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Moisture adsorption isotherms properties of dried overripe barlin fruit (*Musa acuminata AA.*) powder

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Abstract

Moisture sorption of dried overripe barlin banana powder using the static gravimetric method. The materials were conditioned at 5, 10, and 25 °C with a water activity range of 0.16 to 0.86. Five mathematical models to describe the suitability of the phenomenon of adsorption experimental data using non-linear regression. The thermodynamic properties such as isosteric heat and Gibbs free energy are evaluated in this paper. The experimental results indicated that the equilibrium moisture content of dried overripe barlin banana powder increased with increasing water activity and decreasing temperature. The results of the model are based on the goodness of fit, and the Peleg model is the most suitable for describing the adsorption phenomenon with the type II sigmoidal shape. Monolayer moisture content and adsorption surface area using the GAB model are in the range of 6.18-7.81%d.b and 219.09-246.39 m^2/g d.b, respectively, which increases with increasing temperature. Moreover, isosteric heat decreases with an increase in equilibrium moisture content and is opposite to the value of Gibbs free energy. In addition, the hygroscopicity of the dried overripe barlin banana powder increased with temperature and water activity.

Keywords: Barlin banana; Hygroscopicity; Moisture adsorption isotherms; Thermodynamic properties

1. Introduction

Barlin bananas (*Musa acuminata AA.*) is a type of bananas with a smaller size than other types of bananas. This type of banana is only intended as a guest dish and cannot be processed into product diversification due to the extremely high water content (85-90%w.b). There must be more information regarding this type of banana regarding the amount of production, land, content, and physical properties. Bananas generally contain high fibre and vitamins, driving them a functional food [1].

Moisture sorption isotherms are fundamental reference data in the sorption mechanism, optimizing the drying process, determining the critical moisture content point, and selecting the suitable packaging material. In the development of this study, many theoretical, semi-theoretical, and empirical equations have been published to describe the absorption characteristics of foodstuffs [2]. The phenomenon of moisture sorption isotherm is also significant as a variable for calculating thermodynamic properties such as isosteric heat, net isosteric, entropy, spreading pressure, hygroscopicity, adsorption surface area, and Gibbs free energy. This development analysis provides structural information, energy requirements, and the types of water molecules in the product [2].

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Many agricultural products have been reported related to moisture sorption isotherm studies. Commodities that have been studied include butterfly-pea flower powder [3], dried butterfly-pea flowers [4], safflower petals [5], camellia oleifera [6], orange leaves [7], rosemary leaves [8], bay laurel leaves [9], mint leaves [10], longan [11], tomato pulp [12], jambolan [13], sorghum [14], lemon peel [15], potato [16], and modified cassava [17]. However, information on the sorption isotherms of barlin bananas is limited. Taking into account the fact that there are differences in moisture absorption and water absorption of materials because there are differences in the nature and source of water, there has been no study related to the thermodynamic properties of moisture sorption isotherms of barlin bananas. The objectives of the present works are: 1) To investigate and model moisture adsorption isotherms in barlin bananas at a temperature difference of 5, 10 and 25^{o}C; 2) Determining thermodynamic properties such as isosteric heat, surface area, and Gibbs free energy; 3) Identify adsorption surface area and hygroscopicity due to differences in water activity.

2. Material and methods

2.1. Material and sample preparation

Fresh barlin bananas are obtained from farmers in Malang Regency, Indonesia, in an unripe condition. The maximum time for distribution from the field to the laboratory is 4 hours after harvest. Harvesting is done at 08.00 (local time). Unripe bananas are then dried for 1 hour in the sun to reduce bananas sap which can inhibit ripening. Then the bananas are ripened in a sack and tightly closed for up to 5 days, or the bananas are yellow with black spots on the surface of the bananas is called overripe (Figure 1).

Figure 1 Overripe barlin used as the sorption sample in this study

Overripe bananas are peeled and sliced vertically in half. Then in the convection oven (hot air dryer) (Memmert UF110, Germany) at 60^{o}C for 48 hours. Size reduction using a blender with a size of 500 μm. The dry sample is put into an airtight container and placed in a desiccator until the dried bananas powder is analysed. The chemicals used were KOH, $MgCl_2$, $CaCl_2$, NaCl, and KCl of pro-analytical quality (Merck, Germany). The chemical is dissolved in distilled water until it is saturated. A saturated solution was made from 60 g of chemical and added a little distilled water on a magnetic stirrer (speed of 100 rpm). Measuring water activity using the saturated solution requires a digital thermometer-hygrometer (Mainland, China) at temperatures of 5, 10 and 25^{o}C in an airtight container for 4 hours.

