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SORPTION PROPERTIES OF VARIOUS TYPES OF COMMERCIAL WHEAT FLOUR

Aneta Ocieczek

Gdynia Maritime University, ul. Morska 83, 81-225 Gdynia
e-mail: daszenka@am.gdynia.pl

Abstract. The investigated types of wheat flour were characterised by varied chemical composition. The sorption properties were assessed on the basis of monolayer capacity, sorption specific surface, total capacity of capillaries in the area of capillary condensation, the most probable capillary radius and radius of capillaries subject to filling in the first phase of condensation. The initial data were obtained by the static-desiccation method at 20°C and 30°C. The results revealed that the effect of temperature growth on sorption properties of flour is not explicit, although at higher ambient temperature a significant differentiation of sorption properties of individual types of flour is observed, most probably determined by their chemical composition. Sorptivity of wheat flour is correlated to the protein content. On the other hand, the capillary structure is not a feature differentiating individual types of flour.

Keywords: sorption isotherms, surface microstructure, durability, GAB

INTRODUCTION

Flour is a mixture of relatively small (0-150μm) particles of endosperm and particles of the seed coat. The criterion used for assessing the share of particles of the seed coat is the content of mineral compounds (flour type), due to their over 10-fold higher content in the seed coat as compared to the endosperm. Commercial flour of a specified type is a mixture of many machine flours of various compositions and quality, determined by falling number, sedimentation value or water storage capacity (Gąsiorowski 2004, Rothkaehl 2010). The basis for establishing mutual proportions between individual types of machine flours, used for composing the final flour, is the ash content. This parameter is perceived as the most appropriate criterion for assessing flour usefulness for specific purposes, although it does not have a decisive effect on its usable value (Gąsiorowski 2004).

The chemical composition of individual parts of a wheat kernel strongly differs. The central part of the kernel features carbohydrates, represented first of all by starch, and storage proteins (creating gluten), while the amount of enzymes is low. With the increase in the share of external layers of the grain in flour, which is reflected by the growing type of flour, more functional proteins and enzymes are transferred to the flour. The type of flour indicates changing chemical composition and baking properties (Bogaczyński 2010).

In Poland, commercial flours are divided into types, with ash content forming the only classification criterion. Type 450 wheat flour, known as cake flour, is used first of all for baking light cakes, e.g. sponge cakes. Type 550 wheat flour, known as luxury flour, is used for the production of yeast cakes and fried cakes, e.g. drop scones. On the other hand, type 650 wheat flour, known as roll flour, is used for baking rolls and buns. Flours of a higher type are characterised by a higher share of protein, lipids and fibre, and a lower share of carbohydrates, which has a significant effect on their baking properties. Changes in chemical composition can also potentially affect flour sorptivity and, therefore, its shelf-life.

The aim of the study was to compare the characteristics of sorptivity for type 450, 550 and 650 commercial wheat flours, at 20 and 30°C, aimed at assessing the relations between the chemical composition and the stability during storage.

MATERIALS AND METHODS

The examinations involved type 450, 550 and 650 commercial wheat flours, produced under industrial conditions, at the commission of one of the distributors of food products in Poland. The chemical composition of flour is presented in Table 1, on the basis of the data declared by the producer on the product packaging.

Table 1. Basic chemical composition of wheat flour

Parameter	Product type (in grams per 100 grams of the product)		
	450	550	650
Protein	10.10	10.50	11.00
Carbohydrates	74.00	73.00	72.10
of which sugars	0.20	0.50	0.50
Lipids	1.20	1.60	1.70
of which saturated fatty acids	0.30	0.30	0.40
Fibre	2.30	2.20	2.50

Source: on the basis of the producer's declaration on the packaging.

Analytical methods

Determination of water content

Water content was determined by drying samples of about 2 g at 130°C for 1 hour (Krełowska-Kułas 1993). After that time, closed dishes with the samples were placed in a desiccator with $P_2O_{5,}$ until the ambient temperature was reached and then weighed. Water content was calculated in grams per 100 grams of dry matter.

Determination of water activity

Water activity was determined in an AquaLab apparatus (series 3 model TE manufactured by Decagon Devices, Inc., Pullman, WA, USA), with an accuracy of ± 0.003 at 293 K (20°C).

