ISSN 1330-9862
(FTB-1561)

scientific note

Moisture Sorption Isotherms of Sesame Flour at Several Temperatures

Nikolay D. Menkov and Albena G. Durakova*

University of Food Technologies, Department of Process Engineering, 26 Maritza Blvd., BG-4000 Plovdiv, Bulgaria

Received: September 30, 2005
Accepted: April 26, 2006

Summary

Moisture equilibrium data (adsorption and desorption) of sesame flour were determined using the static gravimetric method of saturated salt solutions at three temperatures, 10, 25 and 40 °C. The range of water activities for each temperature was between 0.11 and 0.85. Equilibrium moisture content decreased with the increase in storage temperature at any given water activity. The experimental data were fitted by five mathematical models (modified Oswin, modified Halsey, modified Chung-Pfost, modified Henderson and Guggenheim-Anderson-de Boer (GAB)). The GAB model was found to be the most suitable for describing the sorption data. The monolayer moisture content was estimated using the Brunauer-Emmett-Teller equation.

Key words: sesame flour, sorption isotherm, modelling

Introduction

With recent consumers' interest in functional food, sesame flour has been increasingly used as an ingredient and a functional modifier in many foods. Kang *et al.* (*1*) consider that the full defatted sesame flour decreases oxidative stress. De Fatima *et al.* (*2*) blended wheat flour, soy meal and defatted sesame meal and investigated its acceptability for macaroni, cookies and burgers. Alobo (*3*) replaced millet flour, used for preparing biscuits, with sesame seed flour. Egbekun and Ehieze (*4*) investigated the composition and functional properties of full fat and defatted sesame flour and recommended them for incorporation in the functional food production. El-Adawy (*5*) studied the physical properties of dough and breadmaking properties of a blend from wheat flour and sesame flour. Saldivar *et al.* (*6*) evaluated the nutritional value of table bread fortified with soybean meal and sesame meal. Afolabi *et al.* (*7*) made similar investigations and established that the bread produced with sesame flour had higher protein and fiber contents.

Water plays an important role with respect to the properties of food systems. The water in food influences physical or textural characteristics of the product as well as its chemical stability (*8*). Sorption properties of food (equilibrium moisture content and monolayer moisture) are essential for the design and optimization of many processes such as drying, packaging and storage (*9*). The water activity of the flour as a hygroscopic material exerts a strong influence on its quality and technological properties. Bothast *et al.* (*10*) observed microbiological and sensory properties of wheat flour and corn flour at several temperature and moisture levels. Abdullah *et al.* (*11*) investigated the fungal spoilage of rice flour and wheat flour and recommended the starch based food to be stored at water activity levels not higher than 0.65. Nasir *et al.* (*12*) and Butt *et al.* (*13*) investigated the effect of moisture on the shelf life of wheat flour and established that the moisture has significant effect on crude protein, crude fat, mould growth and insect infestation. Teunou and Fitzpatrick (*14*) determined the effect of temperature and relative humidity on the flour flowability.

---

*Corresponding author; Fax: ++359 32 644 102; E-mail: nimenkov@yahoo.com

A number of models have been suggested in the literature for the dependence between the equilibrium moisture content (EMC) and the water activity (a_W) (*15*). The modified Chung-Pfost, modified Henderson, modified Halsey, modified Oswin and Guggenheim-Anderson-de Boer (GAB) equations, which incorporate the temperature effect, have been adopted as standard equations by the American Society of Agricultural Engineers (ASAE) for the description of sorption isotherms (*16*).

The objectives of this work are: (*i*) to obtain experimental equilibrium sorption isotherms of semi-defatted sesame flour at 10, 25 and 40 °C; (*ii*) to find out a suitable model describing the isotherms; (*iii*) to calculate the monolayer moisture content.

Material and Methods

Material

Commercial sesame flour produced in Bulgaria was used in this study. AOAC (Association of Official Analytical Chemists) standard procedures (*17*) were used for the determination of the proximate chemical composition (% on wet basis): moisture 10.0, crude fat 10.6, protein 57.3, carbohydrate 18.7, and ash 3.4.

