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A Comparison of the Sorption Properties of Selected High-fiber Preparations from Cereal Bran, Vegetables, and Root Plants in the Context of Their Functional Properties

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Abstract. This study aimed to compare the sorption properties of four dietary fiber preparations made of cereals (wheat and oats), vegetables (carrot), and root plants (potato). The sorption isotherm was determined using the standard static-desiccator method. The initial experimental results were compared statistically and then subjected to transformation using the Brunauer-Emmett-Teller model (BET model). A new approach to the research problem involved determining the high-fiber preparations' functional properties using sorptive methods. These properties, owing to the preparations' various water-binding capabilities, depended on both their chemical composition and physical structure. Carrot fiber is characterized by significantly higher sorption properties compared to the other tested preparations. The study's experimental results indicate a broad range of possibilities for modifying food properties using dietary fibers derived from various raw materials, differing significantly in their susceptibility to interact with water.

Keywords: Functional properties; Micronization; Quality modeling

1. Introduction

Dietary fiber's importance in everyday diets has spurred great interest among dietitians, food scientists, and food technologists alike in Poland. This interest has manifested in updates to dietary guidelines published by the National Institute of Food and Nutrition (IŻŻ) for the entire Polish population (NCEŻ IŻŻ, 2016) and individual population groups, including elders (NCEŻ IŻŻ, 2018) and children and adolescents (NCEŻ IŻŻ, 2019), and dietary fiber intake is deemed essential in these publications. The problem of an insufficient supply of dietary fiber with diets based primarily on processed foods is crucial, given the populations' health (especially in developed countries), and it poses a severe challenge to the industry, which had been closely observing changes in consumption trends and recommended daily intakes for essential food nutrients. In most developed countries, including Poland, dietary fiber intake fails to meet the recommended level of 20 g per day (Krusińska et al., 2017) and correlates with the incidences of many diet-related diseases (Liu et al., 2015).

Dietary fiber is not a homogenous substance but, rather, represents a group of

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compounds metabolized by the human body that are resistant to enzymatic hydrolysis in the gastrointestinal tract. The functional properties of dietary fiber—including health-promoting properties—are strictly linked to its structure, the contents of its individual components, its origins, and its extraction method. Its key properties include water holding capacity (i.e., the ability to absorb and mechanically retain water), binding cations, binding bile acids and their salts in the intestines, and viscous substances' formation (Cieślik and Topolska, 2002; Makała, 2003).

Comparing different fiber preparations is important due to the increasing variety of these types of fiber preparations on the market. Fiber is not a homogeneous substance, and it may show various functional properties. Dietary fiber is a by-product that can be obtained during the processing of various plant materials. Although it has no nutritional properties, it can be used in its pure form (e.g., to shape such food qualities as satiation) (Ocieczek and Urban-Rajniak, 2018). These properties have been revealed to result from the specific microstructure of fiber particles' surface, which allows the fiber to easily interact with water. In its crude form, as a by-product, it can be used as a sorbent in water purification. In this use, the particles' surface microstructure and their ability to interact with water are also important (Ajayi-Banji et al., 2016).

The important role that dietary fiber plays in human nutrition is due to its ability to bind water in the gastrointestinal tract lumen, positively affecting the final stage of the digestive process. In populations whose fiber intake is high, the incidence of cardiovascular diseases and gastrointestinal cancers is significantly lower. Dietary fiber also exhibits prebiotic and texture-forming properties. It is recommended as a modifier of foods' physical properties (e.g., in confectionery, dairy, fat, and meat products, in food concentrates, and in dietetic foods) (Makała and Ocieczek, 2008; O'Shea et al., 2012; Hemati Matin et al., 2013; Zhuang et al., 2016).

The market offers a broad range of high-fiber preparations and foods with added fiber. These products are produced using parts of cereals, fruits, vegetables, wastes from fruit and vegetable processing, and wastes from the grain and milling industry. Their industrial utility is predicated on the sensory properties of the raw material that the fiber was extracted from, production technology, individual fiber content fractions, degrees of micronization, and health-promoting properties.

