DEVELOPMENT OF A VIBROCENTRIC MACHINE FOR RAW GLYCERIN PURIFICATION

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Based on the analysis, it has been established that the existing glycerin purification technology is characterized by low productivity and high energy intensity of the process. To intensify this process, a vibrocentric machine for cleaning glycerin was developed, which combines the implementation of centrifugal filtration and vibration deposition of raw materials. As a result of experimental research the effective working modes of this machine are substantiated. In particular, the angular velocity of the driving shaft is 110-120 rad·s^{-1}, the angular speed of the rotor is 120 rad·s^{-1}, the amplitude of the vibration of the container is 4-4,5 mm.

According to these parameters, the energy consumption is 1.4 kWh. It is proved that glycerin purification is rationally carried out at a temperature of 60-80 °C and the duration of vibrocentric influence is 300-420 s. The before mentioned parameters allow to obtain a product with a mass fraction of pure glycerol 88,5-89,5%. Provided that the process of initial purification of glycerin is increased 4 times to 90-94 kg·h^{-1}.

Keywords: vibrocentric machine, complex influence, primary cleaning of glycerin, regime parameters of the machine, raw material quality indexes.

1. Introduction

Pharmacopoeia or distilled glycerin gains increasing demand in confectionery, microbiological, pharmaceutical, enzymatic and other processing industries. In pharmaceutical practice, glycerin is used to make a wide range of dosage forms, namely: solutions, syrups, elixirs, mixes, suspensions, emulsions, ointments, pastes, candles, etc. It is also used as a drug with a variety of pharmacological effects. Glycerin is a good solvent of iodine, tannin, bromine, phenol and mercury chloride. By using glycerin instead of water, you can prepare highly concentrated medical solutions. Glycerin also has antiseptic properties, which is why it is used to prevent wound infections. The antiseptic and preservative properties of glycerin are associated with its hygroscopicity through which the dehydration of bacteria passes. It is a part of many cosmetic products. A large amount of glycerin is used to prepare nitroglycerin, which has antianginal, vasodilator,

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coronary stimulating effect. In the food industry, glycerin is used to prepare extracts of tea, coffee and other plant substances. This product is widely used in the production of soft drinks. Glycerin is also used in the food industry for sweetening wines and liqueurs, for the manufacture of mustard, jelly and vinegar. It is also used to produce surfactants used as additives that enhance the quality of finished products [1].

In the production of food (pharmacopoeial) glycerin, a number of problems arise that have a significant effect on the productivity of the specified production. In particular, raw glycerin contains an unlit organic residue, fats, acids, salts, and ash, which greatly complicate the process of its purification without a long-term advocacy.

Among the main methods for separating liquid inhomogeneous raw materials, gravity, centrifugal and electrostatic deposition methods were favored. However, these methods are characterized by high cost and the complexity of equipment designs for their implementation. This problem is proposed to be solved by applying a vibrocentric method of purifying raw glycerin. As a result of the application of such combined effects of centrifugal forces and vibration action, it is planned to increase the productivity of the separation process of a liquid inhomogeneous system, provided that the utilized input costs are reduced for its processing. In addition, the simultaneous application of a filtering rotor will not only intensify the process of purifying the raw glycerin, but also provide its partial lightning.

2. Technology for glycerin purification

At present, in the agricultural industry, two main technologies can be identified, in which glycerin is produced: in the process of biofuel and soap production [2-5].

In the production of biodiesel fuel, it is possible to use several technologies. The most efficient biofuel production technologies are cyclic with the use of catalysts and continuous multi-reactor technology. When producing biofuels from plant raw materials, in particular rape, the yield of glycerin is 15% [6-8].

In the process of soap production, subsoap alkaline is formed that contains 83% water, 10% salt and 4-7% glycerol. Further processing involves the dehydration of the subsoap alkaline by the evaporation plant, centrifugation for salt separation and purification operations of the selected glycerin by mechanical and physical mechanical treatment. When glycerinis obtained by means of known methods, its concentration is 80-85%, indicating the need for its further purification and concentration. As the use of glycerin in the medical and food industry requires a higher concentration of 99,5%.

