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듀럼밀 성분의 수분흡습 특성

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Moisture Adsorption Characteristics of Durum Wheat Components

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Abstract

Starch, gluten, sludge and water solubles of durum wheat were isolated and their moisture adsorption characteristics were compared at 30℃. The adsorption data were analyzed to find the best-fit sorption isotherm equation for each component. Water solubles showed the highest sorptive capacity among the durum wheat components. Starch showed higher equilibrium moisture content (EMC) than gluten and sludge in the water activity region below 0.9 and 0.75, respectively. In the higher water activity region, however, gluten and sludge showed higher EMC than starch. The Chung-Pfost equation was the best fitting model for the moisture adsorption isotherm of durum starch, while the Oswin equations were for the gluten and the sludge. For the water solubles, the G.A.B. equation showed the highest goodness-of-fit.

Key words: durum wheat, starch, gluten, sludge, water solubles, moisture adsorption, isotherm equations

Introduction

Hygroscopic materials keep reacting with their ambient environment. A dry material gains moisture from the environment (adsorption) while a wet material loses moisture to the environment (desorption). Adsorption is associated with water entering the material and it occurs when the vapor pressure within the material is lower than the vapor pressure in the surrounding air. In contrast, desorption is associated with water moving out of the material. Desorption occurs when the vapor pressure of the material is higher than that of the environment.

A consideration of the sorption phenomena is essential in understanding the drying and storage principles of foods. The sorption process continues until the vapor pressure of a food reaches that of the environment. The relative vapor pressure of a food compared to that of pure water at the temperature of the food has been defined as water activity (Aw). The degree of the interaction of the water with other components in a food system and its contribution to food quality are affected by the activity and the thermodynamic state of water (Taoukis *et al.*, 1988).

A number of theoretical and empirical equations have been derived to describe the shape of the sigmoidal sorption isotherms of foods. The equations are of special interest in many aspects such as prediction and modelling of drying time, shelf-life prediction and formulation of foods. Van den Berg and Bruin (1981), Chirife (1983) and Chirife and Iglesias (1978) have reviewed the sorption isotherm equations of various food materials.

In the present study the moisture adsorption characteristics of the components of durum wheat were compared at 30℃ to find their interaction with water and the results were analyzed to find the best-fit sorption isotherm equation for each component.

Materials and Methods

Durum Wheat

Two varieties of durum wheat; Vic and Ward,

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possessing a strong mixing property (strong gluten-type) and a weak mixing property (weak gluten-type), respectively, grown at the North Central Branch of the North Dakota State University Experiment Station located in Minot, ND, USA, were used for the isolation of their components.

Isolation of Wheat Components

Starch was isolated from durum wheat samples by the procedure of Adkins and Greenwood (1966) to minimize starch damage. Each wheat sample was steeped for 24 hr at 10°C in 0.02 M acetate buffer into which mercuric chloride had been added to 0.01 M level. The softened grain was washed and wet-milled at low speed for the first 2 min and at high speed for another 5 min in a Waring Blender. The wet-milled magma was screened on a 20 mesh sieve and the overs containing bran and pericarp was discarded. The screened magma was screened again on a 100 mesh sieve to separate most non-starch materials. The starch was then recovered from the filtrate by centrifugation at 1,000×g for 5 min and washed four times by resuspension in distilled water. The recovered prime starch was air dried at room temperature for 3 days and passed through a 70 mesh sieve.

Gluten was isolated from ground durum wheat semolina by a hand-washing method (AACC Approved Method 38-10, 1983) with a slight modification of the drying method. The recovered wet gluten was freeze-dried. Freeze-dried gluten was ground using a burr mill (Laboratory Construction Co., Kansas City, MO, USA) and passed through a 60 mesh sieve.