Determination of sorption isotherms

The course of flour isotherms was determined by the static desiccator method (Tyszkiewicz 1987). The principle of the determination of sorption isotherms is based on the determination of equilibrium moisture between the test sample and the atmosphere of a certain relative humidity, controlled by appropriate saturated salt solutions. The scope of research included water activity from 0.07 to 0.98. Temperature of the examination was 20^{o}C and 30^{o}C. The time for establishing equilibrium of the system was 45 days from placing the samples in desiccators. In desiccators with water activity above 0.7 thymol was placed to protect against the development of microorganisms. Individual samples of flour used for the determination of sorption isotherms in the first phase of the experiment were placed in an amount of about 1 g ± 0.1 mg in dishes with a diameter near 35 mm, so as to completely and evenly cover the surface of the layer with a thickness up to 1 mm. Then the dishes were placed in a desiccator containing P_2O_5 as the desiccant, at room temperature for 3 weeks in order to minimise the moisture content (~2%) in the tested flours. After that the masses of the samples were specified and placed in desiccators with appropriate saturated salt solutions. On the basis of the initial weight of the product (determined after 3 weeks of incubation in a desiccator with P_2O_5) and changes in water content, the equilibrium moisture contents were calculated and sorption isotherms were drawn. Measurement of water activity in the samples, after 45 days from placing them in the desiccators, was taken with the AquaLab apparatus (series 3 model TE manufactured by Decagon Devices, Inc., Pullman, WA, USA), with an accuracy of ± 0.003 at 293 K (20^{o}C). All points of empirical sorption isotherms were determined as an average of 9 repetitions.

Sorption properties based on the GAB model and the Kelvin equation

Mathematical description of the empirically-determined sorption isotherms was carried out with the application of the Guggenheim, Anderson and De Boer (GAB) equation (Paderewski 1999).

$$v = \frac{v_m CKa_w}{(1 - Ka_w)(1 - Ka_w + CKa_w)} \tag{1}$$

where:
a_w – water activity;
v – equilibrium water content (g H_2O (100 g d.m.)$^{-1}$);
v_m – water content in the monolayer (g H_2O (100 g d.m.)$^{-1}$);
C – energy constant of Guggenheim;
K – constant correcting properties of multilayer molecules with relation to the liquid phase.

The parameters of the equation were identified from the empirical data. Identification was performed with non-linear regression, using the Monte Carlo algorithm. Minimisation of the sum of squares of remainders was adopted as the objective function (Ocieczek and Kostek 2009).

Knowing the content of water vapour adsorbed at temperature lower than the boiling temperature, and the so-called water settling surface, the adsorbent specific surface was calculated based on the following equation:

$$a_{sp} = \omega \frac{v_m}{M} N \tag{2}$$

where:
a_{sp} – sorption specific area (m^2 (g d.m.)$^{-1}$);
N – Avogadro number ($6.023 \cdot 10^{23}$ molecules mol^{-1});
M – water molecular weight (18 g mol^{-1});
ω – water setting surface ($1.05 \cdot 10^{-19}$ m^2 molecule^{-1}) (Paderewski 1999).

The size and volume of capillaries of the material under test were determined from the isotherms in the capillary condensation area. Calculations were performed from the Kelvin equation (Paderewski 1999):

$$\ln a_w = \frac{2\sigma v}{r_k RT} \tag{3}$$

where:
σ – liquid surface tension at temp. T (N m^{-1});
r_k – capillary radius (nm).

Graphic interpretation of the obtained pairs of numbers (*V-r*), determined as a structural curve, provided the basis to determine the radius of capillaries (*dV/dr-r*) by the method of graphic differentiation of distribution curves. The maxima on the distribution curves corresponded to the most often occurring radii of pores (Świtka 1992).

