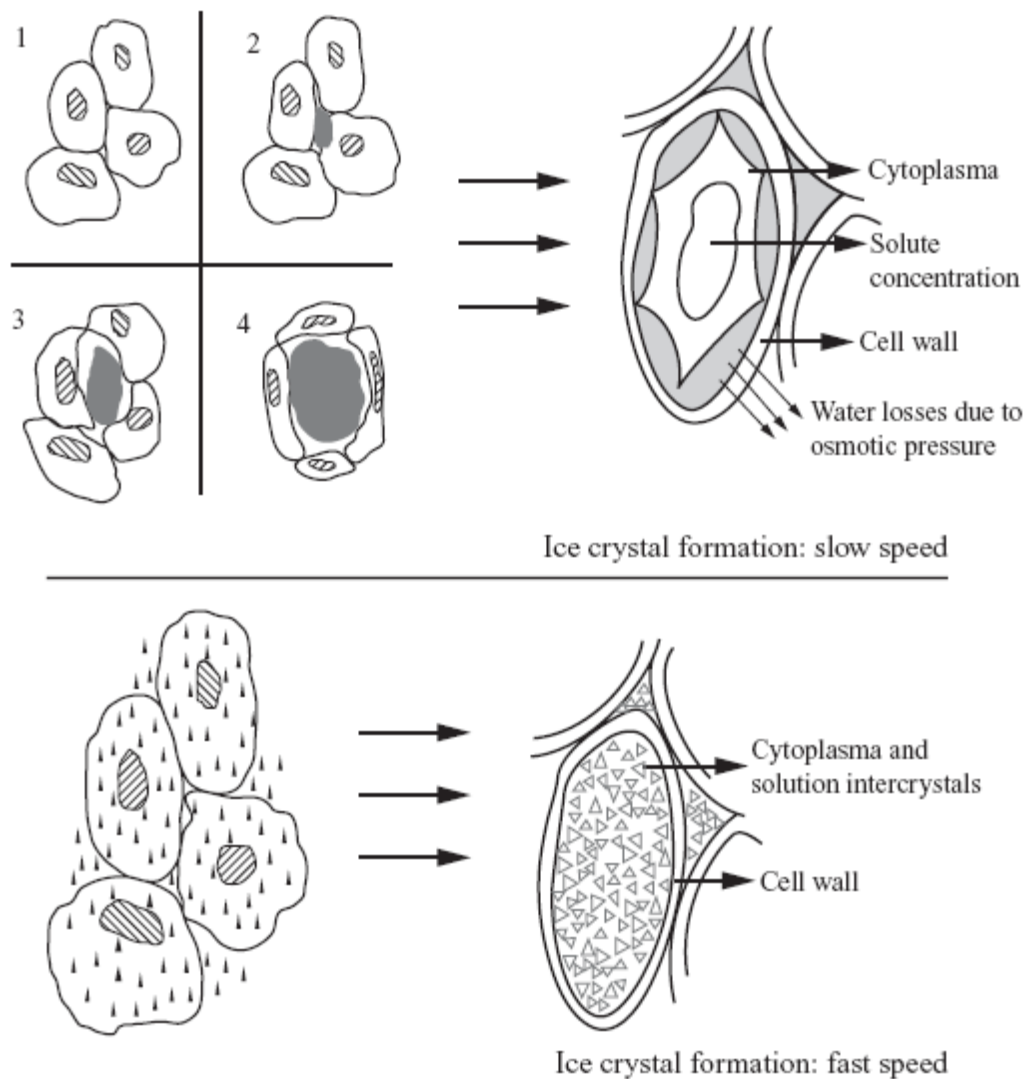


Figure 2. 2 Ice crystal formation in plant tissue

Source: (Hui 2006) Handbook of fruits and fruit processing

But in pre-treatment of fruits before expression, freezing/ thawing pre-treatment have higher cell disintegration index than others pre-treatments which are ohmic heating and pulsed electric field treatment (Mhemdi, Bals et al. 2012). There

are others factors which affect the fruit mass. Fruit quality: ripe fruits yield the best quality and quantity of juice. As fruits ripen, the substances that hold the cells together (hemicelluloses and firm pectic substances) break down and convert to water-soluble pectins which makes the fruit become softer and it can squeeze easily. Enzymes are also used to facilitate juice extraction. Pectolytic and cellulose or starch-splitting enzymes are added to the fruit mash. But enzyme pre-treatment should be done at the optimum pH, temperature, and time. Excessive enzymatic breakdown results in a viscous mash, from which the juice cannot be expressed (Sreenath 1994).

2.5 Modelling the expression behavior

There are various rheological models which can explain the expression theory, mostly, spring model and combination of spring and dashpot model is widely used. In mechanical expression, there are two period, filtration period and consolidation period, as shown in Figure 2.3. During the deliquoring operation, a cake is formed gradually above the filter medium. In the cake, the solids are pressed together to form a porous solid structure. As long as free solids are present, the operation is called filtration. When all the solids are forming a cake, and the deliquoring still continues, the phenomena transfer from filtration period to consolidation period.

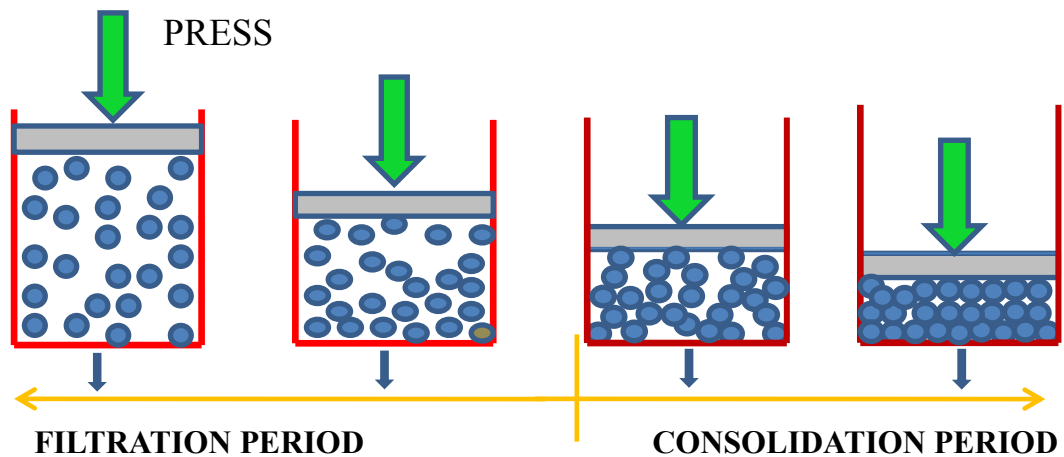


Figure 2. 3 Mechanical expression theory (filtration and consolidation)

2.5.1 Simple Terzaghi model

Spring model and combination of spring and dashpot model is widely used to explain the consolidation period of mechanical expression. However, the simplest model, so-called Terzaghi model, which uses only spring model, has expressed solid cake inside the expression chamber as a spring which carries the increasing applied load while reducing its volume.

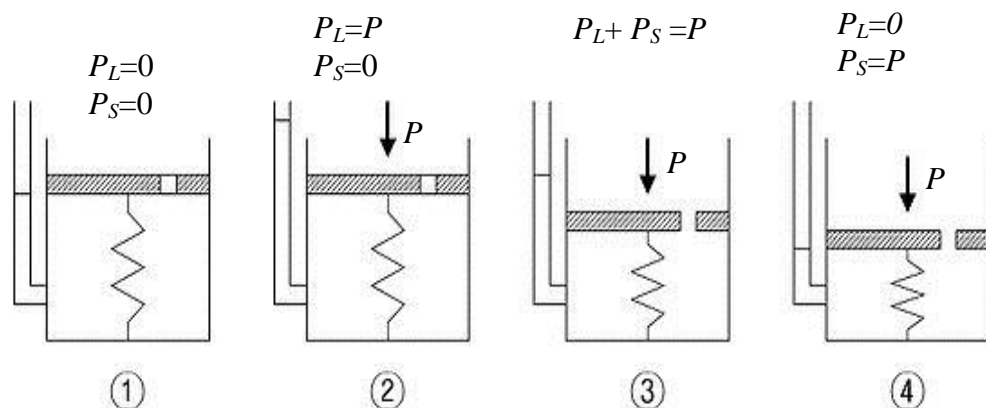


Figure 2. 4 Terzaghi's Spring Model

Source: <http://en.wikipedia.org/wiki/Consolidation>

In Terzaghi's spring model according to the Figure 2.4, stage 1 shows no applying pressure and no resistance. In stage 2, there is an applied pressure (P) with closed stopcock, during this condition the cake doesn't squeezed, the liquid inside the cake receives all the applied pressure (P) or force. It means that the hydraulic pressure or liquid pressure (P_L) should be equal to applied pressure, while compressive pressure or solid pressure (P_S) is zero. In stage 3, with open stopcock, liquid is expelled out, the expression is occurring. The applied pressure (P) is gradually/partly carried by solid particle in the cake. During this stage, the summation of P_L and P_S equals to P , until the change of the cake thickness reach plateau stage as shown as Stage 4, where applied pressure (P) is resisted by solid cake, P_S becomes to P , while P_L becomes zero.

During expression occurs, not only the liquid moves forward to filter medium but also the solid does, so that the position of the boundary of the system will change all the time leads to the difficulty of using fixed x- coordination. Thus, in order to derive the expression (consolidation) equation by using Terzaghi's theory, the theory should be modified by using the solid particle distribution (ω) which expressed as moving plane which contains ω (solid volume $\text{m}^3/\text{drainage area } \text{m}^2$) instead of the fixed x-coordinate (the fixed plane which apart from filter medium).

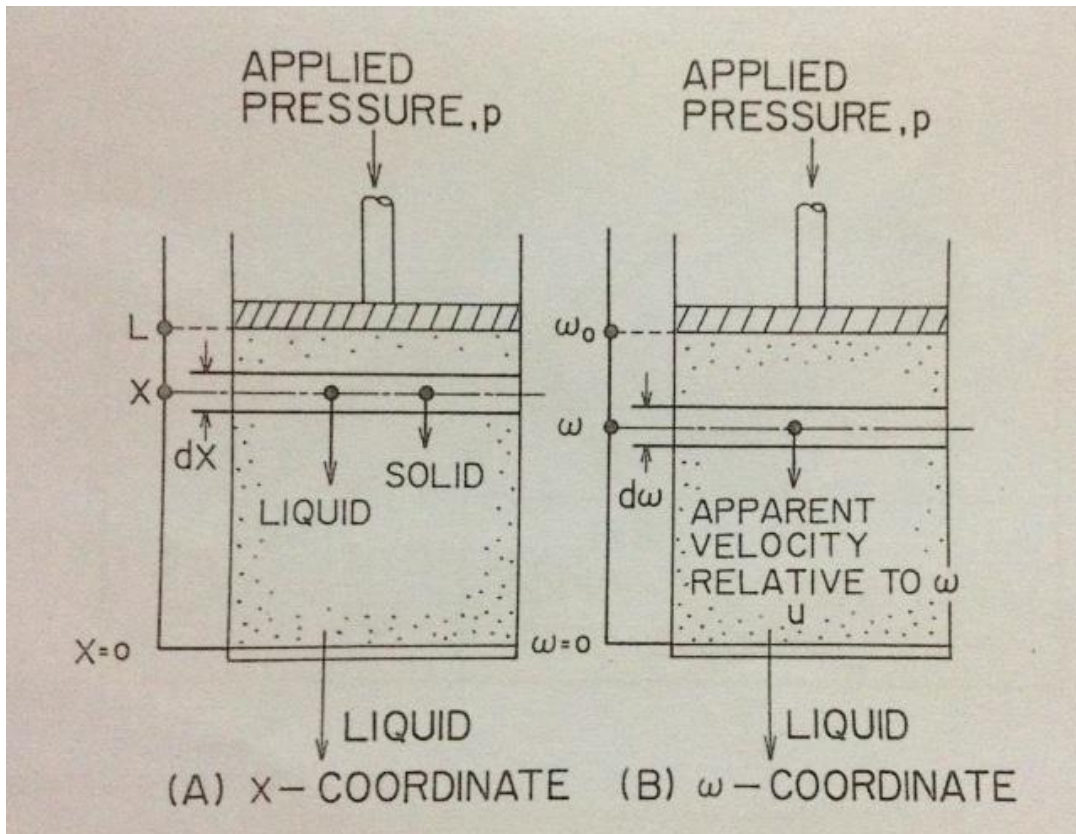


Figure 2. 5 Schematic picture of cake under consolidation
Source: (Shirato 1986)

The liquid mass balance inside the infinitesimal thickness of the layer $d\omega$ can be expressed by Equation (2.2)

$$\partial e \times \partial \omega = \partial u \times \partial \theta_c \quad (2.2)$$

Where:

e = local void ratio (-)

u = apparent relative liquid velocity to solid velocity (m/s)

θ_c = consolidation time (s).

The apparent relative liquid velocity to solid velocity which is the liquid squeezed out rate from the filter medium (u) can be expressed as Equation (2.3) (Shirato 1986).

$$u = \frac{1}{\mu\alpha\rho_s} \cdot \frac{\partial P_L}{\partial \omega} = -\frac{1}{\mu\alpha\rho_s} \cdot \frac{\partial P_S}{\partial \omega} \quad (2.3)$$

Where,

μ = liquid viscosity (Pa s)

α = local specific cake resistance (m/kg)

ρ_s = solid density (kg/m³)

P_L = hydraulic pressure or liquid pressure (Pa)

P_S = Compressive pressure or solid pressure (Pa).

Then the Equation 2.1 becomes as

$$\frac{\partial e}{\partial \theta_c} = \frac{\partial}{\partial \omega} \left(-\frac{1}{\mu\alpha\rho_s} \cdot \frac{\partial P_S}{\partial \omega} \right) \quad (2.4)$$

On the assumption that μ , ρ_s and α are constant values, and the relation between e and P_S is linear, Equation (2.4) becomes modified Terzaghi consolidation Equation (Eq.2.5).

$$\frac{\partial P_S}{\partial \theta_c} = C_e \frac{\partial^2 P_S}{\partial \omega^2} \text{ or } \frac{\partial e}{\partial \theta_c} = C_e \frac{\partial^2 e}{\partial \omega^2} \quad (2.5)$$

$$C_e = \frac{1}{\mu\alpha\rho_s(-de/dP_S)} \quad (2.6)$$

C_e (m²/s) is the modified consolidation coefficient which derived from Terzaghi consolidation theory by using the moving ω -coordinate. C_e is the value

which expresses how fast the applied pressure transfer toward the depth of the cake. It means that the larger C_e value, the sample is easier to squeeze and faster the expression rate.

By using the average value of C_e obtained from the beginning through the end of consolidation, for under constant expression pressure, the degree of consolidation progress as called the average consolidation ratio (U_C) can be expressed as follow Equation (Eq. 2.7)

$$U_C \equiv \frac{L_1 - L}{L_1 - L_\infty} = 1 - \exp\left(-\frac{\pi^2 i^2 C_e \theta_c}{4 \omega_0^2}\right) \quad (2.7)$$

Where

L = thickness of cake at any time θ_c (m)

L_1 = initial thickness of cake (m)

L_∞ = final thickness of cake (m)

i = number of drainage surfaces

θ_c = consolidation time (s)

ω_0 = total solid volume per unit sectional area (m^3/m^2)

U_C is the value to express the degree of the consolidation progress toward the final or equilibrium stage. The value of U_C varies from 0-1. When $U_C = 0$ means the cake is just start to be compressed. And $U_C = 1$ means the cake is completely compressed. The C_e value can be determined easily by linearization the Equation 2.7 as Equation 2.8.

$$\ln(1 - U_c) = -\frac{\pi^2 i^2 C_e}{4 \omega_0^2} \cdot \theta_c \quad (2.8)$$

2.5.2 Terzaghi-Voigt combined model

In Terzaghi model, the void ratio (e) inside the cake was assumed to be dependent only on local compressive pressure (P_S). However, in view of the fact that the change of the void ratio (e) does not cause by only the P_S but also by consolidation time (θ_c) so-called creeping effect too. Thus, in order to obtain more rigorous equation of consolidation, the creep effect (or secondary consolidation), which the void ratio (e) depends on not only P_S but also consolidation time (θ_c), should be combined to Terzaghi model (or primary consolidation) as shown the schematic rheological model in figure 2.6.

