MOISTURE SORPTION BEHAVIOR OF JATROPHA SEEDS AT 20 °C AS A SOURCE OF VEGETABLE OIL FOR BIODIESEL PRODUCTION

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ABSTRACT

This research studied the moisture sorption behavior of jatropha seeds by developing a model that correlates the equilibrium moisture (EMC) and Free Fatty Acids (FFA) contents as a function of water activity and EMC, respectively. Two sets of sorption-isotherm experiments were performed to describe the relationship of EMC and FFA contents with water activity, both for fresh and dried seeds of Banten and Lampung varieties. The seeds are conditioned in a series of vessel with saturated salts at certain water activity. The EMC were increased following an increase in water activity in either desorption or adsorption. The hysteresis effect is more pronounced at Banten variety than Lampung one. The FFA content of fresh seeds was increased with an increase in water activity, while the FFA content of dried seeds is relatively constant. The moisture sorption behavior of jatropha seeds reveals that like most product, it exhibits the sigmoid pattern. The BET, GAB, Harkins-Jura, Halsey and Oswin models are recommended to be adequate in predicting the amount of moisture adsorbed or desorbed at known humidity. The relationship between EMC and FFA contents shows that the polynomial equation is the best for fresh seeds, and constant equation for dried seeds.

Keyword: moisture sorption; jatropha seeds; water activity; equilibrium moisture content;

INTRODUCTION

Jatropha curcas is a drought-resistant shrub or tree belonging to the family *Euphorbiaceae*, which is cultivated in Central and South America, South-East Asia, India and Africa (Gubitz *et al*., 1999). It is a plant with many attributes, multiple uses and considerable potential. The plant can be used to prevent and/or control erosion, to reclaim land, grown as a hedge to protect fields, and be planted as a commercial crop (Openshaw, 2000).

The seed is a part of jatropha plant with the highest potential for utilization. It contains 40-60% of oil and 20-30% of protein. The jatropha seed is generally toxic to human and animal. Phorbol ester and curcin have been identified as the main toxic agent responsible toxicity (Haas and Mittelbach, 2000).

The oil of *Jatropha curcas* is regarded as a potential diesel substitute. Among the advantages of vegetable oils as alternative fuel are that they are non toxic, safely stored and handled because of their high flash point. The fact that the jatropha oil cannot be used for nutritional purposes without detoxification makes its use as an energy source for fuel production very attractive.

Currently, most of biodiesel is produced using methanol and an alkaline catalyst. The difficulty with an alkaline-esterification of the oils is that they often contain large amount of free fatty acids (FFA). These FFA quickly react with the catalyst to produce soaps that are difficult to separate. This may reduce the sum of catalyst available for transesterification lowering the ester yield. It is then important to keep the FFA oil in a low level (< 3%).

Attractive approach to control the FFA in the seed level is by controlling water activity of the seed to a level that disables any undesirable reactions or enzyme activities. A fundamental approach for controlling the water activity of the seeds is by understanding of seed characteristics and its behavior in responding the changes in environmental conditions, particularly the relative humidity. This approach has been successfully applied to several oilseeds such as canola, macadamia nuts (Palipane and Driscoll, 1992), wheat (Larumbe *et al*., 1994), sunflower, soybean, amaranth, hazelnut (Lopez *et al*., 1995). Controlling the FFA in the seed level can thus be achieved by proper handling and storage of the seeds before oil extraction.

The main purpose of this research is to achieve a fundamental understanding of the hygroscopic properties of jatropha seeds. This understanding can then be utilized for developing a model correlating the equilibrium moisture (EMC) and FFA contents as a function of water activity and EMC, respectively. The output of this research includes models of sorption isotherm of jatropha seeds, and sorption isotherm properties of jatropha seeds and its correlation with FFA content.

