



Differential thermal analysis of moisture binding in zephyr with different contents of glucose syrup

Inessa V. Plotnikova^{1,*}, Gazibeg O. Magomedov¹, Dmitry A. Kazartsev²,
Magomed G. Magomedov¹, Konstantin K. Polansky³, Viktor E. Plotnikov¹

¹ Voronezh State University of Engineering Technologies^{ROR}, Voronezh, Russia

² K.G. Razumovsky Moscow State University of Technologies and Management
(the First Cossack University)^{ROR}, Moscow, Russia

³ Plekhanov Russian University of Economics^{ROR}, Moscow, Russia

* e-mail: plotnikova_2506@mail.ru

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Abstract:

When formulating a product, it is just as important to measure changes in free and bound moisture as it is to analyze quality indicators. Zephyr, a Russian whipped dessert, made with sugar dries quickly during storage, gradually losing its moisture. Its crystalline sugar crust thickens and its entire mass saccharifies, resulting in higher firmness and poor appearance. In this study, we aimed to determine the effect of high-conversion glucose syrup on the amount of moisture and its binding forms in zephyr after storage.

We studied four samples of pectin-based zephyr with different carbohydrate profiles after three months of storage. Differential scanning calorimetry, thermogravimetry, and non-isothermal kinetics were applied to assess moisture contents and forms of binding in zephyr.

Thermograms with thermoanalytical curves were used to analyze the thermolysis of zephyr samples with different contents of glucose syrup in the temperature range from 20 to 300°C. We also studied the endothermic effects at various stages of thermolysis and measured free and bound moisture in the samples. Four stages of their dehydration were identified on the basis of graphical dependences between weight changes and heating temperatures.

The control zephyr sample contained more capillary and polymolecular bound moisture, while the experimental samples in which sugar and confectioner's syrup were partially or completely replaced with high-conversion glucose syrup had more polymolecular and monomolecular bound moisture. The use of high-conversion glucose syrup instead of sugar and confectioner's syrup reduced the amount of free moisture and therefore increased the amount of bound moisture, keeping zephyr fresh throughout its shelf life.

Keywords: Zephyr, glucose syrup, differential thermal analysis, moisture content, forms of moisture binding

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INTRODUCTION

Modern quality control methods in Russia are used to evaluate food products with various technological, structural, mechanical, thermophysical, and electrophysical properties throughout their shelf life [1]. They include chromatographic, spectral, optical, and thermovimetric instruments. It is particularly important to control changes in moisture binding, namely the ratio of free and bound moisture in the product based on the content and properties of moisture as it interacts with

dry substances. This also requires a comprehensive determination of the product's thermodynamic, thermophysical, structural, and mechanical characteristics, as well as its mass-exchange properties [2].

Whipped pastille mass has a colloidal capillary-porous coagulation structure. It is a two-phase gas-liquid system, where the dispersed phase is air (or gas) bubbles (up to 25 microns in size) separated by protein films and the dispersion medium is a solution of sugar, glucose syrup, acids, pectin, and other components. The dispersion medium, which is formed due to the

pectin frame of colloidal particles, is a stabilized foamy jelly with certain mechanical strength, plasticity, and elasticity [3].

Zephyr mass has a foamy structure and an intermediate moisture content of 18 to 25% that contributes to its increased plasticity and elasticity. In zephyr mass, water mainly moves along the skeleton of the jelly but it can evaporate and condensate inside the pores (cells).

Zephyr is produced with various types of gelling agents, including pectin, agar-agar, agaroid, furcellaran, gelatin, etc. These agents are high-molecular compounds that are similar to polysaccharides with a chain-like structure of molecules [4, 5]. In a soluble form, they contribute to the stability of foams. In particular, they are adsorbed in the liquid films covering air bubbles and increase their strength. Egg or milk-protein foaming agents are used to stabilize the foam structure.

Strong pectin jelly is produced from pectins, sugar (or its substitutes), sugary starch hydrolysates (glucose syrups of various types), organic acids, salts of alkali metals and weak organic acids, as well as water. Pectin forms a dense, elastic network of the frame which firmly holds the liquid phase of the jelly. To cause pectin substances to coagulate, water polarity is changed and the surface tension is increased by adding sugar or sugar-containing products. The amount of added sugar depends on the amount of pectin.

Greater esterification of molecules correlates with their lower polarity, which requires a lower sugar content in the solution, and vice versa [6]. Higher concentrations of sugar lead to higher surface tension, causing sucrose molecules to bind and hold more water molecules and resulting in lower water polarity. If the concentration of pectin with a medium gelling capacity is 0.8–1.0% of the jelly weight, the amount of sugar should be at least 65%, corresponding to the concentration of a saturated solution at 70°C.

Molded zephyr halves gradually cool down as they mature, with pectin jelly forming in the dispersion medium. As they dry, a thin crystalline crust forms on their surface, preventing them from sticking. As a result, zephyr halves can be easily removed from the trays, fastened together, and sprinkled with powdered sugar.

During storage, zephyr dries out quite quickly, gradually losing its moisture. Its crystalline crust thickens and its entire mass gets saccharified, resulting in better strength but poorer appearance (reduced volume).

To keep zephyr fresh, its moisture should be evenly distributed and retained throughout its entire shelf life [7]. Free moisture is actively involved in various biochemical and microbiological processes. Bound moisture is associated with proteins and carbohydrates through physical and chemical interactions and least contributes to product spoilage. Bound moisture can be increased by using polysaccharide hydrocolloids (dietary fibers, gums, pectin-containing materials, starch, polysaccharides, carrageenans, etc.) [8]. Chemically, they are linear or branched high-molecular compounds with hydrophilic

groups. They easily enter into physical interaction with moisture in the product and ensure its reliable retention.

Glucose syrups, depending on their type, contain from 15 to 60% of polysaccharides (dextrans, tetra- and trisaccharides), which are branched high-molecular compounds [9]. In addition, they contain hygroscopic reducing substances (glucose and maltose), which are good at retaining moisture for a long time.

The traditional zephyr formulation contains 40–50% of confectioner's syrup (by weight of sugar) with 36–44% of reducing substances on a dry basis (dextrose equivalent, DE), 42–34% of dextrans, and 65–75% of sugar (of the total weight). In addition, the syrup contains 0.1–0.4% of minerals, such as potassium, phosphorus, sodium, calcium, magnesium, and iron, as well as organic acids [10]. Its pH varies from 3.5 to 6.0.

In our previous studies, we tried to prolong zephyr's freshness during storage by slowing down its drying and gradual saccharization. For this, we partially or completely replaced sugar and confectioner's syrup in the control sample with various types of syrups [11]: high-conversion glucose syrup (DE = 62.6%) with 15.4% of dextrans, confectioner's syrup (DE = 40.4%) with 37.6% of dextrans, and low-conversion glucose syrup (DE = 32.3%) with 45.7% of dextrans. According to our results, confectioner's or low-conversion syrups used instead of sugar significantly increased the zephyr's viscosity and density, but decreased its shape-holding ability, causing the mass to spread after molding due to a high content of polysaccharides in these syrups. The use of high-conversion glucose syrup provided the zephyr mass with good viscosity and plasticity, as well as a density of $460 \pm 10 \text{ kg/m}^3$, which allowed the mass to be shaped by using a depositor [12].

