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# SORPTION PROPERTIES OF A MODIFIED POWDERED COCOA BEVERAGE

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The purpose of the study was to analyse the effect of changes in the composition of raw material and agglomeration on sorption properties of a multi-component food, in the example of a powdered cocoa beverage. The basic composition of the mixtures was 20% of cocoa and 80% of sucrose. A change in raw material composition involved partial or total replacement of sucrose with a mixture of glucose and fructose, or with maltodextrin. Analysis of sorption properties demonstrated variability in the course of isotherms of water vapour sorption for components of the powdered cocoa beverage. Limiting water activity ($a_w$) was determined for the value of 0.529. The conducted analysis detected no significant effect of agglomeration on water content in the tested products. However, a significant change in the raw material composition was demonstrated.

**Keywords:** cocoa, powder, water activity, isotherms

## 1. INTRODUCTION

Water is one of the main components of food, and its presence, along with the condition of food, affects numerous processes and reactions of chemical, physical and biological character. Those reactions determine the quality and stability of food during its transport and storage (Pałacha, 2007).

Water availability and its impact on the course of reactions occurring in foods can be determined on the basis of water activity ($a_w$), defined as the ratio of water vapor pressure above the solution to the water vapor pressure of pure water, under conditions of constant temperature and pressure. Water activity affects the appearance, texture, flavor and taste and product susceptibility to spoilage. Optimal control of water activity, a characteristic of the product, allows for the highest quality, maximum durability and help to minimize the content of additives such as preservatives. Therefore, water activity plays a key role in quality control of food products, pharmaceuticals, cosmetics and other products (Kowalska and Lenart, 2003).

Reliable measurement methods are required for determination of water state in food. The following methods are established: thermoanalytical, based on principles of thermodynamic equilibrium or nuclear magnetic resonance, as well as infrared spectroscopy with Fourier transformation, Raman spectroscopy, dielectric, roentgen-based, light scatter and neutron diffraction (Pałacha, 2007).

However, the most popular and commonly used method for tests of water state in a product is the gravimetric method based on determination of sorption isotherms (Ostrowska-Ligęza and Lenart, 2008). Mathematical analysis of determined curves allows to provide a definition of pure isosteric heat of water adsorption or desorption. The method is relatively simple, but largely depends on repeatability

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*Corresponding author, e-mail: jolanta_kowalska@sggw.pl of environmental conditions and the expertise of the operator performing the measurement. Literature data confirm repeatability of results obtained using this technique, and their consistency with values obtained from thermoanalytical methods. The most commonly used method of determination of water vapour sorption isotherms is the static desiccator method (Pałacha, 2007).

One of the tasks encountered in food engineering is transformation of non-stable products into stable, easy and fast to prepare ones (Kowalska and Lenart, 2005). These include powdered products having long expiry periods, and which are easy to prepare and store. However, to meet particular expectations those products require certain conditions – temperature and relative humidity of the environment. Despite their stability, powdered products may pose some difficulties for manufacturers, re-packaging companies and consumers. Those difficulties are associated with particular characteristics of that type of product: powdery form, segregation during container filling and emptying, and high hygroscopicity in air, which may lead to compromised liquidity and formation of lumps (Schubert and Behrend, 2003). Unfavourable changes taking place in powdery products may be limited or eliminated using the process of agglomeration (Domian, 2002; Kowalska and Lenart, 2002a; Pietsch, 2003; Domian, 2005). Depending on requirements for the final product, it is possible to reduce dust and dusting level, improve liquidity, add rapid solubility (instant) and reduce hygroscopicity. The agglomeration process also causes an increase in porosity, facilitating water penetration and speeding up its solubility (Rambali et al., 2001; Seville et al., 2000; Bocka and Kraas, 2001). However, it is impossible to improve all the features at once (Dz.U. L 197 z 3.8.2000:19; Faure et al., 2001; Kowalska et al., 2009). Increased stability of agglomerate is usually associated with reduced instant properties expected by manufacturers and consumers. Therefore, agglomeration process parameters should be selected in such a way as to obtain a final product meeting as many requirements as possible.

