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Ananthreddigari, Abhinav R. *Determination of sorption isotherm and shelf life study of hazelnut cracker using GAB model*

Abstract

Moisture sorption isotherm was determined for the hazelnut cracker at 25 °C. The isotherm generated was fitted to the Guggenheim-Anderson-de Boer (GAB) sorption equation. This equation was then used to develop and predict the shelf life of the cracker under standard environmental conditions. The environmental chamber was maintained at 25 °C with 80% relative humidity and the cracker was packed in HDPE (High Density Polyethylene) pouch of 4 ± 0.2 mil thickness. The sorption isotherm of the cracker followed a type II sigmoidal trend having a mono layer (C) and multilayer (k) GAB constants as 131.8 and 0.81 respectively. The GAB model used was observed to have a good fit with a relative percent deviation modulus (E) of 3.9979% and the root mean square error (RSME) of 0.0561. The shelf life of 50 grams of hazelnut cracker was determined to be 16.6 days and 44.9 days in open and closed conditions respectively.

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Chapter I: Introduction

The concept of developing and launching a product into market is not a simple process. It generally starts with an idea creation, market research, voice of the consumer, product development at laboratory scale, shelf life testing and sensory acceptability. This study is focused on the shelf life testing of a cracker using an accelerated shelf life testing method.

The recent concern over the sustainability of food (food for all) going viral, food industries have started thinking of alternatives like value added products by making the best, from the waste. The cracker used for the study was also developed from the hazelnut meal (defatted meal considered as waste after the extraction of the hazelnut oil) and the flaxseed meal (defatted flaxseed meal).

Nuts are considered to be good source of nutritious food as they have healthy lipids (Shahidi, Alasalvar, and Liyana-Pathirana, 2007). Recent epidemiologic and clinical studies suggest that consumption of nuts is associated with favorable plasma lipid profiles and reduce risk of coronary heart disease (CHD), cardiovascular disease and chronic ailments. (Mercanligil *et al.*, 2007). The Food and Drug Administration (FDA) in 2003 has established a relationship between consumption of nuts and reduced risk of CHD. The presence of natural antioxidants and the phenolic compounds in nuts and their respective byproducts like skin, oil, the meal left out after oil extraction and shell are responsible for these health benefits (Siriwardhana and Shahidi, 2002). Also the healthy eating pyramid recommends one to three serving of nuts to be incorporated in the diet each day. One such nut which gained attention in recent years is Hazelnut.

Hazelnut (*Corylus avellana* L.), also known as filberts, belongs to the Betulaceae family and ranks second in tree nut production after almonds (Turkish Hazel, nut Exporter's Union,

2006). Among the world producers of hazelnut, Turkey contributes about 75% of production whereas Italy (12%) and United States of America (6%) are the second and third largest producers respectively (Oliveira *et al.*, 2008). In the United States of America (USA), Oregon state alone contributes over 95% of the commercial hazelnut crops but recent studies conducted on the hybrid hazelnuts grown in Nebraska also showed that “hazelnuts have emerged as a promising oilseed crop”(Xu and Hanna, 2011). Hazelnuts were also recognized as economically feasible as it requires minimal water for its production, sustainable and environmental friendly oil seed crop and for industrial applications (Xu and Hanna, 2011).

Hazelnuts are generally grown to extract the oil and after the oil is extracted the cake and left over meal are considered to be byproduct. This byproduct (Hazelnut meal) is used as poultry feed but recent studies show that this meal had significant health benefits when induced in human diets. A study done on hypercholesterolemic adult men (exceeding levels of cholesterol in the blood) for 8 weeks, showed that inclusion of 40 g/day of hazelnut meal with their diet demonstrated a 5.2% reduction in total cholesterol. Also based on the LDL, HDL and TAG levels the study concluded that the consumption of hazelnut meal decreased the risk of CHD by 13%, 11-17% and 10% respectively (Mercanligil *et al.*, 2007).

“Nutrients”, which play an important role in human metabolism are divided into two classes’ macronutrients and micronutrients. The presence of macronutrients is abundant in our day to day diet but the deficiency of micronutrients is a global concern. Lack of micronutrient in diet can lead to blindness, diminish mental capacity and some cases can even lead to death. Present statistics show that over two billion people around the world experience micronutrient deficient (Tarver, 2013).

Hazelnut meal is not only good source of vitamin B₁, vitamin B₂, vitamin B₆, niacin and vitamin E but also are good source for microelements (Simsek and Aykut, 2007). Microelements like Boron(B), Chromium(Cr), Copper(Cu), Iron(Fe), Cobalt (Co), Lithium (Li), Nickel (Ni), Selenium (Se) and Zinc (Zn) are essential for human body as they perform critical functions like maintaining the cell membrane, essential for metabolism of glucose, insulin, stimulation of new brain cells and many other biological functions. A research based on RDA (recommended dietary allowance) showed that 50grams of hazelnut meal per day provided 6%, 9%, 19%, 9%, and 16% of B, Co, Fe, Ni and Zn respectively and on the other hand Se, Cu, and Cr levels were in excess amounts than RDA (Simsek and Aykut, 2007).

The previous studies discussed about hazelnut meal as a diet, its importance and health benefits. Studies also recommended that hazelnut could exhibit a synergistic health effect when added to another equivalent antioxidant. (Contini, M., 2012) Therefore the crackers used in this study were developed using hazelnut meal and flax seed meal.

The consumption of flaxseed (*Linum usitatissimum* L.) has been growing and is encouraged due its major components like α - linolenic acid (ALA), soluble fiber and lignin which prevent the lung damage and inflammation (Giacomo, S., 2013). Cardiovascular disease (CVD) was the leading cause of deaths in 2005, contributing to 35% of total deaths (Rosamond *et al.*, 2008). High levels of LDL-C (low density lipoprotein cholesterol) could lead to CVD. A study conducted on US adults during 1999 and 2004 estimated that the average LDL-C level was 25.3%. But the past studies conducted on lowering the LDL-C levels suggest that, incorporating 50 grams per day of flaxseed (for 4 weeks) in the diet, reduced the LDL-C levels from 25% to 18 % (Cunnane *et al.*, 1993).

Flaxseed meal is considered to be a prosperous source of bioactive phenolic acids (Kasote, Hedge, and Deshmukh, 2011). Presence of major components like ALA and lignin prevent chronic diseases such as cardiovascular disorders, obesity and hormone-dependent cancers (Giacomo, S., 2013).

Flaxseed meal has a significant amount of ALA in blood which can regulate glucose metabolism and can act as a high quality protein with favorable ratio of essential amino acids. Based on the nutrition and health benefits of flaxseed meal, it can be incorporated in ready to eat snack foods for higher consumer acceptability of the product (Giacomo, 2013). The shelf life of food can vary depending upon the ingredients used, water activity, moisture content, lipid oxidation, enzyme activity and other deteriorating factors. Generally, cereals and crackers are predicted to have more than 9 months of shelf life. A shelf life study for nearly 9 months would be time consuming and also laborious. (Siripatrawan, 2008). Thus an accelerated shelf life study is done for a month or two to predict the actual shelf life of the cracker. Accelerated shelf study is a model where the food is intentionally subjected to factors which aid in faster deterioration. The test can be completed within a short span of time and can be validated later with the exact conditions.