RESULTS AND DISCUSSION

The isotherms of the flour samples tested were classified as sorption isotherms of type II (Fig. 1-3). According to Limousin *et al.* (2007), sigmoid curves of sorption indicate that the observed phenomenon reflects the formation of polymolecular water layers on the surface of the examined flours. “S”-shaped isotherms, characterised by the occurrence of two points of inflection, are used for the description of the three-degree process of adsorption, in which at least two opposing mechanisms are used (Limousin *et al.* 2007). The first section of the isotherm is shaped with the participation of polar groups of hydrophilic components, characterised by a high binding energy. In the case of wheat flour, those components include starch, proteins and pentosans subject to gradual saturation with water molecules. In the second section of the isotherm, formation of water molecules into so-called bunches can be observed. The third section reflects the process of water accumulation in the intermolecular spaces, which leads to partial swelling of matrix macromolecules (mostly proteins) and, consequently, to a loosening of their structure and exposing additional hydrophilic spots (Arêas and Cassiano 2001, Roman-Gutierrez *et al.* 2002, Saad *et al.* 2009).

The analysis of significance of differences ($p<0.05$) using the t-Student test for paired samples revealed no significant differences in the course of the curves ($t_{450/550} = 0.479$; $t_{450/650} = 0.366$; $t_{550/650} = 0.675$; $t_{crit.} = 2.228$) between adsorption isotherms of individual types of flour determined at 20°C. On the other hand, significant differences were found in the course of curves ($t_{450/550} = 3.103$; $t_{450/650} = 15.485$; $t_{550/650} = 8.822$; $t_{crit.} = 2.228$) between adsorption isotherms of individual types of flour determined at 30°C. Therefore, differentiation of sorption properties of the examined samples can be equated with changes in the conformation of macromolecules (protein and starch) conditioned by the influence of ambient temperature. Most probably, flour particles adsorbing water at lower temperature are more easily subject to structural transformations determined by the presence of water particles characterised by higher mobility and activity.

