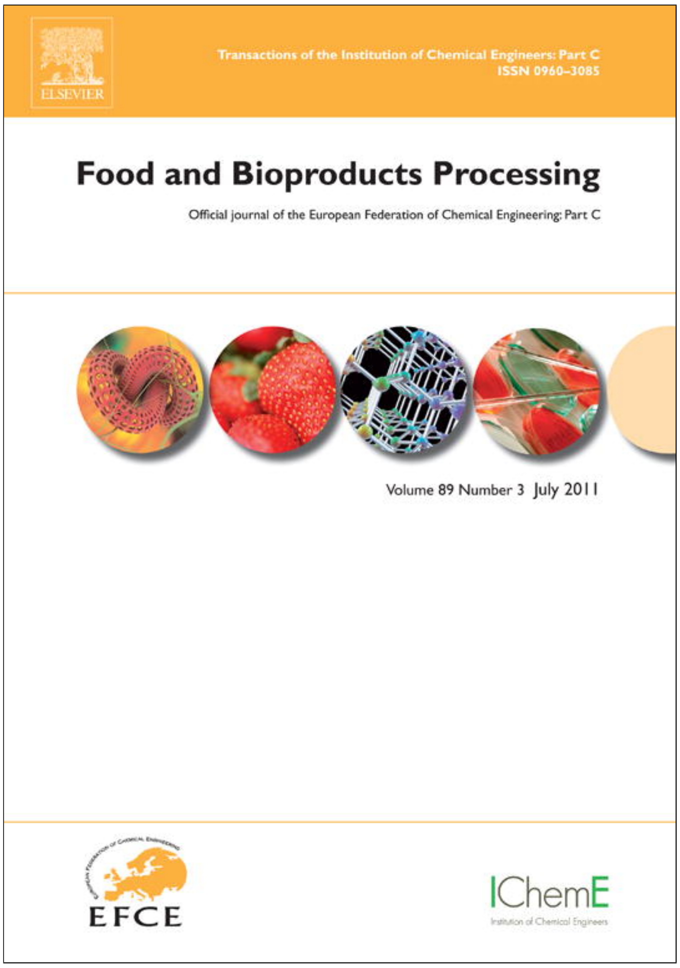
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Moisture sorption isotherms of two varieties of millet

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ABSTRACT

Moisture sorption isotherms of EX-BORNO and SOSAT C88 millets at temperature range of 30–70 °C and water activity range of 0.07–0.98 were determined using the static gravimetric method. The sorption isotherms of both millet varieties decreased with increasing temperature, exhibited type II behaviour according to BET classification and hysteresis having loop size increasing with increasing temperature. This is as a result of the hard nature of the millet varieties. The moisture sorption and the data fitted well with Modified Henderson, Modified Halsey, Modified Oswin and Modified GAB models. The constants of the equations used in fitting were determined by non-linear regression analysis when the models were compared using the standard error of estimate, mean relative percent deviation, fraction explained variation and residual plots. The Modified Oswin model gave the best fit for the whole set of data. The study has provided information and data useful in large scale drying and processing of millet which have remained at the traditional level despite the importance of the drought resistance crop in poverty alleviation.

Keywords: Millet; Sorption isotherm; Sorption hysteresis; Drying; SOSAT C88; EX-BORNO

1. Introduction

Pearl millet (*Pennisetum glaucum*) is an important cereal crop grown extensively in the Northern part of Nigeria. It is an early maturing crop that tolerates drought much better than most other cereals like maize and sorghum. There are two major local varieties of millet available namely; EX-BORNO with a yield potential of 2000–3000 kg/ha and the improved SOSAT C88 variety with a yield potential of 2500–3500 kg/ha (LCRI, 1997). It is consumed locally as *tuwo* (porridge produced from ground wet mash), *fura* (refreshing drink), *koko* (pap food drink made from dried flour or wet mash) and many other forms depending on local dietary habits. Ikwelle et al. (1993) ranked millet as the most important cereal in the Southern Sudan and the Northern Guinea. Nkama (1998) outlined the uses and traditional food preparations of millet in Nigeria. There are technological possibilities of the utilization of millet in such industrial applications as starch production. Drying is a fundamental operation in all of these processes.