Statement of the Problem

Considering the international demand of foods with high nutritional quality which are ready to eat, low in cost and to possess a greater shelf life, the present study focused on the shelf life determination of a cracker developed by two major byproducts, hazelnut meal and flaxseed meal. The cracker was developed mixing equal proportions of hazelnut meal and flaxseed meal with honey, salt and baking powder. The dough thus formed was sheeted onto a sheet with thickness 1/8th of an inch. The sheets were then dehydrated for 12 hours at 145F, cut and then

packed accordingly for the purpose of the study. The Shelf life prediction of the cracker will give the producer an idea about the conditions to be maintained around the product, so that the product reaches the consumer with all the nutritional aspects intended for him or her.

Objective of the study

The basic objective of the study was to determine the shelf life of the cracker (in days) using the integrated GAB model. Specific sub-objectives of the study were

1. To investigate the type of sorption isotherm the hazelnut cracker tends to follow at 25 °C.
2. To verify and quantify the goodness fit of the GAB model to the Sorption isotherm generated for the cracker.
3. To determine in which condition (closed or open) the cracker will have greater shelf life.

Assumptions and Limitations

The assumptions of the study are as follows:

1. We assume that the pressure inside the HDPE pouch in closed or open condition is zero.

Definition of terms

Antioxidant. According to Merriam- Webster (2013), “an antioxidant is a substance that inhibits oxidation or reaction promoted by oxygen, peroxides, or free radicals”.

Critical moisture content. According to Azanha. & Faria. (2005), the critical moisture content is the moisture content at which the product loses its crispiness to a level that would be rejected by the consumer.

Environmental chamber. Environmental chambers are used to create a controlled temperature environment for package and shelf life testing.

Omega 3 fatty acid. According to Ward (2007), omega-3 fatty acid is a polyunsaturated fatty acid that has a double bond between the third and fourth carbon from the end with the methyl group (CH₃).

Shelf life. According to Ward (2007), shelf is the time a food can be stored and still be safe to eat.

Water activity. According to Labuza (1984) “water activity is the ratio of the vapor pressure of water in a food to the saturated vapor pressure of pure water at the same temperature”.

Water vapor transmission rate. “The steady water vapor flow in unit time through unit area of the body, normal to specific parallel surfaces, under specific conditions of temperature and humidity at each surface” (ASTM international, 2003, 6th edition, p.444).

Chapter II: Literature Review

This study aimed at determining the shelf life of the hazelnut cracker. The use of water sorption isotherm data of the cracker, and shelf life simulation with the integrated GAB model lead to the shelf life determination of the cracker.

Moisture Sorption Isotherm

The relation between the moisture content and the water activity at constant temperature is known as the moisture sorption isotherm (Labuza, 1984). Each food material will have unique sorption isotherm at constant temperature and this data plays a crucial role in determining the optimal storage conditions for the food (Guine, 2009). Though the knowledge of sorption isotherms is extensively used to predict the shelf life (Al-Mushtaseb *et al.*, 2004), it can also be used in determining other parameters like design and optimization of drying equipment (Andrade *et al.*, 2011).

The modern methods of determining the sorption isotherms are the impedance spectroscopy technique and the light reflection or infrared spectroscopy technique (Van & Goossens, 2004). But for food products sorption isotherm can be measured in three different methods, gravimetric method, manometric method and hygrometric method (Iglesias & Chirife, 1978). For the purpose of the study the gravimetric method (the weight of the sample is measured with balance) has been used till the samples reach an equilibrium.

Classification of Sorption Isotherms

The sorption isotherms are mainly classified into five types (Brunauer *et al.*, 1940). The types of the sorption isotherms according to Brunauer are as follows. Type 1 is known as the Langmuir isotherm and the sorption curves are convex upwards. Type 2 is known as the sigmoidal sorption isotherm which considers the multilayers and the internal surface of a

material and is concave upwards. Type 3 is used to depict the glass transition temperature of food products. Type 4 sorption isotherm describes swellable nature of the food. Type 5 is known as Brunauer-Emmett-Teller (BET) multilayer adsorption isotherm and is related to the isotherms in type 2 and type 3 (Andrade *et al.*, 2011). The most commonly found isotherms on foods are type 2 and type 4. Therefore one of the objectives of the study was to investigate the type of sorption isotherm the hazelnut cracker tend to follow.

Mathematical Models Used to Determine Sorption Isotherm

Many models can be found in the literature which determines the sorption behavior of the foods. Models which have been proposed and used for food products since decades are the BET (Brunauer-Emmett-Teller) equation, GAB (Guggenheim-Anderson-de Boer) model, Chen, Hasley, Henderson, Smith, Oswin, Lewicki, Chung and Pfost, Iglesias-Chirife equation and the Peleg model (Furmaniak *et al.*, 2009; Guine, 2009).

The GAB and BET models have been considered to be important and versatile sorption models respectively for foods which follow the type 2 sorption isotherm. (Siripatrawan & Jantawat, 2006). Though the BET model has been used for many food products, the GAB model has an advantage over the BET model because of its strong theoretical background and it can be considered as an upgraded version of Langmuir and BET theories (Andrade, 2011). The second important factor of using a GAB model is that it can be used for a wider range of water activities (0 to 0.95) unlike BET model which can only show best results when water activity is less than 0.60. (Siripatrawan & Jantawat, 2006). Hence for the purpose of the study the GAB model has been used to determine the shelf life of the cracker.

Shelf Life Simulation to Predict the Shelf Life of Cracker

The above models are developed to ease the process of shelf life testing of food products, especially of those which are moisture sensitive. The low moisture foods generally have greater periods of shelf life and the actual shelf life testing would be costly and time consuming. Hence the simulation models are widely used in predicting the shelf life of the low-moisture foods. The Simulation models are based on the relationship between moisture adsorption of food product, the barrier properties of the packaging material and the environment in which the food is stored (Azanha & Faria, 2005).

Determination of Critical Moisture Content

According to Azanha. & Faria. (2005), the critical moisture content is the moisture content at which the product loses its crispiness to a level that would be rejected by the consumer. The critical moisture content plays a crucial role in determining the shelf life of the cracker using an integrated GAB model equation.

The critical moisture content (CMC) can be determined in two ways. Most commonly it is determined from a sensory analysis of the cracker. The panel members would be rating the cracker for its crispiness and acceptability. The other way predicting is purely based on the previous literature or studies conducted on similar products. For the purpose of the study, determination of CMC was based on the previous literature available on similar crackers rather than a sensory panel analysis. The CMC of the hazelnut cracker was determined to be ranging between 5.6% and 6.2% (Azanha. & Faria. 2005; Siripatrawan & Jantawat, 2006; Guine, 2009). Lower the CMC lower is the predicted shelf life, therefore selecting the lower range can eliminate the experimental bias of predicting higher shelf life. CMC of 5.6% was used to determine the shelf life of the cracker

Chapter III: Methodology

The purpose of this study was to uncover the sorption characteristics of the hazelnut cracker at room temperature subjected at different relative humidity conditions. More specifically, the study aimed at determining the shelf life of the cracker using the sorption data and integrated GAB model.

Determination of Initial Moisture Content and Critical Moisture Content

Hazelnut crackers of 2-3 grams were put in an aluminum dishes (5 samples). The dishes were placed in the vacuum oven and dried at 98-100 °C and 0.08 Mpa for about 8 hours. After drying the dishes were weighed on dry basis and the initial moisture content of the crackers was calculated according to AACC (2000). On the other hand, the critical moisture content was obtained from the literature review.

$$\%M_i = \frac{W_f - W_p}{W_d} \times 100 \quad (1)$$

Where, M_i is the initial moisture content of the cracker

W_f is the weight of the cracker with the dish weight

W_p is the weight of the empty dish

W_d is the dry weight of the cracker

Sorption Isotherm Determination

The salt solutions used for the purpose of the study were Lithium Chloride (a), Magnesium Chloride (b), Potassium Carbonate (c), Sodium Nitrite (d), Sodium Chloride (e), Potassium Chloride (f) and Potassium Sulfate (g). The salt solutions prepared according to a standard protocol (Appendix A) maintained a relative humidity of 12%, 33%, 45%, 60%, 75%, 85% and 97% respectively in the humidity chambers (Figure 1).

