Sorption characteristics of fondant candies based on tagatose

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Abstract

Introduction. Studies which established the sorption characteristics of sucrose-based fondant candies and fondant candies based on the combination of tagatose with fructose were conducted.

Materials and methods. Fondant candies made on the basis of sucrose and tagatose with the addition of fructose in the ratio of 9:1 were investigated on a weight vacuum adsorption device with spring quartz scales using gravimetric method.

Results and discussion. The sorption curves of both samples of the studied candies were identical in nature and belonged to isotherms of type III, which is characteristic of microporous adsorbents with low energy of adsorbent-adsorbate interaction. The sorption curves were sigmoid, with a well-developed hysteresis, which indicates a completely irreversible process of dehydration. After the desorption process, a small part of the adsorbed moisture remains in the samples on sucrose and tagatose with fructose (0.55 and 0.37%, respectively).

The coefficients of determination of both BET and Friendlich models used for the analysis were in the range of 0.85–0.97, which indicates their suitability for describing the isotherms of the studied products. Analysis of the constant characterizing the adsorption energy showed that in the sample based on tagatose and fructose adsorption was 18% higher than in the control sample.

The amount of adsorbed moisture in the studied samples of candies at low values of a_w had a slight difference, while in the area of capillary moisture ($a_w = 1.0$) this value differed significantly: the amount of adsorbed moisture in the control sample of fondant candies on sucrose was 0.6498 cm^3/g, while in the sample based on tagatose and fructose it was 1.5499 cm^3/g. The amount of strongly bound moisture in the sample based on tagatose and fructose was greater than in the control sample and was 8.33%, while in the control sample only 5.24%.

Conclusions. The sorption isotherms allow to predict the behavior of candy samples during their storage at different values of relative humidity. The obtained data can be used for rational selection of packaging material and packaging method to extend the shelf life of developed fondant candies based on tagatose.

Introduction

Fondant candies are a complex heterogeneous system, which tries to balance and minimize energy, the reduction of which is possible during the transition of the system to a state of crystallization that may occur due to intensive desorption of products causing an increase in part of solids and size of crystals, and this is reflected in deterioration quality of finished products in general (Bund et al., 2010; Hartel et al, 2011; Ivanov et al, 2021).

The previous research aimed to develop fondant candies that would focus on the requirements of modern consumers, namely, having high sensorial characteristics, and low calories and glycemic value.

To achieve this goal, it was proposed to use the innovative sugar tagatose. However, due to the low hygroscopicity of this sugar, it is advisable to make confectionery in combination with a hygroscopic component. Thus, the possibility of making marmalade from orange with complete replacement of sucrose by a combination of tagatose with oligofructose was proposed (Rubio-Arraez et al.,2015). With an increase of tagatose in the recipe of the product, samples of marmalade were characterized by the phenomenon of intense moisture loss during storage. With increasing dosage of hygroscopic oligofructose in the recipe of marmalade with tagatose, the intensity of moisture removal was significantly slowed down. Therefore, it can be assumed that in the development of products which are prone to hardening during storage, based on tagatose, it is desirable to add additional hygroscopic component to the recipe, including hygroscopic monosaccharide fructose.

To expand the range of fondant candies, a product recipe was developed in which sucrose was completely replaced with a combination of low-glycemic and low-calorie tagatose in combination with hygroscopic low-glycemic monosaccharide fructose in a ratio of 9:1.

When developing new types of confectionery, especially those where the main component of the recipe is replaced, in particular the replacement of white crystal sugar with other sugars, in addition to texture and sensory properties, the stability of the product during storage and selection of a rational packaging method must be taken into account. The relationship between moisture content and water activity or relative humidity can be described using the moisture sorption isotherm (Andrade et al., 2011). Information on the behavior of moisture in products is a necessary condition for the choice of packaging material and packaging method, as well as to establish the warranty period for the preservation of their quality characteristics (Chetana et al., 2004; Kuzmyk et al., 2021; Sokolenko et al., 2019). For classic fondant candies based on white crystal sugar it was established that desorption processes and their negative impact on product quality are the dominant factor during their storage (Ozcan et al., 2019), and for developed fondant candies based on tagatose with the addition of fructose study of sorption-desorption properties to predict the behavior during storage is quite an urgent task.