Procedure

The EMC of the sesame flour was determined at 10, 25 and 40 °C. Static gravimetric method was applied (*8,18*). For the adsorption process, flour was dehydrated in a desiccator with P_2O_5 at room temperature for 20 days prior to the beginning of the experiment. The desorption isotherms were determined on samples hydrated in a glass jar over distilled water at room temperature to approximately 23 % dry basis (d.b.) moisture content. Samples of (1±0.02) g were weighed in weighing bottles. The weighing bottles were then put in hygrostats with seven saturated salt solutions (LiCl, $MgCl_2$, CH_3COOK, K_2CO_3, NaBr, NaCl, KCl), used to obtain constant a_W (*8,19*). Water activities of the saturated salt solution at several temperatures are presented in Table 1. All salts used were of reagent grade. At high water activities (a_W>0.70), crystalline thymol was placed in the hygrostats to prevent the microbial spoilage of the flour (*8*). The hygrostats were kept in thermostats at (10, 25 and 40±0.2) °C. Samples were weighed in a balance, sensitivity ±0.0001 g, every three days. Equilibrium was reached when three consecutive mass measurements showed a difference of less than 0.001 g. The moisture content of each sample was determined by the oven method and the measurements were done in triplicate.

Table 1. Water activities of saturated salt solutions at several temperatures (*19*)

Salt	t=10 °C	t=25 °C	t=40 °C
		a_W	
LiCl	0.113	0.113	0.112
CH_3COOK	0.234	0.225	0.201
$MgCl_2$	0.335	0.328	0.316
K_2CO_3	0.431	0.432	0.432
NaBr	0.622	0.576	0.532
NaCl	0.757	0.753	0.747
KCl	0.867	0.843	0.823

Data analysis

The description of the sorption isotherms was verified according to the following five models (*8,20*):

Modified Chung-Pfost $$a_W = \exp\left[\frac{-A}{t+B}\exp(-CM)\right] \quad /1/$$

Modified Halsey $$a_W = \exp\left[\frac{-\exp(A+Bt)}{M^C}\right] \quad /2/$$

Modified Oswin $$M = (A+Bt)\left(\frac{a_W}{1-a_W}\right)^C \quad /3/$$

Modified Henderson $$1 - a_W = \exp[(-A(t+B)M^C] \quad /4/$$

GAB $$M = \frac{AB'C'a_W}{(1-B'a_W)(1-B'a_W+B'C'a_W)} \quad /5/$$

$$B' = B\exp\left(\frac{h_1}{RT}\right) \quad /6/$$

$$C' = C\exp\left(\frac{h_2}{RT}\right) \quad /7/$$

where M is the moisture content; a_W is the water activity; A, B, C, h_1 and h_2 are the coefficients; t is the temperature, °C; T is the temperature, K; R is the universal gas constant, J/(mol·K).

A nonlinear least squares regression program was used to fit the five models to the experimental data (all replications). The suitability of the equations was evaluated and compared using the mean relative error expressed in percentage, standard error of moisture and randomness of residuals (*20*):

Mean relative error $$P = \frac{100}{N}\sum\left|\frac{M_i - \hat{M}_i}{M_i}\right| \quad /8/$$

Standard error of moisture $$SEM = \sqrt{\frac{\sum(M_i - \hat{M}_i)^2}{d_f}} \quad /9/$$

Residual $$e = M_i - \hat{M}_i \quad /10/$$

where M_i and $\hat{M}_i$ are EMC values, experimentally observed and predicted by the model, respectively, N is the number of data points, and d_f is the degree of freedom (number of data points minus number of constants in the model).

The monolayer moisture contents (M_e) for each temperature were calculated using the Brunauer-Emmett-Teller (BET) equation with the experimental data for water activities up to 0.45 (*8,21*):

BET $$M = \frac{M_e C a_W}{(1-a_W)(1-a_W+Ca_W)} \quad /11/$$

Results and Discussion

The obtained mean values of EMC based on the triplicate measurements for the respective water activity and temperature are presented in Table 2 for adsorption, and in Table 3 for desorption. In the water activity interval from 0.1 to 0.5, the EMC values decreased with an increase in the temperature at constant a_W. Similar trends for many foods have been reported in the literature (*9*). This trend may be due to a reduction in the total number of active sites for water binding as a result of physical and/or chemical changes in the product induced by temperature (*22*). At increased temperatures water molecules get activated to higher energy levels, causing them to become less stable and break away from the water binding sites of the material, thus decreasing the EMC (*22,23*). At $a_W > 0.5$ the effect of temperature on the EMC is not distinctly marked. The comparison with the published sorption data for sesame seed (*24*) shows that higher sorption capacity of sesame flour may be due to its lower fat content and higher mass exchange area. Fig. 1 gives the experimental data obtained for adsorption and desorption at 25 °C. The sorption isotherms have an S-shape profile (a type II isotherm according to Brunauer's classification) and correspond to multilayer formation. The EMC increased rapidly to 0.2 in region I at low water activities, then slowly in region II, between a_W of 0.2 and 0.8, followed by a steep rise in region III, above a_W=0.8. Region I is represented by the monolayer moisture, which is strongly bound to material. Region II includes multilayer moisture which is under transition to natural properties of free water and is available for chemical reactions. The water in region III is in free state, held in the voids, crevices and capillaries. The hysteresis effect is statistically significant in all experimental points but is most pronounced in the $0.2<a_W<0.8$ region.