The water soluble and sludge fractions were isolated according to the procedure of D'Appolonia (1968) with some modifications. Durum wheat flour was mixed in a Waring Blender in a ratio of 1 part flour to 2 parts water for 4 minutes at low speed. The slurry was centrifuged with the aid of an I.E.C. perforated basket rotor (20 cm diameter) on an I.E.C. centrifuge (Model K, International Equipment Co., Needham Hts., MA, USA) operated at 3,000 rpm with filter paper (S&S No. 576 custom filter paper, Schleicher & Schuell Inc., Keene, NH, USA). The recovered liquid was centrifuged at 10,000×g for 10 minutes and the supernatant was freeze-dried for the preparation of the water solubles.

The sludge fraction was scraped off and reslurried in distilled water and centrifuged at 10,000×g for 10 minutes. The recovered sludge was freeze-dried and ground in the same manner as was the gluten fraction.

Moisture Adsorption

A static method using a sulfuric acid solution to maintain a constant relative humidity was used to obtain an EMC at 30°C. One pint Mason jars were filled one-third full with various concentrations of sulfuric acid solutions. A support made of 3.18 mm ID copper tubing was welded through the metal cover of the Mason jar, and a stopcock was attached to the outside end of the support. Duplicate pre-dried samples (ca. 1 g) were put into small hexagonal polystyrene weighing dishes (size 50.0 mm × 50.0 mm, Fisher Scientific Co.) and placed over the sulfuric acid solutions in jars. One magnetic stirring bar was put into each jar.

Jars were evacuated so that measurements were made in an atmosphere of water vapor alone and so that the equilibrium could be reached in a short time. Finally, jars were placed in a constant-temperature cabinet held at 30±0.1°C. Sulfuric acid solutions in jars were stirred daily on a magnetic stirrer. Under the above conditions, an equilibration period of 72 hours was sufficient to establish moisture equilibrium.

The vapor pressures of various concentrations of sulfuric acid solutions and the saturation vapor pressure of water were obtained from a reference table (Perry and Green, 1997). Relative humidity in all experiments ranged from 9.7 to 94.3%.

Moisture Determination

Moisture contents of samples were determined by the method of Chung and Pfost (1967a). Samples were dried in a convection oven at 50°C for 24 hours and finally at 130°C for 2 hours.

Analysis of Data

From a preliminary study, seven isotherm equations were selected on the basis of the goodness-of-fit to experimental data. The equations were rearranged to have independent variable water activity (Aw) and dependent variable moisture content (m) as

Table 1. Moisture adsorption equations used for the adsorption data of durum wheat components

Equation*	Reference
$m=A+B\,Aw+C\,Aw^2+D\,Aw^3$	Alam and Shove (1973)
$m=\frac{1}{B}[\ln A-\ln(-\ln Aw)]$	Chung and Pfost (1967b)
$m=\frac{Aw}{A\,Aw^2+B\,Aw+C}$	G.A.B. (Bizot, 1983)
$m=\left[\frac{A}{B-\ln Aw}\right]^{1/2}$	Harkins and Jura (1944)
$m=\left[\frac{-\ln(1-Aw)}{A}\right]^{1/B}$	Henderson (1952)
$m=A\left[\frac{Aw}{1-Aw}\right]^{B}$	Oswin (1946)
$m=A-B\ln(1-Aw)$	Smith (1947)

*m=equilibrium moisture content (g water/g solid)
Aw= water activity
A, B, C, D=constants

listed in Table 1.

Each set of the sorption data was fitted to the seven isotherm equations using the NLIN procedure, a non-linear regression analysis technique, of the Statistical Analysis System (SAS Institute, 1985). Sums of squares of deviations (SSE) were compared to check the goodness-of-fit.

Results and Discussion

Moisture adsorption isotherms of the components of Vic and Ward durum wheats at 30°C are illustrated in Fig. 1 and 2, respectively. Water solubles showed the highest sorptive capacity among the durum wheat components. Starch showed higher EMC than gluten and sludge in the water activity region below 0.9 and 0.75, respectively. In the higher water activity region, however, gluten and sludge showed higher EMC than starch.

The lower sorptive capacity of gluten than starch in low and intermediate water activity regions explains the results of previous researches in which second clear flour and flour of higher protein content have lower EMC than patent flour and flour of lower protein content, respectively (Bailey, 1920), and the starch of bread wheat have a higher sorptive capacity than the gluten (Bushuk and Winkler, 1957; Gur-Arieh *et al.*, 1967).