Five samples each approximately weighing 3-3.3 grams were placed in an aluminum dishes and placed inside the seven humidity chambers at 25 °C as shown in (Figure 2). The dishes were weighed twice every week until the weights of dishes reached an equilibrium or in other words at least two consecutive weights of the dishes were same (Azanha. & Faria., 2005).

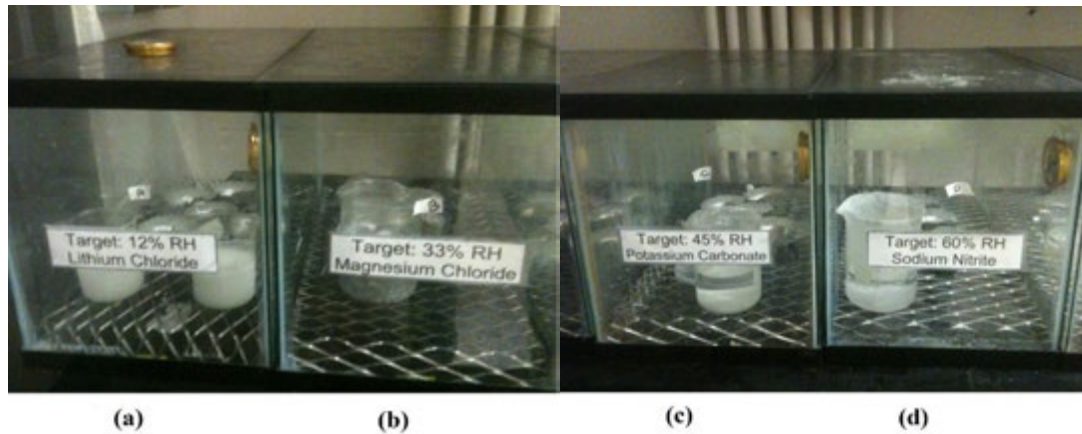


Figure 1. The experimental setup of the relative humidity chambers with the cracker and the salt solution.

The Equilibrium Moisture Content (EMC) was calculated after the cracker samples have attained a constant weight with no exchange of mass with the surrounding.

$$M_e = \left[\frac{W_e}{W_i(M_i + 1)} \right] - 1 \quad (2)$$

Where, W_e was the equilibrium weight achieved

W_i was the initial weight of the cracker

M_i was the initial moisture content of the cracker.



Figure 2. A top view of the samples in a humidity chamber maintained at 60% RH

GAB Calculations

The data obtained corresponding to the water activity (a_w) and the moisture content at the room temperature were adjusted to the GAB (Anderson, 1946; de Boer, 1995; Guggenheim, 1995) equation in order to determine the best fit.

The GAB equation was used to model the sorption isotherm of hazelnut cracker was as

$$\frac{W_m}{M} = \frac{(1 - ka_w) \times (1 - ka_w + Cka_w)}{Cka_w} \quad (3)$$

W_m – Monolayer capacity

M – predicted equilibrium moisture content of the product

k – Kinetic constant related to the multi-layer sorption

a_w – water activity (=RH%/100)

C – Kinetic constant related to the sorption of first layer.

This standard equation can be also rearranged as follows and has been used to model the water sorption of hazelnut cracker (Kaymak and Gedik, 2004) as follows:

$$m_p = \frac{W_m C k a_w}{(1 - K a_w a_w + C k a_w)} \quad (4)$$

The above equation can further be simplified and can be expressed as a polynomial function (Azanha and Faria, 2005) as follows:

$$\frac{a_w}{M_e} = \alpha(a_w)^2 + \beta a_w + \gamma \quad (5)$$

The constants for this equation are derived from the roots of the polynomial equation of the sorption isotherm curve. Hence the above equation can further be broken down to the following equations:

$$\alpha = \frac{k}{W_m} \left[\frac{1}{C} - 1 \right]; \quad \beta = \frac{1}{W_m} \left[1 - \frac{2}{C} \right]; \quad \gamma = \frac{1}{W_m C k} \quad (6)$$

$$T = \frac{\beta^2}{-\alpha\gamma} + 4 \quad (7)$$

$$C = \frac{T + \sqrt{T^2 - 4T}}{2} \quad (8)$$

$$W_m = \left[1 - \frac{2}{C} \right] \times \frac{1}{\beta} \quad (9)$$

$$k = \frac{1}{C W_m \gamma} \quad (10)$$

To determine the goodness of the fit for the GAB model determination coefficient (R^2), mean relative percentage deviation modulus, E (Kaya and Kahyaoglu, 2005) and percentage root mean square error, RSME (Al-Muhtaseb et al., 2004) were calculated as follows:

$$E = \frac{100}{N} \sum_{i=1}^n \frac{|m_e^i - m_p^i|}{m_e^i} \quad (11)$$

$$RSME = \sqrt{\frac{\sum_{i=1}^n (m_e^i - m_p^i)^2}{N}} \quad (12)$$

Where m_e is the experimental EMC

m_p is the predicted value

N is the number of experimental data points

The fit was considered to be a good fit if the mean relative percentage deviation modulus values was less than 10%. The lower the E and RSME values the better the fit or goodness of the model (McMinn and Magee, 2003).

Water Vapor Transmission Rates and Permeability Coefficient

The cracker's shelf life was tested under two conditions, open condition and a closed condition. For both the conditions American Society for Testing and Materials, ASTM 96 E standard (Appendix B) for testing the water vapor transmission rate (WVTR) through the packaging film.

Closed Condition

The pouch was made using an HDPE (High Density Polyethylene) film. To make one pouch, two sheets of 9 in (length) and 8 in (width) film were cut from the HDPE roll, sealed using an impulse heat sealer (figure 3). The heat sealer's jaws heat up to the specified temperature and the seal is created when the material is clamped between the jaws. Three sides of the pouch were sealed with the heat sealer.



Figure 3. Advanced thermal impulse heat sealing equipment, Mansfield, Texas.



Figure 4. Heat Seal Scope

The quality of sealing was evaluated using the heat seal scope (Figure 4) and non-defective pouches were selected for tests. Ten pouches were made of which, 5 were used for permeability test and 5 were used for the validation test. Among the 5 which were used for the permeability test, approximately 10 grams of desiccant (CaCO_4) was weighed into three pouches and the fourth side was sealed as shown in Figure 5. The other two pouches were used as a control (Figure 6) to indicate the amount of water gained by the packaging material itself. All the pouches were weighed (day 0 weight) and placed in an environmental chamber maintained at 25

$^{\circ}\text{C}$ temperature and 80% RH. Care was taken so that the pouches did not overlap each other in the environmental chamber. The pouches were weighed every 4th day for 36 days and the readings were recorded in table 4. The other 5 pouches were used for the validation test.



Figure 5. (a) Closed condition with 10 gm desiccant, (b) Control – no desiccant

Open Condition

Ten readily available pouches in the market were used for the study. Five of them were used for the permeability test and the other 5 were used for the validation test. For the permeability test, 3 pouches were selected and 50 grams of desiccant was placed in them. The fourth side of the pouch was not sealed but was just folded inward and then clipped using a binder clip and placed in a box (Figure 6). The other 5 samples were set for the validation test.