MATERIALS AND METHODS

This research consists of four main works, i.e. jatropha fruit preparation, determination of sorption isotherm, determination of FFA and data analysis. These experiments were carried out in the laboratories of Department of Agroindustrial Technology and Indonesian Center for Agricultural Postharvest Research and Development.

Jatropha fruits used in this research are originated from two different locations, i.e.

Lampung and Banten, and are supplied by Balai Penelitian Tanaman Industri (Sukabumi-Indonesia). All solvent and chemicals are analytical grades that were obtained from Sigma-Aldrich, AppliChem and J.T. Baker, Indonesia.

The seeds are characterized for the chemical composition of the seeds. The sorption isotherm properties of the seeds from each location are determined for both fresh and dried seeds, and conducted at temperature of 20 °C. The moisture and FFA contents of the seeds are quantified at equilibrium conditions at given relative humidity to determine sorption behavior of the seeds and its correlation with FFA content.

A model is fitted to the data to provide an estimate of change of moisture content under different conditions of equilibrium relative humidity. Data of sorption isotherm is applied to eight well-known different isotherm equations (Table 1): Halsey, Henderson, Harkins-Jura, Iglesias-Chirife, Oswin, Smith, BET (Brunauer, Emmet and Teller) and GAB (Guggenheim, Anderson and de Boer) in order to determine the best model to predict the sorption behavior for the seeds. The constants for isotherm equations are calculated using linear regression and differently non-linear regression for GAB. The goodness of fit of the different models is evaluated with the mean relative deviation modulus (P) between the experimental (M_i) and predicted moisture content (Mp_i): $P = (100/n)\sum(M_i - Mp_i)/M_i$.

Table 1.Models considered in equilibrium isotherm modeling

Model	Equation
BET	$a_w/(1 - a_w).M = 1/Mo.C + a_w.(C - 1)/Mo.C$
GAB	$M = A.B.Mo.a_w/(1 - B.a_w).(1 - B.a_w + A.B.a_w)$
Halsey	$M = (-A/T.\ln(a_w))^{1/B}$
Harkin-Jura	$1/M^2 = (B/A) - (1/A)\log a_w$
Henderson	$M = -(\ln(1 - a_w)/A.T)^{1/B}$
Iglesias	$\ln(M + ((M^2 + M^{0.5})^{0.5}) = A + B.a_w$
Oswin	$M = A.(a_w/1 - a_w)^B$
Smith	$M = A - B.\ln(1 - a_w)$

A, B, C are parameters pertinent to each equation; Mo is monolayer moisture content; T is temperature absolute (K).

Jatropha fruit collection and preparation

The ripe fruits are peeled, and the seeds are removed and collected. The seeds from each location are separated into two groups; one is directly transferred into series of desiccators containing saturated salts for determination of sorption isotherm, and the other one goes into oven drying (50 °C, 48 hours) to reduce its moisture content (± 3-5%) then transferred into desiccators for the same treatment.

Characterization of jatropha seeds

Chemical compositions of the seeds are determined to provide the initial conditions of the seeds before conditioning for sorption isotherm determination. Moisture content of both fresh and dried seeds is determined using the constant-temperature-oven method. The seeds are dried at 105 °C for 15 h, after which they are cooled in a desiccator containing silica gels and reweigh to quantify moisture loss in weight on drying.

Oil content of the seeds is determined using solvent extraction method. Ground seeds are placed into an extraction thimble, and the oil is extracted using Soxhlet extraction apparatus with hexane for 6 h. The solvent is evaporated using rotary vacuum evaporator, and the oil is weighed. Seed oil content is expressed as percent by mass of the dry matter. The oil is then sent to the Gas Chromatography to determine the fatty acid compositions.

Initial FFA content of the seeds is determined using a method described in section d. Protein, ash and crude fiber are determined according to AACC approved methods.

