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Sorption characteristics of Bulgarian Einkorn flakes

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Abstract. The current scientific research is focused on the Einkorn flakes of Bulgarian origin. The aim is to determine their moisture adsorption and desorption isotherms. The flakes are obtained of milling Einkorn grains with a reconstructed mill roll. The applied pressure was between 200 and 280 tons. The experimental database of equilibrium moisture content (EMC) are determined at three different temperatures - 10°C, 25°C and 40°C through the standard static gravimetric method in the compulsory presence of water activity within the range from 0.1 to 0.9. The obtained adsorption and desorption isotherms have an S-shape profile i.e. they are from the IInd class typical of the majority of food products. The results confirm a regularity that the sorption capacity decreases with increasing of the temperature in the presence of constant water activity which is valid for the both processes. The mathematical three-parametric modified models of Chung-Pfost, Oswin, Halsey and Henderson are evaluated for the description of the obtained sorption data. The monolayer moisture content (MMC) of Einkorn flakes is calculated through the linearization of Brunauer-Emmett-Teller equation. The values varied from 3.26% to 4.92%.

Keywords: sorption isotherms, adsorption, desorption, monolayer moisture content, Einkorn, Triticum monococcum, flakes

1. Introduction

Consumption of cereals as a staple food containing essential bioactive compounds contributes to the good and the normal human body functioning. Modern food production offers neglected regional highnutrition energy products on consumers to meet their needs [1-3].

According to literature reviews, the Triticum monococcum as well-known as Einkorn is a variety of wild wheat species and is considered as one of the most ancient Bulgarian wheat [4-5]. It has a low gluten content compared to ordinary modern wheat that makes it suitable for people with celiac disease [6-7]. Einkorn grain is a good source of micro and macronutrients including vitamins A, B and E, minerals such as zinc, magnesium, manganese, phosphorus, lipids, fructans, and high content of protein, carotenoids and tocopherols as well as some antioxidant compounds [8-10]. Numerous scientific investigations are focused on the creation of new products with therapeutic and prophylactic properties based on the Einkorn flour [11]. Furthermore, Einkorn's products are recommended against the risk of coronary heart disease, cholesterol, chronic and intestinal diseases as well as their prevention [9, 12-13].

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Nakov et al. 2018 are created biscuits based on Einkorn flour. The study is confirmed a high content of polyphenols, carotenoids, antioxidants and beta-glucans of the new product. Based on the results obtained the authors propose to consider the new Einkorn biscuits as functional food [14]. The positive consumer continuity leads to an increase in searching for biscuits and bread based on the whole Einkorn flour on the market. Izambaeva et al. 2016 are studied the Einkorn flour and bread. The presence of antioxidant activity in the investigated products is proven by three analytical methods ABTS, FRAP and CUPRAC, the lutein content of the Einkorn flour is 207.4 μ g.100g⁻¹ and in the Einkorn bread is 226.8 μ g.100g⁻¹[15].

The varied opportunities for Einkorn flour applications give new perspectives to the food industry. For example - the production of flakes - an integral part of modern balanced ready-to-eat breakfast cereals consumed daily by adolescents and adults in countries such as the United States, Germany, France and Bulgaria [16]. Einkorn flour and flakes are a hygroscopic material. The essential sorption characteristic is the equilibrium moisture content (EMC). EMC provides important information on the product which in turn is an important indicator determining storage regimes [17-19].

2. Materials and methods

2.1. Raw Material

Einkorn flakes were delivered by experimental institute located in Parvenets, Bulgaria. They were milled with a reconstructed mill roll [using 2 smooth shafts with a differential difference of 0.05/each roller rotates at different speeds, the difference between them for 1 rpm (rotation per minute) is 1.5 mm to 5 mm]. The applied pressure was between 200 and 280 tons.