The aim of the study was to determine the relationship between the equilibrium moisture content in fondant candies, which were made according to the classic recipe based on sucrose and using a combination of tagatose sugar and fructose in a ratio of 9:1. Analysis of changes in the hygroscopicity of the studied product provided a basis for the study of phase transitions in the product depending on its carbohydrate composition and moisture content.

In all cases, the suitability of several mathematical models for describing the sorption isotherms of a multicomponent food product with a high content of mono- and disaccharides was evaluated and those offering the most accurate description were identified.

Identification of the sorption model, which describes the change in water content in the product as dependent on water activity in the widest possible range, can be useful for predicting changes in product quality during storage and to extend its shelf life, and therefore to optimize production and improve product quality.

Materials and methods

Materials

Fondant candies made by boiling sucrose-based fondant syrup (control sample) and completely replacing sucrose with a combination of tagatose and fructose sugars in a ratio of 9:1, followed by cooling and whipping to obtain a fine crystalline fondant mass, which was cast in polymeric forms in the laboratory.

Sorption properties of candies

Determination of the sorption properties of the studied samples was carried out by gravimetric method using a sorption vacuum device with spring quartz scales. (Sylchuk et al., 2021).

Data analysis

To select the model that best describes the sorption isotherms of sucrose fondant candy (control sample) and a sample based on tagatose with fructose, two popular models (BET and Freindlich) are considered. It should be noted that they have a theoretical basis (based on the theory of adsorption), and their parameters have a physical meaning.

Table 1

Equations to describe sorption isotherms

Title of the equation	Equation	Nomenclature
BET	$a = \dfrac{a_m \cdot K \cdot P/P_s}{\left(1-\frac{P}{P_s}\right)\{1+(K-1)\cdot\frac{P}{P_s}\}}$	a – the amount of adsorbed moisture a_m - monolayer capacity K – equilibrium constant of polyslayer adsorption P/P_s - relative equilibrium water vapor pressure
Freundlich	$G = K \cdot P^{\frac{1}{n}}$	G - the amount of adsorbed moisture P – equilibrium water vapor pressure K i n – empirical constants

Results and discussion

Sorption properties of fondant candies

Isotherms of adsorption-desorption of water by samples of fondant candies based on sucrose (control sample) and a combination of tagatose and fructose in the ratio 9:1 are shown in Figure 1 in such coordinates: the amount of adsorbed water – its activity, which is directly related to the relative equilibrium vapor pressure $a_w = P/Ps$.

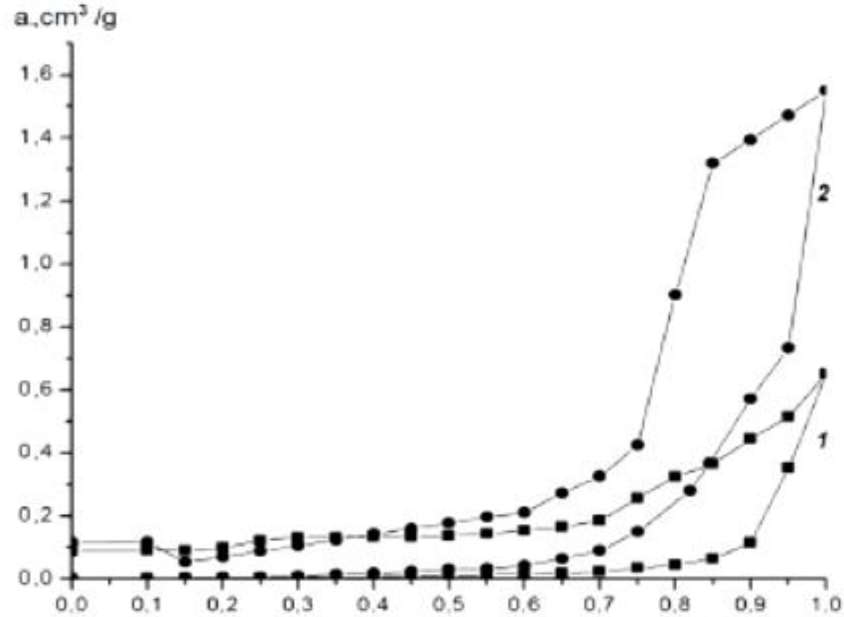

Figure 1. Isotherms of adsorption-desorption of water by samples of fondant candies

Products with high content of sugar such as caramel (Hadjikinova et al., 2003, Netramai et al., 2011) and fruit powders (Rodriguez-Bernal et al., 2015), usually have a J-shaped isotherm or isotherm of type III (Bell et al., 2000). The sorption isotherms of the tested samples also correspond to the characteristic form of food products with high sugar content, which adsorbs a small amount of water at low values of water activity. At the bottom value of a_w the slope of the curve is smaller, while with increasing a_w the slope increases rapidly, which can be explained by the increase in hygroscopicity of the studied sugars (Dorokhovich, 2013).