Determination of sorption isotherm

An isopiestic method is employed for the determination of sorption isotherms by exposing the seeds to atmospheres of known relative humidity. A 200 g seeds is taken randomly from 5 kg of fresh seeds then divided into ten of 20 g for sorption isotherm determination. Each 20 g seed is placed into a desiccator containing saturated salts with certain relative humidity. Saturated salt solutions (equilibrium relative humidity at 20 °C shown in parentheses) comprise $ZnCl_2$ (6%), NaOH (10%), CH_3COOK (23%), $MgCl_2$ (32%), K_2CO_3 (44%), NaBr (57%), NaCl (75%), $(NH_4)_2SO_4$ (79%), KNO_3 (94%), K_2SO_4 (97%). Seeds are removed every day, reweighed and returned to the desiccators. When seeds reach constant weight (i.e. in equilibrium with the saturated salt solution (2-4 weeks)), their moisture content is determined using oven method. The same treatment is repeated for dried seeds. The experiments are conducted in two replications.

FFA determination

Two gram seeds is grounded, and placed into an Erlenmeyer flask containing 50 ml ethanol 96%. The acid value (FFA content) is determined by titration using KOH (0.1 N) with phenolphthalein as an indicator. The end point of titration is the appearance of permanent pink in the solvent mixture.

Results and discussion

The physicochemical characteristics of the two jatropha varieties used in these experiments are shown in Table 2. The moisture content of jatropha seeds is 39-46% for fresh seeds and 3.2-4.7% for dried seeds. The oil content of jatropha seeds are 33-43%, and they contain 1-3% Free Fatty Acids (FFA). Lampung variety is rich in oleic and linoleic fatty acids, while Banten variety is rich in palmitic and oleic fatty acids. The triglycerides of Banten variety are thus more stable than Lampung variety. Protein, ash and crude fiber of jatropha seeds are respectively 21-22%, 3.6-4.2% and 49.1-53.3%.

Table 3 shows the equilibrium moisture and FFA contents for Lampung variety of jatropha seeds, while Figure 1 shows typical adsorption and desorption isotherm. The equilibrium moisture content increases with an increase in water activity. The adsorption and desorption curves for jatropha seeds follow the characteristic sigmoidal shape common to many hygroscopic product, as observed for hazelnut (Lopez *et al*., 1995) and macadamia nuts (Palipane and Driscoll, 1992). In addition, the hysteresis phenomenon between adsorption and desorption is small. The FFA content of fresh seeds increases with an increase in water activity, while the FFA content of dried seeds is relatively constant (Figure 2).

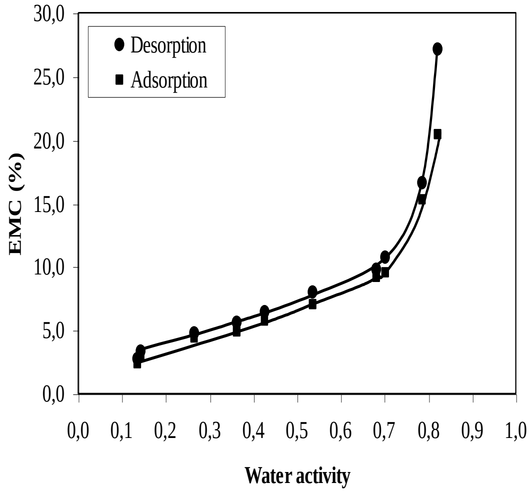

Figure 1. Adsorption-desorption isotherm for Lampung variety of jatropha seeds at 20 °C

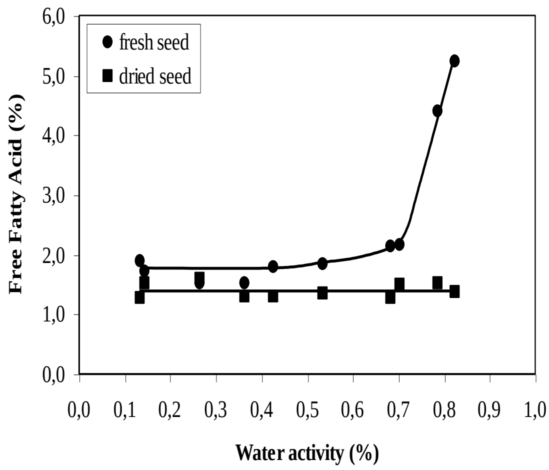

Figure 2. FFA content of fresh and dried jatropha seeds for Lampung variety after sorption isotherm at 20 °C