Table 1 A_w (water activity) of saturated solutions at different temperature

Saturated Solutions	**a_w**		
	5^{o}C	**10^{o}C**	**25^{o}C**
KOH (potassium hydroxide)	0.16	0.17	0.18
$MgCl_2$ (magnesium chloride)	0.33	0.34	0.35
$CaCl_2$ (calcium chloride)	0.61	0.62	0.64
NaCl (natrium chloride)	0.74	0.76	0.77
KCl (potassium chloride)	0.83	0.84	0.86

2.2. Adsorption procedure

The sorption test was determined at a conditioning temperature of 5°C, 10°C and 25°C using the static gravimetric method [2], [3]. This method uses five saturated solutions and can condition a_w treatments, as shown in Table 1. Samples weighing 4-5 g are placed in airtight containers. The sample container is placed in a controlled room with an uncertainty of ±2°C. Sample mass was measured daily until it was balanced after three days of controlled incubation. Mass was weighed using a high-precision balance (Fujitsu FRS-A-300, Japan). Samples in equilibrium were measured for water content at 105°C (4 hours) to determine the equilibrium moisture content (EMC).

2.3. Mathematical modelling

The EMC data from the adsorption phenomenon of dried bananas at three temperatures was modelled using the five mathematical models shown in Table 2. The model equation shows the relationship between EMC and water activity [2]. Constants and coefficients, i.e., a, b, C, n, and M_o. M_o is the predicted monolayer moisture content (d.b) from the BET and GAB models based on experimental data.

Table 2 The mathematical equation selection for fitting [4]

Model Type	Equations
Peleg	$EMC = a{a_w}^b + C{a_w}^n$
Oswin	$EMC = C\left(\frac{a_w}{1-a_w}\right)^n$
Halsey	$EMC = \left(-\frac{C}{ln\, a_w}\right)^{\frac{1}{n}}$
BET	$EMC = \frac{M_o C a_w}{(1-a_w)(1+(C-1)a_w}$
GAB	$EMC = \frac{M_o C K a_w}{(1-Ka_w)(1-Ka_w+CKa_w)}$

2.4. Adsorption surface area

The adsorption surface area is crucial to determine because it is related to the nature of water bonding [2]. This parameter is determined using the monolayer water content approach using the GAB and BET model approaches with the following equation [2], [18].

$$S_A = \frac{M_o N_A A_{H2O}}{M_{H2O}} = 3545.11\, M_o$$

Where SA is the adsorption surface area (m^2/g d.b), Mo is the monolayer moisture content of model (d.b), NA is Avogadro's number ($6.02x10^{23}$ molecules/mol), A_{H2O} is the area of the water molecule ($1.06x10^{-19}$ m^2), and M_{H2O} is the molecular weight of water (18 g/mol).

2.5. Sorption isosteric heat

Isosteric heat (Q_{st}, J/mol) is the sum of the net isosteric (q_{st}, J/mol) with the heat of vaporization of water (H_L). The value of H_L is 43 kJ/mol from the temperature system [2]. Isosteric heat is expressed in the following equation.

$$Q_{st} = q_{st} + H_L$$

The net isosteric was determined by plotting the ln(a_w) versus 1/T curve at the constant moisture content [4], [19].

$$ln_{aw} = -\left(\frac{qst}{R}\right)\left(\frac{1}{T}\right) + C$$

Where, R is the gas constant (8.314 J/mol.K), T is the absolute temperature (K), and C is the constant.

2.6. Gibbs free energy

The change in Gibbs free energy (ΔG) shows the affinity value of the water adsorbent and the spontaneous adsorption process. The best model approach includes temperature and predicted water activity as variables for calculating Gibbs free energy as in the following equation [2], [19].

$$\Delta G = RT\, ln(a_w)$$

2.7. Hygroscopicity

Hygroscopicity is the final level after the equilibrium moisture content [20]. The hygroscopicity value determines the adsorption data for each water activity and temperature level. Hygroscopicity is determined using the following equation [21].

$$H_i = \frac{M_i + (\Delta m - mt)}{1 + (\Delta m - mt)}$$

Where H_i is hygroscopicity (%), Δm is mass increase after the experiment (g), mt is the initial mass (g), and M_i is free moisture content (d.b).