Manufacturers offer a broad spectrum of various products in the form of powders – gelatine desserts, soups, teas, coffees, and cocoa. All those groups of products, except for cocoa, are characterised by easy and fast preparation for consumption. Cocoa powder is broadly used in the confectionery and cake industry. But the most popular are beverages prepared on the basis of cocoa powder. Due to the poor solubility of cocoa powder in liquids, and its characteristic, bitter taste, it is combined with other components (sugar, milk powder), and then it is subject to an engineering process (blending, agglomeration or spray drying) in order to achieve a readily soluble product. The product dissolved in water or milk gives a cocoa beverage (Kunachowicz et al., 1998). Powdered cocoa beverages offered by manufacturers contain approximately 20% of cocoa powder and approximately 80% of sucrose. They also contain lecithin or another emulsifier used to reduce surface tension on the phase interface. Available cocoa beverages are consumed mostly by children. Therefore they are often enriched with vitamins and/or minerals, to supply necessary nutritional elements.

Powdered cocoa in the form of a mixture with other components, pursuant to Directive 2000/36 (PN-A-74859:1994) referred to as “sweetened powdered cocoa”, and commonly referred to as “cocoa beverage” or “instant cocoa”, is characterised by high caloricity (approximately 370-400 kcal/100g) (Domian and Lenart, 2000). Also the glycaemic index of this product is high – over 60. As instant cocoa is consumed mostly by children, development of a product with modified raw material composition is recommended, by total or partial replacement of sucrose with another saccharide, for example maltodextrin or a mixture of glucose and fructose (added in the form of a glucose-fructose syrup). The caloricity and glycaemic index of these sugars are close to values characteristic for sucrose. In the case of glucose and fructose, owing to their more intensive sweet taste it is possible to add a quantity reduced by half compared to sucrose. Therefore the caloricity of the final product is also significantly lower. Maltodextrin is characterised by low sweetness, but similarly to glucose, favours perception of sweet taste and intensifies the taste and aroma of products. Moreover, saccharides are easily absorbable.

Recommended conditions of storage of powdered food, including cocoa beverages, depend on raw material composition and type of packaging. Packages for these products are labelled with recommendations for storage in a dry place with the package tightly closed. Storehouses where cocoa products are stored should have a temperature of 12-18°C and relative humidity below 75% (Kowalska and Lenart, 2002b). Obligatory quality systems require that these parameters be maintained by manufacturers, storage and transporting companies and retailers. However, the conditions are often not kept by consumers, especially after the packaging has been opened. Therefore, physical, chemical and biological changes may occur. Storage at too high temperature may lead to separation of fat on the surface of granulated material, while excessive humidity favours lumping and compromises the solubility of a product, and also constitutes favourable conditions for development of microorganisms.

Water vapour sorption by food products is an important phenomenon in the food processing industry. The water vapour sorption rate depends on the relative humidity and temperature of the environment, porosity of the product and pore size, and on the chemical composition and physical status of individual components (Fennema, 1996).

Knowledge of sorption isotherms is necessary for ensuring optimum conditions of food storage (Brunauer et al., 1938). Humidity of the environment should be kept at a level protecting the material against water vapour adsorption or desorption. Stability of hygroscopic food may also be achieved by appropriate packaging. Well selected package protects food before it is opened and for a consumer it is important that a product maintains its properties during storage at home. Therefore, selection of an appropriate raw material composition, processing and storage conditions is an important factor to guarantee stable product properties throughout the whole shelf life.

The purpose of the study was to analyse the effect of a change in raw material composition and agglomeration on sorption properties of multi-component food, in the example of powdered cocoa beverage.

