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STUDYING THE PROPERTIES OF GRAPE POMACE AS OF AN OBJECT OF DRYING

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Abstract. Grape pomace contains a complex of valuable and biologically active compounds. Drying is one of the main ways of microbiological stabilisation and preservation of the nutritional value of this secondary raw material. Kinetic parameters of dehydration of grape pomace from different industrially cultivated and processed varieties have been studied, namely, of the red grape varieties Cabernet and Shiraz, and of the white varieties Chardonnay and Riesling. Dependences of the moisture content in the process of convective drying at different drying agent rates have been obtained at a regulated temperature of 80 °C. The components of such an important technological parameter as the drying time have been determined. These components include the duration of the constant drying rate period Φ_2 and the time of the decreasing drying rate period Φ_1 of the two zones of the second drying period. The coefficients of the dehydration process have been calculated depending on the type of grape pomace processing. It has been shown that the discrepancy between the calculated and experimental results does not exceed ±3.9%. The specific features of the moisture yield of the pomace have been revealed, the pomace being viewed as a complex heterogeneous system with colloidal and capillary-porous properties. There are different types of its technological preparation: it can be fresh, frozen, fermented, and this makes for the fact that the drying time and drying rate may differ by 1.32–1.46 times. High preservation of valuable properties of grape pomace has been shown. Thus, the concentration of biologically active substances (BAS) in the total polyphenolic compounds is up to 69% of their initial concentration in the grape pomace samples, and the microbial contamination of the samples after drying is reduced by 51–82% of their initial contamination.

Key words: grape pomace, convective drying, regression equations, moisture yield characteristics, biosafety, microbiological stability.

ДОСЛІДЖЕННЯ ВЛАСТИВОСТЕЙ ВИНОГРАДНИХ ВИЧАВОК ЯК ОБ'ЄКТА СУШІННЯ

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Анотація. Вивчено кінетичні параметри зневоднення виноградних вичавків із різних промислово вирощуваних і перероблюваних сортів – ягід червоних сортів винограду Каберне і Шираз, білих сортів Шардоне і Рислінг. Отримано залежності показника вмісту вологи в процесі конвективного сушіння з різними швидкостями сушильного агента за регламентованої температури 80°C. Визначено складові важливого технологічного параметру – часу сушіння, який включає тривалість періоду постійної швидкості процесу сушіння Φ_1 і час періоду спадаючої швидкості сушіння Φ_2 двох зон другого періоду висушування. Розраховано коефіцієнти процесу зневоднення залежно від виду технологічної обробки виноградних вичавків і показано, що розбіжність між розрахунковими і експериментальними результатами не перевищує ±3,9%. Виявлено особливості вологовіддачі вичавків як складної гетерогенної системи, яка володіє колоїдними і капілярно-пористими властивостями, з різними видами технологічної підготовки: свіжої, замороженої, збродженої, що обумовлює відмінності в тривалості і швидкості сушіння в 1,32–1,46 разів. Показано високе збереження цінних властивостей виноградних вичавків. Концентрація біологічно активних речовин (БАР) за сумарними поліфенольними сполуками становить до 69% до первісної їхньої концентрації в зразках виноградних вичавків, а мікробна контамінація зразків після висушування знижується на 51–82% до їх вихідного обсіменіння.

Ключові слова: виноградні вичавки, конвективне сушіння, рівняння регресії, особливості вологовіддачі, біобезпека, мікробіологічна стабільність

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Introduction. Formulation of the problem

Grapes are widely distributed in the Mediterranean and Black Sea regions. The grape varieties most suitable for obtaining juice and grape wines are grown in the coastal areas of the Kuban, Ukraine (Odessa and Kherson regions), Moldavia, France (Beaujolais, Burgundy) [1,2]. The chemical composition of grape berries depends on climatic conditions, soil composition, and varietal characteristics [3,4]. During processing, a significant part of the grape pomace is formed. It contains valuable food ingredients and biologically active substances, due to which they are of considerable interest for various branches of the food industry, as well as for other types of industry [5,6].

The paper considers the problem of rational processing of grape pomace using methods that optimise moisture removal. The research is based on the data obtained about the structural and mechanical properties of grape pomace, on the ratio between the forms of free and bound moisture.