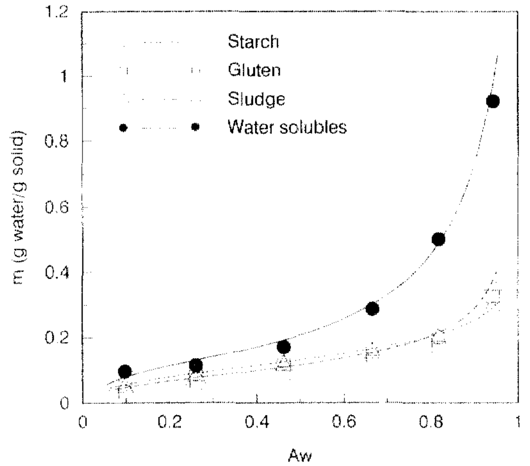

Fig. 1. Moisture adsorption isotherms of Vic durum components at 30°C.

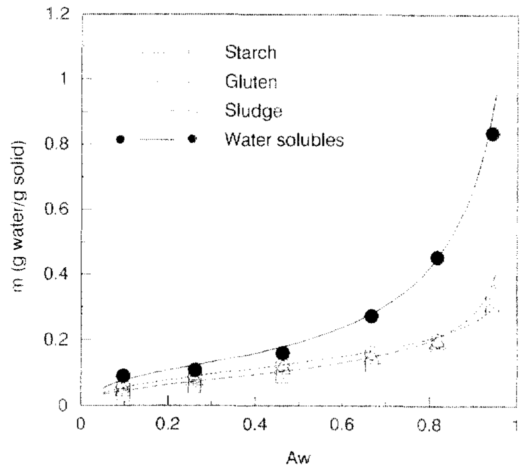

Fig. 2. Moisture adsorption isotherms of Ward durum components at 30°C.

The higher sorptive capacity of gluten at the extremely high water activity region explains the higher water absorption with increasing protein content in bread dough preparation (Bloksma and Bushuk, 1988). Baker *et al.* (1946) have also reported that starch binds water up to 30.1% of its own weight while gluten binds water up to 39.3% of its weight.

The differences in sorptive capacity of the components between Vic and Ward durum are shown in Table 2. There was no difference in mean sorptive capacity of starch between Vic and Ward at 1% level of confidence. The mean sorptive capacities of gluten, sludge and water solubles of Vic

Table 2. Sorptive capacities of durum wheat components (g water/g solid)

Variety	N	Starch	Gluten	Sludge	Water solubles
Vic	12	0.15309	0.13415**	0.15800**	0.34790**
Ward	12	0.15344	0.12873	0.15292	0.32015

**Significance at 0.01 level.

durum wheat possessing a strong mixing property were higher than those of Ward durum possessing a weak mixing property, indicating that the differences in sorptive capacities of these components might also contribute the mixing properties in addition to their absolute amounts (Bloksma and Bushuk, 1988).

Table 3. Constants of the isotherm equations for durum wheat starch

Equation	Parameter	Variety	
		Vic	Ward
Alam	A	0.01951	0.01117
	B	0.45736	0.52336
	C	-0.83626	-0.98454
	D	0.68641	0.78533
Chung-Pfost	A	5.74008	5.39337
	B	16.25314	15.81009
Oswin	A	0.12652	0.12576
	B	0.29606	0.30409
Smith	A	0.06466	0.06248
	B	0.07946	0.08172
G.A.B.	A	-9.01910	-9.18032
	B	11.41797	11.46088
	C	0.58578	0.63334
Harkins-Jura	A	0.01187	0.01156
	B	0.08573	0.07687
Henderson	A	33.13873	29.92022
	B	1.90817	1.85183

Table 4. Sum of squares of deviations (SSE) between observed and predicted equilibrium moisture contents for durum wheat starch

Equation	Variety		Mean[a]
	Vic	Ward	
Alam	0.000326	0.000445	0.000386 C
Chung-Pfost	0.000137	0.000251	0.000194 D
Oswin	0.000267	0.000282	0.000275 D
Smith	0.000826	0.000905	0.000866 A
G.A.B.	0.000290	0.000463	0.000377 C
Harkins-Jura	0.000427	0.000599	0.000513 B
Henderson	0.000717	0.000862	0.000790 A

[a]Means with same letter are not significantly different (α=0.05).