Figure 6. Open condition – cracker placed in a HDPE pouch, clipped and placed in box

The permeability of the HDPE pouch in both the conditions was calculated based on the following equation:

$$WVTR = \frac{q}{t} \quad (13)$$

Where, q/t is the slope of the equation when time in days is plotted versus weight gained in grams

$$P = \frac{WVTR}{\Delta P} \quad (14)$$

Where P is the permeability coefficient, ΔP is the difference of vapor pressures outside the surface of the material and surface inside the pouch.

Validation Test

The validation test was conducted to predict the results in a real time scenario and to validate the GAB and WVTR model results. It was done simultaneously with WVTR test with same test parameters and conditions but for the fact that the pouch had 50 grams of hazelnut cracker in both open and closed conditions. Weight gain profiles were recorded for 36 days taking readings every 4th day.

Shelf Life Determination Using Integrated GAB model

The shelf life of the cracker was calculated using the GAB model by using the following equation (Diosady *et al.*, 1996).

$$t = H/\varphi \quad (15)$$

$$H = [M_f - M_i + \frac{2W_m C}{\varepsilon} \ln \left(\frac{\varepsilon M_f - 2W_m C}{\varepsilon M_i - 2W_m C} \right)] \quad (16)$$

$$\varepsilon = (1 - C)[(2K a_w 0) - 2]$$

$$\varphi = \frac{Pp_s}{2k(1-C)W_d}$$

Where, t is the shelf life of the cracker in days

W_m , K and C are the GAB constants

W_0 is the storage humidity

M_f is the critical moisture content

M_i is the initial moisture content

p_s is the saturated vapor pressure at 25 °C

P is the Permeability coefficient

W_d is the product weight

Data Analysis

All the data was analyzed using Microsoft® Excel 2010 (Microsoft Corporation, Cambridge, Massachusetts, USA). The Microsoft Excel was extensively used to calculate all the averages, standard deviation, comparing the two variables and to generate the graphical representation of the variables.

Chapter IV: Results and discussion

The study was focused on determining the shelf life of the hazelnut cracker using the integrated GAB model. All the experiments were done for a span of 36 days at 25 °C in controlled atmospheres.

The initial moisture content of the hazelnut cracker was 3.059% (dry basis) with a variation of 0.327%. The equilibrium moisture content (EMC) of the cracker calculated at different relative humidity (RH) conditions based on equation (1) are shown in Table 1.

Table 1

Calculated (experimental) EMC for the hazelnut cracker

Relative humidity RH	Average EMC %
12	3.4398
33	4.4398
45	5.4398
60	6.4398
75	7.4398
85	8.4398
97	9.4398

These experimental points were plotted on a graph with RH or water activity (RH%/100) on X-Axis and EMC % (dry basis) on Y-axis (Figure 8). An experimental sorption isotherm curve for the cracker at 25°C followed a type II sigmoidal pattern.

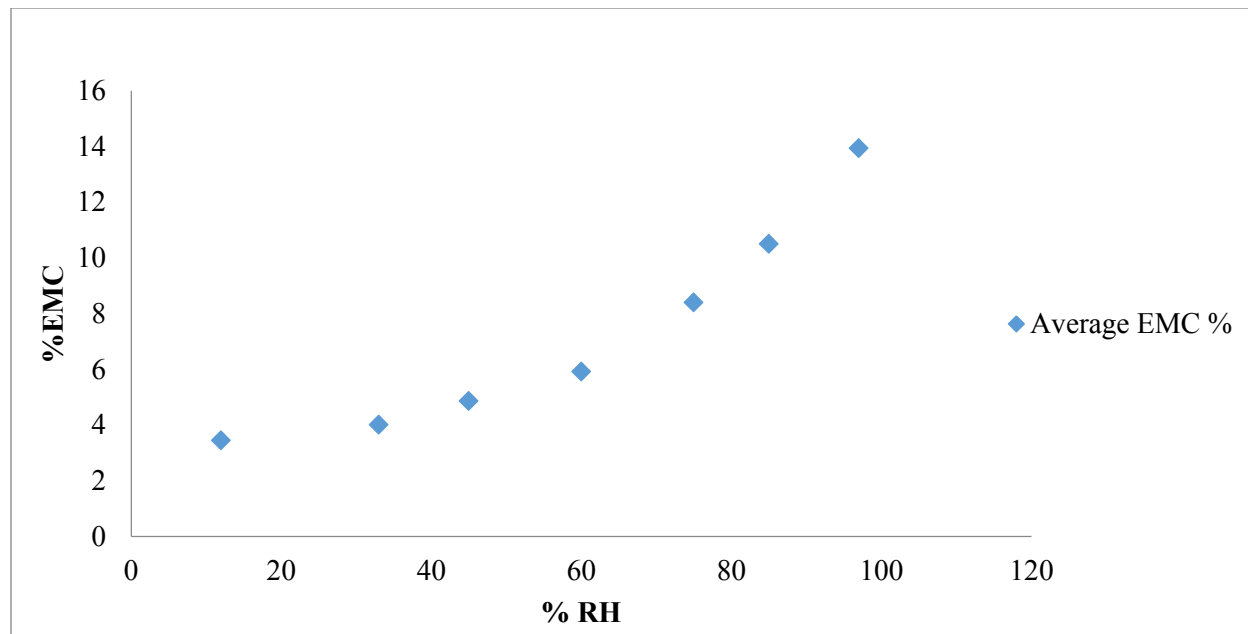


Figure 7. Experimental sorption isotherm of hazelnut cracker.

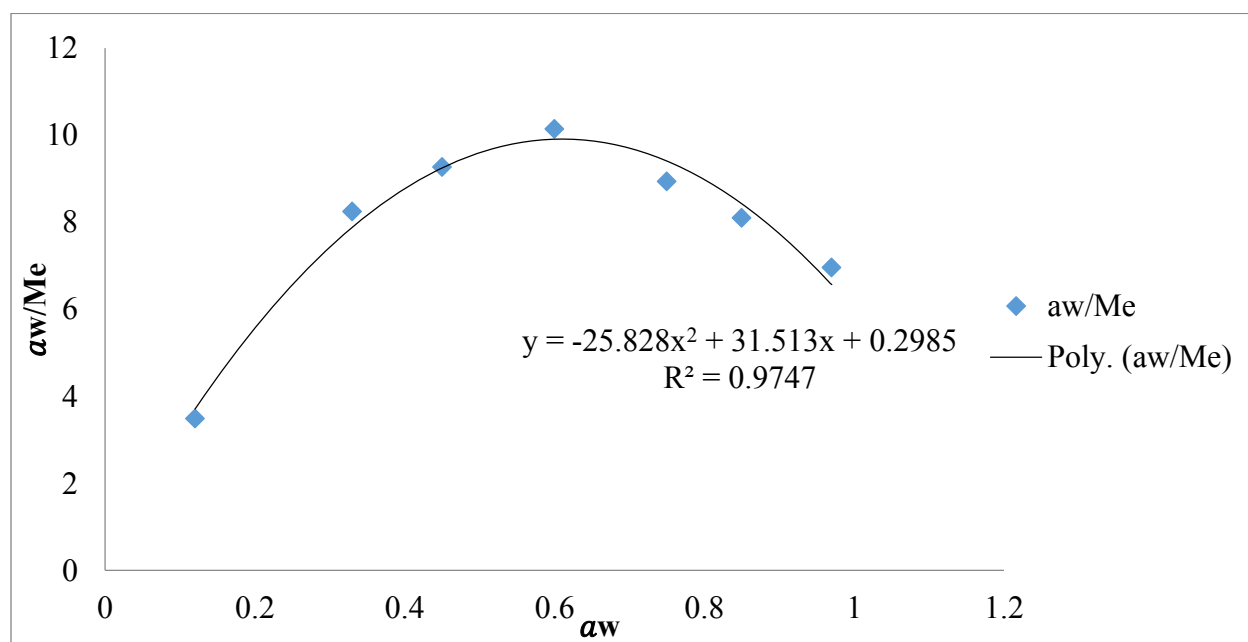


Figure 8. Polynomial version of GAB model.