Analysis of recent research and publications

Scientific and technical literature presents broad analysis of how grape pomace is processed in different countries. It is known that the pomace that is formed in the course of separation of grape juice can be quickly damaged by microorganisms and become unusable. Most technological solutions deal with the preparation of grape pomace for further processing. Thus, for example, a variant was suggested of preparing grape pomace for extracting valuable components by the enzymatic hydrolysis method [7]. This method allows increasing the yield of extractive substances from pomace. In order to maximise the antioxidant properties of the thermolabile components of pomace, the gentle drying method was suggested [8]. A detailed analysis of the drying characteristics of grape pomace by the convection method was carried out [5,9-12].

A quantitative chemical analysis has been described, as well as biotesting of products of sweet red grapes processing (Cabernet variety) [3]. Powder and extracts can be obtained from the secondary resources of processing grapes and used in bakery and confectionery products [13-15]. Considerable attention has been paid to the analysis of the physicochemical characteristics of grape pomace [4]. Foreign experts in winemaking have assessed the chemical composition of seeds and skins of red grapes from Libya and other countries [1,2,6,16]. Special aspects of drying physically pretreated Perlette grapes are given in [17]. A way of determining the end of the drying process in plant objects by the intensity of chemiluminescence decay was described in [20], and it was confirmed by the results of biotesting [21]. However, the existing methods of grape pomace processing do not allow obtaining high-quality food products due to using inadequate equipment, high temperatures, and long duration of pomace drying.

The aim of the work was to study the kinetic parameters of the dehydration process of the pomace of red and white grape varieties pre-prepared in different ways (fresh, fermented, frozen) to obtain a dried product with high-level consumer properties and nutritional value.

The purpose of the work made it necessary to solve these basic **tasks**:

- to investigate the rate of grape pomace drying at different drying agent speeds and a fixed temperature;
- to characterise the kinetics of the drying process by the proportion of moisture forms in the objects of drying, and calculate the coefficients that determine the duration of the process;
- to characterise the safety of the products obtained and the preservation of BAS in the dried products.

Research Materials and Methods

The objects of the research are grape pomace that were formed at canneries and wineries in the course of processing grapes of different varieties. Grape pomace of industrially cultivated red grape varieties (Cabernet, Shiraz) and white varieties (Chardonnay, Riesling) was investigated.

The red grape varieties (Shiraz, Cabernet) after fermentation were processed on a screw press to select the must (yield 70%). Pre-prepared freshly harvested Chardonnay grapes were processed on a membrane press, yielding about 20% of the pomace and 80% of the must.

Convective drying was studied on a bench-scale ring dryer by Sidorenko’s method [11]. In the work, modern methods were used to plan the experiment mathematically [10,18], to evaluate the proportion of free and bound moisture in the grape pomace of the varieties under study [5,9], and to determine the moisture content in the grape pomace at the initial, intermediate, and final drying points [5,10]. The equilibrium properties of plant objects in the hygroscopic region were determined by mathematical modelling [18,19]. The end of the drying process of plant objects was determined by the intensity of chemiluminescence decay [20]. The microbial contamination of the samples was determined by the number of mesophilic aerobic and facultatively anaerobic microorganisms (MAFAnM) [11, 21]. The BAS retention was studied for the phenolic complex of samples (concentration of total polyphenolic substances), and the integral security of dried products was confirmed by the results of biotesting [21].

Results of the research and their discussion

Convective drying of the grape pomace of red (Cabernet and Shiraz) and white (Chardonnay and Riesling) varieties was carried out on a bench-scale ring dryer by the method developed by O. V. Sidorenko. The dryer (Fig. 1) consists of a drying chamber (1), an electric air heater (2), and a fan (3). They are connected to each other by air ducts (4) in which movable shutters (5) are installed to adjust the air speed in the drying chamber. Inside the drying chamber, there is a movable frame (6) which is connected to one of the dishes of electronic scales (7). A metal mesh box (8), with a width of 120 and a length of 420 mm (the cells are 6x6 mm, the diameter of the wire 0.6 mm), was set on the frame, and into it, a grape pomace sample with a layer thickness of 10 mm was placed.

To measure the rate of the material's loss in weight, the time was tagged with a stopwatch every time the mass of the material to be dried reduced by 2 g (±0.1 g). The experiment was finished with the achievement of a state close to equilibrium, which was defined as the state when the material ceased to lose in weight. The intensity of chemiluminescence decay was an additional indicator to determine the appropriate moment of the end of drying.