Foamy zephyr mass can be obtained by two methods: 1) by prolonged mechanical whipping of an apple-sugar-pectin mixture at atmospheric pressure in the presence of a foaming agent, followed by adding a glucose syrup and 2) by saturating the mass from an apple-sugar-pectin mixture and a glucose syrup with air at excess pressure. In this study, the zephyr mass was obtained in laboratory conditions using the first method using a Kitfort KT-1338 planetary mixer at a rotation speed of 4.3 s^{-1} .

We aimed to study how the content of high-conversion glucose syrup changed the amount of water and its binding forms in zephyr during storage by using differential thermal analysis.

STUDY OBJECTS AND METHODS

We studied zephyr samples based on pectin with different carbohydrate profiles resulting from partial or complete replacement of sugar and confectioner's syrup with high-conversion glucose syrup. After manufacturing, the samples were packed in food-grade plastic transparent containers made of polyethylene terephthalate and stored for three months under controlled external conditions ($21 \pm 1.5^\circ\text{C}$, relative humidity of $82 \pm 2\%$). The control sample was prepared according to the traditional method by using sugar and acid confectioner's syrup at a ratio of 1:0.2. Samples No. 1 and No. 2

Table 1 Zephyr formulations with various carbohydrate profiles

Ingredient	Dry matter, %	Zephyr samples							
		Control		No. 1		No. 2		No. 3	
		Content of ingredients, g							
		Natural	Dry matter	Natural	Dry matter	Natural	Dry matter	Natural	Dry matter
		Syrup							
White sugar	99.85	34.65	34.60	34.65	34.60	34.65	34.60	–	–
Confectioner's syrup (DE = 40.4%)	83.40	13.98	11.65	13.98	11.65	13.98	11.65	–	–
High-conversion syrup (DE = 62.6%)	84.20	–	–	–	–	–	–	48.63	40.95
Total	–	54.41	46.25	53.78	46.25	53.16	46.25	46.53	40.95
Dry matter in syrup, %		85.0		86.0		87.0		88.0	
		Whipped mixture							
White sugar	99.85	30.87	30.82	15.42	15.40	–	–	–	–
High-conversion syrup (DE = 62.6%)	84.20	–	–	15.45	13.01	30.87	25.99	30.78	25.92
Pectin citrus	92.00	1.98	1.82	1.98	1.82	1.98	1.82	1.98	1.82
Apple puree	10.00	29.30	2.93	26.20	2.62	22.9	2.29	21.90	2.19
Egg white	12.00	6.33	0.76	6.33	0.76	6.33	0.76	6.33	0.76
Sodium lactate	40.00	0.65	0.26	0.65	0.26	0.65	0.26	0.65	0.26
Lactic acid	40.00	1.17	0.47	1.17	0.47	1.17	0.47	1.17	0.47
Flavoring	–	0.13	–	0.13	–	0.13	–	0.13	–
Total	–	70.43	37.06	67.33	34.34	64.03	31.49	62.94	31.42
Dry matter in mixture, %		52.6		51.0		49.2		50.0	
Total	–	124.84	83.31	121.11	80.59	117.19	77.74	109.47	72.37
Dry matter in zephyr mass, %		66.7		66.5		66.3		66.1	

Control contained sugar and acid confectioner's syrup at a ratio of 1:0.2 (traditional formulation)

Samples No. 1 and No. 2 included 50 and 100% of high-conversion glucose syrup instead of sugar

Sample No. 3 had sugar and acid confectioner's syrup completely replaced with high-conversion glucose syrup

had sugar partially (50%) and completely (100%) replaced with high-conversion glucose syrup in the whipped formulation mixture, respectively. Sample No. 3 had sugar and acid confectioner's syrup completely replaced with high-conversion glucose syrup in the whipped mixture and syrup.

The control sample was prepared as formulated in Table 1 by whipping an apple-sugar-pectin mixture with egg white and then adding, with constant stirring, confectioner's syrup with a moisture content of $85.0 \pm 0.5\%$ at $85\text{--}90^\circ\text{C}$ until the zephyr mass reached a moisture of $33.0 \pm 0.5\%$ and a density of $440 \pm 10 \text{ kg/m}^3$.

The partial or complete replacement of sugar with high-conversion glucose syrup gradually decreased the sweetness of the zephyr mass, intensified the aroma and taste of the fruit puree and flavoring additives (acids, flavors), and increased the mass's viscosity and strength. The content of reducing substances increased from 16.6% (control) to 38.7% (sample No. 3). Sucrose crystallization slowed down or stopped completely. Larger amounts of high-conversion glucose syrup in the zephyr samples increased the contents of mono- and disaccharides (glucose and maltose), whose molecules are highly hydrophilic, compared to those of sucrose. In particular, they bind moisture better and retain it for a long time, keeping zephyr fresh [13].

Using high-conversion glucose syrup instead of sugar increased the moisture content in the zephyr mass. To obtain the required moisture of $33.5 \pm 0.3\%$, the syrup was boiled down to a higher dry matter content of 86–88%. The content of apple puree was reduced from 23.5% (control) to 20% (sample No. 3) of the total mass in line with State Standard R 702.1.015-2021. After three months of storage, the control and experimental samples No. 1, 2, and 3 had moisture contents of 10.1, 11.5, 12.1, and 14.3%, respectively. Moisture was determined by the refractometric method in accordance with State Standard 5900-2014.

Changes in moisture and its binding forms in the zephyr samples after storage were determined by the differential thermal method involving differential scanning calorimetry (DSC), thermogravimetry (TG), and non-isothermal kinetics. Thermoanalytical curves (thermogravimetry and differential scanning calorimetry), which were used for quantitative processing by the method of non-isometric kinetics, showed changes in temperature, weight, and enthalpy [14]. Particularly, the thermogravimetry curve indicated weight losses caused by increased temperatures; the derivative thermogravimetry (DTG) curve showed the rate of weight losses; and the differential scanning calorimetry curve characterized the thermal effects of reactions at linearly increasing temperatures.

The differential scanning calorimetry method determines the direction and magnitude of enthalpy changes associated with changes in the product's moisture caused by heating. It registers thermal effects in the form of absorbed energy during physical, chemical, and structural changes in the product. The experimental curves show the dependence of the heat flux of absorbed energy on the exposure temperature [15]. The differential scanning calorimetry method measures differences in heat fluxes and temperatures between the experimental samples and the control.

The thermogravimetry method measures changes (losses) in the weight of a sample during its heating in a wide temperature range (from 20 to 300°C), corresponding to various phase transformations of moisture [16].

Thermal curves, or thermograms, resulting from thermal analysis depend on the chemical composition and structure of the sample. The differential scanning calorimetry and thermogravimetry curves are processed using the MS Excel and NETZSCH Proteus software [17]. The resulting DSC and DTG curves show the rate of absorbed energy and the rate of weight loss due to moisture removal by heating, respectively. The dDSC curve determines the rate of absorbed energy at different stages of sample heating, the initial and final temperatures of thermal reactions, and the temperature peaks with maximum rates of moisture evaporation or volatilization of other substances. The region of the derivative thermogravimetry curve with a constant drying rate corresponds to the removal of free moisture, while the region with a decreasing drying rate characterizes the removal of bound moisture [18].

In this study, we used an STA 449 F3 Jupiter synchronous thermal analysis (TG-DSC) apparatus (NETZSCH, Germany) for various gas atmosphere values. This apparatus has the advantages of a highly sensitive thermobalance and a differential scanning calorimeter.