The most common drying method for this crop in the tropics is the open-air sun-drying. However, this drying technique is slow, uncontrolled, exposed to environmental contamination and requires hard labour (Doymaz, 2005). Furthermore, direct exposure to solar radiation may affect the quality of the dried product significantly. The large scale production of millet and the wide range of possible products from it will require that the open-air sun-drying method currently practiced be replaced with efficient drying systems.

In order to control the drying process and storage conditions, besides the knowledge of thin layer drying equations (Sun and Woods, 1994), it is necessary to know the relationship between the equilibrium moisture content (EMC) in millet and the equilibrium relative humidity (ERH) of the drying air at a given temperature (Sun and Woods, 1997, 1998). This relationship is normally described by the moisture sorption isotherm equations. The moisture sorption isotherm of food graphically relates its equilibrium moisture content in either desorption or adsorption, to the water activity (a_w) at a definite temperature. These isotherms are extremely important quantitative measures in food preservation, storage, packaging and drying (Chen, 1997; Arslan and Togrul, 2005; Medeiros et al., 2006).

Several researchers have developed EMC/ERH equations to describe the moisture sorption isotherms of food and agricultural materials (Van den Berg and Bruin, 1981). Despite the availability of the large number of equations, no single equation is found to have the ability to describe accurately the

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Table 1 – Five commonly used moisture sorption isotherm models.

Model	Equilibrium moisture content model
Modified Henderson	$M \approx \left[\frac{-\ln(1-a_w)}{A(T+B)}\right]^{1/C}$
Modified Chung-Pfost	$M \approx \frac{-1}{C}\ln\left[\frac{(T+B)}{A}\ln a_w\right]$
Modified Halsey	$M \approx \left[\frac{-\ln a_w}{\exp(A+BT)}\right]^{-1/C}$
Modified Oswin	$M \approx (A+BT)\left[\frac{a_w}{1-a_w}\right]^{1/C}$
Modified GAB	$M \approx \frac{AB(C/T)a_w}{(1-Ba_w)(1-Ba_w+(C/T)Ba_w)}$

M, moisture content, % (d.b.); a_w, water activity (decimal); T, absolute temperature (K), A, B and C, constants specific to individual equations. *Source*: Yu et al. (1999).

EMC/ERH relations for different grains over a broad range of relative humidity and temperature (Sun and Woods, 1993). Therefore, for a specific crop there is need to search for the most appropriate EMC/ERH equation (Chen and Morey, 1989; Sun and Woods, 1994; Sun and Bryne, 1998). However, the five commonly used equations (Yu et al., 1999; Aviara et al., 2004) have been noted to be the Modified Henderson model (Thompson, 1972; Gely and Santalla, 2008), Modified Chung-Pfost model (Pfost et al., 1976; Okunola and Igbeka, 2007), Modified Halsey model (Iglesias and Chirife, 1976; Menkov et al., 2004), Modified Oswin model (Chen and Morey, 1989; Sanjeev and Singh, 2006), and the GAB model (Van den Berg, 1984; Timoumi et al., 2004) as modified by Jayas and Mazza (1993). Table 1 presents these equations for both the equilibrium moisture content and equilibrium relative humidity (water activity).

The theoretical implications of moisture sorption hysteresis range from adsorption of the irreversibility of the sorption process to the question of validity of thermodynamic functions determined from such a system (Kapsalis, 1987; Aviara et al., 2006). The explanation for occurrence of moisture sorption hysteresis which ranged from the ink bottle theory, the molecular shrinkage theory (Chung and Pfost, 1967) and the capillary condensation and swelling fatigue theory (Ngoddy and Bakker-Arkema, 1975) and its consequences have been discussed (Kapsalis, 1981; Karel, 1989; Yan et al., 2008). Its practical implications deal with the effect on storage stability. Kapsalis (1987) grouped the nature and variations of moisture sorption hysteresis in food into three types and noted that the phenomenon decreases with increase in temperature, while Benado and Rizvi (1985) and Ajisegiri (1987) showed that it can be eliminated through several desorption and adsorption cycles.

This study therefore determined the desorption and adsorption moisture isotherms of EX-BORNO and SOSAT C88 millets at drying temperatures of 30, 40, 50, 60 and 70 °C, evaluated the suitability of four commonly used three-parameter moisture isotherm model equations and investigated the nature of the moisture sorption hysteresis.