2.2. Method

2.2.1. Physicochemical parameters

Moisture, (%) of the product studied - standard method via drying of 5g of flour at 105°C to constant weight, according to AOAC, 1990 [20]; Protein – Direct Kjeldahl method analysis (determination of nitrogen content/ nitrogen determination method) at 25±2°C, according to BDS EN ISO 14431:1978 [21]; Fat content, (%) – Soxhlet method via solvent extraction with petroleum ether at 105±2°C. BDS EN ISO 6997:1984 [22]; Carbohydrates - at 25±2°C, according to BDS EN ISO 7169:1989 [23]; Ash, (%) – The method used for the determination of ash content in flour is according to BDS EN ISO 7646:1982 at 580±2°C [24]; Energy value, kcal/kJ - Regulation EU 1169:2011, Appendices XIV; XV [25]. All physico-chemicals analyzes were carried out in the Food Testing Laboratory at The Institut of Food Preservation and Quality – Plovdiv, Bulgaria.

2.2.2. Sorption characteristics database

2.2.2.1 Preparation of the sample

The adsorption and the desorption isotherms are studied at 10°C, 25°C and 40°C using a gravimetric - static method [26-28]. One part of the samples is dehydrated in a vacuum desiccator over P₂O₅ before beginning the adsorption analysis. Simultaneously, the other part of the samples is hydrated in a desiccator over distilled for the desorption analysis. Eight saturated salt solutions (LiCl, CH₃COOK, MgCl₂, K₂CO₃, MgNO₃, NaBr, NaCl, KCl) providing constant water environment activities are prepared in a glass jar for each temperature and for each process used [29]. The full explanation of the method used is presented in an article of Bogoeva, 2020 [30].

2.2.2.2. Mathematical modelling of the data

According to a literature review four different modified three-parametric models were selected for fitting of the sorption isotherms data, namely modified Chung-Pfost, modified Halsey, modified Oswin, modified Henderson, recommended by American Society of Agricultural Engineers (ASAE) [31]:

Modified Chung-Pfost	$a_w = exp\left[\frac{-A}{t+B}exp(-CM)\right]$	(1)
Modified Halsey	$a_w = exp\left[\frac{-exp(A+Bt)}{M^C}\right]$	(2)
Modified Oswin	$M = (A + Bt) \left(\frac{a_w}{1 - a_w}\right)^C$	(3)
Modified Henderson	$1 - a_w = exp[-A(t+B)M^C]$	(4)

where: M is the average moisture content, %; a_w is the water activity, decimal; A, B and C are coefficients; t is the temperature, $^{\circ}$ C.

The different modified models were fitted through a non-linear least-squares regression program "Statistica". According to Lomauro et al. (1985) one of the basic criteria for determination of the models' equations is the average relative error (P, %). If the value P is < 10% the model is considered fit [32]. Moreover, Chen and Morey's research included two more criteria to compare the suitability of the models, namely the standard error of moisture (SEM) and the randomness of residuals [33]. Nowadays, the P (%), SEM and the randomness of residuals are taken into consideration in order to determine the suitability of the models [29-30]:

The average relative error
$$P = \frac{100}{N} \sum \left| \frac{M_i - \hat{M}_i}{M_i} \right|$$
(5)

 $SEM = \sqrt{\frac{\sum (M_i - \hat{M}_i)^2}{df}}$ (6)

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The standard error of moisture

distribution of residuals
$$e_i = M_i - \widehat{M}_i$$
 (7)

where:

The

 M_i and \hat{M}_i are experimentally observed and predicted by the model value of the equilibrium moisture content:

N is the number of data points;

df is the number of degree of freedom (number of data points minus number of parameters in the model).

According to a literature review the monolayer moisture content (MMC) is calculated through the linearization of Brunauer-Emmett-Teller (BET) equation (Eq. 8) including water activities over 0.45 for the three selected temperatures at 10°C, 25°C and 40°C [34-35]:

$$M = \frac{M_e C a_w}{(1 - a_w)(1 - a_w + C a_w)}$$
(8)

where:

M is the equilibrium moisture content, %;

Me is the MMC, %;

a_w is the water activity, decimal;

C is the coefficient.

