

Moisture sorption isotherms cassava starch kaffir lime bioplastic films

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Abstract—Cassava starch can be used in the formation of biodegradable films and containers, as an alternative for polymer derived from petrol. Kaffir lime oil act as antimicrobial agent that can inhibit the growth of bacteria in the food packaging. Bioplastic films from cassava starch with 35 w/w glycerol were produced by casting method. The concentration of glycerol which is 35 w/w as a plasticizer. Equilibrium moisture content at temperature 25°C and the water activity at range 0.05 to 0.9 by using static gravimetric method was determined. Moisture sorption isotherms were measured through a standard saturated salt slurry method. Five saturated salt solutions which are KOH, NaCl, BaCl₂, KCl, NaNO₃ were prepared corresponding to wide range of water activities. The result of monolayer moisture contents of bioplastic films was evaluated using Brunauer Emmett and Telle (BET) sorption equation. The isotherms exhibited Type III behaviour. Type III behaviour, in which a small amount of water was adsorbed in low water activity and the amount absorbed in larger water activity. The moisture sorption data were fitted to BET models and a non-linear regression method was used to evaluate the value constant of the sorption equations. The highest R² values ranging between 0.9439 to 0.9850 and the value of MRPD values ranging between 9.1882 to 18.3558. The monolayer, M₀ value in this study was determined by using the BET equation and was obtained between range 0.0429-0.0469 gram water per gram dry matter following adsorption at 25°C. The bioplastic film is suitable as packaging because the microorganism unable to grow below water activity of 0.91. There is no significant effect in moisture content with an increase of concentration of kaffir lime.

Keywords— Cassava starch, Kaffir lime, Moisture sorption isotherms, bioplastic films

I. INTRODUCTION

Bioplastic film is a thin layer material to cover food products, which can be produced from biopolymers. Biopolymers have been studied extensively regarding their film-forming properties to produce bioplastic films and coatings intended as food packaging. Different biopolymers have been used in the development of bioplastic film and coatings, such as proteins, lipids, and polysaccharides. Bioplastic films can improve mechanical barrier properties and can also prolong shelf life of food [1]. Bioplastic films can be produced from a mixture of high molecular weight polymer, plasticizer and solvent. Bioplastic films are comprised of edible biopolymers and food-grade supplement. Plasticizers and any components such as starch, wheat starch are mixed with the biopolymers solutions to modify the functionality or physical properties of the films. The biopolymers may be classified into three categories, which are proteins, lipids and polysaccharides.

Starch is a naturally occurring polymer that is less expensive and plentifully obtainable. Starch is the primary storage of carbohydrate in plants and is broadly consumed polysaccharide in the human diet. Native starch, which produce in a granular form,

is one of the most important carbohydrate resource found in cereals and tubers plant such as potatoes and maize, respectively. Starch is one of the polysaccharides used to develop bioplastic film. It can be obtained from several raw materials of tropical origin such as corn, sweet potato and cassava [2]. Starch is the second most abundant natural compound, which produce about five million tons each year in Europe after cellulose. It is low cost, abundantly available, eco-friendly, biodegradable and renewable. Cassava presents a high potential to be utilized as raw material for fabricating biodegradable plastics. [3] found that cassava starch has been significantly used to produce biodegradable film with properties comparable to those prepared using other starches. Cassava is the most economical source to produce good film forming and casting properties because of high amylose content [4]. Other than that, starch-based films are edible, transparent, odourless, tasteless and colourless, which are properties for food packaging, including bioplastic films and coatings for vegetable and fruit. Cassava starch can form transparent coatings and flexible films with none preceding chemical treatment, neither plasticizer addition [5].