2.8. Statistical Analysis

The EMC and hygroscopicity data were tested statistically using two-way ANOVA with a 95% confidence level. The test was continued with LSD (Least Significance Different) with a 95% confidence level. All data was repeated three times (n=3). Tests for accuracy in evaluating model results are the coefficient of correlation (R^2), the average relative deviation (P), and the root of mean square error (RMSE) [2]. All calculations were performed using Microsoft Excel 2016 software (Microsoft, USA).

3. Results and discussion

3.1. Adsorption isotherm

The adsorption isotherm of dried overripe barlin can be seen in Figure 1. The EMC increases slowly at low a_w conditions. At high a_w (more than 0.7), EMC increases rapidly. Table 3 provides the EMC experimental data during adsorption at temperature and humidity levels. Decrease in temperature results in a reduction of the adsorption EMC. Then, the increase in humidity affects EMC's rise during adsorption. Based on the LSD statistical test with a confidence level of 95%, it shows that temperature and humidity have a significant effect on the equilibrium moisture content.

Table 3 EMC of adsorption during sorption of dried overripe barlin bananas

Saturated Solutions	**EMC, db**		
	5°C	**10°C**	**25°C**
Potassium hydroxide	7.34±0.61^{aA}	8.59±0.92^{aB}	9.84±0.80^{aC}
Magnesium klorida	8.70±0.41^{bA}	9.97±0.41^{bB}	11.24±0.54^{bC}
Calcium chloride	9.39±0.46^{cA}	12.74±1.14^{cB}	15.64±1.03^{cC}
Natrium klorida	10.88±1.87^{dA}	15.63±1.18^{dB}	23.58±1.82^{dC}
Potassium chloride	14.76±1.83^{eA}	20.67±1.41^{eB}	28.63±1.50^{eC}

* Columns with lowercase denoted significant (p<0.05) differences between saturated solutions. Columns with uppercase denoted significant (p<0.05) differences between temperatures. Data represents the mean (n=3) with LSD's test.

Based on the BET classification [22], the isothermal form of sorption from dry overripe bananas is type II or sigmoid in shape. This type of curve has also been reported for several food products such as butterfly pea flowers [4], cassava [18], chestnuts [23], and tiger nuts [2]. The sorption characteristics under conditions of high water activity allow for an increase in the melting of sugar in water due to the influence of temperature. This event is an endothermic process [24].

3.2. Fitting of sorption model

Many models reported so far can be used to predict the sorption phenomenon of dried overripe barlin bananas. Five standard models are used and give good results for various materials. Start equations from experimental data with non-linear regression. The modelling results are presented in Table 4. The model results are accepted when the R^2 fit test value exceeds 0.99. In addition, the P and RMSE values are less than 0.1. Based on the acceptance standards set, the Peleg model provides the best modelling results in describing the adsorption isotherms of dried overripe barlin bananas.

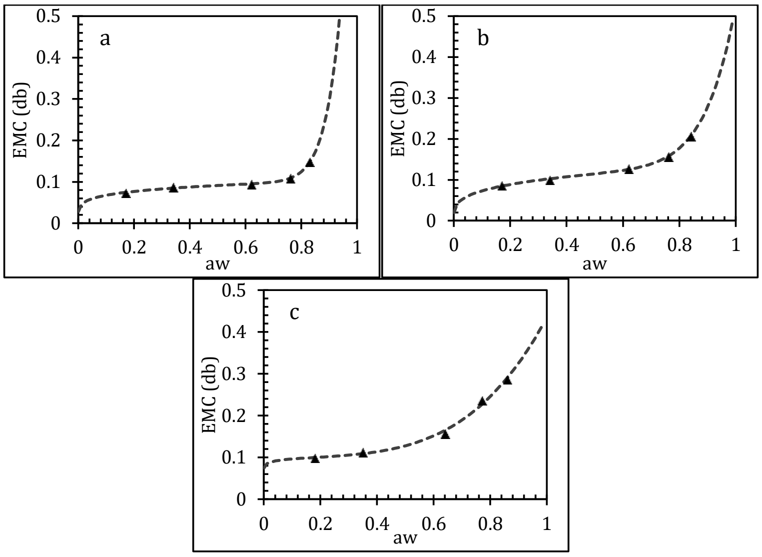

Figure 2 Adsorption isotherms of dried overripe barlin bananas and fitting with Peleg model, (▲) experiment data, (┅┅) prediction data (a) 5°C, (b) 10°C, and (c) 25°C

