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INVESTIGATION OF THE MOISTURE BINDING FORMS IN PLANT RAW MATERIALS AND THERMAL CHARACTERISTICS DETERMINATION

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Abstract. *In this article, the forms of moisture connection and the thermal characteristics of plant materials are studied. Rosehip and hawthorn fruits have been chosen as the object of the research. In the heat analyzer, a decrease in the mass of the product and an increase in temperature have been found when the temperature was controlled in a certain gas environment. The mass and temperature dependence curves of the dog rose and hawthorn samples are presented in the article. DTA-1/10 galvanometer and DTG-1/5 galvanometer sensitivity, the maximum heating temperature is 300° C and the rate of change of the furnace heating temperature is 3° C / min. The temperature features of the plant product thermolysis have been studied. As a result of the study, the initial temperature, the highest and the final temperature of endothermic action have been determined. The thermophysical properties of the rosehip and hawthorn fruit have been studied on the basis of the experiments and the results are reflected in the tables below. The thermal conductivity coefficients and specific heat of mass at the temperature range of 20-80 ºC are given in the article as well.*

Keywords**:** *rosehip, hawthorn, thermal analyzer, derivatograms, temperature, product, endothermic effect.*

With the growth of the world's population, the problems of providing high-quality, biologically complete, environmentally safe food, rational use of raw materials and fuel and energy potential are becoming more and more urgent. The main direction of increasing the production of concentrates is the creation of new, progressive, energy-saving technologies and improvement of the existing ones that ensure high quality of finished products based on the introduction of new technology that helps save raw materials, energy, materials, and the creation of highly-efficient machines and devices that provide full automation and mechanization of technological processes.

The correct option of drying mode depends on the nutritional value and quality indicators of the finished product, which are the result of structural, mechanical, biological and physico-chemical transformations of substances. To implement the drying process of food plant raw materials effectively, it is necessary to study the nature of the moisture connection with the areas where substances are converted when the temperature rises. A rosehip and hawthorn fruit have been taken as the research objects. All samples have been pre-sorted in order to align the granulometric composition and ensure uniformity of the product structure.

Experimental part. Estimation of the thermal effect on food plant raw materials have been studied by non-isothermal analysis using a complex thermal analyzer TGA-DSC of Mettler-Toledo STARe [1,2,9] when the atmosphere had been heated up to the temperature of 423 degrees K, with a heating rate of 3 K/min. The thermal analyzer have been used to determine the product mass decrease and temperature increase during the controlled process in the given gas environment. This device includes an experimental module, a minicompressor, a thermostat, a rotameter, a gas controller, specialized software, and a personal computer that controls and regulates the device and processes the experimental data obtained. Experiments were carried out in duralumin containers, where a product with a total weight of 10.55 mg has been applied. The thermoanalytic curves used for quantitative processing by non-isothermal kinetics simultaneously register changes in temperature, sample mass, rate of temperature change or enthalpy, and mass changes (TA, TG, DTA, and DTG curves in Fig. 1). the Adjustment device made it possible to evenly heat the furnace, and achieving the linearity of the furnace heating program ensured the reproducibility of TA, TG, DTA, and DTG curves.