To produce good bioplastic films, addition of plasticizer such as glycerol is usually used. Glycerol is the most popular plasticizers in film making technique due to stability and compatibility with hydrophilic biopolymeric packaging chain [6]. The utilize of plasticizer is often needed to produce a bioplastic film. Normally used is glycerol because might be considered to be non-toxic and compatible to be used in the food industry [7]. Bioplastic film with glycerol addition as plasticizer was proven to produce good plasticity properties and can increase elasticity of the film. After adding up the glycerol, this film was not too brittle and rigid. The addition of plasticizing agents to bioplastic films is to overcome film brittleness caused by extensive intermolecular forces [4]. Plasticizers can prevent fracture of the film during handling and storage [8]. Moreover, plasticizer can affect all the physical properties of film [9].

According to [10] corn starch films consist of less than 10% w/w glycerol brittle and those with glycerol content ranging from 10% w/w to 20% w/w were not adequate flexible for effective use. The concentration of glycerol relies on applications, for example the minimum amount of glycerol for desired mechanical resistance and heat transparency is set at 30% w/w for sago starch films [7]. [11] discovered that glycerol ranges below 30% w/w may reduce the ductility of sago starch films. If the quantity of glycerol exceeded 50% w/w, the plasticity increased and its permeability. The higher the content of glycerol, the higher the ductility. However the lower the barrier and solubility effectiveness [12].

Other than that, essential oil such as kaffir lime is also added into formulation of bioplastic film. Citrus hystrix is the ordinary name of Kaffir lime. It is a tropical fruit that native to variety parts of Asia. The fruit, peel, leaves and oil of kaffir limes are all use for different purposes. According to [13], [14] peel and leaves of kaffir lime contains significantly higher amounts of essential oil compared to other plant parts. In addition, kaffir lime oil act as

antimicrobial agent [15]. Antimicrobial refers to substances that can kill or inhibit the growth of microorganisms. Antimicrobials was a substance produced by microorganisms that can prevent or reduce growth of bacteria in the food. A study reported the antibacterial activity of essential oil from kaffir lime peel against bacteria such as *S. aureus*, *S. epidermidis*, *P. aeruginosa* and *E. coli* by [16]. According to [17] the addition of an antimicrobial compound in packaging materials is required to enhance bioplastic film function. The combination of bioplastic film and antimicrobial compound may retard expansion of spoilage and pathogenic microbial that might prolong the time shelf life of food. Essential oils are plant extracts, which antimicrobial activity have been proven from previous research. Many researcher discovered that spoilage microorganisms, food borne and post-harvest pathogens are sensitive to essential oil [18]. A study by [19] had proven the antimicrobial activity of bioplastic films incorporated with essential oil against microorganism and molds.

Besides the formulation of bioplastic film, moisture content is an important property. The correlation between moisture content and water activity in the food at constant temperature and pressure is demonstrated as moisture sorption isotherms [20]. Sorption isotherm is necessary to predict stability and changes throughout packaging and storage for food products [21]. Moisture sorption is to estimate the equilibrium moisture content, which is vital to predict the properties of the films. Water activity is the ratio of the partial pressure of water over the wet solid system to the equilibrium vapor pressure of water at the same temperature. Moisture sorption isotherms are effective thermodynamic tools to determine the interaction between water and materials especially food products. The curve of the sorption isotherms might change depending on the kind of product and reflect the manner in which water binds to the system. Sorption isotherms also used to analyse the structural features of a food product such as surface area, pore size and crystallinity. Equilibrium moisture content the food material is described as moisture content when the vapour pressure in foodstuff has reached an equilibrium with its environment [20]. The curve of the sorption isotherm may undergo a change relying on the type of product and reflect the way in which water binds the system [22]. This isotherm curve can be acquired in one of two ways, which are adsorption or desorption. The adsorption and desorption processes are not entirely reversible, consequently a difference can be made between the isotherms through identifying whether the moisture levels inside the product are increasing or decreasing.