2. Materials and methods

2.1. Sample preparation

The SOSAT C88 and EX-BORNO millets used in this study were obtained from the Lake Chad Research Institute (LCRI), Maiduguri in Borno State, Nigeria. Triplicate samples, each weighing 15 g were used to determine the moisture content of whole grains using the oven drying method as recommended in the ASAE standard. This involved drying in an oven at 103 ± 2 °C until constant weight was obtained (ASAE, 1983). The initial moisture contents were found to be 10.80 and 10.27 ± 0.22% (db). The bulk quantity of grains was cleaned and divided into two portions. One portion was prepared for use in determining the desorption equilibrium moisture content, by rewetting it to a higher moisture content. A calculated amount of water was added to this portion and the grains were then sealed in polyethylene bags and stored for 24 h. This enabled the moisture content to be raised to stable and uniform levels of 32.02 and 35.30% (db) for SOSAT C88 and EX-BORNO, respectively. The polyethylene bags were marked and transferred into a refrigerator at 4 °C and when needed for experiments, the grains were allowed to equilibrate in the ambient condition for 6 h.

The other portion was prepared for use for experiments on adsorption. The sample was dried at 80 °C for 2 days to obtain a lower moisture content of about 5.87 and 4.83% (db) for SOSAT C88 and EX-BORNO, respectively. The grains from this portion were also sealed in marked polyethylene bags and kept in a refrigerator. They were equally allowed to equilibrate in the ambient conditions for 6 h, when needed for experiments.

2.2. Equilibrium moisture content determination

The desorption equilibrium moisture contents of the two millet varieties were determined at temperatures of 40, 50, 60 and 70 °C over a water activity range of 0.07–0.98 using the static gravimetric method. This method involves the use of saturated salt solutions to maintain constant relative humidity (r.h.) in enclosed still moist air at a certain temperature to obtain the complete sorption isotherms. The triplicate samples of SOSAT C88 and EX-BORNO each weighing about 10 g were put into specimen baskets and placed inside glass desiccators. Saturated solutions of salts as used by Brooker et al. (1974) and Rizvi (1986) presented in Table 2 were used to maintain constant relative humidity levels in the desiccators (Young, 1967; Ajibola, 1989). Excess salt was maintained in each solution. The samples were placed in an open flat container and arranged in each desiccator bottle so that they will not be in contact with the salt solution. The desiccators containing the salt solutions and samples of millets were marked and placed inside temperature-controlled Gallenkamp DV 400 ovens (Weiss Gallenkamp UK) which were set at 30, 40, 50, 60 and 70 °C. The oven temperatures were monitored to within ±1.0 °C to maintain constant temperature for each setting.

The samples were weighed daily using a Mettler PC2200 DeltaRange analytical balance (Mettler-Toledo Inc., USA) with an accuracy of 0.001 g. Equilibrium was considered to have been attained when three identical consecutive measurements were obtained. This took between 7 and 12 days. The dry matter content was then determined by oven drying the sample at 103 ± 2 °C until constant weight was attained (ASAE, 1983). The equilibrium moisture content was calculated on a dry basis from the weight change and dry matter weight, and the average values at each temperature and water activity were determined. At higher water activity above 0.85, microbial growth may occur in sample and ruin it. Two methods are often used to control this: (i) using antimicrobial agents but these could change the a_w and moisture profile; (ii) vacuum method was used in this study to create an anaerobic environment in the dessicator. Where the occurrence of mould

Table 2 – Saturated salt solution and relative humidities (%) at different temperatures.

Salt	Temperature (°C) 30	Salt	Temperature (°C) 40	50	60	70
Sodium hydroxide	7.0	Sodium hydroxide	7.0	7.0	7.0	7.0
Lithium chloride	11.0	Lithium chloride	11.0	11.0	11.0	11.0
Potassium acetate	22.0	Calcium chloride	22.0	21.0	21.0	20.0
Magnesium chloride	33.0	Magnesium chloride	32.0	31.0	31.0	30.0
Magnesium nitrate	53.0	Manganese chloride	51.0	50.0	50.0	50.0
Sodium bromide	58.0	Sodium nitrite	61.0	60.0	60.0	60.0
Strothium chloride	71.0	Sodium nitrate	71.0	70.0	70.0	70.0
Ammonium sulphate	81.0	Potassium chloride	82.0	81.0	80.0	80.0
Potassium nitrate	93.0	Barium chloride	89.0	89.0	88.0	88.0
Potassium sulphate	97.0	Potassium sulphate	98.0	96.0	95.0	94.0

Source: Brooker et al. (1974) and Rizvi (1986).

was noticed, during the experiment such specimen pan was discarded.