To our knowledge, this is the first scientific research considering the determination of sorption characteristics of the Einkorn flakes of Bulgarian origin and its physicochemical parameters and sorption characteristics.

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The food label demonstrates the physicochemical percentage distribution of the basic nutritional substances in the product. Initial analysis of the Einkorn flakes begins with the determination of proteins, carbohydrates, moisture, fat, ash content and energy values. The results are as follows 13.1%; 72.8%; 8.7%; 3.4%; 2.0% and 95.41kcal /399.20kJ, respectively. According to the literature review, our obtained results are approximative to the reported data [3-4, 14].

The initial moisture content of the sample before starting the adsorption process is 5.73%, for the desorption process, it is 17.22%, respectively. The results obtained from the Bulgarian Einkorn flakes for both processes are presented in table 1 and table 2.

	10°C			25°C			40°C		
Sel	a_w	EMC ^a	SDb	a_w	EMC ^a	SDb	a_w	EMC ^a	SD ^b
LiCl	0.113	3.99	0.18	0.113	3.37	0.09	0.112	3.00	0.10
CH ₃ COOK	0.234	4.27	0.07	0.225	4.96	0.16	0.201	4.38	0.07
MgCl ₂	0.335	6.10	0.08	0.328	5.39	0.11	0.316	5.67	0.16
K ₂ CO ₃	0.431	7.31	0.04	0.432	6.60	0.04	0.432	6.59	0.16
MgNO ₃	0.574	8.35	0.17	0.529	7.61	0.08	0.484	7.31	0.09
NaBr	0.622	8.97	0.03	0.576	8.34	0.13	0.532	7.68	0.12
NaCl	0.757	10.98	0.11	0.753	10.57	0.17	0.747	10.31	0.19
KCl	0.868	14.21	0.16	0.843	13.07	0.23	0.823	13.07	0.11

Table 1. Equilibrium moisture content EMC (%) of the Bulgarian Einkorn flakes for adsorption process at different water activities (a_w) and at different temperatures t (°C).

^a Mean values based on three replications

^b Average standard deviation values based on three replications

Table 2. Equilibrium moisture content EMC (%) of the Bulgarian Einkorn flakes for desorption process at different water activities (a_w) and at different temperatures t (°C).

	10°C			25°C			40°C		
Sel	a_w	EMC ^a	SD ^b	a_w	EMC ^a	SD ^b	\mathbf{a}_{w}	EMC ^a	SDb
LiCl	0.113	4.91	0.17	0.113	4.14	0.06	0.112	3.48	0.08
CH ₃ COOK	0.234	5.96	0.15	0.225	5.79	0.06	0.201	5.18	0.03
MgCl ₂	0.335	6.98	0.08	0.328	6.60	0.05	0.316	5.99	0.15
K ₂ CO ₃	0.431	8.30	0.12	0.432	7.80	0.06	0.432	7.81	0.12
MgNO ₃	0.574	9.51	0.09	0.529	8.21	0.05	0.484	8.20	0.04
NaBr	0.622	10.26	0.14	0.576	9.11	0.06	0.532	7.89	0.09
NaCl	0.757	10.89	0.21	0.753	10.83	0.10	0.747	10.46	0.18
KCl	0.868	13.54	0.09	0.843	11.71	0.14	0.823	12.47	0.37

^a Mean values based on three replications

^b Average standard deviation values based on three replications

The obtained values of equilibrium moisture content for both processes are in the range of 3.00% to 14.21%. If we were selected the storage regime under condition relative air humidity from 45% to 55% and temperature from 18°C to 25°C, we could recommend a moisture content from 6.59% to 9.51%. Furthermore, our results confirmed the thesis of many similar conclusions that with the increase of temperature, the EMC decrease in the exclusive presence of constant water activity [29-30, 36-37].