The moisture sorption isotherms of food material can be described by several models. Model of the moisture sorption isotherms can be separated into some categories which are kinetic models based on a monolayer (Brunauer, Emmett and Teller BET) model, kinetic models based on a multilayer and condensed film (Guggenheim-Anderson-de Boer GAB) model and empirical models (Smith and Oswin) [23]. These models are widely used for it multi-purpose, comparatively easy, mathematical. computations and acceptable food system. The models show the moisture content leads to the most stability of food throughout its storage. However, limited literature were found on moisture sorption of cassava starch- kaffir lime film. So, the aims of this work were to investigate the moisture sorption of cassava starch kaffir lime bioplastic film as affected by kaffir lime concentration and its modelling to moisture sorption model.

II. METHODOLOGY

A. Materials and methods

Cassava starch and was purchased from local market at Section 7, Shah Alam, Selangor. Kaffir lime oil were extract from its peel via

hydrodistillation method. Glycerol (Merck) will be used as a plasticizer.

B. Extraction of Kaffir Lime Oil

Essential oil will be extracted from kaffir lime peel by using a hydrodistillation technique. Fresh kaffir lime peel and distilled water will be added at the proportion of 1:5. The extraction will be done for 3 hours at temperature 150°C. The oil will be collected in a vial and will be kept at 4°C [24].

C. Preparation of Film

Cassava starch based films were prepared by casting technique. The film solution were prepared by mixing cassava starch (5% w/v), glycerol (35% w/w), and the remaining is distilled water [25]. The solution will be gradually heated on the hot plate with continuous stirring until the temperature reached to 70°C and maintained for 20 minutes. Different concentration (0, 0.5, 1, 1.5 and 2 % v/v) [26] of kaffir lime oil will be added and continuously by stirred at 300 rpm. The solution will be casted over petri dish and then dried in an oven at 40°C for 48 hours [4]. The dried film will be peeled off and stored in a plastic bag and keep in the desiccators for further analysis.

D. Measurement of Moisture Sorption Isotherms

The relationship between water activity (a_w) and moisture content at a given temperature is called the moisture sorption isotherm. Moisture sorption isotherms were measured through a standard gravimetric method which is saturated salt slurry method. Five saturated salt solutions which are (KOH, NaCl, BaCl₂, KCl, NaNO₃) were prepared corresponding to wide range of water activities ranging between 0.05 to 0.9 were obtained from table 1. The experimental apparatus consisted of five plastic beaker which inside the plastic beaker were consist of five bioplastic film that consist of different kaffir lime concentration and five beaker of salt solution. Every beaker of salt solution were quarter filled with saturated salt solution [27]. Bioplastic film samples for adsorption were weighed and placed on beaker containing saturated salt solution. The five samples of different salt solution were weighed every two days. The equilibrium moisture content of each sample was acknowledged when three consecutive weight measurements that showed difference less than 0.001 or until weighed is constant. The moisture content curves versus water activity were plotted to determine the moisture content of the cassava starch at constant temperature [28]. The moisture content were used to calculate the moisture content at various water activity.

Table 1: The water activities (a_w) of saturated salt solutions at 25°C [29], [30].

Saturated Salt Solution	25°C
Potassium Hydroxide (KOH)	0.083
Barium Chloride (BaCl ₂)	0.9
Sodium Nitrate (NaNa ₃)	0.623
Sodium Chloride (NaCl)	0.783
Potassium Chloride (KCl)	0.863

E. Model of Moisture Sorption Isotherms

Sorption isotherms of the edible film were modelled using kinetic models based on a monolayer (Brunauer, Emmett and Teller BET) model. The quality of the fit of the proposed models were assessed by the coefficient of determination (R^2) value. The BET models are expressed and rearranged as below.

$$M = \frac{M_0 C a_w}{(1 - a_w)(1 + (C - 1)a_w)} \quad (1)$$

M_0 = is the moisture of product corresponding to the monomolecular layer of the adsorbed water

a_w = water activity

C = characteristic constant of the material related to the heat release in this sorption process

M = moisture content at equilibrium (gram water per gram dry matter)

The linearized form of the equation can be expressed as below

$$\frac{a_w}{[(1-a_w)m]} = \frac{1}{(m_0 \cdot C)} + \left[\frac{(C-1)}{m_0 \cdot C} \right] a_w \quad (2)$$