The desorption and adsorption equilibrium moisture contents of EX-BORNO and SOSAT C88 millets were fitted to four of the moisture sorption isotherm models given in Table 1, using SPSS 14.0 for Windows for non-linear regression, which minimizes the sum of squares of deviations between experiment and theory in a series of iterative steps. All the models are three-parameter equations, which can be solved explicitly for equilibrium moisture content as a function of temperature. The non-linear regression procedure required that initial parameter estimates be chosen close to the true values. The initial parameter estimates were obtained by linearization of the models through logarithmic transformation and application of linear regression analysis, or solving a quadratic form of the equation in the case of modified GAB model.

The least-squares estimates or coefficients of the terms were used as the coefficients of the terms of the sorption models tested for the initial parameter estimates in the non-linear regression procedure. Model parameters were estimated by taking the equilibrium moisture content (EMC) to be the dependent variable. The goodness-of-fit of each model was evaluated using the standard error of estimate; mean relative percentage deviation, fraction explained variation and nature of the residual plots.

The standard error of estimate is denoted by SE and defined as

$$SE = \sqrt{\frac{\sum_{i=1}^{N}(Y_i - Y_i')^2}{d_f}} \quad (1)$$

The mean relative percent deviation (P) is defined as

$$P = \frac{100}{N}\sum_{i=1}^{N}\frac{(Y_i - Y_i')}{Y} \quad (2)$$

The fraction explained variation (FEV) is defined as

$$FEV \approx \frac{SSM}{SST} \quad (3)$$

$$\text{with } SSM = \sum_{i=1}^{N}(\hat{Y}_i - \bar{Y})^2 \text{ and } SST = \sum_{i=1}^{N}(Y_i - \bar{Y})^2$$

where Y is the measured value; Y', the value predicted by the model; $\bar{y}$, the mean value; N, the number of data points; d_f, the degree of freedom in the regression model; SSM, the sum of squares due to the model and SST, the total sum of squares. The differences between measured and predicted EMC values $(Y - Y')$ at various equilibrium relative humidities were defined as residuals, and plotted against measured values of EMC. These plots indicate the datapoint distribution around the origin. Residual plots is the plot of the difference between Experimental, i.e. observed values of EMC and predicted values from models tested and are further used to decide the predictive ability of sorption models in addition to SE, P and FEV. Low values of P and SE, and higher FEV and high degree of randomness in the residuals indicate a superior model. The fraction of variance explained is the square of the correlation coefficient, i.e. R^2.

3. Results and discussion

3.1. *Moisture sorption isotherms*

The experimental results obtained after adsorption and desorption performed on both millet varieties at 30–70 °C over the a_w range of 0.07–0.98 are presented in Figs. 1a, b and 2a, b. The sorption isotherms have the sigmoidal-shaped profile according to the BET classification. These curves are typical of legume and oil seeds as reported by Vertucci and Leopold (1987), Pappas and Rao (1987), Mazza and Jayas (1991), Menkov (2000) and Tarigan et al. (2006). The equilibrium moisture content (EMC) increased with increase in a_w and were lower at higher temperatures. This can be explained by the higher active state of water molecules at higher temperature thus the attractive forces between them decreasing (Yan et al., 2008). Similar trends for many seeds have been reported (Mazza and Jayas, 1991; Suthar and Das, 1997; Walters and Hill, 1998; Menkov and Dinkov, 1999; Menkov, 2000; Tarigan et al., 2006; Akanbi et al., 2006; Yan et al., 2008). The EMC values of millet seeds are similar to those obtained from other legume seeds; cowpea (Pappas and Rao, 1987), *Lathyrus* pea (Mazza and Jayas, 1991) and vetch seeds (Menkov, 2000).

