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Equilibrium Moisture Characteristics of Wheat Flour at Higher Temperatures

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ABSTRACT

Equilibrium moisture content (EMC) of wheat flour was obtained by equilibrating them at 10-78% equilibrium relative humidity (ERH) at 50, 60, 70, 80 and 90 °C above saturated inorganic salt solutions. Seven EMC-ERH models namely Henderson, modified Henderson, modified Chung-Pfost, modified Oswin, modified Halsey, modified GAB and Chen-Clayton were fitted to the observed data and were evaluated using mean relative percent error, standard error of estimate and residual plots. At a constant relative humidity, equilibrium moisture content decreased with increasing temperature. The Chen-Clayton model described the EMC data the best, modified Henderson and modified Chung-Pfost equations gave good fit. The heat of vaporization (h_{fg}) of wheat flour at different flour moisture contents and temperatures was estimated from EMC-ERH data by using the Clausius-Clapeyron equation.

Keywords: *Equilibrium moisture content; Heat of vaporization and Wheat flour.*

Moisture sorption isotherms describe the relationship between moisture content and equilibrium relative humidity. The isotherms are useful in drying and storage of the products. Isotherms help in designing the thermal processes. Wheat flour (WF) is used to prepare various types of breads and pastries, pasta, breakfast cereals and other products used for human consumption. The WF is subjected to the thermal treatments for improving the safety without significantly affecting the functionality (Boreddy, 2015; Boreddy et al., 2019).The milling of wheat do not produce sterile WF. However, the traditional thermal treatments take long time to reach the treatment temperature because of lower thermal conductivity. Apart, WF is given thermal treatments in packaged form to avoid the cross-contamination in order to prepare recipes for speciality customers. Traditional thermal processing methods either take long time or not suitable for package processing. Hence, novel thermal processing methods such as radio-frequency and microwave methods were found to reduce processing times. These methods raise the temperature of the packaged produce many times faster. Taking the advantage of these novel methods, pasteurization methods for the flour are found suitable to carry at higher temperatures up to 90 °C in order to reduce processing times and to improve productivity (Boreddy *et al.*, 2016). As the product is being processed in package and heating rates are faster, there is a possibility of moisture losses from the product and deposition of moisture on inside package surfaces. The phenomenon of loss of moisture and deposition depends on the product moisture content and temperature which is not clearly understood (Boreddy *et al.*, 2016). To understand such phenomenon the EMC-ERH data is needed. The EMC-ERH data of wheat flour is available at lower temperatures. However, EMC-ERH data at higher temperatures throughout the relative humidity range is not available. Keeping in view the need for an EMC-ERH data of wheat flour, the present study is undertaken with the following objectives.

1. To determine the equilibrium moisture content data of wheat flour at higher temperatures by employing static gravimetric method
2. To evaluate the applicability of general isotherm models for description of EMC-ERH data of wheat flour and
3. To calculate the heat of vaporization (h_{fg}) of wheat flour as a function of moisture content and temperature.

MATERIALS AND METHODS

Wheat flour (Brand name: Aashirwad wheat atta, manufactured by: ITC Ltd.) was purchased locally in Bapatla and used in the experiments. The initial moisture content of the wheat flour was in the range of 9.66% d.b.

Experimental procedure

The static gravimetric method was used for determination of equilibrium moisture content of wheat flour. Eleven different saturated solutions of various inorganic salts were employed to generate the controlled humidities ranging from 10 to 78% (seven levels) in a closed chamber. Studies of sorption process were carried out at five temperatures of 50, 60, 70, 80 and 90 °C for wheat flour. Each desiccator was provided with a perforated and raised platform and the level of saturated salt solutions in it was kept below the perforated platform in order to avoid contact of the salt solution with the sample holder. For each of these experiments about 3 g sample of wheat flour was taken separately into the respective weighing bottles. Samples were allowed to equilibrate with the environment inside the desiccators until there was no discernible weight change. It took about 15-30 days, depending upon the nature of the samples. The equilibrated moisture content of the samples was calculated from the initial moisture content, initial and final equilibrated weights of the samples.