Table 4 shows the equilibrium moisture and FFA contents for Banten variety of jatropha seeds. Like for Lampung variety, the equilibrium moisture content increases with an increase in water activity (Figure 3). The hysteresis phenomenon between adsorption and desorption is relatively high. The FFA content of fresh seeds increases with an increase in water activity, while the FFA content of dried seeds is relatively constant (Figure 4). Compared with Lampung variety at same water activity, the equilibrium moisture contents for Banten variety are smaller both for desorption and adsorption. However, their FFA contents are higher than Lampung variety both for fresh and dried seeds. For sorption isotherm of jatropha seeds, the variety of jatropha seeds affects thus the equilibrium isotherm and FFA contents of seeds.

Table 2. Physicochemical characteristics of jatropha seeds and fatty acid composition of jatropha oils

Parameter	Variety			
	Lampung		Banten	
	Fresh seeds	Dried seeds	Fresh seeds	Dried seeds
Moisture (%, wb)	44.15 ± 1.79	4.13 ± 0.50	42.63 ± 1.87	3.60 ± 0.34
FFA (%)	2.02 ± 0.68	1.55 ± 0.38	2.27 ± 0.44	1.85 ± 0.52
Oil (%, db)	37.51 ± 2.73		35.73 ± 1.47	
Protein (%, db)	21.05		21.98	
Ash (%, db)	3.67		4.17	
Crude fiber (%, db)	49.13		53.31	
Fatty acid composition (%):				
1. Lauric	1.02		0.95	
2. Myristic	0.00		0.73	
3. Palmitic	7.01		16.66	
4. Stearic	1.49		0.00	
5. Oleic	46.84		80.39	
6. Linoleic	43.64		1.28	

Table 3. Equilibrium moisture content (EMC) and FFA for Lampung variety of jatropha seeds

Salt	a_w	Fresh Seeds		Dried Seeds	
		EMC (%)	FFA (%)	EMC (%)	FFA (%)
$ZnCl_2$	0.137	2.58	1.87	2.25	1.26
NaOH	0.146	3.25	1.69	2.83	1.49
CH_3COOK	0.268	4.72	1.50	4.34	1.57
$MgCl_2$	0.366	5.53	1.50	4.72	1.27
K_2CO_3	0.429	6.38	1.77	5.63	1.29
NaBr	0.537	7.86	1.81	6.89	1.33
NaCl	0.684	9.71	2.11	9.08	1.24
$(NH_4)_2SO_4$	0.705	10.68	2.13	9.49	1.46
KNO_3	0.789	16.54	4.38	15.16	1.49
K_2SO_4	0.825	27.01	5.21	20.30	1.34

Table 4. Equilibrium moisture content (EMC) and FFA for Banten variety of jatropha seeds

Salt	a_w	Fresh Seeds		Dried Seeds	
		EMC (%)	FFA (%)	EMC (%)	FFA (%)
$ZnCl_2$	0.137	2.10	2.40	2.52	1.87
NaOH	0.146	2.72	2.76	2.57	2.06
CH_3COOK	0.268	4.61	3.23	3.99	1.94
$MgCl_2$	0.366	5.74	2.58	4.54	1.99
K_2CO_3	0.429	6.41	2.57	5.03	2.21
NaBr	0.537	7.84	3.11	6.62	1.99
NaCl	0.684	9.87	2.81	8.51	1.95
$(NH_4)_2SO_4$	0.705	10.36	2.87	9.42	1.87
KNO_3	0.789	15.63	2.80	14.33	1.94
K_2SO_4	0.825	26.58	4.68	17.53	2.03