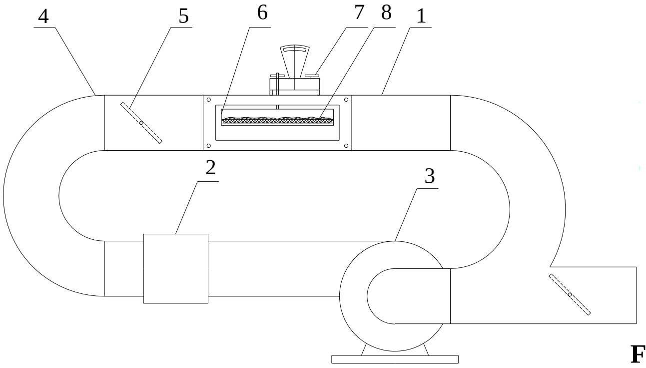


Figure 1. Ring dryer scheme

Figures 2-3 illustrate the dependencies of the duration of convective drying on the rate of the drying agent $N(\tau)=dU/d\tau$. The rate of the drying agent varied from 6.0 to 11.0 m/s at a temperature of 80°C. According to the experiments, a drying agent rate over 11.0 m/s is undesirable, as the particles of the material to be dried start being drawn away. It is known that with an increase in the temperature of the drying agent (hot air), the oxidative processes intensify sharply. This is due to the fact that there is no skin to protect grape pomace flesh from contact with air oxygen, unlike, for example, when whole grape berries are dried to obtain sultanas, raisins, or Zante currants as the final product. As recommended, the maximum temperature of the drying agent does not exceed 80°C [11]. The increase in the temperature significantly worsens not only the nutritional qualities of the resulting dry grape pomace due to the caramelisation of sugars, the thermal destruction of biologically useful substances, and the deterioration of the appearance. It also intensifies the oxidative processes of lipids in grape seeds, which worsens the quality of the grape-seed oil obtained [3]. On the other hand, a decrease in the temperature below 70°C activates the enzyme system, including lipase, which leads to an increase in the acid number of the oil [4].

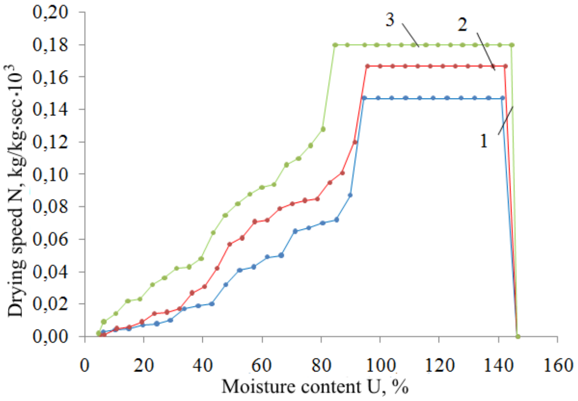


Figure 2. Rate curves of convective drying of the Shiraz grapes pomace at different rates of the drying agent (air temperature t = 80°C): 1 – 6.0 m/s; 2 – 8.5 m/s; 3 – 11.0 m/s

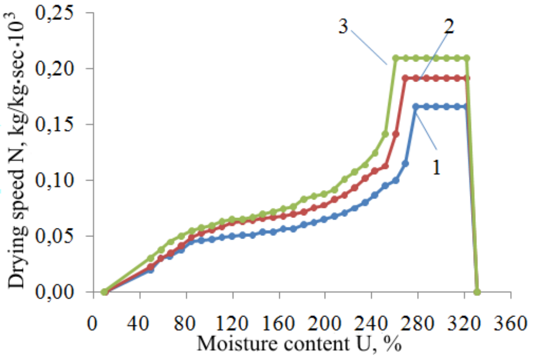


Figure 3. Rate curves of convective drying of the Riesling grapes pomace at different rates of the drying agent (air temperature t = 80 °C): 1 – 6.0 m/s; 2 – 8.5 m/s; 3 – 11.0 m/s

Research experiments have confirmed that lowering the lower limit of the rate and the temperature of the hot air or gas is economically impractical, because it lengthens the drying time and ends in extra energy costs.

As shown in Fig. 2-3, in the initial drying period, the moisture content changes insignificantly, and the dehydration rate rises to the maximum. This corresponds to the stage of warming up the grape pomace. The line of constant drying rate is characterised by a linear decrease in the moisture content in the raw material. The free moisture is removed from the surface layer of the grape pomace, and the rate of dehydration depends on the parameters of the drying agent. At the end of this drying period, the moisture content is at a critical level. For the pomace samples selected, it ranges 73 to 87% of absolutely dry matter.