Sorption isotherms for both samples of fondant candies were identical. In the interval of a_w = 0–0.7 the sorption and desorption curves were located close to each other, and in the interval of a_w = 0.75–1.0 there was a hysteresis loop.

The hysteresis loop indicates that during the sorption in this period there was a pseudo-phase transition associated with changes in the crystal structure of fondant candies. There is also a noticeable difference in the height of the isotherms, which depends on the amount of adsorbed moisture. Thus, the isotherm of the control sample was much lower compared to the isotherm of fondant candies based on tagatose and fructose. The increase in the water absorption capacity of the candy sample based on the combination of tagatose and fructose can be explained by the presence of different sugars with different sorption properties in the recipes of candies. The sorption properties of the used sugars were studied and it was inserted that the rate of sorption of water vapor increases significantly for sucrose and tagatose by

P/Ps 0.8; and for fructose at lower pressure values 0.62, which indicates higher hygroscopicity of this sugar (Dorokhovich, 2013; Han, 2021). Due to the different rate of sorption, sugars are able to attach different amounts of moisture at the maximum possible adsorbent pressure (P/Ps =1.0). Among the used sugars, sucrose has the lowest content, while fructose has the highest content of moisture, and tagatose has an intermediate value, which is consistent with obtained data for candies using these sugars.

The influence of prescription components on the formation of sorption properties of the finished product is also evidenced by the results of previous studies. The sorption properties of caramel made from polyols (isomaltitol and sorbitol) were considered. The sample based on isomaltitol had a lower water absorption capacity than the sample on sorbitol due to the greater hydrophobicity of this sugar substitute (Hadjikinova et al., 2011).

Analysis of sorption models

The rate of chemical reactions in food is highly dependent on the water content. It is assumed that the maximum amount of moisture at which most reactions have a low rate, corresponds to the water content in the monolayer. The monolayer can be determined using theoretical models, where it is one of the constants of the equation describing the physical properties of the material. Two-parameter BET and Freundlich models were considered in the search for the best fit of the sorption isotherm model to the experimental data. The BET model (Figure 2) was used for a_w range from 0.02 to 0.25.

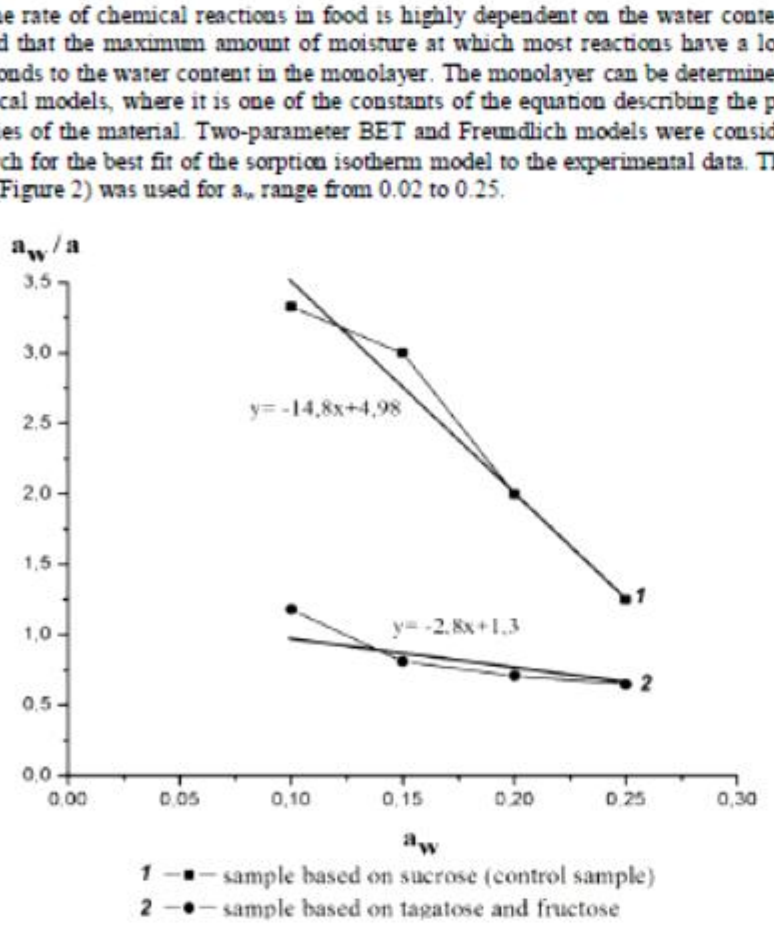

Figure 2. Graphic representation of the BET equation for samples of fondant candies