By plotting moisture content vs a_w , the slope is $\left[\frac{(C-1)}{m_0 \cdot C} \right]$ and intercept is $\frac{1}{(m_0 \cdot C)}$

F. Statistical Analysis

The experimental sorption isotherms data were fitted using BET model as shown in equation (2). To evaluate the ability of BET model to fit the experiment data, the mean relative percent deviation (MRPD) [31] and the coefficient of correlation R^2 [32] between the experimental and the BET model (predicted data) were determined by using the following equation bellows:

$$\text{MRPD (\%)} = \frac{100}{N} \times \sum \left| \frac{y_{j\text{cal}} - y_{j\text{exp}}}{y_{j\text{exp}}} \right| \quad (3)$$

$$S_t = \sum (\bar{y} - y_{j\text{exp}})^2 \text{ being } \bar{y} = \frac{\sum y_j}{N} \quad (4)$$

$$\text{SCE} = \sum (y_{j\text{cal}} - y_{j\text{exp}})^2 \quad (5)$$

$$R^2 = \frac{S_t - \text{SCE}}{S_t} \quad (6)$$

Where $y_{j\text{cal}}$ and $y_{j\text{exp}}$ are respectively the calculated and the experimental values of y where is the moisture content. The models were compared according to their mean relative percent deviation (MRPD) and the correlation R^2 at the studied temperature.

III. RESULTS AND DISCUSSION

A. Moisture Sorption Isotherms

Moisture adsorption is a significant indicator of the sensitivity of material to moisture. The physical and barrier characteristics of characteristics of the starch based films that can be significantly affected by moisture content. The moisture content curves of cassava starch films at 25°C are represented at Figure 1. From figure 1 show that these graph represented a (Type III) according classification of [22]. According to the classification of [22], all the sorption isotherms obtained exhibit Type III behaviour, in which a small amount of water was adsorbed in low water activity and the amount absorbed in larger water activity. When the bulk humidity factor was achieved, rapid powders absorb a large amount of water vapor, causing it to be dissolved and leading to a steep rise in the third part of the curve, corresponding to the formation of hydrate. Foods that are rich in soluble components exhibit isotherms with Type III behavior due to the solubility of the components in water.

Linear form in the first part of isotherm is due to water adsorption in biopolymer, the sharp increase in water content in high water activity is due to gradually dissolution of solutes such as sugars and salt. According to [22], food rich in soluble components show isotherms with Type III behavior due to the solubility of the components in water. Comparison of the initial moisture content with the final equilibrium moisture content gives the sample the ability to adsorption or desorption moisture at different of water activities.

From figure 1 indicate that at constant temperature, the equilibrium moisture content increase with increasing the water activity. Two mechanisms are responsible for this sorption phenomenon, at low relative humidity values, water molecules are attached to the pore walls forming a thin water film, as relative humidity rises, this film becomes thicker and capillary

condensation starts taking place in the narrow pores, the two mechanisms overlap each other, but at high relative humidity, the capillary condensation becomes dominant. Equilibrium moisture content of bioplastic films increases with the rise of relative humidity the same temperature. That was due to the vapor pressure deficit (VPD) decreases with increasing relative humidity which creates an atmosphere close to saturation and that increases the ability of bioplastic films to absorb more moisture from the surrounding atmosphere.

Water activity has an effect on equilibrium moisture content film that bioplastic film from cassava starch. Equilibrium moisture content of bioplastic film increased slowly until water activity 0.63 and a minor increase in humidity caused a large increase in equilibrium moisture content of high water activity at constant temperature. This increase of equilibrium moisture content in cassava starch bioplastic films with increased water activity can be attributed to the fact that in low water activity, water can only be absorbed on the surface site.

[33] in their study said that, when water activity increases, the dissolving of dissolved components increases the moisture properties. Furthermore, increased in equilibrium moisture content with increased water activity can be attributed to film inability to maintain vapor pressure on unity by reducing moisture content. Other than that, the increase in water activity results in the increased availability of polar sites into water molecules [23], as shown in figure 1 for temperature 25°C. It also supported by [34], [35] at low water activity protein adsorbed more water from sugar and other soluble components while at higher water activity, sugar and soluble components absorb more water.