The parameter estimates for the EMC models in which the desorption and adsorption EMCs were taken as dependent variables for the two millet varieties, respectively, when the data were used in the expressions of the four chosen moisture sorption isotherm equations for the EMC are as presented in Tables 3 and 4. The four moisture sorption isotherm models fitted well with the experimental data. However, the modified Oswin model fitted best with the least standard error of estimate, least mean relative percentage deviation and highest fraction explained variation on the desorption and adsorption

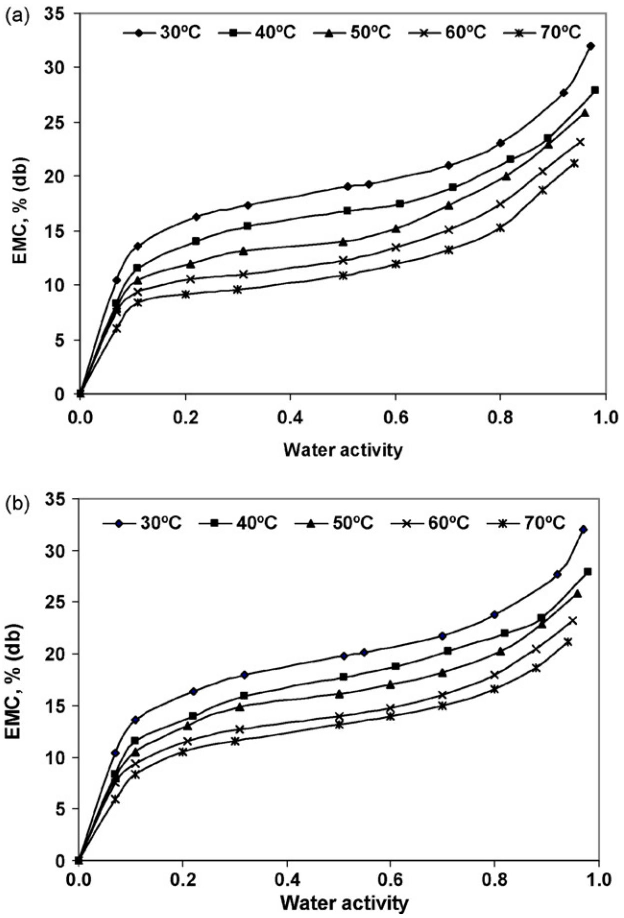

Fig. 1 – (a) Moisture adsorption isotherms of SOSAT C88 millet. (b) Moisture desorption isotherms of SOSAT C88 millet.

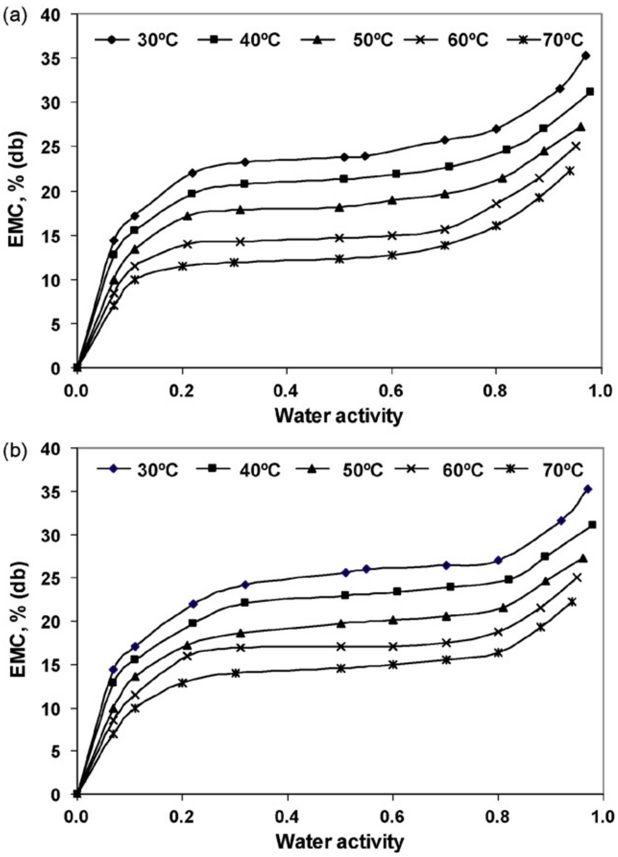

Fig. 2 – (a) Moisture adsorption isotherms of EX-BORNO millet. (b) Moisture desorption isotherms of EX-BORNO millet.