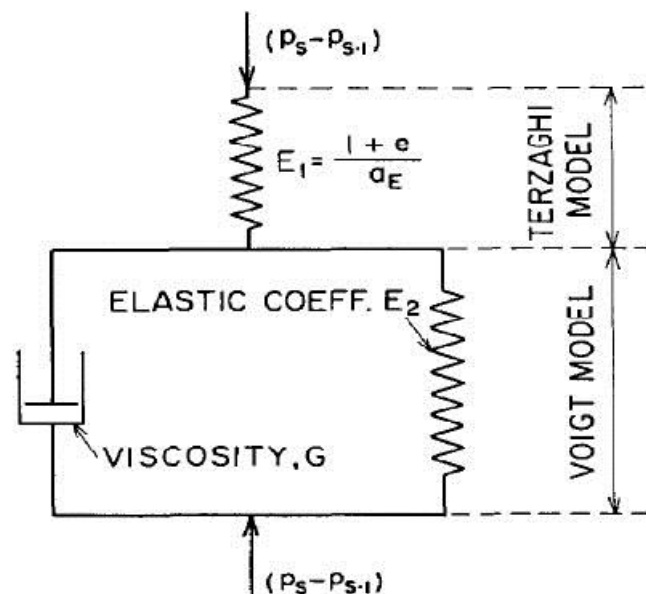


Figure 2. 6 Diagram of Terzaghi-Voigt combined model

Source: (Shirato 1986)

The above model shows that Terzaghi-Voigt combined model can be used to explain the consolidation phenomena where the simple Terzaghi model fails to describe the consolidation phenomena of the cake. Creep or secondary consolidation is the slow movement of the solid particles towards the filter medium in very slow rate by taking the void space under local solids compressive pressure. Up to 70% of the consolidation stage was secondary consolidation (Shirato 1986). However, there was no transition point between primary and secondary consolidation, they occurred simultaneously.

In Terzaghi-Voigt combined model, U_C can be expressed as Equation (2.9).

$$U_C = (1 - B) \left\{ 1 - \exp \left\{ -\frac{\pi^2 i^2 C_e \theta_c}{4 \omega_0^2} \right\} \right\} + B \{ 1 - \exp \{ -\eta \theta_c \} \} \quad (2.9)$$

Where:

B = Creep constant (-), defined by $\frac{V_{SC-max}}{V_{max}}$, where V_{SC-max} is the maximum liquid volume removed by the secondary consolidation and V_{max} is the maximum liquid volume at $\theta_c = \infty$

η = creep constant (s^{-1}), which expresses the velocity of progress of the creep defined by $\frac{E_2}{G}$, where E_2 is the elastic coefficient of the spring of Voight's model and G is the viscosity of the dash pot of Voight's model

According to the Equation (2.9), it is clearly that the creep constant (η) is the factor which dominates the velocity of the secondary consolidation.

2.5.3 Multi-staged creep model

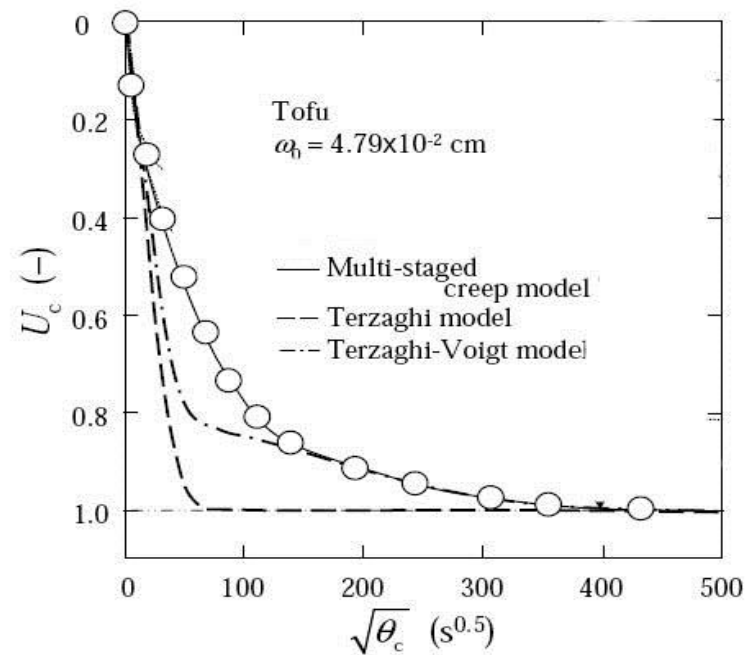


Figure 2. 7 Application of rheological model to tofu

Source: (Iritani, Katagiri et al. 2014)

The Figure 2.7 showed the expression behavior of tofu by the consolidation ratio (U_c) and time. The dashed line and dashed-dotted line are the predictions based on the Terzaghi and Terzaghi-Voigt combined model respectively. Both models cannot fit the experimental data of tofu. So, more Voigt elements were added to Terzaghi-Voigt combined model as schematic rheological model in Figure 2.8 to describe the experimental data.

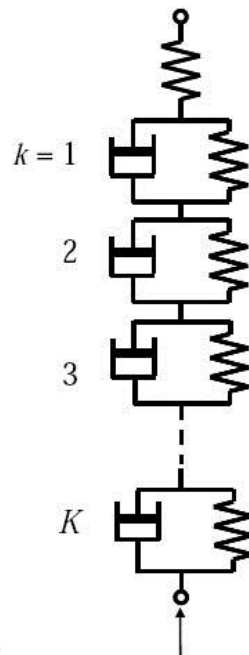


Figure 2. 8 Terzaghi and multi-staged Voigt combined model

Source: (Iritani, Katagiri et al. 2014)

2.6 Properties of Juice Quality

In terms of quality issues, the soluble solids content and titratable acidity are the major indicators to be taken into account when identifying the status and suitability of a juice product for use in an application.

The soluble solids content will relate directly to both sugars and fruit acids which are the main contributors. Pectins, glycosidic materials and minerals will also consider as a small but insignificant influence on the solids figure. Brix-calibrated optical refractometer was conveniently using for direct reading of degrees Brix.

The acidic character of a juice is another factor which contributes to juice flavor type and is taken into consideration when assessing the value of the juice. There are various acids present in fruit juices (e.g. oxalic, citric, tartaric, malic, etc.),

and it is usual to record acidity in terms of citric acid or malic acid for the majority of fruit juices.

As a general rule, the acidity of juice will decrease with increasing maturity of the fruit source, or with increasing levels of sugars in the resulting juice. Hence, the ratio of soluble solids (Brix values) to acidity is an important value in the assessment of juice quality. The ratio of Brix/acid is used to be when determining standard sensory or taste, qualities, and to minimize the effect of seasonal variation. The higher the Brix value in relation to the acid content of the juice, the higher the ratio and the 'sweeter' the taste.

The large number of juices contain ascorbic acid or vitamin C, which is quantitatively the most important vitamin in fruit juices ranging from a negligible level to 200mg/100g in some berries. Vitamin C performs a valuable function as an antioxidant in fruit juices. But vitamin C is highly degradable antioxidant when it expose to higher ambient temperature, low temperature storage, light, and oxygen.

CHAPTER III

METHODOLOGY

3.1 Materials

3.1.1 Fruits

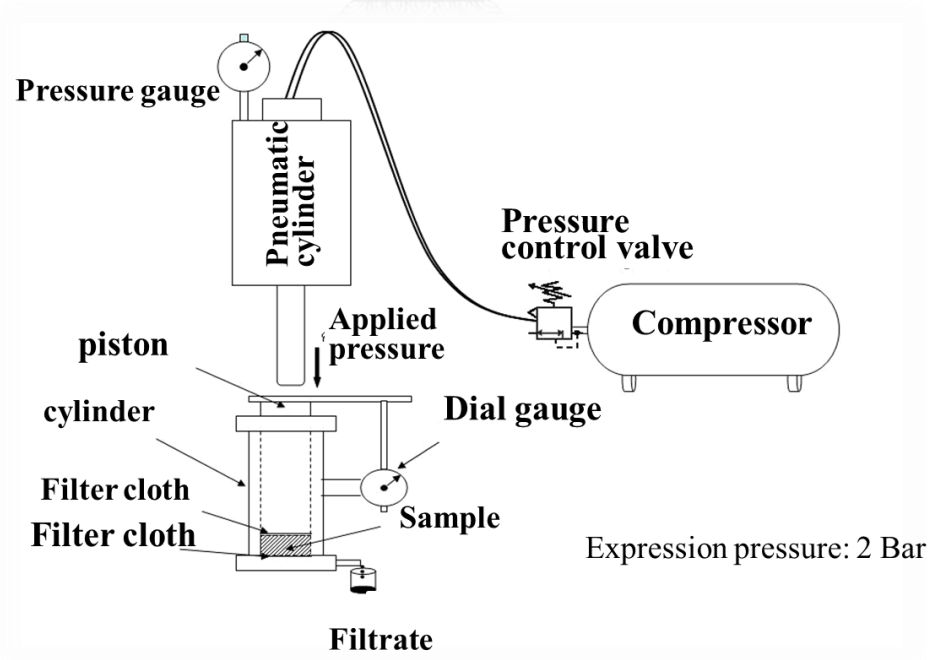
Two fruits, apples and pineapples, were chosen for the systematic experimental study. Fuji apples (*Malus domestica*) and Siracha pineapples (*Ananas comosus*) were taken into account. The choice of the two fruits: pineapple and apple, varieties and maturities was made considering their expected differences in nature, composition and quality properties. The initial composition of fresh apples and pineapples were presented in Tables 2.1 and 2.2. Both apple and pineapple fruits for the experiment were purchased from local market, Bangkok, Thailand.

3.1.2 Expression equipment

Expression permeability cell (expression chamber) is made from a stainless steel acting cylinder with a stainless steel piston inside the chamber. The diameter of the chamber is 6 cm. as shown in Figure 3.1(a). Two drainage surfaces were conducted by fixing the filter cloth at the bottom of the cylinder and the bottom of the piston contacting to sample. The piston of the cylinder is pressed by 16 cm. of diameter of pneumatic cylinder as shown in figure 3.1(b). The compressed air applied to the pneumatic cylinder was kept constant at 2 bar by a pressure regulator (pressure control valve) and a pressure gauge, therefore the expression pressure was applied to the sample was 14.2 bar, constantly. The changes of cake thickness with time were continuously measured by dial-gauge which mounted to the expression chamber cylinder.



(a)



(b)

Figure 3. 1 Expression permeability cell

3.1.3 Instruments

The following instruments were used in this study:

- Digital meter – Mitutoyo corporation
- pH meter – Mettler-Toledo (Thailand) Ltd.
- Refractometer – ATAGO 0033044, made in Japan.
- Hot air oven – Conthermthermotec 2000, Contherm Scientific Ltd, Hutt city, New Zealand.
- Filter papers – 70 mm, Cat No. 1001-070, Whatman international Ltd.
- Digital Caliper – 0.01-100mm, Model 19974, Shinwa Rules Co., Ltd.

3.1.4 Chemicals

The following chemicals were used in this study:

- CuSO₄ Copper II Sulphate – Batch No. 1208611, 1007-500G, Unilab Ajax Finechem Pty Ltd.
- 2,6 – Dichlorophenol Indephenol Sodium Salt – Lot BC BB7107, Fluka Analytical Sigma-Aldrich Co., Ltd.
- L – Ascorbic Acid – Batch No. 1007089, 79-500G, Fluka Analytical Sigma-Aldrich Co., Ltd.
- Sodium Hydroxide Pellets – UN No. 1823, Lot #SG53401301, LobaChemie Co., Ltd.
- Acetone – Batch No. A1084-1-2500, 112784-0714, QReC Co., Ltd.

- Sodium Carbonate – P.C: S0156, CAS No. 497-19-8, Rankem RFCL Co., Ltd.
- Meta-phosphoric Acid – Lot No. 0000483659, EC No. 253-433-4, #135324.1209, AppliChemPanreac ITW Co., Ltd

3.2 Methods

3.2.1 Raw materials selection and preparation

Commercially available mature pineapples and apples were purchased from local supermarket, Tesco Lotus, Bangkok, Thailand. The stage of ripeness of pineapples was chosen as stage 5 (Selvarajah, Bauchot et al. 2001). Fruits was selected on the basis of size and color uniformity, and blemished and diseased fruits were discarded. Fruits were washed, peeled, chopped and weighed 100 grams for each replication. Chopped fruits were put into the plastic bags and vacuumed before freezing.

3.2.2 Pre-treatments

3.2.2.1 *Slow freezing*

Chopped fruits of 100 grams were put into the plastic bags and vacuumed and froze in the freezer with temperature of -18°C for 24 hours Appendix B.1.

3.2.2.2 *Rapid freezing*

Chopped fruits of 100 grams were put into plate and froze by liquid nitrogen with temperature of -90°C for 5 minutes Appendix B.2.

3.2.2.3 Thawing

Frozen fruits were thawed at laboratory room with temperature of $28\pm 2^{\circ}\text{C}$ for 30 minutes before expression.

3.2.3 Expression operation

Pre-treated fruits were put in the expression chamber of expression permeability cell with constant pressure at 1.42 MPa (14.2 bar). Initial height and weight before expression were noted and expression data was read from the digital meter attached to the body of expression chamber. The total duration of each experiment was set at 3 hours. After expression, juice and final cake weight, and final cake thickness were recorded for further investigation. After thawing of frozen/thawed samples, dripping juice weight was measured separately to know the freezing/thawing effect on extraction of liquid inside the fruit tissues. Dripping means juice obtained after thawing. Total obtained juice weight means total weight of juice from dripping and expression. Percentage yield was determined by $\text{percentage yield} = (\text{total weight of obtained filtrate}) / (\text{initial weight of sample}) \times 100\%$. Percentage recovery was determined by $\text{percentage recovery} = (\text{total weight of obtained filtrate}) / (\text{initial weight of sample} - \text{weight of insoluble solid in sample}) \times 100\%$.

3.2.4 Determination properties of pineapple and apple juice

3.2.4.1 pH

pH were determined by using digital pH meter. pH meter was calibrated using standard solution and pH of apple and pineapple juices obtained from expression experiments were measured by dipping the pH meter into juices. The values of pH were read from digital meter.

3.2.4.2 Acidity

Acidity was determined by titration method and reported as percentage of citric acid (g citric acid/100 ml). (AOAC 2005). It was determined by direct titration against standardized alkali solution (e.g. 0.1M sodium hydroxide) to an end-point and it can find out accurately by using phenolphthalein as an indicator.

3.2.4.3 Soluble solid

Soluble solid was determined by using hand refractometer reported as degree Brix.

3.2.4.4 Sugar Acid Ratio

It was determined by dividing the sugar concentration in terms of soluble solids (Brix) by the citric acid concentration.

$$\text{Sugar Concentration (Brix) / Citric Acid Concentration} = \text{Sugar/Acid ratio}$$

3.2.4.5 Moisture content

Moisture content was determined by drying oven method until the samples reached a constant weight. (AOAC 2005). The final cakes after expression experiment were put into the hot air oven until the weight became constant. Moisture content was calculated by as follows:

$$\text{Moisture content} = (\text{Weight final cake} - \text{Weight after dry}) / \text{Weight final cake}$$

3.2.4.6 Vitamin C

Vitamin C content was determined by titration method according to AOAC and reported as mg/100 ml. (AOAC official method 967.21, 2000).

3.2.5 Storage determination

Slow freezing pre-treatment method was used in determination of storage effect for 3, 6, and 9 weeks. Normal freezer was used to store the samples. And expression operation was performed according to the step 3.2.3.

3.2.6 Model fitting

In order to estimate the U_c , the best fit values of B , η and C_e were determined by non-linear solving program; Solver add-in software of Microsoft Excel 2013 as following steps.

a) Determination of initial values of B and η

According to the Terzaghi-Voight combined Equation (2.9), normally, $\frac{\pi^2 i^2 C_e}{4 \omega_0^2} \gg \eta$ then it becomes approximately Equation (3.1), when $\theta_c \gg 0$. (Shirato 1986)

$$U_c \cong 1 - B \exp(-\eta \theta_c) \quad (3.1)$$

Thus, B and η can be determined by linear regression of the experimental data plot between $\ln(1-U_c)$ versus θ_c as Figure 2.7. These obtained values of B and η will be used as initial values for best fitting in the next step.