Pressure also has an effect on water activity of food system, but its effect is slightly than temperature effect. In the most cases, the pressure effect can be neglected unless high pressure applied. In this case, there is no pressure is applied in this experiment. Similar resulted had been obtained by [20], [32].

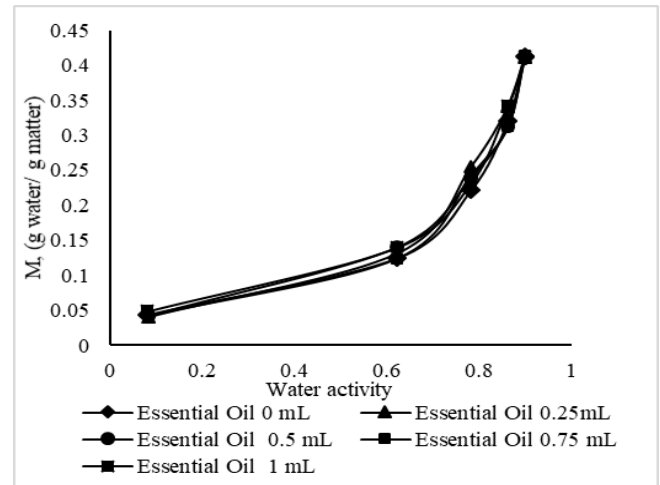


Figure 1 Equilibrium moisture sorption isotherms of cassava starch kaffir lime bioplastic film with 35 w/w of glycerol content, with different kaffir lime oil at 25°C.

B. Modelling of Sorption Isotherms

The constants of sorption isotherms models for cassava starch films with the same glycerol content at temperature of 25°C. The quality of the fit of the proposed models was assessed by the coefficient of determination (R^2), the mean relative percent deviation (MRPD) and estimated parameter of BET models in table 2. The results show that the highest probability of fitting the experimental data with the highest values for the R^2 and MRPE was obtained with the BET model for all cassava starch with different concentration of kaffir lime bioplastic films samples. It can be concluded that the higher the R^2 value, indicate that the better is the fit. It can be seen that the BET model gives the best fit for experimental data for the entire range of water activity investigated in this experiment at constant temperature which is at 25°C, the

highest R^2 values ranging between 0.9439 to 0.9850 and the value of MRPD values ranging between 9.1882 to 18.3558 were obtained from table 2. The fitted sorption isotherms for the BET model with experimental data are illustrated in Figure 2.

The most recognized models for calculation of monolayer moisture content, M_o of foods materials is BET. This may be caused by the fact that BET is more related to moisture sorption in the first layer that makes it more suitable dry materials. The monolayer value is proof of the quantity of water that can be bound to a single layer per gram of dry film. Basically, the monolayer of moisture content, M_o is represents the moisture content of the material, a_w is water activity, C is characteristic constant of the material related to the heat release in this sorption process, M is moisture content at equilibrium (gram water per gram dry matter). The parameters of the sorption models were estimated from experimental result by using a non-linear regression.

From the linearized BET equation, the graph moisture content has been plotted against a_w in figure 2 that results in a slope and a straight line intersection used to determine the monolayer value of M_o as shown in equation 2. The M_o value in this study was determined by using the BET equation and was obtained between range 0.0429-0.0469 gram water per gram dry matter following adsorption at 25°C from table 2. The higher monolayer moisture content of cassava starch kaffir lime could be associated to higher of equilibrium moisture content at adsorption. However, the Monolayer value of the BET model is in good accordance with the results found in the literature [23]. [36] in their study on water adsorption isotherms and thermodynamic analysis state that the monolayer moisture content, M_o , can be seen as moisture content that affects the longest period of time with minimal loss of quality at a certain temperature. By using the BET equation to predict the most safe water activity in this experiment, it is found that the average value is 0.65.