EMC of SOSAT C88 and EX-BORNO millets. This was closely followed by the modified Halsey model except for desorption in SOSAT C88 where the Modified Henderson gave the least SE followed by Modified Oswin.

According to Aviara et al. (2006) the standard error of estimate alone may not be sufficient evidence for the goodness-of-fit of a moisture sorption isotherm model based on experimental data and its temperature dependence. The nature of the residual plots should be considered in addition. From the above, although, Modified Henderson model had the least SE for desorption in SOSAT C88, it may not be considered the best for predicting the desorption EMC and its temperature dependence for SOSAT C88 at 30, 40, 50, 60 and 70 °C.

The results of the residual plot of the distribution of the data are also presented in Tables 3 and 4. Three of the models were found to give random plots which are indications of good fit for adsorption and desorption. Modified Oswin, however, gave random plots for desorption and adsorption for both varieties. The predictive performance of the modified Oswin model on the adsorption and desorption EMC of SOSAT C88 millet variety at 30, 40, 50, 60 and 70 °C are shown in Fig. 3a and b with the experimental plots. The isotherms fits well with the experimental data and are sigmoidal in shape showing that it adequately described and predicted the temperature dependence of the EMC data. A similar trend

Table 3 – Estimated parameters and comparison criteria for the EMC models of SOSAT C88 millet.

Parameter and criteria	Equilibrium moisture content models							
	Desorption				Adsorption			
	Modified Halsey	Modified Henderson	Modified Oswin	Modified GAB	Modified Halsey	Modified Henderson	Modified Oswin	Modified GAB
A	27.702	0.001	60.420	12.238	27.453	92677.421	63.024	9.518
B	−0.044	−333.000	−0.139	0.539	−0.047	1×10^9	−0.149	0.656
C	5.088	3.533	6.215	18284.92	4.786	3.331	5.858	-9×10^{10}
SE	1.6795	0.6227	1.1281	2.5118	1.5301	2.8011	1.1336	2.7815
P	9.639	15.008	5.932	12.991	8.840	99.956	5.998	15.963
FEV	0.991	0.996	0.996	0.980	0.992	0.974	0.996	0.974
Residual plot	Patterned	Patterned	Random	Random	Patterned	Patterned	Random	Random

A, *B*, *C* are the model constants; SE, the standard error of estimate; *P*, the mean relative percentage deviation; FEV, the fraction explained variation.

Table 4 – Estimated parameters and comparison criteria for the EMC models of EX-BORNO millet.

Parameter and criteria	Equilibrium moisture content models							
	Desorption				Adsorption			
	Modified Halsey	Modified Henderson	Modified Oswin	Modified GAB	Modified Halsey	Modified Henderson	Modified Oswin	Modified GAB
A	44.701	415.271	93.621	17.851	43.340	1103.079	75.895	10.275
B	−0.080	8×10^8	−0.231	0.346	−0.081	8×10^8	−0.241	0.621
C	6.612	4.761	8.155	20378.81	6.169	4.464	7.667	34644.71
SE	2.0562	3.8793	1.6143	3.8845	1.7799	4.0826	1.4417	2.7383
P	10.364	99.969	8.231	17.687	9.075	99.980	6.724	21.015
FEV	0.990	0.965	0.994	0.965	0.992	0.959	0.995	0.975
Residual plot	Random	Patterned	Random	Patterned	Random	Patterned	Random	Patterned

A, B, C are the constants; SE, the standard error of estimate; P, the mean relative percent deviation; FEV, the fraction explained variation.

was noticed in EX-BORNO (not shown). Of the remaining two models, the Modified GAB model was found acceptable for describing the adsorption and desorption EMC of SOSAT C88 millet and adsorption for EX-BORNO using the residual plot criterion with a random plot distribution. It, however, did not describe and predict well the temperature dependence of the EMC data adequately as shown in Fig. 4 for adsorption.

