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Development and application of an automatic system for determining seed volume kinetics during soaking

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To the Graduate Council:

I am submitting herewith a thesis written by Shan Xu entitled "Development and application of an automatic system for determining seed volume kinetics during soaking." I have examined the final electronic copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Food Science and Technology.

Federico M. Harte, Major Professor

We have read this thesis and recommend its acceptance:

John Mount, John Wilkerson, Paul Angelino

Accepted for the Council:
Dixie L. Thompson

Vice Provost and Dean of the Graduate School

(Original signatures are on file with official student records.)

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Accepted for the Council:

Carolyn R. Hodges
Vice Provost and Dean of the Graduate School

DEVELOPMENT AND APPLICATION OF AN AUTOMATIC SYSTEM FOR DETERMINING SEED VOLUME KINETICS DURING SOAKING

A Thesis Presented for the
Master of Science
Degree
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Shan Xu
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ABSTRACT

Soaking is an important unit operation during the processing of seeds used for direct consumption. The change in seed volume over time during soaking (volume kinetics) relates to water uptake and the quality of final product, and affects the design of the entire processing operation. Since volume determination is labor-intensive and time consuming, volume kinetics is usually not well monitored throughout seed hydration. The first chapter of this thesis is a review on the importance of soaking and volume kinetics monitoring during seed processing, the factors affecting hydration in seeds, current volume measurement methods and models used to determine and describe the change in volume over time in seeds during soaking. The second chapter describes the design, construction and evaluation of a bean volumetric auto tester (B-VAT) for volume kinetics determination of seeds during soaking. Evaluation tests suggested the system can generate reliable, reproducible, and detailed volume kinetics results for seeds soaking at different conditions with limited labor requirements. In the third chapter, the volume kinetics of 6 pinto, 5 navy and 3 black bean cultivars were tested during soaking at 25 ºC and 55 ºC. Significant differences were observed among varieties and cultivars at both temperatures ($p<0.01$). As temperature increased, the hydration efficiency was enhanced for all cultivars, but to a various degree. In the fourth chapter, we tested the hypothesis that a thin hydrophobic layer on the seed coat was responsible of the extended initial lag phase observed during the soaking of pinto beans. Hexane pre-

treatment before soaking was used for all cultivars and contact angle measurement were done to determine the surface hydrophobicity of the beans. Good correlations were found between surface hydrophobicity and hydration efficiency of beans. Hexane effectively reduced the hydrophobicity of bean surface and improved the hydration efficiency of pinto beans. The fifth chapter covers the overall conclusion of this study and states recommendations of future work regarding the improvement of the developed system and further exploration of the bean hydration process.

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CHAPTER I
INTRODUCTION AND LITERATURE REVIEW

Food Legume

A legume is a plant in the Fabaceae family (or *Leguminosae*), or a fruit of Fabaceae plants such as alfalfa, clover, beans, peas, lupins, lentils, soy and peanuts. Legume seeds and foliage are richer in protein than other non-legume plant materials, due to the additional nitrogen that forms via nitrogen-fixation symbiosis. Iraq, Algeria, Morocco, and China are the major Leguminous food producing countries, each with more than 120,000 million metric tons production per year (FAOSTAT, 2007).

Grain legumes, including beans, lentils, lupins, peas, and peanuts (Kurlovich and Repyev, 1995) are usually cultivated for their seeds that are widely used as a major food source for human and animals. Rich in protein (25%-35% on average), minerals (especially calcium, potassium, iron, zinc and magnesium), vitamins (especially thiamine, riboflavin, and niacin) and dietary fiber, legume seeds are commonly produced and consumed in developing countries as a substitute source for animal protein to bridge the gap of protein insufficiency and reduce cholesterol intake (Salunkhe et al., 1985).

The common bean, *Phaseolus vulgaris*, is native to the tropical areas of South and Central America (Haytowitz and Ruth, 1986). In 2007, there were 18.3 million tons of dry common beans grown worldwide, with Brazil and India being the leading producers (FAOSTAT, 2007). The world production of dry beans and other major legume seeds in top producing countries in 2007 are listed in Table 1.1. (Note: all tables and figures are listed in the appendices)

The common beans are also the most consumed legume species, especially in Mexican and Brazilian diets where they are the primary source of protein (Broughton et al., 2003). The major varieties of common beans are navy, pinto, red kidney, black, pink, and French. Navy and pinto beans are two of the most produced varieties, constituting approximate 33% and 30% of the worldwide production of common beans, respectively (Ruth, 1989). In U.S. and Mexico, navy and pinto beans are commonly used in homemade recipes and industrial processed products such as canned foods.

Seed Hydration

Significance of hydration

Hydration is an integral unit operation which enables volume and texture recovery of dehydrated foods, and in some cases brings about more benefits such as saving cooking time and energy (Clemente et al., 1998; Frias et al., 2000). There are three simultaneous steps in the hydration process: 1) the water uptake into dry food matrix, 2) the expansion in volume, and 3) the leaching of soluble solids (Jiang and Zhang, 2005; Khazaei and Mohammadi, 2009; Lee et al., 2006; Lewicki, 1998). In processing of seeds, including soybeans, beans, lentils, peas, rice, corn and barley, it is very common to dry the seeds after harvest in order to retain the maximum quality of the grain and suppress the growth of bacteria and fungi during storage, therefore rehydration of seeds is required before cooking or other major unit operations

in industrial processing. In Figure 1.1, the manufacturing processes of common seed-based food products where hydration is incorporated are described (Salunkhe et al., 1985). In these cases, hydration, also called soaking, reduces the required cooking time (Molina et al., 1975) due to the even distribution of water inside the beans before cooking, leading to a better texture of the final product and increased nutrition value by 1) leaching the antinutrients such as tannins, phytic acid, some oligosaccharides, and trypsin inhibitors (Lestienne et al., 2005); and 2) shortening the cooking time where most nutrient degradation occurs. The effect of soaking at room temperature on reduction of antinutrient factors in lentil seeds are shown in Table 1.2 (Abousamaha et al., 1985). As soaking time increased, the antinutritional factors of lentil seeds were reduced bya greater amount, especially for tannins. Egounlety and Aworh (Egounlety and Aworh, 2003) found that soaking at room temperature for a period of 12 hr can reduce the amount of oligosacharrides in soybean, cowpea and groundbean that relate to the flatulence by 17 – 35%.

Practical examples of hydration in seed processing include the soaking prior to milling for soybeans when performing oil extraction, the soaking prior to cooking for many common beans and cereals, and the soaking in preparing many tradition bean-based foods such as miso, natto, soy-bulgur, and tempeh. (Bayram et al., 2004; Johnson et al., 2008).

Factors affecting hydration

Hydration under desired condition improves final food quality attributes such as nutritional value, volume recovery and texture improvement. It usually requires between 6 to 48 hours at room temperature depending on type of legume seed. Legumes like mung bean need shorter time (about 6 hr), whereas soybeans take prolonged periods (24 to 48 hr) (Salunkhe et al., 1985). Researchers have studied many factors that affect the hydration efficiency and the final product quality including intrinsic factors i.e. seed chemical composition, seed size and extrinsic factors as summarized and listed in Table 1.3.

Temperature

Temperature is the primary factor affecting the hydration rate and the equilibrium state. Based on Fick's diffusion Law, the diffusivity increases as the temperature increases, leading to a faster water imbibition rate. The dependence of diffusivity on temperature has been described by the Arrhenius Equation as shown in Eq. (1):

$$D_e = D_0 e^{(-\frac{E_a}{RT})} \qquad (1)$$

Where D_e is the diffusion coefficient (m^2/s), D_0 is a pre-exponential factor (m^2/s), E_a is the activation energy of diffusion (J), R is the universal gas constant 8.314kJ/ml K and T is the absolute temperature. Maldonado et al., (Maldonado et al., 2010) suggested that diffusion is the dominant mechanism

in the rehydration process of dehydrated mangoes. The same conclusion was drawed in studies of Peleg (Peleg, 1988b), Abu-Ghannam (Abu-Ghannam, 1998b), Turhan and coworkers (Turhan et al., 2002) and Muramatsu and coworkers (Muramatsu et al., 2006) on soybean, red bean, chickpea and brown rice, respectively. In Figure 1.2, the water absorption curve of chickpea at ambient temperature is shown (Turhan et al., 2002). Some other legume seeds e.g. dry beans, (Sopade and Obekpa, 1990) and red bean (Abu-Ghannam, 1998a) exhibit a similar behavior. According to the theory that hydration is a diffusion dominated process, as temperature increases, the rate of water diffusion in seeds increases as well.

The effect of temperature in the hydration behavior of legume seeds, cereals and other dehydrated food materials has been intensively studied. Prasad and his coworkers studied the hydration kinetics of chickpea split at temperatures of 40, 50 and 60 °C, and they suggested that the water absorption was more rapid as temperature increased, due to the increase of diffusivity (Prasad et al., 2010). Sopade and Obekpa found that the water absorption rate and capacity were enhanced when temperature increased up to 40 °C in soybean and cowpea (Sopade and Obekpa, 1990). Khazaei and the collaborates soaked sesame seeds at four temperatures and observed a similar trend: when temperature increased, the soaking rate and water absorption capacity both increased (Khazaei and Mohammadi, 2009).

However, Maldonado (Maldonado et al., 2010) studied the effect of temperature on the rehydration of dehydrated mangoes and found that at 40°C, the diffusivity was larger than either at 25 or 60 °C and so was the

amount of water absorbed. They indicated that at 60°C, the less efficient rehydration was probably caused by the damage of cellular tissue. Similarly, Sayar and his collaborates (Sayar et al., 2001a) concluded that above 55°C, the water diffusion rate of chickpea decreased probably due to the gelatinization of starch at higher temperature as shown in Figure 1.3. Similarly, Haladjian observed a reduction in the maximum water holding capacity of faba bean when soaking temperature was increased from 50°C to 65°C in all pHs (Haladjian et al., 2003). Kon and collaborates (Kon et al., 1973) investigated the nutrient loss of small white bean soaking at elevated temperatures and found that the nutrient loss increased three to four-fold when temperature was increased to 60 °C, but remained very small when temperature was below 50 °C as shown in Table 1.4. As an example, the effect of soaking temperature on water absorption behavior of chickpea (5 temperatures tested) is shown in Figure 1.4 (Sayar et al., 2001a). Although the hydration rate was significantly improved at elevated temperature, the water holding capacity decreased as temperature increased.

pH of hydration medium

The pH of the hydration medium is also an important factor because it influences seed components such as protein, starch and antinutritional factors (Abousamaha et al., 1985; Negi et al., 2001). Most previous researchers proposed that an alkaline solution is preferred for soaking beans when considering the product nutritional value, while a few opposite conclusions exist. Aranda and coworkers (Aranda et al., 2004) soaked faba beans in pH

2.6, pH 5.3 and pH 8.4 solutions at room temperature and results suggested that soaking in a basic solution caused lowest mineral losses from soaked beans with no differences observed when Ca or Mg were used for soaking in solutions of different pH. Similarly, Nestares and coworkers (Nestares et al., 2001) used the same pH solutions for soaking common beans (*Phaseolus vulgaris L*.) and evaluated the nutritive utilization of calcium, phosphorus and magnesium. They stated that as pH increased, mineral absorption and apparent digestibility coefficient also increased, probably due to the lower losses of soluble minerals. In another report, they further studied the protein digestibility and utilization using the common beans at the same pHs and concluded that soaking in basic solution also improved both indices (Frias et al., 2000; Nestares et al., 2001). In contrast, Aparna and his coworkers (Aparna et al., 2000) found that adding sodium bicarbonate (pH increased to 6.4-7.2) and sodium chloride (did not change pH, remained 5.5-6.7) in the cooking media for various legumes (blackgram, field bean, lentil, and moth bean) reduced the *in vitro* starch digestibility (IVSD) while adding tartaric acid (reduce pH to 4.4-4.9) and citric acid (reduce pH to 5.0-5.6) increased IVSD significantly. All four substances added into cooking media brought down the *in vitro* protein digestibility (IVPD) to different levels.

Salts

Salts are often used during the industrial processing of beans to improve hydration because of their low costs and better availability. The addition of salts to the soaking medium affects the water uptake, texture and nutrition

value of the final product. This is attributed to the alterations in permeability of cell membrane in salt solutions. El-Adaway et al. studied the effect of addition of sodium bicarbonate at a 0.5% concentration in soaking medium for soybean, lupine and bean seeds and found that soaking reduced antinutritional factors and protein solubility but increased available lysine and *in-vitro* protein digestibility probably because of the partial removal of tannins as a protein precipitant and thus could serve as a nutrient supplement (El-Adawy et al., 2000). Sodium bicarbonate was also effectively utilized as an effective additive to reduce the levels of phenolics and tannins of *Bauhinia purpurea L.* seeds and velvet beans by some researchers (Vadivel and Pugalenthi, 2008; Vijayakumari et al., 2007). Abousamaha and his collaborates stated that soaking lentil seeds in 4% saline solution led to an increase in nitrogen solubility and protein digestibility (Abousamaha et al., 1985). Varrianomarston and his collaborates (Varrianomarston and Deomana, 1979) found that the binding of phosphorus facilitates the softening of beans during cooking and recommended adding sodium triphosphate to the bean soaking medium to get better overall quality. However, it was found that the water absorption rate did not increase when applying 0.5% sodium bicarbonate into soaking medium for faba beans but reduced as the concentration of sodium bicarbonate increased from 0.5% to 1% and 5% (Kader, 1995) possibly caused by higher viscosities and lower water activity.

Pressure

The use of hydrostatic pressure during food hydration enhances the moisture uptake rate at different temperatures compared to atmospheric pressure. Ramaswamy and coworkers suggested that moderate hydrostatic pressure (33 MPa) on navy beans facilitated the initial water absorption rate and reduced the loss of solids (Ramaswamy et al., 2005). Bello concluded that hydration of rice kernels under pressure 0.25 - 0.7Pa increased the water absorption rate four times and the absorption capacity slightly more than that under atmospheric pressure at all three temperatures tested: 15, 35 and 55 °C (Bello et al., 2008). This is because that water absorption is related to the hydrostatic pressure and osmosis pressure differences across the cells (Fisher, 1955; Mees and Weatherley, 1957a, b).

Soaking time

Soaking time is also considered in relation with hydration because it affects the nutrition value of soaked product. Soluble components in food could leach out into soaking medium upon time, altering the nutritional composition in the final product. Prolonged soaking time could cause the leaching of minerals and nutrients, especially at high temperature. Shi et al. (Shi et al., 2009) found that soaking time significantly affected the stability of saponin B in navy beans which is a controversial component that has antifungal and antibacterial function, lowers blood cholesterol and imbibes cancer cells (Matsuura, 2001), however has antinutritional effects as well (Gurfinkel and Rao, 2003; Khalil and Eladawy, 1994).

Storage condition

In seed processing, seeds are stored after dehydration until use. They storage condition can affect the water reabsorption efficiency during hydration process and the quality of final seed products i.e. color, flavor, texture (Coelho et al., 2007). In general, beans stored at temperature higher than 25 ºC and relative humidity greater than 65% lead to a hard-to-cook (HTC) defect, increasing the cooking time and energy use. A few researchers kept the beans under similar undesired conditions and studied the hardness and cooking time of the beans. They found out that storage condition with higher temperature (>35 ºC) and relative humidity (>65% RH) dramatically accelerate the aging of beans, causing longer cooking time and greater hardness (Nasar-Abbas et al., 2008). Some researchers pointed out that storage at 40 º C under 76% RH for 20 days lead to a hardness equal to the natural aging for a year under cool and dry ambient condition (S et al., 2009). In addition, the development of HTC defect was suggested to reduce nutritional value of beans in *in vitro* protein digestibility and *in vitro* starch digestibility due to stronger interactions between phytic acid and proteins or carbohydrates (Nyakuni et al., 2008). Many previous researches reported a positive correlation between HTC defects and reduction in phytate content of beans during storage (MartinCabrejas et al., 1997; Medeiros Coelho et al., 2007; Nasar-Abbas et al., 2008).

Volume kinetics during hydration

Importance of volume change in hydration

It is known that dry seeds swell during hydration up to more than 250% of their original volume, with volume changes varying depending on the variety. For instance, most legume seeds are able to swell up to ~ 250% of their original volumes, e.g. kidney beans (Tagawa et al., 2002), soybean (Bayram et al., 2004)，and chickpea (Chenoll et al., 2009b), whereas canola seeds to ~ 165% (Thakor et al., 1995) and rice up to ~140% (Muramatsu et al., 2006). The volume change curve of chickpea soaking at room temperature which is also representative of the trend of most legume seeds is shown in Figure 1.5. Volume expansion during hydration is suggested to be proportional to the amount of water absorbed in most biological materials (Steffe and Singh, 1980). In view of industrial processing, volume becomes an important parameter that relates to the design of the facilities and the processing; the recovered volume of legume seeds is also a key parameter for canning processing that has direct impact on cost. Volume is also an important quality factor for the rehydrated food products which not only indicates water uptake rate during hydration, but also correlates with critical quality-related parameters. Thakor found that the volume change of canola during hydration at three different temperatures had a positive and linear correlation with its moisture content described by Eq. (2)

$$\frac{V - V_0}{V_0} = \beta_V (M - M_0) \quad (2)$$

where *V* is the volume, *M* is the moisture content, subscription *0* represent initial value and β_V, the empirical coefficient was calculated to be around 1.032, and the R^2 to be 0.99 (Thakor et al., 1995). Tagawa and his collaborates proposed an equation describing the relationship between volume and both moisture content and temperature for kidney beans during soaking with a first-order relationship between volume and moisture content. They later found that the experimental volume data had a quadratic relationship with moisture content as shown in Eq. (3):

$$V_b = a_1 M_w^{\ 2} + b_1 M_w + c_1 \quad (3)$$

where V_b represents the bulk specific volume, M_w represents the moisture content, and a, b, c are empirical parameters (Tagawa et al., 2002).

The R^2 was above 0.975 for all three varieties of kidney beans tested. Furthermore, volume of the final product itself is also an aesthetic parameter that affects the preference of consumers according to many sensory experiments. In conclusion, a good description of the change in volume kinetics during hydration could help understand the hydration process and also better monitor the change in product quality

Methods for volume determination

There are three major types of volume determination methods for seeds with different shapes in previously reported literatures. One method is to

measure the particle in two or three axis, approximate the volume to an ellipsoid or a spheroid to calculate the volume. Ways to measure the two dimensional lengths of the sample include the use of a micrometer (Bayram et al., 2004; Khazaei and Mohammadi, 2009), a digital caliper (Chenoll et al., 2009b) or an imaging equipment (Shahin et al., 2006). The manual measurement method takes longer time and is less accurate and repeatable even with the assistance of imaging equipment. Another major drawback of this method is that approximating volume using the width and length measurement reduces accuracy because of the variations in shapes of different food materials. The other common method used is based on Archimedes's principle that is, the volume of the material is equal to the volume of water expelled from a container full of liquid after placing the material. And the volume equates to the quotient of buoyancy force the material suffers when immersing inside the liquid divided by the density of that liquid. An example is measuring volume of water chestnut using n-heptane liquid and calculate volume with Eq. (4) (Moreira et al., 2008),

$$V = \frac{m - m_{hep}}{\rho_{hep}} \qquad (4)$$

where V is the volume of sample, m is the mass of sample in the air, m_{hep} is the mass of sample in the heptane liquid, and ρ_{hep} is the density of heptane. Other researchers tried to measure the bulk density or particle density of the samples to calculate volume using a pycnometer filled with toluene (Muramatsu et al., 2006). The procedures are extremely labor-intensive and time consuming. The third type of method involves liquid displacement in a

graduated cylinder (Thakor et al., 1995). Again, it requires intensive work and also lacks accuracy and repeatability due to manual operation.

Math models for seed hydration

The models that have been used for food hydration include mechaistic models which are based on the physical interaction and empirical models. Empirical models are adopted much more often than mechanistic models because of their simplicity (fewer parameters) and utility. Among mechanistic models, diffusive models based on Fick's second law are commonly used by researchers (Khazaei and Mohammadi, 2009; Sanjuan et al., 1999; Vega-Galvez et al., 2009). Recent studies suggest that the hydration process can not be simply considered a diffusion process because of the energy potential inside the porous or fibrous food (Saguy et al., 2005; Weerts et al., 2003). Capillary flow theory based models were also introduced but had not been widely adopted (Lee et al., 2006; Ni and Datta, 1999; Weerts et al., 2003). In terms of empirical and semi-empirical models, the exponential model, Peleg model and Weibull distribution were applied to describe food hydration process where the last two being the most popular models in the description of hydration kinetics.

Mechanistic model

Diffusion model

The diffusion model was based on Fick's second law of diffusion. It is applicable when assuming that 1) diffusion dominates the water transfer process, 2) no external resistance to heat and mass transfer exist, 3) initial moisture content is uniform, and 4) the shape of seed can be approximated to a sphere. Bello proposed Eq. (5) for accurately estimate the saturation moisture content of rice grain at different soaking conditions when assuming the sample shape to be a sphere (Bello et al., 2004).

$$\frac{M_t - M_s}{M_0 - M_s} = \frac{6}{\pi^2}\exp(-\frac{D_e \pi^2 t}{R^2}) \qquad (5)$$

Where M_t, M_s, M_0 are the moisture content at time t, saturation moisture content and initial moisture content, respectively, D_e is the diffusion coefficient, R is the radius of the sphere. Khazaei and Mohammadi successfully applied this model on hydration kinetics of sesame seeds. However, another form of Fick's second law, shown as Eq. (6) did not fit the actual data well after the rapid moisture uptake stage (Khazaei and Mohammadi, 2009)

$$\frac{M_t - M_0}{M_s - M_0} = \frac{2}{\sqrt{\pi}}(S/V)\sqrt{D_e t} \qquad (6)$$

where M_t, M_s, M_0 are the moisture content at time t, saturation moisture content and initial moisture content, respectively, S is the surface area, t is the soaking time. The test data and estimated data using the two models are shown in Figure 1.6 as below.

Capillary-flow theory based model

Researchers recently brought up the concept that the hydration process of

dry porous foods is not a diffusion dominated process but rather capillary-flow dominated. Due to the complexity of this model, only a few studies used this model to characterize the hydration. This process was described by the Lucas Washburn equation (7)

$$\frac{dh(t)}{dt} = \frac{r\gamma\cos\theta}{4h(t)\eta} - \frac{r^2 g\rho}{8\eta} \qquad (7)$$

where ρ is the liquid density, γ is the surface tension, η is the fluid viscosity, θ is the advancing liquid contact angle, t is the time, g is the gravitational constant, r is the pore radius and h(t) is the height of liquid rise. The equation is valid based on these assumptions: the food could be simplified as to consist of multiple pores, the water flow is Newtonian, one dimensional, steady state and fully developed (Lee et al., 2006). There were two parameters in this model: $k_1 = \frac{r\gamma\cos\theta}{4\eta}$; $k_2 = \frac{r^2 g\rho}{8\eta}$. The parameter k_1 (m^2s^{-1}) is the dominant factor of the initial rate and k_2 is dominant when hydration approaches equilibrium. Weerts and collaborates used capillar flow based model for hydration of black leaf tea and the result suggested that the fitting was not good compared to other models, as shown in Figure 1.7 (Weerts et al., 2003). However, few articles was found applying the model on seed hydration process.

Empirical models

Peleg equation

Peleg equation was first brought up to the describe moisture sorption (Peleg, 1988a) and was later successfully used for modeling hydration processes of many food materials including soybean (Wardhani et al., 2008), cowpea, peanut (Sopade and Obekpa, 1990), sesame seeds (Khazaei and Mohammadi, 2009), chickpea splits (Prasad et al., 2010), aloe vera (Vega-Galvez et al., 2009), okras (Apar et al., 2009), chestnut (Moreira et al., 2008), apple (Bilbao-Sainz et al., 2005), and wheat (Maskan, 2002). The Peleg model is described as Eq. (7)

$$X_t = X_0 + \frac{t}{k_1 + k_2 * t} \qquad (8)$$

In the equation, X_t, X_0 are the moisture content at time t and in the beginning, respectively, t is hydration time, k_1 is the rate constant which equates to the reciprocal of hydration rate while k_2 is related to the end moisture content and is more constant except for the cases when the sample is subjected to structural or compositional alterations. In Figure 1.8, Peleg model is shown to be able to fit the hydration curve of wheat at different temperatures with R^2 > 0.98-0.99.

Weibull distribution

The Weibull distribution is a mathematical probabilistic equation which has a physical basis for the use of modeling an event that has certain degree of variations (Brown and Wohletz, 1995). It is described as Eq. (8)

$$X_t = X_e + (X_0 - X_e)\exp[-(\frac{t}{\alpha})^{\beta}] \qquad (8)$$

where X_t, X_e, X_0 are moisture content at time t, equilibrium moisture content and initial moisture content; t is hydration time; α is the scale parameter; β is the shape parameter. So far many researchers have proved that the Weibull distribution is an effective model for hydration kinetics of sesame seeds (Khazaei and Mohammadi, 2009), chickpea splits (Cunningham et al., 2007), breakfast cereal (Machado et al., 1999), caroba slices (Corzo et al., 2008), and tropical fruits (Marques et al., 2009). A normalized Weibull distribution was later introduced for carrots (Marabi et al., 2003) to account for different shapes of food materials. Some researchers suggested that the reciprocal of α represents the initial rate of the hydration process (Machado et al., 1999) and β suggests the time required to complete absorbing 63% of total water uptake (Cunningham et al., 2007). The use of Weibull distribution in fitting the data on hydration of tropical fruits is shown in Figure 1.9 (Marques et al., 2009), the reported R^2 was > 0.95.

Exponential equation

This is the simplest empirical model usually expressed as Eq. (9),

$$M = e^{-kt} \qquad (9)$$

where M is the moisture content, t is the hydration time. This model is a simpler version of Weibull distribution and k is the only parameter in the model, indicating the hydration rate constant. The use of this model is less frequent than the other two empirical models (Apar et al., 2009; Khazaei and Mohammadi, 2009; Prasad et al., 2010). However, in a study on chickpea split,

the model had shown a good fit, with R^2 > 0.99, as shown in Figure 1.10 (Prasad et al., 2010).

Conclusions

Although the hydration characteristics and factors affecting hydration of seeds have been studied in many previous researches, there are few studies related to pinto, navy and black beans. Due to the time consuming and labor intensive volume determination methods for the irregular-shaped seeds, few reports are available to adequately describe the volume kinetics during hydration. It is necessary to find a more effective and reliable volume determination technique for better understanding the process of water uptake and the differences among different types of seeds during soaking. No literature reported the very different hydration behavior of different bean varieties and the specific treatment needed to shorten the hydration time. In order to better characterize the hydration process, i.e. to quantify the water uptake rate, math models were heavily applied. Empirical models such as Peleg model and Weibull distribution are more often used than mechanistic models. Peleg model predicts better than other models for most of samples at different temperature in previous reports.

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Appendix

Table 1.1 Production from major food legume producing countries (metric tons) (FAOSTAT, 2007).

Nation	Common bean, dry	Soybean	Peas, dry	Chickpea	Lentil
Brazil	3169360	57857200	721347	-	721347
India	3930000	10968000	800000 *	6333700	-
U.S.	1150808	72860400	874000 *	-	154584
China	1233005 *	12725147	-	-	135000 *

* Unofficial data; F FAO estimate; - production value comparatively low.