As a result of the application of one of the above technologies, raw glycerin is obtained. However, to obtain distilled (pharmacopoeial) glycerin, raw glycerin requires additional cleansing. The basic scheme of the processing of raw glycerin is presented in Figure 1.

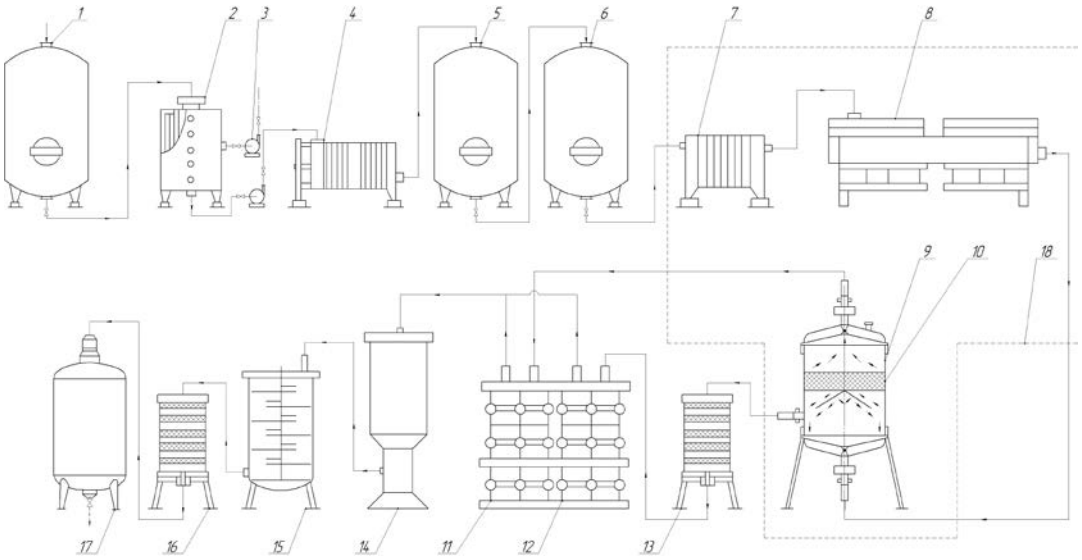


Fig. 1. Technological scheme of the process of obtaining distilled (pharmacopoeial) glycerin from raw glycerin (vegetable origin): 1 - tank for long-term storage of glycerin; 2 - tank for heating glycerin; 3 - bubbler pump; 4 - press filter; 5 - storage tank; 6 - tank for preparation of glycerin for distillation; 7 - heat exchanger; 8 - drying plant; 9 - separator; 10 - the demister; 11, 12 - condensators; 13 - thin-film apparatus; 14 - deodorizer; 15 - tank with mixer; 16 - filter; 17 - tank for loading off; 18 - distiller

The raw (untreated) glycerin is fed into a storage tank 1 for prolonged storage, in which the first stage of processing is carried out by mechanical deposition. For further purification of glycerin, it concentrates to a tank of at least 80%. Such glycerin requires mechanical treatment using chemicals (aluminum sulfate $Al_2(SO_4)$), which is the second stage of treatment with the use of the absorption process. From the long-term storage tank, partially refined glycerin is poured into a tank of 2, and heated to a temperature of 80-120 °C by steam, which is fed into special heat exchangers. Then aluminum sulfate is added to the working power, intensifying this process by means of pneumatic mixing. Subsequently, the raw material obtained with the help of a centrifugal pump is fed to a press filter 4, from which the glycerin substance merges with the storage tank 5. The next step in glycerin purification is molecular distillation. Pre-added to the mixture is caustic soda (NaOH), which greatly improves the separation conditions.