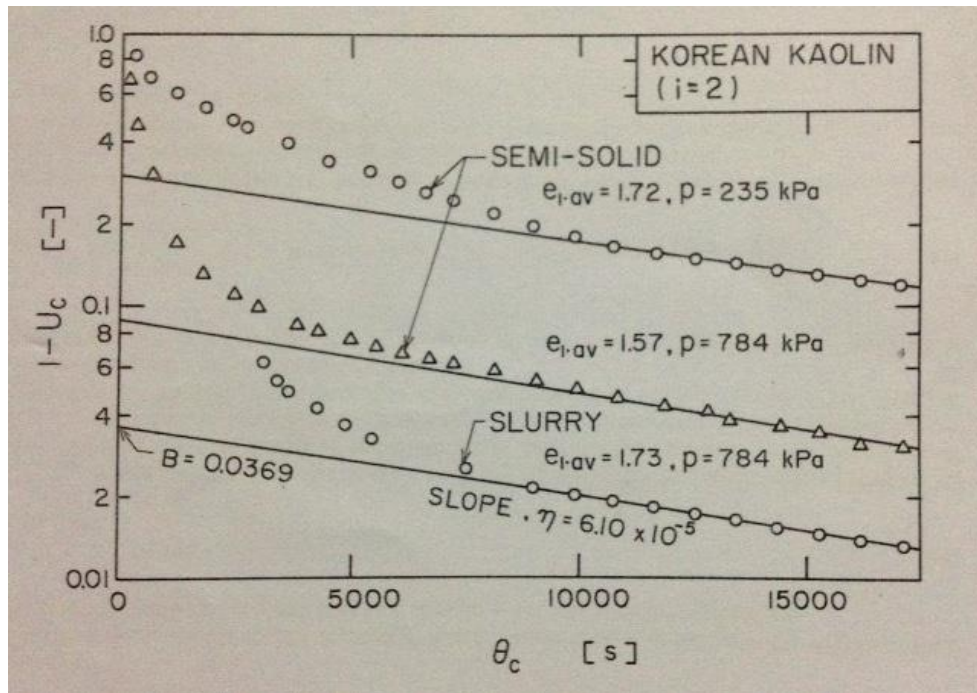


Figure 3. 2 Determination of creep constants, B and η
Source: (Shirato 1986)

b) Determination of the initial value of C_e , and the best fit values of B , η and C_e .

The C_e value of Terzaghi model as expressed in Equation (2.7) was determined by linearization method as Equation (2.8). The value was used as initial value of C_e which should be put in non-linear solving program.

Non-linear solving program; Solver add-in software of Microsoft Excel 2013 was applied to determine the best fit values of B , η and C_e of the Terzaghi-Voight combined model by using of the initial value of C_e , and initial values of B and η in a step a). The minimum of summation of error square was used as constrain for determining these values.

3.2.6.1 Simple Terzaghi Model

Obtained data on the changes of cake thickness as U_c was fitted by Equation (2.7) by using Solver add-in software of Microsoft Excel 2013.

3.2.6.2 Terzaghi-Voight Combined Model

Obtained data on the changes of cake thickness as U_c was fitted by Equation (2.9) by using Solver add-in software of Microsoft Excel 2013.

3.2.6.3 Empirical equation

Two parameters equation as following equation (Equation. (3.2)) was used to fit the normalized cake thickness ($\frac{L}{L_1}$) changes with time.

$$\frac{L}{L_1} = 1 - \frac{a\theta_c}{(b+\theta_c)} \quad (3.2)$$

Where:

L = cake thickness at anytime

L_1 = initial cake thickness

a = change in cake thickness

b = constant

θ_c = expression time

3.2.7 Statistical Analysis

Effects of pretreatment will be studied on two types of fruits containing soluble and insoluble fibers. Effects of pre-treatment and storage time on properties of juice were studied. Data will be analyzed by using RCBD and analysis of variance at 95% confidence level.

Note: Experiments and analysis of composition will be conducted in triplicate



CHAPTER IV

RESULTS AND DISCUSSION

4.1 Expression experiments

In this research experiments, pineapple was chosen as fruit rich in insoluble fiber and apple as fruit rich in soluble fiber although they contained both soluble and insoluble fiber. It can be considered that because of final cake thickness of frozen/thawed pineapple samples were thicker than the apple samples.

There were many ways of defining the dietary fiber including botanically, chemically and digestibility. Plant foods are the only source of dietary fiber. All the fractions (cellulose, lignin, hemicellulose, pectins, gums and mucilages) of dietary fiber are the major constituents of plant cell wall (Selvendran 1984). Dietary fiber is subdivided into Insoluble and soluble dietary fiber depending on their solubility in water. Generally, most of the fruits except pineapple have a balance ratio of soluble and insoluble fiber. Data on the amount of dietary fiber in foods varies according to the definition of dietary fiber chosen and the measurement method used. There can be considerable variation between different methods to measure 'total' dietary fiber content in some foods. However, pineapple was considered to be rich in insoluble fiber than apple fruits.

4.1.1 Effect of pre-treatments

The cylinder position obtained from expression tests of apple and pineapple samples were shown in Figures 4.1 and 4.2 respectively. These figures were plotted between changes in depth of cylinder thickness and expression time for 3 hours. In addition, effects of pre-treatments were compared in the graph. The solid

line in the graph described as the final depth of cylinder at 49.07 mm. The length between the data that plotted in the graph with the solid line represented the cake thickness. As shown in Figure 4.1, the rate of the change in cake thickness in first 10 minutes was very first and the slope of initial part of the graph went down rapidly and reached to plateau and steadily changes until the end of experiment. Among three types of pre-treatment, slow freezing pre-treated sample reached to plateau faster than the control and cryogenic freezing pre-treated samples. This is because slow freezing have greater effect of breaking down the cell wall than cryogenic freezing method (Fennema 1975). When compare with control and frozen/thawed samples, the frozen/thawed samples reach to the point where the final cake thickness reduced more than 95% in first ten minutes while the control took 3 hours to reach that point. Frozen/thawed samples almost reached to the end of cylinder depth at 49.07 mm.

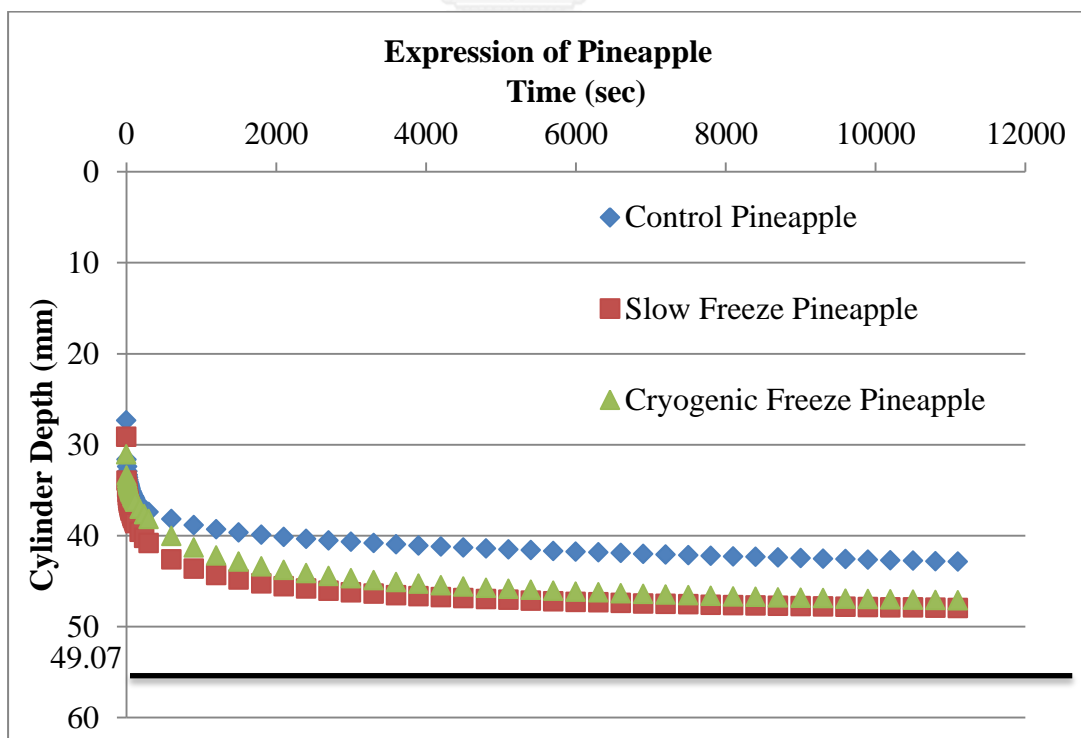


Figure 4. 1 Effect of pre-treatments on expression of pineapple

As shown in Figure 4.2, huge effect of freezing/thawing was seen in apple samples with 3 types of pre-treatment. It was also clearly found out that the point after 3 hours expression of control sample was easily achieved by frozen/thawed samples within 5 minutes. And it was clearly seen that freezing pre-treatment gave better expression performance, faster expression rate and more yield. Control sample gave only 23% and 59% of thickness change in 3 hours expression of apple and pineapple respectively. While frozen/thawed samples gave more than maximum of 99% and 89% for apple and pineapple in the same period of expression as shown on Table 4.1 ($P \leq 0.05$).

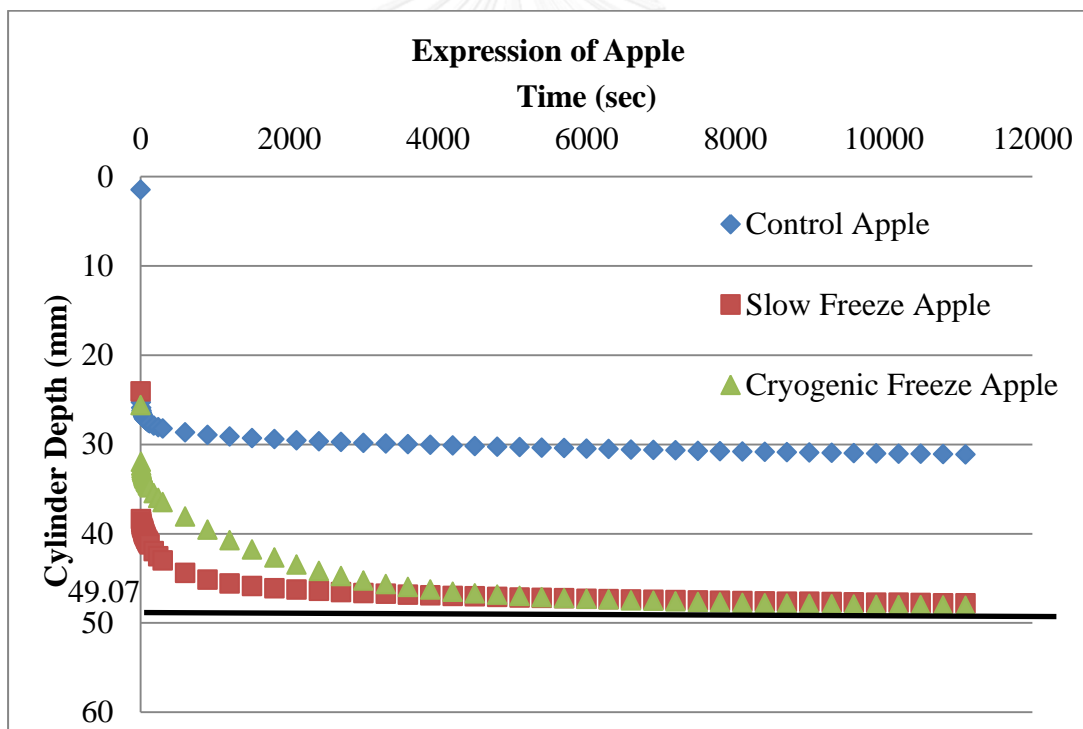


Figure 4. 2 Effect of pre-treatments on expression of apple

The rapid changes in the initial stage of consolidation of frozen/thawed samples may be because freezing and thawing induced the breakdown of cell wall and

formation of ice crystal during freezing destroyed the cellular compartments resulting in a reduction of fruit firmness (Chassagne-Berces 2009, Chassagne-Berces 2010).

When comparing between apple and pineapple expression without any pre-treatments between the L/L_1 and expression time of apple and pineapple samples was shown in Figure 4.3. It was observed that expression of pineapple was easily squeezed than apple. For the same weight of apple and pineapple expression, apple was expressed only 23% of total expression ratio while the pineapple was expressed up to 60% and the initial part of the slope went down fast in pineapple. It may be because of their different native tissue structure. In the initial state of apple before expression was very rigid compare to pineapple. Apple was mainly composed of pectin which support the tissue while pineapple native structure was soft in texture and composed of fiber (Hassan, Othman et al. 2011).

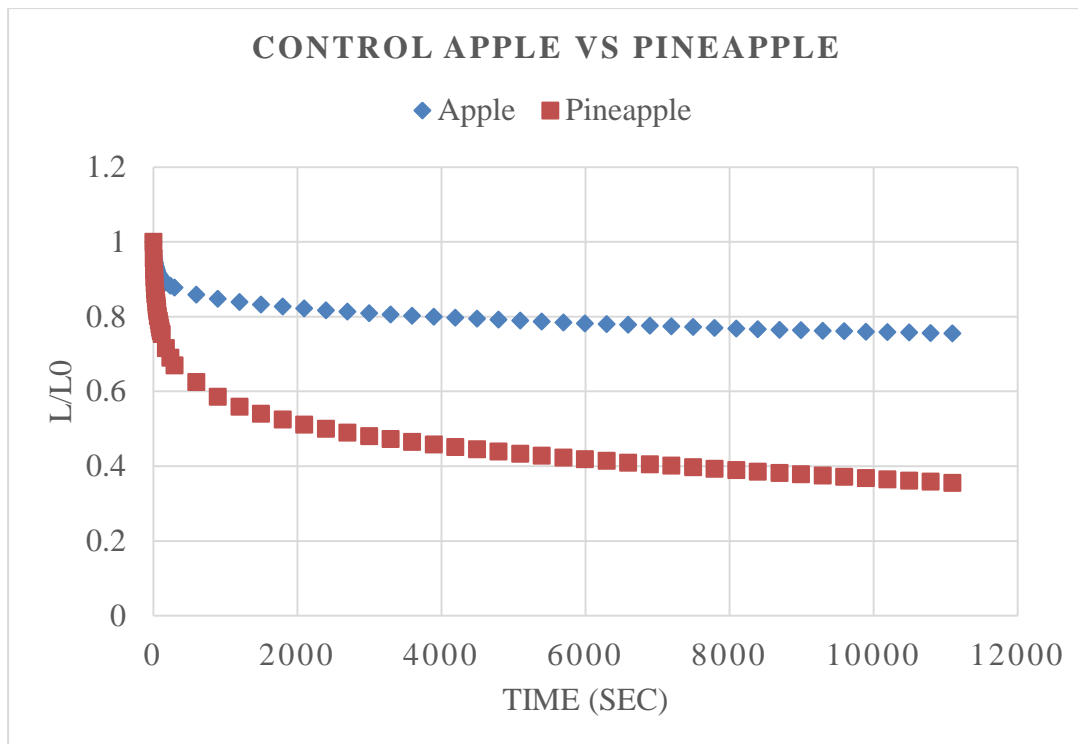


Figure 4. 3 comparison between normalized fitting of cake thickness between control apple and pineapple

Moreover, the rate of expression in fruits containing insoluble fiber was faster than the fruit rich in soluble fiber. But the expression behavior of apple (representative of soluble fruit) was fitted by both Terzaghi and Terzaghi-Voight combined model while the expression behavior of pineapple fruit was fitted only by Terzaghi-Voight combined model.

4.1.2 Juice yield and recovery

The effect of pre-treatments and storage time on percentage yield and recovery of pineapple and apple expression experiments are shown in Tables 4.1 and 4.2. It was noticeably found out that frozen/thawed samples of pineapple had significantly higher percentage yield of 82-83% against 65% and percentage recovery of 84-86% vs. 69% than the control samples.

Table 4. 1 the effect of pre-treatment on percentage yield and recovery of apple and pineapple expression

Types of Fruit	Pre-treatment (°C)	Percentage Yield (%)	Percentage Recovery (%)
Pineapple	Control	64.8 ^a ±5.87	68.73 ^a ±6.38
	-18°C	82.11 ^b ±3.71	84.42 ^b ±3.88
	-90 °C	82.89 ^b ±2.8	85.58 ^b ±2.93
Apple	control	32.64 ^a ±3.98	36.68 ^a ±4.19
	-18 °C	82.68 ^b ±3.38	85.27 ^b ±3.57
	-90 °C	88.15 ^b ±0.87	90.58 ^b ±0.89

Values are mean ± standard deviation of triplicate sample determinations

Means in the same column in same fruits with different superscript letters are significantly different ($p \leq 0.05$)

All determinations were performed in triplicate

It was clearly found out that frozen/thawed samples of apple had significantly higher percentage yield of 83-88% against 33% and percentage recovery of 85-91% vs. 37% respectively than the control samples. Dripping juice represented by-products of freezing/thawing and data of dripping juices after thawing was shown in Table 4.2 ($p \geq 0.05$). It was markedly noticed that freezing/thawing facilitated the extraction of intracellular liquid, dripping was resulted from breakdown of cell wall and leakage of intracellular liquid. It was because frozen/thawed samples were easily squeezed than control samples. Without beginning any expression operation, nearly one fourth of total obtained juice can be collected just by freezing/thawing. Comparing between fruits containing soluble and insoluble fibers, pineapple fruits were easily damaged by freezing/thawing because weight of dripping juice of pineapple was significantly higher than the apple dripping juice.