Cereal preparations rich in dietary fiber feature fractions of both water-soluble and insoluble fiber. The insoluble components (i.e., cellulose and hemicellulose) are used as dietary supplements, supporting weight loss mainly through their influence on peristalsis stimulation and faster food passage (Anioła, 2019). In turn, the β-glucans of—for example—oats, positively affect carbohydrate metabolism and reduce the risk of gastrointestinal cancer development, particularly colon cancer (Álvarez and Barbut, 2013).

High-fiber preparations produced from vegetables—usually from pomace, a waste product from the vegetable processing industry—have relatively high soluble dietary fiber content, mainly including pectin. Therefore, they can improve carbohydrate and lipid metabolism in the human body. They also positively influence food products' sensory properties (Lara-Espinoza et al., 2018).

Fiber preparations produced from root vegetables—including, for example, potato pulp, which is used as a raw material to produce potato fiber—promote the development of beneficial bacterial strains in the intestines (Anioła, 2019). Moreover, they bind and remove toxins from the alimentary tracts (Anioła, 2019).

The functional properties of all the high-fiber preparations mentioned above differ, including the health-promoting properties. Despite dietary fiber's vast importance and major role in nutrition and industry, few studies have been devoted to assessing and comparing the properties of commercial dietary fiber preparations derived from various raw materials. Therefore, this study aimed to compare the sorption properties of selected preparations of dietary fiber originating from cereals and vegetables, including roots, to identify differences in their abilities to interact with water and in their particle surface microstructures.

2. Methods

This study's experimental material included two dietary fiber preparations produced from cereals—wheat fiber (WF 200; Rettenmaier and Söhne GmbH+Co. KG) and oat fiber (HF; Rettenmaier and Söhne GmbH+Co. KG)—and two preparations produced from vegetables—carrot fiber (BM; AMCO Sp. z o.o.) and potato fiber (KF 200; POTEX, LYCKEBY STARKELSEN FOOD@FIBRE AB). The wheat and potato fiber preparations had the same degree of micronization. All analyzed preparations were commercial samples provided for the study in air-tight unit packages of approximately 100–200 g by their producers.

The scope of the study included: (1) a determination of the preparations' initial water content and activity; (2) a determination of their equilibrium water content and activity after a 60-day sample of incubation in desiccators with controlled relative humidity; (3) a comparison of the position of the tested preparations' sorption isotherms; (4) a comparison of parameters (monolayer capacity-v_m and energy constant-C) of the BET model used for isotherm transformation; and (5) a determination of the specific sorption area (a_{sp}) on the basis of v_m.