The thermal analysis of the zephyr samples was carried out in the analytical center of Voronezh State University of Engineering Technologies at atmospheric pressure, maximum temperature of 573 K (300°C), and a temperature change rate of 5°C/min. Zephyr samples were placed in 20- μ L oxidized aluminum crucibles with a pierced lid. Since zephyr mass foams when heated, the sample weights were lower than the allowable volume of a crucible (12 mg), namely 6.3930, 7.8803, 5.1045, and 8.0919 mg for the control and samples No. 1, 2, and 3, respectively. The crucibles were placed in a nitrogen gas atmosphere of class 5.0, with a flow rate of 60 and 20 mL/min for the active sweeping gas and the protective gas, respectively.

RESULTS AND DISCUSSION

When heated, zephyr mass gradually loses its free moisture and then bound moisture. This is accompanied by wide endothermic effects characterizing moisture loss and decomposition of nutrients. Moreover, these processes depend on the chemical composition, quantity, and ratio of carbohydrates introduced with sugar and

glucose syrup; pectins and dietary fibers introduced with apple puree; protein substances of the foaming agent; and sugar decomposition products forming during the boiling of syrups [19].

High temperature causes significant physical and chemical changes in organic compounds, resulting in moisture release which transforms nutrients inside the product [20]. The weight of zephyr samples decreases due to moisture evaporation, protein denaturation, and carbohydrate decomposition [21].

The thermolysis of the zephyr samples in the temperature range from 20 to 300°C is shown in the thermoanalytical curves (Figs. 1–4). As can be seen, the weight of the control sample decreased by 55.83%, with the residual weight of 2.8238 mg or 44.17%; the weight of sample No. 1 decreased by 54.80%, with the residual weight of 3.5619 mg or 45.20%; that of sample No. 2, by 55.03%, with the residual weight of 2.2955 mg or 44.97%; and that of sample No. 3, by 53.31%, the residual weight of 3.7781 mg or 46.69%.

The initial heating of the zephyr samples was in the temperature range of 20–25°C. Further heating intensified moisture removal and, accordingly, increased the energy of breaking the bond between water and the material. The differential scanning calorimetry curves showed a number of endothermic effects characterized by changes in the enthalpy index and accompanied by heat absorption [22]. The thermogravimetry curves revealed changes in the weights of the zephyr samples and peaks of the endothermic effects (differential scanning calorimetry curves). The endothermic effects identified in various temperature ranges (Table 2) indicated a gradual removal of moisture from the samples according to the forms and energy of moisture binding with the biopolymers of the product [23].

During heat exposure, the zephyr samples passed from a structured to a highly elastic state due to moisture removal and many physicochemical processes [24].

The differential scanning calorimetry curve showed three endothermic effects for the control sample (Fig. 1), namely:

1. In the temperature range from 25.45 to 138.37°C, with a large amount of internal energy (328.9 J/g) absorbed at a medium rate (1.04%/min) at the peak of the derivative thermogravimetry curve ($t = 87.82^\circ\text{C}$) and a weight loss of 0.8596 mg (13.44%) (thermogravimetry curve) due to dehydration with a removal of capillary, poly- and monomolecular bound moisture (0.6460 mg) at the beginning of melting, thermal decomposition of monosaccharides (fructose and maltose), denaturation and subsequent decomposition of protein substances, and the formation of various chemical compounds with volatile substances released in the form of gases (0.2136 mg).

2. In the temperature range from 155.49 to 167.95°C, with a small amount of internal energy (3.833 J/g) absorbed at a low rate (0.12%/min) at the peak of the derivative thermogravimetry curve ($t = 165.99^\circ\text{C}$) and a weight loss of 0.0774 mg (1.21%) (thermogravimetry curve) due

to the melting of glucose and sucrose, the decomposition and caramelization of fructose and maltose, as well as the formation of anhydrides, reversion (condensation) products, hydroxymethylfurfural, organic (formic and levulinic) acids, and colored compounds (carameline, caramylene), with volatile substances released in the form of gases (0.0774 mg).

3. In the temperature range from 188.46 to 231.89°C, with a small amount of internal energy (92.6 J/g) absorbed at a high rate (4.0%/min) at the peak of the derivative thermogravimetry curve ($t = 218.13^\circ\text{C}$) and a weight loss of 1.5044 mg (23.52%) (thermogravimetry curve) due to the thermal decomposition and caramelization of sucrose and glucose, the formation of anhydrides, reversion products, hydroxymethylfurfural, organic acids,

and colored compounds, as well as the decomposition of dextrans and protein substances, with volatile substances released in the form of gases (1.5044 mg).

The differential scanning calorimetry curve revealed two endothermic effects for sample No. 1 (Fig. 2), namely:

1. In the temperature range from 33.72 to 140.85°C, with a large amount of internal energy (336.6 J/g) absorbed at a medium rate (1.05%/min) at the peak of the derivative thermogravimetry curve ($t = 84.26^\circ\text{C}$) and a weight loss of 1.0181 mg (12.92%) (thermogravimetry curve) due to dehydration processes with a removal of capillary, poly- and monomolecular bound moisture (0.9062 mg) and the beginning of melting and thermal decomposition of monosaccharides (fructose, maltose),

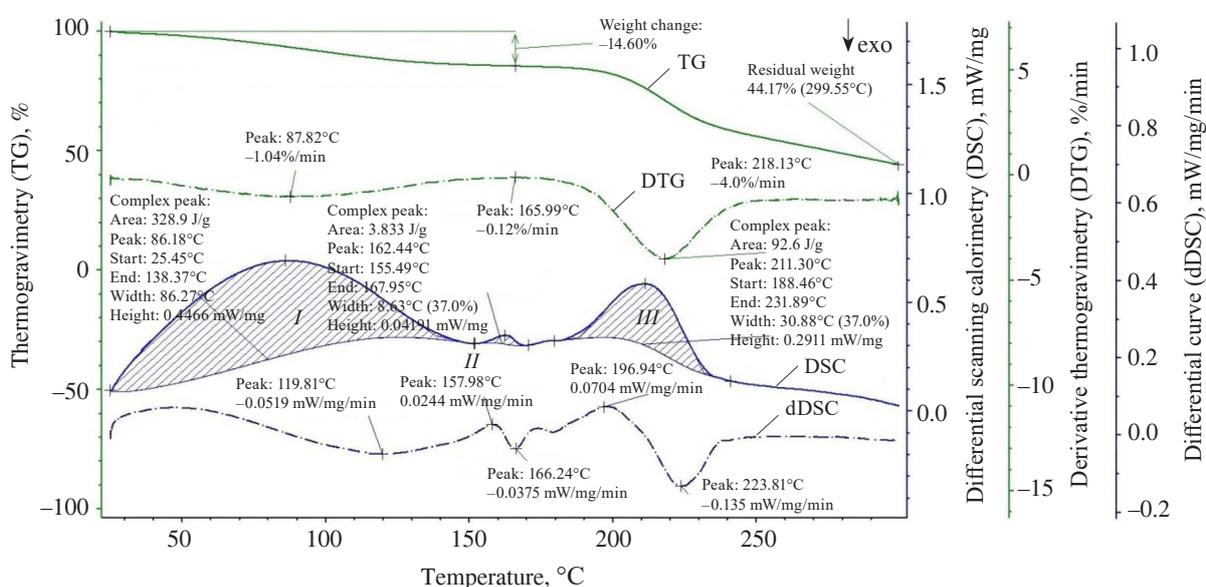


Figure 1 Thermogram of the control zephyr sample made from sugar and confectioner’s syrup (DE = 40.4%) at a ratio of 1:0.2