In the second period, a change in the S-curves for the pomace samples under study is observed. This allows distinguishing the two zones of bound moisture, with the second critical moisture content. In the first zone, the initial drying rate decreases along a curve with a convexity towards the abscissa axis (the moisture content axis), which is characteristic of dehydration of capillary-porous bodies. In the second zone, the drying rate changes along a line curving towards the ordinate axis, which characterises the drying of colloidal materials. Thus, Lykov's classification is valid when applied to the colloidal-physical properties of moist grape pomace which is a complex system as it has both colloidal and capillary-porous properties.

The resulting graphical dependences are equivalent to the initial, intermediate, and final values of the moisture content. The kinetic characteristics at constant drying rates in the first period and the drying rate decrease curves in the 1st and 2nd zones of the second drying period differ significantly in their numeric values, too (Table 1).

An increase in the rate or the temperature of the hot air (gas) shifts the first critical point of the moisture content towards a decrease in the moisture content.

Table 1 – Changes in the moisture content in the grape pomace at the initial, intermediate, and final points of the drying process

Pomace of the grape variety	Drying process parameters				
	Drying agent rate, υ m/s	Initial moisture content, $U_{0\%}$ mass	Critical moisture content 1,U_{cr1}% mass	Critical moisture content 2, $U_{cr2\%}$ mass	Equilibrium moisture content 2, $U^{*\%}$ mass
Shiraz	6.0 8.5 11.0	146.33	94.29 91.24 86.56	55.0 35.0 30.0	6.11 5.20 4.49
Chardonnay	6.0 8.5 11.0	167.95	103.64 98.29 92.93	28.6 23.3 20.5	6.84 5.75 4.93
Cabernet	6.0 8.5 11.0	128.0	91.52 82.40 73.28	39.08 34.52 29.96	4.51 4.03 3.52
Riesling	6.0 8.5 11.0	340.82	287.18 278.37 269.55	140.5 141.1 141.7	9.02 9.21 9.34

The previously obtained data [5,12] about the change in the curves of the experimental equilibrium isotherms (with a wet material as the drying agent) confirm this effect. A comparison of the drying rate curves at different temperatures shows that the equilibrium moisture content and the critical moisture content decrease when the temperature increases.

The decrease in the value of critical moisture content 1 is accompanied by an increase in the proportion and amount of moisture removed in the first drying period. This effect is explained as follows.

With the increase in the drying potential (defined as a difference in the temperature between the dry and wet thermometers), or due to a temperature rise of the dry thermometer from 60°C to 80°C, or due to a temperature drop on the wet thermometer, in line with an increase in the drying agent rate (6.0–11.0 m/s), drying on the surface of and inside the grape pomace layer becomes non-uniform, less even. The reason for this is the unevenness and complexity of its structural composition. With high rates of the hot air, drying leads to cracking and flaking of skin fragments on the surface of the grape pomace layer. Due to the cracks, new exposed surfaces of the dried raw materials appear. In this connection, part of the internal (bound) moisture turns into the surface moisture (not bound). As a result, the amount of moisture evaporated in the 1st period increases, the first critical moisture content decreases, and the line of the constant drying rate increases with a shift to the left, towards a decrease in the moisture content. Thus, it is advisable in the drying chamber to implement the technologies that will ensure the stirring and mixing of the moving layer of grape pomace, which will lead to an increase in the efficiency of evaporation of moisture both in the first and in the second drying periods. Flaking and the formation of cracks on the surface of the upper layer of grape pomace can also explain the decrease in the value of the second critical moisture content.

In practice, in most cases, – for example, when drying grain, – the formation of cracks sharply reduces the quality of the finished product and leads to its destruction [18,19]. However, when drying grape pomace, the formation of cracks and flaking of the material must be viewed as a positive side of the drying process, since this phenomenon reduces the overall drying time, which, in turn, helps preserve the quality of drying.

As the object of our research, dry grape pomace is to be ground into powder that has a much longer shelf life, and can be used as a valuable food additive in bakery.

An important technological parameter of drying is the total drying time, which includes the duration of the constant drying rate Φ_1 and the time period $\Phi_2$of the decreasing rate of drying [11]

$$\Phi_{tot}=\Phi_1+\Phi_2=\Phi_1+\Phi_{21}+\Phi_{22} \quad (1)$$

Where $\boldsymbol{\Phi_{21}}$ and $\boldsymbol{\Phi_2}$ correspond to the drying rates in the 1st and the 2nd zones od the second drying period.