Isotherm model fitting

Seven general forms of EMC/ERH (Isotherm) models as described in (Table 1) were chosen to fit the experimental EMC-ERH data determined in this study. The experimental data at all temperatures and relative humidities was fitted into a chosen model once at a time with the help of Microsoft Excel Solver. "GRG Nonlinear" solving method was selected for solving the general isotherm equations with the experimental data. The other parameters set while using solver were: constants and sum of squares of residuals. The constants that were yielded after fitting the data were used to calculate the predicted EMC values of the respective products.

Analysis of data

The above EMC-ERH models were evaluated for their suitability in predicting EMC of the WF on the basis of mean relative percent error (P_e), standard error of estimate (SEE) and residual plots.

The mean relative percent error (P_e) is defined as: P_e (%) =

$$\frac{100}{\text{No. of data points}} \sum \frac{|\text{Predicted EMC-Measured EMC}|}{\text{Measured EMC}}$$

Standard error of estimate (SEE) is defined as:

$$SEE = \sqrt{\frac{\sum(\text{Predicted EMC} - \text{Measured EMC})^2}{\text{Degrees of freedom of regression model}}}$$

The differences between measured and predicted equilibrium moisture content values at various ERHs were defined as residuals. The residuals were plotted against predicted values of EMC. The residual plots were assessed for pattern or randomness. If the residuals in the plot are dispersed randomly the model is considered as the suitable and, then looked for SEE and P_e values to judge the best fit.

Heat of vaporization

The following equation derived from the Clausius-Clapeyron equation was used in calculation of heat of vaporization of wheat flour (Howell and Buckius, 1987).

$$\frac{h^1{}_{fg}}{h_{fg}} = \frac{\ln(RH_1 P_{vs1}) - \ln(RH_2 P_{vs2})}{\ln P_{vs1} - \ln P_{vs2}}$$

Where,

$h^1{}_{fg}$- heat of vaporization of product, kJ kg^{-1},

h_{fg} - heat of vaporization of pure water, kJ kg^{-1},

P_{vs1} and P_{vs2} are saturated vapour pressures at temperatures T_1 and T_2.

In computing the heat of vaporization from this equation the values of RH_1 and RH_2 at a given equilibrium moisture levels will be obtained from the best fit modified Henderson isotherm equation for the two temperatures T_1 and T_2. The above equation expresses the ratio of the heat of vaporization of the moisture in a product at particular moisture content to that of the heat of vaporization of pure water at the same temperature. The HV of the moisture in the respective product at different moisture contents were determined for different product temperatures from 50 to 90 °C.

RESULTS AND DISCUSSION

Equilibrium Moisture Content (EMC) of wheat flour

Experiment was conducted in the ERH range from 10.23% to 78.68% (Greenspan, L. 1977) and temperature of 50, 60, 70, 80, and 90 °C values. The time taken to reach the equilibrium conditions for WF were minimum of 15 days at 90 °C and maximum of 34 days at 50 °C. Generally, the number of days taken for reaching equilibrium conditions in static gravimetric method was about 3 weeks to 35 days. The time taken for reaching equilibrium for commercial wheat flour was 35 days at 29 °C (Clement *et al*., 2018), and about 3 weeks for obtaining the sorption isotherms of Durum wheat flour (Chuma*et al*., 2012), and soy and wheat flours (Riganakos*et al*., 1994). The criterion of equilibrium condition of the food product with the environmental relative humidity considered by the various authors were different. Most of the researchers considered no discernible weight change in the sample (Clement *et al*., 2018; Reddy and Chakraverty, 2004), some authors considered change in mass between two successive readings was less than 2 mg (Li *et al*., 2011) and also less than 1 mg/g (Martin-Santos *et al*., 2012).The initial, final sample weights together with the initial moisture content of the WF sample were used to calculate the EMC at that condition.