At higher values of a_w, the research results did not form a straight line, which made it impossible to calculate the parameters of the equation, because in parallel with the adsorption in this zone there is dissolution of the crystal structure. Similar results were demonstrated in the selection of the BET model for powdered sugar. According to the presented graph, it was noted that this equation was valid in the range a_w from 0.0 to 0.3 (Clément et al., 2018). In practice, to describe the phenomena of adsorption in such complex systems, it is more appropriate to use the empirical Freindlich equation, which is well valid for medium humidity. The division of adsorption curves into 3 zones is more clearly observed, and in each zone the experimental results are quite accurately described by straight lines (Figure 3).

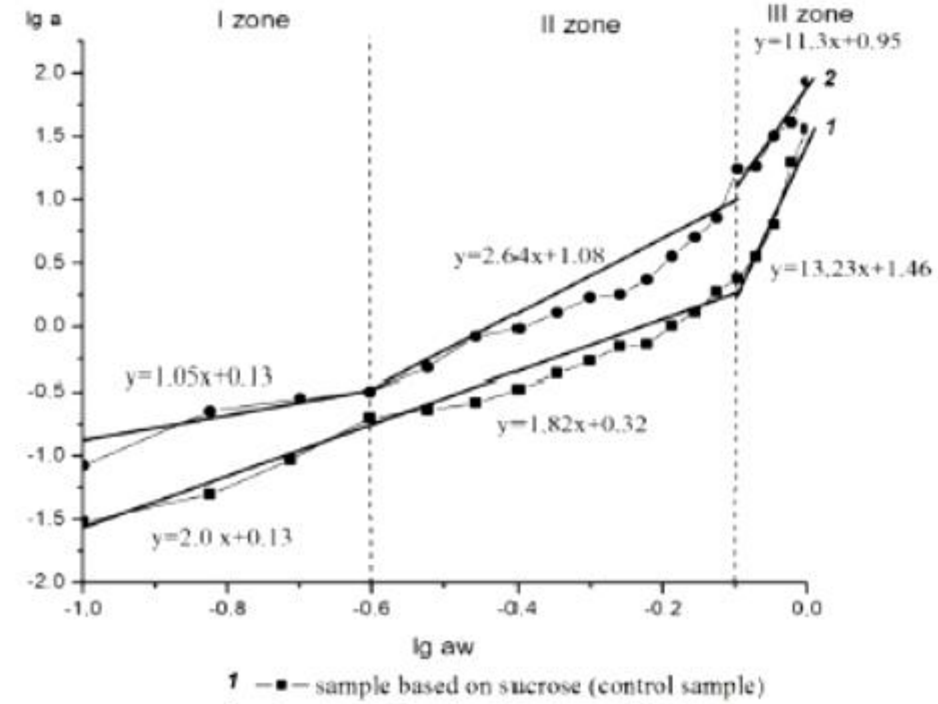

Figure 3. Graphic representation of the Friendlich equation

Table 2 shows the calculated model parameters and the coefficient of determination R^2.

The values of the capacity of monolayer of the adsorbed moisture obtained using the BET model were significantly lower for sucrose-based fondant candies compared to the sample on tagatose and fructose, which can be explained by the presence of hygroscopic fructose, which begins to absorb moisture at lower values than sucrose. It was explored that the water content in the monolayer tended to increase with decreasing sucrose content in the studied samples of candied orange (Witczak, T. et al., 2016). Regarding the analysis of the constant K, which characterizes the energy of adsorption, in the sample based on tagatose and fructose it was 18% higher than in the control sample.