The drying time needed to achieve the given moisture content U is calculated (for the first drying period), depending on the rate of the drying agent, by the formula:

$$\Phi_1=(U_0-U)/N_1 \quad (2)$$

Where $\boldsymbol{U}$ and $\boldsymbol{U_0}$ are respectively, the initial moisture contents of grape pomace,% of the mass;

$\boldsymbol{N_1}$ is the drying rate of the first period,%/min. Their calculated dependencies are presented below.

The second period is characterised by a decrease in the drying rate. The temperature of the material rises, approaching the temperature of the hot air (gas). That is why, the reduction in the drying rate in the second period is due to a change in the form of the connection between the moisture and the material, i. e. a change of the limiting stage of the drying process. If the drying rate in the first period was determined by the heat transfer conditions of the drying agent, the drying rate in the second period is determined by the conditions of the diffusion of moisture and moisture vapour in the pores and capillaries of the dried grape pomace. As the moisture evaporation zone deepens, the diffusion path from the evaporation zone to the surface of the material lengthens. Accordingly, the internal mass transfer coefficient and the drying rate are reduced. The latter is now limited by this complex process that depends on the structural, mechanical, and thermophysical properties of the material being dried. It should be noted that for all rate curves of drying the grape pomace of the varieties under study, in the second period, the nature of their change is similar. Due to the fact that for the second drying period of colloidal capillary-porous systems, there is no generalised and reliable calculation theory, it is recommended to calculate such an important technological parameter as the drying time on the basis of experimental data obtained for each kind of the material to be dried [5].

For the 1st and the 2nd zones of the second drying period, linear dependences of the drying rate on the current moisture content have been obtained:

$$N_{21} = A_{21} + B_{21} \cdot U \quad (3)$$

$$N_{22} = A_{22} + B_{22} \cdot U \quad (4)$$

After mathematical processing, we obtained general logarithmic expressions for calculating the drying time in the 1st and 2nd zones of the second period of the grape pomace of the varieties under analysis:

$$\tau_{21} = \frac{1}{B_{21}} \cdot \ln \frac{A_{21} + B_{21} \cdot U_{cr1}}{A_{21} + B_{21} \cdot U} \quad (5)$$

$$\tau_{22} = \frac{1}{B_{22}} \cdot \ln \frac{A_{22} + B_{22} \cdot U_{cr2}}{A_{22} + B_{22} \cdot U} \quad (6)$$

Where A_{21}, A_{22}, B_{21}, B_{22} are coefficients that are calculated from the dependencies presented in Table 2. they are obtained from our own experimental results for each variety of grape pomace with all temperature conditions of drying;

U_{cr2} is the critical moisture content separating the 1st and 2nd of the second period,% mass.

The discrepancies between the calculated values from Eqs. 5,6 and the experimental results range ±8.5 to 16.6%/

Table 2 – Dependences of the coefficients in the equations (5 and 6) for calculating the drying time

Grape variety	$\boldsymbol{B_{21}}$	$\boldsymbol{A_{21}}$	$\boldsymbol{B_{22}}$	$\boldsymbol{A_{22}}$
Shiraz	4.4·ν – 12.4	0.856 + 0.0154·ν	0.04 + 0.64·ν	0.184·ν –0.464
Cabernet	5.92 + 1.392·ν	0.13 + 0.146·ν	3.22 + 0.023·ν	–(0.525 + 0.11· ν)
Chardonnay	9.4 + 0.627·ν	–(0.235 + 0.02·ν)	29.26 + 0.24·ν	–(0.66 + 0.011·ν)
Riesling	7.0 + 0.6·ν	0.2732 + 0.0228·ν	–(6.86 + 0.02·ν)	0.56 + 0.08·ν