## 2. MATERIALS AND METHODS

Raw materials for the tests were powdered products with the following trade names: low fat cocoa powder, sucrose (instant), medium-saccharified maltodextrin, glucose and fructose. Cocoa powder and sucrose were bought from a manufacturer of powdered cocoa beverages, maltodextrin was bought from a supplier of additives for the food industry, and glucose and fructose were bought at a retail point, the so-called “organic food” shop. The basic composition of a mixture was 20% of cocoa and 80% of sucrose. 0.5% of lecithin was added to each mixture, per weight of the product. A change in raw material composition consisted in partial or total replacement of sucrose with maltodextrin or a mixture of glucose and fructose (added in equal amounts). Components, mixtures and agglomerates presented in Table 1 were analysed.

Technology of agglomerate production consisted in mixing components in a tank of the STREA 1 agglomerator (Niro-Aeromatic AG) for 2 minutes, following their transformation into fluid state. After thorough mixing a dosing pump was started which supplied water to a spraying nozzle, through which the wetting liquid was sprayed onto agglomerated material. The optimum flow rate of the liquid at which 0.3 kg of powder was uniformly wetted was accepted at the level of $6 \cdot 10^{-3}$ $kg \cdot s^{-1}$. Time of the process was 15 minutes and compressed air pressure in the spraying nozzle was 0.2 MPa. After the end of wetting of powder mixture the agglomerate was dried for 15 minutes at 68°C (Ostrowska-Ligęza and Lenart, 2008). The obtained products were granulometrically analysed on sieves with mesh size ranging from 0.0 mm to > 2.0 mm. Fractions with particle size of 0.2 – 2.0 mm were used for the tests.

Adsorption isotherms were determined using the static desiccator method (Brunauer et al., 1938). $CaCl_2$ with water activity of 0.0 was used as the reference desiccator, and desiccators with saturated solutions of salts with respective water activities: LiCl – 0.113, $CH_3COOK$ – 0.225, $MgCl_2$ – 0.328,

$K_2CO_3$ – 0.432, $Mg(NO_3)_2$ – 0.529, NaCl – 0.753, $(NH_4)_2SO_4$ – 0.810, $BaCl_2$ – 0.903. The samples were weighed 90 days later, and the final equilibrium water content was calculated, *u* [g $H_2O$·(100 g s.s.)$^{-1}$]. Temperature in the thermostat room was 20°C. Measurements were completed for components, their mixtures and water-agglomerated mixtures (agglomerates).

Table 1. Components used for manufacture of powdered cocoa beverage and their mixtures

| No. | Product name | notation | |
|---|---|---|---|
| | Components | powder | agglomerate |
| 1 | Cocoa | 1 | 2 |
| 2 | Sucrose | 3 | 4 |
| 3 | Maltodextrin | 5 | 6 |
| 4 | Glucose | 7 | 8 |
| 5 | Fructose | 9 | 10 |
| | Mixtures | mixture | agglomerate |
| 6 | 20% cocoa + 80% sucrose | 11 | 12 |
| 7 | 20% cocoa + 40% sucrose + 40% glucose and fructose | 13 | 14 |
| 8 | 20% cocoa + 40% sucrose + 40% maltodextrin | 15 | 16 |
| 9 | 20% cocoa + 40% maltodextrin + 40% glucose and fructose | 17 | 18 |
| 10 | 20% cocoa + 80% glucose and fructose | 19 | 20 |

All the raw materials, mixtures and agglomerates were stored in tightly closed plastic containers, in a dark room, at room temperature, until the time of analysis. Analyses were completed in triplicate, and the obtained results were statistically analysed using the Statgraphics Plus application (variance analysis and statistical interpretation were performed for the significance level $\alpha = 0.05$).

## 3. RESULTS

Analysis of the course of water vapour sorption isotherms started from raw materials, both in the form of powders, and agglomerated with water. The flattest course was observed for sucrose (No. 3 & 4) and fructose (No. 9 & 10) (Fig. 1A & B). According to the BET classification, the shape of those curves may be qualified as type III, characteristic for crystalline objects (van den Berg, 1985; Rizvi, 1995). It was demonstrated that sucrose is the least susceptible to environmental water and only above the water activity of 0.8 was a very intensive increase in water content observed in that material. This tendency was demonstrated both for powder and agglomerate. In the case of fructose, an intensive increase in water content was observed above $a_w = 0.43$. Also final water contents for both components are interesting. Maximum absorbed water in the environment with $a_w = 0.903$ by sucrose was determined at the level of approximately 36 g H2O·(100 g s.s.)$^{-1}$ and this value is lower by 30% compared to the corresponding value for fructose. At activity of 0.81 fructose becomes liquefied, which may indicate saturation of the solution. The curve corresponding to water content changes in relation to aw was more horizontal.