Table 1.2 Effect of soaking on the antinutritional factors of two varieties of lentil seeds (Abousamaha et al., 1985).

Soaking time (h)	Lentil Giza				Lentil Syrian			
	A	B	C	D	A	B	C	D
1	31.66	0.0	5.02	2.7	35.88	0.54	3.04	1.06
3	34.17	2.27	5.16	7.72	36.47	1.88	3.5	7.06
6	44.17	4.54	5.39	10.03	50.59	3.22	3.73	7.77
9	45.83	6.81	9.78	11.58	51.17	6.18	5.22	11.66
12	55.00	7.27	15.55	15.44	81.17	30.10	11.02	15.19

A, B, C, and D = % reduction in tannins, haemagglutinin, trypsin inhibitor and pentosans, respectively

Table 1.3 A summary of factors affecting food hydration process.

Factor	Description	Effect	Sample	Reference
Temperature	Temperature of hydration medium	Increase hydration rate (diffusivity) Increase final water content (water absorption capacity); Decrease in diffusivity as temperature increases (over 55C) Decrease water holding capacity	Chickpea split Soybean Sesame seed Mango Chickpea Faba bean	K. Prasad, 2010 P.A. Sopade, 1990 Javad Khazaei, 2009 S. Maldonado, 2010 Sedat Sayar, 2001 Nanor Haladjian, 2003
pH	pH of soaking or cooking medium; regulated by adding acid or salt	Alkaline medium increases IVPD, acidic medium increases IVSD; Both alkaline and acidic medium reduces IVPD	Faba bean Common bean blackgram, field bean, lentil, and moth bean	Pilar Aranda, 2004 Nestares et al. 2001 K. Aparna, 2000
Additives	Salts mainly, in some cases affect pH	Sodium bicarbonate increases product nutrition value; high concentration (>1%) has negative effects Sodium triphosphate could soften the texture of soaked product	Soybean, common bean, velvet bean	T.A. EI-Adawy, 2000 Kader, 1995 E. Varriano-Marston, 1979
Pressure	Applied pressure during hydration	Appropriate pressure increase water absorption rate, water uptake capacity, decrease the solid loss	Navy beans Rice kernel	Raghupathy Ramaswamy, 2005 Marcelo O. Bello, 2008)
Soaking time	Nutrients/antinutrients leaching and degradation	Longer soaking time leads to more loss of saponin B	Navy bean	John Shi, 2009

Table 1.4 Nutrients present (%) in California small white bean after soaking at different temperatures. (Kon et al., 1973)

Soak temp. (°C)	Total solids	Total sugars	Oligo-saccharides	N	Total P	Ca	Mg	Thiamine	Riboflavin	Niacin
20	96	92	91	99	99	94	76	98	99	93
40	97	95	96	95	89	93	70	98	94	92
50	94	73	81	93	87	91	64	92	90	87
60	83	61	60	81	74	91	52	69	80	58
70	85	55	59	83	74	93	52	63	70	55
80	85	55	59	84	75	96	52	63	70	52
90	82	55	61	87	82	91	52	63	53	51

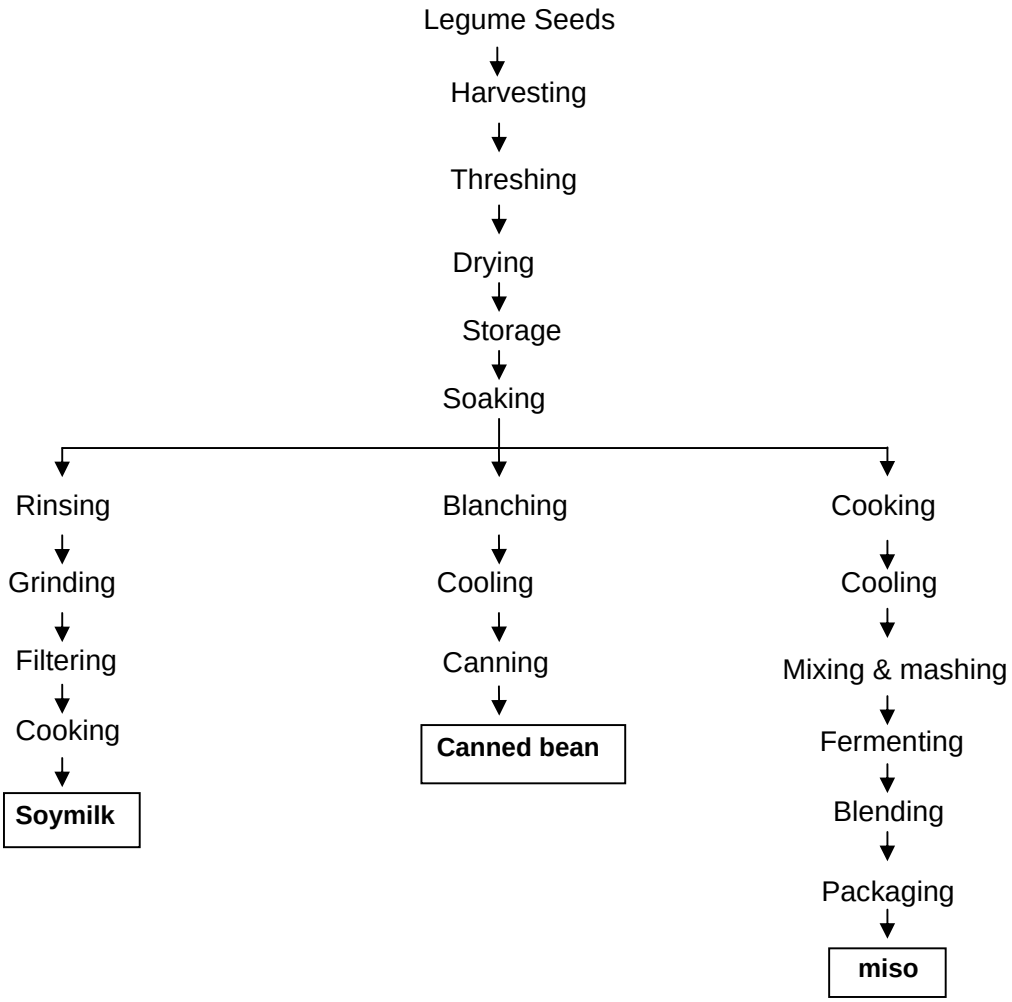

Figure 1.1 Legume seed postharvest processing steps (Salunkhe et al., 1985).

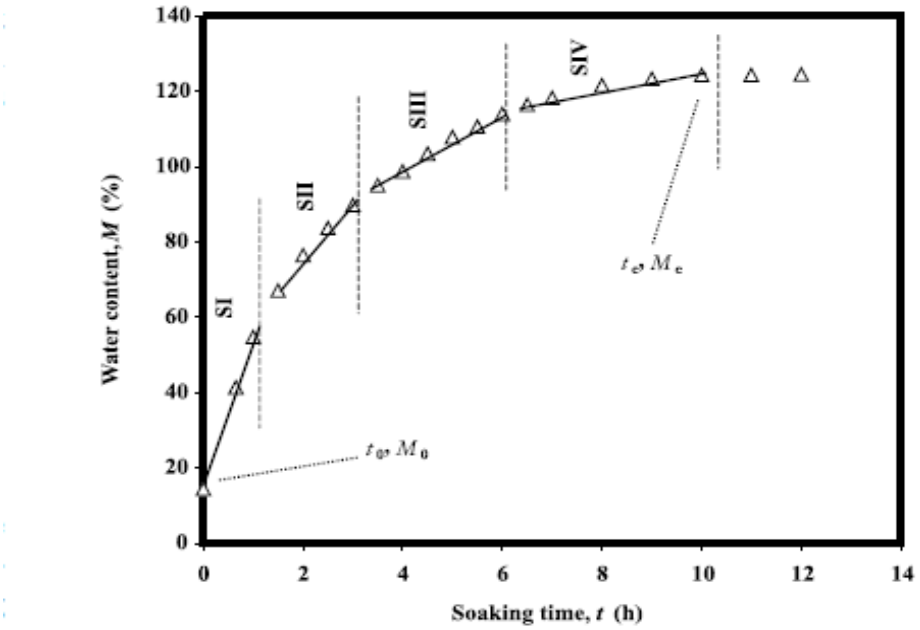

Figure 1.2. water absorption curve during soaking of chickpea at 20 °C. SI, SII, SIII, and SIV represent linear segments of the curve (Turhan et al., 2002).

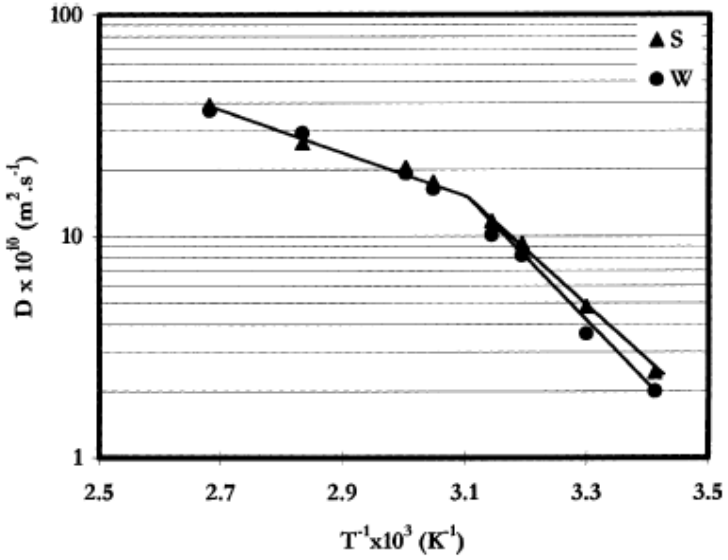

Figure 1.3. Effect of soaking temperature on effective water diffusitivy in chickpea (Sayar et al., 2001a). D is the diffusion coefficient, T is the absolute temperature.

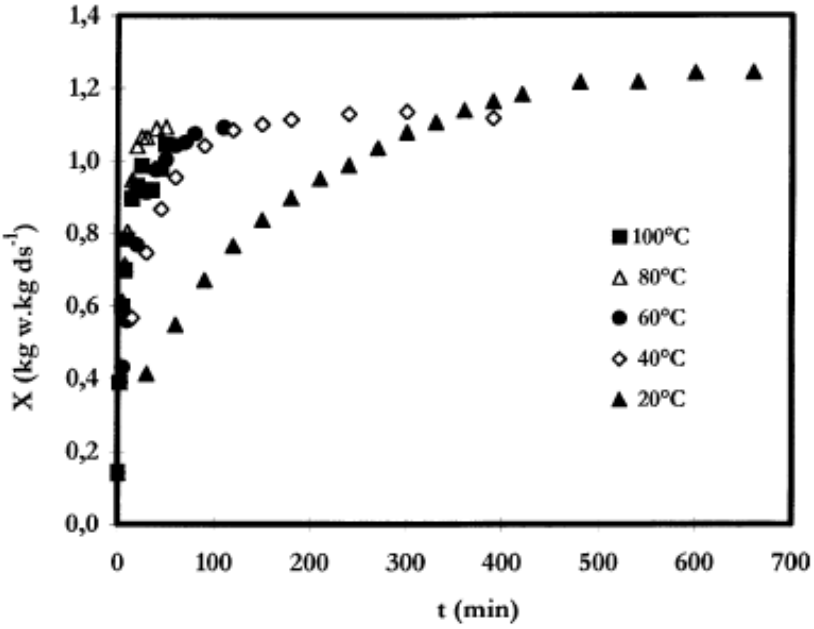

Figure 1.4. Effect of temperature on water absorption of chickpea during soaking (Sayar et al., 2001a). X is the moisture content, t is the time.

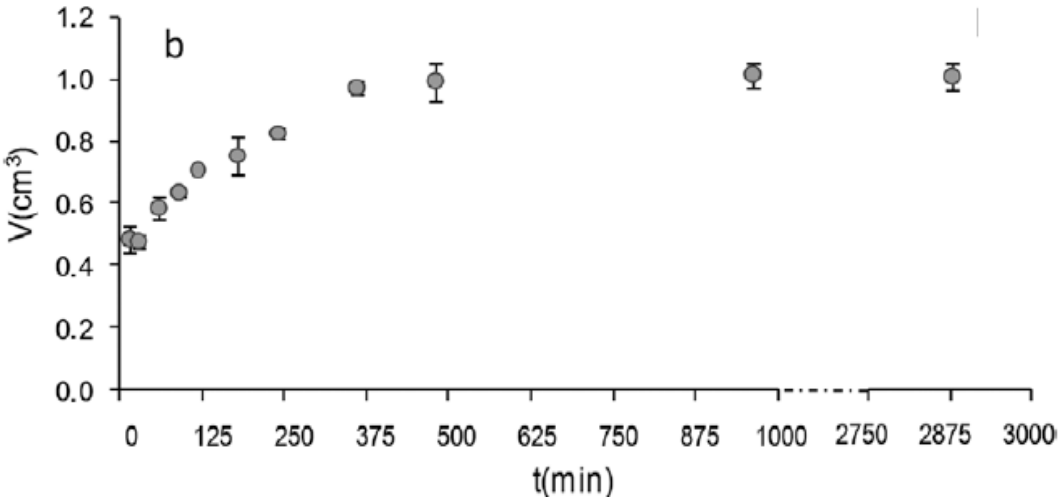

Figure 1.5 Volume change curve of chickpea soaking at 25 °C (Chenoll et al., 2009b). V is the volume, t is the soaking time.

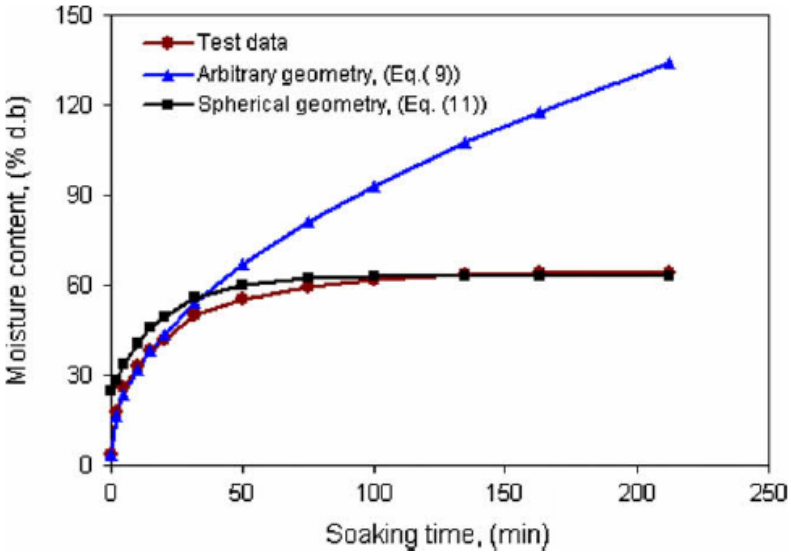

Figure 1.6. Fitting of two forms of Fick second law to water absorption data of sesame seeds at temperature of 50 °C (Khazaei and Mohammadi, 2009).

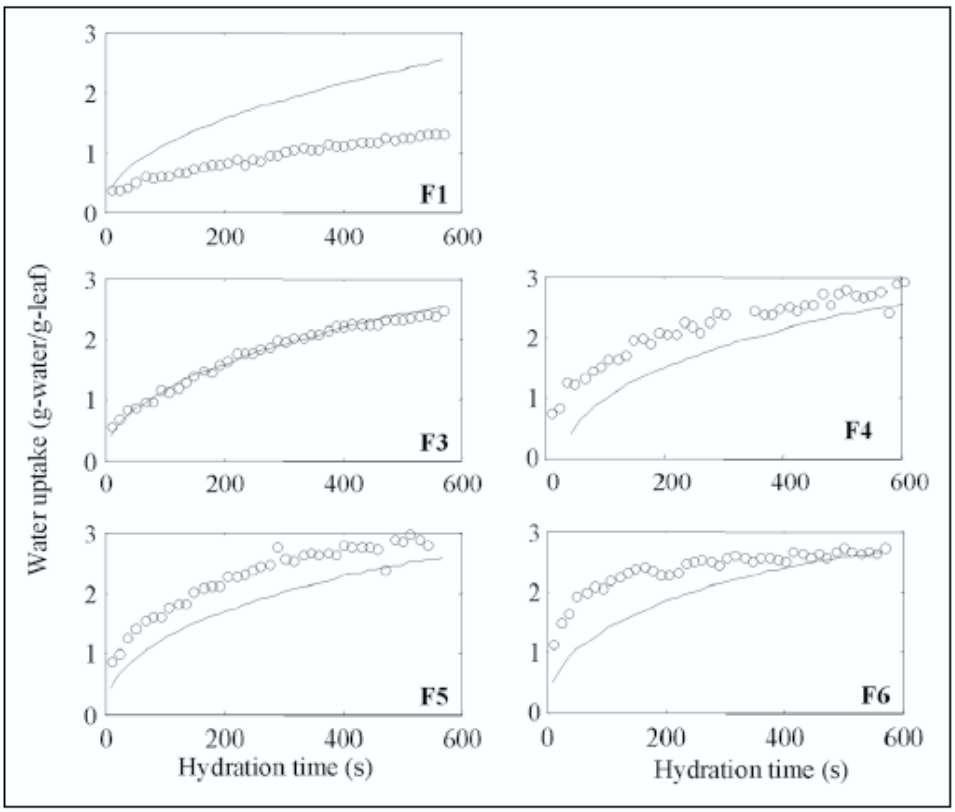

Figure 1.7 Predicted hydration (solid lines) and experimental data (circles) of hydration of black leaf tea Assam at T=333K (Weerts et al., 2003).

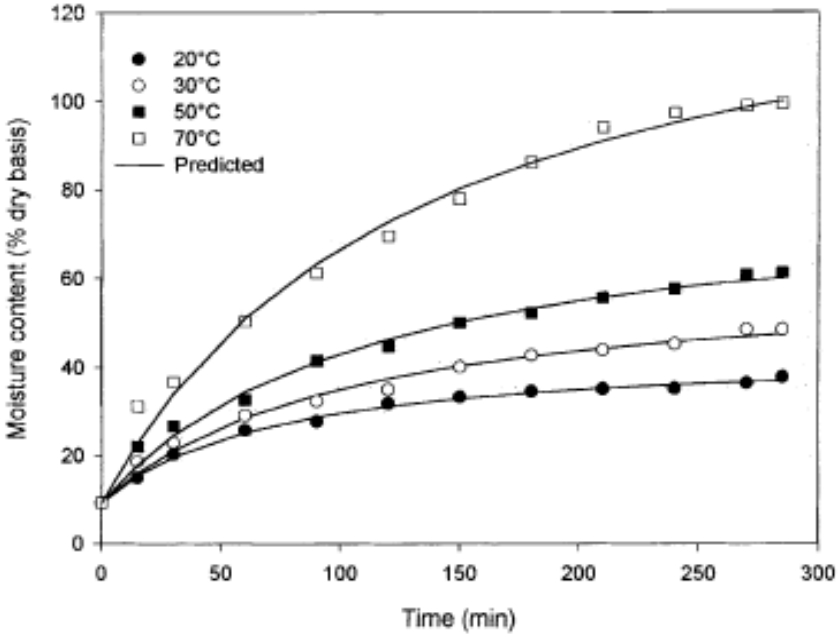

Figure 1.8 Predicted data by Peleg model and experimental hydration curves of wheat when soaking at different temperatures (Maskan, 2002).

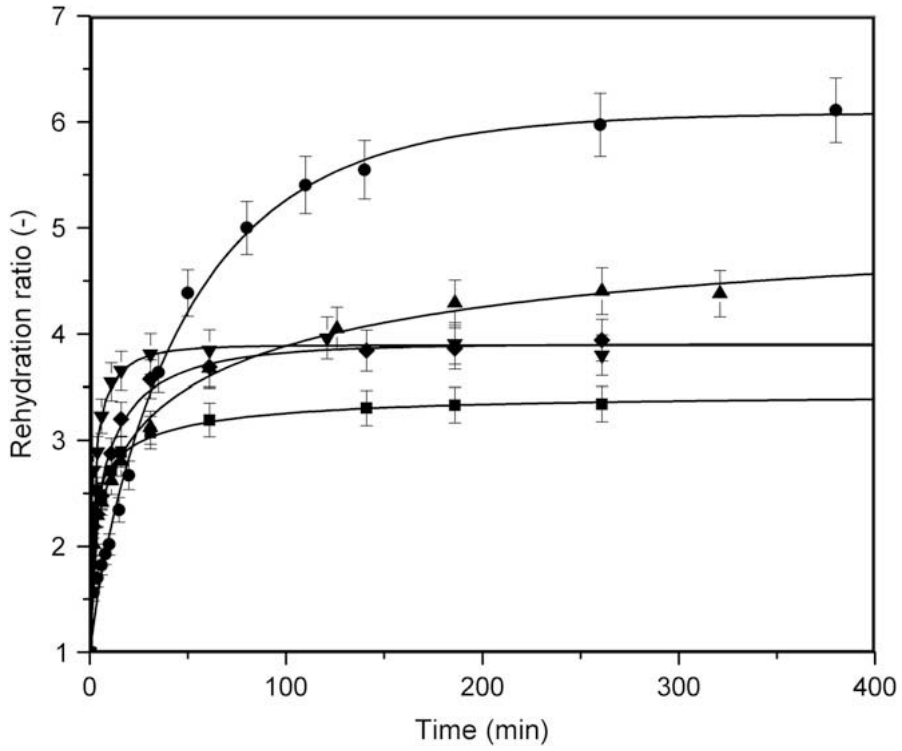

Fig. 1.9 Rehydration ratio of the freeze-dried fruits as a function of time. Experimental data: dots; Predicted values by the Weibull equation: line (Marques et al., 2009).

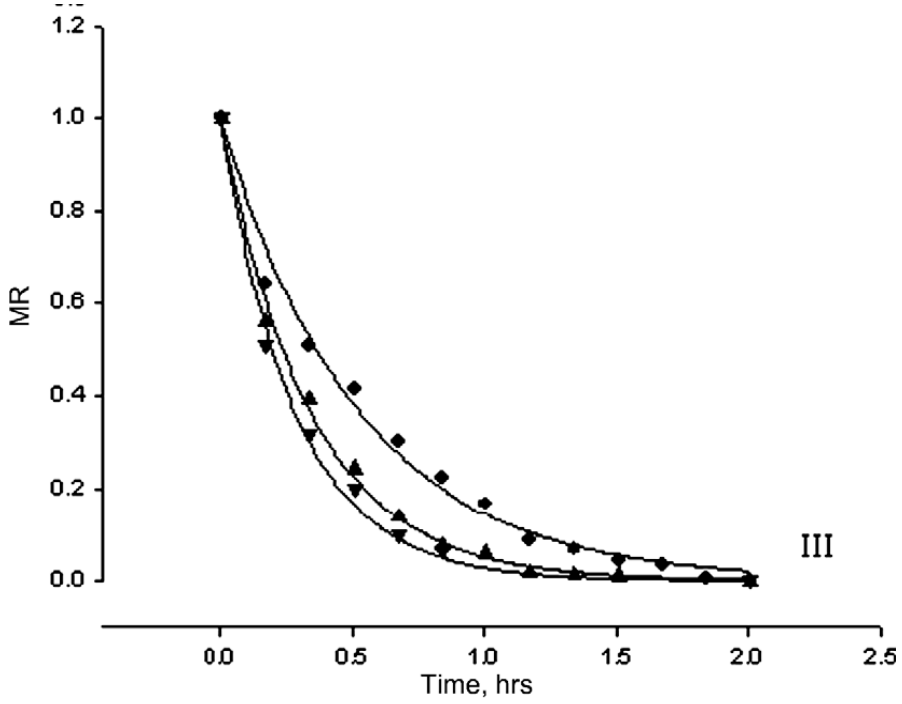

Figure 1.10 Fitting of Exponential model on water uptake of chickpea split at 40, 50 and 60 ºC (Prasad et al., 2010).

CHAPTER II
AN AUTOMATIC MEASURING SYSTEM FOR STUDYING VOLUME KINETICS OF SEED HYDRATION: DESIGN, DEVELOPMENT AND EVALUATION

Abstract

Hydration or soaking is a critical unit operation in the industrial processing of seeds used for human consumption. The volume kinetics during seed hydration is important because volume change is proportional to water uptake and associated with moisture content and aesthetic quality of the product. However, current volume determination methods are extremely labor-intensive and time-consuming when highly reproducible and frequent volume measurements are needed. This study explains the design, development and evaluation of a bean volumetric tester (B-VAT) for seed hydration at controlled conditions. The system integrates a physical device, control and data acquisition systems, and software to automatically measure volume of seeds over time. The evaluation tests showed that the B-VAT system (1) has low systematic variability (0.9% COV) in volume measurements, (2) is able to measure volume at minute-scale intervals, (3) generates highly reproducible results with dry beans tested, (4) can potentially be used for different seeds at different temperature in various media, and (5) requires minimal operator work and time. The system shows good potential for adequately depicting the volume kinetics of seeds during hydration and better characterizing the hydration process.

Introduction

Legume and grain seeds are the top two sources for human diet, providing carbohydrate, protein, and dietary fiber (Ruth, 1989). Seed hydration, also called soaking, is a widely adopted unit operation in preparing seed-based food products. Soaking is commonly adopted prior to milling soybeans for soymilk production, before cooking in bean processing, and for the preparation of many Asian traditional diet such as natto, tempeh and rice (Kashaninejad, 2007; Muramatsu, 2006; Bilgicli, 2009; Bayram et al., 2004). The optimum soaking time needed for some popular legume seeds at different temperatures is shown in Table 2.1.

The adoption of soaking shortens the required cooking time by introducing water evenly into the dry seeds before cooking, which saves cost and energy and reduces nutrient degradation. The water distribution between starch and protein fractions also reduces time required to obtain an acceptable food texture (Gowen et al., 2007). Another beneficial aspect of soaking under optimal conditions, is to facilitate the leaching of anti-nutrients that exist in legume seed e.g., tannins, phytic acids, tripsin inhibitors and oligosaccharides that cause flatulence (Lestienne et al., 2005). The soaking is usually considered to be composed of three simultaneous steps: 1) water uptake into the material; 2) volume expansion; 3) leaching of solids (Lee et al., 2006).

Nomenclature

V1 V2 V3	solenoid two-way valves 1, 2, and 3.
P1, P2	fill pump, drain pump
S1	ultrasonic sensor
S2	optical level switch
MT	measuring tank
SC	seed chamber
ST	surrounded tank
TE	time elapses
B1, B2	water bath 1, 2
ON/OFF	status for valves and pumps
n	iterations of measuring cycles
x	measurement interval (min)
a	max fill level signal (V)
A	water tank cross section area (cm2)
b	drain time

CV – chamber volume WV- water input volume SV- sample volume

The major changes happening during soaking are not limited to changes in weight, moisture content and nutrient content but also to volume which is usually proportional to the water uptake (Steffe and Singh, 1980). The characteristics of volume kinetics thus are related to the water absorption properties and can contribute to the understanding of the overall hydration process. The volume of the final product also affects its aesthetic quality, influencing consumers' preferences. Some previous studies suggest a positive linear or quadratic correlation between volume and moisture content, the latter an essential quality indicator of soaked seeds (Tagawa et al., 2002; Thakor et al., 1995). Moreover, the seed volume can increase to almost three times the original size during

soaking and the volume expansion rate changes over time depending on the hydration characteristics. A complete study of volume kinetics during soaking facilitates understanding the characteristics of the hydration process, controlling part of the quality attributes of the final product, and designing the processing facilities.