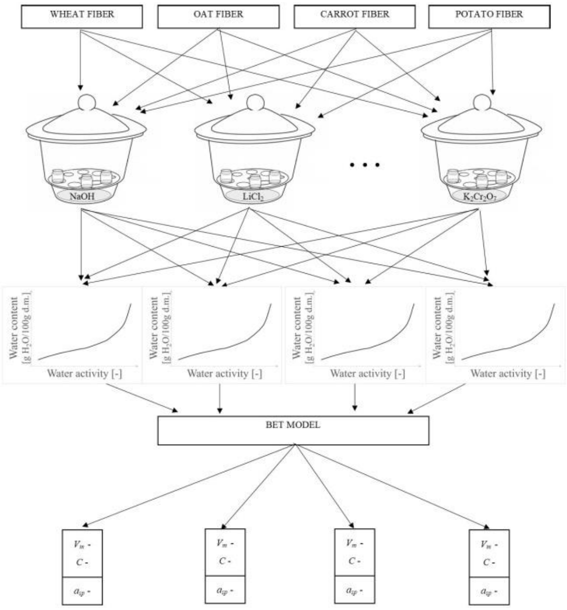

Figure 1 Research procedure

The water content in the examined material was determined by drying it to a solid mass at 105°C. Water activity (a_w) at 20°C was determined with an accuracy of ±0.003 in the AquaLab apparatus (ver. AS4 2,14.0 2017, Series 4TE and 4TEV, manufactured by Decagon Devices, Inc., Pullman, WA, USA). The sorption isotherms were determined using the standard static-desiccator method, which is based on an evaluation of the humidity balance between a tested sample and the atmosphere with a given relative humidity regulated by saturated solutions of the following respective substances: NaOH (0.0698), LiCl (0.1114), CH_3COOK (0.2310), $MgCl_2$ (0.3303), K_2CO_3 (0.4400), $Na_2Cr_2O_7$ (0.5480), KJ (0.6986), NaCl (0.7542), KCl (0.8513), and KNO_3 (0.9320) (Ocieczek and Zięba, 2020). All parameters were tested in three parallel replicates. The significance of differences between the average water content and water activity values in the examined samples was assessed with an analysis of variance (ANOVA) and Tukey's test for posthoc analysis. However, the significance of differences between sorption isotherms was assessed using Student's t-test for bonded pairs. Sorption differences among the tested preparations were determined by identifying and comparing the BET model's parameters:

$$v = \frac{v_m C a_w}{(1-a_w)[1+(C-1)a_w]} \tag{1}$$

where: a_w is the water activity (–); v is the equilibrium water content (g H_2O/100g d.m.); v_m is the water content in the monolayer (g H_2O/100g d.m.); and C is the energy constant (Figura and Teixeira, 2007; Pałacha and Sitkiewicz, 2010).

The BET equation parameters were determined based on empirical data using nonlinear regression with a Monte Carlo algorithm, which prevented inhibition of the estimation process by a local minimum. The residual sums of squares (RSS) of the determined BET equation parameters were estimated using the SolverAid macro command, based on the Hessian matrix. The usability of the models tested for a description of experimental data was evaluated based on the root mean square (RMS) error expressed as a percentage (Ocieczek and Zięba, 2020).

$$RMS = \sqrt{\frac{\sum\left(\frac{v_e - v_o}{v_e}\right)^2}{N}} \cdot 100\% \tag{2}$$

where: N is the number of data; v_e is the experimental equilibrium water content (g H_2O/100 g d.m.); and v_o—predicted equilibrium water content (g H_2O/100 g d.m.).

Given the volume of water vapor adsorbed at a temperature lower than the boiling point and the so-called water cross-section area, the adsorbent's specific surface area was computed based on Equation 3:

$$a_{sp} = \omega \frac{v_m}{M} N \tag{3}$$

where a_{sp} is the specific sorption area (m^2/g); N is the Avogadro's number (6.023×10^{23} molecules/mol); M is the molecular weight of water (18 g/mol); and ω is the water cross-section area (1.05×10^{-19} m^2/molecule) (Figura and Teixeira, 2007; Pałacha and Sitkiewicz, 2010).

3. Results and Discussion

The characteristics of the tested dietary fiber preparations are presented in Table 1. Their chemical composition (including dietary fiber and protein contents) and physical properties (including water-binding capacity and oil absorption) indicated significant differences in their functional properties. All of the preparations featured similar sensory traits (i.e., neutral tastes and aromas and light colors), making them attractive for food producers and allowing for their wide applications to various food products.

Table 1 Characteristic of the dietary fiber preparations analyzed in this study

Preparation	Dietary fiber content (%)	Protein content (%)	Water-binding capacity (g H_2O/g)	Oil absorption (g oil/g d.m.)	Nutrition facts
WF200	> 97	0.4	7.4	4	Wheat fiber
HF	> 96	0.25	4.0	3	Oat fiber
BM	92	3.0	1:18	< 4	Carrot fiber, carrot isolate
KF200	70	5.0	—	—	Potato fiber

Prepared based on producers' declaration. "—" indicates the lack of a producer's declaration

The water contents of the dietary fiber preparations analyzed in this study differed and fit within a broad range of values (Table 2). The same finding held true for their water activity values, as the statistical analysis results showed (Table 2). However, importantly, the preparations' determined water activity range ensured their stability and microbiological safety, as indicated by a_w values lower than 0.6 (Rahman, 2009).