Other analysed materials showed a sigmoid course of water vapour sorption isotherms (Fig. 1A & B). According to the BET model they may be classified as type II, characteristic for the majority of food products (Rizvi, 1995). Analysis of water vapour sorption isotherms made it possible to determine a limit of water sorption by tested products at $a_w = 0.529$. Tested components showed a different course of curves above and below the value accepted as the limiting one. A change in water content in relation to aw for glucose is notable. Glucose (No. 7 & 8) is a sugar, and according to accepted assumptions it should give a shape similar to that for sucrose and fructose. However, the shape of the isotherm obtained for glucose is not flat. A tendency for increased water content following agglomeration was observed for tested materials, compared to powders of the same raw material composition. An opposite relation was shown only by glucose. Up to water activity of 0.529 water content in agglomerated glucose was lower compared to the mixture by approximately 20 to 50%. Above the activity of 0.529 glucose powder showed a tendency for equilibrium. The tested powder was a disintegrated crystalline form, which could affect the behaviour of that component at aw over 0.5.

A significant effect of cocoa powder agglomeration was demonstrated below $a_w = 0.529$ (Fig. 1A & B). Those differences were from over 50% at $a_w = 0.113$ to approximately 12% at $a_w = 0.529$. Further increase in water activity had no effect on differences in the course of isotherms for cocoa powder and its agglomerate. Up to the limiting value of water activity (0.529) maltodextrin powder and its agglomerate showed the highest water content compared to other components. However, statistical analysis showed no effect of maltodextrin agglomeration on water content.