The polynomial version of the GAB model (Figure 9) was obtained by taking the ratio of water activity to the equilibrium moisture content on Y axis and its relative water activity on X-axis. From the equation of the curve, the roots of the rearranged GAB equation (4) α , β and γ

were interpreted to be -25.828, 31.513 and 0.2985 respectively. The coefficient of determination R^2 was found to be 0.9747.

The GAB constants C and K calculated based on equation (5), (6), (7), (8), and (9) were 131.8 and 0.8133 respectively. The predicted EMC of the hazelnut cracker thus calculated based on equation (3) were compared to the experimental EMC values in Table 2. Figure 10 depicts the trend followed by both the sorption isotherms subjected at similar conditions.

Table 2

Experimental EMC versus Predicted EMC

RH%	Experimental M_e	GAB M_e
12	0.034398445	0.032361266
33	0.040006285	0.041850976
45	0.048555271	0.048652962
60	0.059169415	0.060555781
75	0.083931523	0.079744817
85	0.10496307	0.10090434
97	0.139329332	0.147763482

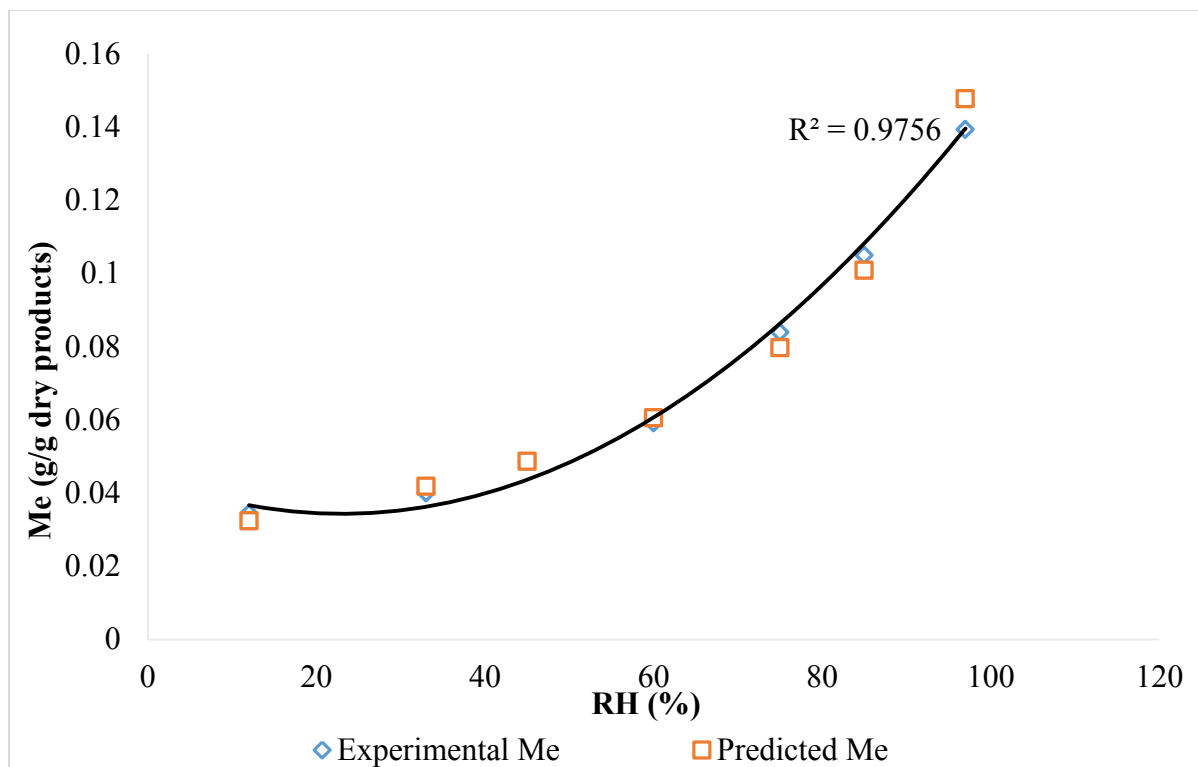


Figure 9. Moisture sorption isotherm for the cracker fitted to GAB model.

Goodness of the Fit

The relative percent deviation modulus (E) was determined to be 3.9979% and the root mean square error (RSME) was determined to be 0.0561 according to the equations (10) and (11) respectively. Based on Figure 10, the coefficient of determination was also found to be 0.9756. All the parameters when compared to the parameters in a similar study conducted on jasmine crackers, were falling within the range, $1 < \%E < 10$, $RSME < 1$ and R^2 close to the value one as shown in table 2 (McMinn and Magee, 2003). Also another study which analyzed the fitting ability of the GAB model concluded that the sorption isotherm would follow a type II sigmoidal shape and would be a good fit if the range of the GAB parameters was: $0.24 < k \leq 1$ and $5.67 \leq C \leq \infty$ (Lewicki, 1997). The results of the study show that the k and C values were 0.813 and

131.80 respectively. (Shown in Table 2). Thus the GAB model was considered to be good fit for the experimental sorption isotherm of the hazelnut cracker.

Table 3

Coefficients of GAB isotherms for hazelnut cracker at 25 °C

Estimated parameters	Experimental results
W_m	0.0312
C	131.80
k	0.813
R^2	0.9756
%E	3.9979
RSME	0.0561

Water Vapor Transmission Rate Test

The average weight gain (G) of the pouch at 25 °C and 80% relative humidity were recorded in Table 3. The notation G4 means the weight gained by the pouch on day 4, C HDPE means closed condition for high density polyethylene and O HDPE means open condition.

The average water vapor transmission rate values of the packaging material (HDPE pouch) were calculated for each time interval by subtracting the control weight (no desiccant) from its corresponding average weight gain of pouches and recorded in Table 4.

Table 4

Pouch weight gain at different time intervals

Weight gain	Pouch		
	CHDPE	OHDPE	Control
G ₀	0	0	0
G ₄	0.17±0.036	0.27±0.063	0
G ₆	0.27±0.032	0.437±0.045	0.01±0.02
G ₈	0.373±0.026	0.563±0.057	0.025±0.055
G ₁₀	0.527±0.041	0.81±0.098	0.03±0.05
G ₁₂	0.607±0.048	0.913±0.058	0.025±0.045
G ₁₄	0.717±0.041	1.253±0.037	0.005±0.045
G ₁₆	0.85±0.045	1.573±0.073	0.025±0.045
G ₂₂	0.93±0.048	1.99±0.11	0.015±0.035
G ₂₆	1.02±0.054	2.33±0.138	0.02±0.02
G ₂₈	1.11±0.059	2.653±0.2	0.025±0.015
G ₃₂	1.19±0.059	3.067±0.175	-0.005±0.035
G ₃₆	1.28±0.045	3.467±0.143	0±0.04

CHDPE means closed condition HDPE pouch

OHDPE means open condition HDPE pouch

G₄ means the amount of weight gained by the pouch on day 4

Table 4 shows that the average water transmission rates of the HDPE pouch for open treatment were higher than that of the closed treatment at any given time interval. This was predicted as the open treatment pouches were just folded and clipped whereas the closed treatment pouches were thermo sealed.