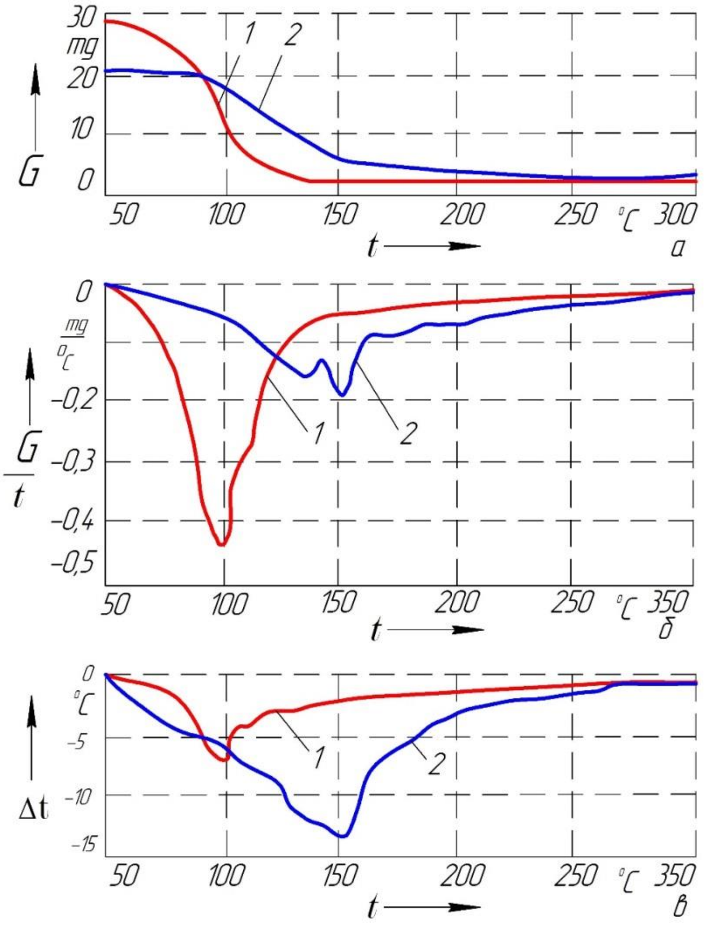

Fig. 1. Experimental change dependences in the sample TGA mass, the rate of DTA temperature change, and the rate of DTG mass change: 1-hawthorn, 2 – rosehip

The mode option for recording derivatograms has been determined using the method [4,9]. The following modes of derivatogram removal have been selected: the sensitivity of the DTA galvanometer is 1/10; the sensitivity of the DTG galvanometer is -1 / 5; the sensitivity OF the TG galvanometer is 500 mg; the rate of change in the furnace heating temperature is 3 °C / min; the maximum heating temperature is 300 °C.

In the heat exposure process, particles of the plant products undergo significant physico-chemical change, which releases water of a specific product and determines the nature occurring within the product during the transformations of substances. Due to moisture evaporation and sugar decomposition, fiber and other organic compounds (lysine, methionine), their mass is reduced by 65 ... 73 %. In this case, the strength of the structure is weakened due to partial hydrolysis of fiber, cellulose and other complex carbohydrates. The kinetic characteristics of the process is the initial temperature of the beginning of thermolysis T1 (deviation from the baseline of the DTA curve). The temperature T2 corresponds to the point of greatest deviation of this curve. The section of the increase in the DTA curve, starting from the peak, corresponds to the equalization of the temperature field of the samples to a new quasi-stationary state, which was previously disturbed by the thermal effect of transformation [19].

Quantitative evaluation of kinetically unequal water molecules in the product was carried out using experimental curves obtained by thermogravimetry. The section of the mass change curve corresponding to the dehydration process was transformed into a dependence of the degree of mass change or substance transformation on temperature. To do this, every 5 °C on the TG curve at certain temperature values, a change in the mass mi of the sample was found, corresponding to the amount of water released at temperature Ti. The degree of change in mass (α) was calculated as the ratio of mass mi to the total amount of water contained in the product (m), determined from the TG curve at the end of the dehydration process [18].

Obtained TG curves in the coordinates "α – T" are S-shaped, reflecting the complex interactions of water and dry matter of the product, and suggests a difference in the rate of release of water in different parts of the data curves (Fig. 2). Consequently, the curves of degree of conversion of a substance from temperature allow to study various kinetically not equivalent forms of communication of moisture and require different speed of dehydration.

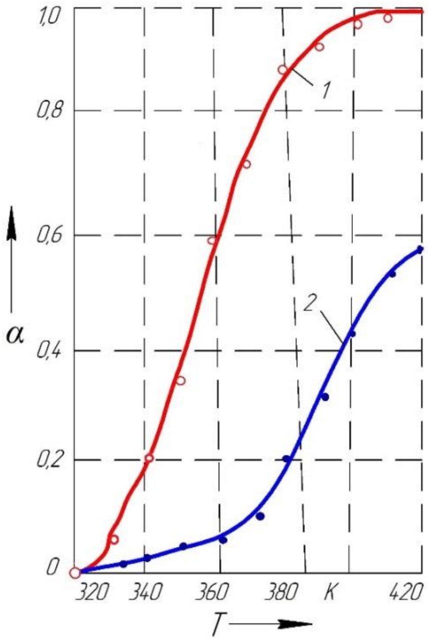

Fig. 2. Dependence of the degree of transformation α on the temperature T: 1-hawthorn when heated at a rate of temperature rise of 2 K / min; 2-rosehip when heated at a rate of temperature rise of 10 K / min

Derivatograms (Fig. 1) have characteristic temperatures of hydration stages, degradation of substances, and temperature intervals of stability of intermediate compounds determined by peaks of endothermic effects accompanied by evaporation of moisture and possible separation of gaseous fractions (table 1).

Table 1.

Temperature characteristics of the thermolysis process of plant products

Product	Kinetic characteristics		
	Temperature of the beginning of the endothermic effect, K	The temperature peak of the endothermic effect, K	Endothermic effect termination temperature, K
Rosehip	316…320	403	433…437
Hawthorn	304…308	368	378…382

To obtain data from the water removal mechanism based on the obtained curves, determine the temperature range and the amount of moisture desorbed at approximately the same rate, we used curves in the coordinates " (- lga) – (103/T)". The dependences (–lga) on the value (103 / T) (Fig. 3) are made for the range 298...458 K.