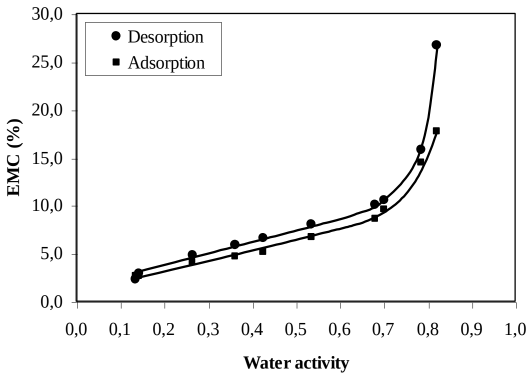

Figure 3. Adsorption-desorption isotherm for Banten variety of jatropha seeds at 20 °C

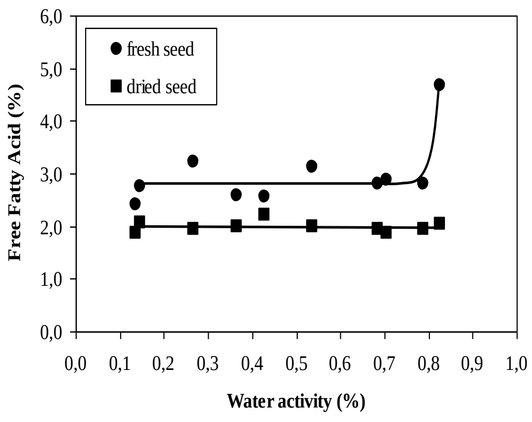

Figure 4. FFA content of fresh and dried jatropha seeds for Banten variety after sorption isotherm at 20 °C

Bell and Labuza (2000) describe that one aspect of moisture sorption leading to hysteresis is how water interacts within pores or capillaries. During moisture adsorption, water moves into the capillaries. However, these capillaries may empty differently upon desorption. Narrow ends of surface pores can trap and hold water internally below the water activity where the water should have been released. In addition, during adsorption the pure water will dissolve solutes present in the dry product. The dissolution of solutes may increase the surface tension such that upon desorption, a lower water activity results at given moisture content. Similarly, the wetting angle is lower for desorption as compared to adsorption, also resulting in a lower water activity at given moisture content for desorption.

Deteriorative changes in oilseeds may be either oxidative or hydrolytic, resulting in the production of free fatty acids (Christensen, 1974). Fats in seeds are readily broken down by lipases into free fatty acids and glycerol during storage, particularly when the temperature and moisture content are high. This type of change is greatly accelerated by mold growth because of high lipolytic activity of the molds. The kinds of molds are influenced by the initial moisture, temperature and oxygen concentration. *Aspergillus niger*, *A. candidus* and *A. versicolor* are the predominant species present.

Free fatty acids are carboxylic acids released from triglycerides through the effect of lipase or oxidation. The risk of oxidation is high in jatropha seeds due to its high unsaturated fatty acids content, especially oleic fatty acids (Table 2). Fatty acids containing one or more of the conjugated pentadiene systems, $-CH=CH-CH_2-CH=CH-$, are sensitive (Taub and Singh, 1998). Bell and Labuza (2000) describe that lipid oxidation begins to increase above a water activity of 0.3. At this water activity, the amount of water adsorbed on surface and in capillaries is enough to affect the overall dielectric properties such that the water can behave as a solvent. Thus, chemical species can dissolve, become increasingly mobile and react. The higher water activity, the faster the reaction rate because of the greater solubility and increased mobility.