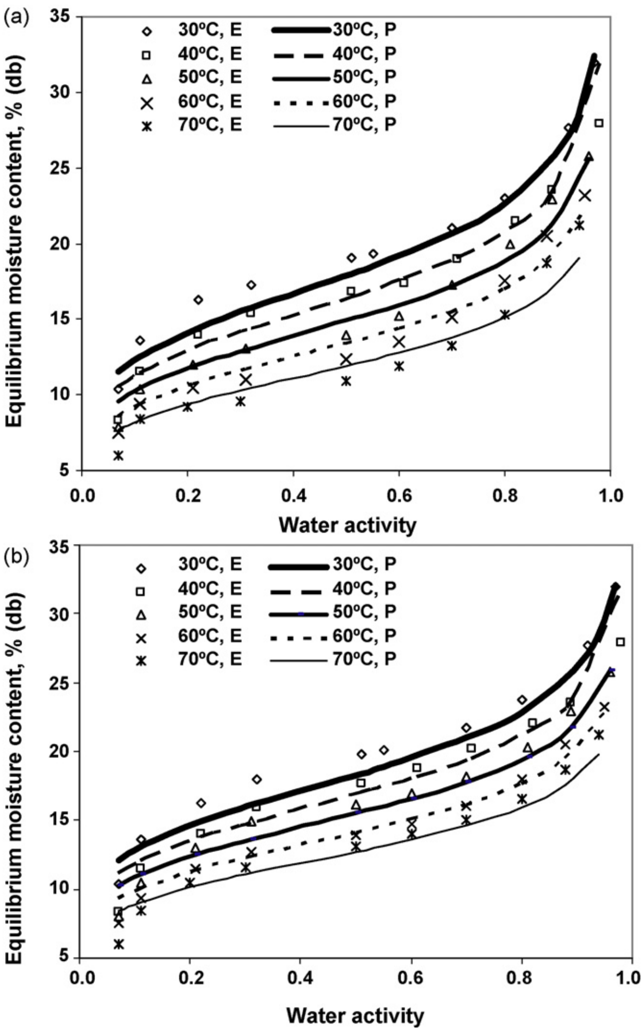

Fig. 3 – (a) Modified Oswin Experimental (E) and Predicted (P) adsorption isotherms of SOSAT C88. (b) Modified Oswin Experimental (E) and Predicted (P) desorption isotherms of SOSAT C88.

The desorption prediction also follow the same pattern of same prediction for all temperatures as indicated in the plot for all falling on the same path in Fig. 4. Modified Henderson though had the least SE for desorption in SOSAT showed a patterned residual plot all through indicating that it is not well fitted for the two millet varieties. Therefore Modified Oswin model that gave the least mean relative percent deviation, highest fraction and all random plots is therefore considered the best for predicting the adsorption and desorption EMC and its temperature dependence for the millet varieties.

3.2. *Moisture sorption hysteresis*

The combined plots of the desorption and adsorption isotherms of both millet varieties are presented in Figs. 5 and 6 showing evidence of the occurrence of moisture sorption hysteresis in sorption processes for the millet varieties. The adsorption isotherms at the temperatures lay below their desorption counterparts and both enclosed a hysteresis loop of the third type according to Kapsalis (1987) classification figures. Yan et al. (2008) stated that hysteresis has been related to the nature and state of the components in a food, reflecting their potential for structural and conformational rearrangements. When water moves out from the capillaries of biomaterials, during moisture desorption, the narrow ends of surface pores trapped and held water internally below the water activity where the water should have been released,

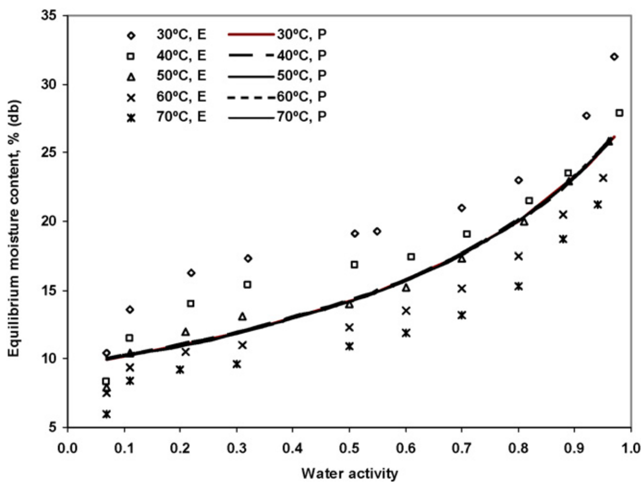

Fig. 4 – Modified GAB model Experimental (E) and Predicted (P) adsorption isotherms of SOSAT C88.