Table 2

Parameters of BET and Friendlich models for samples of fondant candies

Model	Parameter	Sample	
		Candies based on sucrose	Candies based on a combination of tagatose and fructose
BET	a_m	0.0682	0.2500
	K	0.2660	0.3250
	R^2	0.969	0.841
Friendlich	n	0.4230	0.5211
	K	2.9806	9.6095
	R^2	0.939	0.925

The studied sorption curves were signoid, with a well-developed hysteresis and can be divided into three zones: the first zone, corresponding to a_w <0.25, which refers to the adsorption of monomolecular film of water; the second zone corresponding to the adsorption of additional layers over this monolayer at a_w = 0.26–0.75 and the third zone for a_w> 0.76 corresponds to the condensation of water in the pores of the sample with subsequent dissolution of the soluble material (Mathlouthi, 2001).

Since there were conditionally identified three zones on the sorption isotherm, the moisture content of the adsorption zones was determined to characterize the amount of water adsorbed by the samples. The results are shown in Table 3.

Table 3

Amount of adsorbed moisture by the investigated samples of fondant candies

Sample of candies	Amount of adsorbed moisture, cm^3/g					
	First zone a_w = 0.02–0.25		Second zone a_w = 0.26–0.75		Third zone a_w = 0.76–1.0	
	Sorption	Desorption	Sorption	Desorption	Sorption	Desorption
Control sample	0.0005–0.0036	0.1226–0.0874	0.0040–0.0340	0.2549–0.1229	0.0341–0.6498	0.6498–0.2552
Sample based on tagatose and fructose	0.0005–0.0057	0.1207–0.1138	0.0086–0.1291	0.4091–0.1219	0.1308–1.5499	1.5498–0.4138

The first zone corresponds to the hydration of the most active adsorption centers, which include hydrophilic -OH groups of sugars. It is represented by the adsorption of the monomolecular layer. Since adsorption occurs on the surface of the adsorbent, the larger the surface, the higher its adsorption capacity. But adsorption can occur only in certain areas of the adsorbent – active centers: corners, ribs of crystals which have a greater excess surface energy compared to the total surface area (Clément et al., 2018). Despite the fact that fondant candies have a fine crystalline structure, which is filled with active centers, in this area of pressure sorption is almost non-existent. This can be explained by the fact that in samples with a crystalline structure, moisture can interact only through hydrogen bonding on the crystal surface, because the location of a dense crystal lattice excludes foreign molecules such as water (Bell et al., 2000). Thus, the moisture content remains low and almost constant until the a_w value becomes high enough to cause the crystal surface dissolution at the point of contact. Above this relative humidity, water is able to dissolve the crystals, and the content of adsorbed moisture increases rapidly (Ergun et al., 2010; Sokolenko et al., 2020).

The amount of adsorbed moisture in the studied samples of candies at low values of a_w had a slight difference. This can be explained by the fact that sucrose and tagatose form a dense surface which minimizes the permeability of water to the central layers of candies.

The next stage of hydration of candies (zone II) is manifested in the appearance of a small rise of the adsorption curve in the range of activities a_w = 0.25-0.75. This is due to the gradual dissolution of the outer crystals of fondant with the gradual penetration of water molecules into the central layers of candy. The increase in the sorption properties of confectionery in this zone is due to the fact that the dissolution of sugars begins, similar patterns were observed in determining the sorption properties of rahat-lukum (Gostus et al., 1998). It is known that for pure crystalline sucrose the increase in moisture is very small, as long as the value of a_w does not exceed 0.8, and below this value water is connected by hydrogen bonds with groups -OH, which are on the crystal surface (Labuza et al., 2008). No similar results were found for tagatose.

It can be assumed that the faster the process of dissolving sugar crystals, the faster the moisture will be able to penetrate into the central layers of fondant samples. In this zone, the lowest amount of adsorbed moisture was in the control sample of fondant candies on sucrose – 0.0340 cm³/g, compared to the sample based on tagatose and fructose – 0.1291 cm³/g. This is due to the higher solubility of fructose compared to other studied sugars and its ability to absorb moisture at a_w=0.45-0.50.

In zone III there is a process of active absorption of moisture by samples of candies. The amount of water adsorbed in the third zone was 91.67 – 94.76% of the total amount of moisture (at a_w = 1.00). The difference between the indicators of adsorbed moisture in the samples at a_w = 0.76… 1.0 had a pronounced value, which can be explained by the different chemical composition of the studied samples of candies. Thus, in the developed sample the sorption capacity was higher than in the control sample due to the higher hygroscopicity of not only fructose but also tagatose in this moisture range. Thus, in the zone of capillary moisture (a_w = 1.0) the lowest amount of adsorbed moisture was in the control sample of fondant candies on sucrose – 0.6498 cm³/g, compared to the sample based on tagatose and fructose – 1.5499 cm³/g.