Table 2 Water content and activity of the dietary fiber preparations analyzed in this study

Preparation	Water content (g H_2O/100g d.m.)	SD	Statistical parameters	Water activity (-)	SD	Statistical parameters
WF200	7.4735	± 0.014	F = 14083 NIR = 0.0708	0.564	± 0.001	F = 1068.4 NIR = 0.0041
HF	6.6029	± 0.032		0.548	± 0.003	
BM	10.8201	± 0.019		0.536	± 0.001	
KF200	9.0589	± 0.036		0.495	± 0.001	

The results of ample studies have indicated that practically no microorganism can develop under conditions of water activity lower than 0.6 (Sandle, 2016) because of insufficient water volumes.

The water content in a solid body's matrix most strongly determines water activity. The water-content-to-water-activity ratio indicates that the free water fractions contained in the studied carrot fiber preparation (BM) and potato fiber preparation (KF200) were significantly more strongly bound to the product's matrix than the free water fractions of the wheat fiber preparation (WF200) and oat fiber preparation (HF). Apart from water content, water activity is affected by interactions between a solid body's surface and the water contained in this body, water vapor condensation in capillaries, and finally the concentrations and types of water-soluble substances (Ocieczek and Zięba, 2020).

Presumably, the stronger water-binding with the matrix of BM and KF200 preparations compared to the two other samples was due to their particles' higher affinity to water molecules (a higher number of hydrophilic functional groups). The carrot fiber (BM) and potato fiber (KF200) preparations contained significantly more protein (Table 1) and less crude fiber (Table 1); however, vegetable-derived dietary fiber usually represents the water-soluble fraction, mostly in the form of pectin (Lara-Espinoza et al., 2018). The protein fraction was also showed an exceptionally high affinity to water, as Hebrard et al. (2003) reported earlier when they demonstrated an approximately five-fold higher hydration capacity for wheat flour proteins versus wheat flour starch.

Sorption properties are also affected by the physical parameters of particles exposed to surface interactions with water molecules in a vaporous state. The binding of the free water fraction with the matrices of wheat fiber (WF200) and potato fiber (KF200) differed despite their particles' identified micronization degrees (200). Therefore, speculation suggests that the chemical properties—including various content fractions, revealing different affinities to water due to raw material properties—play a decisive role in sorption properties' changes. Ocieczek and Zięba (2020) drew the same conclusions from their study on powder shampoos' sorption properties using *Sapindus Mukurossi* and *Acacia Concinna* fruits.

Knowledge of the sorption isotherm shape allows for identification of the water-binding mechanism in an individual preparation. The determined isotherms (Figure 2) reflected the process of physical adsorption occurring on porous bodies (Rahman, 2009; Peleg, 2020). This process resulted in the curve shape typical of type II isotherms, according to Brunauer classification. The sigmoid sorption curves indicated the formation of multi-molecular water layers on the preparations' surface. This finding means that the tested powders adsorbed water on their specific hydrophilic surfaces under the influence of increased environmental water vapor content (Pałacha and Sitkiewicz, 2010; Ocieczek and Otremba, 2020).

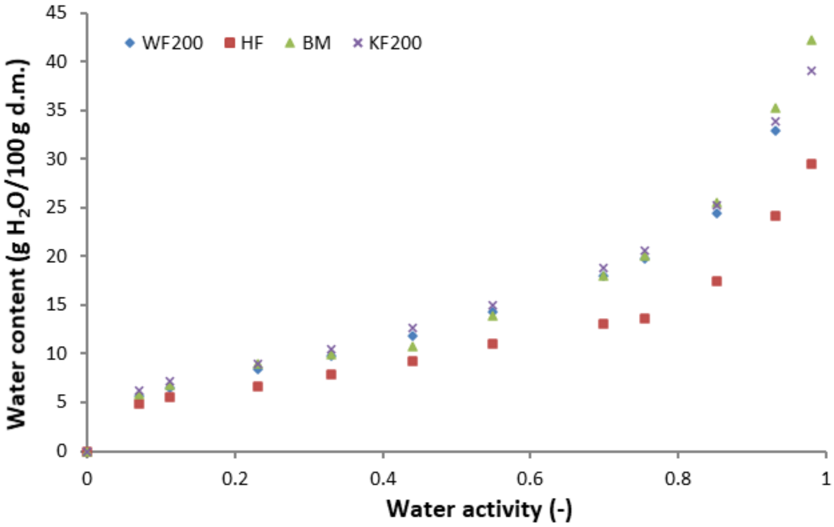

Figure 2 Sorption isotherms of the dietary fiber preparations analyzed in this study