Because of the irregular and inconsistent shapes of seeds, and the complex volume change throughout the hydration process, volume determination becomes time consuming and labor intensive, especially when a detailed picture of volume kinetics is needed. The methods used so far to measure the volume change in seeds are: 1) to measure the dimension of a seed with a caliper or micrometer, approximate its shape to an ellipsoid, then use equation $V = \frac{4}{3} * \pi * a * b * c$ to calculate the volume where a, b, c are the lengths in three axis (Chenoll et al., 2009a); 2) to utilize imaging equipment for getting two dimensional image, then calculate the volume as previous described; 3) to apply Archimedes' principle, using n-heptane as immersing liquid and calculate by equation $V = \frac{m - m_{hep}}{\rho_{hep}}$ (Moreira et al., 2008); and 4) to determine bulk density and particle density using a pycnometer, then calculate volume (Muramatsu et al., 2006). Volume measurements are done in most cases only at a few time points during the process, providing an unclear depiction of volume kinetics. Furthermore, all these methods are time consuming, labor intensive, and prone to error introduced due to the differences among operators. The imaging analysis

method, although reduces labor work in measuring seed dimensions, still brings about error due to shape approximation. Moreover, these methods typically do not have a large sampling size leading to lack of representation of the population. In summary, although the volume kinetics plays an important role in hydration characterization, quality of product and process design, it is not well monitored during hydration due to the lack of efficient and reproducible techniques.

The objectives of this study were 1) design and develop a stand-alone system to automatically conduct volume measurements at minute-scale time intervals and provide a detailed depiction of volume kinetics during seed hydration under controlled conditions and 2) evaluate system accuracy and repeatability.

System Description

Design overview

The volume change of irregular-shaped seeds during soaking is measured as the difference in the volume for a given chamber and the volume of water to fill the chamber with seeds at pre-defined time intervals. A device shown in Figure 2.1.A contains a seed chamber (SC) where the sample stays, a water bath (B1) as the source of the input water, and a measuring tank (MT) where the water volume can be determined with an ultrasonic level sensor. The soaking temperature is maintained constant by two water baths with a circulating tank surrounding the seed chamber to reduce heat loss. Three valves and two pumps

are utilized in the device for water flow control. An ultrasonic sensor is used to measure the volume changes throughout the testing procedure. The repetition of volume measurements at desired time points is realized by a PC-based programmable data acquisition system that has control over the valves and pumps.

Basic principle and process

The fundamental parts of the physical device responsible for repeated volume measurements are shown in Figure 2.1.A. The functionalities of these parts and the basic principle and process of the multiple volume determinations are described below. A cylindrical measuring tank (MT) is equipped with an ultrasonic sensor (S1) on its top to continuously measure inner water level. So the volume of water in the measuring tank is calculated by multiplying the S1 level reading by the cross section area of the measuring tank. A seed chamber (SC) is equipped with an optical level switch (S2) to indicate whether water has reached the level of the switch (Ls) and a mesh at the bottom to retain the sample during hydration. A water bath (B1) is the source for the hydration medium. The three main tanks are connected via flexible tubing indicated as bold black line with 3 valves (V1, V2, V3) and 2 pumps (P1, P2) for flow control. A surrounding tank (ST) where water is continuously circulating with a second water bath (B2) is used during the test in order to keep hydration temperature constant. The volume measurements

over time are conducted through the repetition of three major steps described as below.

Step 1: Fill MT. Valve 1 (V1) is opened and water is pumped into the MT until a preset level (L0) is reached as shown in Figure 2.1.B. Then V1 is closed.

Step 2: Transfer to SC. Valve 2 (V2) is opened and pump 1 (P1) is started, water is then transferred into the empty seed chamber until water filled up SC, at which moment the inner water level of MT dropped to a different level (L1) as shown in Figure 2.1.C. V2 is closed and P1 is turned off once water reaches Ls. The inner volume of SC is equal to the volume of water that has been transferred out from MT which can be calculated by equation (1)

$$V_{chamber} = A*(L0 - L1) \quad (1)$$

where A is the cross section area of MT.

Step 3: Drain SC. Valve 3 (V3) is opened, pump 2 (P2) is turned on so that water is draining from SC to B1 as shown in Figure 2.1.D. After water is completely drained, V3 is closed and P2 is turned off.

After seeds are loaded, step 1, 2 and 3 are repeated as shown in Figure 2.1.E to 2.1.G. The initial seed volume can be calculated using Equation 2 where L2 is the level reading in MT after water transferred to SC and $V_{chamber}$ was calculated using Eq. (1) as described previously.

$$V_{0-seed} = V_{chamber} - A*(L0 - L2) \quad (2)$$

After initial seed volume is obtained, a preset amount of time elapses (TE) between step 2 and 3 for soaking the seeds, and the seed volume at any time t can be calculated by Equation 3.

$$V_{t-seed} = V_{chamber} - V_{t-water} \quad (3)$$

where the volume of water can be calculated as the production of the differences of level reading in step 2 and the cross section area of the MT.

All valves and pumps are controlled electronically via a computer, a data acquisition system and an electronic circuit board. Repeated Fill-Transfer-Drain iterations are conducted at pre-determined time intervals to obtain near real-time volume measurements during seed soaking.

System Setup

The system setup is shown in Figure 2.2. The system consists of a physical device, an electronic control board, a data acquisition system (National Instruments, USA) and a notebook computer equipped with LabView® software (v 8.6.1). The specifications of all the parts used to develop the physical device are described as below (manufacturer details are listed in the Appendix Part II):

1) Measuring tank: PVC

2) Sample chamber and surrounded tank: CPVC; inner volume: around 290 ml

3) O-rings: Buna-N size: 1-1/4''ID and 1-3/8''OD; 3-7/8''ID and 4'' OD

4) Mesh: Tri-Clamp Screen Gasket

Specs: 3” diameter, 10 mesh, material: EDPM

5) Tubing: Cole-Parmer C-flex tubing #06422-15.

Specs: 3/8’’ ID * 1/2” OD; temperature rating: 135 ºC; material: polypropylene

6) Quick connectors: CPC HFC12 series coupling body and inserts; materials**:** Polypropylene; connector size: 3/8”

7) Water bath 1: Fisher Scientific Isotemp 3016D upright refrigerating heating circulator, 6L

8) Water bath 2: Fisher Scientific Isotemp 3028P programmable refrigerated circulator, 28L

9) Ultrasonic sensor: Baumer Ultrasonic Sensor UNAM 12U9914/S14

Specs: range 2-20cm; repeat accuracy:<0.5mm; resolution: <0.3mm; voltage supply: 15-30VDC; cone range: 1 cm radius; operating temperature -10-60 ºC

10) Level switch: Honeywell Liquid Level Sensor LLE103101

Specs: voltage supply: 5-12VDC; operating temperature: -40-125 ºC; output: high (5VDC) in air.

11) Valves: Clark Solution Solenoid two-way Valve Model 1335

Specs: voltage supply: 12VDC, port size: 3/8’’NPT; body material: brass; seal material: viton; type: normally closed, combined acting.

12) Fill pump: Little Giant in-line submersible centrifugal pump BPLA 33

Specs: voltage supply:12VDC; Amp: 2A; inlet size:1/2’’; outlet size: 3/8’’; HP: 350GPH @ 1’’ head; dimension: 5” x 3.5” x2.5”

13) Drain pump: Flojet diaphragm pump Series 2P366

Specs: voltage supply: 12VDC; inlet and outlet: 3/8’’; HP: 129 GPH @ 10 psi

The LabView® software and DAQ system are the core of the control system, responsible for communicating with the electronic control board to either send command for valve operation and pump control or receive signal from level sensors. The output voltage signal from the ultrasonic sensor is read by the DAQ system and software. The National Instrument cRIO-9401 digital I/O module was used for discrete input and output control and the National Instrument cRIO-9215 was used for processing the analog voltage output from the ultrasonic sensor to digital values as well as receiving output of the level sensor directly. The power supply was a DC converter which transforms AC power (88-120VAC, 50/60 Hz) to 12VDC with a maximum output current of 12.5 Amp which sufficiently provide power to all electronic parts that need to be energized simultaneously. A 12-to-24 V DC/DC converter was used to power the ultrasonic sensor.

A circuit interface board was designed and implemented by the UT Department of Biosystems Enginering & Soil Science to energize/de-energize the valves and turn pumps on/off at appropriate times as defined by the software. The circuit board was powered from the 12V power supply, for energizing pumps and solenoid valves. A voltage regulator was utilized to regulate the 12V power input

to 5 VDC output for the optical level sensor. A pulse-width modulation was incorporated to vary the power supply for the two pumps for speed control (0 to 100%). Two inverter gates with NPN transistors (β>1000) enabled the control module to switch the power on and off for the valves and pumps. The LabView® program was used for system control and served as the user interface where the following parameters could be set by the user prior to the test:

1) Measurement interval: 1 to 30 min;
2) Number of iterations: 1 to 1000;
3) Preset volume of water for testing: L0 (in voltage) in MT: from 0.5 to 5.0V;
4) Setup output data file: name, location and attribute variables.

The following information can also be observed on the PC display during the test:

1) SC volume (ml), current seed volume (ml) and water input volume (ml);
2) Current measuring iteration and remaining time of TE in current iteration (sec);
3) Ultrasonic sensor level readings (V) from last iteration and percent coefficient of variation (%COV) from repeated readings (1000);
4) A graph showing volume change of seed over time where y-axis is volume (ml) and x-axis is time (min).

The LabView® flowchart is shown in Figure 2.3. There are two main procedures in the program, priming and testing. The priming precedes the testing in order to fill the gaps and rinse the surfaces in the B-VAT system for accurate volume determination.

Special features

The design and selection of the parts to build the physical device contained several special thoughts in order to improve the utility of the system (AutoCAD designs are listed in Appendix Part III). The sample chamber was built in the workshop in UT with shape and size designed to reduce he water trapped in the chamber and to reduce the error in volume measurement. The middle part of the SC had a larger cross section area (~62.04 cm^2) so that the seeds could be widely spread-out without bridging. The cross section area where the optical level sensor was mounted was smaller (~19.82 cm^2) to increase the precision for water volume control.

Rounded corners were used instead of straight ones to reduce water entrapment. The bottom part of the lid of the SC had a cone structure for the same reason.

The ultrasonic sensor was selected for its high resolution in level sensing. Each level reading output was the average of 1000 sample readings to reduce the random measurement error by a factor of 31.6 . A centrifugal pump used between the MT and the SC transferred water at a maximum speed of 1.6L/min with minimal pressure. A diaphragm pump was used to evacuate the SC in seconds. The speed of the centrifugal pump was optimized so that water did not flow too fast for the level switch to detect the water level and also not too slow to lengthen the processing time.

Performance Evaluation

Calibration of ultrasonic sensor and the measuring tank

The ultrasonic sensor performance was tested for linearity and hysteresis. The ultrasonic sensor's voltage output should be linearly correlated with the water level as the product description implies. Nonlinear correlation leads to inaccurate volume measurements. Moreover, the linear correlation should be the same path with water going up and down in the MT, which indicates no hysteresis. Ten points spread across the measurement range were used for a linear calibration. The approximate tank diameter was 7.62 cm, 1/10 of the sensor measurement range was 1.8 cm. The water volume to fill 1/10 of the measurement range was about 82 ml. Therefore, in order to have 10 points for calibration within the range, 80 ml of water measured by a 100ml volumetric cylinder was poured into the tank 10 times to get 10 corresponding water level readings. Similarly, 80ml of water were removed each time until the tank was empty. The ten points collected each path while water going up and down in the MT was checked for hysteresis. In Figure 2.4, the calibration data for the ultrasonic sensor are shown. The variance of signal readings at the same level when filling and removing water were less than 0.5% on average with a maximum of 1%, indicating negligible hysteresis. The R^2 of the ten data points and its linear regression line was >0.99, suggesting good linearity.

Also, in order to obtain an accurate cross section area (A) for volume determination, the calibrated cross section area of the tank was calculated by $A = \frac{V}{L}$ with sensor voltage outputs and corresponding water volumes inside MT at that time. Different ranges of voltage output were chosen for calculating the average cross section area of the MT which was 47.335 cm^2. This value was used in LabView® program to calculate the actual chamber and sample volumes.

System accuracy

The system accuracy was defined as the system volume measurement resolution. The resolution for the volume measurement of the system was calculated to be 0.045 ml, based on water level reading resolution and the cross section area of the measuring tank. The resolution of level readings were calculated using the specifications of the ultrasonic sensor (Resolution: 0.3mm; FS: 18cm/10V) and the National Instrument analog module (FS: 20V; Resolution: 16 bits, sampling No. per reading: 1000). However, other factors could also affect the actual volume accuracy including the water retained on the inner surface of the system. Thus, we further evaluated the overall system repeatability.

System repeatability

The system repeatability was tested by measuring the water input volume into the SC using glass marbles (approximately 100 ml) for 30 times. The glass marbles had a diameter of 0.671mm. System variability was determined by the

coefficient of variation for the thirty repeated measurements of the water input volume. The smaller the coefficient of variation among replicates, the higher the system repeatability was. The repeatability of the system was evaluated at three different times during usage (more than one month apart from each other) and checked for any drift.

The coefficient of variations (COV) of the multiple measurements for system variability evaluation is shown in Table 2.2. The average COV of the system was less than 0.9% and considered to be adequate for measuring the volume change in seeds.

Data reproducibility

The reproducibility of bean volume kinetics determined by B-VAT was evaluated at both 25 and 55 ºC. A 70 gram sample of navy cultivar C beans was placed into the system for soaking in d.i. water at 25 °C for 5 hrs. A 70 gram sample of black bean (cultivar C) was soaked in d.i. water at 55 °C for 5 hrs. Three replicates were taken for both tests. The volume measurements were conducted by the system at 90 seconds intervals. The beans were harvested in the fall of 2009 from ADM and then stored in a dry and cool condition until tested. Very small or split beans were removed prior to testing.

In Figure 2.5, the volume of navy beans soaking at 25 ºC measured at 1.5 min intervals for a period of ~ 6 hr is shown with the average values as dots and standard errors as bars. The volume change of black beans soaking at 55 ºC is

shown with the average values as dots and standard errors as bars in Figure 2.6. The coefficient of variation among the three replicates of the navy bean and black bean at different temperatures were 2% and 3%, respectively, suggesting a high data reproducibility given the highly variable biological samples.

System versatility

The water displacement principle of the system enables to measure volume change of most irregular shaped seeds (such as lentil, rice, corn seed, barley, faba bean) during hydration without shape approximation. With the high reproducible and reliable results the system can yield at a wide range of temperatures, the B-VAT system has a high potential application in food industries as an effective tool for improvement of processing condition parameters and hydration behavior classification or prediction on the basis of a good depiction of volume kinetics during hydration.

System feasibility

The system as a whole offers no difficulties in measuring volume kinetics at temperature ranging from 20 to 60 ºC according to the temperature rating of each system part. It is well known that temperature is the major leading factor that affects the hydration rate of foods. Usually a preferred soaking temperature for foods to have better quality and structure ranges from 40 °C to 55 °C (Maldonado

et al., 2010; Haladjian, et al., 2003). And the soaking temperature can easily be controlled and regulated via water baths (B1 and B2).

The water chemistry e.g., pH and additives for soaking seeds can be altered easily by changing the medium in B1. In previous researches, it is suggested the addition of salts in the hydration medium, the variance in pH of hydration medium and other chemical properties of the medium influence the seed hydration (El-Adawy et al., 2000; Kader, 1995; Nestares et al., 2001; Vadivel and Pugalenthi, 2008; Vijayakumari et al., 2007). The system allows study on effect of water chemistry of hydration medium on the volume kinetics of seeds during hydration.

The capacity of the B1 is 6 liters which is large enough for minimizing the change in chemical property of the hydration medium during testing. The current sampling size for beans can go up to 100 gram while 70 gram was determined to be sufficient in most cases. If needed, rings with the same diameter of the SC and a certain length made with CPVC materials can be added on top of the SC to increase the sampling capacity. The different sampling capacity of this system also enables it to be applicable for studying on various seeds during the hydration process.

The length of hydration process can theoretically be set to days and the minimum measuring interval as low as 1 minute, being more than enough for most application. In the evaluation tests, the 1.5 min measuring interval used can provide a detailed depiction of the hydration process under both room

temperature and elevated temperature. The quick connectors used in system allow faster assembly and disassembly of the system for maintenance.

Conclusion

The automatic volume determination system is able to provide a comprehensive, accurate and reproducible depiction of the volume kinetics of seeds during hydration under various controllable conditions with limited training and labor operations. The hydrating medium temperature can range from ambient temperature to 65 °C. Sampling capacity can be range from 50 grams to 150 grams. The minimum volume measurement interval is 1 minute which is more than enough for industrial purposes. System variation was proved to be less than 0.9% and result repeatability with a 70 grams sample load was less than 3%. It can potentially be used not just for measuring volume kinetics during soaking of legume seeds, but also for other seeds such as rice and corn.

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Appendix

Part I. Tables and figures.

Table 2.1 Required time for soaking various types of seeds at different temperatures

Variety	Time	Temperature	References
Chickpea	>10 hr ~ 3 hr	25 ºC 60 ºC	(Chenoll et al., 2009a; Sayar et al., 2001b)
Wheat	> 5 hr > 5 hr	20 ºC 50 ºC	(Maskan, 2002)
Lentil seed	>10 hr	RT	(Abousamaha et al., 1985}
Soybean	>14 hr ~ 5 hr	30 ºC 50 ºC	(Jiang and Zhang, 2005)
Sesame seed	> 6 hr ~ 3 hr	27 ºC 50 ºC	(Khazaei and Mohammadi, 2009)
Kidney bean	> 8 hr	30 ºC	(Tagawa et al., 2002)
Faba bean	> 20 hr > 14 hr	20 ºC 50 ºC	(Haladjian et al., 2003)

Table 2.2 Coefficient of variations of repeated system measurements of the water input volume

# test	# measurements	Mean (ml)	Standard deviation (ml)	COV (%)
1	30	200.53	1.43	7.1%
2	30	199.79	1.72	8.6%
3	30	199.10	1.81	9.1%

No. 1, 2, 3 are three individual tests of the volume of three different amounts of glass marbles at three different time.

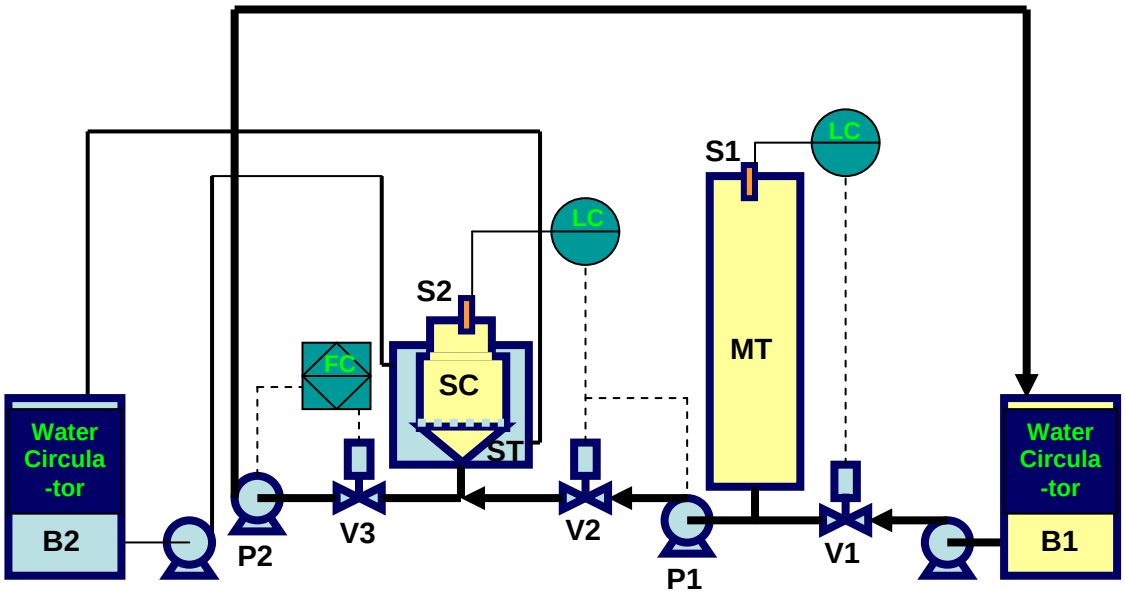


Figure 2.1.A. The major components of the physical device responsible for taking repeated volume measurements.

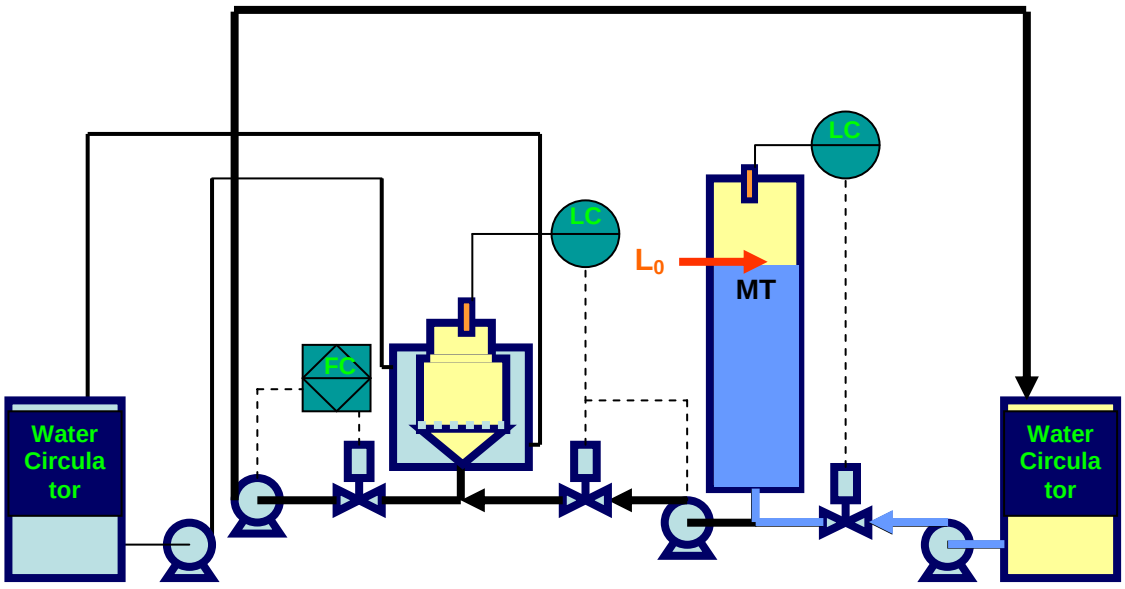


Figure 2.1. B. Water filled up measuring tank (MT) till preset level 0 (L0). Arrows show the incoming flow direction.

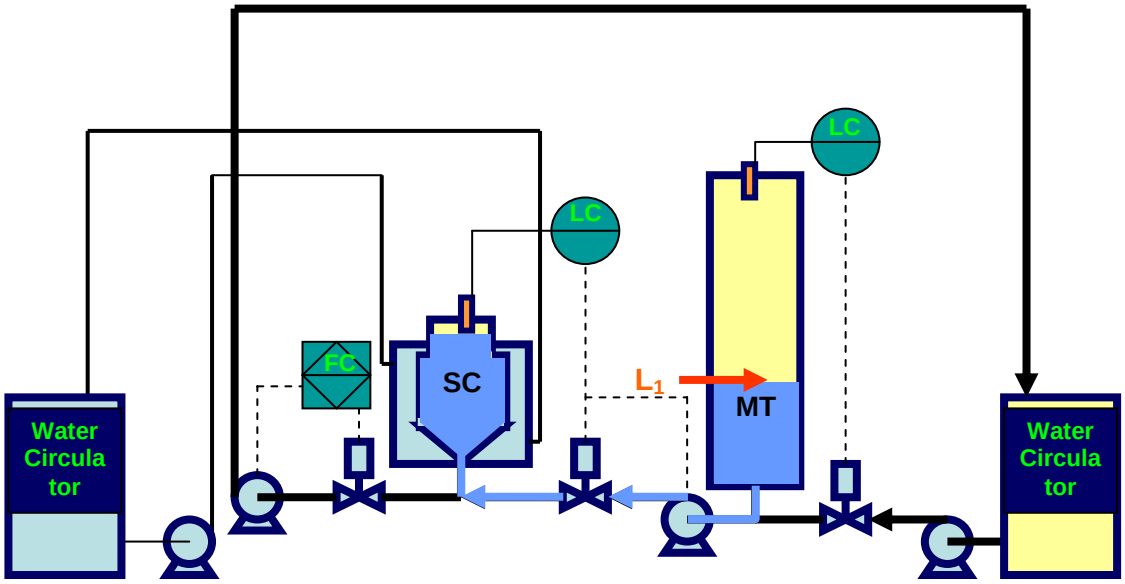


Figure 2.1. C. Water transferred to sample chamber (SC) till level of the S2 (Ls). The water level in measuring tank (MT) dropped from L0 to L1

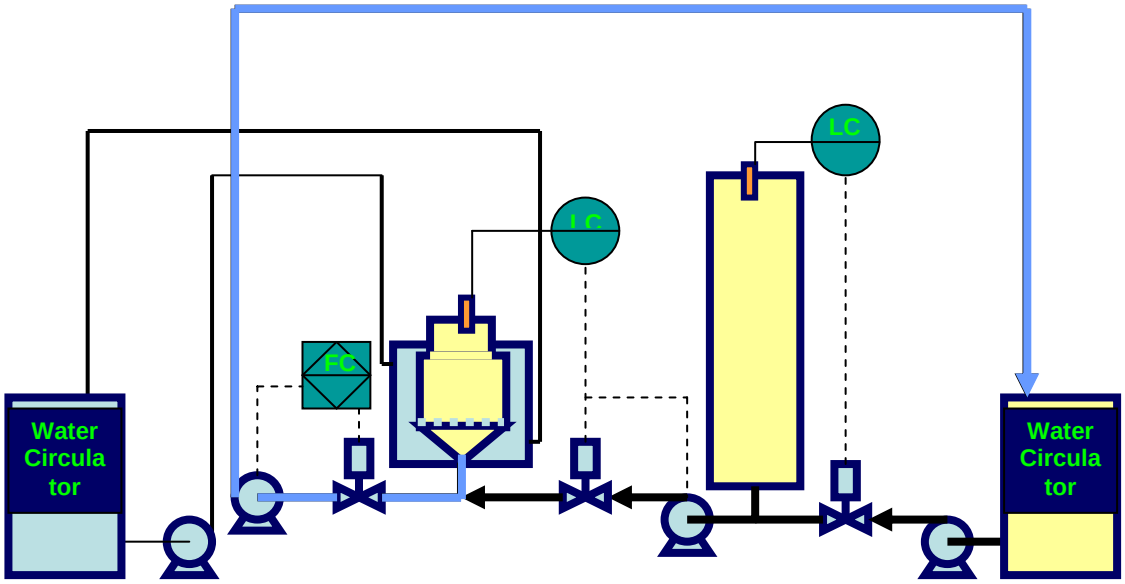


Figure 2.1. D. Water drained from sample chamber (SC).