For each variety's pomace – Cabernet (A_1= 0.061; B_1 = 0.008), Chardonnay (A_1= 0.075; B_1= 0.0076) and Riesling (A_1= 0.115; B_1= 0.009) – a linear equation in the general form has been obtained:

$$N_1 = A_1 + B_1 \cdot v, \quad (7)$$

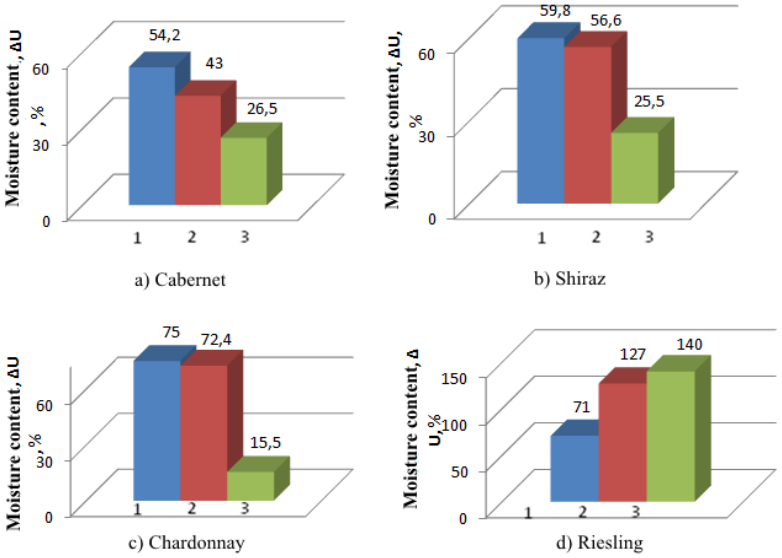
Where the hot air rate (with t = 80 °C) is v = 6.0 – 11.0 m/s.

The discrepancy between the calculated values of the equation (7) and the experimental results does not exceed ±3.9%.

When comparing the technological and kinetic parameters of convective drying of grape pomace with its efficiency, the following is evident. For the fermented pomace of red grapes (Fig. 4, 1 – chemical, 2 – physico-chemical, 3 – physico-mechanical form of the connection of moisture with the material), the content of free and bound moisture is approximately comparable, but the content of capillary-porous and colloidal moisture in the pomace obtained from frozen Riesling grapes is, respectively, almost 2 and 9 times more than in fresh sweet pomace of Chardonnay grapes.

When grape pomace is dehydrated to the 10% moisture content, the drying time is calculated according to the equations (1, 2, 5, 6). For the pomace (after fermentation) of the Cabernet and Shiraz varieties, it is, respectively, 27.8 min and 32.9 min, for fresh raw materials 53.2 min, and for frozen grapes 92 min. The time of the first drying period for the grape pomace of the varieties in question was 5.4–7.9 minutes.

Kinetic parameters, too, noticeably differ. For example, the drying rate in the first period (at υ=11 m/s и t=80°C) for the grape pomace of the Riesling variety is 1.32 times greater than for the Chardonnay variety, and for Shiraz and Cabernet varieties, the drying rate is, respectively, 1.43 and 1.46 times greater.



a) Cabernet b) Shiraz

c) Chardonnay d) Riesling

Figure 4. Dependence of the moisture content on the form of bound and not bound moisture in grape pomace of the varieties: a) Cabernet; b) Shiraz; c) Chardonnay; d) Riesling

Conclusions

The work shows a possibility of increasing the efficiency of drying grape pomace, according to its structural and mechanical properties, to the ratio between free and bound moisture, and to the peculiarities of grapes processing. Specific features of the moisture yield of the pomace have been revealed, the pomace being viewed as a complex heterogeneous system with colloidal and capillary-porous properties. There are different types of its technological preparation: it can be fresh, frozen, fermented, and this makes for the fact that the drying time and drying speed may differ by 1.32–1.46 times. For fermented red grape varieties with different types of connection between the moisture and the material, the content of free and bound moisture has been established to be approximately the same. However, in frozen grape pomace, the content of capillary-porous and colloidal moisture is much larger (by almost 2 and 9 times respectively), than that in fresh sweet pomace. The coefficients of the process have been calculated depending on the type of grape pomace processing. It has been shown that the discrepancy between the calculated and experimental results does not exceed ±3.9%.

The studies confirmed that the drying of the grape pomace to a moisture content of less than 9.5–10.0% is impractical, due to the biochemical changes observed in the objects of drying by the reaction of the cells of the raw material dried by the chemiluminescence methods. The intensity of chemiluminescence decay was an additional indicator to determine a reasonable moment for the end of the drying process, either for grapes or for other plant objects. These indicators were also confirmed by the results of biotesting and by studying the BAS retained in the phenolic complex of samples – the concentration of total polyphenolic compounds is up to 69% of their original amount in the grape pomace samples. Besides, a positive characteristic of the pomace quality after drying is a 51–82% decrease in microbial contamination of the samples. Thus, valuable consumer properties, safety, and microbiological stability of the dried products obtained have been established.

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