Table 4 Parameters of the moisture adsorption isotherms models

Models	Temperature	C	M_o	k	n	a	b	R^2	P	RMSE
GAB	5°C	4.96x10^6	0.0618	0.6507	-	-	-	0.9173	0.0761	0.0093
	10°C	3.48x10^9	0.0695	0.7718	-	-	-	0.9671	0.0483	0.0073
	25°C	1.97x10^8	0.0781	0.8479	-	-	-	0.9860	0.0368	0.0078
BET	5°C	1.14x10^7	0.0286	-	-	-	-	0.9739	0.2442	0.0268
	10°C	1.14x10^7	0.0378	-	-	-	-	0.9949	0.2109	0.0391
	25°C	1.14x10^7	0.0466	-	-	-	-	0.9615	0.2101	0.0351
Halsey	5°C	0.0023	-	-	0.1692	-	-	0.9732	0.3208	0.0359
	10°C	0.0027	-	-	0.1501	-	-	0.9916	0.3060	0.0415
	25°C	0.0030	-	-	0.1361	-	-	0.9640	0.3143	0.0498
Oswin	5°C	0.0950	-	-	0.2108	-	-	0.9113	0.0764	0.0096
	10°C	0.1199	-	-	0.2895	-	-	0.9461	0.0573	0.0092
	25°C	0.1446	-	-	0.3706	-	-	0.9673	0.0633	0.0121
Peleg	5°C	1.2186	-	-	17.413	0.1036	0.1861	0.9988	0.0091	0.0011
	10°C	0.3871	-	-	9.4376	0.1384	0.2780	0.9984	0.0112	0.0016
	25°C	0.3249	-	-	3.8561	0.1093	0.0560	0.9984	0.0255	0.0056

The Peleg model can be visualized in Figure 2. The Peleg model is suitable for most agricultural products, such as olive leaves [25], loquat and quince fruits [19], green tea [26], Camellia oleifera [6], safflower seed [27], orange peel and leaves [28], Cucumis melo L. seeds [29], butterfly-pea flower [4], and tiger nuts [2].

3.3. Monolayer moisture content

The monolayer water content (M_o) is the amount of water that is very tightly bound. This type of water is at the most profound specific sites in the material. Monolayer moisture content is a consideration in determining the stability and length of storage time. When below the moisture content of each level, the absorbed water cannot be used as a solvent, or other reactions (including lipid oxidation and enzyme activity) can be minimized [2].

The water content of the monolayer can be predicted through the GAB and BET equations. However, manually identifying the water content of the monolayer is done by looking at the first curved line of the absorption curve. This method is more convenient for all equation models regardless of the specific model. The amount of monolayer water content in dried overripe barlin bananas can be seen in Table 4. The predicted results of the GAB model are 6.18% d.b (5^{o}C), 6.95%d.b (10^{o}C), and 7.81%d.b (25^{o}C). Then the BET model identifies monolayer water content, namely 2.86%d.b (5^{o}C), 3.78%d.b (10^{o}C), and 4.66%d.b (25^{o}C). The water content predicted by BET and GAB is similar to pineapple [30], cassava[18], banana [31], and tiger nuts [2].

The results of the prediction model show that the higher the temperature, the water content of the monolayer increases [3], [17], [28], [32]. This phenomenon contradicts the results of several published reports [2], [17], [23], [33], [34]. This case indicates it is not more hydrophilic at low temperatures [2]. This increase indicates that the high temperature absorbs more water so that the value of the monolayer water content is directly proportional. The amount of energy required is also smaller at high temperatures. At low temperatures (5-10^{o}C, cold conditions), the monolayer's water content is lower, indicating more energy is required.

3.4. Sorption isosteric heat

The interaction between water content and food ingredients can be known, one of which is the thermodynamic parameters, such as isosteric sorption heat. Isosteric heat well describes and analyzes the drying process and the stability of the material under storage conditions. Isosteric determination using the Clausius-Clapeyron equation approach and this equation has been widely used in various types of previous research [4], [24], [33], [35], [36]. The isosteric heat curve for the adsorption of dried overripe barlin bananas is presented in Figure 3. The curve illustrates the effect of equilibrium moisture content on isosteric heat.