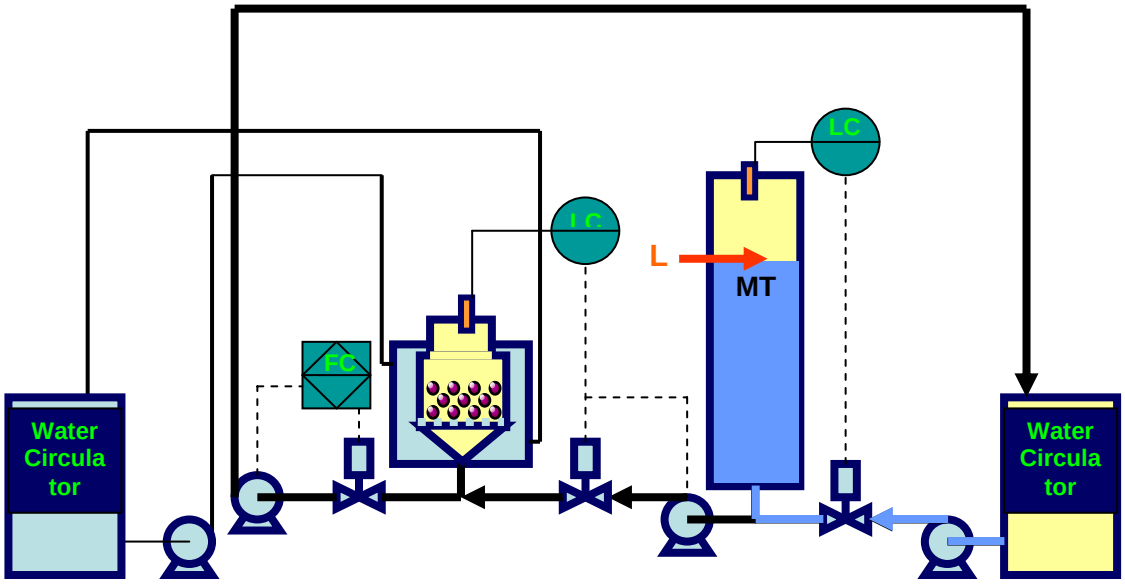


Figure 2.1. E. Water filled up measuring tank (MT) again to level L0 after seeds are loaded.

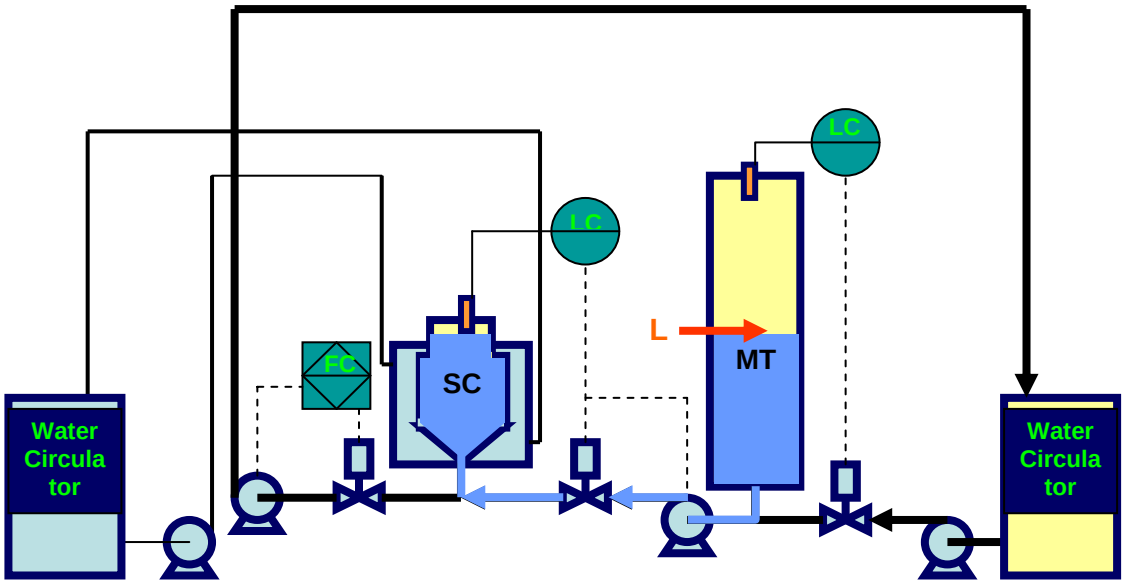


Figure 2.1. F. Water transferred to sample chamber until L3. Water level in water tank dropped from L0 to L2 (L1 shows the previous water level when samples are not present).

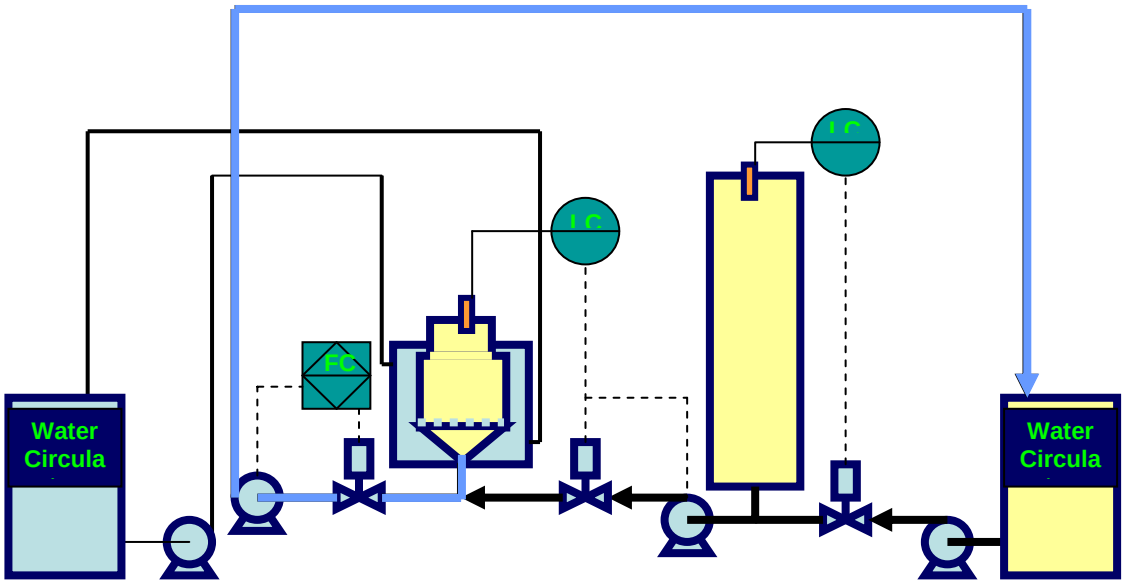
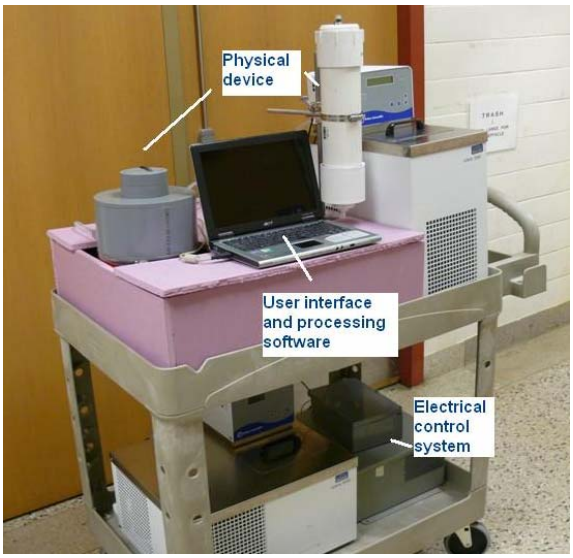


Figure 2.1.G Water drained from sample chamber (SC).



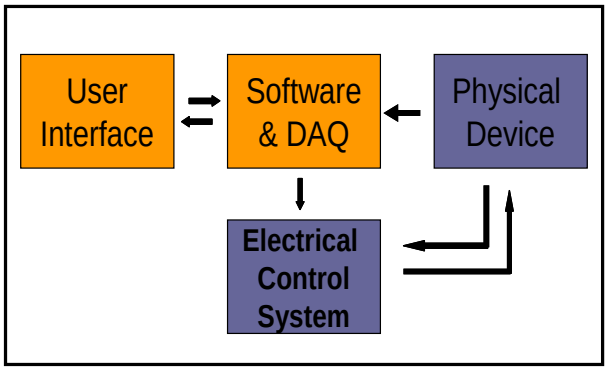


Figure 2.2 The picture of the system and its diagram showing main parts: physical device, electrical control system, software with DAQ and user interface.

FLOWCHART OF LABVIEW SOFTWARE

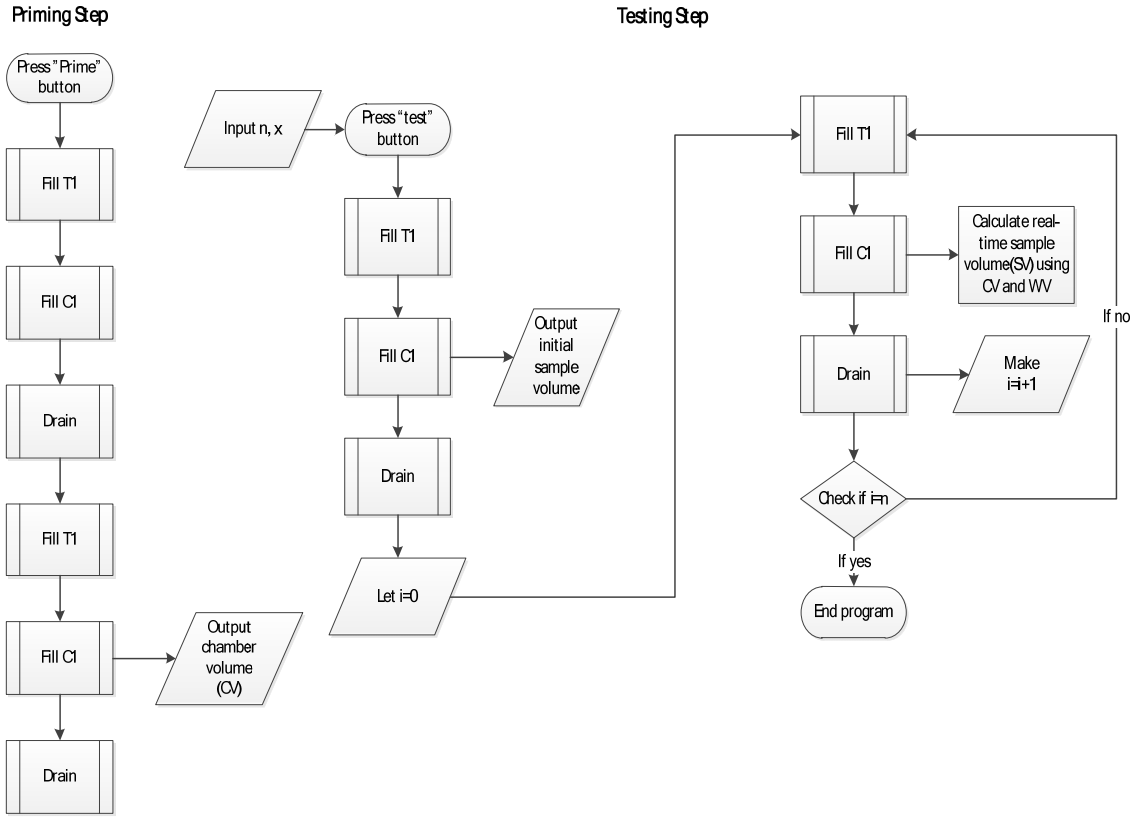


Figure 2.3 The flowchart of Labview program that automatic controls the repeated measurements over time.

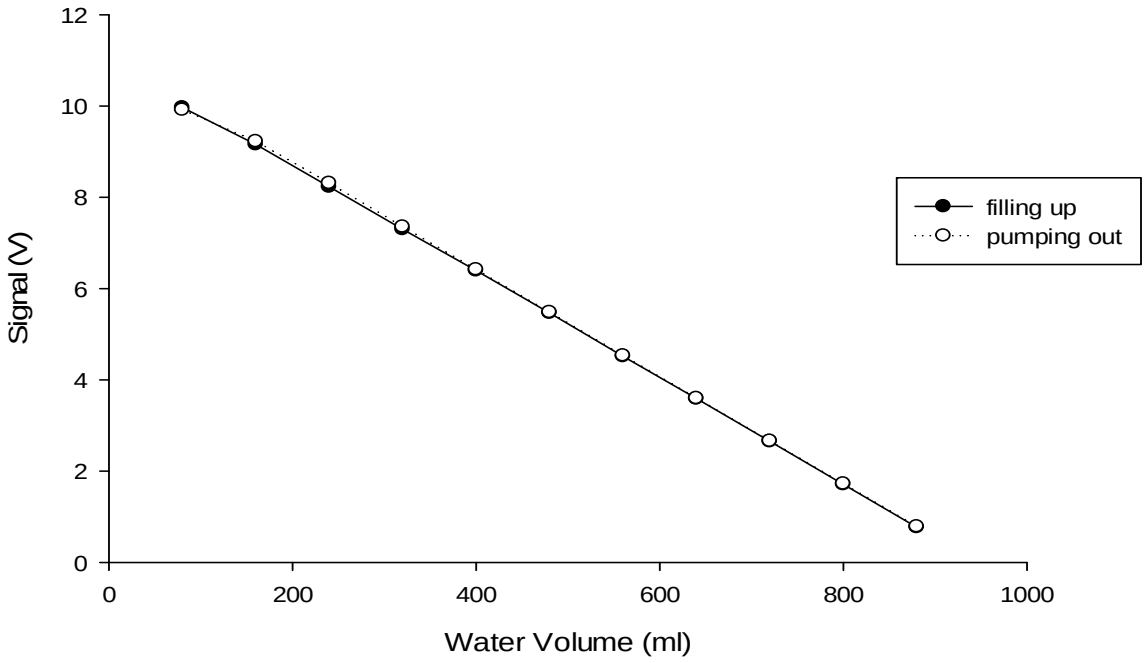


Figure 2.4 Calibration for sensor hysteresis and voltage-volume correlation

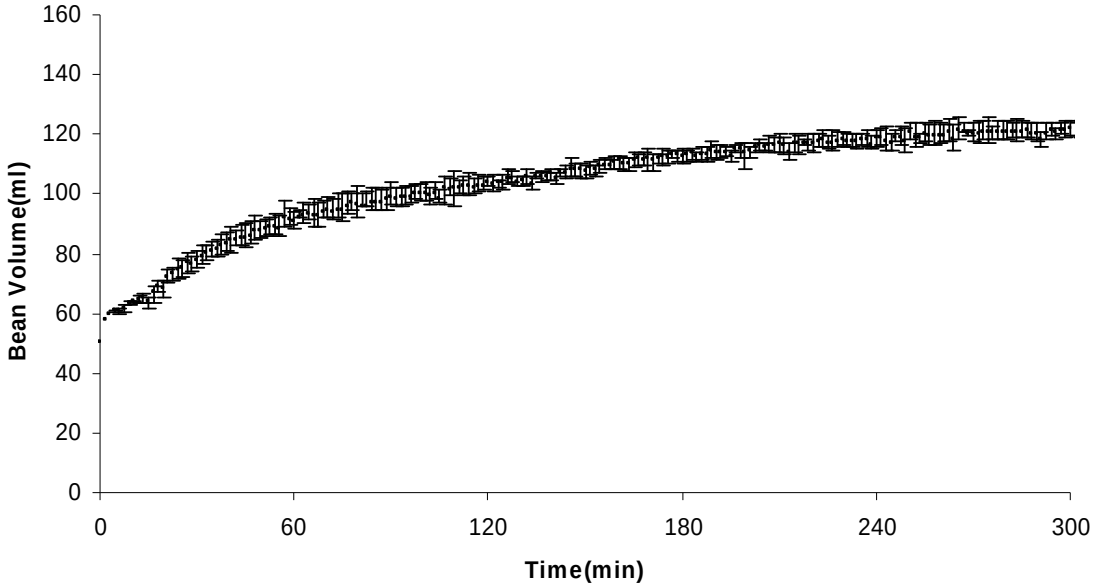


Figure 2.5. Volume kinetics of Navy cultivar C at 25 °C. Average volumes of volume are shown as dots and standard errors are shown as bars.

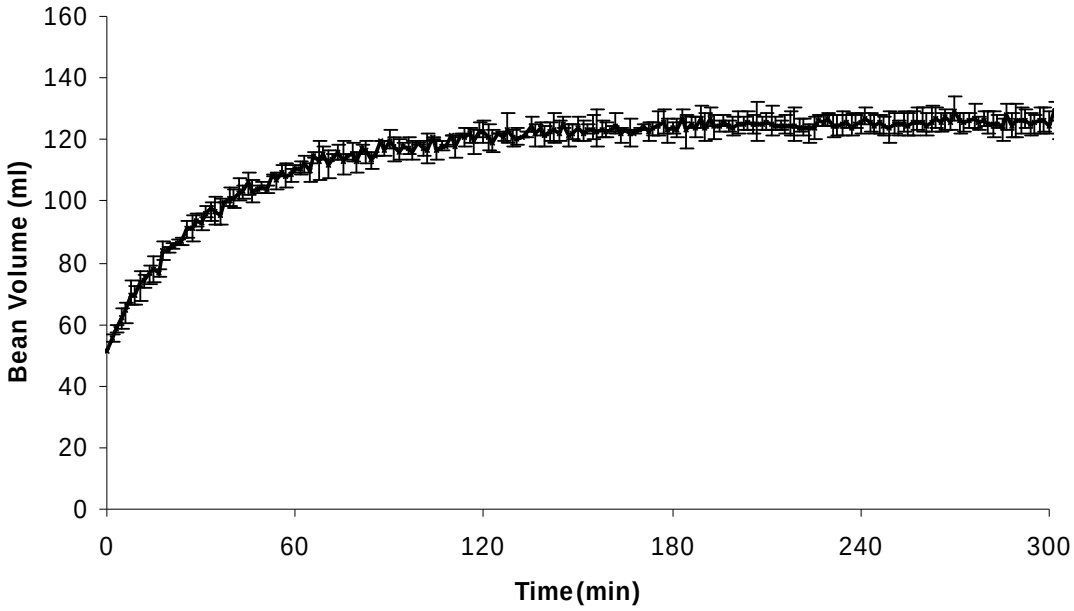


Figure 2.6 Volume kinetics of Black cultivar C at 55 °C. Average volumes of volume are shown as dots and standard errors are shown as bars.

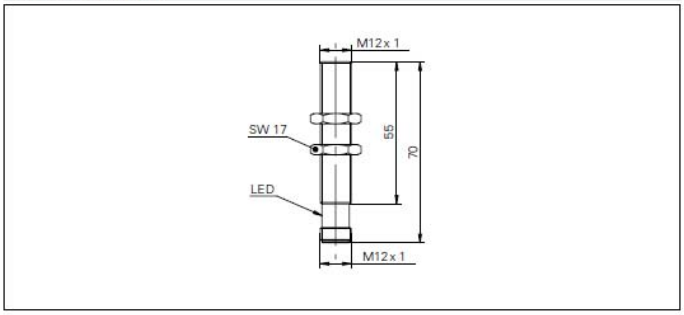
Part II Product catalog of the major parts of the system
Ultrasonic sensor

Baumer — Ultrasonic sensors

Ultrasonic proximity sensors with analog output

UNAM 12U9914/S14

dimension drawing



general data	
sensing range sd	20 ... 200 mm
scanning range close limit Sdc	20 ... 200 mm
scanning range far limit Sde	20 ... 200 mm
repeat accuracy	< 0,5 mm
resolution	< 0,3 mm
adjustment	external Teach-in
sonic frequency	380 kHz
response time ton	< 30 ms
release time toff	< 30 ms
alignment aid	target display flashing
light indicator	yellow LED / red LED
temperature drift	< 2 % of distance to target So

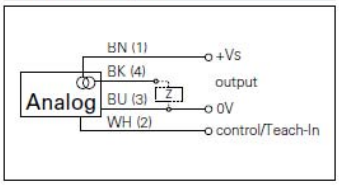
electrical data	
voltage supply range +Vs	15 ... 30 VDC
current consumption max.	35 mA
output circuit	voltage output
output signal	0 ... 10 V / 10 ... 0 V
output current	< 20 mA
residual ripple	< 10 % Vs
short circuit protection	yes
reverse polarity protection	yes

mechanical data	
type	cylindrical
housing material	brass nickel plated
width / diameter	12 mm
height / length	70 mm
connection types	connector M12

ambient conditions	
operating temperature	-10 ... +60 °C
protection class	IP 67

photo

connection diagram



- Analog voltage output 0 - 10 V

Optical level sensor

Honeywell

LLE Series

Liquid level sensors

DESCRIPTION

The enhanced series of liquid level sensors incorporates a photo-transistor trigger which provides a digital output that denotes the presence or absence of liquid.

The mode of operation is derived from the principle of total internal reflection. An LED and photo-transistor are housed within a plastic dome at the head of the device. When no liquid is present, light from the LED is internally reflected from the dome to the photo-transistor. When liquid covers the dome, the effective refractive index at the dome-liquid boundary changes, allowing some light from the LED to escape. Thus the amount of light received by the photo-transistor is reduced and the output switches, indicating the presence of liquid. This method of liquid level sensing is very fast, and almost instantaneous for water.

FEATURES

- Solid state technology
- Small size
- Digital output
- Pre-wired
- Electrically robust

BENEFITS

- Accurate, repeatable switching point
- Can be mounted in applications where space is limited
- Microprocessor compatible
- Easy to install, saving assembly time
- Reverse polarity, over voltage, short circuit and transient protection

TYPICAL APPLICATIONS

- Home appliances
- Spa baths
- Vending machines
- Food and beverage
- Medical
- Compressors
- Machine tools
- Automotive

ORDER GUIDE

		Catalogue Listing	
Description		**Standard temperature**	**High temperature**
Screw In, M12 Thread, Plastic	(Type 1)	LLE101000	LLE101101
	(Type 2)	LLE102000	LLE102101
	(Type 3)	LLE103000	LLE103101
Push In, Plastic	(Type 5)	LLE105000	LLE105100
Screw In, ⅛ in, Metal	Nickel plated brass	LLE205000	LLE205100
	Stainless steel	LLE305000	LLE305100

LLE Series

TECHNICAL INFORMATION

Specifications			
Operation mode		User defined single point on/off switch (Output is high in air)	
Repeatability (mm)		± 1	
Hysteresis (mm)		2 (dependant on liquid)	
Response time		Rising liquid level - 50 µs Falling liquid level - 1 s max (in ethanol) Response in other liquids dependent on viscosity	
Mechanical			
Mounting		Type 1 and 2 - mounted from outside; Type 3 and 5 - mounted from inside	
Termination		250 mm flying leads (180 mm for metal versions)	
		Blue 0 V	
		Red +5 V to +12 V supply	
		Green Output	
Material [Note 1]		Polysulphone	
Dimensions		**Plastic** **LLE101/102/103 Series**	**Metal** **LLE205/305 Series**
	Dome	3,5 mm radius (includes LLE105 Series)	
	Thread	M12x1	½ in BSPT
	Hex	19 mm	24 mm (See mounting drawings on page 3)
Environmental		**Standard temperature**	**High temperature**
Operating temperature (°C)		-25 to 80 (-13 °F to 176 °F)	-40 to 125 (-40 °F to 257 °F)
Storage temperature (°C)		-30 to 85 (-22 °F to 185 °F)	-40 to 125 (-40 °F to 257 °F)
Thermal testing		As per BS EN60068-2-33	
Humidity		As per BS EN60068-2-30	
Vibration		As per BS EN60068-2-6 Part S3: 1996	
Mechanical shock		As per BS EN60068-2-27 Part 2 Ea: 1987	
Pressure range (bar)		0 to 5 (plastic housing) [Note 2] 0 to 25 (metal housing)	
Ambient IR light limit (@ 940 nm) [Note 3]		10 mW/cm² in operation	
Electrical		**Standard temperature**	**High temperature**
Supply voltage (Vcc)		+5 Vdc to +12 Vdc ± 5 %	
Supply current (mA)		15 mA nominal @ +5 Vdc	5 mA nominal @ +5 Vdc
Output sink current [Note 4]		@ 25 °C 10 mA max.	@ 25 °C 40 mA max.
@ 5 Vdc supply		@ 80 °C 3 mA max.	@ 125 °C 7 mA max.

Notes:

[Note 1] Material compatibility information available on request.

[Note 2] Threaded sensors only.

[Note 3] For other ambient light environments the user should test the sensor under application conditions to verify compatibility.

[Note 4] The output is intended as a TTL compatible output signal, for interfacing to logic systems. For interfacing with other types of circuitry an appropriate buffer circuit must be used.

Drain pump

WATER SYSTEM PUMPS

Marine & Bilge

Automatic Marine/RV Potable Water Supply Pump

FLOJET Dayton

No. 2P366

No. 2XE90

No. 1P811

- 12VDC
- **Max. water temperature: 160°F**

Uses: For intermittent use on boats, camper-trailers bilge, or water system pump.

Note: Not for use with hydrocarbon-based or other chemicals.

FLOJET

Run-dry ability protects pump if water supply is lost. Permanent magnet ball bearing motor. Built-in pressure switch automatically turns pump on when pressure drops to 20 psi; turns off at 30 psi. Corrosion-resistant polypropylene with a Duplex Santoprene™ diaphragm and meet United States Coast Guard (USCG) Electrical Standards.

DAYTON

High-impact plastic and stainless steel (FDA Approved). Mounts in any position—submerged or in open air— as long as inlet is at or below fluid level. Cannot run dry and is not self priming.

Inlet (In.)	Outlet (In.)	Self-Priming To	GPH of Water @ Total Discharge Pressure* (psi) 0	10	20	30	Max. Pressure (psi)	Dim. (In.) L	W	H	Brand	Mfr. Model	Item No.		$ Each
3/8	3/8	6 Ft.	156	129	120	111	30	8⅛	3¼	3¾	Flojet	0210012C	2P366	✓	87.90
1/2	1/2	9 Ft.	174	156	132	114	50	9	6	4¾	Flojet	R3526144A	2XE90	✓	85.05
1/2	3/8	—	339	225	—	—	6.0	5⅛	3⅝	2⅝	Dayton	—	1P811		35.00

(*) To determine feet of head, multiply discharge pressure by 2.31.

Fill centrifugal pump

Little Giant 33BPLA 12V DC - Submersible or In-Line Utility Pump, 56" power cord (556001)

12 VDC Submersible or In-Line Utility Pump
Designed to fit into tight spaces, the BPLA utility pump delivers up to 350 GPH. Uses include boat bilges, campers, RV trailers, mobile homes, farm utility tractor cab coolers, bait tank water systems and many others.

Little Giant 33BPLA 12V DC - Submersible or In-Line Utility Pump, 56" power cord (556001) Features:
• 12 VDC, 56" Power Cord
• Operates submerged or in-line
• Inlet options include hooded and 1/2" male pipe thread
• Discharge is 3/8" male pipe thread (accepts 5/8" I.D. hose)
• Nylon volute and impeller
• ABS thermoplastic housing and cover
• Stainless steel motor shaft
• Buna-N shaft seal

Little Giant 33BPLA 12V DC - Submersible or In-Line Utility Pump, 56" power cord (556001) Specification:
• Flow: 350 GPH @ 1' of Head
• Cord Lenght: 56"
• Shut Off: 11'
• Voltage: 12VDC
• Amps: 2

Valves

CLARK SOLUTIONS

Model 1335, 2-Way, NC & NO Solenoid Valve

3/8 to 3/4" Pipe Size, Piloted, Combined & Direct Acting Solenoid

DESCRIPTION

Model 1335 two-way normally closed and normally open solenoid valves are available in forged brass or 316 stainless steel bodies. A variety of seal and seat materials including Acrylo-Nitrile, Neoprene®, Ethylpropylene, and Viton® satisfy many general industry applications.