The amount of caustic soda depends on the quality of the glycerin itself and is determined by the laboratory method. From the tank 6 glycerin enters the heat exchanger 7, where it is heated to a temperature of 150 °C. The warmed glycerin is then fed to the drying unit 8, where the instantaneous evaporation of water passes. Thus, the thermophysical processing stage is carried out. Dehydrated glycerin is fed to a separator 9 pump. At high temperatures (150 °C), a vapor of purified glycerin

under vacuum acts through the demister 10 and enters the cooling condenser 11. The rest of the glycerin stands in the bottom of the separator and the screw pump is taken to the thin-film apparatus 13 for further purification. Next, glycerin is fed to a second cooling condensator 12. From both condensators, glycerin enters deodorizer 14, where it is purified from unwanted odors; after which it is directed to the storage tank 15. This tank contains activated carbon, the quantity of which is determined by the properties of the processed glycerin. In order to improve the adsorption of glycerin, the adsorbent coal is turbulized with stirrers or an oscillating support surface.

An important problem in the production of glycerin is the first stage of purification, in which the process of mechanical upkeep occurs. This process takes two about three months, depending on the quality of the original product. Raw glycerin is loaded in a tank with a capacity of 20-30 m^3, where after a certain period its bundle is divided into three parts. The most undesirable part of the separated raw material is the lower and upper parts, in which the salts accumulate, resinous substances (non-volatile organic residue or tar) and others. There are cases when an unplanned order of food glycerin in large quantities comes into production, then raw glycerin does not undergo primary purification (advocacy), but immediately goes to the second stage. In such cases, the greatest problem is the cleaning of non-isolated glycerin, since the presence of tar, salts and other unwanted impurities greatly complicates the entire subsequent distillation process (periodic filling of filters, the need for additional time for subsequent purification steps, increase of chemical reagents, etc.). Therefore, it is expedient to intensify the method of cleaning glycerin in the first stage of its production.

3. Equipments and methods

On the basis of the analysis of technological dissipation of the purification process of raw glycerin, a vibrocentric machine was developed [9]. The design and principal scheme of this machine is presented in Fig. 2.

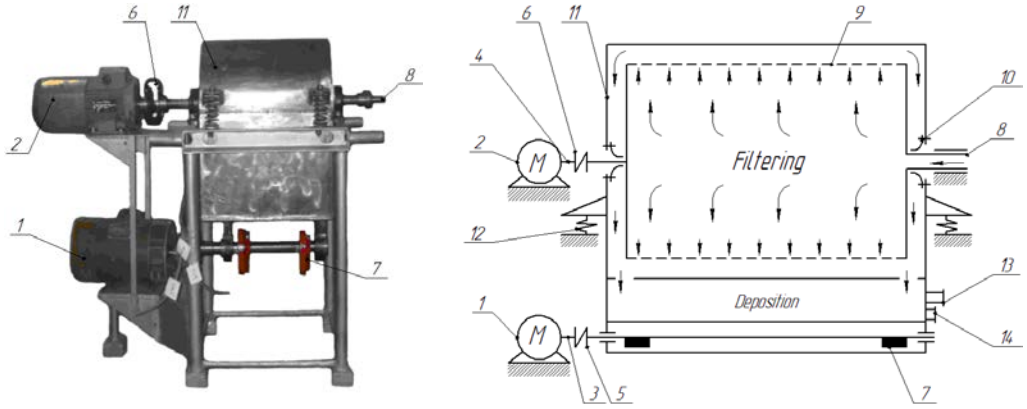
The main structural elements of a vibrocentric machine include two electric motors 1, 2, drive shaft 3, on which the elastic coupling 5 and the balancers 7 are mounted, drive shaft 4 on which an elastic coupling 6 is mounted that transmits a torque to a hollow shaft 8 on which the perforated rotor 9 is mounted; elastic elements 10, which allow leveling the transmission of vibrations on the shaft 8, tank 11 connected to elastic elements 12, drain pipes 13, 14, reservoir 15. The sidewall of the tank is transparent, which makes it possible visually to observe the process of vibration dampening. Using the developed mechanisms of regulation of balancers 7, elastic elements 12 and electric motors 1 and 2, it is possible to adjust the working parameters of the investigated vibration machine.

