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Effect of micronization on sorptive properties of wheat cellulose

Introduction

Micronization is an advanced modern method of thermal processing of plant-originated raw materials aimed at modifying the availability of dietary components and at improving the rheological properties of a food product [1]. Such an effect is accomplished as a result of changes induced in the physical structure of chemical compounds.

Plant fibres, consisting mainly of polysaccharides, are capable of absorbing considerable amounts of water and possess a variety of nutritional properties depending on the degree of their solubility. Cereal grains are constituted predominantly by insoluble fibre. The outer layer of wheat is likely to contain up to 50% of plant fibres. Wheat bran contains mainly polysaccharides (47%), i.e. cellulose and hemicellulose, proteins (15%), starch (8%), and minerals (6%). Substances that incrust cellulose do not form chemical compounds with it, but are mechanically-bound to it. Micronization of plant fibres enables altering their functional properties, thus increases their availability for various branches of the food industry.

Aim

The study was aimed at determining differences in the sorptive properties of preparations of wheat cellulose subjected to the micronization process.

Experimental material

The experimental material were preparations of wheat cellulose (WF 200, WF 400 and WF 600) earlier subjected to the micronization process in order to diversify their functional properties (Tab. 1.).

Table 1
Characteristics of the analyzed preparations of wheat cellulose

Preparation	Bulk density, [g/l]	Mean length of fibres, [µm]	Water binding capacity, [%]
WF200	75	250	800
WF400	40	500	1100
WF600	210	80	550

Own elaboration after Lander [2] and producer's declaration

All reagents used for analyses and preparation of saturated solutions of salts were high-purity reagents used in chemical analysis.

Analytical methods

The sorption isotherms of the cellulose preparations examined were plotted with the gravimetric method using saturated solutions of salt [3]. The preparations were stored under conditions of constant relative humidity ($0.07 \le a_w \le 0.98$) and at a constant temperature of 20°C. The equilibrium settling time of the system accounted for 30 days. Thymol was added to hygrostats with water activity over 0.7 in order to protect the preparations against the development of microflora [4–6].

Parameters of the sorption process, including: capacity of the monolayer with corresponding water activity and an energy constant, were determined based on the GAB model of sorption described by the following equation:

$$\frac{\nu}{\nu_m} = \frac{c_e k a_w}{(1 - k a_w)(1 - k a_w + c_e k a_w)} \tag{1}$$

where

ν – adsorption [kg/kg];
ν_m – monolayer capacity [kg/kg];
c_e – energy constant;
k – constant;
a_w – water activity [7–9].

Parameters of the above equation were identified from empirical data. Identification was conducted by means of non-linear regression using a non-gradient algorithm. Such an approach avoids stopping the estimation process by a local minimum. Simultaneously, it enables fitting the GAB model into $a_w - v$ coordinates, in contrast to the standard method applied in this research field. The GAB model was fitted in the least-squares sense. This criterion is the most extensively applied in the statistical analysis.

The specific surface area of an adsorbent was calculated based on the volume of adsorbed water vapor and the so-called water cross-section, using the following equation:

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

where

a_{sp} – specific surface area of sorption [m^2/g];
N – *Avogadro's* number [$6.023 \cdot 10^{23}$ molecules/mol];
M – molecular weight of water [18 g/mol];
ω – water cross-section [$1.05 \cdot 10^{-19}$ m^2/molecule] [7–9].

Results and discussion

A comparison of the sorptive properties of the investigated cellulose preparations was based on the evaluation of adsorption isotherms and a comparative analysis of parameters of adsorption isotherms plotted for the GAB model and their derivatives.

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All isotherms plotted were characterized by the same shape, being typical of biopolymers constituting wheat cellulose (Fig. 1). The course of the sorption isotherms demonstrated inflexion over the range of low water activities. Such isotherms belong to type II isotherms according to *Brunauer's* classification. The same type of isotherms was reported for cellulose preparations obtained from other raw materials [1, 4, 9].

The shape and location of a water vapor sorption isotherm may be different depending on the type of product, its components, affinity to water and active surface. In the discussed cases, it may be speculated that the shape of the locations of the isotherm of water vapor sorption were due to differences in the physical structure of cellulose fibres (the length of the fibres was subject to shortening) evoked by micronization [4, 10].