Table 1. EMC-ERH (Isotherms) models used to fit the experimental EMC data of wheat flour

S.No.	Name of the model	General form	References
1	Henderson	1-ERH = $exp(-ATM^{B})$	Henderson, 1952
2	Modified Henderson	1-ERH = $exp[(-A(T+B)M^{C})]$	Thomson *et al*., 1968
3	Modified Chung-Pfost	ERH = $\left[\frac{-A}{T+B} exp(-CM)\right]$	Chung and Pfost 1967
4	Modified Halsey	ERH = $exp\left[\frac{-exp(A+BT)}{M^{C}}\right]$	Halsey, 1948
5	Modified Oswin	ERH= $\frac{1}{1+\left(\frac{A+BT}{M}\right)^{C}}$	Chen and Morey, 1989
6	Modified GAB	EMC = $\frac{A\left(\frac{C}{T}\right) * B * ERH}{(1 - B * ERH)(1 - B * ERH + \left(\frac{C}{T}\right) B * ERH)}$	Anderson, R.B. 1946; Guggenheim, E.A. 1966
7	Chen-Clayton	ERH= exp $\left[-\frac{A}{T^{B}} * \exp(-CT^{D}M)\right]$	Chen and Clayton,1971

Table 2. The EMC-ERH model co-efficients, standard error of estimate (SEE), mean relative percent error (P_e) and pattern of residual plots (random / patterned) for wheat flour

Name of the Model	Model Coefficients				SEE	P_e(%)	Pattern of Residual plot
	A	B	C	D			
Henderson	9.13183×10^{-6}	3.452769	-	-	0.098	16.82	Patterned
Modified Henderson	1.622×10^{-4}	1.42×10^{-3}	1.988271	-	0.032	5.32	Random
Modified Chung-Pfost	349.4608	1.39×10^{-3}	0.251647	-	0.035	4.77	Random
Modified GAB	21.42574	0.048292	1725.782	-	0.046	7.21	Patterned
Modified hasley	3.845592	9.998×10^{-8}	2.083367	-	0.107	17.96	Random
Modified Oswin	7.448181	5.75×10^{-4}	2.612176	-	0.102	14.47	Random
Chen Clayton	5.1952636	9.88×10^{-4}	0.012631	0.712443	0.03	4.82	Random

Table 3. Heat of vaporization of wheat flour in kJ/kg at different temperatures and moisture contents (MC)

Moisture content % d.b.	Temperature				
	50 °C	60 °C	70 °C	80 °C	90 °C
	Heat of vaporization of wheat flour kJ/kg				
3	3432.1	3330.3	3244.6	3169.9	3102.4
6	3339.7	3236.3	3149	3072.8	3003.9
9	3199.6	3095.1	3007.1	2930.3	2861.1
12	3030.2	2927.8	2842.2	2768	2701.7
15	2854.4	2759.4	2681.1	2614.1	2554.9
	Water kJ/kg				
	2382.9	2358.6	2333.9	2308.9	2283.2

The EMC of WF at any particular equilibrium relative humidity (ERH) decreased with an increase in environmental temperature. The relationship between experimental values of EMC and ERH of WF at 50, 60, 70, 80 and 90 °C is plotted in Fig. 1. As the environmental temperature increased the amount of EMC decreased. The EMC values ranged from 2.05 to 4.01 at lower ERH value of about 10% whereas the EMC values ranged from 9.21 to 13.81 at higher ERH value of about 78% when the environmental temperature increased from 50 to 90 °C. This may be attributed to the adsorption of higher moisture at lower temperatures when compared to the lower adsorption of moisture at higher temperatures. In other words, as the environmental temperature increases, the water vapour pressure of the moisture within the wheat flour sample increases that hastens the diffusion of water vapour from wheat flour to the surrounding environment (Siripatrawan and Jantawat, 2006). The trend is exhibited throughout the ERH values range.

The experimental EMC values obtained for WF in this study were lower than that of the values obtained at 29 °C by Clement *et al*., (2018), and EMC values at 10, 20, and 30 °C by Martin-Santos *et al*., (2012). The EMC values of WF are also lower than that of the whole wheat at the respective conditions as reported by Li *et al*., (2011).