Options include weather proof coils & housing, manual override and energized coil indicator light.

SPECIFICATIONS

GENERAL

Operation: Normally closed or normally open
Valve Body Materials: Forged brass, investment cast AISI 316 stainless steel
Diaphragm: Metal core with choice of seat material
Valve Seats: Acrylo-Nitrile, Neoprene®, Ethylpropylene, Viton®
Valve Life: > 1,000,000 cycles, field rebuild kits available
Connections: BSP or NPT
Operating Voltage: 12 VDC; 24 VDC/VAC; 120 VAC, 60Hz
Standard Solenoid Housing: Encapsulated coil, DIN 43650 connection (PG-9)
Optional Weather Proof Solenoid Housing: NEMA 4, IP65
Power Consumption: Class F coil to 80°C: 60 Hz, 13 W; DC, 19 W
Options: Manual operation, energized coil indicator light

Table 1

Wetted Materials						
Body	Plunger	Plunger Tower	Springs	Diaphragm	Inner-Diaph. Material	Piston
Brass	AISI 430F	304L or 305 SS	Copper	See Table 2	-	AISI 304
AISI 316	AISI 430F	304L or 305 SS	Silver & 302 SS	See Table 2	AISI 316	AISI 316

Table 2

Seat Material	Acrylo Nitrile	Neoprene®	Ethyl-propylene	Viton®
Maximum Temperature	+80°C	+80°C	+150°C	+150°C
Uses	Water, air, light oils, kerosene. Low and medium vacuum.	Oxygen, alcohol, argon, other non-corrosive light gases and liquids. Freon 12.	Water steam, hot water, acetone.	Benzene, naphtha, aromatics, etc.. Hot gases. High vacuum.

Connection (NPT or BSP)	Orifice Dia. (mm)	Cv Coef. (GPM)	Kv Coef. (m^3/h)	Weight (kg)
Brass Body, Pilot Operated, Normally Closed: Minimum Differential, 0.1 Bar; Maximum Differential Pressure, 10.0 Bar				
3/8"	14	2.75	2.35	0.76
1/2"	14	3.10	2.65	0.76
3/4"	18	5.03	4.30	0.98
Brass Body, Combined Acting, Normally Closed: Minimum Differential, 0 Bar; Maximum Differential Pressure, 7.0 Bar				
3/8"	14	2.75	2.35	0.76
1/2"	14	3.10	2.65	0.76
3/4"	18	5.03	4.30	0.98
Brass Body, Pilot Operated, Normally Open: Minimum Differential, 0.1 Bar; Maximum Differential Pressure, 10.0 Bar				
3/8"	14	2.75	2.35	0.76
1/2"	14	3.10	2.65	0.76
3/4"	18	5.03	4.30	0.98

Clark Solutions • 10 Brent Drive • Hudson, MA 01749 • Tel. 978 / 568 3400 • Fax 978 / 568 0060

Power supply

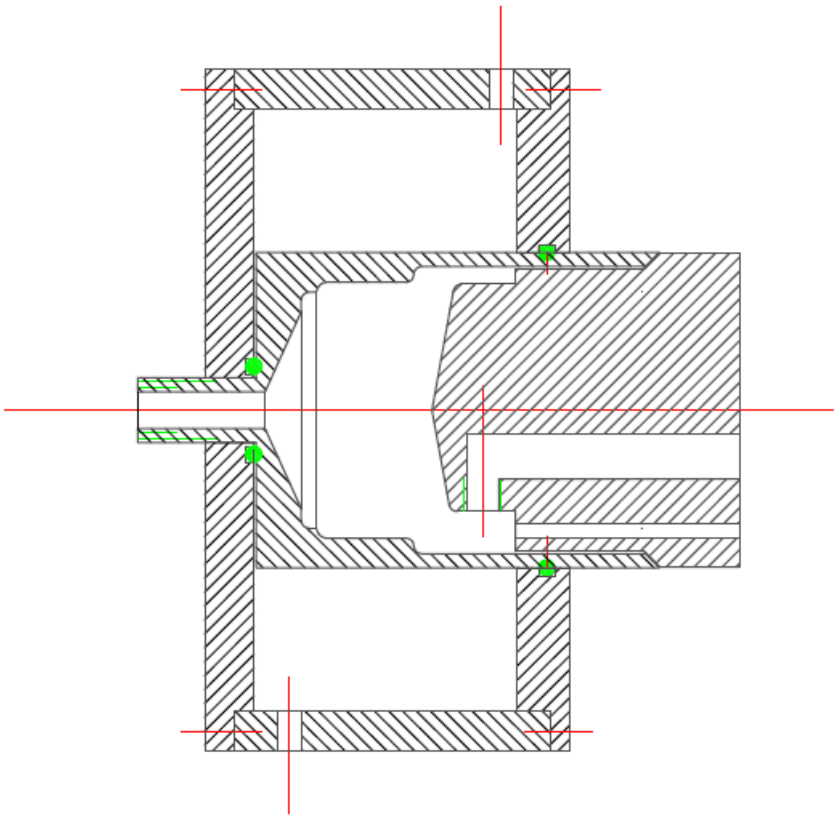
150W Single Output Series - with approval

. High reliability
. AC input range selected by switch
. Approvals: UL, CE
. Protections: over current / over voltage / short circuit
. 1 year warranty
. F615UL 199 x 110 x 50(mm)

AC input voltage range	88~132 / 176~264VAC selected by switch
Input frequency	47~63Hz
Inrush current	cold start, 20A/115V, 40A/230V
Input leakage current	< 0.7mA/230VAC
Line regulation (full load)	<= 0.5%
Output voltage adjust range	+ - 10%
Output over current protection	110~130% current limiting, auto recovery
Output over voltage protection	115-150%
Withstand voltage	I/P -O/P: 3.0KVAC/1min
	I/P -F/G: 1.5KVAC/1min
	O/P-F/G: 0.5KVAC/1min
Rise time	50ms @ full load (typical)
Hold up time	20ms @ full load (typical)
Operating temp. & humidity	-10°C ~ +50°C, 20%~93% RH (non condensing)
Storage temp. & humidity	-20°C ~ +85°C, 20%~95%RH (non condensing)
Safety standards	GB4943, UL60950, EN60950
EMC standards	GB9254, EN55022 class B
	EN61000-3-2,3, EN61000-4-2,3,4,5,6,8, 11
Cooling method	convection

Model	DC Output		Load regul.	R&N	Effiency
PS1-150W-5	5V	30A	0.5%	120mV	78%
PS1-150W-7.5	7.5V	20A	0.5%	120mV	80%
PS1-150W-9	9V	16.7A	0.5%	120mV	80%
PS1-150W-12	12V	12.5A	0.5%	120mV	82%
PS1-150W-24	24V	6.5A	0.5%	150mV	85%
PS1-150W-28	28V	5.5A	0.5%	100mV	85%
PS1-150W-36	36V	4.2A	0.5%	200mV	86%

Part III AutoCAD drawing of the SC with ST.



CHAPTER III
EFFECT OF VARIETY, CULTIVAR AND TEMPERATURE ON VOLUME KINETICS OF PINTO, NAVY AND BLACK BEANS DURING SOAKING

Abstract

Common beans are one of the most highly planted and consumed legumes in the world, especially in the Central and South America. The industrial processing of beans include a soaking step prior to cooking in order to reduce the cooking time required and enhance the nutritive quality of the final product. The change in volume over time is a good indicator of soaking behavior because it is proportional to the amount of water absorbed by the seeds. In this study, differences in volume kinetics among cultivars of three common bean varieties: pinto, navy and black during soaking at 25 ºC and 55 ºC for a 5 hr period were investigated using the B-VAT system. Among the three bean varieties, pinto showed the slowest soaking at both temperatures and had unique volume change patterns. Within all varieties, significant differences were observed among cultivars at both temperatures ($p<0.01$). Pinto cultivar B, black cultivar A behaved as “outliers” because of their distinct hydration patterns. As temperature increased, all cultivars had faster changes in volume (at an initial rate up to 6.8 times faster) and larger end relative volumes (up to 113% more). Temperature had the greatest impacts on improving soaking of pinto cultivars and the least impacts on navy cultivar C and cultivar D.

Introduction

The common bean (*Phaseolus vulgaris*) , one of the most consumed legume species, is one of the most important diet supplements for protein and dietary fiber in Central and South America, especially in Mexico and Brazil (Broughton et al., 2003). Common bean include varieties navy, pinto, red kidney, black, pink, and French. Navy and pinto bean are the two most produced varieties in common bean, consisting of more than 60% of the total production of common bean in the world (Ruth, 1989). The pinto bean, also called molted bean due to its appearance, are traded and consumed heavily in southwest United States and Mexico, taking 30% of the whole production of common bean in the world. The navy bean, also called white bean or pea bean, is the most popular common bean and accounts for 33% production of common bean. It is very popular in Britain and US, and is mainly used for making baked beans. The black bean, famous for its meaty texture and rich antioxidant content, is also a fundamental part of the diet in Latin America.

A soaking step is usually necessary for processing dry beans; because the antinutrients contained in dry beans, i.e., hemagglutinin, trypsin inhibitors and phytate substances leach out during soaking and enhance the nutritive value of the product. Another reason is to introduce water evenly into beans prior to cooking to save the time and energy required for cooking. Normally the soaking step for common beans to equilibrium at room temperature could range from 8 to 18 hr, becoming an economic burden for manufacturers. In order to process dry beans more efficiently, studies have been done to

investigate factors that could shorten the soaking time of faba bean, kidney bean and soybean (Abousamaha et al., 1985; Aparna et al., 2000; Chenoll et al., 2009b; Haladjian et al., 2003; Jiang and Zhang, 2005; Kader, 1995; Tagawa et al., 2002).

Temperature is one of the key factors affecting the hydration process of beans, and has been heavily studied in many food materials, such as legume seeds (Haladjian et al., 2003; Sayar et al., 2001a; Turhan et al., 2002), cereals (Cunningham et al., 2007; Muramatsu et al., 2006) and dehydrated fruits (Maldonado et al., 2010; Marques et al., 2009). As temperature increases, the water absorption rate also increases, but extreme temperature (> 50 - 60°C) will cause membrane denaturation or structure disruption, leading to less water diffusivity and lower holding capacity (Haladjian et al., 2003; Maldonado et al., 2010; Sayar et al., 2001a). Only a few researchers also investigated the impact of temperature on volume development (Chenoll et al., 2009b; Khazaei and Mohammadi, 2009; Muramatsu et al., 2006). However, few reports were found depicting the volume kinetics of pinto, navy or black beans during soaking, despite or their broad utilization. Also, differences in hydration behavior among bean varieties and cultivars have not been thoroughly investigated.

During soaking, volume expansion changes significantly in dry beans and as water is absorbed, beans expand up to more than 2.5 times of their original volume. Volume is an important quality parameter which is also indicative of water uptake during hydration (Steffe and Singh, 1980). It is also cost related, critical for processing design and key to the aesthetic quality of product which

directly affect the consumers' preference. A comprehensive depiction of volume change is able to provide a better understanding of the soaking process in view of water uptake and volume development. However, the current volume determination methods, ranging from image analysis (Shahin et al., 2006) or manual measurement of length and width, approximating the shape of beans to simplify calculation of volume (Bayram et al., 2004; Khazaei and Mohammadi, 2009), to using Archimedes principle (Moreira et al., 2008) are either labor intensive or lack of accuracy and repeatability to adequately describe the volume kinetics during soaking. Hence, the automatic system for measuring volume kinetics which was introduced in the previous chapter was adopted in order to capture the differences in volume development pattern among variety, cultivar and temperature treatment groups in detail.

The objective of this study was to study how pinto, navy and black bean differ in volume kinetics among varieties and cultivars at two different temperatures.

Materials and Methods

Materials

Six cultivars of pinto beans (cultivar A, cultivar B, cultivar C, cultivar D cultivar E and cultivar F), five cultivars of navy beans (cultivar A, cultivar B, cultivar C, medalist and cultivar E), and three cultivars of black beans (cultivar A, cultivar B and cultivar C) harvested and then dried in fall season, 2009,

were provided by AMD's facility in St. Thomas, ND and used for this experiment. Beans were stored at a dry place in ambient conditions.

Volume kinetics determination during soaking

70 grams of each cultivar of beans were weighed after removing the split beans and then put into B-VAT system as described in the previous chapter to determine volume kinetics at both 25 ºC and 55 ºC with at least two replicates. The volume measurements were taken by the system automatically at 1.5 min intervals for at least 5 hrs. Recirculated deionized water was used as the soaking medium.

Initial moisture content of each cultivar was tested using a Motomco Moisture Meter (Model 919). 250 grams of beans were weighed and placed in the dump cell in the moisture meter, the measure button was then pressed. The sample automatically fell into the test cell containing the temperature-sensing probe accurate to 0.5ºC. Within seconds, the tester displayed the percentage of moisture. The variability of the moisture measurements is ±0.05% and for weight measurement is ±0.02%.

Data analysis

The experimental data were fitted by Peleg model to calculate the initial water uptake rate for each cultivar at each temperature. Also, the time to reach 2 times the original volume and the end relative volume were selected as indices to characterize the soaking behavior. The experiment was conducted with CRD. The differences among varieties, cultivars and

temperature treatments on the selected hydration indices were tested using ANOVA and Tukey test by SAS 9.0 (SAS Institute, 2009)

Results and Discussions

Initial moisture content

The average value of the moisture content of each bean cultivar before hydration test were listed in Table 3.1. The initial moisture content of the beans varied in the range of 8% to 9.5% except for Black cultivar B. Although their initial moisture contents were relative low, no obvious differences in soaking behavior were observed even for Black cultivar B.

Effect of variety and cultivar

Differences of the volume development pattern of pinto navy and black cultivars hydrated at both room temperature and elevated temperature were observed, as shown in Figures 3.1 – 3.6 , respectively. Relative volume, y axis variable in the figures, represents the ratio of current volume of beans to the initial volume of beans. The three indices introduced to characterize the hydration behavior are listed in Table 3.2. With these three indices, the hydration patterns of different samples became more comparable in how they start, develop and end. The initial rate was calculated as the reciprocal of parameter k_1 , that was obtained from Peleg model as Eq. (1).

$$\frac{V_t}{V_0} = 1 + \frac{t}{(k_1 + k_2 * t)} \qquad \text{--------（1）}$$

The k_1 and R^2 of fitting Peleg model for each cultivar at both temperatures were shown in Table 3.3. Generally, at 25 ºC, pinto beans hydrated much slower than the other two varieties, showing a linear increase pattern of volume kinetics, suggesting that the water transfer was not diffusion dominated motion but with an external resistance. The initial water uptake rate of pinto beans and the end relative volume after a 5.5 hr soaking process are significantly smaller than that of navy and black beans ($p<0.01$), and the times needed to reach $2V_0$ were much longer ($p<0.01$). Among cultivars, difference were also observed for initial rate and end relative volume ($p<0.01$). Due to the prolonged time for pinto beans to expand to one size bigger, the indice for time did not have a accurate value and thus was not used as part of the statistic analysis. Pinto cultivar B hydrated at a rate of 0.161 ml/ml/hr, significantly higher than all other cultivars ($p<0.01$). Pinto cultivar F hydrated significantly faster than pinto cultivar D and pinto cultivar C and pinto cultivar E significantly faster than pinto cultivar C ($p<0.01$). The end relative volume of pinto cultivar B after soaking for 5.5 hrs are significantly higher than all other cultivars, with a ratio of 174%. The expansion of volume of pinto cultivars after soaking 5.5 hr at room temperature could go down to 125%. Moreover, most of the pinto cultivar did not fully hydrated after 16 hr hydration except for cultivar B. Apparently for pinto bean, it does not make any sense to use room temperature for its hydration. Navy beans have a different hydration pattern from pinto beans, a quadratic increase curve with initial water uptake rate much higher than later. Generally, navy beans hydrated much faster than pinto beans at room temperature, approaching equilibrium in about 6 hr.

Among navy cultivars, all the three indices as listed in Table 3.2, were significantly different. Cultivar C had the highest initial rate at 0.924 ml/ml/hr, significantly higher than cultivar A, cultivar B and cultivar E ($p<0.01$). Cultivar cultivar D showed a statistically significant higher rate than cultivar B and cultivar E, the two that initially hydrated slowest ($p<0.01$). However, cultivar A and cultivar E had the longest time to reach $2V_0$, approximately 7.5 hr while cultivar C took the shortest time of 2.5 hr ($p<0.01$). Again, cultivar C has the biggest end relative volume after soaking ends, increased to 2.3 times of the initial volume whereas cultivar A and cultivar E expanded least, only to less than 2 times of their initial volumes. Black beans, had volume kinetics pattern more closer to navy beans at room temperature. However, cultivar A had a linear increase pattern of volume that is different from the other two cultivars with quadratic increase patterns. The initial rate among black cultivars were significantly different, while the fastest being black cultivar B, followed by cultivar C, than cultivar A. The other two indices were not significantly different among black cultivars ($p>0.05$).

At 55 ºC, differences among cultivars for all three varieties were still observed. For pinto beans, cultivar B still hydrated significantly higher than other cultivars ($p<0.01$), at a rate of 0.58 ml/ml/hr. Cultivar E took the longest time to reach $2V_0$, which was about 4.5 hr, and cultivar B took significantly less time than cultivar A, cultivar D and cultivar E ($p<0.01$). The end relative volume of cultivar A was the biggest while cultivar E was the smallest. It suggested that the faster initial rate of moisture uptake does not necessarily link to a bigger water absorption amount at the end of soaking process. For

navy beans, cultivar C still hydrated the fastest at a initial rate of 5.219 ml/ml/hr ($p<0.01$). Cultivar cultivar E and cultivar A took significant longer time to reach $2V_0$ than the other cultivars ($p<0.01$). And cultivar C hydrated up to 237% of initial volume, significantly higher than cultivar A of a 214% end relative volume. Black was the most homogeneous variety where only the initial rate was significantly different while cultivar A being the slowest cultivar ($p<0.01$). The previous reported hydration behavior of legume seeds all showed a quadratic increase pattern, close to most of navy and black cultivars, while the linear increase pattern of pinto beans has never been reported, even at room temperature (Haladjian et al., 2003; Khazaei and Mohammadi, 2009; Sopade and Obekpa, 1990). To sum up, navy and black beans generally hydrated well at room temperature or elevated temperature but pinto beans hydrated much slower although they do not have a smaller water holding capacity. Differences among cultivars were observed for all varieties at both temperatures. The very distinct behavior of pinto beans revealed that they had a much bigger resistance than the other two varieties. The differences among bean varieties and cultivars in soaking behavior could be attributed to their differences in chemical compositions such as the content and structure of starch and seed coat properties (Kader, 1995; Vega-Galvez et al., 2009).

Effect of temperature

When increasing temperature to 55 °C, the volume change pattern of all bean cultivars changed significantly: the volume expansion rate increased so did the ending volume, as shown in Figures 3.7-3.20. This finding was in

agreement with some other previous studies (Khazaei and Mohammadi, 2009; Prasad et al., 2010). However, the effect of temperature on volume development changed differently depending on the variety and cultivar. The three indices that were used to characterize the volume kinetics curve for all cultivars at 55 ºC were listed in Table 3.2 as well. The volume kinetics pattern of most pinto cultivars changed to a three-phase volume development pattern at elevated temperature, differing from other two varieties. There were five cultivars (except cultivar B) having a distinct initial lag phase up to 150 min before entering the rapid water uptake phase which the other two varieties started immediately upon soaking. The initial water uptake rate increased with a range of 1.32 to 4.36 fold, compared to that at room temperature. There followed a rapid water uptake phase and then the phase when volume approaching equilibrium which resembles the behavior of navy and black beans. The end volume after 5 hr reached a similar range as that of navy and black beans at 55 °C. However, the initial rate were still significantly less than other two varieties, while the time to reach 2V being significantly longer than the other two varieties ($p<0.01$). For navy and black beans, the effect of temperature on them was similar, showing as the increase in initial rate up to 6.56 fold for navy cultivar B and 6.84 fold for black cultivar A, a decrease in time to reach $2V_0$ by 100 to 360 min, and a slight increase in the end relative volume (less than 13%). Differing from pinto beans, the increase in initial rate were at least 3.5 fold and most of all around 5 fold. The effect of temperature for navy and black beans to shorten the time required to reach $2V_0$ and increase the end relative volume were much lower than those of pinto beans.

Clearly, increasing temperature improves the hydration efficiency for all three varieties but at different level for different cultivars. It is necessary for pinto beans due to their very slow water uptake behavior at lower temperature. But for cultivars such as navy cultivar C, which hydrate quickly at room temperature, the temperature treatment became less energy-efficient.

Conclusions

Hydration behavior differs among bean varieties and cultivars. Pinto beans have an initial water uptake rate more than 5 times slower than navy and black beans, showing special resistance to initial water absorption. However, their behaviors at elevated temperature suggested that pinto beans have a similar water holding capacity as navy and black beans. Within varieties, some cultivars behave as “outliers” compared to other cultivars at same soaking conditions. Examples include the linear swelling pattern for black cultivar A at room temperature compared to the quadratic increase pattern for other black cultivars and the lack of initial lag phase and much higher water uptake rate for pinto cultivar B compared to other pinto cultivars.

Increasing temperature from 25 to 55 °C enhanced the initial water uptake and the end relative volume for all bean cultivars after 5 hrs soaking, but with different increments. It changes the hydration curve of pinto beans from linear increase pattern to a three phase increase pattern. The time to reach $2V_0$ for some pinto cultivars were shortened up to more than 15 hr whereas less than

2 hr for some navy cultivars. Pinto beans need the higher soaking temperature to assure the efficiency of hydration than navy and black beans.

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Appendix

Table 3.1 Initial moisture content of all bean cultivars

Bean cultivar	Moisture content (%)
Pinto cultivar A	9.53
Pinto cultivar B	9.09
Pinto cultivar C	8.87
Pinto cultivar D	9.53
Pinto cultivar E	9.19
Pinto cultivar F	9.09
Navy cultivar A	8.64
Navy cultivar B	8.75
Navy cultivar C	8.06
Navy cultivar D	8.87
Navy cultivar E	9.09
Black cultivar A	8.23
Black cultivar B	13.80
Black cultivar C	8.23

Table 3.2 Effect of temperature on volume kinetics of all bean cultivars

	Initial rate, ml/ml/hr		Time to reach $2V_0$, min		End relative volume	
	25 °C	**55°C**	**25 °C**	**55°C**	**25 °C**	**55°C**
Pinto cultivar A	0.0585	0.238	>1000	190	132%	245%
Pinto cultivar B	0.161	0.58	700	122.5	174%	233%
Pinto cultivar C	0.0385	0.168	>1000	157.5	125%	229%
Pinto cultivar D	0.0514	0.171	>1000	200	142%	232%
Pinto cultivar E	0.071	0.094	>1000	267.5	128%	219%
Pinto cultivar F	0.075	0.136	>1000	165	127%	230%
Navy cultivar A	0.451	2.16	465	105	192%	214%
Navy cultivar B	0.383	2.512	305	65	204%	216%
Navy cultivar C	0.924	5.219	150	35	230%	236%
Navy cultivar D	0.702	3.399	222.5	52	214%	220%
Navy cultivar E	0.22	0.985	435	135	186%	234%
Black cultivar A	0.2	1.369	327.5	85	200%	231%
Black cultivar B	0.871	3.042	265	62.5	214%	233%
Black cultivar C	0.58	2.902	217	60	205%	230%

Table 3.3 k1 and R-square of Peleg model fitting the initial phase of different cultivars at two temperatures

	25 ºC		55 ºC	
	k1 (min/ml/ml)	R-square	k1 (min/ml/ml)	R-square
Pinto cultivar A	1026.68	0.984	252.1008403	0.981
Pinto cultivar B	373.51	0.994	103.4482759	0.994
Pinto cultivar C	1560.96	0.98	357.1428571	0.986
Pinto cultivar D	1168.32	0.979	350.877193	0.981
Pinto cultivar E	854.07	0.97	638.2978723	0.985
Pinto cultivar F	804.35	0.98	441.1764706	0.989
Navy cultivar A	136.04	0.995	27.77777778	0.986
Navy cultivar B	156.66	0.994	23.88535032	0.995
Navy cultivar C	65.94	0.997	11.49645526	0.992
Navy cultivar D	86.47	0.995	17.65225066	0.99
Navy cultivar E	276.73	0.993	60.91370558	0.993
Black cultivar A	301.75	0.995	43.8276114	0.987
Black cultivar B	68.89	0.994	19.72386588	0.99
Black cultivar C	105.45	0.995	20.67539628	0.985