Table 4. 2 Effect of freezing/thawing and storage time on dripping juice

Types of fruits	Storage Time	Dripping juice (mg)	Total Obtained Juice (mg)
Pineapple	Control/0 week	-	-
	-18°C/0 week	23.77 ^{AB} ±0.42	82.38 ^A ±3.7
	-90°C/0week	21.78 ^A ±1.38	82.46 ^A ±3.96
	3 weeks*	23.53 ^a ±1.11	77.17 ^a ±1.52
	6 weeks*	24.43 ^a ±1.66	83.90 ^b ±1.54
	9 weeks*	24.25 ^a ±0.87	93.85 ^c ±0.67
Apple	Control/0 week	-	-
	-18°C/0 week	13.58 ^A ±0.97	83.1 ^A ±3.57
	-90°C/0 week	12.17 ^A ±0.46	88.52 ^B ±0.86
	3 weeks*	12.85 ^a ±0.46	81.81 ^a ±1.49
	6 weeks*	13.3 ^a ±0.58	86.39 ^b ±0.86
	9 weeks*	12.89 ^a ±0.65	92.76 ^c ±1.51

Values are mean ± standard deviation of triplicate sample determinations

Means in the same column in same fruits with different superscript letters are significantly different ($p \leq 0.05$). * Means samples were froze at -18°C for 24 hours.

All determinations were performed in triplicate.

4.1.3 Effect of storage time

As shown in Table 4.3 ($P \leq 0.05$) that frozen/thawed stored samples of pineapple have significantly higher percentage yield at longer storage time of 3, 6, and 9 weeks with 77, 83, and 93% respectively. The results of percentage recovery of 3, 6, and 9 weeks were 81, 88, and 98% respectively. But in Table 4.2 showed that

there is no significant different of dripping juice between 3, 6, and 9 weeks of pineapple and apple. And highest obtained juice was obtained in 9 weeks stored pineapple and apple.

It was observed that frozen/thawed stored samples of apple have significantly higher percentage yield at longer storage time of 3, 6, and 9 weeks with 81, 86, and 92% respectively. The results of percentage recovery of 3, 6, and 9 weeks were 85, 90, and 97% respectively. Percentage yield and recovery values of apple and pineapple samples were rapidly increasing according to longer storage time up to 93% yield and 97% recovery for pineapple and 92% yield and 96% recovery for apple. But there were significant differences on storage effect based on dripping which can be concluded that 3 weeks of storage time was enough to destroy the cell wall of fruits. The longer the storage time, the higher the percentage yield was observed because of low temperature destroyed cell wall of the fruits and the longer storage time the more degree of damage was occurred (Hui 2006).

Table 4. 3 the effect of storage time on percentage yield and recovery of apple and pineapple expression

Types of Fruit	Storage Time (week)	Percentage Yield	Percentage Recovery
Pineapple	3	76.81 ^a ±1.49	81.38 ^a ±1.88
	6	83.48 ^b ±1.58	87.9 ^b ±1.57
	9	93.24 ^c ±0.66	97.58 ^c ±0.86
apple	3	81.49 ^a ±1.63	85.26 ^a ±1.82
	6	85.95 ^b ±0.85	90.14 ^b ±0.85
	9	92.25 ^c ±1.58	96.51 ^c ±1.78

Values are mean ± standard deviation of triplicate sample determinations

Means in the same column in same fruits with different superscript letters are significantly different ($p \leq 0.05$).

All determinations were performed in triplicate

4.2 Model fitting by Simple Terzaghi and Terzaghi-Voight combined model

Tables 4.4 and 4.5 showed the parameters that obtained by fitting method.

Table 4. 4 Model fitting on exprossion behavior of apple on *Uc*

Model	Treatment	Ce/ω_0^2 (1/s)	B (-)	η (1/s)	Error ²	r^2
Terzaghi	Control	$4.11 \times 10^{-05} \pm$ 1.75×10^{-06}	-	-	$3.44 \times 10^{-01} \pm$ 3.59×10^{-02}	0.936
		$1.01 \times 10^{-04} \pm$ 2.05×10^{-05}	-	-	$2.68 \times 10^{-01} \pm$ 3.09×10^{-02}	0.956
Terzaghi Voight	Control	$6.72 \times 10^{-05} \pm$ 1.15×10^{-05}			$3.51 \times 10^{-02} \pm$ 1.87×10^{-02}	0.995
		$7.6 \times 10^{-04} \pm$ 4.43×10^{-06}	$6.77 \times 10^{-01} \pm$ 196×10^{-02}	$2.57 \times 10^{-04} \pm$ 6.95×10^{-06}	$1.09 \times 10^{-02} \pm$ 2.24×10^{-04}	0.998
	-18°C	$3.97 \times 10^{-04} \pm$ 1.56×10^{-05}	$4.29 \times 10^{-01} \pm$ 4.09×10^{-02}	$3.16 \times 10^{-04} \pm$ 1.45×10^{-05}	$3.44 \times 10^{-03} \pm$ 5.45×10^{-04}	0.999
		$6.85 \times 10^{-04} \pm$ 3.49×10^{-04}	$8.23 \times 10^{-01} \pm$ 1.26×10^{-01}	$5.27 \times 10^{-04} \pm$ 2.49×10^{-05}	$3.39 \times 10^{-3} \pm$ 2.74×10^{-03}	1

Values are mean \pm standard deviation of triplicate sample determinations

Means in the same column with different superscript letters are significantly different ($p \leq 0.05$)

All determinations were performed in triplicate

Table 4. 5 Model fitting on expression behavior of pineapple on Uc

Model	Treatment	Ce/ω_0^2 (1/s)	B (-)	η (1/s)	Error ²	r^2
Terzaghi	Control	$5.67 \times 10^{-05} \pm 3.34 \times 10^{-06}$	-	-	$4.25 \times 10^{-01} \pm 1.93 \times 10^{-02}$	0.921
	-18°C	$8.12 \times 10^{-05} \pm 1.06 \times 10^{-05}$	-	-	$1.94 \times 10^{-01} \pm 1.59 \times 10^{-02}$	0.969
	-90°C	$6 \times 10^{-05} \pm 7.37 \times 10^{-06}$	-	-	$1.16 \times 10^{-01} \pm 2.41 \times 10^{-02}$	0.982
Terzaghi-Voight	Control	$6.68 \times 10^{-04} \pm 4.93 \times 10^{-06}$	$5.79 \times 10^{-01} \pm 1.63 \times 10^{-02}$	$2.77 \times 10^{-04} \pm 3.85 \times 10^{-06}$	$8.77 \times 10^{-03} \pm 2.15 \times 10^{-04}$	0.998
	-18°C	$4.13 \times 10^{-04} \pm 1.28 \times 10^{-05}$	$5.44 \times 10^{-01} \pm 3.85 \times 10^{-02}$	$3.76 \times 10^{-04} \pm 7.87 \times 10^{-06}$	$5.1 \times 10^{-03} \pm 1.48 \times 10^{-03}$	0.999
	-90°C	$3.07 \times 10^{-04} \pm 1.34 \times 10^{-05}$	$6.35 \times 10^{-01} \pm 6.37 \times 10^{-02}$	$3.57 \times 10^{-04} \pm 1.41 \times 10^{-05}$	$3.42 \times 10^{-03} \pm 5.56 \times 10^{-04}$	0.999

Values are mean \pm standard deviation of triplicate sample determinations

Means in the same column with different superscript letters are significantly different ($p \leq 0.05$)

All determinations were performed in triplicate

The results of model fitting by Terzaghi and Terzaghi-Voight combined model were shown in Tables 4.4 and 4.5 for apple and pineapple. Terzaghi model was failed to apply the expression mechanisms of frozen/thawed apple and pineapple as shown in Figures 4.4 and 4.5 where the graph plotted between the U_c and expression time with 3 pre-treatments. For control samples, the experiment did not reach an equilibrium stage and only 30% of data were recorded after 3 hours experiment and it cannot be used to fit by models. To prove that, normalized fitting of cake thickness was done at step 4.3.

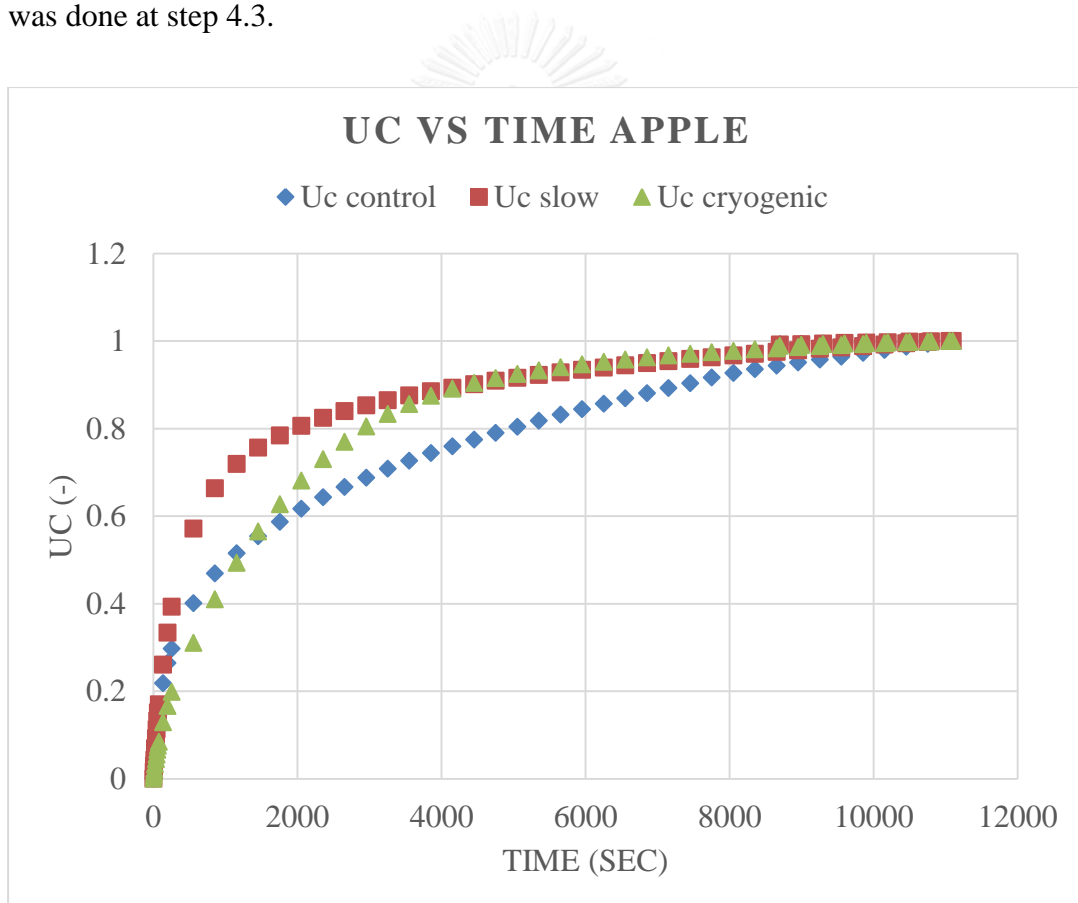


Figure 4. 4 U_c vs time of apple samples with 3 types of pre-treatment

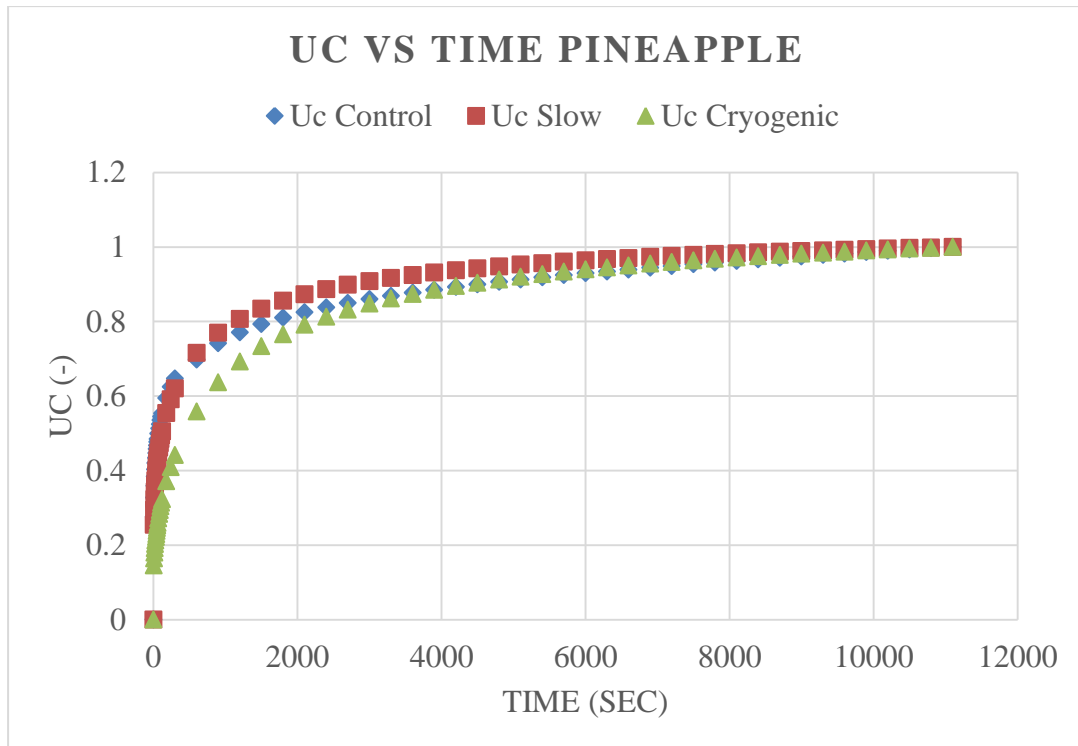


Figure 4. 5 U_c vs time of apple samples with 3 types of pre-treatment

Figures 4.4 and 4.5 show that experimental U_c plotted against expression time of slow freezing pre-treated apple and pineapple, gave good results than those of control and cryogenic freezing pre-treated samples. Terzaghi-Voight combined model was being able to fit better than Terzaghi model in apple and pineapple as shown in Figures 4.6 and 4.7. In Terzaghi-Voight combined equation, it consists of two terms of primary consolidation and secondary consolidation that are happening parallel during the expression mechanisms with parameters of C_e , B and η . Where B represented which mechanisms dominate the responsibility of maximum juice removed and η represented rate of expression dominated by secondary consolidation and C_e represented the index where ratio of cake thickness multiplied by expression time.

As shown in Tables 4.3 and 4.4 ($P \leq 0.05$), the least values of error and highest values of R^2 , C_e and η were used to find. The B value was recorded minimum of 50% and up to 70% for pineapple and minimum of 39% and up to 90% for apple respectively. The highest r^2 values were observed in Terzaghi-Voight combined model and least error values were observed in Terzaghi-Voight combined model of apple and pineapple. Moreover, smaller B values were used to find to compare between slow and cryogenic freezing pre-treatment. According to the equation, B value represented the percentage of expression mechanisms dominated by secondary consolidation so called creep effect. Lower values of B were observed in slow freezing pre-treated samples of apple and pineapple. This is because it was mainly dominated by spring effect and less affected by secondary consolidation or creep effect.

Apple and pineapple data fitted by Terzaghi model and Terzaghi-Voight combined model were shown in Figures 4.6 and 4.7 where the graph plotted between U_c and expression time of frozen/thawed apple and pineapple samples fitted by Terzaghi model (dotted line) and Terzaghi-Voight combined model (dash line). It was observed that simple Terzaghi spring model dotted line in Figures 4.6-4.7 cannot fit the expression mechanisms of fruit samples. Terzaghi-Voight combined model can fit the experiments very well. Secondary consolidation has big effect to expression mechanisms of fruit samples.