Figure 3 clearly shows three linear sections, which indicates a stepwise release of water. At the first stage, the "water – water" bond is destroyed - heating and removing "free" water mechanically and osmotically bound moisture, which has a low binding energy with the product (AC section). Water is released, forming an openwork grid of associates of water molecules connected by hydrogen bonds. During heating, part of the osmotically and immobilizationally bound moisture held in closed cells of protein micelles is released when their polypeptide chains are deployed at the evaporation temperature of adsorptively bound moisture as a result of violation of micellar and hydrophobic interactions of proteins and carbohydrates with water [17].

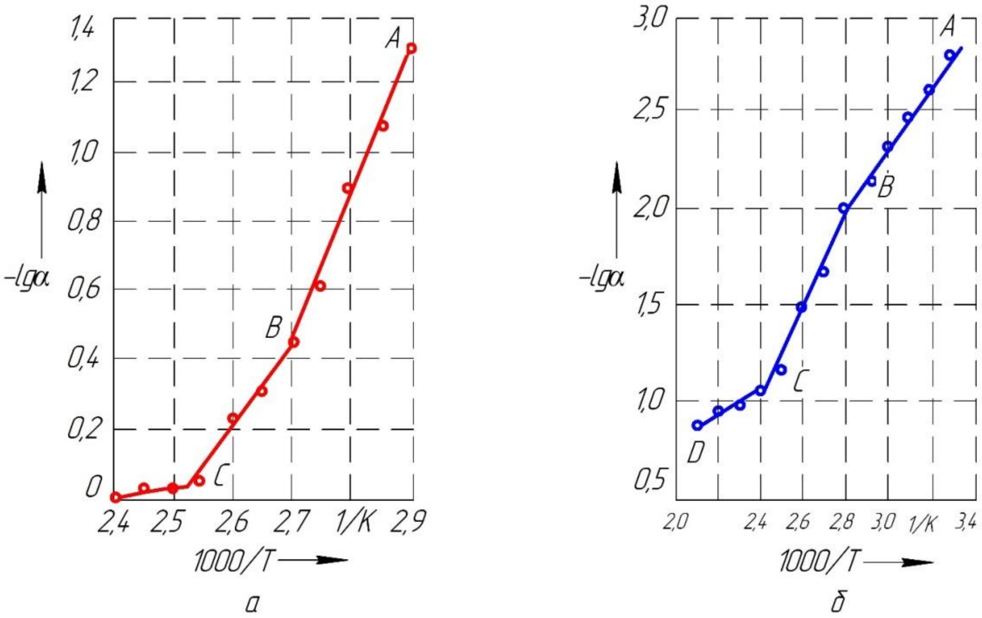

Fig. 3. Dependence of-lg α on the value of 103/T: a-hawthorn; b-rosehip

The water released at the second stage (intermediate) forms several subsequent layers of molecules that are more strongly bound to the product (CD section). These molecules are influenced by active groups of dry substances and have a more distorted structure compared to water in volume [7].

The peak of the endothermic effect on the DTG curve, accompanied by the end of intense mass loss, corresponds to the release of water molecules with a physicochemical bond. Further, the destruction of substances is observed, followed by thermal decomposition of the product (section DE).

Thus, the analysis of the data obtained made it possible to distinguish three periods of water dehydration and dry matter transformation under thermal influence on plant products, as well as to identify temperature zones that correspond to the release of moisture with different forms and binding energies. The effect on the DTA curve is accompanied by a change in mass (Tg curve) and an effect on the DTG curve, which allows us to determine the beginning and end of the enthalpy change [7].

On the DTA curve, a significant endothermic minimum is observed at temperature, which corresponds to the processes of product dehydration and is accompanied by a loss of sample mass, and is also associated with the transformation of substances and the onset of gaseous fractions.

At the first stage, water is released, forming an openwork grid of associates of water molecules connected by hydrogen bonds. During heating, part of the osmotically and immobilizationally bound moisture held in closed cells of protein micelles is released when their polypeptide chains are deployed at the evaporation temperature of adsorptively bound moisture as a result of violation of micellar and hydrophobic interactions of proteins and carbohydrates with water. The water released at the second stage (intermediate) forms several subsequent layers of molecules that are more strongly bound to the product (CD section). These molecules are influenced by active groups of dry substances and have a more distorted structure compared to water in volume [7].

Thus, the analysis of the obtained data allowed us to distinguish three periods of water dehydration and dry matter transformation under thermal influence on medicinal raw materials, as well as to identify temperature zones that correspond to the release of moisture with different forms and binding energies.