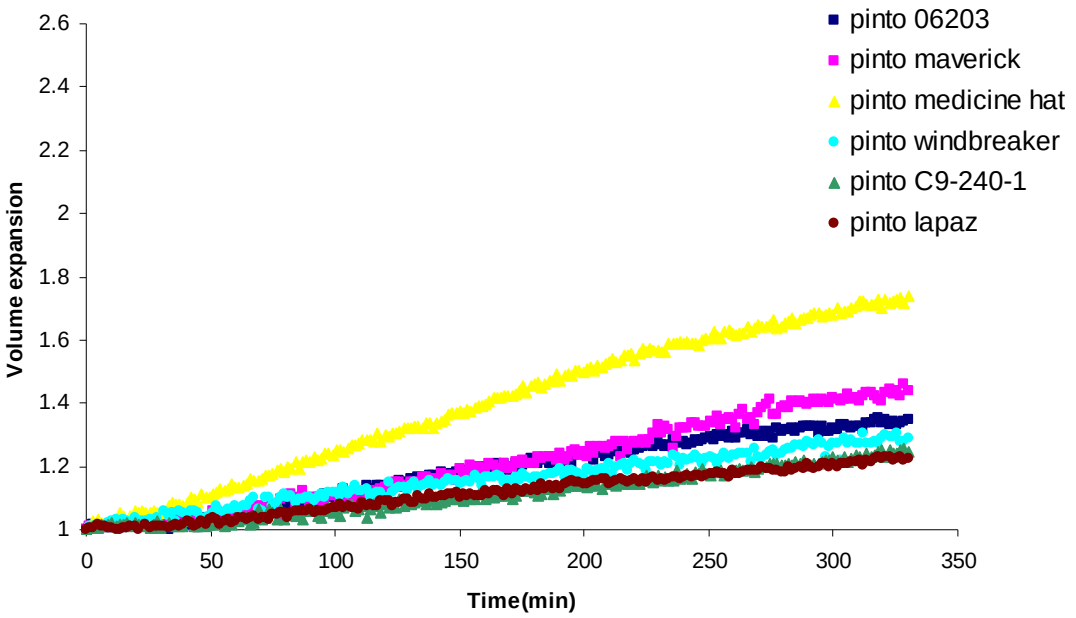

Figure 3.1 Volume kinetics of pinto beans at 25 °C

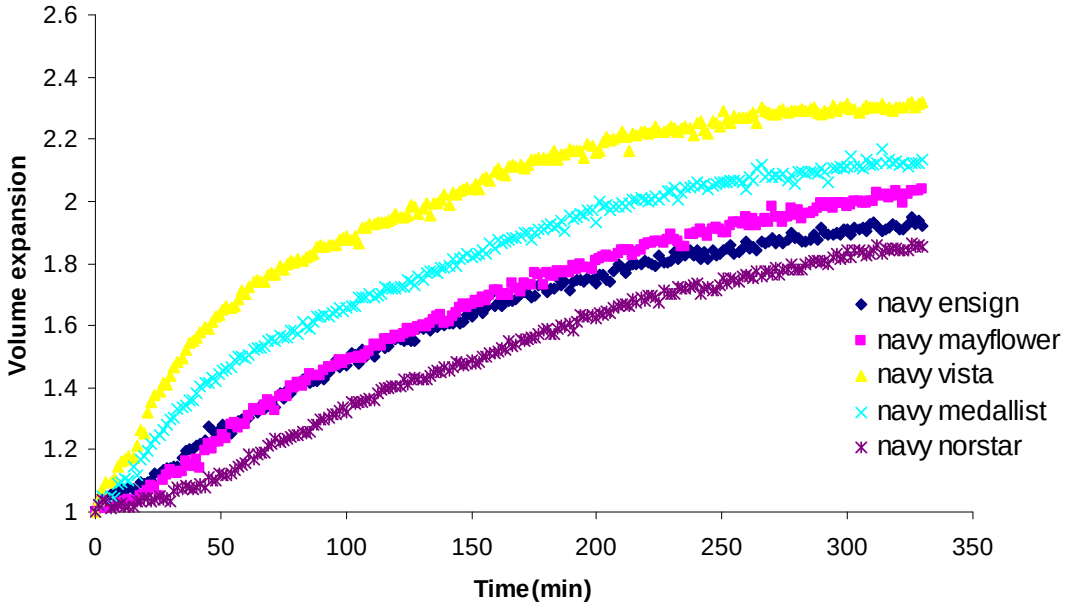

Figure 3.2 Volume kinetics of navy beans at 25 °C

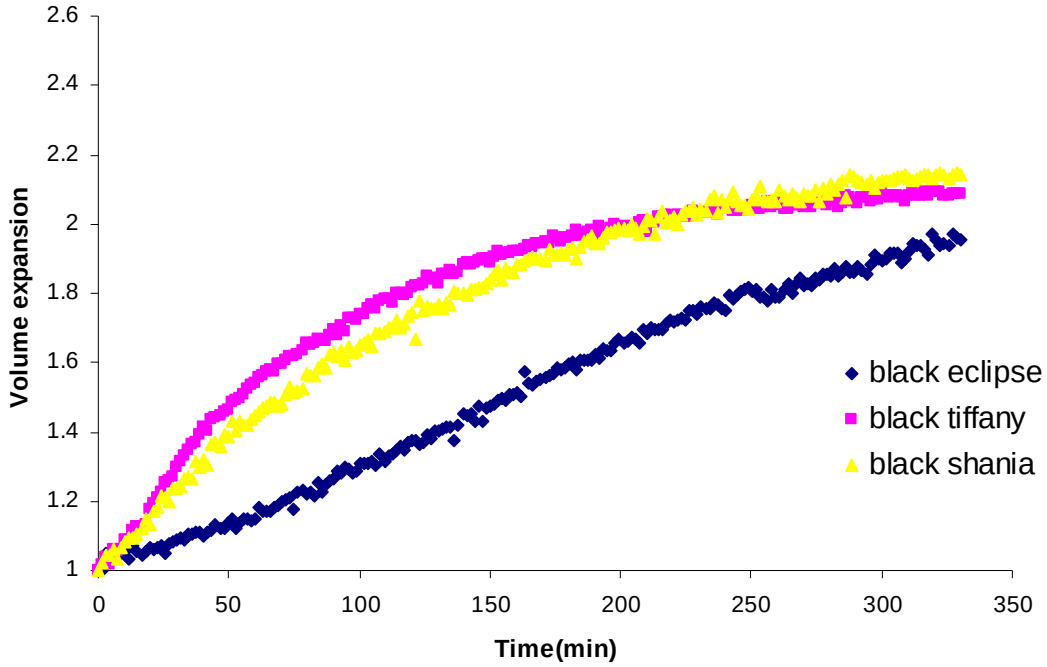

Figure 3.3 Volume kinetics of black beans at 25 °C

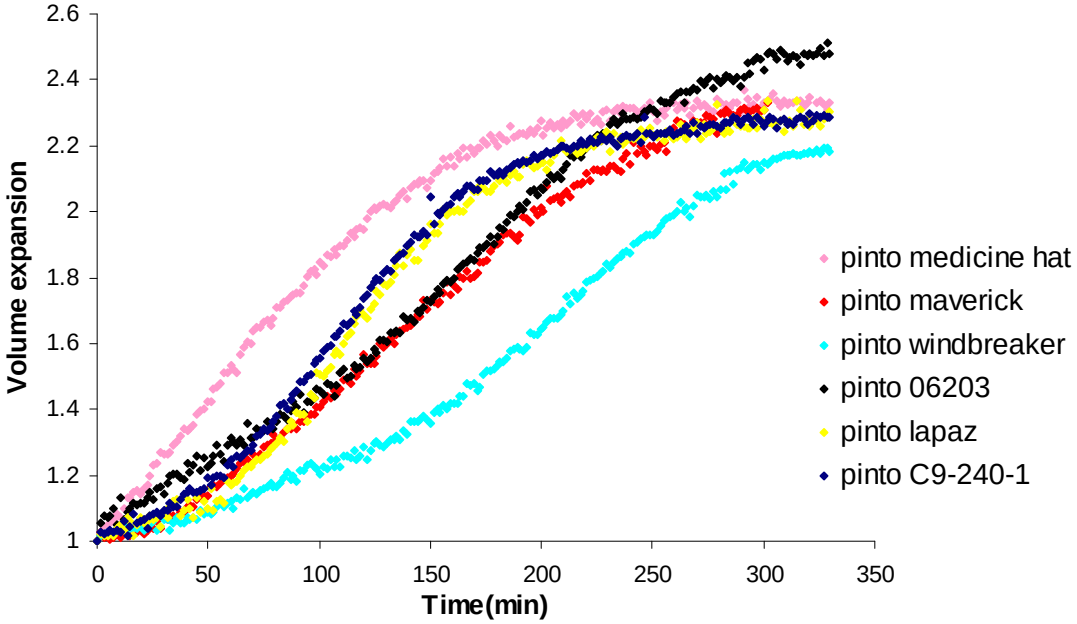

Figure 3.4 Volume kinetics of pinto beans at 55 ºC

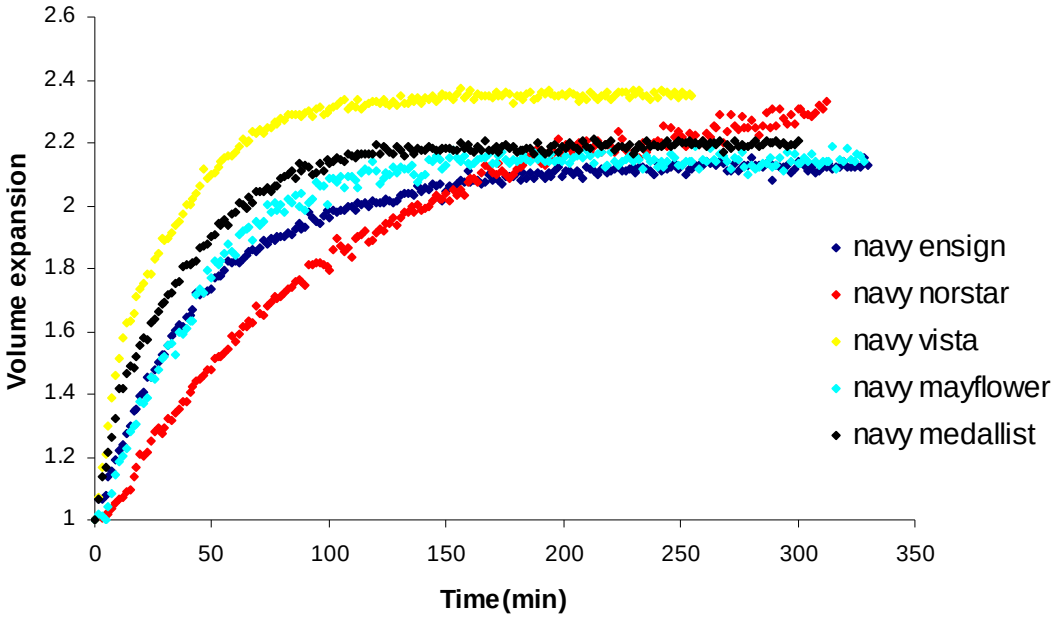

Figure 3.5 Volume kinetics of navy beans at 55 ºC

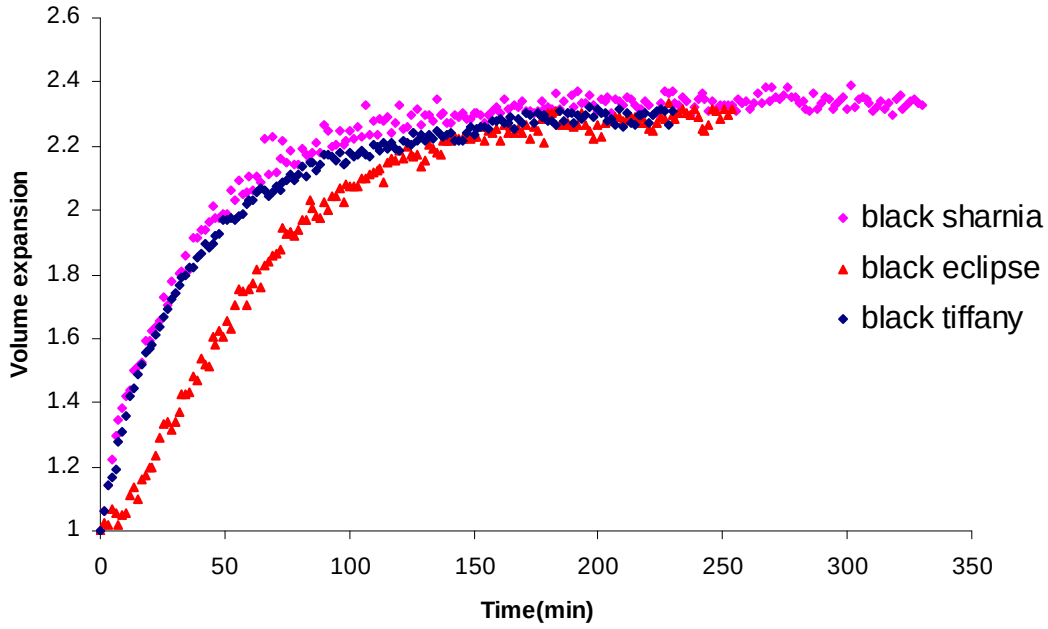

Figure 3.6 Volume kinetics of black beans at 55 ºC

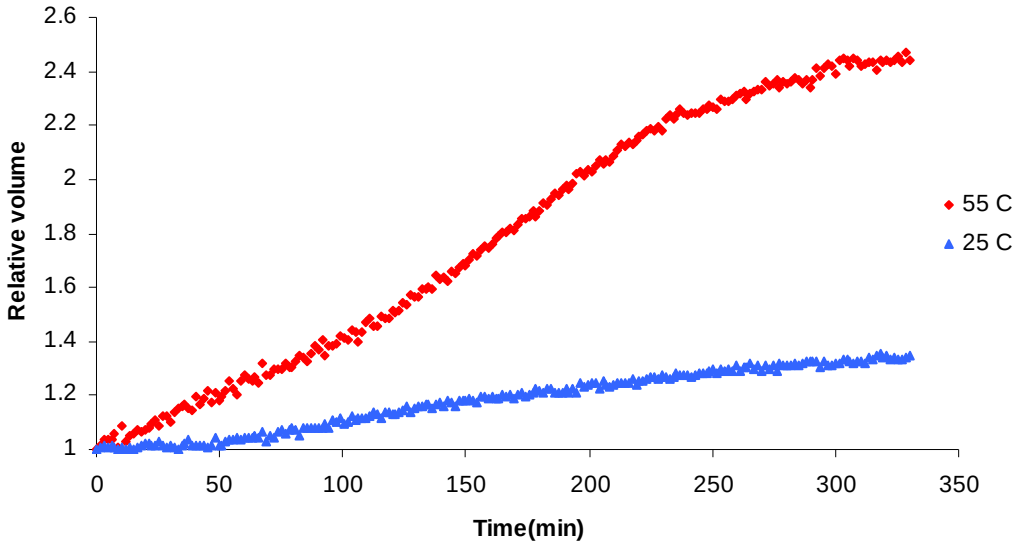

Figure 3.7 Volume kinetics of pinto cultivar A at different temperatures

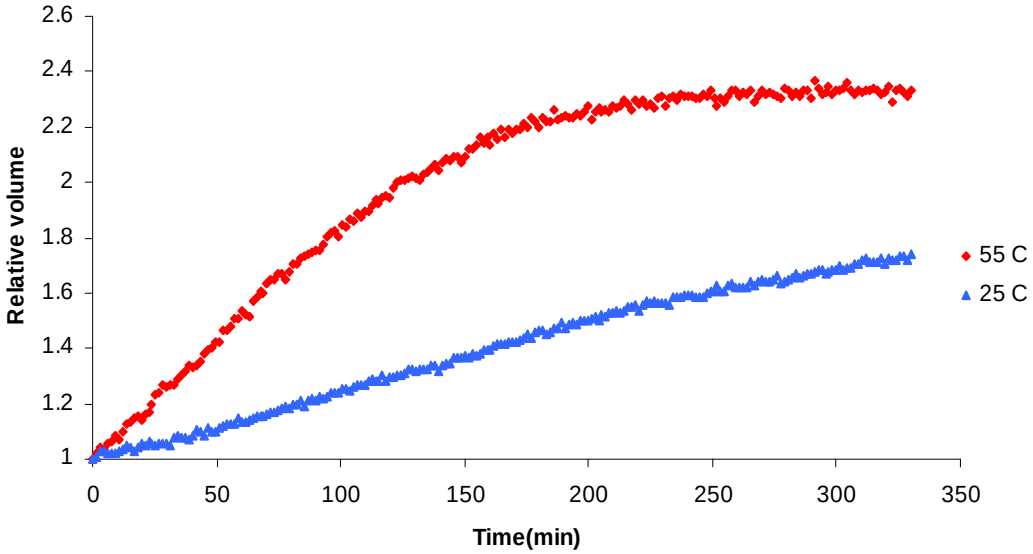

Figure 3.8 Volume kinetics of pinto cultivar B at different temperatures

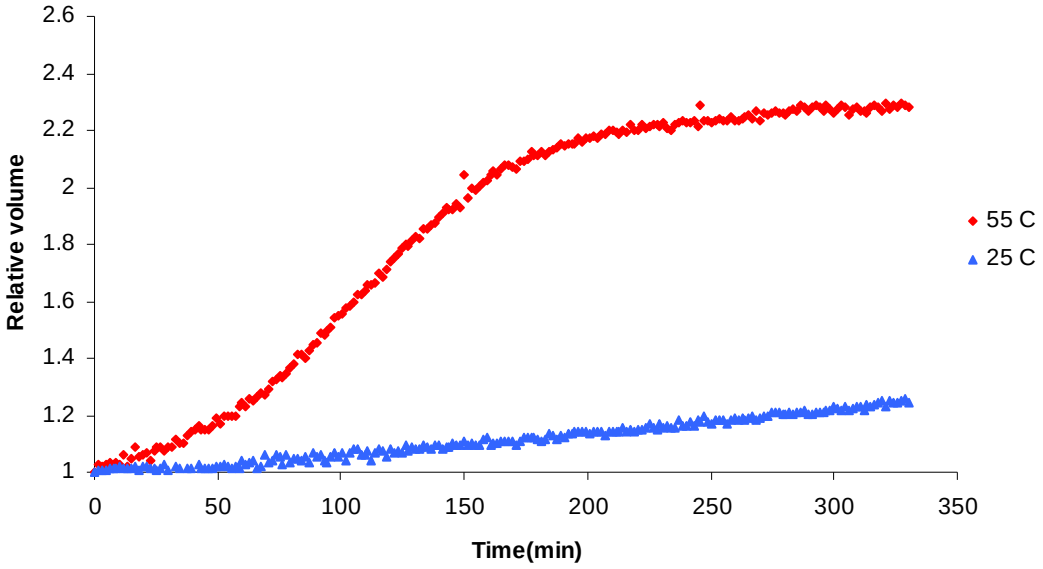

Figure 3.9 Volume kinetics of pinto cultivar C at different temperatures

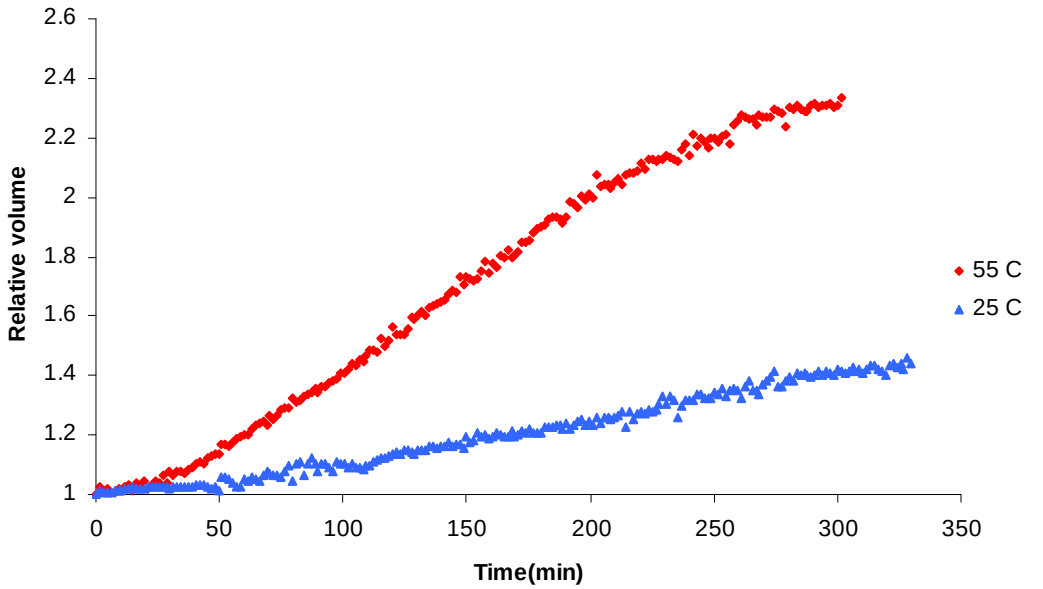

Figure 3.10 Volume kinetics of pinto cultivar D at different temperatuers

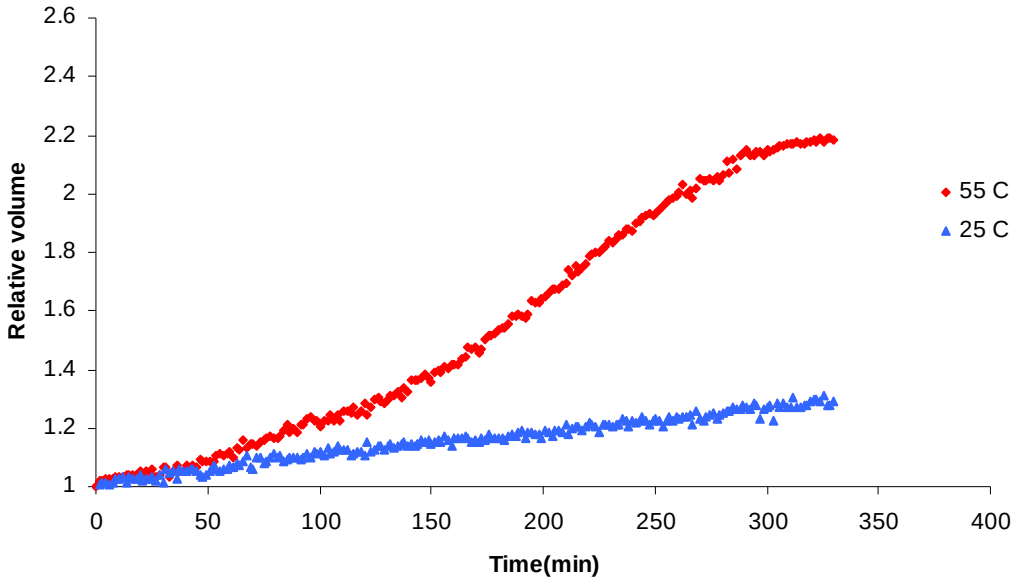

Figure 3.11 Volume kinetics of pinto cultivar E at different temperatures

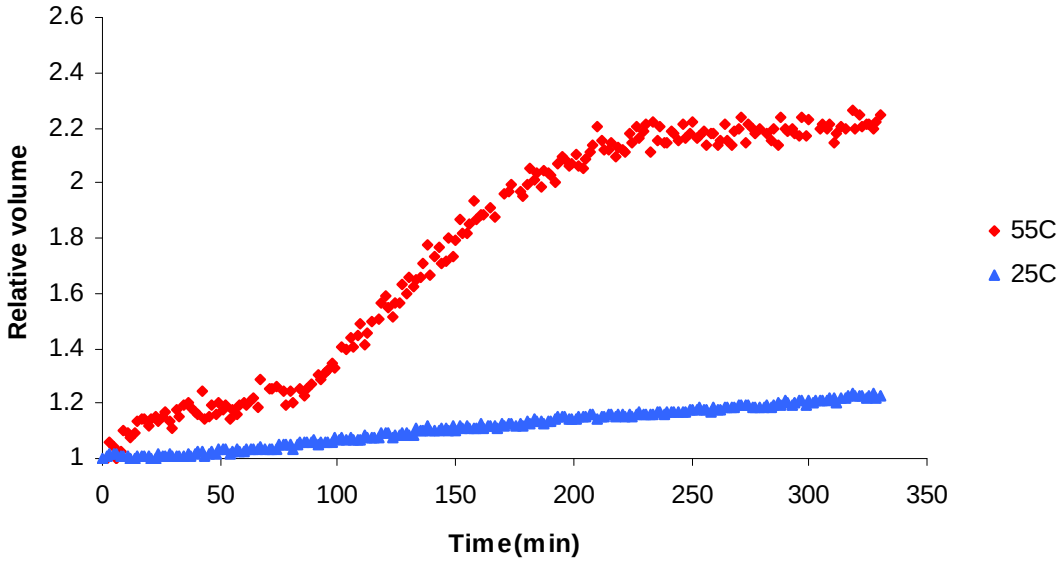

Figure 3.12 Volume kinetics of pinto cultivar F at different temperatures

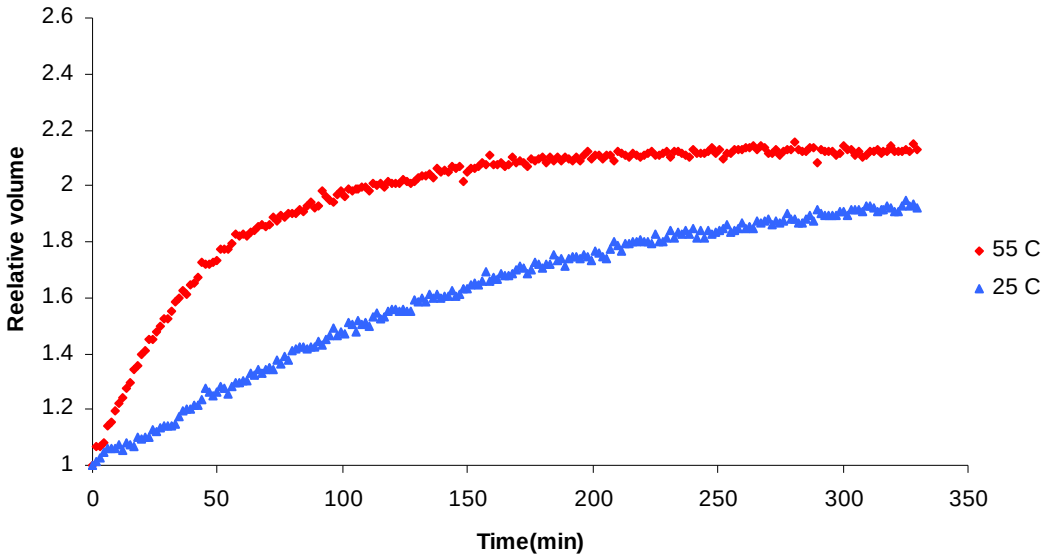

Figure 3. 13 Volume kinetics of navy cultivar A at different temperatures

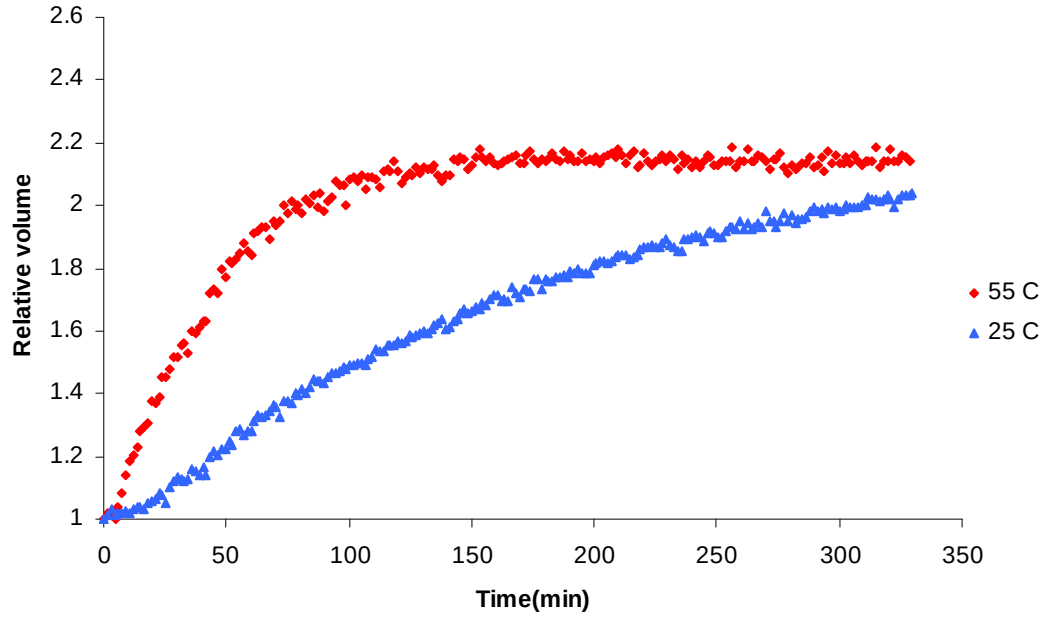

Figure 3.14 Volume kinetics of navy cultivar B at different temperatures

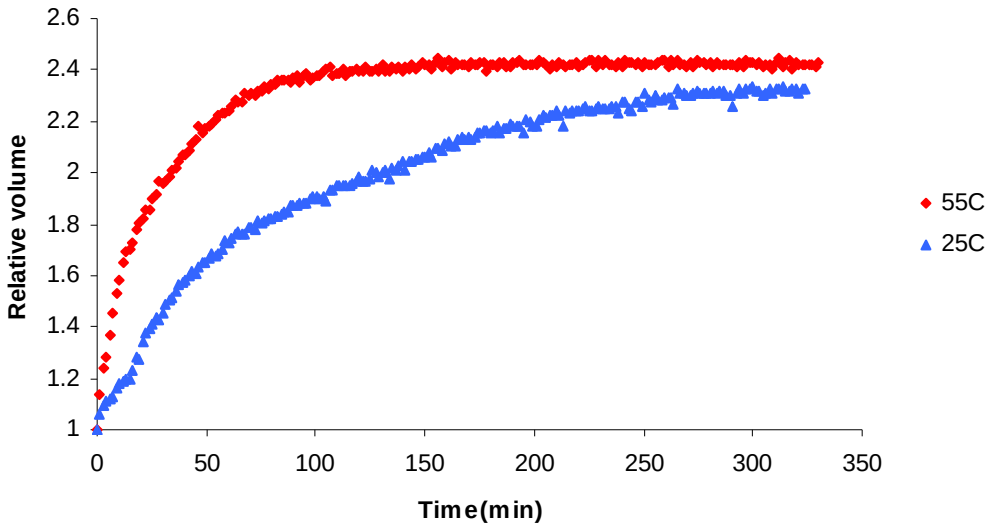

Figure 3. 15 Volume kinetics of navy cultivar C at different temperatures

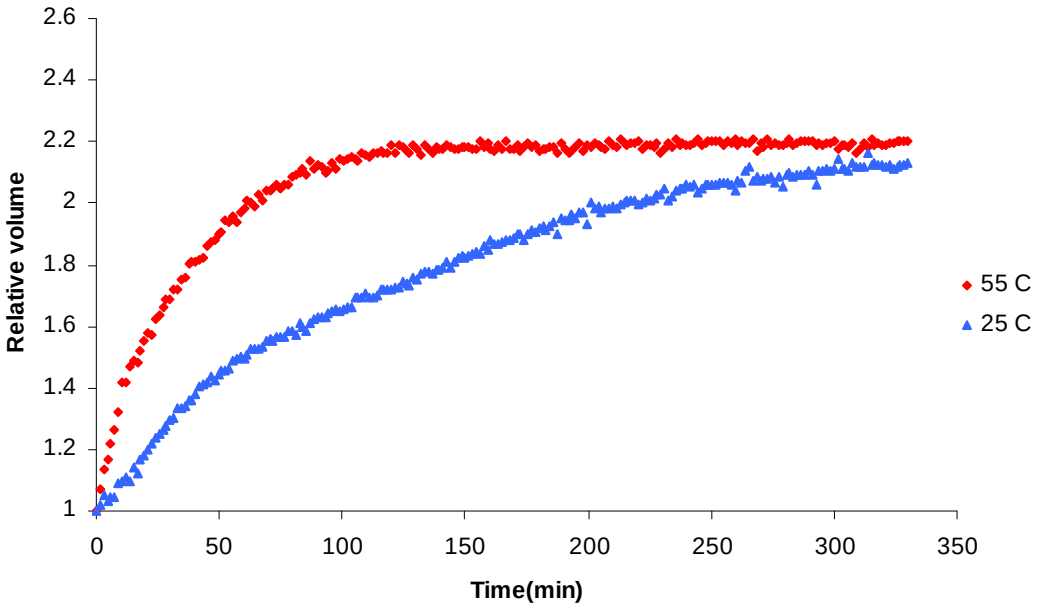

Figure 3.16 Volume kinetics of navy cultivar D at different temperatures

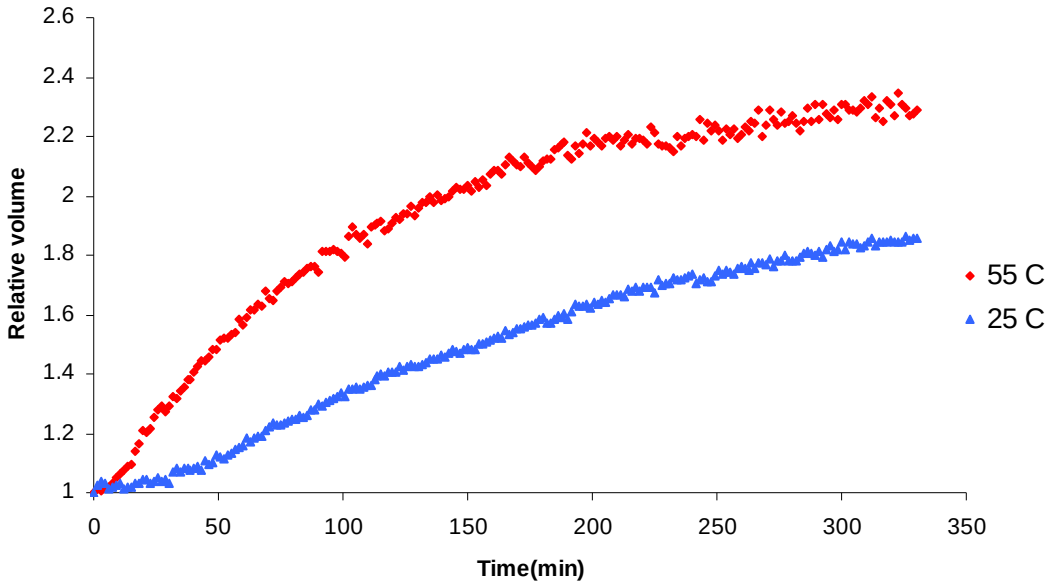

Figure 3.17 Volume kinetics of navy cultivar E at different temperatures

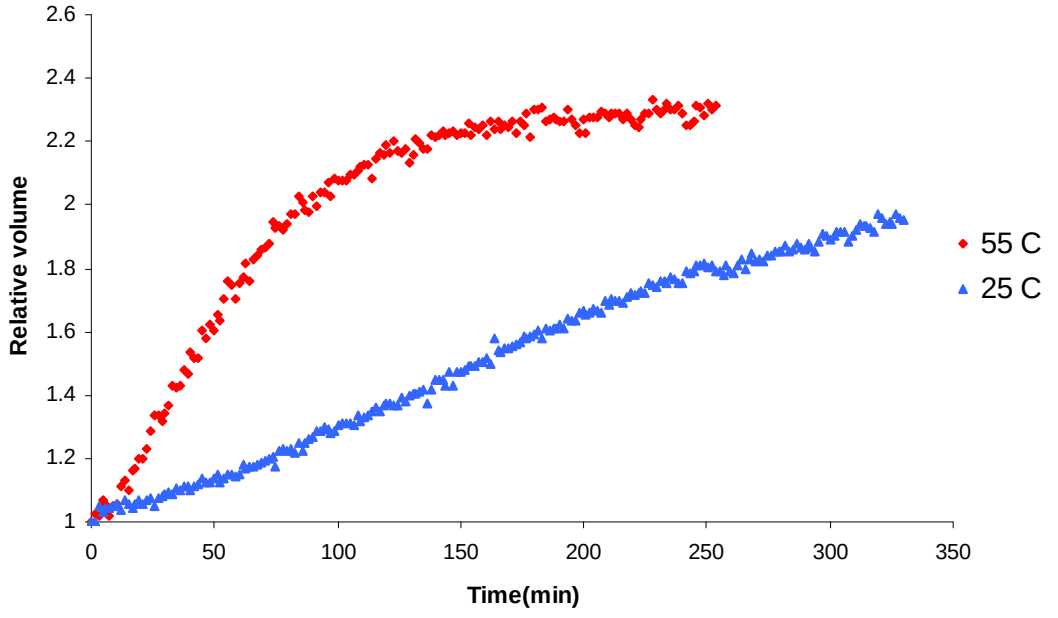

Figure 3.18 Volume kinetics of black cultivar A at different temperatures

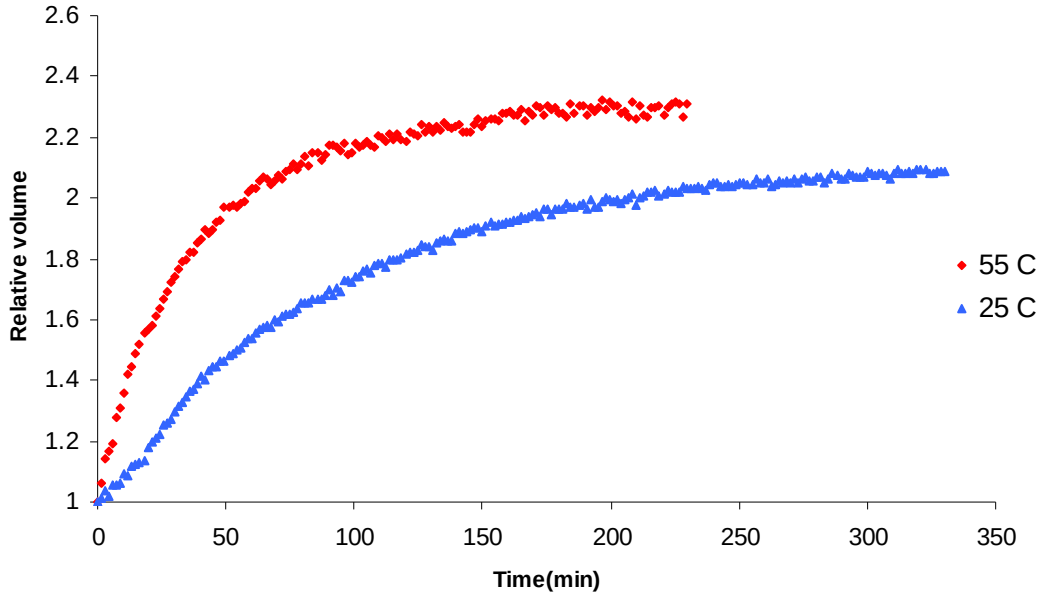

Figure 3.19 Volume kinetics of black cultivar B at different temperatures

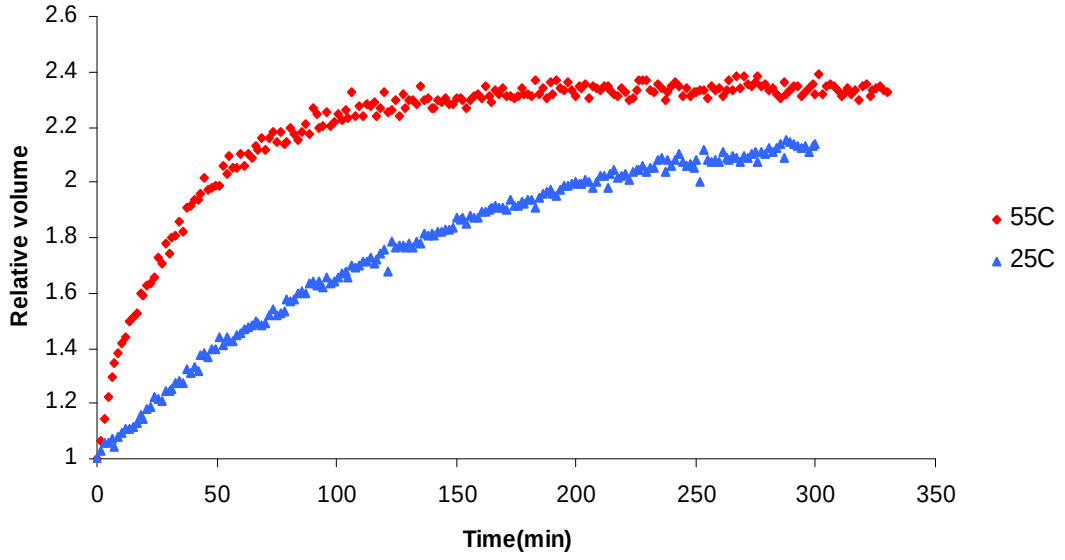

Figure 3.20 Volume kinetics of black cultivar C at different temperatures

CHAPTER IV
EFFECT OF SURFACE HYDROPHOBICITY ON BEAN HYDRATION EFFICIENCY

Abstract

Pinto beans require prolonged soaking time resulting in increased costs and energy. At elevated temperature, the water uptake of pinto beans exhibits a sigmoidal behavior with an initial lag phase not observed d in many other seeds. It was hypothesized in this study that a hydrophobic layer on the bean's surface becomes a key limiting factor for early water penetration. A 1 min hexane immersion treatment was adopted for 6 pinto cultivars in order to reduce the surface hydrophobicity prior to soaking. The surface hydrophobicity of beans was determined before and after the treatments via surface contact angle measurement. Soaking behavior at 55 ºC was determined by four indices: 1) initial volume increase rate; 2) length of lag phase; 3) end relative volume and 4) time to reach 2 times the initial volume. Hexane pre-treatment method adopted in this study was able to effectively reduce the hydrophobicity of bean surfaces (up to 42% reduction in contact angle) and shorten the hydration time required to reach 2 times the initial volume (up to 2.5 hr). Results also indicated that surface hydrophobicity had moderate correlations with rate of water uptake in initial lag phase ($r=-0.628$, $r<0.05$) and end relative volume ($r=-0.81$, $p<0.05$). The reduction in hydrophobicity due to pre-treatment was moderately correlated with the reduction in length of lag phase ($r=0.749$, $p<0.09$) and the increase in end relative volume ($r=0.736$, $p<0.1$)

Introduction

Pinto beans (*Phaseolus vulgaris L.*), also called molted beans due to their appearance, are traded and consumed heavily in South and Central America and are an important diet supplement due to its high nutritive value and low calorie content. Pinto beans account for 30% of the total production of dry bean in the world. The soaking step is usually necessary for processing dry beans to leach out antinutrients (e.g., hemagglutinin, trypsin inhibitors and phytate substances), thus enhancing the nutritive value of the product. More importantly, by introducing water evenly into beans prior to cooking, soaking also saves the time and energy required for cooking considerably. Pintos are one of the varieties that require the longest soaking time prior to cooking. It takes more than 24 hr for soaking pintos under room temperature conditions. Thus, studies on methods to facilitate the water transfer into beans are needed. Factors that could shorten the soaking time in faba, kidney, and navy beans, soybeans, sesame seeds, and rice have been previously investigated (Abousamaha et al., 1985; Aparna et al., 2000; Chenoll et al., 2009b; Haladjian et al., 2003; Jiang and Zhang, 2005; Tagawa et al., 2002). However, few reports exist regarding the hydration kinetics of pinto beans. In the previous chapter, an extended lag phase was observed during the soaking of pinto beans at elevated temperature.

Many plants tissues have natural wax on their surfaces to protect them from environmental stresses such as ultraviolet radiation, insects and pathogens as well as to limit moisture loss (Eigenbrode and Espelie, 1995; Schreiber et al.,

2001). The cuticular wax is found to be on the surface of leaves, seeds and fruits (Chang et al., 2006; Chowdhary et al., 1982). Based on the observation of the initial lag phases during soaking of pinto beans, it was hypothesized that a hydrophobic layer on the surface of pinto bean (possibly wax) was responsible of delaying initial water penetration into the bean. At room temperature, this layer can hardly be removed, leading to the linear and slow volume expansion. When temperature increased, this resistance was gradually removed, reducing the lag phase. The higher the temperature, the faster this layer breaks down and thus shortens lag time and increases water absorption rate. The thickness and intensity of the wax could vary among cultivars, causing the different hydration behaviors between the various pinto cultivars.

In order to test the hypothesis, hexane was used as a media to reduce the intensity of the hydrophobic layer on the surface of beans. Hexane is widely used as a cheap, relative safe and easily evaporated non-polar solvent. Despite requiring extra safety measures when used in the food industry, it is heavily used as a solvent for oil extraction from grains and soybeans. The code of federal regulation (21 CFR 173.270) allows its presence in spice oleoresins and hops extracts as residues but with amount less than 25ppm and 2.2 % weight, respectively. In this study, hexane was used as a pre-treatment for soaking to reduce hydrophobicity of the surface of pinto beans.

A very common way to determine the hydrophobicity/wettability of a solid surface is to measure the contact angle of a water droplet sitting on the surface. Previous reports used this method to quantify the hydrophobicity of

solid surfaces such as stainless steel, glass, polyethylene, Gallium Asenide, cell walls of various bacteria, and polymeric materials (Gulec et al., 2006; Matsushita et al., 1998; O'Connell et al., 2009; Vanloosdrecht et al., 1987). The smaller contact angle, the more hydrophilic a surface is. Usually, a surface having contact angle <90º is considered to be hydrophilic, between 90º to 150º hydrophobic and >150º superhydrophobic.

The objective of this study was to 1) accurately describe and quantify the unique soaking behavior of pinto beans; 2） to validate the hypothesis that the hydrophobicity of bean surface is a major barrier for water penetration, leading to the initial lag phase; and 3) prove that hexane is an effective treatment to reduce the surface hydrophobicity and improve bean hydration efficiency.

Materials and Methods

Materials

Dry seeds from six cultivars of pinto beans: cultivar A, B, C, D, E, and F, harvested in fall season, 2009 and provided by ADM's facility in St. Thomas, ND were used for this experiment. Beans were stored in dry and cool place in ambient condition until testing.

Volume kinetics determination during soaking

70 grams of each bean cultivar were weighed after removing the split beans and then put into the system described in Chapter 2 to determine volume kinetic at 55 °C for 4.5 hr, with two replicates at each temperature. The

volume measurements were taken by the system automatically at 1.5 min intervals. Deionized water was used as soaking medium. The soaking process were divided into three phases: lag phase, rapid water uptake phase, and phase approaching equilibrium. In order to differentiate the transit point from initial lag phase (LP) into the rapid water uptake phase (RP) and highlight the change in volume change rate, the volume kinetics curves were divided into two linear sections and the rates of the two phases were calculated as the slopes of the respective linear models fitted in the two phases. To better characterize and compare the hydration behavior, several indices were defined and determined: 1) initial rate (ml/ml/min) presents the volume ratio increase every minute; 2) length of lag phase (min); 3) end relative volume (%); 4) time to reach 2 V_0, where V_0 is the initial volume.

Hexane pre-treatment

70 grams of each cultivar of pinto beans were immersed in 100 ml n-hexane (Fisher H207-4, assay 60-66%) in a 250 ml beaker for 1 minute under agitation. Hexanes were drained and then kept until evaporated. Beans were then put into the automatic system for testing the volume kinetics as described before.

Hydrophobicity determination