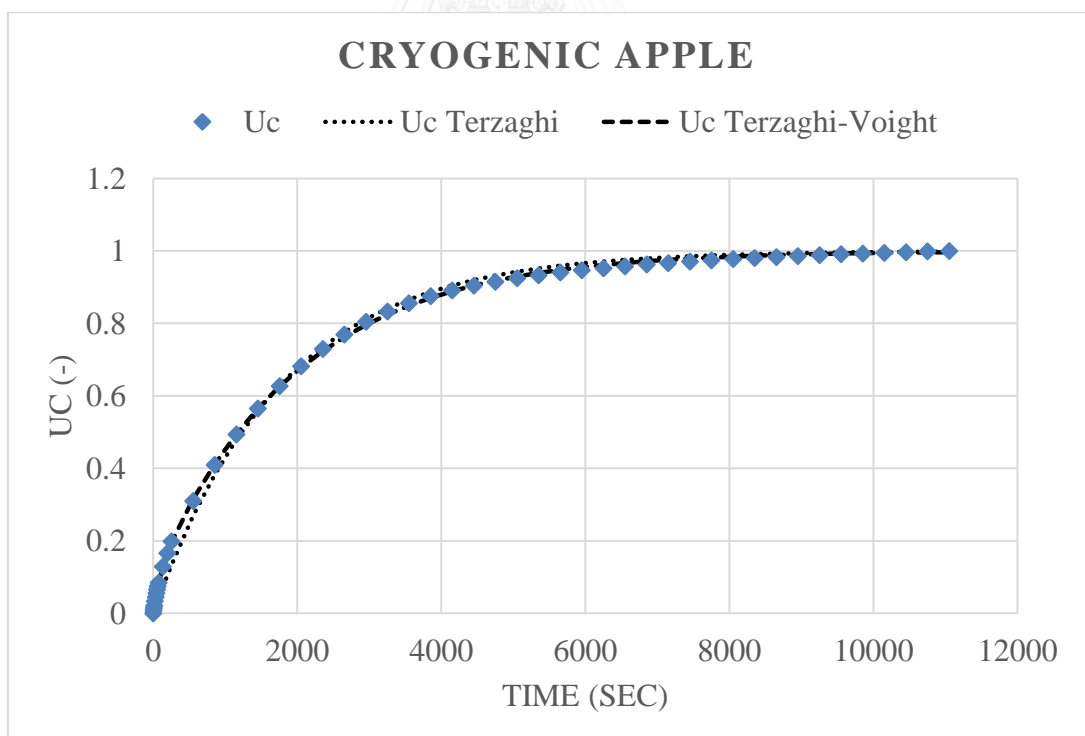
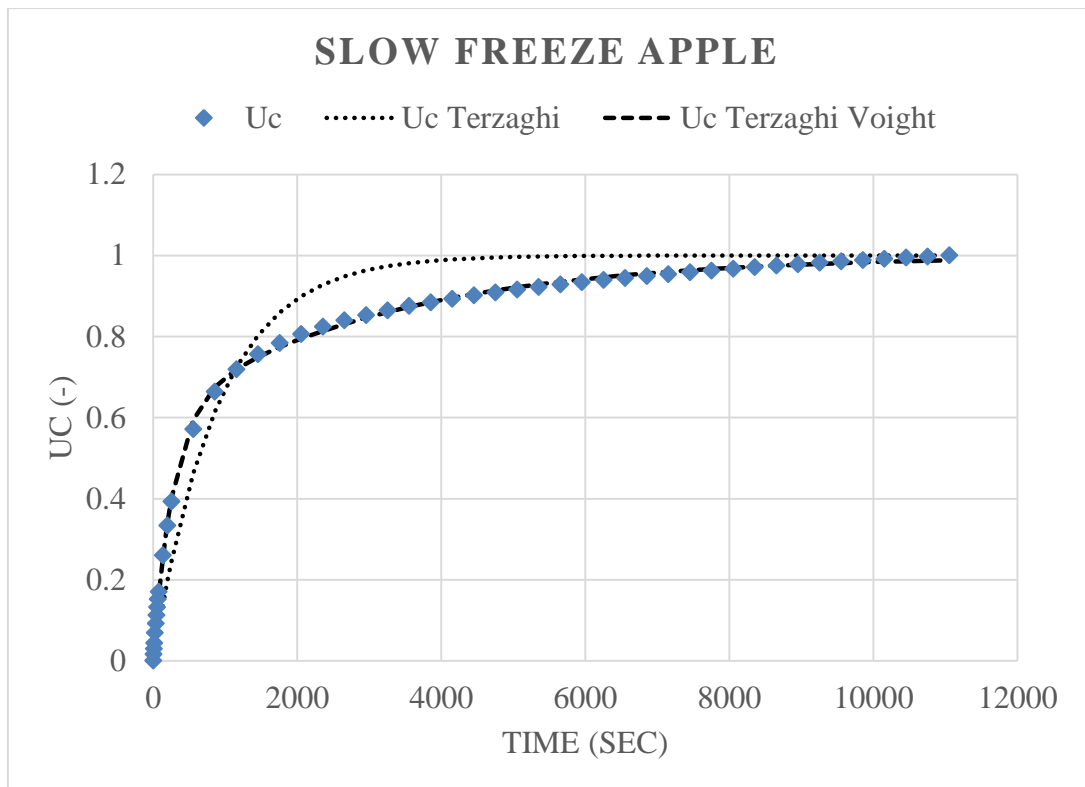


Figure 4. 6 Model fitting of frozen/thawed apple samples by Terzaghi, and Terzaghi-Voigt combined model

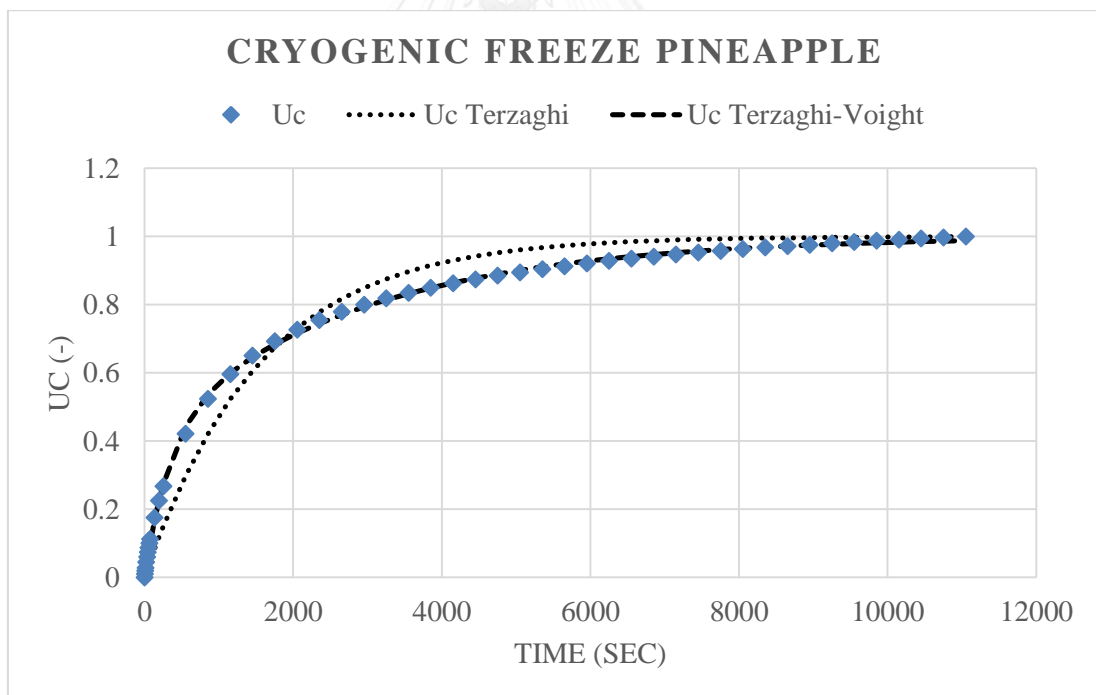
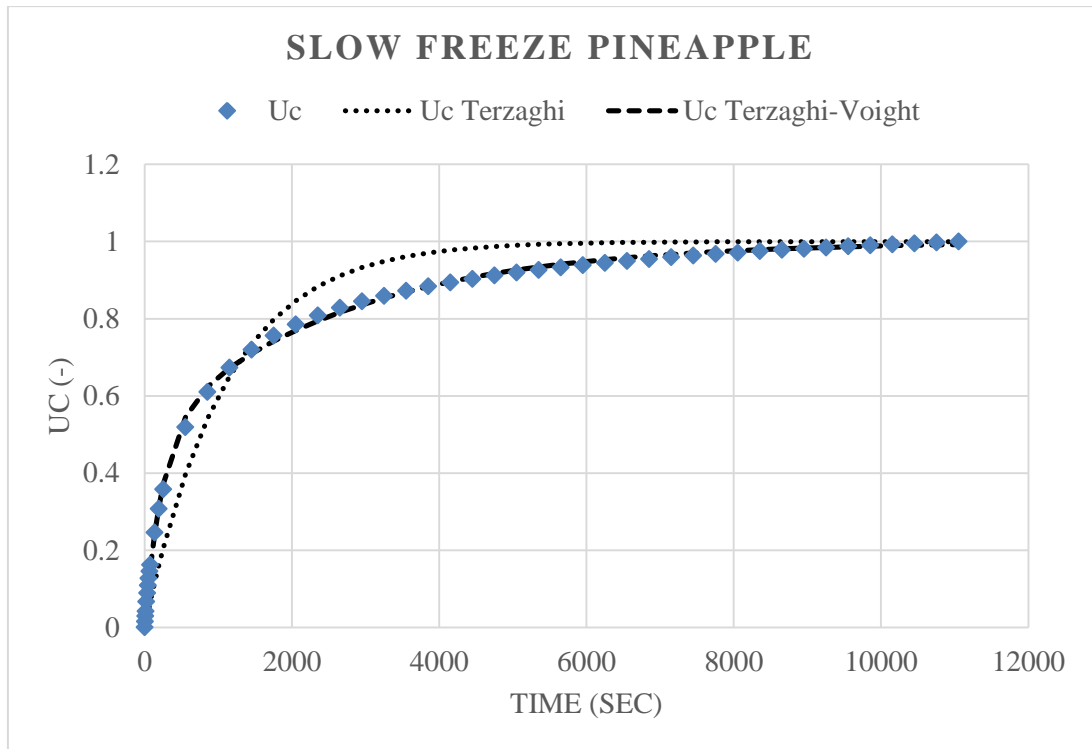


Figure 4. 7 Model fitting of frozen/thawed pineapple samples by Terzaghi and Terzaghi-Voight combined model

Table 4. 6 model fitting on expression behavior of storage apple

Model	Storage	Ce/ω_0^2 (1/s)	B (-)	η (1/s)	Error ²	r^2
Terzaghi	3 weeks	$1.47 \times 10^{-04b} \pm$ 3.2×10^{-06}	-	-	$0.21^{ab} \pm 0.226$	0.97
	6 weeks	$1.44 \times 10^{-04b} \pm$ 7×10^{-07}	-	-	$0.23^b \pm 0.003$	0.96
Terzaghi	9 weeks	$1.1 \times 10^{-04a} \pm$ 3×10^{-07}	-	-	$0.19^a \pm 0.001$	0.97
	3 weeks	$3.96 \times 10^{-04b} \pm$ 1.56×10^{-05}	$0.35^a \pm 9.1 \times 10^{-03}$	$3.46 \times 10^{-04a} \pm$ 7.62×10^{-06}	$2.72 \times 10^{-03b} \pm$ 5.1×10^{-04}	0.99
Voight	6 weeks	$4.16 \times 10^{-04b} \pm$ 1.98×10^{-07}	$0.37^a \pm 4.66 \times 10^{-04}$	$3.41 \times 10^{-04a} \pm$ 3.35×10^{-06}	$2.18 \times 10^{-03ab} \pm$ 8.92×10^{-05}	0.99
	9 weeks	$3.59 \times 10^{-04a} \pm$ 4.68×10^{-07}	$0.42^b \pm 1.67 \times 10^{-03}$	$3.6 \times 10^{-04b} \pm$ 1.3×10^{-06}	$1.75 \times 10^{-03a} \pm$ 1.1×10^{-05}	0.99

Values are mean \pm standard deviation of triplicate sample determinations

Means in the same column with different superscript letters are significantly different ($p \leq 0.05$)

All determinations were performed in triplicate

Table 4. 7 model fitting on expression behavior of storage pineapple

Model	Storage	Ce/ω_0^2 (1/s)	B (-)	η (1/s)	Error ²	r ²
Terzaghi	3 weeks	$9.26 \times 10^{-05a} \pm 0.000$	-	-	$0.21^a \pm 0.000$	0.97
	6 weeks	$9.3 \times 10^{-05a} \pm 3 \times 10^{-07}$	-	-	$0.21^a \pm 0.001$	0.97
	9 weeks	$8.5 \times 10^{-05a} \pm 1.4 \times 10^{-05}$	-	-	$0.27^a \pm 0.092$	0.96
Terzaghi Voight combined	3 weeks	$4.26 \times 10^{-04a} \pm 1 \times 10^{-07}$	$0.50^a \pm 1.23 \times 10^{-04}$	$3.79 \times 10^{-04a} \pm 2 \times 10^{-06}$	$4.28 \times 10^{-03a} \pm 3.7 \times 10^{-05}$	0.99
		$4.27 \times 10^{-04a} \pm 1.9 \times 10^{-07}$	$0.52^a \pm 1.42 \times 10^{-04}$	$3.81 \times 10^{-04a} \pm 2.8 \times 10^{-06}$	$4.26 \times 10^{-03a} \pm 5.49 \times 10^{-05}$	0.99
	6 weeks	$3.98 \times 10^{-04a} \pm 5.01 \times 10^{-05}$	$0.48^a \pm 3.59 \times 10^{-02}$	$3.29 \times 10^{-04a} \pm 8.68 \times 10^{-04}$	$7.49 \times 10^{-03a} \pm 5.56 \times 10^{-03}$	0.99
		9 weeks				

Values are mean \pm standard deviation of triplicate sample determinations

Means in the same column with different superscript letters are significantly different ($p \leq 0.05$)

All determinations were performed in triplicate

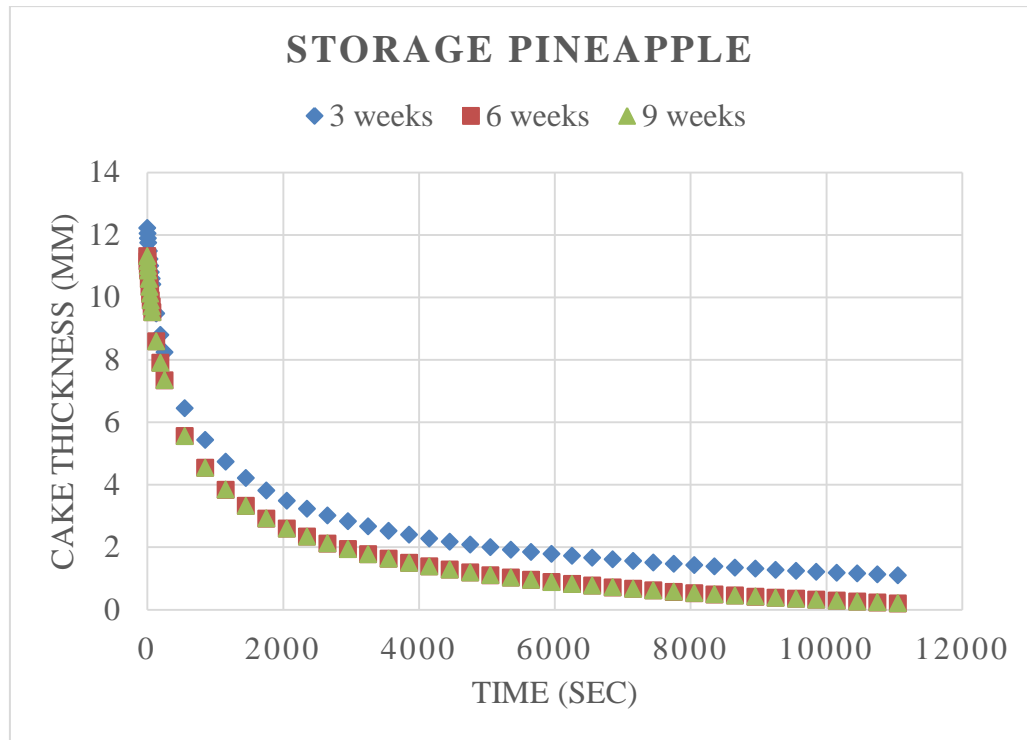


Figure 4. 8 cake thickness versus expression time of stored pineapples

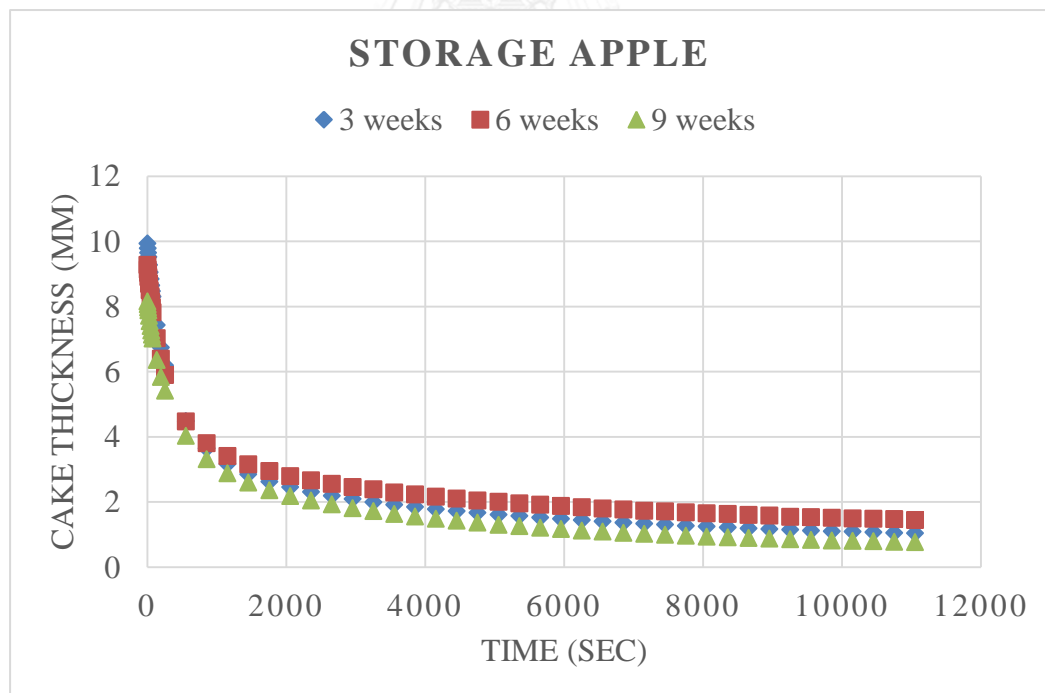


Figure 4. 9 cake thickness versus expression time of stored apples

Figure 4.8 and 4.9 shown that experiments of cake thickness over expression time of 3 hours of stored pineapple and apple samples with 3 different storage time: 3,

6, and 9 weeks. Among 3 different storage time, there were significant differences between storage times. It can be describe that 3 weeks of storage duration was enough according to the graph.