Data on structural changes in plant products during thermal decomposition are shown below (table 2).

Table 2.

Structural changes in the plant products during thermal decomposition

Product	Temperature values of plant products T, K	
	Extraction of the main mass of TB moisture, K	Beginning of degradation of product substances, TD, K
Rosehip	303…347	474…478
Hawthorn	303…368	432…436

The thermophysical characteristics of the studied products are functions of the state and properties of the substance, which depend on many factors, including the chemical composition and structure. For proper organization of the drying process, it is important to know the nature of changes in the thermal characteristics of products.

The method of non-stationary thermal regime based on the solution of the problem of thermal conductivity for the initial stage of the process, namely the method of two temperature-time points developed by V. S. Volkenstein [8,9], is used to determine the thermophysical characteristics.

The study of the dependences of the thermophysical characteristics of the studied types of products was determined on a measuring device for obtaining rheological and thermophysical characteristics of viscoelastic liquids of the Coesfeld RT-1394H brand (national Instruments).

The obtained experimental data were processed on a computer in the "Statistica-5.0" environment, and as a result, equations (1 - 6) were obtained. The values of the thermophysical characteristics of the studied rosehip and hawthorn fruits for the temperature range 293-353 K are shown in table 3.

The obtained experimental data were processed on a computer in the Microsoft Excel environment. as a result, equations (1 - 4) describing the thermophysical properties of the studied types of food plant raw materials were obtained, with the approximation confidence value R2 = 0.9892...0.999:

for the temperature range 293-353 K:

For rosehip:

by $W = 13{,}54$ %:

$$c = 3309{,}8+1{,}1188t\,;\ \lambda = 0{,}3115+0{,}0002t\,;\ a = 9{,}165+0{,}0032\times t\,; \quad (1)$$

by $W = 60{,}07$ %:

$$c = 3363{,}3+0{,}6338t\,;\ \lambda = 0{,}3405+0{,}0002t\,;\ a = 9{,}76+0{,}0031t\,; \quad (2)$$

For hawthorn:

by W = 13,23 %:

$c = 1359{,}7 + 3{,}5918\, t$; $\lambda = 0{,}083 + 0{,}0005\, t$; $a = 5{,}52 + 0{,}\ 0126\, t$;

(3)

by W = 82,2 %:

$c = 1664{,}6 + 3{,}3483t$; $\lambda = 0{,}1345 + 0{,}0004t$; $a = 7{,}585 + 0{,}0063t$ (4)

In formulas (3-6), the t values are given in °C. The data analysis shows that with increasing temperature, the specific heat capacity, thermal conductivity and thermal diffusivity coefficients of the studied samples of plant raw materials increase.

On the example of rosehip and hawthorn fruits, the values of these coefficients from temperature and humidity are obtained (table 3-4.)

Table 3.

Thermophysical characteristics of rosehip and hawthorn fruits

Temperature values, ° C	Samples of rose hips		Samples of hawthorn	
	W = 60,07 %	W = 13,54 %	W = 82,17 %	W = 13,23 %
	Thermal diffusivity, a=10^8, $м^2/с$			
20	9,82±0,02	9,23±0,02	7,73±0,02	5,78±0,02
40	9,89±0,02	9,29±0,02	7,83±0,02	5,99±0,02
60	9,95±0,02	9,36±0,01	7,91±0,02	6,32±0,02
80	10,01±0,02	9,42±0,02	8,12±0,02	6,51±0,02
Thermal conductivity, λ, Вт/(м×К)				
20	0,344±0,003	0,316±0,002	0,142±0,01	0,092±0,01
40	0,348±0,003	0,320±0,002	0,151±0,01	0,101±0,02
60	0,351±0,002	0,325±0,002	0,158±0,01	0,112±0,01
80	0,355±0,002	0,329±0,002	0,166±0,02	0,119±0,02
Mass specific heat, s, j/(kg×K)				
20	3376,0±0,04	3332,76±0,04	1723,0±0,01	1424,01±0,0
40	3389,2±0,04	3356,09±0,04	1804,5±0,01	1508,5±0,02
60	3401,1±0,04	3381,97±0,04	1879,0±0,01	1587,3±0,02
80	3414,6±0,04	3402,1±0,04	1921,4±0,01	1637,2±0,02

Table 4.

Values for the density of rosehip and hawthorn fruits

Product	Humidity W, %	Density, kg/m^3
Hawthorn	82,17	1065,6
	13,23	1173,4
Rosehip	60,07	1026,0
	13,54	1037,4

Conclusion. The obtained dependences of the coefficients of thermal conductivity, thermal diffusivity and heat capacity of food plant raw materials are necessary for physical and mathematical modeling of the drying process under variable heat supply.

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