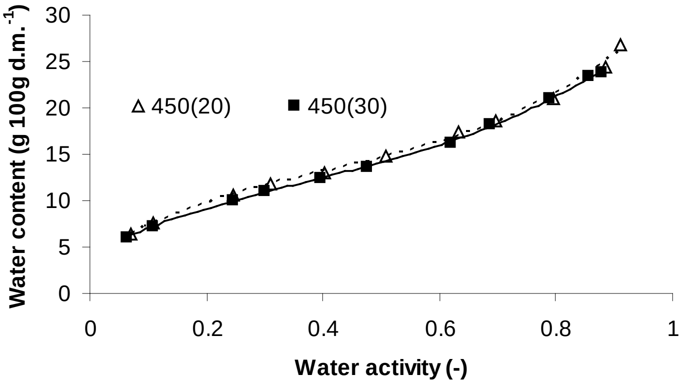

Fig. 1. Sorption isotherms of wheat flour type 450 set at 20°C and 30°C

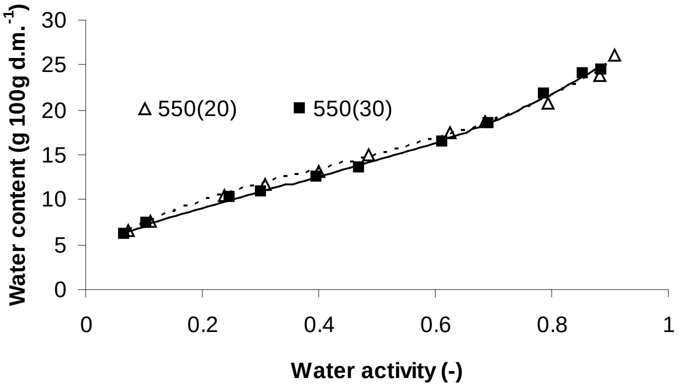

Fig. 2. Sorption isotherms of wheat flour type 550 set at 20°C and 30°C

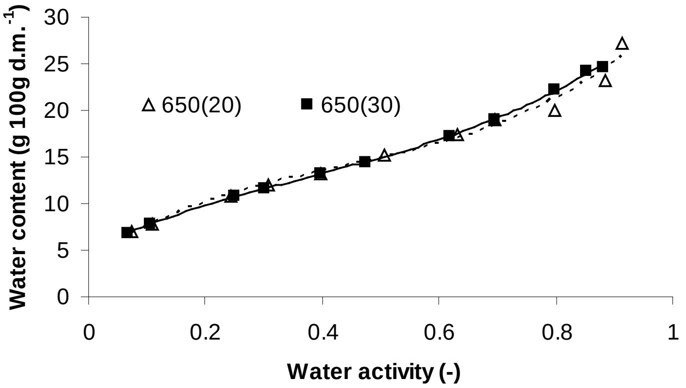

Fig. 3. Sorption isotherms of wheat flour type 650 set at 20°C and 30°C

Moisture adsorption by machine flours grew in the entire range of a_w, and no areas of discontinuity were observed, which indicates that the process of water adsorption from the environment was not accompanied by changes leading to an increase in the degree of macromolecule arrangement. Adsorption capacity of flour is determined by arrangement of polar groups in the external part of flour particles, accessibility of those groups for water, determined by the folding of biopolymers forming chains, crystallisation rate of the matrix and the relative force of water-water and water-macroparticle interaction (Elizalde *et al.* 1996).

The experiment also determined the effect of temperature on equilibrium moisture content corresponding to specific levels of water activity. At 30°C, at a specific level of a_w, the equilibrium moisture content assumed lower values than at 20°C. Increase in temperature moves the equilibrium toward desorption (Brunauer *et al.* 1938). At the higher temperature, samples of flour adsorbed less water, which most probably resulted from the exothermic nature of the sorption process (Erbas *et al.* 2005). However, some publications include data indicating that an increase in temperature does not result in a significant effect on the equilibrium moisture content in wheat flours (Hébrard *et al.* 2003). In order to verify the hypothesis of significant differences between 20°C and 30°C, an analysis of significance of differences ($p<0.05$) was carried out using the t-Student test for paired samples. The results of the analysis indicate that there are significant differences in the course of curves ($t_{450} = 3.441$; $t_{550} = 2.356$; $t_{crit.} = 2.228$) between adsorption isotherms for type 450 and 550 flour determined at 20°C and 30°C. However, the existence of statistically significant differences in the course of curves determined at 20°C and 30°C in the case of flour type 650 was not confirmed ($t_{650} = 0.266$; $t_{crit.} = 2.228$).

It should be also emphasised that with higher environmental water activity, type 550 and 650 flours demonstrated higher hygroscopicity at 30°C than at 20°C. It can be assumed that at higher temperature and relatively higher ambient humidity, macro-molecules (e.g. protein) swelled, as a result of which additional hydrophilic groups were exposed. An argument in favour of this thesis is the presence of a higher protein content in type 550 and 650 flours than in type 450.

On the basis of clearly visible water absorption from the environment at $a_w = 0.75$, initiation of capillary condensation was determined.

Sorption isotherms can be described by various mathematical equations, although the highest importance in food research is currently attributed to the Guggenheim, Anderson and De Boer (GAB) equation (Timmermann 2003). This equation correctly maps the course of sorption isotherms in food systems (in a_w range between 0.00 and 0.90) and it can be used to determine the physical properties of food or energy phenomena accompanying sorption. This equation is recommended by the European Project Group COST 90 (Timmermann 2003, Caurie 2006, Blahovec and Yanniotis 2008).

Table 2. Parameters of the GAB equation describing the sorption properties of wheat flour

Product	Parameters of GAB equation								Error estimate SSE	
	v_m (gH_2O (100g d.m.)$^{-1}$)		a_w		C		K			
	20°C	30°C	20°C	30°C	20°C	30°C	20°C	30°C	20°C	30°C
450	10.67	9.94	0.25	0.24	23.85	28.26	0.66	0.68	1.81	0.44
550	10.91	10.04	0.26	0.24	26.51	27.10	0.64	0.69	1.51	0.96
650	10.94	10.49	0.25	0.23	29.93	31.97	0.62	0.67	14.5	0.49

Source: own research. SSE – the sum of squares for error.

The advantage of the GAB equation is that specific physical values have been assigned to its parameters. At the same time, it should be also emphasised that there are specific limitations to the application of this equation, resulting from its mathematical nature (Ocieczek and Palich 2004). This problem was partially resolved as a result of research carried out by Lewicki (1997). The parameters of the GAB equation are presented in Table 2.