Table 5

Average water vapor transmission values

Weight gain	Pouch	
	CHDPE	OHDPE
G ₀	0	0
G ₄	0.17	0.25
G ₆	0.26	0.36
G ₈	0.35	0.50
G ₁₀	0.50	0.74
G ₁₂	0.58	0.83
G ₁₄	0.71	1.20
G ₁₆	0.83	1.52
G ₂₂	0.92	1.95
G ₂₆	1	2.27
G ₂₈	1.09	2.60
G ₃₂	1.20	3.03
G ₃₆	1.28	3.42

CHDPE means closed condition HDPE pouch

OHDPE means open condition HDPE pouch

G₄ means the amount of weight gained by the pouch on day 4

The average water vapor transmission rates of the pouches shown in Figure 11 were plotted against the time in days on X-axis and weight gain in grams on Y-axis. The Determination of coefficient (R^2) in open condition was 0.9695 and in closed condition was 0.9959.

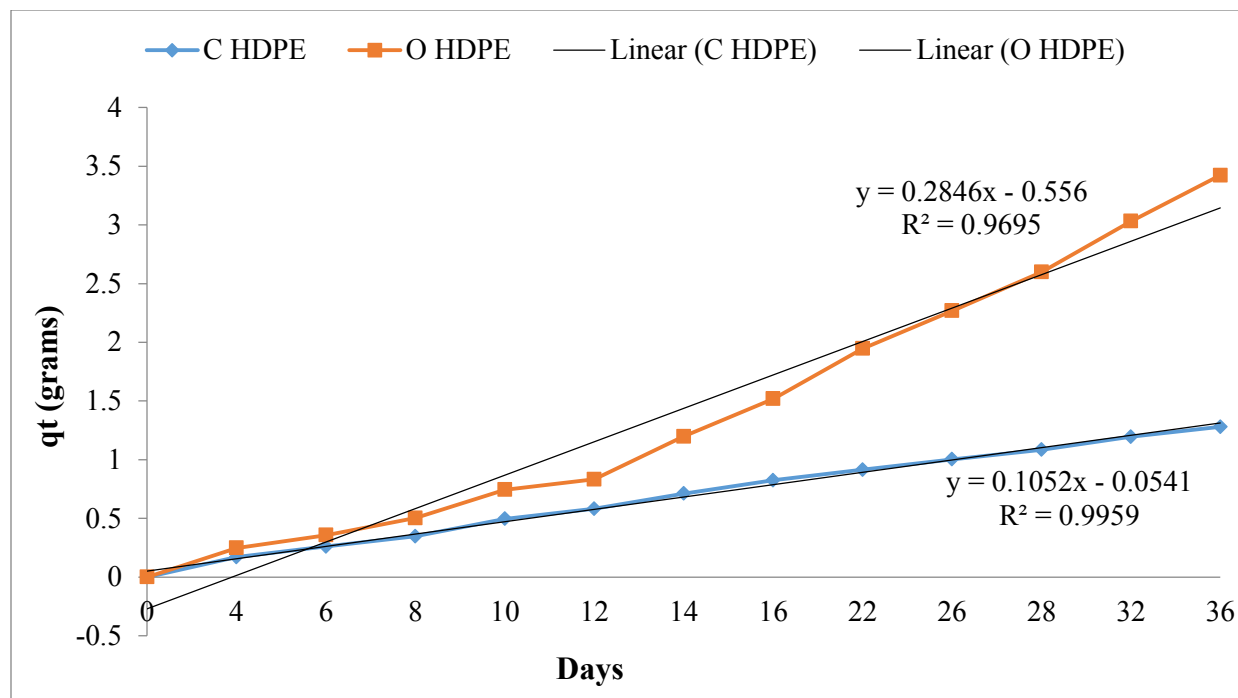


Figure 10. Water vapor transmission rate of the packaging material in closed and open treatments.

The average water vapor transmission rates of the pouches shown in Figure 11 were plotted against the time in days on X-axis and weight gain in grams on Y-axis. The Determination of coefficient (R^2) in open condition was 0.9695 and in closed condition was 0.9959.

Permeability Coefficient of the Pouches

The permeability coefficient calculations were done based on equations (12) and (13) which were derived from the slope of the lines obtained from Figure 11. The permeability coefficient for closed (sealed) pouch was determined to be 0.0055 g/pkg.day.mmHg and for open (non-sealed and boxed) pouch was determined to be 0.01497 g/pkg.day.mmHg.

Shelf Life Determination

The shelf life of the cracker was determined by integrated GAB model using the equations (14) and (15). The pouch permeability values and the GAB constants calculated, the initial moisture content, the critical moisture content, experimental conditions and the weight of the sample used for the study were used to determine the shelf life of the hazelnut cracker. The shelf life (in days) of 50 grams of cracker at 25⁰C and 80% relative humidity in open condition was determined to be 16.6 or 17 days and in closed conditions was 44.9 rounded of to 45 days.

Validation Test

The validation test was conducted to verify the correctness of the results obtained from the shelf life calculations. The results of this test are shown in Table 5. The percent moisture gain for both the conditions increased gradually for each time interval, according to Table 5. The cracker under open conditions attained a moisture content of 5.12% in 14 days and reached to 5.76% in 16 days. The critical moisture content for the hazelnut cracker obtained from literature and which has been used for the shelf life calculation was 5.6% (%CMC) suggesting that the shelf life of the cracker is predicted to be between 14 to 16 days. Comparing this result with the shelf life calculation we can conclude that the validation test held good for the open condition.

The percent moisture gain for the closed condition was at much lower rate than the open condition and hence the cracker didn't reach the critical moisture content during the test period. At the end of 36th day the moisture gain was 4.20% (%MC 36th day).

Table 6

Percent Moisture gain for respective time intervals in open and closed condition

%	Day	Day	Day	Day	Day	Day	Day	Day	Day	Day	Day	Day	Day
Mg	0	4	6	8	10	12	14	16	22	26	28	32	36
O	4.02	4.3	4.58	4.8	5	5.12	5.52	5.76	6.16	6.46	6.82	7.14	7.3
C	2.11	2.19	2.31	2.36	2.45	2.59	2.93	3.06	3.38	3.58	3.88	4.09	4.20

Where % Mg means Percent Moisture Gain

O is for Open condition samples

C is for Closed condition samples

To predict at what day the cracker might have attained a moisture gain of 5.6%, an average moisture gain for each time interval was calculated. This was further divided by the number days in the time interval, gave the average weight gain percent for each day (%AMGPD). The weight gain percent thus calculated was 0.174% for each day.

The estimated number of days (ED) to be added to the 36th day to determine the shelf life of the hazelnut cracker in the closed condition was calculated based on the following formula:

$$ED = \frac{\%CMC - \%MC \text{ 36th day}}{\%AMGPD} \quad (17)$$

The estimated number of days was determined to be 8.04 days rounded of to 8 days. Hence the validated shelf of the hazelnut cracker for closed condition was found to be 44 days (36+ED). The validation shelf life calculation of the cracker (44 days) had a close match with the predicted shelf life calculation (45days) suggesting that the integrated GAB model used for the study had a good fit. Because the EMC for the cracker was reached within 36 days the test was terminated and the data was analyzed but if the test was prolonged through 46 days then the predicted shelf life would have been more accurate.

Chapter V: Conclusion

The sorption isotherm of the cracker followed a type II sigmoidal trend and the GAB model used was successful in predicting the shelf life of the cracker. The results of the study were in accordance with the previous studies conducted on similar products like the jasmine rice crackers and cereals (Siripatrawan & Jantawat, 2006).