It is worth noting that the tank and perforated rotor is made of stainless steel, suitable for use in the food and processing industry.

In the process of purifying, the fraction of the technological mass under the action of centrifugal forces pass through a perforated drum and press against the inner walls of the container. As a result of such a combined technological effect of the rotary motion of the perforated rotor and of the vibrational movement of the working container, it is possible to substantially improve the effect of cleaning the raw glycerin.

The principle of the machine is to combine the filtration of raw glycerin under the action of centrifugal forces and its deposition as a result of the action of vibration. As a result of the rotation of the perforated rotor, the process of separating glycerin into a liquid fraction and a precipitate is carried out. After that, the fluctuations of the container ensure that the glycerin is removed from the resulting precipitate.

Fig. 3 shows the design of the drive mechanism. It consists of the filter rotor drive and the vibroexciter. Both drives are driven by electric motors of direct current, which makes it possible to control the speed from 0 to 120 rad·s^{-1}. In order to avoid unwanted vibration of electric motors in the drive system of the rotor and vibration drive, an elastic coupling is provided. Which transmits the torque from the electric motor to the drive shaft of the vibrator and locates the vibrations of the container itself.



a) b)

Fig. 2. Construction a) and the basic scheme b) vibrocentric machine for raw glycerin purification: 1, 2 – electric motors; 3, 4 – drive shafts; 5, 6 – couplings; 7 – balancers; 8 – hollow shaft; 9 – perforated drum; 10 – elastic spring elements; 11 – container; 12 – elastic elements; 13, 14 – pipes for the discharge of purified raw materials; 15 – separator

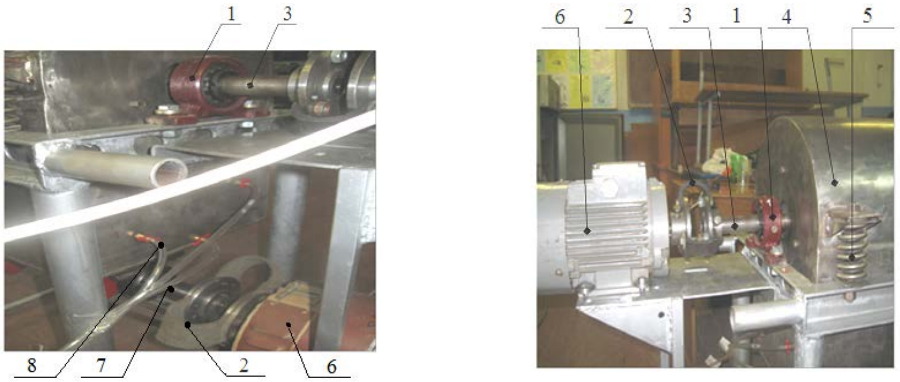


Fig. 3. Drive mechanism of the experimental vibrocentric machine: 1 – bearings; 2 – elastic coupling; 3 – driven rotor shaft; 4 – the tank; 5 – elastic elements; 6 – electric motors; 7 – drive shaft with balances; 8 – pipes for purified raw materials; a – vibration drive of the container; b – drive filter rotor

The structural and technological parameters of a developed vibrocentric machine for cleaning raw glycerin are shown in Table. 1

Table 1

Constructive parameters of the vibrocentric machine

Parameters	Units of measurement	Value
Volume of the tanks, Vc	dm^3	23
Working volume of the rotor, V_R	dm^3	8,36
Vibration accelerator shaft rotation frequency, n_R	min^{-1}	1500
Rotational speed of rotor shaft, n_R	min^{-1}	3000
Power of the vibroexciter drive	kW·h	1,1
Power of rotor drive	kW·h	0,55
Productivity	$dm^3 \cdot h^{-1}$	90
Consumed power consumption	kW·h	1,65

To determine the amplitude-frequency characteristics, an accelerometric analyzer based on the STMicroelectronics accelerometer LIS3DH was used. The principle of operation of the developed device is that after connecting the sensor to the surface of the container, the actuating mechanism is activated, creating vibrational changes of the executive body. These oscillations result in the automatic activation of the built-in accelerometer, which begins recording the amplitude-frequency characteristics of the machine under study.