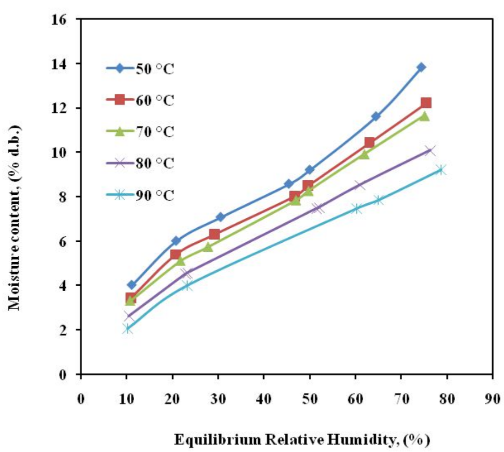


Fig.1 *Experimental values of equilibrium relative humidity and equilibrium moisture contents of wheat flour at different temperatures*

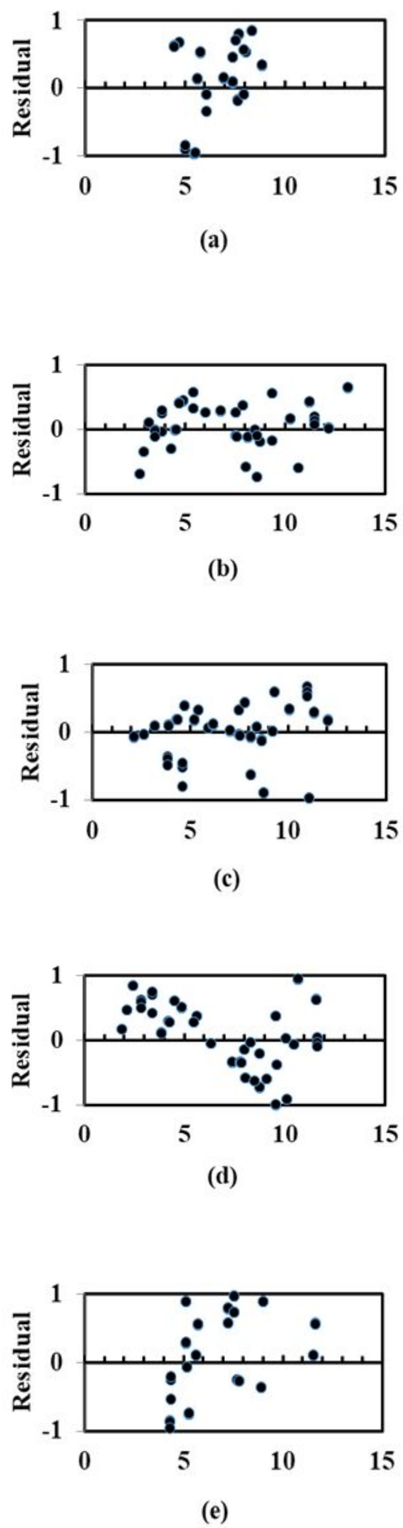
The isotherms of WF were plotted for experimental EMC-ERH values at different environmental temperatures in the Fig. 1. The isotherm of WF at all the environmental temperatures depicts the Type 2 sorption isotherm. In other words, the Type 2 isotherm is a sigmoidal and curves are concave upwards. The Type 2 isotherm takes into account of filling moisture in multi-layers of material's internal surfaces (Brunauer*et al.*, 2009; Mathlouthi and Roge, 2003).

Model fitting EMC-ERH data of WF

The experimental EMC-ERH data of WF was fitted in to seven sorption isotherm models as described in the Table 1. The models were chosen to fit with the experimental data of WF in order to have the EMC-ERH relationships at other conditions also with in the experimental range. The coefficients of the EMC-ERH models namely, Henderson, modified Henderson, modified Chung-Pfost, modified Halsey, modified Oswin, modified GAB and Chen-Clayton were determined by fitting the experimental EMC-ERH data obtained at all the experimental ERHs and environmental temperatures (used triplicated values). The suitability of the each model to predict the EMC-ERH relationship of WF was determined from the values of standard error of estimate (SEE), mean relative percent error (P_e), and residual plots. The EMC-ERH model co-efficients, SEE, P_e and pattern of residual plots (random / patterned) are presented in Table 2. The trend of the residual plots against the predicted values of different models was also plotted in Fig. 2.