The shelf life of the cracker tested under accelerated deteriorating conditions exhibited a shelf life 16.6 days and 44.9 days in open and closed conditions respectively for 50 grams of cracker sample. These results change drastically under real time situations due to the variations in relative humidity or the sample size to the package size ratio. The study was conducted with 50 grams of cracker in a pouch with approximately 140 inch² surface area. The sample weight was low compared to the available space inside the pouch. Hence increasing the sample size would definitely increase the shelf life of the cracker.

Also, the experimental testing was done maintaining 80% RH in the environmental chamber. But the average RH during processing, distribution, shipping, at the grocery shelf or even in the warehouse would range between 50% and 60% RH. Hence reducing the experimental RH to predict the real time shelf life would increase the predicted shelf life. Table 6 shows the shelf life under different conditions taking the base as the experimental result. Since the cracker size is too small for the package surface and ideal sample weight to be 200 grams and the RH around the pack to be 60% the shelf life can be estimated to be approximately 302 days. This result can be correlated with a similar study on shelf life simulation (Siripatrawan, 2008).

Table 7

Shelf life variation based on experimental conditions and real time conditions

Conditions RH %	Shelf life in days*					
	Open condition			Closed condition		
	50 grams	100 grams	200 grams	50 grams	100 grams	200 grams
80**	17**	34	67	45**	90	180
70	20	39	77	52	104	207
60	28	56	112	76	151	302
55	49	98	196	133	265	529

*Shelf life in days was rounded off to the next nearest integer

**Means experimental condition

Recommendations

The following recommendations can be made based on the results of the study

1. The study was conducted only at 25 0 C as the cracker was supposed to be stored at room temperature. But testing the cracker at least two more temperatures, one higher and the other lower than 25 0 C would have established either a decreasing or increasing trend of shelf life.
2. Determining the shelf life based on critical moisture through literature was accurate enough to predict the shelf life but determining it based on the sensory testing prior to the study would have given more accurate results and created a robust methodology.
3. Though both the meals (Hazelnut meal and the Flaxseed meal) used to develop the cracker were defatted, an extended study can be conducted to determine if the presence of fat affects the shelf life.

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Appendix A: Raw Data for Initial Percent Moisture Content (%MC) of the Hazelnut

Cracker

Sample no.	Empty dish wt	Sample wt	Total wt	After 8 hours	%MC dry basis
1	2.285	3.503	5.788	5.6836	3.0720
2	2.277	3.488	5.765	5.6427	3.6337
3	2.28	3.492	5.772	5.6672	3.0940
4	2.28	3.48	5.76	5.6646	2.8186
5	2.292	3.498	5.79	5.6988	2.6770
Average					3.0590
Std Dev					0.3272

Appendix B: Raw Data for the Permeability Test

	No	D0	D4	D6	D8	D10	D12	D14	D16	D22	D26	D28	D32	D36
CHDPE	1	21.34	21.56	21.65	21.75	21.92	21.99	22.11	22.25	22.33	22.42	22.51	22.59	22.67
	2	21.29	21.43	21.52	21.65	21.81	21.92	21.96	22.09	22.16	22.24	22.32	22.40	22.51
	3	21.08	21.23	21.35	21.43	21.56	21.62	21.79	21.92	22.01	22.12	22.21	22.29	22.37
	AVE	21.24	21.41	21.51	21.61	21.76	21.84	21.95	22.09	22.17	22.26	22.35	22.43	22.52
	SD	0.11	0.14	0.12	0.13	0.15	0.16	0.13	0.13	0.13	0.12	0.12	0.12	0.12
Control	1	11.49	11.49	11.52	11.57	11.57	11.56	11.54	11.56	11.54	11.53	11.53	11.52	11.53
	2	11.41	11.41	11.40	11.38	11.39	11.39	11.37	11.39	11.39	11.41	11.42	11.37	11.37
	AVE	11.45	11.45	11.46	11.48	11.48	11.48	11.46	11.48	11.47	11.47	11.48	11.45	11.45
	SD	0.04	0.04	0.06	0.09	0.09	0.09	0.09	0.09	0.07	0.06	0.05	0.08	0.08
OHDPE	1	73.02	73.23	73.40	73.51	73.71	73.87	74.23	74.50	74.89	75.20	75.46	75.90	76.34
	2	69.44	69.80	69.93	70.07	70.37	70.43	70.69	71.02	71.41	71.72	72.04	72.46	72.86
	3	69.04	69.29	69.48	69.61	69.85	69.94	70.34	70.70	71.19	71.55	71.96	72.34	72.70
	AVE	70.50	70.77	70.94	71.06	71.31	71.41	71.75	72.07	72.50	72.82	73.15	73.57	73.97
	SD	1.79	1.75	1.75	1.74	1.71	1.75	1.76	1.72	1.69	1.68	1.63	1.65	1.68
Control	1	19.82	19.87	19.99	19.98	20.00	20.02	19.99	19.97	19.98	19.98	19.98	19.97	19.97
	2	18.45	18.45	18.44	18.41	18.40	18.41	18.39	18.41	18.39	18.40	18.40	18.37	18.39
	AVE	19.14	19.16	19.22	19.20	19.20	19.22	19.19	19.19	19.19	19.19	19.19	19.17	19.18
	SD	0.69	0.71	0.77	0.79	0.80	0.81	0.80	0.78	0.80	0.79	0.79	0.80	0.79

Appendix D: Pouch Weight Gain at Time Intervals Raw Data

	No	G0	G4	G6	G8	G10	G12	G14	G16	G22	G26	G28	G32	G36
CHDPE	1	0.00	0.22	0.31	0.41	0.58	0.65	0.77	0.91	0.99	1.08	1.17	1.25	1.33
	2	0.00	0.14	0.23	0.36	0.52	0.63	0.67	0.80	0.87	0.95	1.03	1.11	1.22
	3	0.00	0.15	0.27	0.35	0.48	0.54	0.71	0.84	0.93	1.04	1.13	1.21	1.29
	AVE	0.00	0.17	0.27	0.37	0.53	0.61	0.72	0.85	0.93	1.02	1.11	1.19	1.28
	SD	0.00	0.04	0.03	0.03	0.04	0.05	0.04	0.05	0.05	0.05	0.05	0.06	0.06
Control	1	0.00	0.00	0.03	0.08	0.08	0.07	0.05	0.07	0.05	0.04	0.04	0.03	0.04
	2	0.00	0.00	-0.01	-0.03	-0.02	-0.02	-0.04	-0.02	-0.02	0.00	0.01	-0.04	-0.04
	AVE	0.00	0.00	0.01	0.03	0.03	0.03	0.00	0.03	0.01	0.02	0.02	-0.01	0.00
	SD	0.00	0.00	0.02	0.05	0.05	0.04	0.04	0.04	0.03	0.02	0.01	0.04	0.04
OHDPE	1	0.00	0.21	0.38	0.49	0.69	0.85	1.21	1.48	1.87	2.18	2.44	2.88	3.32
	2	0.00	0.36	0.49	0.63	0.93	0.99	1.25	1.58	1.97	2.28	2.60	3.02	3.42
	3	0.00	0.25	0.44	0.57	0.81	0.90	1.30	1.66	2.15	2.51	2.92	3.30	3.66
	AVE	0.00	0.27	0.44	0.56	0.81	0.91	1.25	1.57	2.00	2.32	2.65	3.07	3.47
	SD	0.00	0.06	0.04	0.06	0.10	0.06	0.04	0.07	0.12	0.14	0.20	0.17	0.14
Control	1	0.00	0.05	0.17	0.16	0.18	0.20	0.17	0.15	0.16	0.16	0.16	0.15	0.15
	2	0.00	0.00	-0.01	-0.04	-0.05	-0.04	-0.06	-0.04	-0.06	-0.05	-0.05	-0.08	-0.06
	AVE	0.00	0.03	0.08	0.06	0.06	0.08	0.05	0.05	0.05	0.05	0.05	0.04	0.04
	SD	0.00	0.03	0.09	0.10	0.12	0.12	0.11	0.09	0.11	0.11	0.11	0.11	0.10