Monolayer capacity is used as an indicator of the availability of polar sites for water vapour, determined not only by the number of components abundant in polar sites (in the case of wheat flour, starch and protein above all), but also their physical status. The structure of starch granules is varied and can additionally be subject to changes as a result of their disaggregation or interactions between them and other flour components, which has been reported in numerous studies (Jurga 2005, Erbas *et al.* 2005, Roman-Gutierrez *et al.* 2002, Raeker *et al.* 1998, Saad *et al.* 2009). The monolayer value helps to specify the amount of water which is strongly adsorbed and it is considered to be the optimal value at which food is more durable (Mathlouthi 2001). The increase of sorption temperature resulted in reduction of the monomolecular layer capacity in all examined flours, which can be interpreted as a result of the exothermic nature of the sorption process.

Water activity corresponding to the monolayer is determined by the degree of water binding with food matrix and it is used for assessing the structure of hydrophilic groups forming a monolayer. Not all hydrophilic groups reveal the same degree of water affinity. The strongest reaction is observed between water particles and –COOH and –OH groups, and a weaker one with =CO, –CHO, and =N– groups (Świtka 1992). Water activities corresponding to the monolayer were characterised by low diversity, and no relationship was found between monolayer capacity and water activity.

The energy constant *C* indicates the difference between enthalpy of vaporisation from the monolayer and enthalpy of vaporisation for liquid adsorbent. The value of the *C* parameter is, according to Lewicki (1997), an indicator of appropriateness for choosing the GAB model to describe empirical data. This parameter, ranging from 5.67 to infinity, indicates the proper choice of the model. This condition was satisfied in all variants of the experiment. The obtained values of *C* were positively correlated with the protein content in the examined flours and the temperature of the experiment. According to Diosady *et al*. (1996), strong adsorbent-adsorbate relations, of an exothermic nature, lead to a reduction of the temperature of the process and to an increase in the value of *C*. On the other hand, research carried out by Iglesias and Chirife (1982) indicates that for most food products one cannot talk about an increasing tendency of *C* constant depending on the decrease of temperature. Pérez-Alonso *et al*. (2006) indicate the possibility of the *C* constant losing its physical sense as a result of compensation of parameters.

The *K* parameter is used for adjusting properties of molecules located in the adsorption monolayer as compared to the liquid phase. The value of the *K* parameter also indicates the scope of application of the GAB equation (Lewicki 1997), and diversifies monomolecular ($K \leq 0.5$) and multilayer adsorption ($K > 0.5$) (Caurie 2006). With increase in temperature, the *K* parameter assumed higher values, which suggest a change in the energy status of water molecules forming a multilayer system. This results in lowering the sorption of the solid body under conditions of higher ambient temperature.

Another area of the comparative analysis was the characteristics of surface microstructure of the particles forming the flours under examination. The assessment of the sorption specific area was made on the basis of previously determined monolayer capacity. The results obtained indicate that the sorption specific area assumed higher values for a flour of a higher type. At the same time, it should be assumed that the factor determining the expansion of the surface was a higher protein content. As follows from research carried out by Hébrard *et al*. (2003), protein content is the key determinant of hydrophilic properties of wheat flours.

An increase in the temperature of the experiment resulted in reducing the sorption specific area, which can be explained by the reduction of the number of sorption active centres as a result of physical and chemical changes in the product, including protein-protein reactions, non-enzymatic browning and conformational changes of proteins (Bizot *et al.* 1985).