Table 2. Equilibrium moisture content (*M*, % d.b.) of sesame flour at several temperatures (*t*) and water activities (a_W) for adsorption

t=10 °C			*t*=25 °C			*t*=40 °C		
a_W	*M**/%	s.d.**	a_W	*M**/%	s.d.**	a_W	*M**/%	s.d.**
0.113	6.24	0.09	0.113	5.33	0.10	0.112	4.64	0.17
0.234	7.26	0.18	0.225	6.36	0.22	0.201	5.62	0.07
0.335	8.83	0.20	0.328	7.16	0.87	0.316	6.15	0.11
0.431	9.29	0.12	0.432	8.96	0.89	0.432	8.17	0.15
0.622	10.85	0.12	0.576	9.32	0.18	0.532	8.51	0.20
0.757	12.87	0.10	0.753	10.36	0.17	0.747	10.23	0.12
0.867	20.30	0.23	0.843	16.17	0.27	0.823	15.94	0.11

*mean of *N*=3 replications; **standard deviation based on *N*=3 replications

Table 3. Equilibrium moisture content (*M*, % d.b.) of sesame flour at several temperatures (*t*) and water activities (a_W) for desorption

t=10 °C			*t*=25 °C			*t*=40 °C		
a_W	*M**/%	s.d.**	a_W	*M**/%	s.d.**	a_W	*M**/%	s.d.**
0.113	6.81	0.06	0.113	5.52	0.09	0.112	4.80	0.14
0.234	9.30	0.11	0.225	8.73	0.06	0.201	6.35	1.17
0.335	10.16	0.16	0.328	8.98	1.48	0.316	6.82	0.09
0.431	10.95	0.12	0.432	9.98	0.18	0.432	9.18	0.32
0.622	12.37	0.18	0.576	10.70	1.00	0.532	10.08	0.11
0.757	14.52	0.11	0.753	12.66	0.12	0.747	13.02	0.09
0.867	21.61	0.24	0.843	16.32	0.20	0.823	15.94	0.13

*mean of *N*=3 replications; **standard deviation based on *N*=3 replications

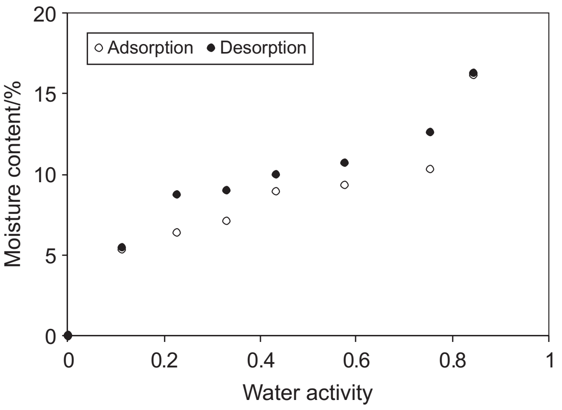

Fig. 1. Comparison of sorption isotherms at 25 °C

The coefficients for the three-parameter models, *P* and *SEM* values are presented in Table 4 for adsorption and Table 5 for desorption. The coefficients for the five-parameter GAB model, *P* and *SEM* values are presented in Table 6. For adsorption and desorption, the *P* and *SEM* values obtained by the GAB models were lower. Fig. 2

Table 4. Coefficients (*A*, *B*, *C*), mean relative error (*P*) and standard error for moisture (*SEM*) of three-parametric models for adsorption

Model	*A*	*B*	*C*	*P*/%	*SEM*
Modified Chung-Pfost	460.11	27.742	0.281	7.76	1.27
Modified Oswin	10.306	–0.0413	0.325	8.70	0.87
Modified Halsey	4.789	–0.0191	2.144	8.13	1.13
Modified Henderson	0.000128	2.071	2.433	13.70	2.08

Table 5. Coefficients (*A*, *B*, *C*), mean relative error (*P*) and standard error for moisture (*SEM*) of three-parametric models for desorption