As presented in the Table 2 and the trend of the residual plots in Fig. 2, the trend of the residuals was found to be random for the models namely modified Henderson, modified Chung-Pfost, modified Halsey, modified Oswin, and Chen-Clayton models except for Henderson and modified GAB models for which the residuals were patterned. Although modified Halsey and modified Oswin showing random residual plots, the P_e values are higher when compared to the other models that have shown the random residuals. Hence, the models namely modified Henderson, modified Chung-Pfostand Chen-Clayton were found suitable to describe the EMC-ERH relationships of WF in the experimental range. Among these suitable models, Chen Clayton model exhibited the lowest SEE and P_e values and the residuals were distributed randomly throughout the predicted EMC range. Hence, it may be concluded that the Chen Clayton model is the best fit to describe the EMC-ERH relationships of WF at all studied temperatures (50, 60, 70, 80, and 90 °C).

Clement *et al*., (2018) found BET, Khun and Halsey models to describe the EMC-ERH data of commercial wheat flour at 29 °C adequately. GAB model was found to describe the isotherms of Durum Wheat flour adequately (Chuma*et al*., 2012). GAB model was also found to describe the isotherms of whole wheat flour adequately by in the environmental temperature range of 10 to 30 °C by Martin-Santos *et al*., (2012). However, Li *et al*., (2011) reported the BET, modified Chung-Pfost and modified Oswin models were the best to describe the sorption isotherms of whole wheat at 10, 20, 25, 30 and 35 °C. Hence, it may be concluded that the EMC-ERH relationships of wheat flour at higher temperatures are different than that of the lower temperatures, and the EMC models that describe the EMC-ERH relationships of WF are also different.



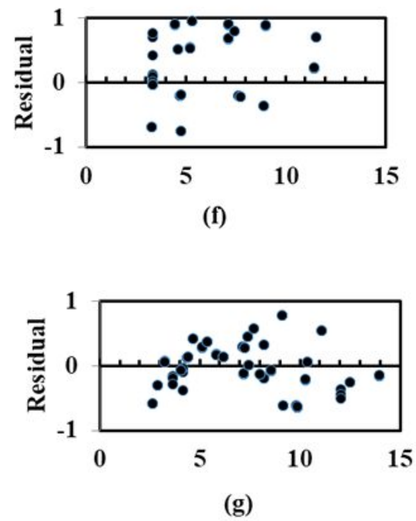


Fig.2 *Residual plots of different models of WF : (a) Henderson, (b) Modified Henderson, (c) Modified Chung-Pfost, (d) Modified GAB, (e) Modified Halsey, (f) Modified Oswin and (g) Chen-Clayton*

The effect of temperature on the sorption isotherms is of great importance given that foods are exposed to a range of temperatures during storage and processing and water activity or EMC changes with temperature. Temperature affects the mobility of water molecules and the dynamic equilibrium between the vapour and absorbed phases (Al-Muhtaseb*et al*., 2002). The plots of predicted EMC from the suitable models in the ERH range were plotted at each temperature separately in the Figures from 3 to 7. From the figures it may be observed that the closer predictions of EMC values through modified Henderson, modified Chung-Pfost and Chen-Clayton models when compared with the experimental EMC values. The prediction of EMC for WF through modified Chung-Pfost model shows the high deviation from the experimental values at higher ERH values at the lowest 50 °C (Fig. 3) and the highest 90 °C (Fig. 7) environmental temperature. However, as described earlier the modified Henderson and Chen-Clayton models described the EMC-ERH relationships of WF the best throughout the ERH range at all environmental temperatures.

Heat of Vaporization of wheat flour

The heat of vaporization (HV) for WF was calculated with the help of Clausius-Clapeyron equation. While calculating the HV values, the relative humidity values (RH_1 and RH_2) at given EMC levels were obtained from the Chen-Clayton (which was found to be the best to predict the EMC-ERH values for WF) at the two temperatures T_1 and T_2.The heat

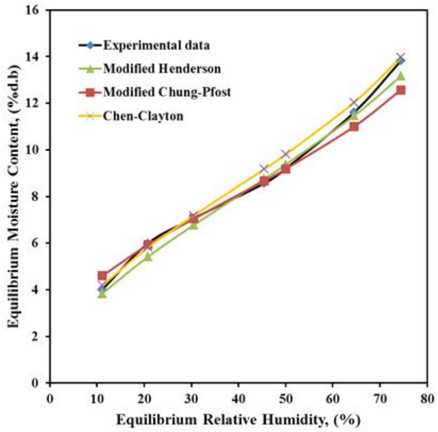


Fig. 3 *Experimental and predicted (modified Henderson, modified Chung-Pfost and Chen-Clayton) Equilibrium moisture contents of wheat flour at 50 °C*