Appendix E: Raw Data for Validation Test

	No.	D0	D 4	D 6	D8	D10	D12	D14	D16	D22	D26	D28	D32	D36
Closed	1	63.42	63.46	63.52	63.57	63.72	63.77	63.81	63.89	64.05	64.15	64.26	64.37	64.43
	2	61.69	61.76	61.80	61.87	61.98	62.01	62.15	62.23	62.37	62.49	62.65	62.72	62.77
	3	61.27	61.33	61.39	61.43	61.52	61.59	61.67	61.78	61.93	62.03	62.18	62.32	62.39
	AVE	62.13	62.18	62.24	62.29	62.41	62.46	62.54	62.63	62.78	62.89	63.03	63.14	63.20
	SD	0.93	0.92	0.92	0.92	0.95	0.94	0.92	0.91	0.91	0.91	0.89	0.89	0.89
Control	1	11.02	11.02	11.03	11.06	11.08	11.06	11.04	11.03	11.03	11.04	11.03	11.04	11.04
	2	11.12	11.16	11.13	11.16	11.28	11.26	11.12	11.18	11.16	11.16	11.15	11.14	11.15
	AVE	11.07	11.09	11.08	11.11	11.18	11.16	11.08	11.11	11.10	11.10	11.09	11.09	11.10
	SD	0.05	0.07	0.05	0.05	0.10	0.10	0.04	0.08	0.07	0.06	0.06	0.05	0.06
Open	1	70.5	70.6	70.7	70.8	70.9	71.0	71.2	71.3	71.5	71.6	71.8	71.9	72.0
	2	67.0	67.1	67.2	67.3	67.4	67.4	67.6	67.7	67.8	67.9	68.1	68.2	68.3
	3	68.4	68.6	68.6	68.8	68.8	68.9	69.1	69.2	69.4	69.5	69.7	69.8	68.9
	AVE	68.6	68.8	68.9	68.9	69.0	69.1	69.3	69.4	69.5	69.7	69.9	70.0	69.7
	SD	1.4	1.4	1.4	1.4	1.4	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.7
Control	1	19.82	19.87	19.99	19.98	20.00	20.02	19.99	19.97	19.98	19.98	19.98	19.97	19.97
	2	18.45	18.45	18.44	18.41	18.40	18.41	18.39	18.41	18.39	18.40	18.40	18.37	18.39
	AVE	19.14	19.16	19.22	19.20	19.20	19.22	19.19	19.19	19.19	19.19	19.19	19.17	19.18
	SD	0.69	0.71	0.77	0.79	0.80	0.81	0.80	0.78	0.80	0.79	0.79	0.80	0.79

Appendix F: Raw Data for Calculating %E and RSME

RH%	Experimental Me	Predicted Me	Me-Mp	abs(Me-Mp)
12	0.034398445	0.032361266	0.002037	0.00203718
33	0.040006285	0.041850976	-0.00184	0.001844691
45	0.048555271	0.048652962	-9.8E-05	9.76909E-05
60	0.059169415	0.060555781	-0.00139	0.001386366
75	0.083931523	0.079744817	0.004187	0.004186706
85	0.10496307	0.10090434	0.004059	0.00405873
97	0.139329332	0.147763482	-0.00843	0.008434151

Appendix G: Standard Protocol for Salt Solutions

Relative Humidity (%RH)			
Temperature °C	Lithium Chloride	Potassium Acetate	Magnesium Chloride
0	11.23 ± 0.54		33.66 ± 0.33
5	11.26 ± 0.47		33.60 ± 0.28
10	11.29 ± 0.41	23.28 ± 0.53	33.47 ± 0.24
15	11.30 ± 0.35	23.40 ± 0.32	33.30 ± 0.21
20	11.31 ± 0.31	23.11 ± 0.25	33.07 ± 0.18
25	11.30 ± 0.27	22.51 ± 0.32	32.78 ± 0.16
30	11.28 ± 0.24	21.61 ± 0.53	32.44 ± 0.14
35	11.25 ± 0.22		32.05 ± 0.13
40	11.21 ± 0.21		31.60 ± 0.13
45	11.16 ± 0.21		31.10 ± 0.13
50	11.10 ± 0.22		30.54 ± 0.13
55	11.03 ± 0.23		29.93 ± 0.16
60	10.95 ± 0.26		29.26 ± 0.18
65	10.86 ± 0.29		28.54 ± 0.21
70	10.75 ± 0.33		27.77 ± 0.25
75	10.64 ± 0.38		26.94 ± 0.29
80	10.51 ± 0.44		26.05 ± 0.34
85	10.38 ± 0.51		25.11 ± 0.39
90	10.23 ± 0.59		24.12 ± 0.46
95	10.07 ± 0.67		23.07 ± 0.52
100	9.90 ± 0.77		21.97 ± 0.60

Relative Humidity (%RH)						
Temperature °C	Potassium Carbonate	Magnesium Nitrate	Sodium Chloride	Potassium Chloride	Potassium Nitrate	Potassium Sulfate
0	43.13 ± 0.66	60.35 ± 0.55	75.51 ± 0.34	88.61 ± 0.53	96.33 ± 2.9	98.77 ± 1.1
5	43.13 ± 0.50	58.86 ± 0.43	75.65 ± 0.27	87.67 ± 0.45	96.27 ± 2.1	98.48 ± 0.91
10	43.14 ± 0.39	57.36 ± 0.33	75.67 ± 0.22	86.77 ± 0.39	95.96 ± 1.4	98.18 ± 0.76
15	43.15 ± 0.33	55.87 ± 0.27	75.61 ± 0.18	85.92 ± 0.33	95.41 ± 0.96	97.89 ± 0.63
20	43.16 ± 0.33	54.38 ± 0.23	75.47 ± 0.14	85.11 ± 0.29	94.62 ± 0.66	97.59 ± 0.53
25	43.16 ± 0.39	52.89 ± 0.22	75.29 ± 0.12	84.34 ± 0.26	93.58 ± 0.55	97.30 ± 0.45
30	43.17 ± 0.50	51.40 ± 0.24	75.09 ± 0.11	83.62 ± 0.25	92.31 ± 0.60	97.00 ± 0.40
35		49.91 ± 0.29	74.87 ± 0.12	82.95 ± 0.25	90.79 ± 0.83	96.71 ± 0.38
40		48.42 ± 0.37	74.68 ± 0.13	82.32 ± 0.25	89.03 ± 1.2	96.41 ± 0.38
45		46.93 ± 0.47	74.52 ± 0.16	81.74 ± 0.28	87.03 ± 1.8	96.12 ± 0.40
50		45.44 ± 0.60	74.43 ± 0.19	81.20 ± 0.31	84.78 ± 2.5	95.82 ± 0.45
55			74.41 ± 0.24	80.70 ± 0.35		
60			74.50 ± 0.30	80.25 ± 0.41		
65			74.71 ± 0.37	79.85 ± 0.48		
70			75.06 ± 0.45	79.49 ± 0.57		
75			75.58 ± 0.55	79.17 ± 0.66		
80			76.29 ± 0.65	78.90 ± 0.77		
85				78.68 ± 0.89		
90				78.50 ± 1.0		
95						
100						