Model	*A*	*B*	*C*	*P*/%	*SEM*
Modified Chung-Pfost	541.22	37.093	0.256	4.64	0.81
Modified Oswin	11.116	–0.0454	0.296	5.24	0.67
Modified Halsey	5.204	–0.0147	2.293	5.94	0.99
Modified Henderson	0.000221	1.737	2.059	18.25	3.09

Table 6. Coefficients (A, B, C, h_1, h_2), mean relative error (P) and standard error for moisture (SEM) of Guggenheim-Anderson-de Boer (GAB) model

	A	B	1000 C	h_1	h_2	P/%	SEM
Adsorption	5.178	0.280	270.3	2597.9	2065.2	7.59	0.83
Desorption	7.164	0.105	38.8	4623.9	176.6	4.32	0.69

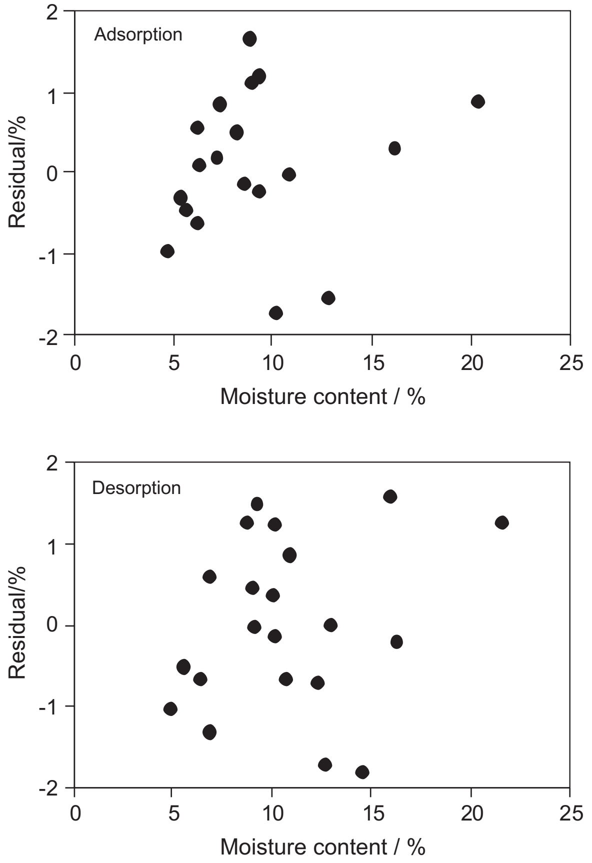

Fig. 2. Distribution of residuals for the Guggenheim-Anderson-de Boer (GAB) model

shows that the distribution of the residuals is random. Therefore, we recommend the GAB model for description of the sesame flour equilibrium isotherms.

The calculated values of monolayer moisture content at the three temperatures are presented in Table 7. The values decreased with the increase in temperature. A similar effect has been reported for many foods (*25*). The temperature dependence of the monolayer moisture has been linked to a reduction in sorption active sites as a result of physicochemical changes induced by the temperature.

Table 7. Brunauer-Emmett-Teller (BET) monolayer moisture content (M_e) of sesame flour at several temperatures (t)

t/°C	Adsorption		Desorption	
	M_e/%	R^2	M_e/%	R^2
10	5.50	0.9884	5.89	0.9842
25	4.90	0.9969	5.37	0.9701
40	3.69	0.9898	5.01	0.9852

Conclusions

The sorption capacity and monolayer moisture content of sesame flour decreased with an increase in temperature at constant water activity. The GAB model is suitable for describing the relationship between the equilibrium moisture content, the water activity, and the temperature for the sesame flour.

Acknowledgements

This study was conducted with the kind support of the National Research Fund, Ministry of Education and Science, Bulgaria.

References

1. M.H. Kang, K.S. Min, S.N. Ryu, J.K. Bang, B.H. Lee, Effects of defatted sesame flour on oxidative stress induced by ethanol-feeding in rats, *J. Kor. Soc. Food Sci. Nutr. 28* (1999) 907–911.

2. F. de Fatima, M. Marques, P.S. Bora, N. Narain, Development of some high-protein conventional foods based of wheat and oilseed, *J. Food Sci. Technol. 37* (2000) 394–399.

3. A.P. Alobo, Effect of sesame seed flour on millet biscuit characteristics, *Plant Food Hum. Nutr. 51* (2001) 195–202.

4. M.K. Egbekun, M.U. Ehieze, Proximate composition and functional properties of fullfat and defatted beniseed (*Sesamum indicum* L.) flour, *Plant Food Hum. Nutr. 51* (1997) 35–41.