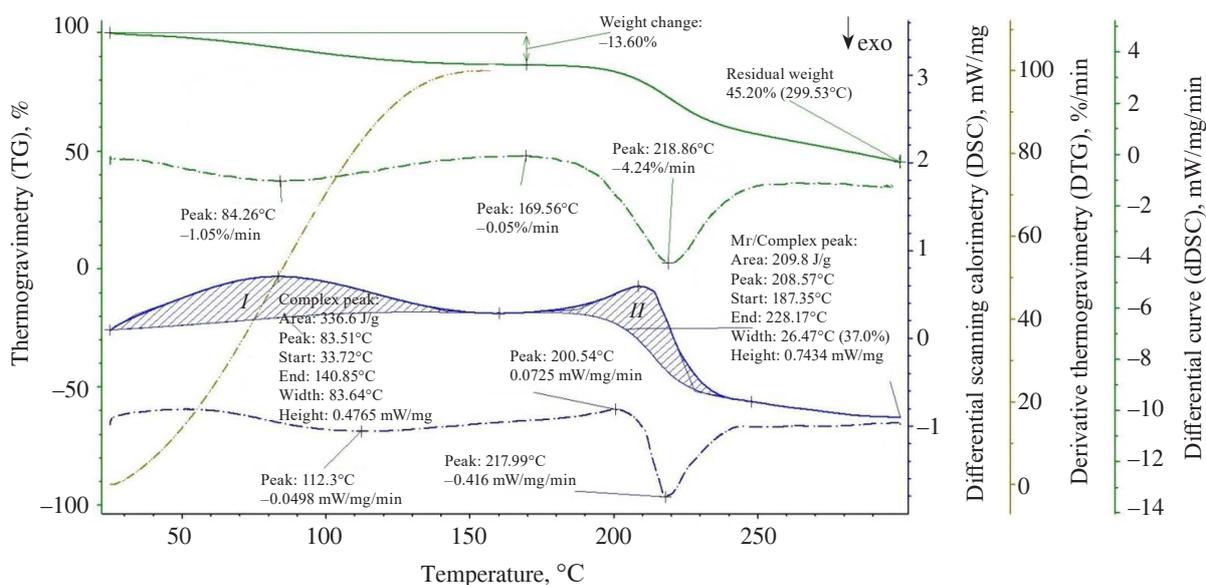


Figure 2 Thermogram of zephyr sample No. 1 with 50% sugar in the whipped mixture replaced with high-conversion glucose syrup (DE = 62.6%)

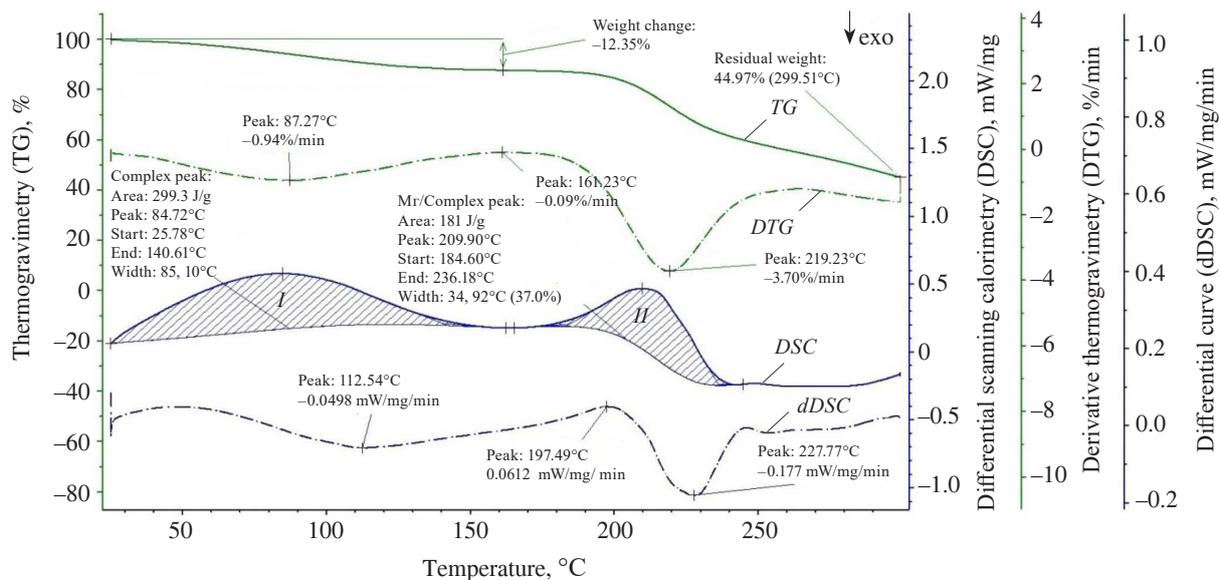


Figure 3 Thermogram of zephyr sample No. 2 with 100% sugar in the whipped mixture replaced with high-conversion glucose syrup (DE = 62.6%)

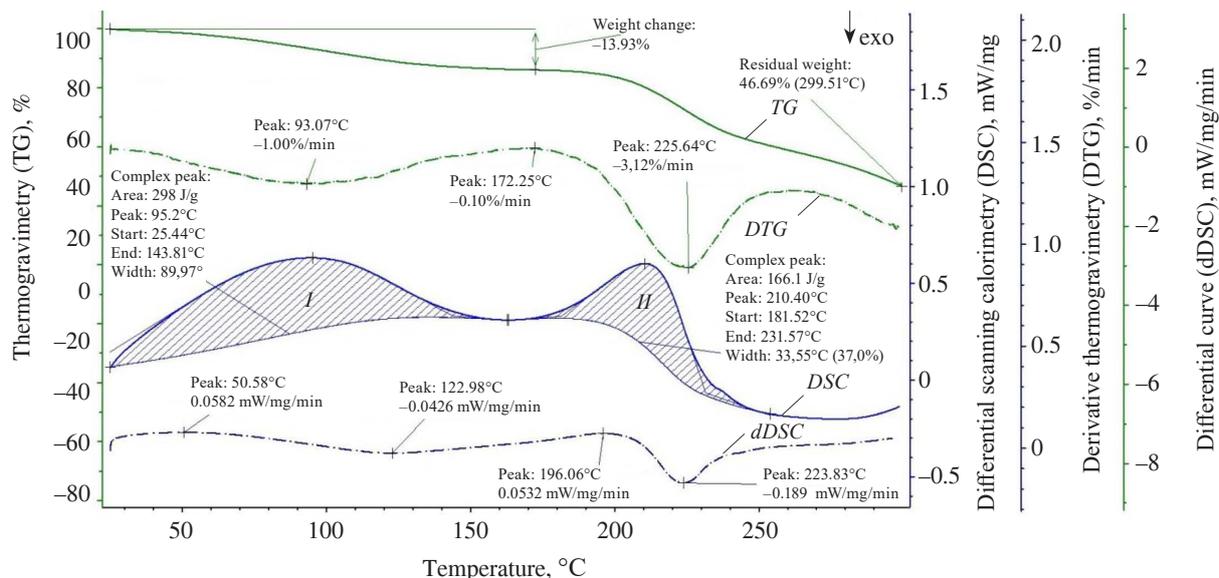


Figure 4 Thermogram of zephyr sample No. 3 with 100% sugar and confectioner's syrup (DE = 40.4 %) replaced with high-conversion glucose syrup (DE = 62.6%) in the whipped mixture and syrup

denaturation and subsequent decomposition of protein substances, and the formation of various chemical compounds with a release of volatile substances in the form of gases (0.1119 mg).