The total capacity of capillaries subject to filling after initiating capillary condensation ($a_w = 0.75$) was positively correlated with the sorption specific area, therefore, with the capacity of the monomolecular layer. However, it should be assumed that the total capacity of capillaries should be determined by the tissue characteristics of the wheat grain from which the flour was obtained. The glassiness of the wheat grain is most often identified with high protein content. However, the process of fertilisation, grain drying, storage, or significant changes in moisture content in the grain result in reducing the glassiness with increased mealiness, despite the high protein level. This change is related to a change in the protein matrix structure. The continuity of the protein matrix is, therefore, conditioned by the occurrence of small amounts of free spaces and determines the characteristic light dispersion (Haddad *et al.* 2001). According to numerous authors (Hébrard *et al.* 2003, Erbas *et al.* 2005, Ocieczek 2007), protein content plays a significant role in forming the surface of the monomolecular layer, which does not have to be at the same time directly related to the total capacity of capillaries in mixture particles forming the flour. On the other hand, the research demonstrated the existence of a significant positive correlation ($0.97<r<0.99$) between the total capacity of capillaries and the radius of pores subject to filling at water activity equal to 0.75. It can be assumed that pores of higher radius determine a higher total capacity of capillaries in the product. Products of the microcapillary structure can demonstrate a more expanded monomolecular area, which most probably results from the significant expansion of the sorption area by the area of small capillaries (Świtka 1992).

Table 3. Characteristics of the surface microstructure of wheat flour

Product	Sorption specific area (m^2 g^{-1})		Total capacity of capillaries at a_w = 0.75 (cm^3 100 g d.m.$^{-1}$)		Size of capillaries at a_w = 0.75 (nm)		Most probable capillary radius (nm)	
	20°C	30°C	20°C	30°C	20°C	30°C	20°C	30°C
450	374.8	349.1	19.87	19.93	4.35	4.22	0.21	0.19
550	383.3	352.7	19.96	20.56	4.38	4.34	0.21	0.19
650	384.4	368.7	19.55	20.76	4.27	4.44	0.21	0.20

Source: own research.

The most probable radius of capillaries was determined by subjecting the structural curves to graphical differentiation. The results indicate that the examined flours were characterised by an almost identical structure of capillary distribution. At 30^{o}C, the examination revealed the occurrence of smaller radii of dominating capillaries than at 20^{o}C, which could suggest a more intensive swelling of the protein-starch matrix with an increase in temperature.

CONCLUSIONS

1. Sorption isotherms of type 450, 550 and 650 wheat flours were characterised by a sigmoid shape and continuous course across the entire range of water activity, which indicates a multi-layer process of surface adsorption of water molecules, not accompanied by an increase in the degree of the arrangement of the solid body matrix.
2. No significant differentiation of sorption properties was determined between individual types of wheat flour at 20°C. At temperature increased to 30°C, the sorption properties of individual types of flour were subject to significant differentiation. The increase in temperature resulted in a significant lowering of sorptivity for type 450 and 550 flours, while this reduction was not significant in the case of type 650 flour.
3. Flours characterised by higher protein content revealed a higher level of equilibrium moisture, a higher monolayer capacity and a higher sorption specific area.
4. The capillary structure of the examined types of flours was not significantly diversified.

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WŁAŚCIWOŚCI SORPCYJNE RÓŻNYCH TYPÓW MĄKI PSZENNEJ HANDLOWEJ

Aneta Ocieczek

Katedra Organizacji Usług Turystyczno-Hotelarskich, Akademia Morska w Gdyni
ul. Morska 83, 81-225 Gdynia
e-mail: daszenka@am.gdynia.pl

Streszczenie. Badane typy mąki pszennej charakteryzowały się zróżnicowanym składem chemicznym. Właściwości sorpcyjne oceniano na podstawie: pojemności monowarstwy, powierzchni właściwej sorpcji, ogólnej objętości kapilar w obszarze kondensacji kapilarnej, najbardziej prawdopodobnego promienia kapilar oraz promienia kapilar ulegających wypełnieniu w początkowej fazie zjawiska kondensacji. Dane pierwotne pozyskano metodą statyczno-eksykatorową w temperaturze 20^{o}C i 30^{o}C. Uzyskane wyniki wskazują, że wpływ wzrostu temperatury na właściwości sorpcyjne mąki nie jest jednoznaczny, choć w wyższej temperaturze otoczenia następuje istotne zróżnicowanie właściwości sorpcyjnych poszczególnych typów mąki zdeterminowane najprawdopodobniej ich składem chemicznym. Sorpcyjność mąki pszennej skorelowana jest z zawartością białka. Natomiast struktura kapilarna nie jest cechą różnicującą poszczególne typy mąki.

Słowa kluczowe: izotermy sorpcji, mikrostruktura powierzchni, trwałość, GAB