Each set of adsorption data was fitted to seven isotherm equations, and the degrees of the goodness-of-fit were compared using sum of squares of deviations (SSE) between actual and predicted EMC. The estimated parameters and the SSE of each isotherm equation for durum starch are listed in Tables 3 and 4, respectively. The Chung-Pfost equation was the best fit for the adsorption of durum starch showing smallest SSE, although there was no significant difference in SSE with the Oswin equation. The predicted isotherms are plotted in

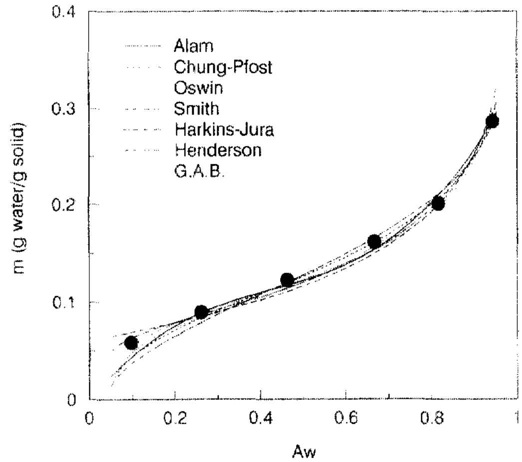

Fig. 3. Predicted isotherms for the moisture adsorption of Vic durum starch at 30°C.

Table 5. Constants of the isotherm equations for durum wheat gluten

Equation	Parameter	Variety	
		Vic	Ward
Alam	A	-0.02890	-0.02923
	B	0.76269	0.73165
	C	-1.80569	-1.69944
	D	1.46961	1.37315
Chung-Pfost	A	2.76457	2.80056
	B	13.10177	13.75448
Oswin	A	0.09142	0.08870
	B	0.44951	0.44256
Smith	A	0.02185	0.02198
	B	0.10090	0.09590
G.A.B.	A	-17.77364	-17.82855
	B	19.00363	19.06051
	C	0.80583	0.95572
Harkins-Jura	A	0.00566	0.00531
	B	-0.00559	-0.00355
Henderson	A	10.83319	11.84993
	B	1.13811	1.16421

Table 6. Sum of squares of deviations (SSE) between observed and predicted equilibrium moisture contents for durum wheat gluten

Equation	Variety		Mean[a]
	Vic	Ward	
Alam	0.001700	0.001756	0.001728 C
Chung-Pfost	0.005768	0.004727	0.005248 A
Oswin	0.000270	0.000302	0.000286 D
Smith	0.001882	0.001625	0.001754 C
G.A.B.	0.000702	0.000830	0.000766 D
Harkins-Jura	0.000595	0.000744	0.000670 D
Henderson	0.003447	0.002994	0.003221 B

[a]Means with same letter are not significantly different (α=0.05).

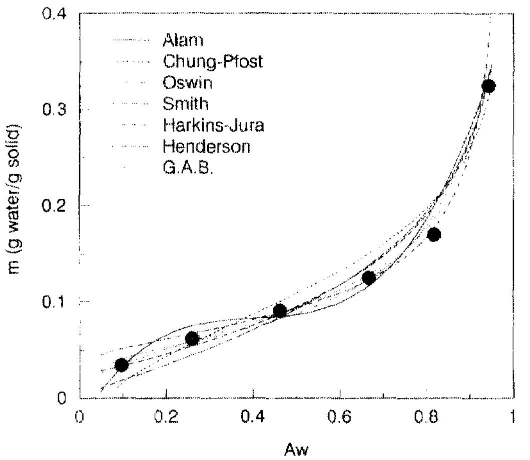

Fig. 4. Predicted isotherms for the moisture adsorption of Vic durum gluten at 30°C.