5. T.A. El-Adawy, Effect of sesame seed protein supplementation on the nutrition, physical, chemical and sensory properties of wheat flour bread, *Food Chem. 59* (1997) 7–14.

6. S.O.S. Saldivar, J.R. Abril-Dominguez, G. Lopez-Ahumada, R. Ortega-Ramirez, Nutritional evaluation of table bread fortified with defated soybean and sesame meals, *Arch. Latinoam. Nutr. 49* (1999) 260–264.

7. W.A.O. Afolabi, C.R.B. Oguntona, B.B. Fakunmoju, Acceptability and chemical composition of bread from beniseed composite flour, *Nutr. Food Sci. 31* (2002) 310–313.

8. L. Bell, T. Labuza: *Moisture Sorption: Practical Aspects of Isotherm Measurement and Use*, American Association of Cereal Chemists Inc, St. Paul, USA (2000) pp. 33–36.

9. A.H. Al-Muhtaseb, W.A.M. McMinn, T.R.A. Magee, Moisture sorption isotherm characteristics of food products: A review, *Food Bioprod. Process. 80* (2002) 118–129.

10. R.J. Bothast, R.A. Anderson, K. Warner, W.F. Kwolek, Effect of moisture and temperature on microbiological and sensory properties of wheat flour and corn meal during storage, *Cereal Chem. 58* (1981) 309–311.

11. N. Abdullah, A. Nawawi, I. Othman**,** Fungal spoilage of starch based foods in relation to its water activity, *J. Stored Prod. Res. 36* (2000) 47–54.

12. M. Nasir, M.S. Butt, F.M. Anjum, K. Sharif, R. Minhas, Effect of moisture on the shelf life of wheat flour, *Int. J. Agr. Biol. 5* (2003) 458–459.

13. M.S. Butt, M. Nasir, S. Akhtar, K. Sharif, Effect of moisture and packaging on the shelf life of wheat flour, *Internet J. Food Saf. 4* (2004) 1–6.

14. E. Teunou, J.J. Fitzpatrick, Effect of relative humidity and temperature on food powder flowability, *J. Food Eng. 42* (1999) 109–116.

15. C. Van der Berg, S. Bruin: Water Activity and Its Estimation in Food Systems: Theoretical Aspects. In: *Water Activity: Influences on Food Quality*, L.B. Rockland, G.F. Stewart (Eds.), Academic Press, London, UK (1981) pp. 189–200.

16. Moisture relationship of plant-based agricultural products, Standard D245.5, ASAE, St. Joseph, USA (2000).

17. Official Methods of Analysis of AOAC, Washington DC, USA (1990).

18. W. Wolf, W.E.L. Spiess, G. Jung: Standardization of Isotherm Measurements (COST-Project 90 and 90 bis). In: *Properties of Water in Foods in Relation to Quality and Stability*, D. Stimatos, J.L. Multon (Eds.), Martinus Nijhoff, Dordrecht, The Netherlands (1985) pp. 661–679.

19. L. Greenspan, Humidity fixed points of binary saturated aqueous solutions, *J. Res. Nat. Bur. Stand. 81A* (1977) 89–96.

20. C. Chen, R.V. Morey, Comparison of four EMC/ERH equations, *Trans. ASAE*, *32* (1989) 983–990.

21. S. Brunauer, P.H. Emmett, E. Teller, Adsorption of gases in multimolecular layers, *J. Am. Chem. Soc. 60* (1938) 309–319.

22. A.H. Al-Muhtaseb, W.A.M. NcMinn, T.R.A. Magee, Water sorption isotherms of starch powders, Part 1: Mathematical description of experimental data, *J. Food Eng*. *61* (2004) 297–307.

23. K.B. Palipane, R.H. Driscol, Moisture sorption characteristics of inshell macadamia nuts, *J. Food Eng*. *18* (1992) 63–76.

24. N.A. Aviara, O.O. Ajibola, U.O. Dairo, Thermodynamics of moisture sorption in sesame seed, *Biosyst. Eng. 83* (2002) 423–431.

25. N.D. Menkov, H. Paskalev, D. Gelyazkov, M.K. Kerezieva--Rakova, Applying the linear equation of correlation of Brunauer-Emmett-Teller (BET)-monolayer moisture content with temperature, *Nahrung*, *43* (1999) 118–121.