It can be shown that the BET model gives the best fit to experimental data for adsorption isotherms of cassava starch kaffir lime films for a wide range of water activity between 0.05 to 0.91. Besides that, the parameters of BET model can be recommended for the prediction of adsorption isotherms of cassava starch kaffir lime films. Similar finding were obtained by [37], [38].

Table 2: Estimated value of BET model fitted to the adsorption of cassava starch kaffir lime films for different kaffir lime concentration at 25°C.

Model Constant		Kaffir lime concentration				
		0 mL	0.25 mL	0.5 mL	0.75 mL	1 mL
BET	M_o	0.0442	0.0469	0.0429	0.0448	0.046
	C	-99.855	-113.77	22.709	-117.77	-30.405
	R^2	0.9850	0.9438	0.9620	0.9679	0.9562
	MRPD	9.1482	14.5929	13.6372	12.1614	18.3558

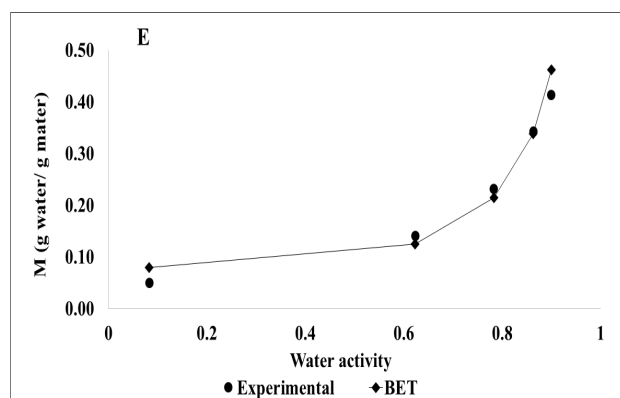
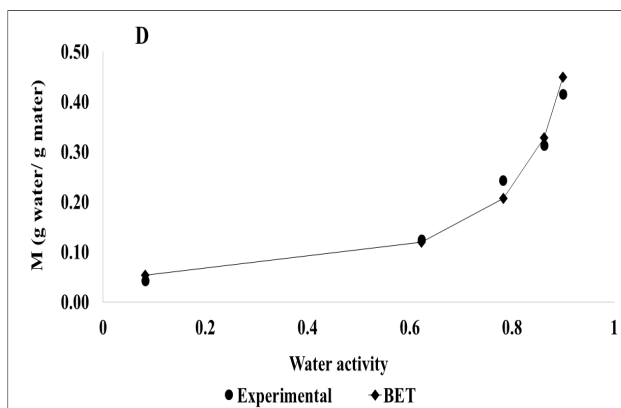
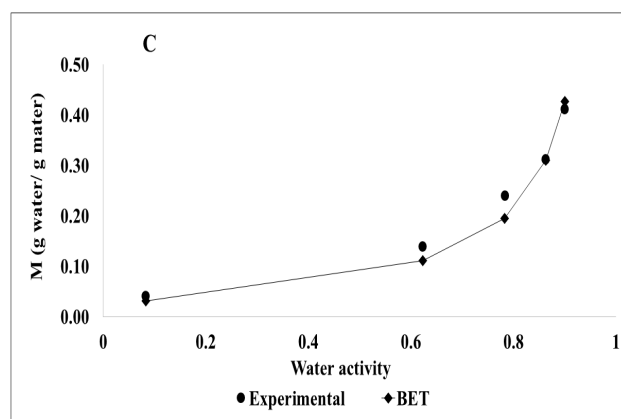
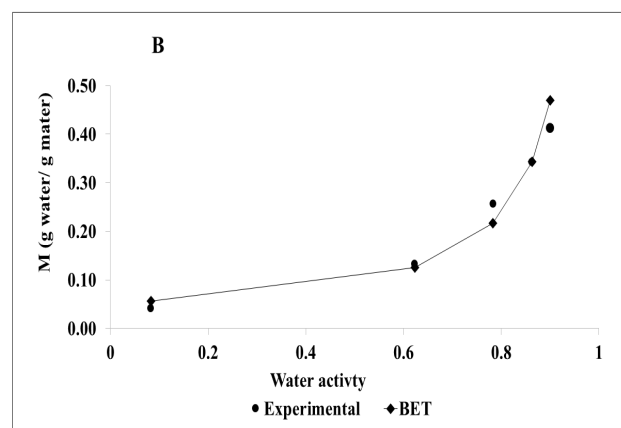
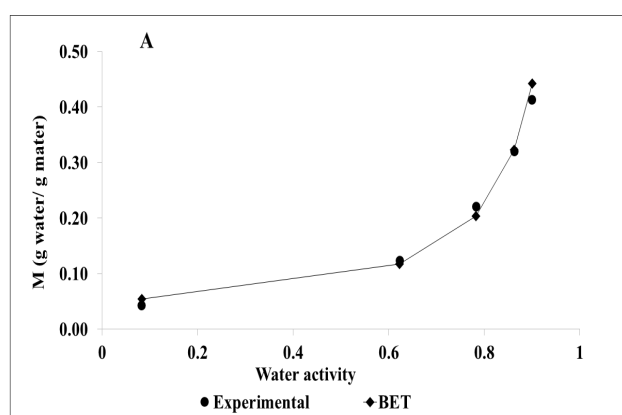