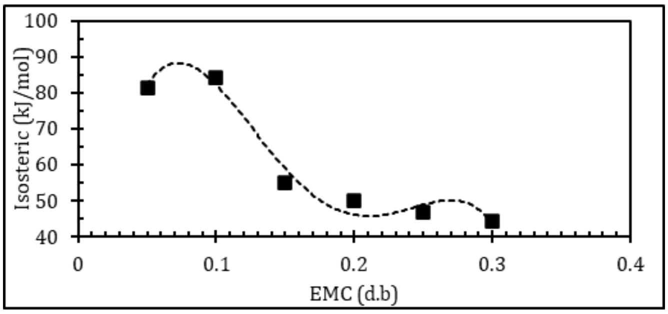

Figure 3 Isosteric heat of adsorption

The isosteric value is positive in the a_w range of 0.05-0.30 d.b, indicating that the adsorption process is experiencing an endothermic reaction [2]. A low isosteric value indicates a high water content, and vice versa. This phenomenon also occurs in Chestnut Flour [23], orange juice powder [37], and chironji [38]. At a water content of 10%d.b, the required isosteric heat is 84.21 kJ/mol. This condition justifies that under low water content conditions, many active polar sites on the material's surface bind to water molecules. However, the state has the form of a single molecular layer. Thus, the energy required to break these strong bonds is linearly correlated with an increase in isosteric heat.

As the water content increases to 15%d.b, the isosteric heat decreases drastically to 11.50 kJ/mol. After that, it tends to be constant up to a very high water content (>15%d.b). Several references show that the isosteric heat of sorption is higher than the evaporation energy of pure water [2], [24], [37], [38]. This phenomenon indicates that the bond between water molecules and sorption sites is higher than in the liquid phase [2]. The relationship between isosteric heat of adsorption and equilibrium moisture content of dried overripe barlin bananas is expressed by a polynomial equation (order 4) in the following equation. This model can predict well in isosteric heat loss with an appropriate test level of R^2 0.9743.

$$Q_{st} = -190235\ EMC^4 + 140227\ EMC^3 - 34877\ EMC^2 + 3139.7\ EMC - 4.2104$$

3.5. Gibbs free energy

Gibbs free energy interprets the maximum amount of energy released under constant pressure and temperature conditions. This parameter determines the predictive affinity between the solid matrix and spontaneous water during adsorption. Spontaneously is characterized by a negative Gibbs free energy value [39]. This condition indicates that no energy is required for the adsorption process from the environment when the reaction occurs. The value of Gibbs free energy for water content at each temperature level is presented in Figure 4. Identifying Gibbs free energy data increases with increasing water content and is stable at 10%d.b water content. The value of Gibbs free energy ranges from -17.12 to 0.0023 kJ/mol. At water content below 10%d.b, the value of Gibbs free energy increases rapidly [2]. This phenomenon is due to the weakening affinity between water and materials [40], [41]. It should be noted that the Gibbs free energy increases with decreasing temperature. The Gibbs free energy depends on temperature. However, this dependence is low because the increase in water content has no relationship to temperature, and it can be seen that the overlapping curves tend to be the same.