The preliminary assessment of the sorptive properties was conducted by comparing the reciprocal location of isotherms. The adsorption of moisture by all wheat cellulose preparations analyzed was observed to increase along with increased values of water activity a_w. Having contact with water occurring in the environment, soluble fibres, i.e. pectins and natural resins, are subject to strong hydration, thus forming a viscous and thick gel, whereas the insoluble fibres, i.e. cellulose, some hemicelluloses and lignin, remain in the suspension and while swelling absorbs water up to twenty times their mass [10].

The sorptive properties of the analyzed preparations of wheat cellulose were additionally compared by means of a goodness of fit test of distribution for many populations. The hypothesis on the homogeneity of the sorptive properties of the wheat cellulose preparations differing in the length of fibres was verified at a significance level of $\alpha = 0.005$. Since $\chi^2 = 98.87 > \chi^2_\alpha = 10.59$, the H_0 hypothesis assuming that the examined preparations are homogenous in terms of sorption capacity should be rejected. The preparations differed significantly in their sorptive properties, which was most likely due to the application of the micronization of wheat cellulose which led to the shortening of fibre lengths.

Once the water activity value exceeded $a_w = 0.60$, the effect of absorption turned out to be especially tangible. It may be speculated that above the level of a_w, the phenomenon of capillary condensation occurred in all the analyzed preparations.

The course of sorption isotherms over the entire a_w range enabled determining the GAB equation, by assaying the goodness of its fit (mean square error, *Pearson's* linear correlation coefficient) to empirical data, as well as calculating parameters (v_m, c, a_w, k) of the GAB equation. The results obtained are presented in table 2.

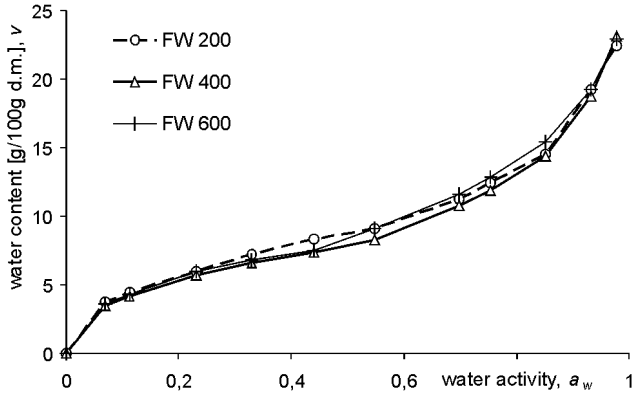

Fig. 1. Sorption isotherms of the investigated cellulose preparations

Table 2

Sorptive properties of cellulose preparations

Product	v_m	a_w	c	k	a_{sp}	RMS	R^2
WF 200	5.9996	0.3317	7.99048	0.7877	164.9967	0.0281	0.9993
WF 400	4.6962	0.1532	49.7157	0.8109	210.7909	0.1116	0.9972
WF 600	5.3203	0.1899	32.7018	0.7837	186.9229	0.0382	0.9991

Source: collation of own data

The highest monolayer capacity was reported for the WF 200 preparation. The v_m capacity is determined not only by the number of particular constituents rich in polar sites, resulting most of all from the origin of preparations, but also by their physical condition. Thus, the evaluation was conducted for the strength of correlation between the length of cellulose fibres and monolayer capacity. The result obtained ($r_{xy} = -0.57$) indicates the occurrence of a strong negative correlation between these values.

In the analyzed cellulose preparations, values of water activity corresponding to monolayer capacity ranged from 0.1532 (WF 400) to 0.3317 (WF 200). The results obtained for the energy constant enable assuming that the investigated process was physical in nature [5, 6]. The v_m value was used to calculate specific surface area of sorption. The results achieved (Tab. 2) demonstrated the greatest specific surface area for WF 400 preparation.

Summary

Sorption isotherms of the analyzed preparations were characterized by a typical sigmoidal shape. Monolayer absorption, multilayer absorption and capillary condensation were observed during absorption of water occurring in the environment by cellulose. The process of micronization did not elicit any changes in the sorption mechanism. However, by leading to the shortening of wheat cellulose fibres (producer's data), it evoked significant differences in the sorptive properties of the analyzed preparations. It should be emphasized, however, that the shortening of cellulose fibres contributes to the increasing capacity of the monolayer and, consequently, the specific surface area of sorption only to a limited extent. The substantial shortening of cellulose fibres does not result in any significant increase in monolayer capacity. In summary, it may be concluded that micronization is likely to elicit significant changes in the sorptive properties of wheat cellulose preparations.

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