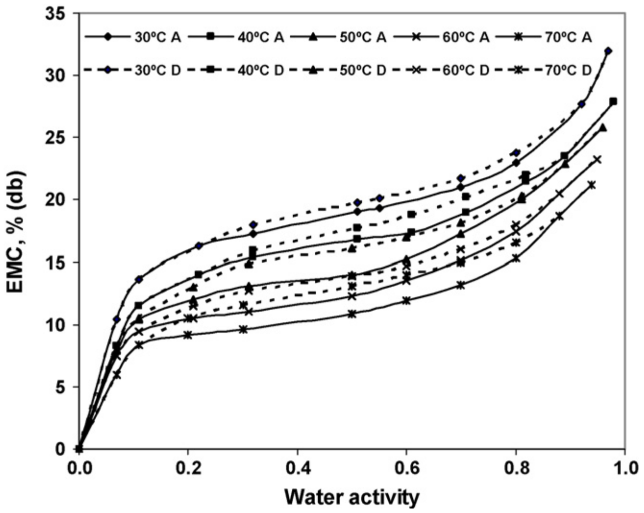

Fig. 5 – SOSAT C88 moisture sorption hysteresis (A is adsorption and D is desorption).

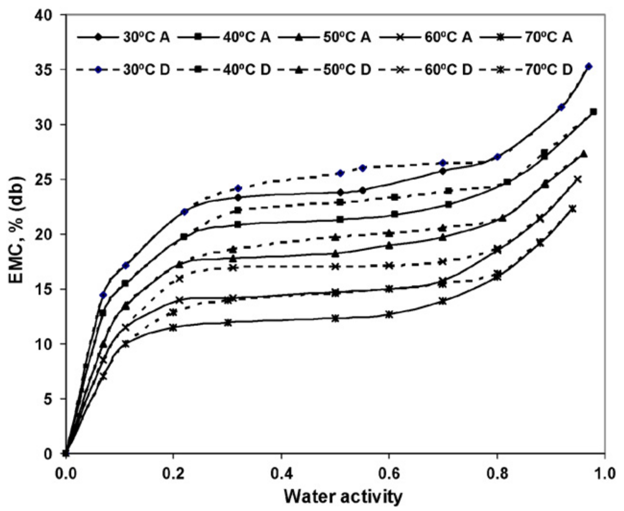

Fig. 6 – EX-BORNO moisture sorption hysteresis (A is adsorption and D is desorption).

thus there was greater moisture content at a low range of water activity. During adsorption the pure water would dissolve solutes present in the material and the dissolution of solutes increased the surface tension, resulting in lower water activity at given moisture content. The swelling of polymeric materials during moisture adsorption can also lead to hysteresis.

It can be seen that while hysteresis commenced in both varieties, at an a_w above 0.2 for 30 °C, it commenced below an a_w of 0.2 at 70 °C. The termination of the hysteresis followed the same pattern. The higher the temperature, the longer the termination point. In the higher range of a_w, the hysteresis loop terminated at about the same a_w for all temperatures. This is an indication that the span of moisture sorption hysteresis in millet increases with increase in temperature. The loop becomes wider with temperature as evident in the two presented the size of the loop is larger at 70 °C than at 30 °C, indicating that the total hysteresis in millet increases with temperature. This may be as a result of the nature of the millet materials which are hard in nature. Higher temperature therefore leads to expansion of the hard solid seed and the pores hence faster release of moisture during desorption.

4. Conclusions

The moisture sorption isotherms of SOSAT C88 and EX-BORNO millets at temperatures 30–70 °C have been established. The desorption and adsorption isotherms are sigmoidal in shape and showed a marked temperature effect. The Modified Oswin model, explained variation on the adsorption and desorption equilibrium moisture content and is therefore the best model for prediction of the sorption phenomena among four commonly used models investigated. The desorption and adsorption isotherms show the occurrence of moisture sorption hysteresis in millet and are of type three with the span increasing with increase in temperature. The size of the loop appears to be larger at 70 °C than at 30 °C and implies that the total hysteresis in millet increases with temperature as a result of the hard nature of the millet. This information are useful in handling the drying and processing operations of this highly used but less researched drought resistant cereal crop whose processing is still at the traditional manual level. Further investigation into the number of repeated adsorption and desorption require for closure and disappearance of the hysteresis loop with temperature need to be carried out to obtain the information for millet.

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