The hydrophobicity of the outer surface of beans with and without hexane pre-treatment was tested via contact angle measurement by the static sessile water drop method using an EasyDrop Standard Drop Shape Analysis System

(KRUSS, Germany). A 2 μl water droplet was deposited on the comparatively more even surface of one bean and a picture was captured immediately after by the camera in this equipment. Due to the curved surface of beans, the contact angles θ was measured based on the definition in Young's equation (Gulec et al., 2006; Van Oss, 1994) as shown in Figure 4.1 which describes the equilibrium state between the three phases: the liquid phase (L), the solid phase (S) and the gas/vapor phase (V) using AutoCAD 2007 software (Autodesk, San Rafael, CA) and used to indicate the surface hydrophobicity. At least three replicates were taken for each treatment.

Statistical analysis

The experiment was designed with CRD and the contact angles of all cultivars before and after hexane pre-treatment were analyzed with ANOVA and Tukey test using SAS 9.0 (SAS Institute, 2009). Pearson correlation among contact angle, reduction in contact angle and hydration parameters: rate of lag phase (LP), rate of rapid phase (RP), ratio of rate of LP for treated beans and untreated beans; ratio of rate of RP; length of LP, reduction in length of LP, end relative volume, ratio of end relative volume of treated beans and untreated beans were determined by SAS 9.0 (SAS Institute, 2009)

Results and Discussions

Description of lag phase

As mentioned in Chapter 3, the volume change when beans were soaked at 25 ^{o}C followed a linear pattern indicating that external resistance existed against water penetration, which is different from what has previously reported in cases where diffusion dominates the water transfer (Abu-Ghannam, 1998a; Maldonado et al., 2010; Turhan et al., 2002). In this study, the volume kinetic curves changed dramatically at 55 °C, from linear to sigmoidal curves, suggesting the hydration of some pinto cultivars is a three-phase process. Initially, water was absorbed very slowly, shown as the lag phase; then after a certain point, water uptake rate increased significantly; afterwards the rate reduced as approaching the equilibrium. All cultivars except cultivar B showed the three-phase volume kinetics. The hydration rates in the two phases (LP and RP) and the length of the initial lag phase for all 6 pinto cultivars are shown in Table 4.2 (control groups). Aside from pinto cultivar B, all other cultivars showed a lag phase and rapid water uptake phase where the rate of RP was up to 4 times of that of the LP, proving the differences in rates during the two phases. The biggest increment occurred on pinto cultivar F where rates increased from 2.5 * 10^{-3} ml/ml/min (the relative volume increase rate) in the lag phase to 10.3 * 10^{-3} ml/ml/min in the rapid phase. The length of lag phases ranged from 0 (cultivar B) to 135 min (cultivar E). Due to the comparatively slower hydration rate during the lag phase and its considerable

duration of time, treatments targeting at reducing the length of lag phase or enhancing its rate are needed.

Effect of hexane pre-treatment

Comparison of contact angle

The hydrophobicity of the bean surfaces was tested on pinto beans before and after the hexane treatment. One picture captured by the Drop Shape Analysis System for each cultivar before and after hexane pre-treatment is given in Figures 4.2 – 4.7. There were notable changes in the morphology of the water droplet on bean surfaces after the treatment. The contact angle of each cultivar and the percent reduction in contact angle are listed in Table 4.2. The percent reduction was calculated using Eq. (10):

$$\% \text{ reduction} = \frac{\theta_b - \theta_a}{\theta_b} \quad (10)$$

The results showed that all five cultivars having an initial lag phase during soaking at 55 ºC exhibited a great reduction in contact angle after hexane treatment, in the range from 29% to 42%. The hexane pre-treatment adopted in this study was able to significantly decrease the hydrophobicity of the surface of the beans for all cultivars ($p<0.01$). Cultivar B, which did not have a distinct lag phase, had a significantly smaller contact angle before the hexane pre-treatment than most of other cultivars except cultivar F ($p<0.01$), and a significantly smaller percent reduction in contact angle after the pre-treatment than all other cultivars ($p<0.01$). Cultivar D, A, E and C had higher surface

hydrophobicity than cultivar B and F ($p<0.01$). The former four cultivars all had hydrophobic surfaces before hexane pre-treatment (contact angle > 90 ºC) and changed to hydrophilic surfaces after the treatment (contact angle < 90. ºC).

Comparison of soaking behavior

All six pinto cultivars with and without hexane pre-treatment were tested for volume kinetics at 55 ºC for 4.5 hr soaking, and the results are shown in Figures 4.8 – 4.13. The water uptake of all cultivars was improved with the hexane pre-treatment. The indices introduced to describe hydration behavior and compare hydration efficiency are listed in Table 4.2: K, the rate of relative volume increase for both LP and RP; the length of lag phase, time to reach 2 times of original volume and relative volume. The R-squares of the linear model fitting LP and RP sections were around 0.97-0.99. All cultivars hydrated more efficiently after hexane pre-treatment while cultivar B received the smallest improvement due to its original less hydrophobic surface. The rate of initial water uptake increased up to 6.5 times (pinto cultivar D) after the hexane treatment. This could be attributed to the partial removal of the hydrophobic layer on the surface of beans. The length of lag phase reduced by up to approximately 1 hr for several cultivars, strongly suggesting that the hydrophobic layer was impaired after the hexane pre-treatment. Also, the time required to reach 2 times the initial volume decreased up to approximately 2.5 hr which is considered a long processing time in industrial manufacturing. Except for pinto cultivar B (the only cultivar that showed no initial lag phase

during soaking at 55 ºC), all cultivars exhibited huge reductions in soaking time after a 1 minute hexane pre-treatment.

Correlations

The statistical analysis suggested that there was a negative moderate correlation between contact angle and the rate of LP ($r=-0.628$, $p<0.05$), between contact angle and rate of RP ($r=-0.726$, $p<0.05$), and a negative high correlation between contact angle and end relative volume ($r=-0.81$, $p<0.05$). These correlations suggest that beans with larger hydrophobic surfaces tend to have lower rate of lag phase and rapid phase, and lower end relative volume after a period of soaking before reaching equilibrium. The hydrophobic bean surface is a critical barrier for water penetration during the soaking process: the more hydrophobic the surface, the slower the transfer of water into the beans and the slower the overall rate of water uptake. Due to the faster water absorption rate after hexane pre-treatment, the beans reached a higher end relative volume after 4.5 hr. Moreover, a positive moderate correlation was observed between the decrease in contact angle and length of LP ($r=0.749$, $p<0.09$), and between the decrease in contact angle and the increase in end volume ($r=0.736$, $p<0.1$). These correlations implies that a bigger reduction in surface hydrophobicity can lead to a shorter lag phase and a bigger end relative volume during soaking of beans. Thus how to effectively reduce the surface hydrophobicity of beans prior to soaking become the major way to improve the soaking efficiency. Furthermore, a negative moderate correlation was found between rate of lag phase and length of lag phase (r=-

0.711, $p<0.05$), implying that treatments shorten the lag phase probably are able to effectively increase the rate of lag phase. There was also a positive high correlation found between rate of lag phase and rate of rapid phase ($r=0.922$, $p<0.001$), suggesting that the rates of the two phases probably are affected by the similar factors during processing.

Conclusions

Pinto beans require prolonged soaking time even at elevated temperature. The soaking of pinto beans at 55 ºC could be characterized as a three phase process: 1) initial lag phase, which ranges from 0 to 2.5 hr 2) rapid water uptake phase at a rate 2 to 4 times faster than initial lag phase and 3) equilibrium approaching phase. There is a hydrophobic layer on the surface of pinto beans, retarding the water penetration. The more hydrophobic the surface, the lower rate of water uptake the bean has during soaking. Hexane pre-treatment is able to decrease the hydrophobicity of the surface of beans and enhance the efficiency of hydration. The more the hydrophobicity is reduced, the better effect it has on shortening the length of lag phase and enhancing the end relative volume. Pretreatment of the beans with hexanes for 1 min is an effective method to shorten the lag time, in increasing the hydration rate and reducing the required soaking time to reach an acceptable end volume for pinto beans.

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Appendix

Table 4.1 Contact angles of six pinto cultivars before and after hexane pre-treatment.

Cultivar	Contact angle (Before)	Contact angle (After)	Reduction percent (%)
Pinto cultivar B	75.47±1.55 b	65.33±1.62 a	13.4%
Pinto cultivar D	104.57±2.62 a	63.53±3.42 a	39.2%
Pinto cultivar A	98.97±4.41 a	70.27±4.67 a	29.0%
Pinto cultivar E	107.93±2.24 a	68.23±0.38 a	36.8%
Pinto cultivar F	81.27±6.75 b	53.13±4.40 b	34.6%
Pinto cultivar C	104.50±3.08 a	60.60±5.14 ab	42.0%

Table 4.2.A Effect of hexane pre-treatment on the hydration behavior of pinto cultivars at temperature of 55 ºC.