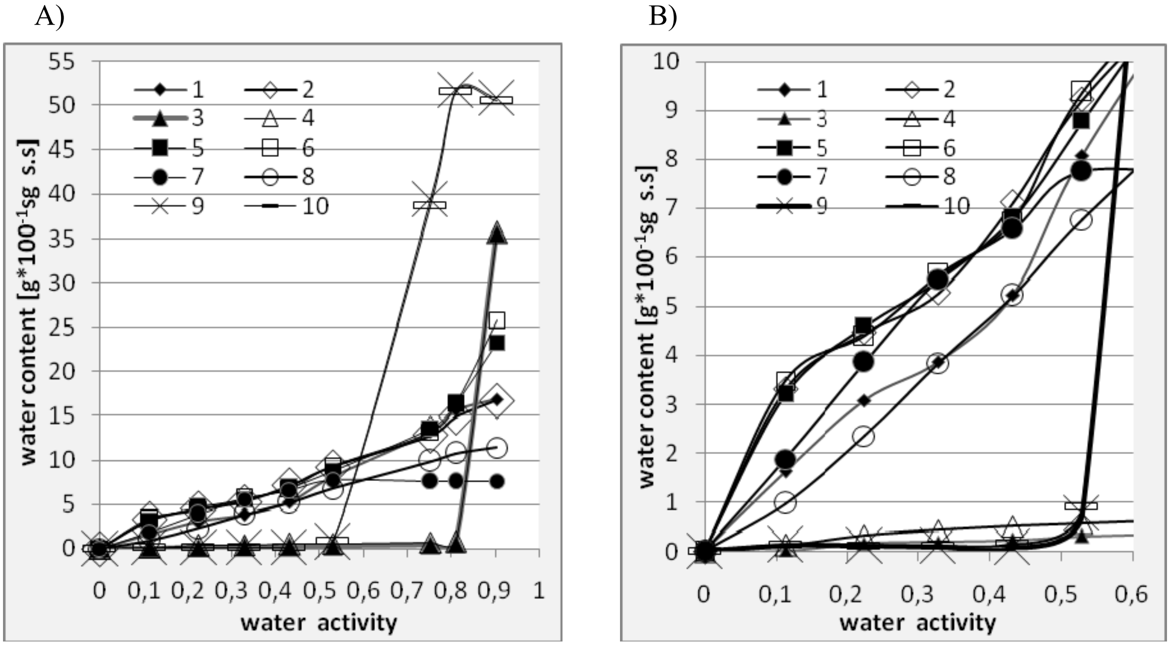

Fig. 1. Water vapour sorption isotherms for components of the powdered cocoa beverage (notation as in Table 1). Water content range: A – 0-55; B – 0-10

Analysis of the course of sorption isotherms of components is important for possible assessment of an effect of raw material composition change on water sorption abilities shown by their mixtures. A sample containing 20% of cocoa and 80% of sucrose was accepted as the basic composition (No. 11 – Table 1) (Fig. 2). The composition is comparable to powdered cocoa beverages available on the market. Dominating sucrose content determined the course and shape of the isotherm for that mixture.

As in the case of components, also for mixtures a limit water activity of 0.529 was accepted. The lowest water adsorption compared to other mixtures was shown by the basic mixture, and shape of the isotherm was flat. Above the limit water activity a significant increase in absorbed water was observed, but it was still the lowest value compared to other products.

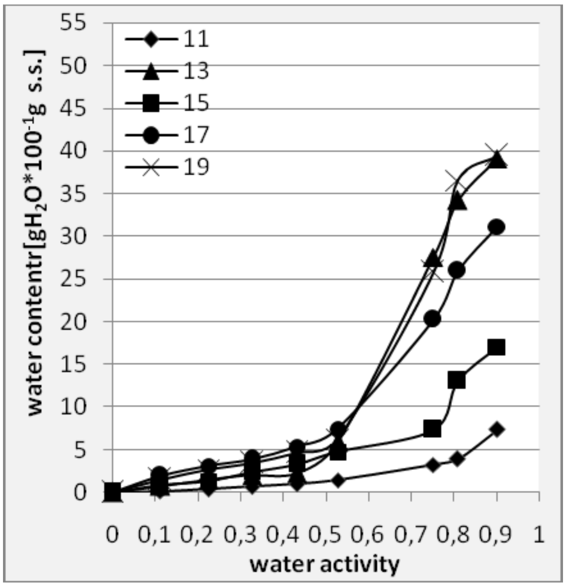

Fig. 2. Effect of change in raw material composition on course of water vapour sorption isotherms for a mixture of powdered cocoa beverage (notation as in Table 1)

Partial or total replacement of sucrose with a mixture of glucose and fructose and/or maltodextrin had a significant effect on the course of sorption isotherms. Mixtures containing 20% of cocoa, 40% of sucrose, 40% of glucose and fructose (No. 13 – Tab. 1) and 20% of cocoa, 80% of glucose and fructose (No. 19) showed the highest water content at activity over 0.529, and sorption isotherms of both those products had a very similar course (Fig. 2). That is associated with water adsorption properties of fructose. A strong effect of that compound on the course of curves and a significant increase in the amount of adsorbed water are visible in the case of mixtures. The final water content for those mixtures is over 5 times higher than that of the basic mixture. Partial replacement of sucrose with maltodextrin (No. 15) resulted in 4.5-fold increase in the amount of adsorbed water for values below $a_w = 0.529$, and over 5-fold increase above that value. Analysis of mixtures showed the most dominating effect of addition of fructose. Presence of that compound in the composition was reflected by very high amounts of absorbed water, especially for values over $a_w = 0.529$. Glucose and maltodextrin sorption isotherms whose water content lower by approximately 50% had no significant effect on the course of curves.

Manufacturers offer powdered cocoa beverages as a soluble instant products, obtained by spray drying and agglomeration. An attempt was made to assess the effect of the agglomeration process on sorption properties of tested products, considering a change in their raw material composition.