To analyze the frequency of rotation of the drive shaft, the UNI-T UT372 tachometer was used. Then, the control of the rotational speed of the electric motor shaft was carried out using the autotransformer AOSN-20-220-75 [10].

The power characteristics of the machine under investigation were investigated using the EMF-1 electronic wattmeter.

Establishing the percentage of mass fraction of pure glycerin is as follows. Place the glycerin sample in a volumetric flask of 500 cm^3. Glycerin in the flask is diluted with distilled water to the separation. From the solution obtained by pipette, we take 25 cm^3 of solution and transfer it to a conical flask with a polished cork. To the selected sample add pipette 25 cm^3 of solution of iodine acid and after mixing we defend for 10 minutes in a dark place. Then, to the oxidized solution, pour 20 cm^3 of potassium iodide, 20 cm^3 of sulfuric acid solution and 100 cm^3 of distilled water, thoroughly washing the walls of the flask. The separated iodine is immediately titrated with sodium thiosulfate solution in the presence of starch. At the same time, under the same conditions, we conduct a control experiment, adding 25 cm^3 of distilled water instead of a glycerin sample.

The percentage of mass fraction of ash is as follows. Weigh the sample of glycerin on the scales in a crucible (for distilled glycerin we use only a high crucible), pre-tempered to constant mass. Gently heat on an electric tile until the vapor is released and the glycerin is cooled. The residue is calcined in a muffle furnace for 2 hours at a temperature (700 ± 50) °C. After quenching, cool the crucible in a desiccator and weigh it. Place the crucible again in a muffle furnace and harden for 30 minutes. We weight the cooled crucible. If constant mass is reached, then the difference between weighing after the first and second quenching must not exceed $2 \cdot 10^{-4}$ g. If constant weight is not reached, then the operation is repeated, having quenched every 30 minutes.

The percentage of the mass fraction of the non-volatile organic residue is as follows. Place the glycerin sample in a volumetric flask and dilute with a double amount of distilled water. The resulting solution is neutralized with a solution of sodium carbonate or a solution of hydrochloric acid to a certain amount. Dissolve the volume of the solution with distilled water to 100 cm^3, measure the pipette with 10 cm^3 of solution and transfer it to a cup pre-dried at a temperature of (170 ± 2) °C to a constant mass. Place the solution with a solution of glycerin on an asbestos plate in a drying cabinet, heat to a temperature of 105-110 °C, after which the temperature in the cabinet is brought for 1 hour to (170 ± 2) °C. At this temperature, evaporate glycerin, making sure that it is not put up, for which we periodically open the door of the cabinet. After completion of the evaporation, place the cup with a dry residue in a desiccator until it is completely cooled and then, without weighing, the residue is dissolved in 3-8 cm^3 of water. The cup with the solution is cooled to 105-110 °C and placed in a drying oven. To evaporate the temperature again raise to (170 ± 2) °C. Operation of dissolution of the residue and evaporationwill repeat twice. The cup with the residue after the two-time dissolution and evaporation is cooled in a desiccator and weighed. Continue dissolving operations until the difference between the weighings for 1 year of drying will be no more than $1 \cdot 10^{-3}$ g.

4. Results and discussion

To establish rational modes of work of the developed machine, a series of experimental studies was conducted. The first part of the experiment was based on the analysis of the amplitude-frequency characteristics of the executive body of the machine and the power charteristics of its vibration drive. The second part was to study the technological features of the glycerine clearing process under the condition of a comprehensive vibrocentric effect.

A comprehensive analysis of the indicated parameters allows us to find rational modes of the machine, which correspond to the maximum dynamic state of the investigated system and provide the maximum efficiency of the glycerine clearing process. The amplitude-frequency and energy characteristics of the developed machine are presented in Fig. 3.