2. In the temperature range from 187.35 to 228.17°C, with a smaller amount of internal energy (209.8 J/g) absorbed at a high rate (4.24%/min) at the peak of the derivative thermogravimetry curve ($t = 218.86^\circ\text{C}$) and a weight loss of 1.7321 mg (21.98%) (thermogravimetry curve) due to the thermal decomposition and caramelization of mono- and disaccharides (sucrose, glucose, fructose, and maltose), the formation of anhydrides, reversion products, hydroxymethylfurfural, organic acids, and colored compounds, as well as the decomposition of

dextrins and protein substances into by-products with a release of volatile substances in the form of gases (1.7321 mg).

The thermolysis of sample No. 2 had two endothermic effects, as shown on the differential scanning calorimetry curve (Fig. 3), namely:

1. In the temperature range from 25.78 to 140.61°C, with a large amount of internal energy (299.3 J/g) absorbed at a medium rate (0.94%/min) at the peak of the derivative thermogravimetry curve ($t = 87.27^\circ\text{C}$) and a loss weight of 0.5972 mg (11.70%) (thermogravimetry curve) due to dehydration processes with a removal of capillary, poly- and monomolecular bound moisture (0.5972 mg) during the denaturation of protein

Table 2 The kinetic characteristics of endothermic effects and weight changes during the thermolysis of zephyr samples with different carbohydrate profiles

Zephyr sample	Endo-thermic effect	The beginning and end of the endothermic effect, ΔT , °C (DSC curve)	Enthalpy ΔH , J/g (DSC curve)	The peak of the endothermic effect, °C (DSC curve)	Weight loss during thermolysis (TG curve)		
					Weight loss range, %	Weight difference in the range, %	Total weight loss after thermolysis, %
Control	I	25.45–138.37	328.9	86.18	0–13.44	13.44	38.17
	II	155.49–167.95	3.833	162.44	13.44–14.65	1.21	
	III	188.46–231.89	92.6	211.3	14.65–38.17	23.52	
No. 1	I	33.72–140.85	336.6	83.51	0–12.92	12.92	34.90
	II	187.35–228.17	209.8	208.57	12.92–34.90	21.98	
No. 2	I	25.78–140.61	299.3	84.72	0–11.70	11.70	37.01
	II	184.6–236.18	181.0	209.9	11.70–37.01	25.31	
No. 3	I	25.44–143.81	298.0	95.2	0–12.90	12.90	31.46
	II	181.52–231.57	166.1	210.4	12.90–31.46	18.56	

Control contained sugar and acid confectioner's syrup at a ratio of 1:0.2 (traditional formulation)

Samples No. 1 and 2 included 50 and 100% of high-conversion glucose syrup instead of sugar

Sample No. 3 had sugar and acid confectioner's syrup completely replaced with high-conversion glucose syrup

substances and the beginning of melting of monosaccharides (fructose and maltose).

2. In the temperature range from 184.6 to 236.18°C, with a smaller amount of internal energy (181.0 J/g) absorbed at a high rate (3.70%/min) at the peak of the derivative thermogravimetry curve ($t = 219.23^\circ\text{C}$) and a weight loss of 1.8892 mg (25.31%) (thermogravimetry curve) due to dehydration with a removal of monomolecular bound moisture (0.0205 mg) during the thermal decomposition and caramelization of mono- and disaccharides (sucrose, glucose, fructose, and maltose), the formation of anhydrides, reversion products, hydroxymethylfurfural, organic acids, and colored compounds, as well as the decomposition of dextrans and protein substances into by-products with a release of volatile substances in the form of gases (1.292 mg).

The differential scanning calorimetry curve showed two endothermic effects for sample No. 3 (Fig. 4), just as for samples No. 2 and 3, namely:

1. In the temperature range from 25.44 to 143.81°C, with a large amount of internal energy (298.0 J/g) absorbed at a medium rate (1.0%/min) at the peak of the derivative thermogravimetry curve ($t = 93.07^\circ\text{C}$) and a weight loss of 1.0439 mg (12.9%) (thermogravimetry curve) due to dehydration processes with a removal of capillary, poly- and monomolecular bound moisture (1.0439 mg) during the denaturation of protein substances and the beginning of melting of monosaccharides (fructose and maltose).

2. In the temperature range from 181.52 to 231.57°C, with a smaller amount of internal energy (166.1 J/g) absorbed at a high rate (3.12%/min) at the peak of the derivative thermogravimetry curve ($t = 225.64^\circ\text{C}$) and a weight loss of 1.5018 mg (18.56%) (thermogravimetry curve) due to dehydration processes with a removal of monomolecular bound moisture (0.1132 mg) during the thermal decomposition and caramelization of mono- and disaccharides (sucrose, glucose, fructose, and maltose), the formation of anhydrides, reversion products, hydroxy-

methylfurfural, organic acids, and colored compounds, as well as the decomposition of dextrans and protein substances into by-products with a release of volatile substances in the form of gases (1.3886 mg).

According to our data, the temperature ranges at which the first endothermic effect occurred were wider for the experimental samples than the control, indicative of a higher degree of moisture binding in them. At the beginning of thermolysis, after the first endothermic effect, when the largest amount of free moisture was removed, the enthalpy values were higher for the control and sample No. 1 (328.9 and 336.6 kJ/g, respectively) than for samples No. 2 and 3 (299.3 and 298.0 kJ/g, respectively). This explains the greater decrease in the weight of the control and sample No. 1 (13.44 and 12.92%, respectively) compared to samples No. 2 and 3 (11.7 and 12.9%, respectively). At this stage of dehydration, the control and sample No. 1 not only lost capillary, poly- and monomolecular bound moisture, but also began to undergo the processes of melting and thermal decomposition of monosaccharides and protein substances into by-products. With further heating, the weight of the control and sample No. 1 decreased by 24.73 and 21.98%, respectively, while the weight of samples No. 2 and 3 reduced by 25.31 and 18.56%, respectively. The total weight loss after thermolysis amounted to 38.17, 34.90, 37.01, and 31.46% for the control and experimental samples No. 1, 2, and 3, respectively. The total amount of by-products was 1.7954, 1.844, 1.292, and 1.3886 mg for the control and samples No. 1, 2, and 3, respectively.

To quantify the changes in moisture and the forms of its binding with the sample components, the dependence between weight change on the thermogravimetry curves and dehydration in the specified temperature ranges is converted into the dependence between the degree of weight change, or the degree of substance transformation (α , mg/mg), and the heating temperature (T , K) [25]. For this, weight changes (Δm_i , %) were registered on the thermogravimetry curves at certain temperatures after

every rise of 5°C, which corresponded to the amount of water released at temperature T_i . The indicator α was calculated as a ratio of the current weight change (Δm_i) at a certain time (τ_i) to the total weight change at the end of dehydration (Δm_{max} , %) according to the formula [26]:

$$\alpha = \frac{\Delta m_i}{\Delta m_{max}} \quad (1)$$

Based on the thermogravimetry curves (Figs. 1–4), we presented the initial weights of the zephyr samples (m_i), their changes during thermolysis (Δm_i), and the α indicator in the temperature range from 24 to 183°C

(Table 3). After three months of storage, the moisture contents, as determined by the arbitration method, were 10.1, 11.5, 12.1, and 14.3% in the control and samples No. 1, 2, and 3, respectively.