The suitability of eight sorption models (Table 5 and 6) in describing the sorption data of jatropha seeds is examined. The sorption data fit quite well in the 0.13-0.7 water activity ranges for Oswin, Smith, Iglesias-Chirife and Henderson isotherm equations, and the 0.13- 0.8 water activity ranges for BET, GAB, Halsey and Harkins-Jura isotherm equations. However, the best fitting (smallest P and highest R^2) is obtained in polynomial equations. Both for Lampung and Banten varieties, the mean relative deviation modulus (P) of all tested sorption models for adsorption are smaller than for desorption. The adsorption data give thus more suitable fit for sorption models than desorption data.

On the basis of P, the Halsey, BET and GAB models are the most efficient and versatile models for adsorption of Lampung and Banten varieties. For desorption of Lampung variety, the BET, GAB and Harkin-Jura models are the most appropriate, while for Banten variety the GAB and Henderson are the most suitable fitting.

The BET, GAB, Harkins-Jura and Halsey equations correspond to multilayer adsorption models. In this research, the best fitting obtained with BET and GAB equations, and Halsey and Harkins-Jura equations indicate that water adsorption is likely to be a multilayer adsorption in Jatropha-water system. Thus, the surface force distribution may be considered to act by causing the adsorbed film to behave as liquid in a two-dimensional state in this adsorption system. Moreover, the sorption data obtained fit the Harkins-Jura model well which indicates that the isotherm has the characteristics of a type II isotherm (sigmoidal-shaped curve).

An important parameter that is usually obtained from sorption data is the monolayer moisture content (Mo). In the BET and GAB isotherm equations, the monolayer moisture content is regarded as the sorption capacity of the adsorbent and as the indicator of usefulness of polar sites for water vapor. In addition, it defines the safest moisture content for storage. Below its monolayer value, for most dry product, the rate of quality loss due to chemical reaction is negligible. In this research, for Lampung variety, the monolayer moisture content is 3.6-4.5% (db), while it is 3.3-4.8% (db) for Banten variety. Both for BET and GAB, the monolayer moisture content for desorption isotherm is higher than for adsorption isotherm, as observed by many researchers (Palipane and Driscoll, 1992; Ajisegiri *et al*., 1994; Sopade *et al*., 1996; Sopade, 2001).

Table 5. Estimated parameters of tested sorption models, mean relative deviation modulus (P) and regression coefficient (R^2) for Lampung variety

Model	Desorption						Adsorption					
	A	**B**	**C**	**Mo**	**P**	**R^2**	**A**	**B**	**C**	**Mo**	**P**	**R^2**
BET	-	-	7.27	4.48	11.57	0.77	-	-	8.79	3.78	8.77	0.90
GAB	12.42	1.02	-	3.98	11.21	0.76	10.90	1.00	-	3.64	8.77	0.81
Halsey	1884.41	1.08	-	-	13.39	0.95	1749.55	1.12	-	-	9.73	0.97
Harkin-Jura	12.59	-0.073	-	-	10.89	0.99	10.06	-0.068	-	-	10.60	0.99
Henderson	0.000078	1.64	-	-	13.39	0.99	0.00010	1.61	-	-	13.38	0.99
Iglesias-Chirife	1.71	2.08	-	-	14.55	0.99	1.57	2.12	-	-	13.32	0.99
Oswin	7.77	0.47	-	-	12.18	1.00	6.87	0.48	-	-	11.05	0.99
Smith	2.41	7.63	-	-	14.15	0.99	2.01	6.94	-	-	12.60	0.99
Polynomial :	**A**	**B**	**C**	**D**	**P**	**R^2**	**A**	**B**	**C**	**D**	**P**	**R^2**
[a]$1/M = f(a_w)$	1.66	2.67	1.62	0.48	4.89	1.00	1.77	2.90	1.81	0.55	5.63	0.99
[b]$M = f(\ln(-\ln a_w)$	4.04	1.08	2.45	6.23	8.23	0.99	3.61	0.81	2.17	5.44	5.57	0.99