Analysis of sorption isotherms of agglomerated products showed a higher effect of a raw material composition change compared to the effect of the agglomeration process itself (Fig. 3).

A significant difference in the amount of water absorbed by agglomerates was observed below and above the value $a_w = 0.529$, which was considered the limiting value. But after the limiting value is exceeded, a very intensive increase in water content was observed for the agglomerate containing glucose and fructose, and they were values approximately 3-fold higher compared to those obtained for the agglomerate containing 40% maltodextrin (No. 16).

The lowest water content for the whole range of water activity, as in the case of mixtures, was obtained for the agglomerate of the basic composition (Fig. 4A). Partial replacement of sucrose with glucose and fructose (No. 14 – Tab. 1) or maltodextrin (No. 16) caused a significant increase in water content compared to the product with the basic composition. Below $a_w = 0.529$ agglomerated mixtures containing 20% of cocoa + 40% of sucrose + 40% of glucose and fructose, and 20% of cocoa + 40% of sucrose + 40% of maltodextrin had isotherms with a similar course, showing similar amounts of absorbed water at particular activities (Fig. 4B).

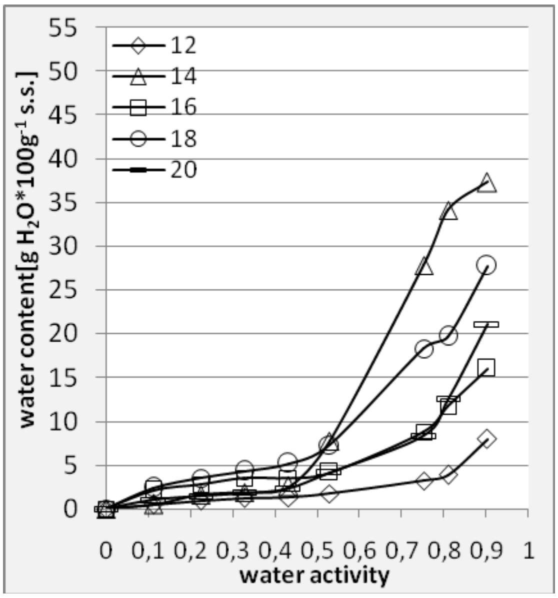

Fig. 3. Effect of a change in raw material composition on the course of water vapour isotherms for agglomerated powdered cocoa beverage (notation as in Table 1)