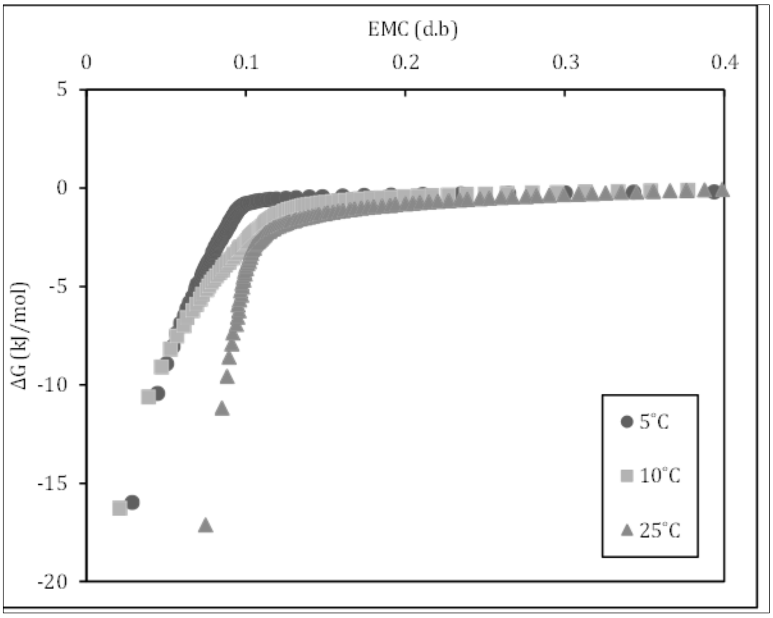

Figure 4 The relationship between the $\Delta \boldsymbol{G}$ and EMC

3.6. Adsorption surface area

Adsorption surface area is an indicator in determining the bonding characteristics of food ingredients. These parameters for dried overripe barlin bananas were determined by correlating with the monolayer moisture content of the GAB and BET models (Figure 5). Surface area values of dried overripe barlin bananas at temperatures of 5, 10, 25°C were 101.39, 134.01, 165.20 m^2/g d.b (BET) and 219.09, 246.39, 276.87 m^2/g d.b (GAB). Identical to the results of a previous study by Zhang et al. [2], which has a specific surface area in the range of 100-250 m^2/g d.b. Figure 5 shows a positive linear relationship between temperature and adsorption surface area with a coefficient of determination of 0.9392 (GAB) and 0.9161 (BET). This case indicates that the hydrophilic bonds increase in the surface area of the material as the temperature increases. This behaviour describes an increase in physicochemical-related active sites affected by temperature [2], [42]. In addition, it is also associated with surface interactions, structures, and chemical constituents related to moisture sorption capacity [2], [43]. However, the increase in temperature induces the water molecules adsorbed at the polar sites to condense into the material through the capillary tracks. This possibility causes swelling of the material.

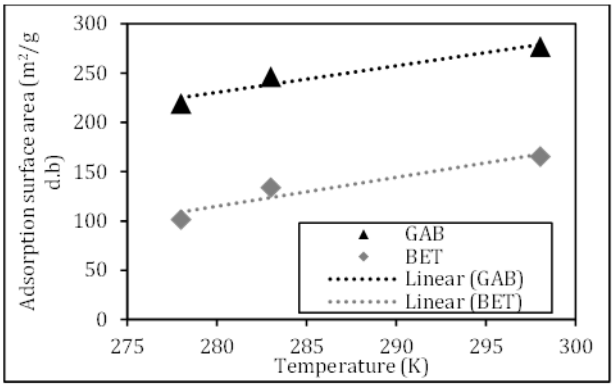

Figure 5 The relationship between the adsorption surface area and temperature

3.7. Hygroscopicity

Differences in the hygroscopicity values of dried overripe barlin bananas powder during sorption with variations in different humidity conditions can be seen in Table 5. The greater humidity and temperature conditions cause an increase in hygroscopic properties. Based on the LSD statistical test with a 95% confidence level, temperature and humidity significantly affect hygroscopicity. Oliveira et al. [32]. They reported that hygroscopicity was only found in the phenomenon of moisture adsorption. They also revealed that hygroscopicity could indicate unwanted caking and agglomeration on the material's surface. However, hygroscopicity is also affected by the presence of admixtures and chemical constituents that cause changes [20]. Differences in hygroscopicity values also inform the strength of the attractive capacity of water molecules [44].

Table 5 Hygroscopicity with variations in humidity during sorption

Saturated Solutions	**Hygroscopicity (%)**		
	5°C	**10°C**	**25°C**
Potassium hydroxide	7.31±0.59^{aA}	8.54±0.91^{aB}	9.69±0.78^{aC}
Magnesium klorida	8.48±0.31^{bA}	9.67±0.37^{bB}	10.68±0.52^{bC}
Calcium chloride	9.09±0.46^{cA}	11.96±1.05$_{cB}$	14.08±0.73^{cC}
Natrium klorida	10.21±1.68^{dA}	14.03±0.96^{dB}	19.56±1.20^{dC}
Potassium chloride	13.21±1.33^{eA}	17.70±0.92^{eB}	22.66±2.06^{eC}

* Columns with lowercase denoted significant (p<0.05) differences between saturated solutions. Columns with uppercase denoted significant (p<0.05) differences between temperatures. Data represents the mean (n=3) with LSD's test.

4. Conclusion

Research on moisture adsorption isotherms properties of dried overripe barlin bananas powder has been successfully carried out in this study. Equilibrium moisture content increases with increasing temperature and water activity. The adsorption curve has a type II shape based on the Brunauer classification (sigmoid). Peleg's model is the best model based on goodness of fit. Monolayer moisture content and adsorption surface area tend to increase with increasing temperature. Isosteric heat is positive, and Gibb free energy tends to increase but is still negative as the water content increases. Based on the results of the best model, storage of dried overripe barlin bananas powder is good at a moisture content range of 0.09-0.15 d.b.

Compliance with ethical standards

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Disclosure of Conflict of interest

The authors declare that they have no conflict of interest.

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