[a]$1/M = D - C.a_w + B.a_w^2 - A.a_w^3$

[b]$M = D - C.\ln(-\ln aw)) - B.(\ln(-\ln aw))^2 - A.(\ln(-\ln aw))^3$

A, B, C, D are parameters pertinent to each equation; Mo is monolayer moisture content

Table 6. Estimated parameters of tested sorption models, mean relative deviation modulus (P) and regression coefficient (R^2) for Banten variety

Model	Desorption						Adsorption					
	A	**B**	**C**	**Mo**	**P**	**R^2**	**A**	**B**	**C**	**Mo**	**P**	**R^2**
BET	-	-	6.06	4.45	13.72	0.75	-	-	11.28	3.45	5.05	0.96
GAB	5.66	0.97	-	4.74	12.79	0.62	14.19	1.00	-	3.32	5.50	0.92
Halsey	1595.14	1.02	-	-	16.22	0.95	1807.93	1.18	-	-	5.90	0.99
Harkin-Jura	12.48	-0.074	-	-	17.81	0.98	8.57	-0.071	-	-	9.45	0.99
Henderson	0.00011	1.48	-	-	12.80	1.00	0.00012	1.57	-	-	11.21	0.99
Iglesias-Chirife	1.39	2.69	-	-	15.87	0.96	1.37	2.48	-	-	9.09	0.98
Oswin	7.67	0.52	-	-	14.40	0.99	6.47	0.50	-	-	8.33	0.99
Smith	2.22	7.78	-	-	17.94	0.99	1.67	6.91	-	-	9.14	0.99
Polynomial	**A**	**B**	**C**	**D**	**P**	**R^2**	**A**	**B**	**C**	**D**	**P**	**R^2**
[a]$1/M = f(a_w)$	2.71	4.50	2.62	0.65	5.31	1.00	1.83	3.08	1.98	0.60	4.37	0.99
[b]$M = f(a_w)$	3.61	1.57	3.14	6.28	7.48	0.99	2.88	0.12	2.26	5.00	3.28	1.00

[a]$1/M = D - C.a_w + B.a_w^2 - A.a_w^3$
[b]$M = D - C.\ln(-\ln aw)) - B.(\ln(-\ln aw))^2 - A.(\ln(-\ln aw))^3$
A, B, C, D are parameters pertinent to each equation; Mo is monolayer moisture content

Table 7. Mean relative deviation modulus (P) for relation between EMC and FFA of fresh and dried jatropha seeds

Model	Mean Relative Deviation Modulus (P)			
	Lampung Variety		BantenVariety	
	fresh seeds	dried seeds	fresh seeds	dried seeds
[a]FFA = constant	21.40	7.20	11.11	3.62
[b]Linier	15.90	7.41	9.87	3.46
[c]Polynomial	3.73	9.97	5.85	4.70

[a]$y = c$; [b]$y = a.x + b$; [c]$y = a.x^3 + b.x^2 + c.x + d$, where y and x are FFA and EMC, while a, b, c and d are constant parameters for each equation.

CONCLUSION AND RECOMMENDATION

Conclusion

The importance of sorption isotherm in drying and storage of product demands that sorption information adequately describes the product. A study of moisture sorption behavior of jatropha seeds shows that like most it exhibits the sigmoid pattern. Although empirical, the BET, GAB, Harkins-Jura, Halsey and Oswin models are recommended as adequate in predicting the amount of moisture adsorbed or desorbed at known humidity, and consequently estimating the likely changes in moisture content at varying humidity during handling. This will be valuable in planning strategies to minimize post-harvest losses of the jatropha seeds, as source of vegetable oil for biodiesel production. The relationship between EMC and FFA content of jatropha seeds showed that the polynomial equation is the best fitting for fresh seeds, and constant equation for dried seeds.

Recommendation

To develop this research, the study of jatropha seeds storage at different packaging products is critically required. In addition, the effect of storage duration on jatropha seeds quality should been studied.

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