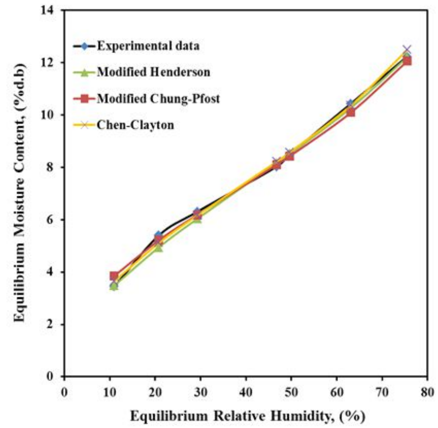


Fig. 4 *Experimental and predicted (modified Henderson, modified Chung-Pfost and Chen-Clayton) equilibrium moisture contents of wheat flour at 60 °C*

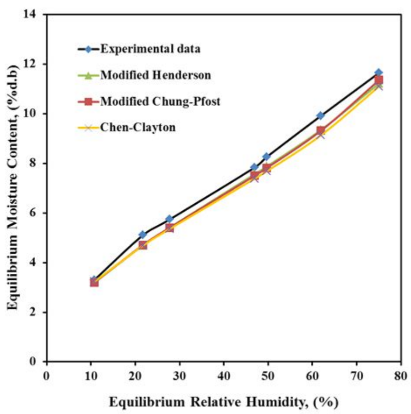


Fig. 5 *Experimental and predicted (modified Henderson, modified Chung-Pfost and Chen-Clayton) equilibrium moisture contents of wheat flour at 70 °C*

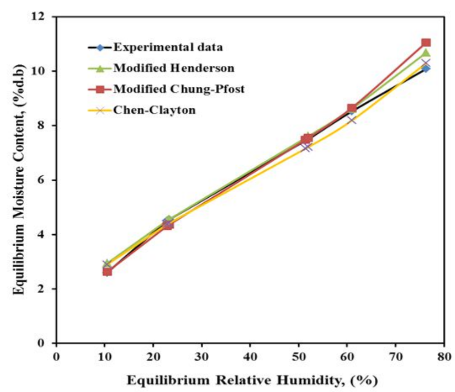


Fig. 6 *Experimental and predicted (modified Henderson, modified Chung-Pfost and Chen-Clayton) equilibrium moisture contents of wheat flour at 80 °C*

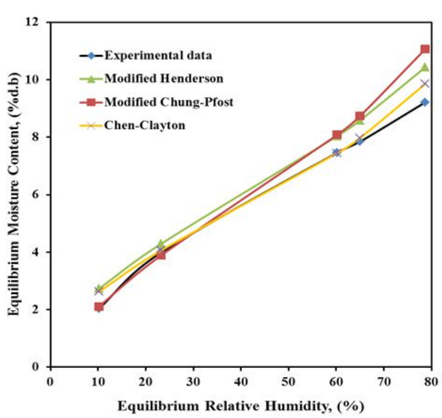


Fig. 7 *Experimental and predicted (modified Henderson, modified Chung-Pfost and Chen-Clayton) equilibrium moisture contents of wheat flour at 90 °C*

of vaporization of WF was calculated for moisture contents from 3 to 15% (d.b.) and the temperatures 50, 60, 70, 80 and 90 °C. The values of HV of water from the wheat flour and the values of pure water are presented in Table 3. The data show that the heats of vaporization of wheat flour increase as moisture content and flour temperature decrease, and these values were always higher than that of pure water.

CONCLUSION

The EMC increased with an increase in ERH at all the experimental temperatures. The shape of the isotherms obtained for WF was sigmoidal in shape, which is a characteristic for most of the food products. The EMC values obtained for WF was different and lower than that of the literature values which were reported at lower environmental temperatures. The isotherm models namely modified Henderson, modified Chung-Pfostand Chen-Clayton were found suitable to describe the EMC-ERH relationships of WF in the experimental range. Among these suitable models, Chen Clayton model was found to be the best for WF. The heat of vaporization of WF increased as the moisture content and product temperature decreased, and these values were always higher than that of the pure (free) water.