Figure 5 shows graphical dependences between moisture changes in the zephyr samples and thermolysis temperatures in the range from 24 to 183°C.

The dependence curves $\alpha = f(T)$ (Fig. 6) have an S-shaped form [27] which shows a complex nature of interaction between water and dry substances in the zephyr samples and the process of dehydration with different rates of release of moisture and binding energy at different stages of thermal analysis [28]. These curves

Table 3 Changes in the weight of zephyr samples during thermolysis according to thermogravimetry curves

Dehydration temperature, T_i , K (°C)	Zephyr samples											
	Control			No. 1			No. 2			No. 3		
	m_i , %	Δm_i , %	α , mg/mg	m_i , %	Δm_i , %	α , mg/mg	m_i , %	Δm_i , %	α , mg/mg	m_i , %	Δm_i , %	α , mg/mg
297 (24)	100	0	0	100	0	0	100	0	0	100	0	0
305 (32)	99.244	0.76	0.075	99.441	0.56	0.049	99.529	0.47	0.039	99.469	0.53	0.037
310 (37)	98.960	1.04	0.103	99.162	0.84	0.073	99.293	0.71	0.059	99.254	0.75	0.052
315 (42)	98.673	1.33	0.132	98.882	1.12	0.097	99.057	0.94	0.078	99.024	0.98	0.069
320 (47)	98.306	1.69	0.167	98.542	1.46	0.127	98.759	1.24	0.102	98.741	1.26	0.088
325 (52)	97.881	2.12	0.210	98.202	1.80	0.157	98.461	1.54	0.127	98.394	1.61	0.113
330 (57)	97.391	2.61	0.258	97.710	2.30	0.200	98.040	1.96	0.162	97.978	2.02	0.141
335 (62)	96.851	3.15	0.312	97.111	2.89	0.251	97.487	2.51	0.207	97.518	2.48	0.173
340 (67)	96.254	3.75	0.371	96.551	3.45	0.231	96.915	3.09	0.255	97.012	2.99	0.209
345 (72)	95.595	4.41	0.437	95.941	4.06	0.353	96.319	3.68	0.304	96.449	3.55	0.248
350 (77)	94.889	5.11	0.506	95.351	4.65	0.404	95.729	4.27	0.353	95.816	4.18	0.292
355 (82)	94.131	5.87	0.581	94.584	5.42	0.471	95.184	4.82	0.398	95.131	4.87	0.341
360 (87)	93.351	6.65	0.658	93.871	6.13	0.533	94.555	5.45	0.450	94.407	5.59	0.391
365 (92)	92.560	7.44	0.737	93.187	6.81	0.592	93.913	6.09	0.503	93.657	6.34	0.443
370 (97)	91.762	8.24	0.816	92.502	7.49	0.652	93.272	6.73	0.556	92.883	7.12	0.498
375 (102)	90.965	9.04	0.895	91.743	8.26	0.718	92.619	7.38	0.610	92.097	7.90	0.552
380 (107)	90.188	9.81	0.971	90.943	9.06	0.788	91.843	8.16	0.674	91.315	8.69	0.608
382 (109)	89.900	10.10	1.000	90.664	9.34	0.812	91.585	8.42	0.696	91.001	8.90	0.622
385 (112)	89.432	10.57	–	90.225	9.78	0.850	91.094	8.91	0.736	90.544	9.46	0.662
390 (117)	88.722	11.28	–	89.450	10.55	0.917	90.344	9.66	0.798	89.785	10.22	0.715
395 (122)	88.069	11.93	–	88.868	11.13	0.968	89.655	10.35	0.855	89.078	10.92	0.764
400 (127)	87.501	12.50	–	88.515	11.50	1.000	89.195	10.81	0.893	88.471	11.53	0.806
405 (132)	87.011	12.99	–	87.970	12.03	–	88.964	11.04	0.912	87.957	12.04	0.842
410 (137)	86.624	13.38	–	87.697	12.30	–	88.706	11.29	0.933	87.550	12.45	0.871
415 (142)	86.313	13.69	–	87.424	12.58	–	88.324	11.68	0.965	87.223	12.78	0.894
420 (147)	86.059	13.94	–	87.077	12.92	–	88.115	11.89	0.983	86.925	13.08	0.915
425 (152)	85.853	14.15	–	86.904	13.10	–	87.949	11.05	0.913	86.668	13.33	0.932
428 (155)	85.745	14.26	–	86.817	13.18	–	87.866	12.10	1.000	86.538	13.46	0.941
430 (157)	85.682	14.32	–	86.730	13.27	–	87.782	12.22	–	86.464	13.54	0.947
435 (162)	85.519	14.48	–	86.545	13.46	–	87.606	12.39	–	86.308	13.69	0.957
440 (167)	85.381	14.62	–	86.453	13.55	–	87.518	12.48	–	86.185	13.82	0.966
445 (172)	85.244	14.76	–	86.360	13.64	–	87.430	12.57	–	86.000	13.99	0.979
450 (177)	85.073	14.93	–	86.274	13.73	–	87.321	12.68	–	85.886	14.11	0.987
455 (182)	84.832	15.17	–	86.188	13.81	–	87.219	12.78	–	85.755	14.25	0.997
456 (183)	84.769	15.23	–	86.012	13.99	–	87.119	12.88	–	85.700	14.30	1.000

Note: m_i is the initial weight of a sample; Δm_i is a weight change during thermolysis; α is the degree of weight change

Control contained sugar and acid confectioner’s syrup at a ratio of 1:0.2 (traditional formulation)

Samples No. 1 and 2 included 50 and 100% of high-conversion glucose syrup instead of sugar

Sample No. 3 had sugar and acid confectioner’s syrup completely replaced with high-conversion glucose syrup

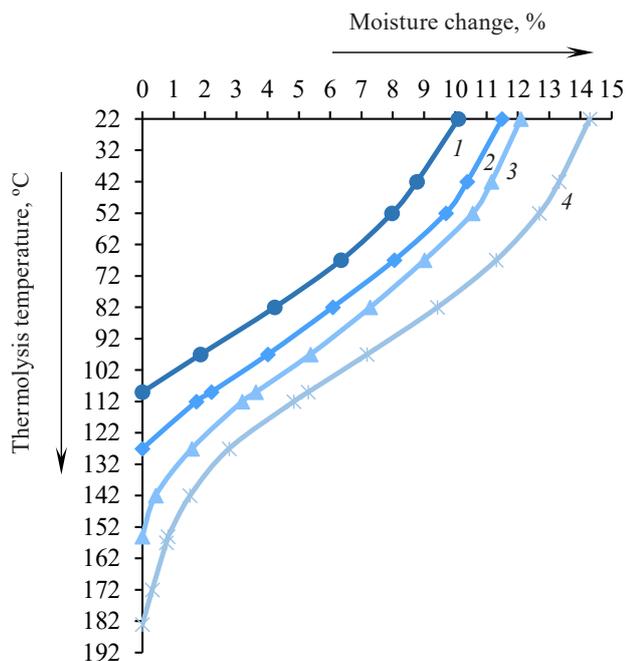


Figure 5 Moisture changes in the zephyr samples with different carbohydrate profiles during thermolysis: 1 – control, 2 – sample No. 1, 3 – sample No. 2, and 4 – sample No. 3