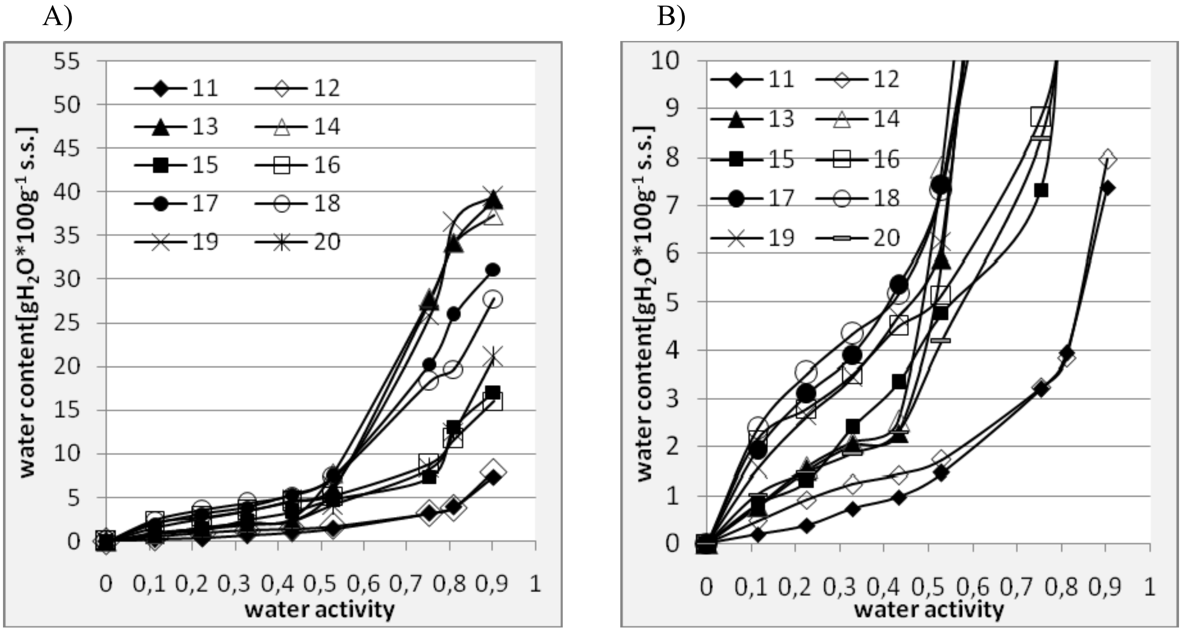

Fig. 4. Effect of a change in raw material composition and agglomeration on the course of water vapour isotherms for the powdered cocoa beverage (notation as in Table 1). Water content range: A – 0-55; B – 0-10

Total replacement of sucrose with glucose and fructose and maltodextrin (No. 18) caused an increase in water content compared to the agglomerate of the basic composition. Below $a_w = 0.529$ those differences were on average twice as high, and above the limit value over 5-fold higher.

The carried out analyses of the effect of agglomeration on the course of water vapour isotherms demonstrated a statistically significant effect of the process only on samples of basic composition, below $a_w = 0.529$ (Fig. 4B). Similarly, the course of curves for that product above the activity accepted as limiting showed no effect of agglomeration on the amount of water absorbed by the mixture and respective agglomerate (Fig. 4A). Analysis of the course of sorption curves for remaining agglomerates also showed no statistically significant effect of the process on the amount of water absorbed by mixtures and agglomerates with the same raw material composition.

## 4. DISCUSSION

Sorption properties of powders are among the most important parameters affecting quality of a product, its suitability for food production and storage stability (Gabas et al., 2007). A material's ability to absorb water is determined by its chemical composition and structure. Sorption isotherms, reflecting the relationship between water content in a product and water activity, constitute a source of numerous valuable data regarding water status in the material. Knowledge of the course of sorption isotherms allows, among other things, determination of the character of factors influencing food spoilage, determination of water content and activity optimal for a material, and design of both food processing processes and composition of a complex matrix of food products. Sorption isotherms also allow theoretical interpretation of physical phenomena occurring at the interface of a product (Gondek and Lewicki, 2005; Sinija and Mishra, 2008).

Various models are used to describe the course of sorption curves, including BET, GAB, and Lewicki's. The shape of isotherms has most often been determined using the classification suggested by Brunauer, Emmett and Teller (BET). However, the BET model describes the course of isotherms for limited ranges of water activity ($a_w$). Pałacha determined water status in a model matrix and in a matrix of apples, using various models (Pałacha, 2007). Based on studies the author found out that GAB and Lewicki's models provide the best description of the course of sorption isotherms. However, considering the significance of constant values and comparing values of mean square error, he chose the GAB model for the description of curves.

Analyses completed for powdered cocoa beverages demonstrated that the majority of tested products were characterised by a sorption curve which could be classified as type II, according to the BET classification. Sucrose and fructose showed a flat course of sorption curves at low and medium $a_w$ values, and a significant increase in water content at high $a_w$, which justifies their classification as type III, according to the BET classification. The course of sorption isotherm curves obtained for the model matrix and the matrix of dried apples was also determined by Pałacha using types provided in the BET model. He confirmed that for the majority of food products, sorption isotherms may be classified as type II, sigmoid, and products containing high quantities of sugar as type III, flat shape, at low and medium values of $a_w$.

Modification of raw material composition had a significant effect on the course of water vapour sorption isotherms for the studied mixtures. Partial or total replacement of sucrose with glucose and fructose caused an increase in water content, especially at water activity of over 0.5. Intermediate (compared to other components) water contents were obtained for maltodextrin. Similar relationships were also obtained for mixtures containing that raw material.