LITERATURE CITED

Al-Muhtaseb A H, McMinn W A M and Magee T R A 2002 Moisture sorption isotherm characteristics of food products. *TransIcheme*, 80: 118-128

Anderson R B1946 Modifications of the BET equation.*Journal of American Chemical Society*, 68: 686-691.

Brunauer S, Deming L S and Teller E 2009 On a theory of Van der Waals adsorption of gases.*Journal of the American Chemical Society*, 62 (7): 1723-1732

Boreddy S R and Subbiah J 2016 Temperature and moisture dependent dielectric properties of egg white powder. *Journal of Food Engineering,* 168: 60-67

Boreddy S R 2015 Radiofrequency-assisted thermal processing for improving microbiological safety of low-moisture food powders. The University of Nebraska-Lincoln

Boreddy S R, Rose D J and SubbiahJ 2019 Radiofrequency-assisted thermal processing of soft wheat flour. *Journal of Food Science*, 84 (9): 2528-2536

Clement Y B Y, Benjamin Y N, Fernande A E and Kablan T 2018 Moisture adsorption isotherm of commercial wheat flour in Cote d'Ivoire. *International Research Journal of Engineering and Technology,* 5(4): 1318-1326

Chakraverty A 1994*Post harvest technology of cereals, pulses and oilseeds*. Third edition.Oxford and IBH Publishing Company Pvt. Ltd. New Delhi.

Chen C S and Clayton J T 1971 The effect of temperature on sorption isotherms ofbiological materials.*Transactions of the ASAE,*14: 927-929

Chen C and Morey R V 1989 Comparison of four EMC/ERH equations.*Transactions of the ASAE,* 32: 983-990

Chung D S and P fost H B 1967 Adsorption and desorption of water vapour by cereal grains and their products. Part II: Development of the general isotherm equation. *Transactions of the ASAE*,10: 549-557

Chuma A, Ogawa T, Kobayashi T and Adachi S 2012 Moisture sorption isotherm of durum wheat flour. *Food Science Technology,* 18(5): 617-622

Greenspan L 1977 Humidity fixed points of binary saturated aqueous solutions. *Journal of Research of the National Bureau of Standards - A. Physics and Chemistry,* 81(1): 89-96

Guggenheim E A 1966*Applications of statistical mechanics*. Oxford: Clarendon Press.

Halsey G 1948 Physical adsorption on non-uniform surfaces.*Journal of chemical physics*, 16: 931-937

Henderson S M 1952A basic concept of equilibrium moisture.*Agricultural Engineering*, 33:29-32

Howell J R and Buckius R O 1987*Fundamentals of engineering thermodynamics*. McGraw-Hill, New York.

Igathinathane C, Womac A R, Sokhansanj S and Pordesimo L O 2005 Sorption equilibrium moisture characteristics of selected corn stover components. *Transactions of the ASABE,* 48(4): 1449-1460

Li X, Cao Z, Wei Z, Feng Q and Wang J 2011 Equilibrium moisture content and sorption isosteric heats of five wheat varieties in China *Journal of Stored Products Research*, 47: 39-47

Martin-Santos J, Vioque M and Gomez R 2012 Thermodynamic properties of moisture adsorption of whole wheat flour. Calculation of net isosteric heat.*International Journal of Food Science and Technology,* 47(7): 1487-1495

Mathlouthi M and Roge B 2003 Water vapor sorption isotherms and the caking of food powders.*Food Chemistry*, 82(1): 61-71

Reddy B S and Chakraverty A 2004 Equilibrium moisture characteristics of raw and parboiled paddy, brown rice and bran. *Drying Technology,* 22(4): 837-851

Riganakos K A, Demertzis P G and Kontominas M G 1994 Water sorption by wheat and soy flour: Comparison of three methods. *Journal of Cereal Science*, 20: 101-106

Siripatrawan U and Jantawat P 2006 Determination of moisture sorption isotherms of jasmine rice crackers using BET and GAB models. *Food Science and Technology International*, 12: 459-465

Thompson T L, Peart R M and Foster G H 1968 Mathematical simulation of corn drying: a new model. *Transactions of the ASAE*, 11(4): 582-586

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