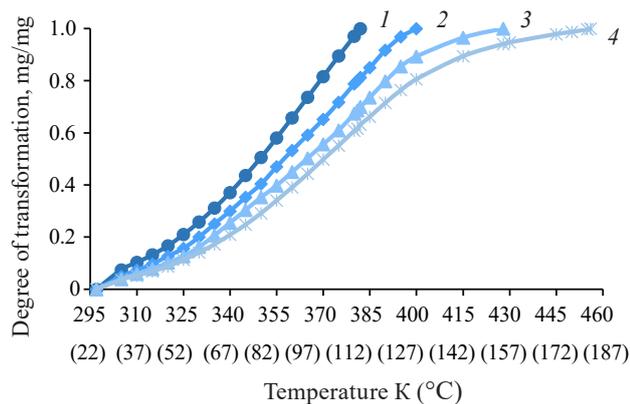


Figure 6 Correlations between the degree of transformation (α) and absolute heating temperatures (T) in the zephyr samples with different carbohydrate profiles: 1 – control, 2 – sample No. 1, 3 – sample No. 2, and 4 – sample No. 3

reveal various kinetically unequal forms of moisture binding in the samples at different rates of dehydration.

To establish the forms of moisture binding and the mechanism of moisture removal in the zephyr samples, we used the curves in the α - T coordinates, as well as the data on temperature ranges and the amounts of bound moisture, and plotted the same curves in the coordinates $(-lg\alpha)$ - $(10^3/T)$. The dependences $-lg\alpha = f(10^3/T)$ were made for the temperature range of 297–456 K (Fig. 7) [29–31].

Based on the curves showing graphic dependences between the changes in α converted into $(-lg\alpha)$ and the changes in temperature ($10^3/T$), we identified four stages of dehydration of the zephyr samples with a removal of

moisture in various forms and with different binding energy. The curves that indicate weight changes at certain temperatures had characteristic inflection points B_i , C_i , and D_i , showing changes in degradation. The graphic dependences (Fig. 7) were approximated in the form of four-line splines in order to establish the rates of moisture removal in relation to the energy levels of moisture binding in the samples [32, 33]. Approximating curves were constructed for each temperature range, which were linear in nature ($R^2 \approx 1$).

Four linear sections marked with a dash-dotted line (Fig. 7) confirm a gradual (stage by stage) removal of moisture depending on the forms of its binding with biopolymers [34, 35] in the zephyr samples with different carbohydrate profiles, namely:

- stage 1 (section A_iB_i) characterizes the removal of free, physically and mechanically bound (capillary-bound) moisture from zephyr micro- and macrocapillaries (pores) that has a low energy of binding with the material (the desorption of capillary moisture has a lower activation energy compared to the moisture released in the second stage) [36];
- stage 2 (section B_iC_i) characterizes the removal of physically and chemically bound (capillary- and polymolecule-bound) moisture that is more strongly bound with zephyr nutrients than capillary moisture and is mainly contained in the form of hydrated moisture in the closed cells of nutrient micelles and around carbohydrate molecules [27];
- stage 3 (section C_iD_i) characterizes the removal of physically and chemically bound (poly- and monomolecular bound) moisture during the partial decomposition of nutrients that is strongly bound with nutrients and is involved in the swelling of proteins, pectins, and dietary fibers [37, 38]; and
- stage 4 (section D_iK_i) characterizes the removal of physically and chemically bound (poly- and monomolecular bound) moisture and gaseous substances formed during the decomposition of nutrients.

Table 4 shows the degrees of transformation ($\Delta\alpha$) corresponding to the four-stage dehydration kinetics of the zephyr samples with different carbohydrate profiles.

As can be seen, the first section (A_iB_i – A_4B_4), which refers to the temperature range of 297–312 K (24–39°C), is where the “water-water” bonds are destroyed and the “free”, physically and mechanically bound (capillary-bound) moisture is released from the surface of the zephyr samples in insignificant amounts: 10.30, 8.26, 6.78, and 5.94% for the control and samples No. 1, 2, and 3, respectively.

The second section (B_iC_i – B_4C_4) refers to a more intensive removal of physically and chemically bound (capillary- and polymolecular bound) moisture in the temperature range of 310–361 K (37–88°C) in the following amounts: 40.20, 40.43, 35.37, and 36.02% for the control and samples No. 1, 2, and 3, respectively.

The third section (C_iD_i – C_4D_4) is where physically and chemically bound (poly- and monomolecular bound) moisture is removed in the temperature range of 349–403 K (76–130°C) in the following amounts: 31.09,