Cultivar	Rate of LP			Rate of RP		
	Control (ml/ml/min)	Treatment (ml/ml/min)	Ratio (%)	Control (ml/ml/min)	Treatment (ml/ml/min)	Ratio (%)
Pinto cultivar B	---	---	---	8.663e-3	2.249e-2	260
Pinto cultivar D	2.012e-3	---	---	5.673e-3	1.871e-2	330
Pinto cultivar A	4.110e-3	5.863e-3	143	6.206e-3	1.030e-2	166
Pinto cultivar E	2.192e-3	4.801e-3	219	5.497e-3	1.078e-2	196
Pinto cultivar F	2.568e-3	8.581e-3	234	1.011e-2	1.335e-2	132
Pinto cultivar C	4.152e-3	8.459e-3	204	9.213e-3	1.870e-2	203

Table 4.2.B Effect of hexane pre-treatment on the hydration behavior of pinto cultivars at temperature of 55 ºC.

Cultivar	End relative volume			Length of Lag phase			Time to reach 2V		
	Control (%)	Treatment (%)	Ratio (%)	Control (min)	Treatment (min)	Difference (min)	Control (min)	Treatment (min)	Difference (min)
pinto cultivar B	231	246	106	0	0	0	120	80	40
Pinto cultivar D	226	275	122	50	0	50	205	60	145
Pinto cultivar A	233	239	102	115	60	55	195	120	75
Pinto cultivar E	198	242	122	135	80	55	270	135	135
Pinto cultivar F	225	259	151	70	45	25	165	85	80
Pinto cultivar C	223	263	118	75	35	40	160	70	90

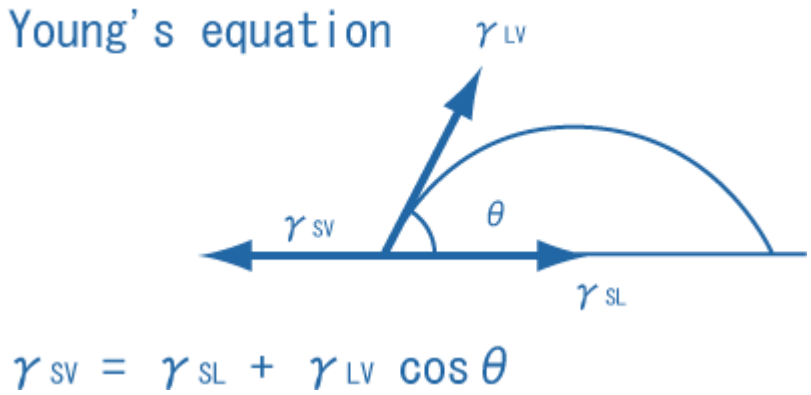
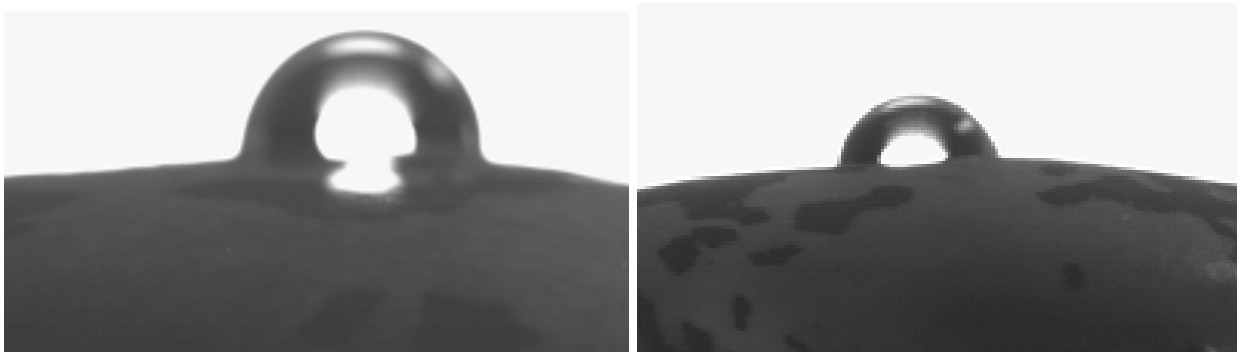
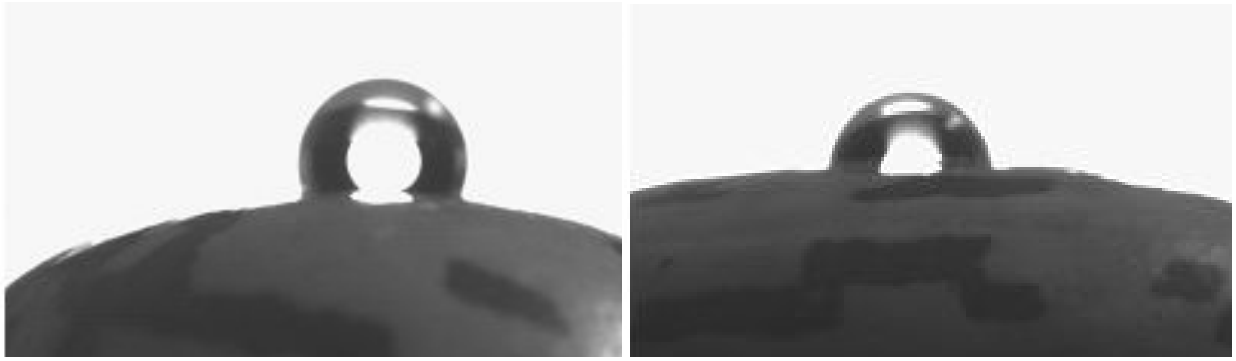


Figure 4.1 Contact angle defined in Young' equation (Gulec et al., 2006).



A B

Figure 4.2 Contact angle of pinto cultivar B before (A) and after (B) hexane pre-treatment.

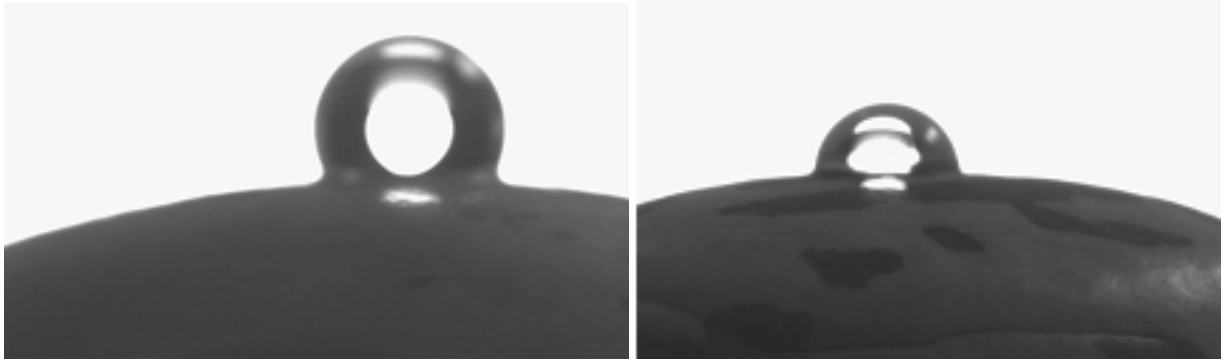


A B

Figure 4.3 Contact angle of pinto cultivar D before (A) and after (B) hexane pre-treatment.

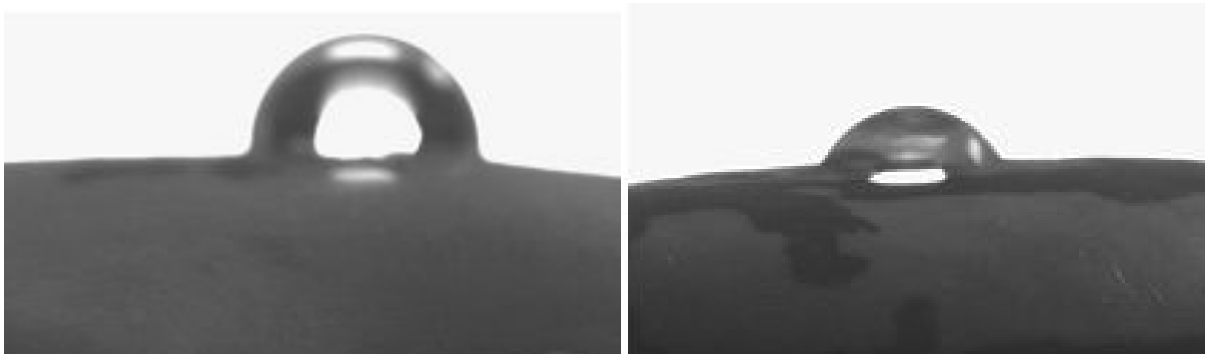
A B

Figure 4.4 Contact angle of pinto cultivar A before (A) and after (B) hexane pre-treatment.



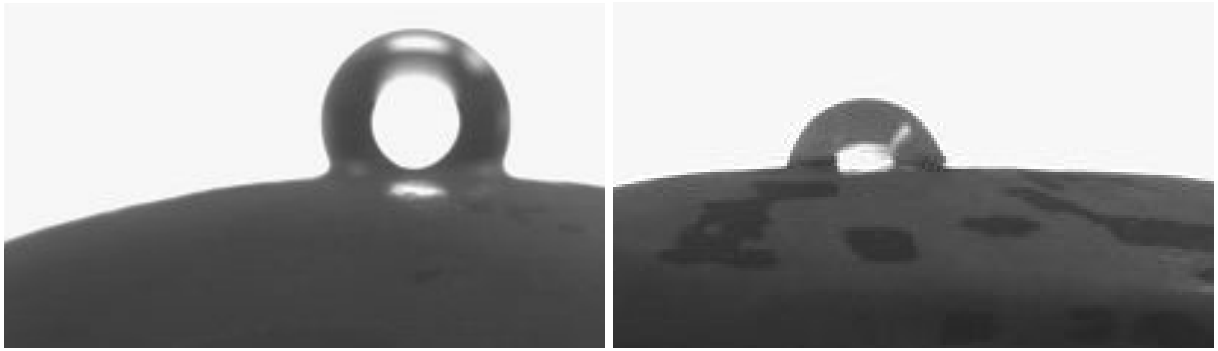
A B

Figure 4.5 Contact angle of pinto cultivar E before (A) and after (B) hexane pre-treatment.



A B

Figure 4.6 Contact angle of pinto cultivar F before (A) and after (B) hexane pre-treatment.



A B

Figure 4.7 Contact angle of pinto cultivar C before (A) and after (B) hexane pre-treatment.

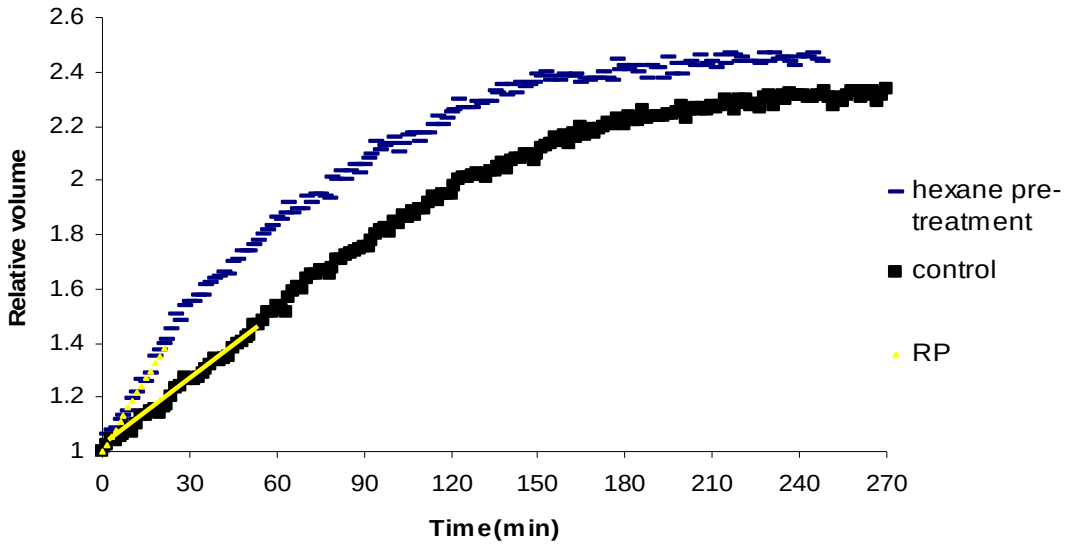


Figure 4.8 Volume kinetics of pinto cultivar B with and without hexane pre-treatment soaking at 55 ºC.

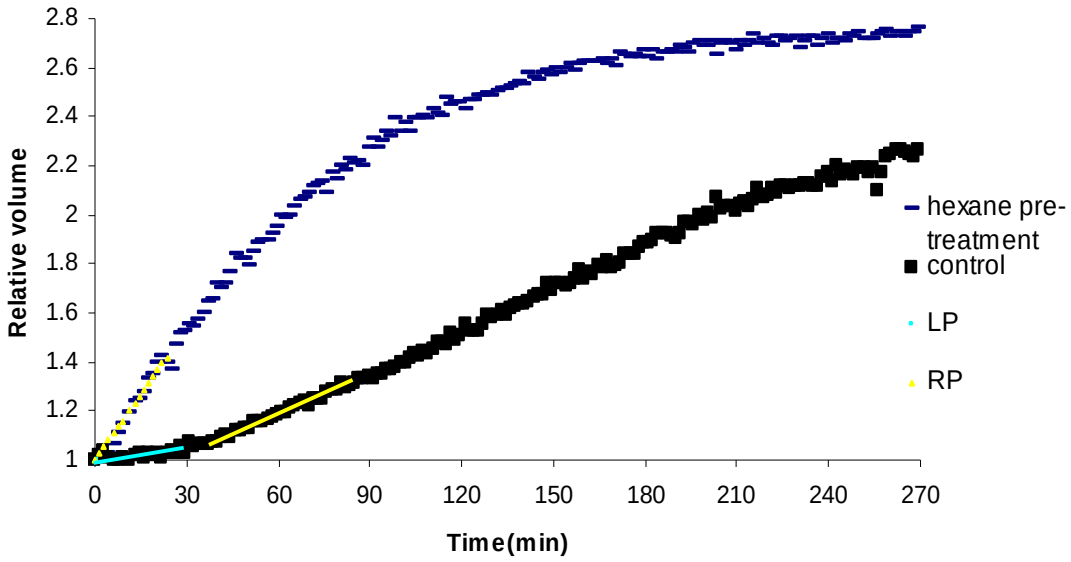


Figure 4.9 Volume kinetics of pinto cultivar D with and without hexane pre-treatment soaking at 55 ºC.

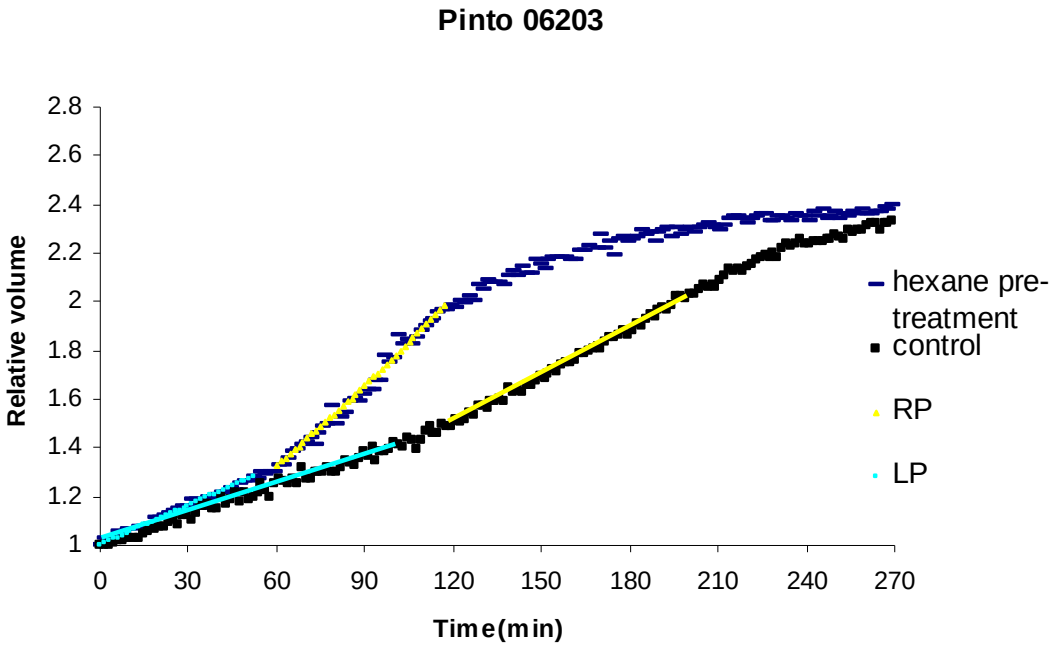


Figure 4.10 Volume kinetics of pinto cultivar A with and without hexane pre-treatment soaking at 55 ºC.

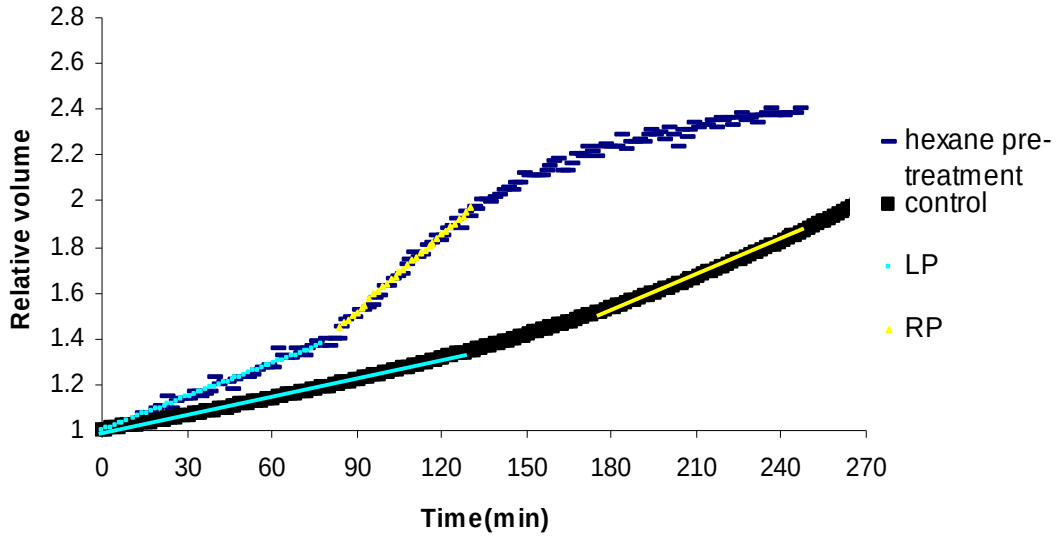


Figure 4.11 Volume kinetics of pinto cultivar E with and without hexane pre-treatment soaking at 55 ºC.

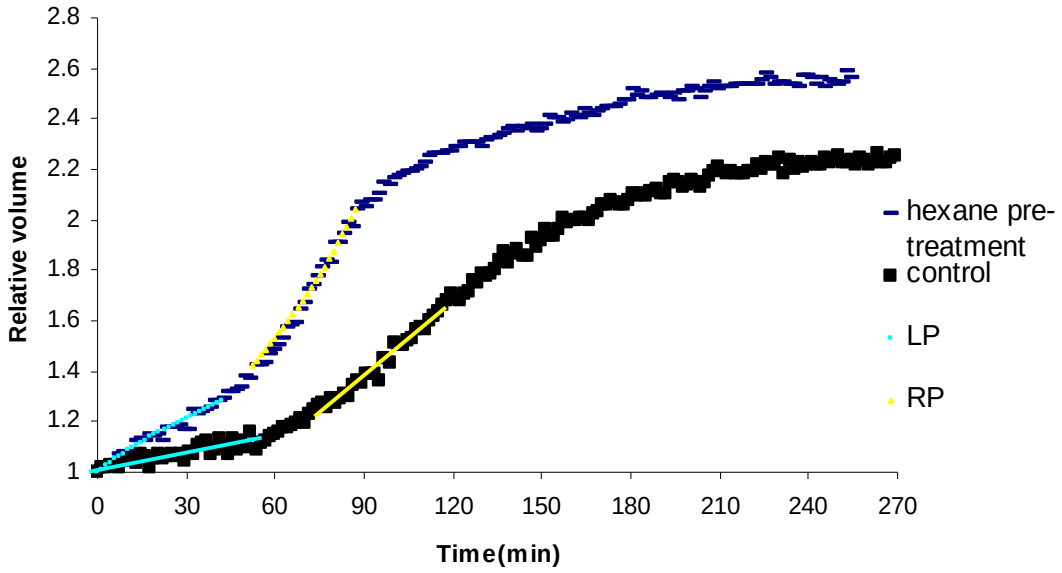


Figure 4.12 Volume kinetics of pinto cultivar F with and without hexane pre-treatment soaking at 55 ºC.

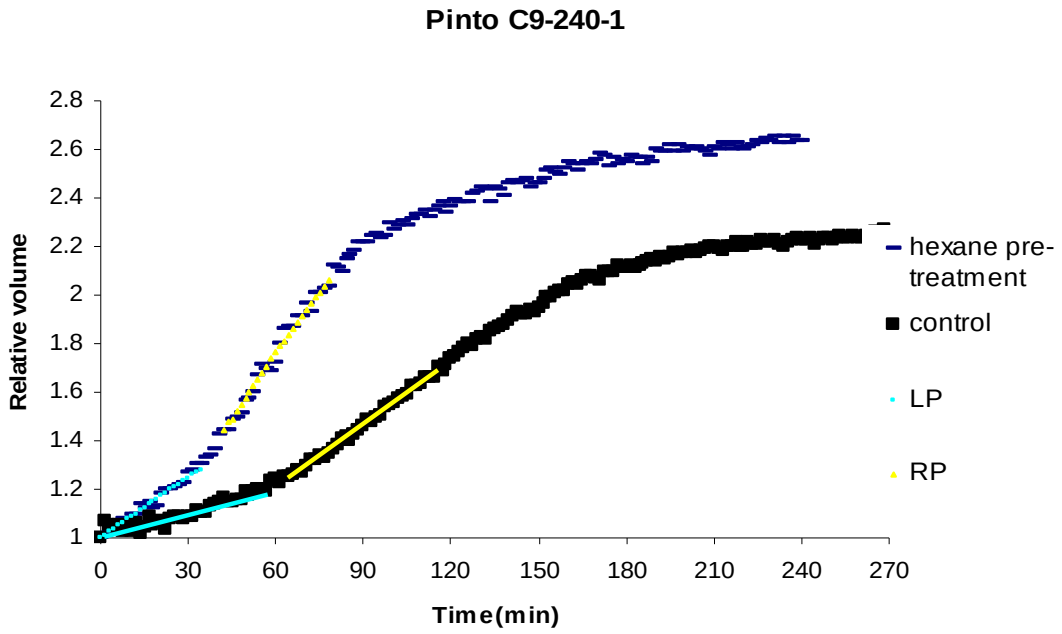


Figure 4.13 Volume kinetics of pinto cultivar C with and without hexane pre-treatment soaking at 55 ºC.

CHAPTER IV
CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE WORK

In order to adequately describe the volume kinetics of seed during hydration, a B-VAT system that automatically conducts volume measurement over time during soaking was developed. The device has minute-scale measuring interval, low system variability (COV< 0.8%), adequate sampling size (mass up to 150 gram), high reproducibility, minimal need for operator intervention during measurements, and it can be used to measure volume during soaking for various types of seeds in controlled soaking media at target temperatures. Significant differences were observed among pinto, navy and black varieties and cultivars during soaking at both 25 and 55 ºC ($p<0.01$). Pinto cultivar B soaked the fastest without initial lag phase at 55 ºC; black eclipase had linear volume expansion at 25 ºC; making them outliers in their respective varieties. Unlike the other two varieties, pinto beans had a linear volume increase pattern at 25^{o}C, a sigmoidal pattern at 55^{o}C, and the least hydration efficiency. Increasing temperature to 55ºC effectively increased the initial water absorption rate for all bean cultivars, in a range from 1.3 to 6.8 fold as well as shortening the time to reach 2 times of initial volume at least by 2 hr. The effect of temperature on pinto cultivars was much more dramatically than navy and black beans in shortening the required soaking time. The reason why pinto beans have extraordinary slow water uptake was partially attributed to their hydrophobic surface. Faster water absorption rates of both lag phase and rapid phase during soaking are expected in beans having less hydrophobic surfaces. Hexane pre-treatment for one minute

immersion of beans was found to effectively reduce the hydrophobicity and enhance the hydration efficiency.

The future work should focus on the following aspects:

(A) Improvements to the B-VAT system. The capability to simultaneously weight samples and monitor turbidity can be incorporated in the system to allow determination of changes in mass (wet basis), and solid loss during soaking. The ultrasonic sensor should be replaced with sensor able to withstand higher temperature ratings; the building materials should be upgraded so that the system can be used to simulated steam soaking. Some of these improvements are being currently developed for a new prototype that will also multiple sample holders to determine volume kinetics in three replicates simultaneously.

(B) Extended temperature range. The optimum temperature for fast water uptake and high equilibrium volume without cell disruption should be determined in a cultivar basis. Also, the effect of temperature on the nutrition value of the final product (e.g. mineral availability, protein digestibility) should be taken into account besides its impact on soaking efficiency.

(C) Other factors. The seed soaking can be affected by other factors, for instance, salts. Sodium bicarbonate and sodium triphosphate were reported previously as additives to enhance the seed soaking efficiency. There are also intrinsic factors related to the beans that might attribute to the different soaking behaviors among varieties and cultivars. For example, the surface area of beans

and the differences in the chemical composition of seed coat. The association of hydration characteristics and these factors could be studied.

(D) New models. The Peleg or Weibull model do not properly fit the hydration curve of pinto beans under elevated temperature, thus a new model incorporating the factors causing the initial lag phase (e.g., the surface hydrophobicity) is needed.

(E) Hydrophobic layer. The original hydrophobic layer on bean surface can probably be dispersed using organic solvent treatments and then analyzed for its composition via the characteristics of its infrared spectrum. A good understanding of the composition of the thin layer definitely facilitate to design the specific treatments. Due to the limitation of using hexane as a processing aid, hot water, steam or other food grade surfactants pre-treatment can be used as a substitute and be investigated for the process optimization.

(F) This study was mainly focus on understanding the physical process of hydration as affected by various factors as discussed. The further study could dig more on what chemical changes occur during soaking e.g., change in the content and structure of starch or seed coat chemical composition.

VITA

Shan Xu was born in Hangzhou, Zhejiang, China in 1985 in a normal but nice family. From kindergarten to undergraduate study, she never lived outside of Hangzhou until she came to U.S. for advanced study. She had a happy and peaceful childhood which gave her the nature of happiness. She was admitted by the top high school in Zhejiang province, the Hangzhou No. 2 high school and had a fulfilled and unforgettable life there. Later on, she got admission from Zhejiang University which tank the third throughout the nation and started her undergraduate study in major of Food Science and Engineering. After getting her bachelor's degree with excellent grades, she dropped her opportunity as a recommended postgraduate to a top university in China but rather chose to go to U.S. to continue her study. During her stay in UT, Knoxville, she majored in Food Science and Technology and worked as a graduate research assistant under the guidance of Dr. Federico Harte. She participated in different research projects regarding to bean hydration, yogurt rheological property, and Maillard reaction but mostly focused on the Food Engineering. She also volunteered in various activities: Relay for Life (2009, 2010), FSC cheese sale fundraising (2010), food safety "lecture" for Kids, etc. In 2009-2010 academic year, she was awarded the IFT Graduate Scholarship from Quality Assurance Division. She can not wait to make use of what she has learned to move on to the next stage of her life.