Gondek and Jakubczyk studied, among other things, the effect of addition of maltodextrin on the course of sorption isotherms of apple powder (Gondek and Jakubczyk, 2009). They found that the isotherm of dried apples containing maltodextrin, starting at the water activity of 0.225, was significantly different from other ones. Addition of 15% maltodextrin caused a shift of the isotherm towards lower water content values for practically all its course. That means that at the same water activity of the environment, water content in a maltodextrin-containing product was lower.

Similar relationships were observed by Gabas et al., who added maltodextrin and gum arabic to pineapple pomace and determined isosteric sorption heat (Gabas et al., 2007). The authors demonstrated that the powder without addition of maltodextrin was characterised by a higher number of polar sites, which resulted in higher absorption of water vapour. At water activity of 0.907 maltodextrin-containing dried pineapple absorbed almost half of the water volume absorbed by the material without any additions.

Powdered products have a structure of dispersed systems of high practical significance. A qualitative characterisation of powders usually involves aspects associated with their reconstitution in a liquid. The purpose of increasing particle size by agglomeration is to improve selected properties, mostly physical, of studied products. Properties of solid matter particles, including bulk density, flow rate, dosability, and avoiding segregation of components or dust formation, was of crucial importance.

It is impossible to improve all qualitative properties at once. Increase in mechanical stability (structure) of an agglomerate is usually associated with reduced instant properties, which are desirable in the case of powdered products. In the case of production of stable agglomerates, primary particles have to be brought into mutual contact, which is usually achieved using external forces, and then particles have to be bound by stable forces – stronger than any disrupting ones. The process in which particles are exposed to strong external forces allows formation of stable, easily turned over and dosed products in the form of smooth, dense granules. However, the instant properties of those products are usually unsatisfactory, due to low porosity of granules and high forces binding primary particles.

Domian tested the effect of various types of agglomeration on physical properties of model mixtures of food products (Domian, 2005). An effect of agglomeration of both pneumatically and mechanically generated substrate was found on improvement of flow rate and reconstitution ability of powders, and on reduction of dust formation. She also demonstrated an effect of raw material composition of agglomerated model mixtures on their physical properties, mostly the ability of reconstitution in liquid.

A process of agglomeration of pneumatically generated substrate was introduced in order to improve instant properties of the tested cocoa beverages. The effect of the process on sorption properties of the analysed products was studied. No significant effect of agglomeration on the shape of sorption curves of products with the same raw material composition was found. However, a statistically significant effect of altered composition of agglomerated products on water vapour isotherm sorption was demonstrated.

## 5. CONCLUSIONS

- Obtained isotherms of water vapour sorption demonstrated the shape classified as type II, according to the BET classification, except curves for sucrose and fructose, which may be classified as type III.
- Analysis of sorption properties showed differentiation of the course of water vapour isotherms for components of powdered cocoa beverage. Below $a_w = 0.529$ the lowest water content was obtained for sucrose and fructose. Those components, above the respective values of 0.81 and 0.43, yielded the highest water contents compared to other components.
- A change in quantity and type of components of powdered cocoa beverage had a significant effect on tested sorption properties. Partial or total replacement of sucrose with other components caused an increase in water content, especially for water activity of over 0.529.
- Agglomeration had a significant effect on increased water content for the product of the basic composition (20% of cocoa + 80% of sucrose). No significant effect of the process was observed for other samples, considering the course of isotherms of agglomerates compared to mixtures having the same composition. A change in raw material composition had a stronger effect on water content compared to the agglomeration process.
- The greatest effect on the course of water vapour isotherms was observed for fructose. Presence of that compound in a mixture or agglomerate caused a significant increase in water content. Based on the conducted analysis of the course of sorption curves for tested components and their mixtures, the value $a_w = 0.529$ was recognised as the limiting one. Some significant differences in the course of isotherms for tested samples were observed below and above it.

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## SYMBOLS

| | |
|---|---|
| $a_w$ | water activity |
| $u$ | water content, g $H_2O\cdot(100\ \text{g s.s.})^{-1}$ |

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