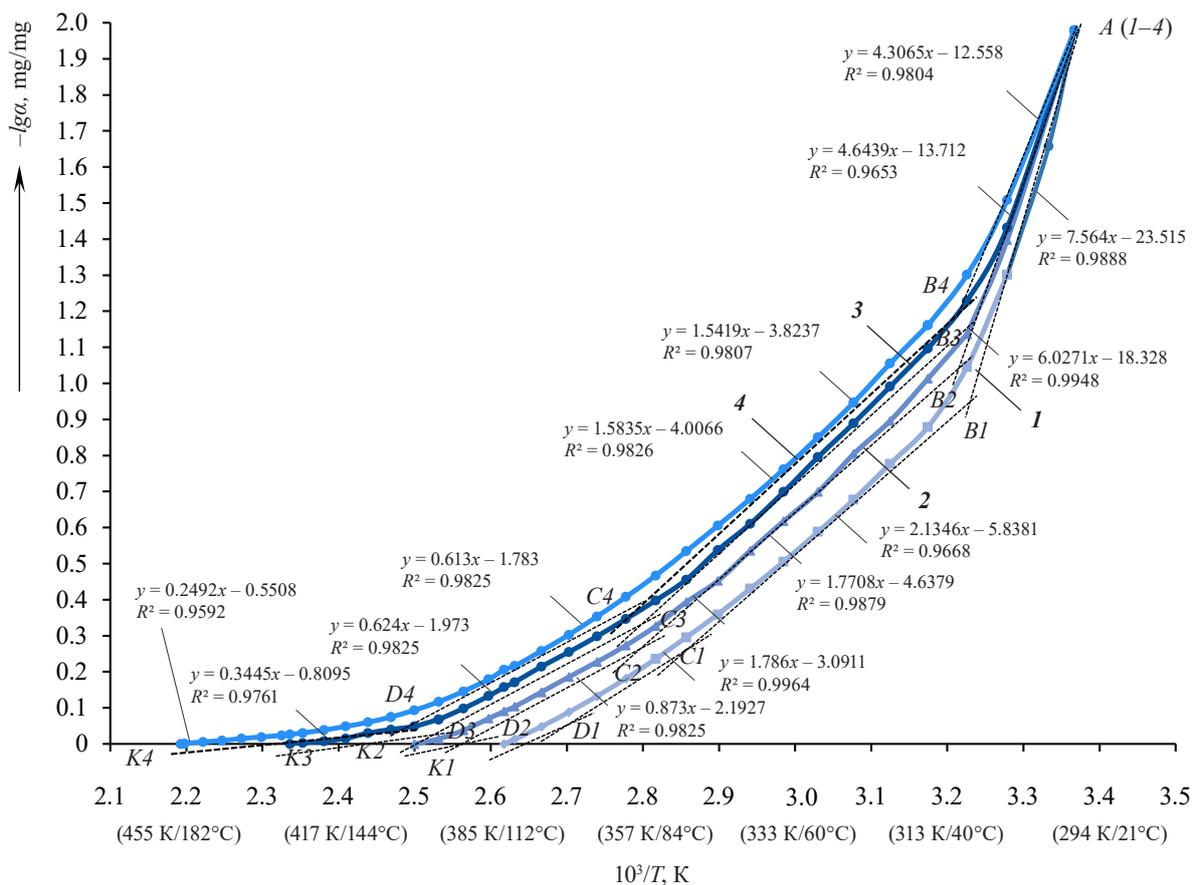


Figure 7 Dependence ($-lg\alpha$) on temperature ($10^3/T$) at an air heating rate of $5^\circ\text{C}/\text{min}$ for the zephyr samples with different carbohydrate profiles: 1 – control; 2 – sample No. 1; 3 – sample No. 2; and 4 – sample No. 3

44.35, 43.80, and 39.86% for the control and samples No. 1, 2, and 3, respectively.

The last section (D_1K_1 – D_4K_4) refers to the removal of physically and chemically bound (poly- and monomolecular bound) moisture and gaseous substances in the temperature range of 370–456 K (97–183°C) in the following amounts: 18.41, 10.44, 14.05, and 18.18% for the control and samples No. 1, 2, and 3, respectively.

CONCLUSION

In this study, we used thermal analysis to determine the content of moisture and forms of its binding in zephyr during storage. The samples in which sugar and confectioner's syrup were partially or completely replaced with high-conversion glucose syrup gradually lost some of their sweetness and had a more pronounced taste and smell of fruit puree, as well as flavoring agents, a higher degree of moisture binding due to an increased content of reducing sugars (glucose and maltose), and slower, or completely eliminated, crystallization of sucrose during storage.

In addition, the samples had a lower content of fructose (or no fructose at all), a highly hygroscopic sugar formed during the boiling of glucose syrup that contributes to a rapid wetting of zephyr. Although glucose and maltose are less hygroscopic, they are highly hydrophilic – they bind moisture firmly and retain it for a long time, keeping zephyr fresh.

rophilic – they bind moisture firmly and retain it for a long time, keeping zephyr fresh.

The thermal analysis of the zephyr samples with different carbohydrate profiles showed that at the initial stage of dehydration, the largest amount of “free”, physically and mechanically bound moisture was removed from the control sample, which had a lower content of reducing sugars and dextrins. The wider temperature ranges in all sections of heating of the experimental samples indicated a higher content of physically and chemically bound moisture in them, compared to the control. Therefore, replacing sugar and confectioner's syrup with high-conversion glucose syrup results in less “free”, capillary-bound moisture and more poly- and monomolecular bound moisture in zephyr. This is due to a higher content of reducing sugars which have high hydrophilic properties allowing them to firmly bind and retain moisture for a long time, prolonging the freshness of zephyr and slowing down its drying during storage [39].

RECOMMENDATION

The differential thermal analysis used to assess moisture and forms of its binding in zephyr samples with different carbohydrate profiles allowed us to determine the effect of high-conversion glucose syrup on the

Table 4 Dehydration kinetics of the zephyr samples with different carbohydrate profiles

Zephyr sample	Stage of dehydration	ΔT , K (°C)	1000/T	$\Delta\alpha$, mg/mg	$-lg\alpha$, mg/mg	Amount of moisture removed during thermolysis (TG curve)	
						Δm , %	% of the total amount
Control	I	297–310 (24–37)	3.37–3.23	0–0.11	1.98–0.95	0–1.04	10.30
	II	310–349 (37–76)	3.23–2.86	0.11–0.51	0.95–0.29	1.04–5.10	40.20
	III	349–370 (76–97)	2.86–2.70	0.51–0.82	0.29–0.08	5.10–8.24	31.09
	IV	370–382 (97–109)	2.70–2.62	0.82–1.00	0.08–0	8.24– 10.10	18.41
No. 1	I	297–312 (24–39)	3.37–3.22	0–0.09	1.98–1.04	0–0.95	8.26
	II	312–357 (39–84)	3.22–2.80	0.09–0.51	1.04–0.29	0.95–5.60	40.43
	III	357–391 (84–118)	2.80–2.55	0.51–0.95	0.29–0.02	5.60–10.30	40.87
	IV	391–400 (118–127)	2.55–2.50	0.95–1.00	0.02–0	10.30– 11.50	10.44
No. 2	I	297–311 (24–38)	3.37–3.10	0–0.07	1.98–1.14	0–0.82	6.78
	II	311–357 (38–84)	3.10–2.80	0.07–0.44	1.14–0.35	0.82–5.10	35.37
	III	357–395 (84–122)	2.80–2.53	0.44–0.91	0.35–0.04	5.10–10.40	43.80
	IV	395–428 (122–155)	2.53–2.34	0.91–1.00	0.04–0	10.40– 12.10	14.05
No. 3	I	297–312 (24–39)	3.37–3.22	0–0.06	1.98–1.23	0–0.85	5.94
	II	312–361 (39–88)	3.22–2.77	0.06–0.41	1.23–0.38	0.85–6.00	36.02
	III	361–403 (88–130)	2.77–2.48	0.41–0.87	0.38–0.06	6.00–11.70	39.86
	IV	403–456 (130–183)	2.48–2.19	0.87–1.00	0.06–0	11.70– 14.30	18.18

Control contained sugar and acid confectioner's syrup at a ratio of 1:0.2 (traditional formulation)

Samples No. 1 and 2 included 50 and 100% of high-conversion glucose syrup instead of sugar

Sample No. 3 had sugar and acid confectioner's syrup completely replaced with high-conversion glucose syrup

content of free and bound moisture in zephyr and to predict a possibility of extending its freshness during storage. In the future, our results may be used to create a database of ingredients that contribute to the product's quality and extended shelf life.

CONTRIBUTION

I.V. Plotnikova developed the research concept, proposed a methodology for the experiment, and edited the manuscript for submission. G.O. Magomedov supervised and monitored the experiment. D.A. Kazartsev processed the experimental data, performed calculations, and edited the manuscript for submission. M.G. Magomedov processed the experimental data and advised on the experiment. K.K. Polansky organized the production tests

and advised on the experiment. V.E. Plotnikov reviewed literary sources on the research problem and conducted the experiment. All the authors were equally involved in writing the manuscript and are equally responsible for plagiarism.

CONFLICT OF INTEREST

The authors declare no conflict of interest regarding the publication of this article.

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ORCID IDs

Inessa V. Plotnikova <https://orcid.org/0000-0001-5959-6652>

Gazibeg O. Magomedov <https://orcid.org/0000-0002-7201-8387>

Dmitry A. Kazartsev <https://orcid.org/0000-0001-6597-2327>

Magomed G. Magomedov <https://orcid.org/0000-0003-2494-4973>

Konstantin K. Polansky <https://orcid.org/0000-0002-8817-1466>

Viktor E. Plotnikov <https://orcid.org/0000-0001-6707-8337>