Table 7. Constants of the isotherm equations for durum wheat sludge

Equation	Parameter	Variety	
		Vic	Ward
Alam	A	-0.02658	-0.02849
	B	0.87467	0.86704
	C	-2.00175	-1.98944
	D	1.58925	1.57674
Chung-Pfost	A	3.29531	3.22231
	B	12.23589	12.49568
Oswin	A	0.11448	0.10998
	B	0.40393	0.40954
Smith	A	0.03825	0.03561
	B	0.10759	0.10540
G.A.B.	A	-13.82558	-14.49540
	B	15.33661	16.00372
	C	0.48748	0.51799
Harkins-Jura	A	0.00865	0.00798
	B	0.00814	0.00597
Henderson	A	11.79844	11.87506
	B	1.31248	1.28985

Fig. 3. Most equations showed greater deviations from observed EMC in the low water activity region than in the high water activity region. The Smith equation generally produced higher EMC than actual EMC, while the Henderson equation produced lower EMC.

The estimated parameters of each isotherm equation for durum gluten are listed in Table 5. The results of Duncan's multiple range test for the SSE (Table 6) showed that the Oswin equation was the best fitting for the adsorption of durum gluten although no differences were found from the Harkins-Jura and the G.A.B. equations. Plots of those equations are shown in Fig. 4.

Results of the analysis of the adsorption data of

Table 8. Sum of squares of deviations (SSE) between observed and predicted equilibrium moisture contents for durum wheat sludge

Equation	Variety		Mean[a]
	Vic	Ward	
Alam	0.001938	0.001968	0.001953 D
Chung-Pfost	0.005478	0.005490	0.005484 A
Oswin	0.000572	0.000571	0.000572 G
Smith	0.002052	0.002093	0.002073 C
G.A.B.	0.001466	0.001429	0.001448 E
Harkins-Jura	0.000817	0.000793	0.000805 F
Henderson	0.004793	0.004643	0.004718 B

[a]Means with same letter are not significantly different (α=0.05).

Table 9. Constants of the isotherm equations for durum wheat water solubles

Equation	Parameter	Variety	
		Vic	Ward
Alam	A	0.00210	0.00587
	B	1.17783	1.04816
	C	-3.39955	-2.98177
	D	3.36310	2.95909
Chung-Pfost	A	2.18530	2.24851
	B	4.37569	4.84466
Oswin	A	0.21172	0.19829
	B	0.52775	0.51615
Smith	A	0.00802	0.01350
	B	0.30541	0.27551
G.A.B.	A	-7.35702	-7.93819
	B	7.38947	8.06582
	C	0.59010	0.57360
Harkins-Jura	A	0.03439	0.02966
	B	-0.01903	-0.01713
Henderson	A	3.11648	3.42935
	B	0.90712	0.93448

Table 10. Sum of squares of deviations (SSE) between observed and predicted equilibrium moisture contents for durum wheat water solubles

Equation	Variety		Mean[a]
	Vic	Ward	
Alam	0.005153	0.007491	0.006322 C
Chung-Pfost	0.076545	0.067515	0.072030 A
Oswin	0.007114	0.005551	0.006332 C
Smith	0.020453	0.015087	0.015976 B
G.A.B.	0.004015	0.002091	0.003053 C
Harkins-Jura	0.019405	0.017664	0.018534 B
Henderson	0.016864	0.015089	0.015976 B

[a]Means with same letter are not significantly different (α=0.05).

sludge of durum wheat samples according to the isotherm equations are shown in Table 7. The Oswin equation was the best fitting equation for the adsorption of the sludge (Table 8).

The estimated parameters of each isotherm equation for water solubles of durum wheat samples are given in Table 9. The results of Duncan's multiple range test indicated that the G.A.B. equation showed the smallest SSE, but there were no significant differences in the goodness-of-fit from the Alam and the Oswin equations (Table 10).

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