The results of model fitting by Terzaghi and Terzaghi-Voight combined model were shown in Tables 4.6 and 4.7 for stored apple and pineapple samples. Terzaghi model (dotted line) was failed to apply the expression mechanisms of stored pineapple and apple samples as shown in figure 4.10 and 4.11 where the graph plotted between the U_c and expression of time. Terzaghi-Voight combined model (dash line) was being able to fit better than Terzaghi model in stored apple and pineapple.

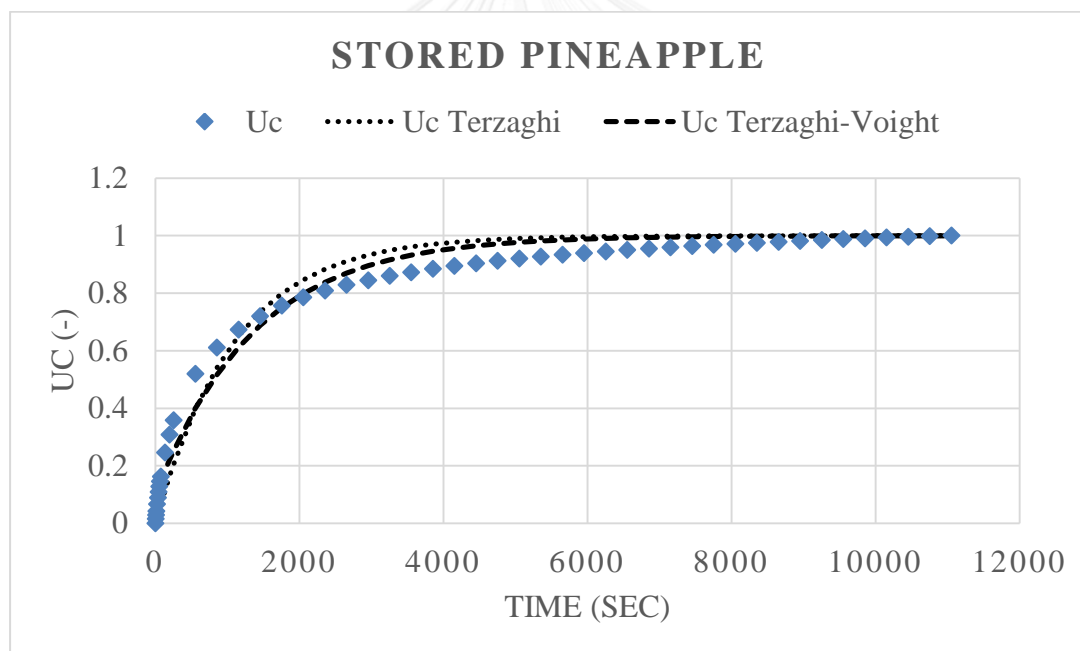


Figure 4. 10 Model fitting of stored pineapple sample by Terzaghi and Terzaghi-Voight combined model

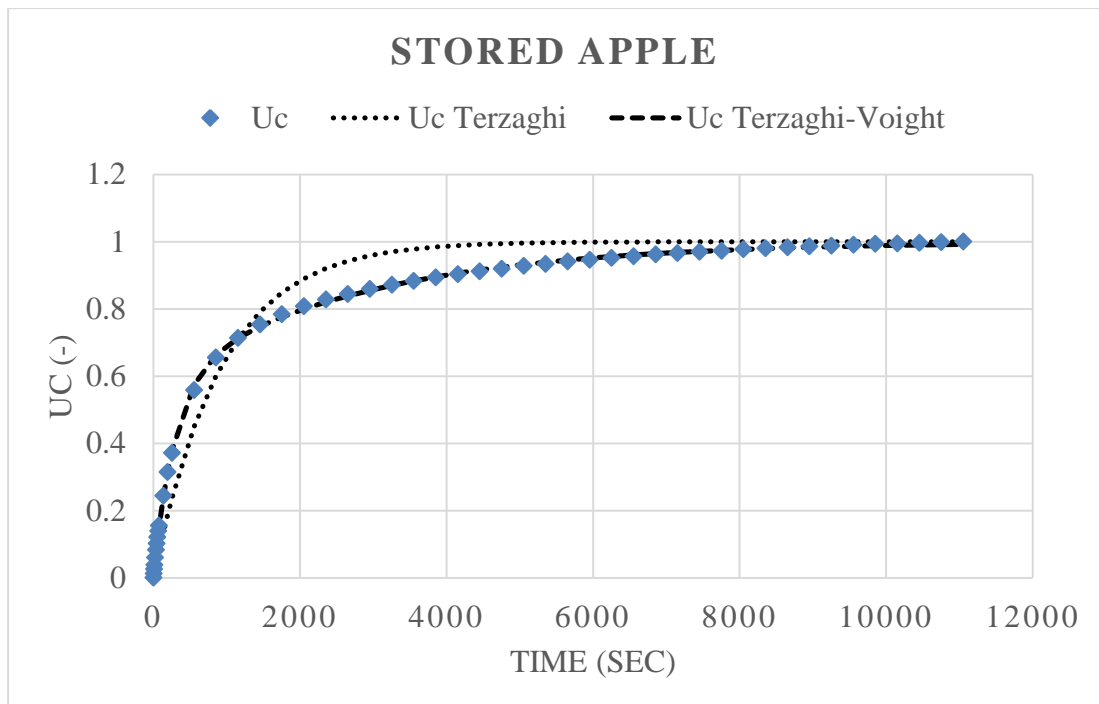


Figure 4. 11 Model fitting of stored apple sample by Terzaghi and Terzaghi-Voight combined model

As shown in Tables 4.6 and 4.7, the least values of error and highest values of r^2 , C_e and η were used to find. The B value was recorded minimum of 35% and up to 42% for apple and minimum of 47% and up to 52% for pineapple respectively. The highest r^2 values were observed in Terzaghi-Voight combined model and least error values were observed in Terzaghi-Voight combined model of stored apple and pineapple.

4.3 Model fitting by empirical equation

The purpose of using this method was to proof that the expression data of control apple and pineapple were did not reach to the equilibrium state after 3 hours of expression time. We used this empirical equation to fit the change in cake thickness

at 3 hours. In empirical Equation (3.2), A represented the maximum ratio of change in cake thickness and B is a constant. The value of A used to indicate the maximum ratio of change in cake thickness reach after 3 hours expression time. As shown in Table 4.6 and Appendix B.31-38 ($p \geq 0.05$), control pineapple reached 59% while slow and cryogenic freezing pre-treated samples reached 89% and 88% respectively. Control sample of apple was reach only 23% while slow and cryogenic freezing pre-treated samples reached 84% and 99% respectively. According to the data, it was clearly seen that expression of frozen/thawed samples were assumed to reach to equilibrium state while control sample did not. To prove this fact the permeability test was conducted. The permeability test was carried out by measuring the rate of water permeated through the obtained cake in chamber after expression test was done. The water from the water reservoir mounted 1 meter high above the chamber was fed through the out let as shown in Figure 4.12.

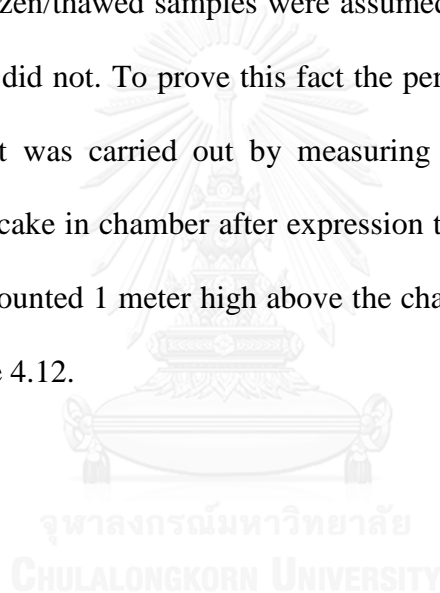


Table 4. 8 fitting of normalized cake thickness of pineapple and apple

Types	Pre-treatment t (°C)	<i>a</i>	<i>b</i>	Error ²	r ²
Pineapple	Control	0.59 ^a ±0.0 3	153.04 ^a ±9.17	0.091 ^{bc} ±0.0 1	0.9687
	-18°C	0.89 ^b ±0.1	694.62 ^c ±181.7 9	0.047 ^{ab} ±0.0 1	0.9923
	-90°C	0.88 ^b ±0.0 4	323.06 ^b ±83.66	0.074 ^{abc} ±0.0 1	0.9871
Apple	control	0.23 ^a ±0.0 1	136.09 ^a ±22.13	0.024 ^a ±0.00 1	0.9232
	-18°C	0.84 ^b ±0.0 5	297.43 ^b ±36.92	0.033 ^a ±0.01	0.9936
	-90°C	0.99 ^c ±0.0 3	760.84 ^c ±193.2 6	0.052 ^a ±0.01 5	0.9931

Values are mean ± standard deviation of triplicate sample determinations

Means in the same column in same fruits with different superscript letters are significantly different ($p \leq 0.05$).

All determinations were performed in triplicate

Table 4. 9 fitting of normalized cake thickness of storage pineapple and apple

Types	Storage	<i>a</i>	<i>b</i>	Error ²	r ²
Pineapple	3 weeks	0.89 ^a ±	232 ^a ±	8.39x10 ^{-02a} ±	0.98
		7.27x10 ⁻⁰²	6.25	1.69x10 ⁻⁰²	
	6 weeks	0.98 ^a ±	234 ^a ±	9.79x10 ^{-02a} ±	
		4.61x10 ⁻⁰²	7.22	1.05x10 ⁻⁰²	
	9 weeks	0.92 ^a ±	233 ^a ±	9.3x10 ^{-02a} ±	
		3.98x10 ⁻⁰²	4.14	6.4x10 ⁻⁰³	
Apple	3 weeks	0.88 ^a ±	225 ^a ±	2.53x10 ^{-02a} ±	0.99
		3.35x10 ⁻⁰²	8.77	4.19x10 ⁻⁰³	
	6 weeks	0.86 ^a ±	231 ^a ±	2.33x10 ^{-02a} ±	
		9.27x10 ⁻⁰³	3.53	1.79x10 ⁻⁰³	
	9 weeks	0.82 ^a ±	322 ^b ±	2.18x10 ^{-02a} ±	
		3.8x10 ⁻⁰²	1.77	2.39x10 ⁻⁰³	

Values are mean ± standard deviation of triplicate sample determinations

Means in the same column in same fruits with different superscript letters are significantly different ($p \leq 0.05$).

All determinations were performed in triplicate

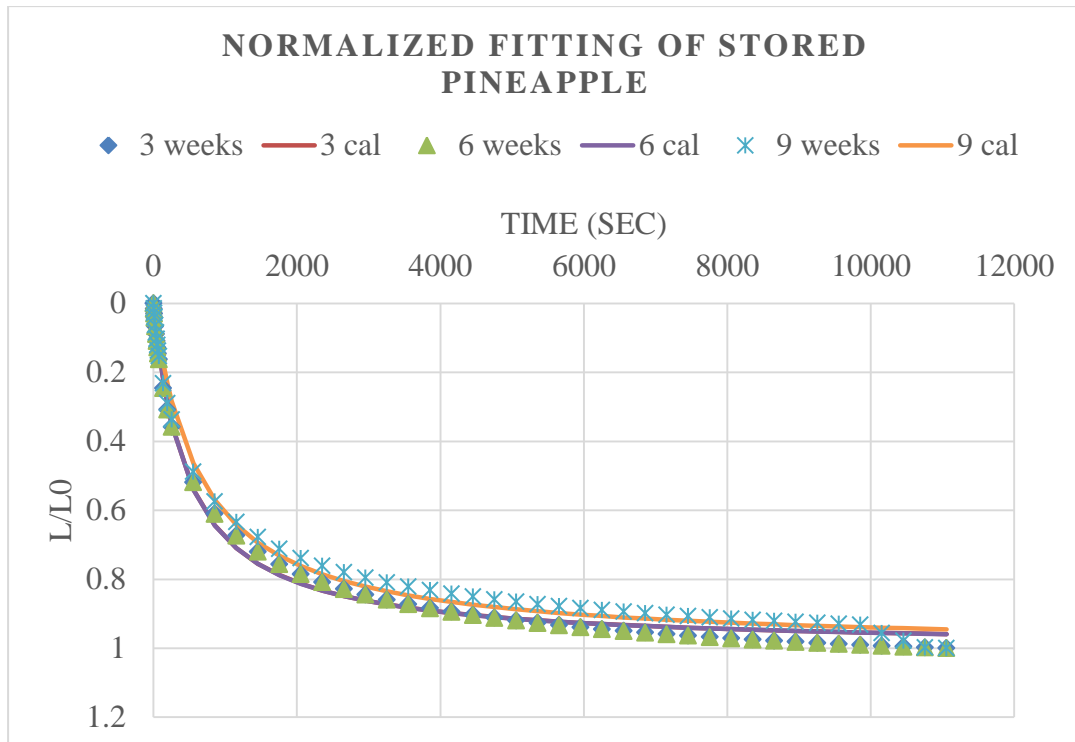


Figure 4. 12 normalized fitting of stored pineapple

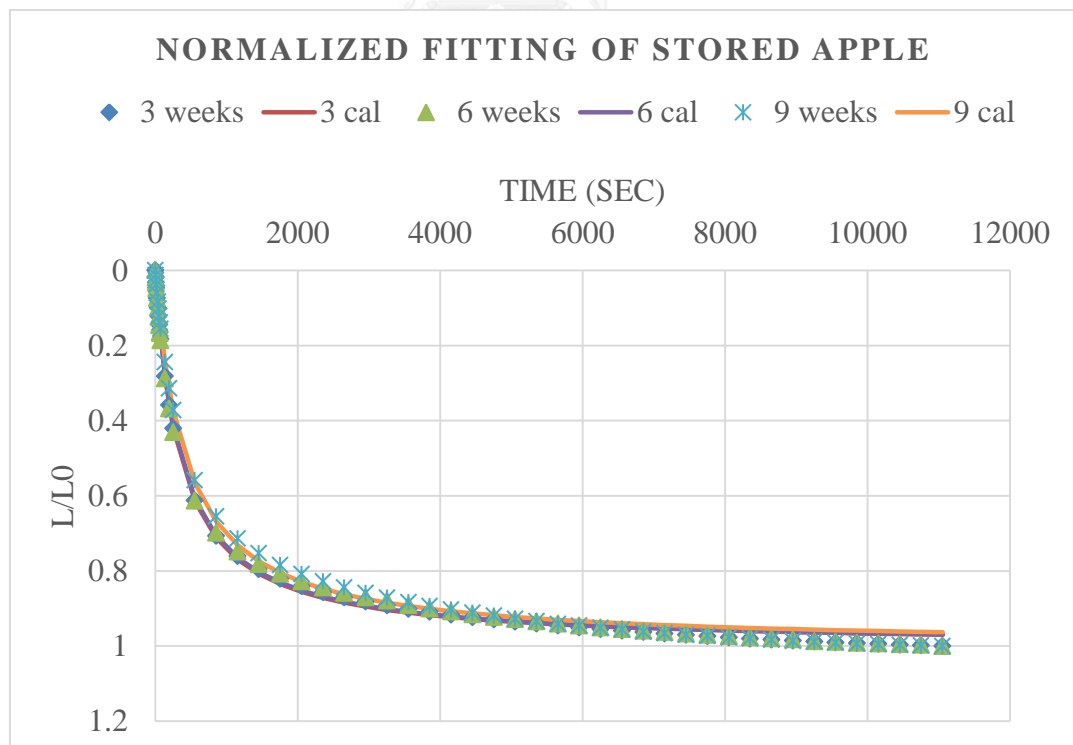
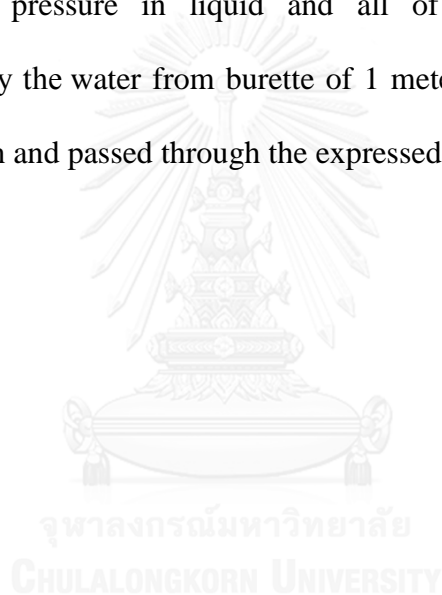