Table 4 shows the ratio of less bound and strongly bound water in the studied samples of candies. Free water has an enthalpy of vaporization almost the same as pure water, it freezes and is a solvent.

Table 4

Amount and ratio of bound and free moisture in the samples of fondant candies

Sample	**Amount of strongly bound and less bound moisture, cm³/h / ratio,%**		
	Zone I and zone II, strongly bound moisture	**Zone III, less bound moisture**	**Total amount of sorbed moisture**
Control sample	0.0340/5.24	0.6158/94.76	0.6498/100
Sample based on tagatose and fructose	0.1291/8.33	1.4207/91.67	1.5499/100.0

Comparative analysis of the obtained isotherms (Figure 1) shows that the isotherms of desorption (drying) in all samples were situated above the isotherms of sorption (humidification). The hysteresis loop covers the entire range of equilibrium vapor pressures. This indicates that the dehydration process is completely irreversible. After the desorption process, a small part of the adsorbed moisture remains in the samples (0.55 and 0.37%). Obtained data may suggest that some part of the adsorbed moisture binds to the sample with very strong bonds, most likely chemical. This moisture is not removed under these desorption conditions.

With the help of sorption analysis it is also possible to establish the equilibrium humidity of the studied samples of unglazed fondant candies with different carbohydrate composition. It is known that during storage of fondant candies on sucrose, they will give off moisture to the environment until the establishment of equilibrium humidity (Opris et al., 2020). Table 5 shows the values of equilibrium humidity of samples of fondant candies at values of relative humidity, which are in the range of 65-80%. This range of values of relative humidity is due to the fact that there are recommendations for storage of most finished confectionery products with a relative humidity of 70-75% (Sweets. General technical conditions, 2015). However, storage conditions may be such that the relative humidity may be lower or higher than the standard, so the range of relative humidity values was expanded from 65 to 80%.

Table 5

Value of equilibrium humidity of samples of fondant candies depending on the relative humidity

Sample	Moisture,%	Value of the equilibrium humidity of samples of candies,% at relative humidity, %		
		70	75	80
Fondant candies based on sucrose	10.0	0.23	3.40	4.32
Fondant candies based on tagatose and fructose	10.5	9.10	12.90	34.10

Analyzing the data in table 5, it should be noted that the sample of fondant candies on sucrose had an equilibrium moisture content below the mass fraction of moisture when stored within a relative humidity of 70-80%, so the phenomenon of desorption will be active in it. But in the sample of candies based on tagatose with fructose, the phenomenon of desorption will be observed at a relative humidity of 70% and below, and at a relative humidity above 75% there will be a phenomenon of sorption of moisture from the environment. It can be assumed that this is due to the hygroscopic properties of the sugars, as it is known that sucrose and tagatose at a_w = 0.75 do not absorb moisture. Fructose begins to absorb moisture at a_w = 0.45-0.55 and at a_w = 0.75 absorbs it up to 36.3% by weight of sugar (Polumbrik, 2011).

To ensure the stability of fondant candies based on tagatose during storage, one of the effective ways can be a rational selection of packaging and packaging material, the use of which will avoid contact with unstable environmental characteristics and prevent undesirable sorption and desorption of unglazed fondant candies.

Conclusion

1. Sorption properties of fondant candies based on sucrose and on low-glycemic sugars of tagatose and fructose were studied by analyzing their sorption-desorption isotherms.
2. The parameters of BET and Friendlich models were determined and it was found that the coefficients of determination of both models were in the range of 0.8 -1.0, which indicates their suitability for describing the isotherms of the studied products. The value of the coefficient of determination R^2 indicates that the obtained experimental values of samples based on sucrose are described with greater accuracy by the considered equations compared to samples based on tagatose and fructose.
3. The adsorbed moisture was distributed by zones and the ratio of less bound and more bound moisture in the studied candies was established, which shows an increase in the amount of more bound moisture in candies based on tagatose and fructose.
4. The equilibrium humidity of candies at different values of relative humidity of ambient air was determined. It was found that the control sample on sucrose is characterized by the phenomenon of desorption, even when stored at relative humidity of 80%, while for the sample of candies based on tagatose and fructose desorption is observed to relative humidity of 70-72%%, and with increasing of this indicator the phenomenon of sorption will occur.

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