The water vapor adsorption isotherm's shape and position may vary, depending on product type, its components, these components' affinity to water, their interactions, and the active surface (Pałacha and Sitkiewicz, 2010). Water adsorption increased alongside an a_w increase, determined by the presence of macromolecular substances that enable significant water vapor adsorption due to a high number of polar sites. When water activity exceeded $a_w = 0.70$, adsorption became very distinct, revealing the phenomenon of capillary condensation in all tested preparations.

The values of the Student's t-test for bonded pairs were estimated and compared to determine differences in the tested dietary fiber preparations' sorption properties. The results showed that the plotted course of the sorption isotherm for the oat fiber preparation (HF) differed statistically significantly compared to all the other fiber preparations ($t_{critic.} = 2.228$; $t_{HF/BM} = 3.942$; $t_{HF/KF200} = 5.380$; $t_{HF/WF200} = 4.377$). Significant differences were also found between the isotherms of the wheat and potato fiber preparations ($t_{WF200/KF200} = 25.974$). In contrast, no differences were observed between the isotherms plotted for the carrot fiber preparation (BM) and for the wheat fiber (WF200) and potato fiber (KF200) preparations ($t_{BM/WF200} = 1.078$; $t_{BM/KF200} = 0.256$).

Sorption isotherms can be described using mathematical equations. One of the most commonly used equations is the BET equation—even though it describes isotherms well only in a limited range of water activities (up to 0.5). This limitation was considered in the current study's calculations (Table 3).

The BET equation was originally derived for inert gas adsorption on solid surfaces. The BET model is based on a distinction between adsorbate molecules settling on a bare surface in a monolayer (Langmuir adsorption) versus settling on already adsorbed molecules, forming multilayers. The BET theory has been successfully used to determine porous catalysts' and fine powders' specific surface areas, and this use continues to this day. The food literature has widely accepted that foods feature a physical water monolayer. However, reports have also suggested that the BET equation used to analyze water vapor sorption by solid food poses three problems (Peleg, 2020). First, the specific surface area of food powders, calculated using the BET model, was indicated to be independent of their particle sizes. Second, the expected shoulder in foods' enthalpy versus their moisture plot was found to be absent. Third, a significant discrepancy between the surface values was argued to have been calculated from the water vapor and nitrogen sorption isotherms. However, some studies' results have challenged these reservations (Ocieczek and Palich, 2007), whereas Peleg's work (2020) has been widely discussed in recent publications.

Table 3 Parameters of the BET equation

Preparation	*RSS*	*RMS*	v_m	*C*	a_{sp}
WF200	1.023 ± 0.584	5.38	4.422 ± 0.777	1.295 ± 0.156	155.3
HF	0.464 ± 0.393	3.99	4.395 ± 0.646	1.112 ± 0.115	154.4
BM	2.136 ± 0.844	7.84	5.718 ± 1.470	1.067 ± 0.195	200.9
KF200	1.098 ± 0.605	5.21	5.373 ± 0.912	1.183 ± 0.140	188.8

RSS—residual sum of squares; *RMS*—root mean square; v_m—water content in the monolayer (g H_2O/100 g d.m.); *C*—energy constant; and a_{sp}—specific surface of sorption (m^2/g d.m.)

The low values of the *RSS* parameter (Table 3) indicate the BET model's good fit to the experimental data. Research in the field of food technology thus far has assessed and discussed the usefulness of the mathematical model to describe the sorption phenomenon based on root mean squares (Pałacha and Sas, 2016; Ocieczek et al., 2020). These studies have demonstrated that RMS values below 10% indicate a good fit of the model to empirical data in the studied range of water activities. In this context, the sorption properties of the tested fiber preparations expressed by the BET model parameters (v_m and *C*) can be concluded to be reliable and mutually comparable. Moreover, the low error values determined for the BET equation's individual parameters indicate data convergence and high confidence in the estimated values of this model's parameters.