Figure 4. 13 normalized fitting of stored apple

The empirical equation is suitable for fitting the relation which change from 0 to 100%. Permeability test was used to check the cake whether it reach to equilibrium or not as shown in Figure 4.12 and Appendix B.4-9. The test is carried out after certain time of expression hose was connect to the outlet of filtrate and let the water goes back into the expression chamber through the expression cake from burette which has water level 1 meter high above the cake chamber. If the expressed was reach to equilibrium stage, there must be zero in liquid pressure inside the cake which means there is no pressure in liquid and all of pressure was received by solid inside that's why the water from burette of 1 meter higher from the expression machine will go down and passed through the expressed cake.



Permeability Test

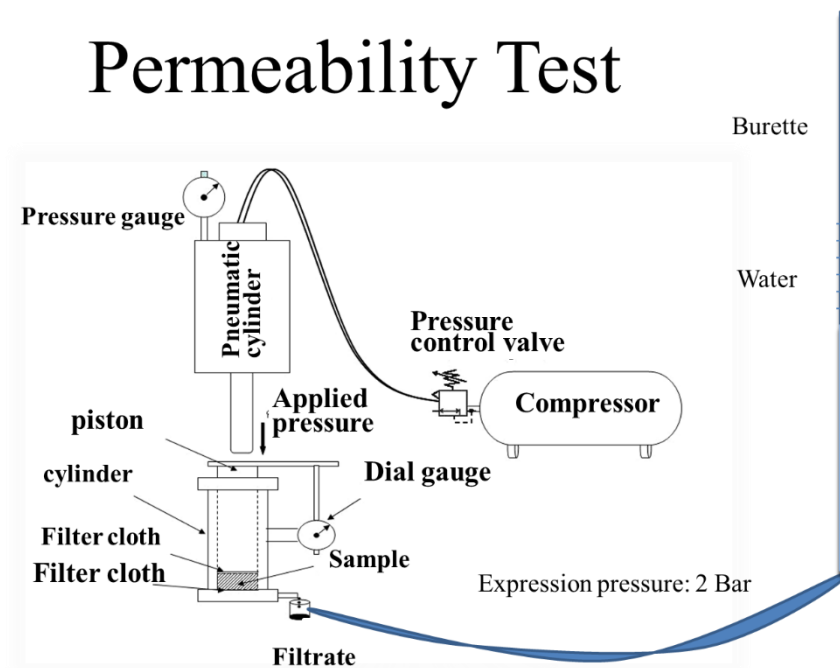


Figure 4. 14 Schematic picture of permeability test

4.4 Properties of juice

4.4.1 pH

The effect of pre-treatments and storage time on pH of pineapple and apple juice samples are shown in Tables 4.10 and 4.11 ($P \leq 0.05$), and analysis of variances are shown in Appendix C.1-C.4.

Significant differences were found out between control, slow and cryogenic freezing pre-treated samples of both apple and pineapple juice. After freezing and thawing, pH value of juices increasing while the value of acidity of juices did not change significantly and it was in good agreement with previous study showing the pH of apple and mangoes increasing after freezing and thawing (Chassagne-Berces 2010).

4.4.2 Acidity

The effect of pre-treatments and storage time on acidity of pineapple and apple juice samples are shown in Tables 4.10 and 4.11 ($P \leq 0.05$), and analysis of variances are shown in Appendix C.5-C.6.

As the results shown, the acidity of apple and pineapple did not differ significantly by the effect of pre-treatment. But higher acidity results were observed during longer storage time but no significant differences were found between 3, 6, and 9 weeks of storage. (Paull 1990) described that the lower storage temperature and a longer storage period may induce acid accumulation.

4.4.3 Soluble solid

The effect of pre-treatments and storage time on soluble solid of pineapple and apple juice samples are shown in Tables 4.10 and 4.11 ($p \leq 0.05$), and analysis of variances are shown in Appendix C.7-C.10.

Soluble solid content of control apple was lower than the frozen/thawed samples while control sample of pineapple had higher soluble solid content than the frozen/thawed samples. It was clearly found out that soluble solid content of frozen/thawed sample was higher than the control sample in apple and these results were in good agreement with previous study showed that soluble solid contents were higher after freezing and thawing (Chassagne-Berces 2010). Because of the cell wall degradation due to ice crystal formation can induce soluble solid extraction (especially sugars) from cells. Indeed, slow freezing rates are well known to produce fewer ice crystals but of larger size which may degrade the cell structure of the product (Fennema 1975) and thus facilitate the extraction of sugars.

4.4.4 Sugar Acid Ratio

The effect of pre-treatments and storage time on sugar acid ratio of pineapple and apple juice samples are shown in Tables 4.10 and 4.11 ($P \leq 0.05$), and analysis of variances are shown in Appendix C.11-C-14.

The sugar acid ratio of frozen/thawed samples and stored samples were found out that significantly higher than the control samples in both apple and pineapple. The results found out that sugar acid ratio of pineapple have 1.89×10^3 in slow freezing pre-treated juice sample and 1.95×10^3 in cryogenic freezing pre-treated juice sample respectively when compare to control samples which had only 1.05×10^3 . For the apple samples, 1.93×10^3 in slow freezing pre-treated juice sample and 1.99×10^3 in cryogenic freezing pre-treated juice sample respectively when compare to control samples which had only 1.71×10^3 . Moreover, the value of sugar acid ratio of stored apple and pineapple fruits juice obtained from expression experiments were significantly lower by storage time. It is because of soluble solid extraction by freezing effect and the data were in good agreement with previous study (Hui 2006).

4.4.5 Moisture content

The effect of pre-treatments and storage time on moisture content of pineapple and apple juice samples are shown in Tables 4.10 and 4.11 ($P \leq 0.05$), and analysis of variances are shown in Appendix C.15-C.18.

Moisture content of control samples of apple and pineapple was significantly higher than the frozen/thawed samples. The noticeable result of low moisture content was observed in stored samples of apple and pineapple compared to 0 week samples. It was clearly observed that frozen/thawed samples has lower moisture content after

expression because freezing and thawing facilitate the extraction of liquid inside the cell by breakdown of cell wall.

4.4.6 Vitamin C

The effect of pre-treatments and storage time on vitamin C of pineapple and apple juice samples are shown in Tables 4.10 and 4.11 ($P \leq 0.05$), and analysis of variances are shown in Appendix C.19-C.22.

Vitamin C is highly degradable when it exposed to light, oxygen and temperature. The vitamin C was barely present with not more than 0.1 mg/100ml in apple and pineapple juices. It was clearly observed on both apple and pineapples samples. It can be destroyed by during steps of preparation (e.g. chopping) and expression experiments which lasted for 3 hours at room temperature of $28 \pm 2^\circ\text{C}$.

Table 4. 10 Effects of pre-treatments on properties of juice of apple and pineapple

Types of fruit	Pre-treatment (°C)	pH	Vitamin C (mg/100ml)	Acidity (citric acid g/100ml)	Soluble solid (Degree Brix)	Sugar Acid Ratio	Moisture content
Pineapple	Control	3.80 ^a ± 0.01	0.076 ^c ± 0.001	0.013 ^b ± 0.000	13.41 ^c ± 0.02	1.05x10 ^{3a} ± 1.50	73.95 ^c ± 1.52
	-18 °C	3.94 ^b ± 0.01	0.059 ^b ± 0.001	0.006 ^a ± 0.000	12.07 ^a ± 0.06	1.89x10 ^{3b} ± 9.02	52.84 ^a ± 2.76
	-90 °C	3.92 ^b ± 0.01	0.05 ^a ± 0.002	0.006 ^a ± 0.000	12.48 ^b ± 0.11	1.95x10 ^{3c} ± 16.74	61.57 ^b ± 2.12
apple	control	3.74 ^b ± 0.02	0.014 ^b ± 0.001	0.0064 ^a ± 0.000	10.93 ^a ± 0.06	1.71x10 ^{3a} ± 9.02	81.34 ^b ± 1.29
	-18 °C	3.72 ^a ± 0.01	0.008 ^a ± 0.000	0.0064 ^a ± 0.000	12.37 ^b ± 0.06	1.93x10 ^{3b} ± 9.02	54.64 ^a ± 4.2
	-90 °C	3.87 ^c ± 0.01	0.017 ^c ± 0.002	0.0064 ^a ± 0.000	12.73 ^c ± 0.12	1.99x10 ^{3c} ± 18.04	52.76 ^a ± 0.93

Values are mean ± standard deviation of triplicate sample determinations

Means in the same column in same fruits with different superscript letters are significantly different ($p \leq 0.05$)

All determinations were performed in triplicate

Table 4. 11 Effects of storage time on properties of apple and pineapple juice

Types of fruit	Storage Time (week)	pH	Vitamin C (mg/100ml)	Acidity (citric acid g/100ml)	Soluble solid (Degree Brix)	Sugar Acid Ratio	Moisture content
Pineapple	3	3.57 ^a ±0.006	0.037 ^b ±0.002	0.0256 ^a ±0.000	14.13 ^a ±0.12	5.521x10 ^{2a} ±4.51	48.2 ^a ±2.15
	6	3.58 ^a ±0.012	0.035 ^{ab} ±0.002	0.0256 ^a ±0.000	14.4 ^b ±0.00	5.625x10 ^{2b} ±0.00	45.2 ^a ±3.29
	9	3.57 ^a ±0.006	0.032 ^a ±0.00	0.0256 ^a ±0.000	14.8 ^c ±0.00	5.781x10 ^{2c} ±0.00	46.4 ^a ±1.57
Apple	3	3.54 ^a ±0.00	0.0173 ^b ±0.02	0.0128 ^a ±0.000	12.07 ^a ±0.06	9.427x10 ^{2a} ±4.51	47.5 ^a ±1.71
	6	3.57 ^b ±0.00	0.005 ^a ±0.002	0.0128 ^a ±0.000	12.1 ^a ±0.00	9.453x10 ^{2a} ±0.00	48.3 ^a ±4.35
	9	3.57 ^b ±0.00	0.004 ^a ±0.00	0.0128 ^a ±0.000	12.2 ^b ±0.00	9.531x10 ^{2b} ±0.00	46.5 ^a ±0.86

Values are mean ± standard deviation of triplicate sample determinations

Means in the same column in same fruits with different superscript letters are significantly different ($p \leq 0.05$).

All determinations were performed in triplicate.

CHAPTER V

CONCLUSIONS AND SUGGESSTIONS

CONSLUSIONS

The expression behavior of fruits containing soluble and insoluble fibers were significantly affected by freezing pre-treatment. Slow freezing pre-treatment has greater effect than the cryogenic freezing pre-treatment. Storage time had no noticeable effect on expression behavior of both apple and pineapple samples in terms of graph. According to fruits physical characteristics, pineapple fruits were easier to squeeze than apple fruits with or without pre-treatment. In apple, Terzaghi model can fit as Terzaghi-Voight combined model in cryogenic frozen/thawed sample.

Terzaghi model was failed to fit the expression mechanisms of frozen/thawed apple and pineapple samples while the Terzaghi-Voight combined model can fit the expression mechanisms successfully at ($r^2 \geq 0.99$).

Since, vitamin C of obtained apple and pineapple juices is highly degradable component and barely present in juice of apple and pineapple after expression. Soluble solid contents were significantly higher in juices of freezing/thawing pre-treatment and slow freezing had greater effect than the cryogenic freezing pre-treatment in apple. There was a significant reduce in moisture content of final cake after expression in frozen/thawed samples of apple and pineapple, and significant increase in sugar acid ratio of frozen/thawed samples of both fruits.

Acidity, soluble solid and sugar acid ratio were extensively higher after longer storage time at low temperature for apple and pineapple samples. But the moisture content of the cake after expression was reduced in stored samples.

SUGGESSTIONS

In order to see a distinguishable effect of expression behavior between control samples and frozen/thawed samples, longer expression time is needed to be observe and in the meanwhile the properties of juice should be separately determined to investigate the effect of freezing pre-treatments on properties of juice. Slow freezing pre-treatment is the best in concern of highest juice yield and faster expression rate. The different method of thawing should be study in future to discover its effect on expression behavior of fruits.