It was performed the graphical comparison between the adsorption and desorption isotherms at 10°C shewed in Figure 1.

These sorption isotherms have an S-shape profile as showed in Figure 1, i.e. they are from the IInd class typical of the majority of food products according to Brunauer's classification [38].



Figure 1. Comparison for both sorption process at 10°C

Coefficients of the three-parameter modified models (A, B, C), the corresponding mean relative error (P, %), standard error of moisture (SEM), residuals distribution and correlation coefficient (R^2) are presented in Table 3 and Table 4 for the two sorption process.

Table 3. Bulgarian Einkorn flakes: model coefficients (*A*, *B*, *C*), mean relative error (P, %), standard error of moisture (SEM), distribution of the residuals and correlation coefficient (\mathbb{R}^2) for adsorption process.

Models	A	В	С	Р	SEM	Residuals	R ²
Chung-Pfost	2067.646	372.129	0.268294	0.85	0.37	Random	0.9906
Oswin	7.451963	-0.004156	0.351038	0.68	0.32	Random	0.9906
Halsey	3.345982	-0.00161	1.861107	7.56	0.97	Non-random	0.9723
Henderson	0.000258	70.06473	1.662762	9.92	0.87	Non-random	0.9541

Table 4. Bulgarian Einkorn flakes: model coefficients (*A*, *B*, *C*), mean relative error (P, %), standard error of moisture (SEM), distribution of the residuals and correlation coefficient (\mathbb{R}^2) for desorption process.

Models	A	В	С	Р	SEM	Residuals	R ²
Chung-Pfost	1186.18	118.2324	0.301697	4.21	0.43	Random	0.9837
Oswin	8.536256	-0.017735	0.266573	5.10	0.49	Random	0.9694
Halsey	4.698046	-0.006131	2.360615	8.04	1.05	Non-random	0.9567
Henderson	0.000103	5.247957	2.421689	19.07	2.54	Non-random	0.6130

Based on the measuring and on the criteria suitability of the models, we could be recommended for satisfactory description of the Einkorn flakes sorption isotherms – the modified Oswin model for both processes. According to Chen and Morey, (1985) it does not exist a uniform model for the description of the sorption isotherms for all food products [33].

Monolayer moisture content (%) at 10°C, 25°C and 40°C for adsorption process are 4.50%, 4.36% and 3.26%, respectively for desorption processes – 4.92%, 3.96% and 3.68%. These results confirm the best condition for the stability of the Einkorn flakes. The values are calculated owing to the linearization of the Brunauer-Emmett-Teller equation and are showed in Figure 2 and Figure 3.



Figure 2. Bulgarian Einkorn flakes: linearization of BET equation for adsorption process.



Figure 3. Bulgarian Einkorn flakes: linearization of BET equation for desorption process.

Based on the obtained results we could continue our experiment with confirmation and determination of the storage regime of Bulgarian Einkorn flakes. We will select the conditions imitated the condition on the store or market – relative air humidity from 44% to 55% and temperature between 18°C and 24°C where the Einkorn flakes will must have the equilibrium moisture content from 6.60% to 9.51%, according to table 1 and table 2. We will compare the duration of storage under MMC and of storage under the condition of EMC as in a study of Durakova et al, 2019 [39].

4. Conclusions

It was determined the equilibrium moisture content (EMC) of the Bulgarian Einkorn flakes (*Triticum monococcum*) for adsorption and desorption process at 10°C, 25°C and 40°C under the condition of different water activity in the range of 0.1% to 0.9%. We were graphical demonstrated that all obtained isotherms have the *S*-shape profile i.e. they are from the IInd class, according to Brunauer's classification.

According to the criteria for the suitability of the modified model we were recommended the modified Oswin model for the description of the obtained sorption data. The monolayer moisture content (MMC) was calculated through the linearization of the Brunauer-Emmett-Teller equation. MMC for the adsorption process is in the range from 3.26% to 4.50% and respectively for the desorption process – from 3.68% to 4.92%.

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