Figure 2: Graph comparison between experimental and predicted (BET model) sorption isotherms of cassava starch kaffir lime bioplastic films at 25°C with 35 w/w of glycerol content and concentration of kaffir lime at 0 mL (A), 0.25 mL (B), 0.5 mL (C), 0.75 mL (D), 1 mL (E).

C. Effect of different concentration kaffir lime on moisture content

The water activity of a product is an important indicator that affects the maximum shelf life of the product. It is indicated the bacteria do not grow under water activity 0.91 [39]. Changes of water activity is demonstrated the relationship between moisture content, and reduces of both water activity and the moisture content of kaffir lime that will inhibit the growth of microorganisms and also can extend the shelf life of the product. Water activities also play a significant role in enzyme activity and vitamins identification in foods that are also important factors affecting the color, taste and aroma of the products. In this experiment the water activity is used is below 0.9. Its indicate that the bioplastic film is suitable as packaging because the microorganism unable to grow below water activity of 0.91. There is a possibility of essential oils cannot eliminated or remove bacteria due to bioplastic films act as a barrier. So that the compound cannot fully permeate through the bioplastic films.

There is no significant change in moisture content an increase of concentration of kaffir lime for all water activity in this study.

IV. CONCLUSION

This study presents data on moisture sorption of Cassava starch kaffir lime bioplastic films by using standard saturated salt solution over a range of water activity from 0.05 to 0.9 at 25°C. The moisture sorption of cassava starch films increased with increase in water activity at constant temperature. The temperature had no significant effect on the equilibrium moisture content. It was found that the bioplastic film exhibited Type III sorption isotherms (J-shaped). Food rich in soluble components show isotherms with Type III behaviour due to the solubility of the components in water. At low water activity, there was very small amount of water was adsorbed onto the active sites and at high water activity, much more water was adsorbed that leading to a rapid increase in equilibrium moisture content. BET adsorption model was found to be the best model to represent the relationship between water activity and equilibrium moisture content because it fits to all films with different concentration of kaffir lime at constant temperature and range 0.05 to 0.9 of water activity. The monolayer, M_0 value in this study was determined by using the BET equation and was obtained between range 0.0429-0.0469 gram water per gram dry matter following adsorption at 25°C. The higher monolayer moisture content of cassava starch kaffir lime could be associated to higher of equilibrium moisture content at adsorption Temperature had significant effect on monolayer moisture content. The bioplastic film is suitable as packaging because the microorganism unable to grow below water activity of 0.91. Bioplastic film containing essential oils can be used to extend shell life and maintaining the food quality.

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