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APPENDIX

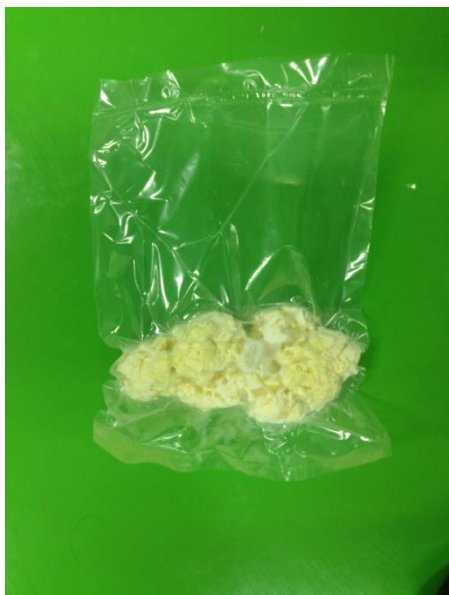


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APPENDIX A**DETAILED INFORMATION ON MATERIALS USED IN THE
EXPERIMENT**

APPENDIX B

ADDITIONAL DATA



Appendix B. 1 Slow freezing pre-treatment condition



Appendix B. 2 Cryogenic freezing pre-treatment condition



(a)



(b)

Appendix B. 3 Differences between final cake thickness of apple and pineapple with and without pre-treatments

APPENDIX C
ANALYSIS OF VARIANCE

Appendix Table C. 1 the effect of pre-treatment on pH of apple

Sum of					
Source	Squares	df	Mean Square	F	Sig.
Treatment	.038	2	.019	314.364	.000
block	.000	2	7.778×10^{-5}	1.273	.373
Error	.000	4	6.111×10^{-5}		
Total	128.332	9			

Appendix Table C. 2 the effect of pre-treatment on pH of pineapple

Sum of					
Source	Squares	df	Mean Square	F	Sig.
Treatment	.038	2	.019	105.812	.000
Block	2.222×10^{-5}	2	1.111×10^{-5}	.062	.940
Error	.001	4	.000		
Total	136.072	9			

Appendix Table C. 3 the effect of storage time on pH of apple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Storage	.002	2	.001	.	.
Error	.000	6	.000		
Total	114.064	9			

Appendix Table C. 4 the effect of storage time on pH of pineapple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Storage	2.222×10^{-5}	2	1.111×10^{-5}	.167	.850
Error	.000	6	6.667×10^{-5}		
Total	114.990	9			

Appendix Table C. 5 the effect of pre-treatment on acidity of pineapple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	8.192×10^{-5}	2	4.096×10^{-5}	.	.
Block	.000	2	.000	.	.
Error	.000	4	.000		
Total	.001	9			

Appendix Table C. 6 the effect of pre-treatment on acidity of apple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	.000	2	.000	.	.
Block	.000	2	.000	.	.
Error	.000	4	.000		
Total	.000	9			

Appendix Table C. 7 the effect of pre-treatment on soluble solid of apple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	5.429	2	2.714	349.000	.000
Block	.009	2	.004	.571	.605
Error	.031	4	.008		
Total	1303.870	9			

Appendix Table C. 8 the effect of pre-treatment on soluble solid of pineapple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	2.844	2	1.422	291.501	.000
Block	.011	2	.006	1.130	.408
Error	.020	4	.005		
Total	1443.330	9			

Appendix Table C. 9 the effects of storage time on soluble solid of apple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	.029	2	.014	13.000	.007
Error	.007	6	.001		
Total	1322.570	9			

Appendix Table C. 10 the effects of storage time on soluble solid of pineapple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	.676	2	.338	76.000	.000
Error	.027	6	.004		
Total	1878.480	9			

Appendix Table C. 11 the effects of pre-treatment on sugar acid ratio of pineapple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	1519264.503	2	759632.252	6.858x10 ³	.000
Block	284.831	2	142.415	1.286	.371
Error	443.070	4	110.768		
Total	2.536x10 ⁷	9			

Appendix Table C. 12 the effects of pre-treatment on sugar acid ratio of apple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	132541.233	2	66270.616	349.000	.000
Block	217.014	2	108.507	.571	.605
Error	759.549	4	189.887		
Total	3.183x10 ⁷	9			

Appendix Table C. 13 the effect of storage time on sugar acid ratio of pineapple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	1030.792	2	515.396	75.988	.000
Error	40.695	6	6.783		
Total	2866334.117	9			

Appendix Table C. 14 The effect of storage time on sugar acid ratio of apple

Source	Sum of		Mean Square	F	Sig.
	Squares	df			
Treatment	176.310	2	88.155	12.997	.007
Error	40.695	6	6.783		
Total	8072331.387	9			

Appendix Table C. 15 the effect of storage time on moisture content of pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	13.398	2	6.699	1.122	.385
Error	35.815	6	5.969		
Total	19586.730	9			

Appendix Table C. 16 The effect of storage time on moisture content of apple

Source	Sum of		Mean		Sig.
	Squares	df	Square	F	
Treatment	4.829	2	2.415	.321	.737
Error	45.121	6	7.520		
Total	20285.961	9			

Appendix Table C. 17 The effect of pre-treatment on moisture content of apple

Source	Sum of		Mean Square	F	Sig.
	Squares	df			
Treatment	1533.038	2	766.519	90.165	.000
Block	6.358	2	3.179	.374	.710
Error	34.005	4	8.501		
Total	37193.672	9			

Appendix Table C. 18 the effect of pre-treatment on moisture content of pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	675.532	2	337.766	47.472	.002
Block	.287	2	.143	.020	.980
Error	28.460	4	7.115		
Total	36183.768	9			

Appendix Table C. 19 The effect of pre-treatment on vitamin C of apple

Source	Sum of		Mean		Sig.
	Squares	df	Square	F	
Treatment	.000	2	6.104×10^{-5}	103.000	.000
Block	3.556×10^{-6}	2	1.778×10^{-6}	3.000	.160
Error	2.370×10^{-6}	4	5.926×10^{-7}		
Total	.002	9			

Appendix Table C. 20 The effect of pre-treatment on vitamin C of pineapple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	.001	2	.001	207.769	.000
Block	1.580x10 ⁻⁶	2	7.901x10 ⁻⁷	.308	.751
Error	1.027x10 ⁻⁵	4	2.568x10 ⁻⁶		
Total	.035	9			

Appendix Table C. 21 The effect of storage time on vitamin C of pineapple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	4.267x10 ⁻⁵	2	2.133x10 ⁻⁵	6.000	.037
Error	2.133x10 ⁻⁵	6	3.556x10 ⁻⁶		
Total	.011	9			

Appendix Table C. 22 The effect of storage time on vitamin C of apple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	.000	2	.000	1.230	.357
Error	.001	6	.000		
Total	.002	9			

Appendix Table C. 23 The effect of pre-treatment on yield of apple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	5615.986	2	2807.993	252.673	.000
Block	11.637	2	5.818	.524	.628
Error	44.453	4	11.113		
Total	47074.830	9			

Appendix Table C. 24 the effect of pre-treatment on yield of pineapple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	625.952	2	312.976	44.574	.002
Block	83.940	2	41.970	5.977	.063
Error	28.086	4	7.022		
Total	53553.678	9			

Appendix Table C. 25 the effect of storage time on yield of apple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	175.270	2	87.635	67.065	.001
Block	6.526	2	3.263	2.497	.198
Error	5.227	4	1.307		
Total	67631.113	9			

Appendix Table C. 26 the effect of storage time on yield of pineapple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	409.825	2	204.912	126.768	.000
Block	3.864	2	1.932	1.195	.392
Error	6.466	4	1.616		
Total	64697.616	9			

Appendix Table C. 27 the effect of pre-treatment on recovery of pineapple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	76.956	2	38.478	1.254	.378
Block	125.468	2	62.734	2.045	.245
Error	122.723	4	30.681		
Total	67402.694	9			

Appendix Table C. 28 The effect of pre-treatment on recovery of apple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	289.762	2	144.881	3.119	.153
Block	70.818	2	35.409	.762	.524
Error	185.814	4	46.454		
Total	68224.417	9			

Appendix Table C. 29 The effect of storage time on recovery of pineapple

Source	Sum of		Mean Square	F	Sig.
	Squares	df			
Treatment	398.632	2	199.316	122.131	.000
Block	6.907	2	3.453	2.116	.236
Error	6.528	4	1.632		
Total	71628.105	9			

Appendix Table C. 30 The effect of storage time on recovery of apple

Source	Sum of		Mean Square	F	Sig.
	Squares	df			
Treatment	191.061	2	95.531	66.408	.001
Block	8.697	2	4.348	3.023	.159
Error	5.754	4	1.439		
Total	74138.748	9			

Appendix Table C. 31 Table of value of a by normalized fitting pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	.184	2	.092	105.818	.000
Block	.002	2	.001	1.330	.361
Error	.003	4	.001		
Total	5.787	9			

Appendix Table C. 32 Table of value of b by normalized fitting pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	460263.388	2	230131.694	22.896	.006
Block	40054.796	2	20027.398	1.993	.251
Error	40203.863	4	10050.966		
Total	1911111.147	9			

Appendix Table C. 33 Table of value of error by normalized fitting pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	.003	2	.002	9.721	.029
Block	.000	2	8.166x10 ⁻⁵	.529	.625
Error	.001	4	.000		
Total	.049	9			

Appendix Table C. 34 Table of value of a by normalized fitting apple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	.996	2	.498	541.081	.000
Block	.003	2	.002	1.673	.297
Error	.004	4	.001		
Total	5.257	9			

Appendix Table C. 35 Table of value of b by normalized fitting apple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	631096.060	2	315548.030	21.314	.007
Block	19184.279	2	9592.140	.648	.571
Error	59219.645	4	14804.911		
Total	2135987.024	9			

Appendix Table C. 36 Table of value of error by normalized fitting apple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	.001	2	.001	3.944	.113
Block	3.767×10^{-5}	2	1.883×10^{-5}	.126	.885
Error	.001	4	.000		
Total	.014	9			

Appendix Table C. 37 Ce/ω_0^2 value of Terzaghi of stored apple

	Sum of				
Source	Squares	df	Mean Square	F	Sig.
Treatment	2.556×10^{-9}	2	1.278×10^{-9}	319.559	.000
Block	.000	2	.000	.000	1.000
Error	1.600×10^{-11}	4	3.999×10^{-12}		
Total	1.625×10^{-7}	9			

Appendix Table C. 38 Table of Error² of Terzaghi of stored apple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	.002	2	.001	6.251	.059
Block	.000	2	.000	.812	.506
Error	.001	4	.000		
Total	.408	9			

Appendix Table C. 39 Table of Ce/ω_0^2 of Terzaghi-Voight combined of stored apple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	5.010×10^{-9}	2	2.505×10^{-9}	30.067	.004
Block	1.551×10^{-10}	2	7.755×10^{-10}	.931	.466
Error	3.333×10^{-10}	4	8.332×10^{-11}		
Total	1.375×10^{-6}	9			

Appendix Table C. 40 Table of η of Terzaghi-Voight combined of stored apple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	6.043×10^{-10}	2	3.022×10^{-10}	10.985	.024
Block	3.210×10^{-11}	2	1.605×10^{-10}	.583	.599
Error	1.100×10^{-10}	4	2.751×10^{-11}		
Total	1.096×10^{-6}	9			

Appendix Table C. 41 Table of error² of Terzaghi-Voight combined of stored apple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	1.428 x10 ⁻⁶	2	7.138 x10 ⁻⁷	8.962	.033
Block	2.160 x10 ⁻⁷	2	1.080 x10 ⁻⁷	1.356	.355
Error	3.186 x10 ⁻⁷	4	7.964 x10 ⁻⁸		
Total	4.614 x10 ⁻⁵	9			

Appendix Table C. 42 Table of Ce/ω_0^2 of Terzaghi of stored pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	1.310 x10 ⁻¹⁰	2	6.548 x10 ⁻¹¹	1.000	.444
Block	1.284 x10 ⁻¹⁰	2	6.421 x10 ⁻¹¹	.981	.450
Error	2.619 x10 ⁻¹⁰	4	6.547 x10 ⁻¹¹		
Total	7.354 x10 ⁻⁸	9			

Appendix Table C. 43 Table of error² of Terzaghi of stored pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	.006	2	.003	1.001	.444
Block	.006	2	.003	.987	.448
Error	.011	4	.003		
Total	.500	9			

Appendix Table C. 44 Table of Ce/ω_0^2 of Terzaghi-Voight combined of stored apple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	1.685×10^{-9}	2	8.423×10^{-10}	.989	.448
Block	1.628×10^{-9}	2	8.142×10^{-10}	.956	.458
Error	3.405×10^{-9}	4	8.513×10^{-10}		
Total	1.571×10^{-6}	9			

Appendix Table C. 45 Table of B of Terzaghi-Voight combined of stored pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	.001	2	.000	.994	.446
Block	.001	2	.000	.964	.455
Error	.002	4	.000		
Total	2.202	9			

Appendix Table C. 46 Table of η of Terzaghi-Voight combined of stored pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	5.069×10^{-9}	2	2.534×10^{-9}	1.000	.444
Block	4.938×10^{-9}	2	2.469×10^{-9}	.975	.452
Error	1.013×10^{-8}	4	2.533×10^{-9}		
Total	1.201×10^{-6}	9			

Appendix Table C. 47 Table of error² of Terzaghi-Voight combined of stored pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	2.070 x10 ⁻⁵	2	1.035 x10 ⁻⁵	1.001	.444
Block	2.052 x10 ⁻⁵	2	1.026 x10 ⁻⁵	.993	.447
Error	4.135 x10 ⁻⁵	4	1.034 x10 ⁻⁵		
Total	.000	9			

Appendix Table C. 48 Table of A value of stored pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	.010	2	.005	1.230	.383
Block	.002	2	.001	.236	.800
Error	.016	4	.004		
Total	7.831	9			

Appendix Table C. 49 Table of B value of stored pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	1.439	2	.719	.042	.960
Block	147.591	2	73.795	4.268	.102
Error	69.159	4	17.290		
Total	490045.334	9			

Appendix Table C. 50 Table of error² value of empirical equation of stored pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	.000	2	.000	.769	.522
Block	8.511x10 ⁻⁵	2	4.256x10 ⁻⁵	.216	.814
Error	.001	4	.000		
Total	.077	9			

Appendix Table C. 51 Table of A value of stored apple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	.001	2	.000	.396	.697
Block	.002	2	.001	.832	.499
Error	.004	4	.001		
Total	6.923	9			

Appendix Table C. 52 Table of B value of stored apple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	18023.616	2	9011.808	207.893	.000
Block	11.773	2	5.887	.136	.877
Error	173.393	4	43.348		
Total	622876.330	9			

Appendix Table C. 53 Table of error² value of empirical equation of stored apple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	1.850x10 ⁻⁵	2	9.249 x10 ⁻⁶	.719	.541
Block	1.609 x10 ⁻⁶	2	8.047 x10 ⁻⁷	.063	.940
Error	5.146 x10 ⁻⁵	4	1.287 x10 ⁻⁵		
Total	.005	9			

Appendix Table C. 54 Table of Ce/ω_0^2 value of Terzaghi of apple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	5.437 x10 ⁻⁹	2	2.718 x10 ⁻⁹	10.253	.027
Block	5.208 x10 ⁻¹¹	2	2.604 x10 ⁻¹¹	.098	.909
Error	1.061 x10 ⁻⁹	4	2.651 x10 ⁻¹⁰		
Total	5.043 x10 ⁻⁸	9			

Appendix Table C. 55 Table of error² value of Terzaghi of apple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	.155	2	.077	838.463	.000
Block	.005	2	.002	26.086	.005
Error	.000	4	9.240 x10 ⁻⁵		
Total	.578	9			

Appendix Table C. 56 Table of Ce/ω_0^2 value of Terzaghi-Voight combined model of apple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	2.200×10^{-7}	2	1.100×10^{-7}	2.586	.190
Block	7.411×10^{-8}	2	3.705×10^{-8}	.871	.485
Error	1.702×10^{-7}	4	4.254×10^{-8}		
Total	3.854×10^{-6}	9			

Appendix Table C. 57 Table of B value of Terzaghi-Voight combined model of apple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	.238	2	.119	15.235	.013
Block	.006	2	.003	.385	.703
Error	.031	4	.008		
Total	3.994	9			

Appendix Table C. 58 Table of η value of Terzaghi-Voight combined model of apple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	1.211×10^{-7}	2	6.053×10^{-8}	221.210	.000
Block	6.608×10^{-10}	2	3.304×10^{-10}	1.208	.389
Error	1.094×10^{-9}	4	2.736×10^{-10}		
Total	1.330×10^{-6}	9			

Appendix Table C. 59 Table of error² value of Terzaghi-Voight combined model of apple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	.000	2	5.611 x10 ⁻⁵	28.754	.004
Block	7.879 x10 ⁻⁶	2	3.939 x10 ⁻⁶	2.019	.248
Error	7.806 x10 ⁻⁶	4	1.951 x10 ⁻⁶		
Total	.000	9			

Appendix Table C. 60 Table of Ce/ω_0^2 value of Terzaghi of pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	1.059 x10 ⁻⁹	2	5.297 x10 ⁻¹⁰	9.374	.031
Block	1.287 x10 ⁻¹⁰	2	6.435 x10 ⁻¹¹	1.139	.406
Error	2.260 x10 ⁻¹⁰	4	5.651 x10 ⁻¹¹		
Total	4.055 x10 ⁻⁸	9			

Appendix Table C. 61 Table of error² value of Terzaghi of pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	.156	2	.078	150.553	.000
Block	.000	2	.000	.339	.731
Error	.002	4	.001		
Total	.699	9			

Appendix Table C. 62 Table of Ce/ω_0^2 value of Terzaghi-Voight combined model of pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	2.051×10^{-7}	2	1.026×10^{-7}	609.329	.000
Block	3.800×10^{-11}	2	1.900×10^{-11}	.113	.896
Error	6.733×10^{-10}	4	1.683×10^{-10}		
Total	2.132×10^{-6}	9			

Appendix Table C. 63 Table of B value of Terzaghi-Voight combined model of pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	.013	2	.006	3.662	.125
Block	.005	2	.002	1.380	.350
Error	.007	4	.002		
Total	3.116	9			

Appendix Table C. 64 Table of η value of Terzaghi-Voight combined model of pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	1.660×10^{-8}	2	8.301×10^{-9}	100.691	.000
Block	2.389×10^{-10}	2	1.194×10^{-10}	1.449	.336
Error	3.298×10^{-10}	4	8.244×10^{-11}		
Total	1.038×10^{-6}	9			

Appendix Table C. 65 Table of $error^2$ value of Terzaghi-Voight combined model of pineapple

Source	Sum of Squares	df	Mean Square	F	Sig.
Treatment	4.501×10^{-5}	2	2.250×10^{-5}	43.551	.002
Block	3.032×10^{-6}	2	1.516×10^{-6}	2.934	.164
Error	2.067×10^{-6}	4	5.167×10^{-7}		
Total	.000	9			



VITA

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