The BET model assumes that the isotherm's sigmoidal course is related to each adsorbed molecule's becoming an adsorption center for the next adsorbate molecule (Pałacha and Sitkiewicz, 2010). Therefore, the model was used to estimate the content of water bound in the monomolecular layer (v_m). The amount of water that allows the monolayer to be filled by the settling of theoretically all hydrophilic functional groups is the optimal amount for preserving dry foods' storage stability (Rahman, 2009). The obtained results indicate that both cereal preparations—that is, wheat fiber (WF200) and oat fiber (HF)—had similar water-binding capacities. In turn, potato fiber (KF200), despite sharing the same degree of micronization with wheat fiber, was characterized by a higher sorption capacity. Carrot fiber (BM) bound the most water in the monomolecular layer (Table 3). The estimated results describing the monolayer's size (Table 3) were compared with the tested preparations' percentage contents of crude fiber and protein (Table 1). This comparison showed that the sorption capacity—expressed through the unfolding of the monolayer—resulted from the percentage contents of the fiber and protein fractions. Fiber's ability to interact with water depends on whether it is dominated by a water-soluble or water-insoluble fraction. Vegetables represent a source of mainly water-soluble fiber, which can bind significant amounts of water and produce highly viscous substances (Skotnicka et al., 2018).

The forces accompanying the development of successive adsorption layers are analogous to the forces that condense vapor into a liquid. The obtained values of the energy constant (C) indicated the physical mechanism of water-binding during the observed phenomenon—regardless of the tested preparation type (Ocieczek et al., 2020). This observation suggests that the surface-phenomenon mechanism was the same in each preparation studied. The differences observed in sorption capacities were related mainly to differences in the tested preparations' chemical compositions (Table 1).

The final element of the present study on the tested preparations' sorption properties was estimating sorption-specific surface areas, a derivative of the monolayer size. The highest value of this parameter was obtained for the carrot fiber preparation (BM), whereas the lowest was obtained for the oat fiber preparation (HF). A generalization of the study results concluded that the fiber preparations obtained from cereals had significantly smaller specific surface areas of sorption compared to the fiber preparations from vegetables and root crops. The oat preparation's very small sorption-specific surface area was probably due to its very low (12–20 times lower) content of protein with strong hydrophilic properties compared to the preparations obtained from carrots and potatoes. Moreover, note that oat is a grain with a naturally high lipid content; therefore, the presence of this hydrophobic fraction in the oat preparation could likely have caused its smaller sorption-specific surface area.

The study results indicate that dietary fiber preparations obtained from various raw materials may differ significantly in their affinities to water, which can be described by the monolayer or sorption-specific surface area. In turn, these parameters enable the prediction of significant differences in their texture-forming properties, which—in addition to modeling foods' physical properties (textures)—may play an important role in promoting satiety after consuming foods that contain these preparations (Skotnicka et al., 2018). Among food ingredients, dietary fiber is the most important factor in inducing satiety. Its water-soluble fractions particularly noteworthy, slowing the stomach's emptying by increasing foods' viscosity (Benelam, 2009) and forcing a feeling of fullness by significantly expanding in the stomach (Lattimer and Haub, 2010; Yuan et al., 2014). In contrast, water-insoluble fiber—such as resistant starch—extends the time needed to chew and swallow food. Although interest in fiber as a food ingredient with high satiating potential appeared relatively recently (in the world for approx. 20 years and in Poland for approx. 10 years), its role in maintaining healthy body weights has been analyzed in many aspects. For instance, it has been investigated as a factor influencing bread's satiating potential (Ocieczek and Urban-Rajniak, 2018), demonstrating that the boosted satiating potential of fiber-enriched bread and stale bread, which is naturally rich in resistant starch, was due to the retrogradation process.

4. Conclusions

Both the raw material types and parameters of the micronization technological process affect the microstructure of a finished dietary fiber preparation and determine its ability to interact with water. Knowledge of the parameters describing the tested dietary fiber preparations' sorption properties will be used to evaluate their capability of causing satiety as an innovative food quality parameter in future research.

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