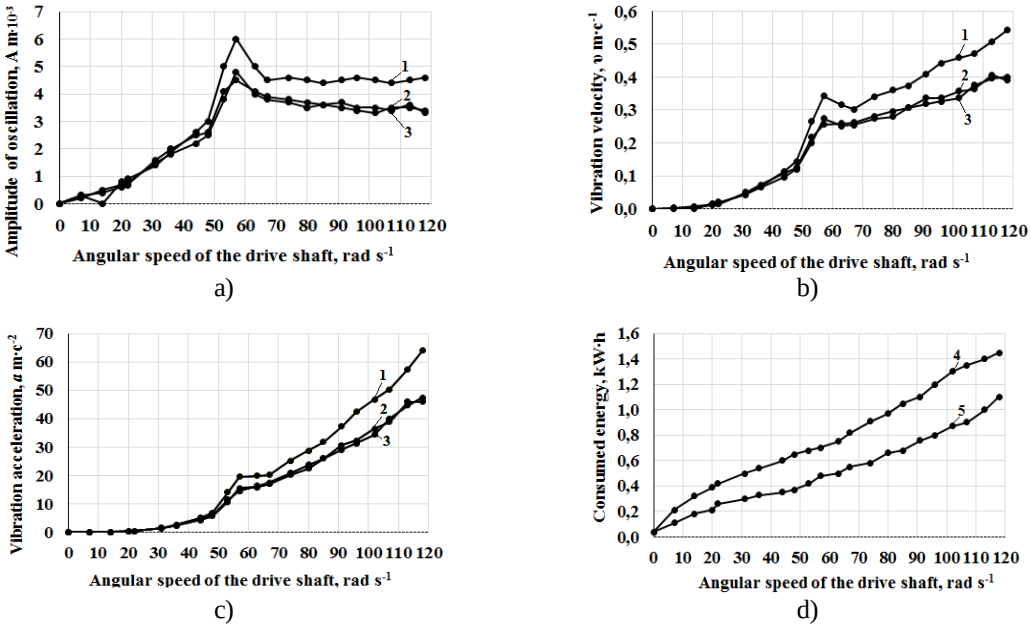


Fig. 3. Amplitude-frequency and power characteristics of the designed vibrocentric machine: 1, 2, 3 – corresponding components of the amplitude of the oscillations Az, Ax, Ay, υz, υx, υy, az, ax, ay; 4 – mode of integrated vibrocentric purification; 5 – mode of vibrocentric filtering

Obtained data in Fig. 3, a showed a gradual increase in the components of the amplitude of the oscillations $Az = 3 \cdot 10^{-3}$ m, $Ax = 2{,}8 \cdot 10^{-3}$ m, $Ay = 2{,}7 \cdot 10^{-3}$ m in the range of values of angular velocity $\omega = 0\text{-}48$ rad·s^{-1}, a sharp increase to $Az = 6{,}0 \cdot 10^{-3}$ m, $Ax = 4{,}8 \cdot 10^{-3}$ m, $Ay = 4{,}6 \cdot 10^{-3}$ m at $\omega = 48\text{-}68$ rad·s^{-1} and the relative stabilization of the curves of the amplitude of oscillations at the level of $Az = 4{,}6 \cdot 10^{-3}$ m, $Ax = 3{,}8 \cdot 10^{-3}$ m, $Ay = 3{,}7 \cdot 10^{-3}$ m at $\omega = 68\text{-}118$ rad·s^{-1}.

This analysis allowed to establish the dominance of the vertical component of oscillations as the main factor for further increasing the dynamic state of processing.

The graphic curves of the components of the vibration velocity υ (Fig. 3, b) and the vibration acceleration a (Fig. 3, c) of the executive body were obtained, the peak values of which are $\upsilon z = 0{,}35\ m \cdot s^{-1}$, $az = 20\ m \cdot s^{-2}$ and observed as a result of resonance at $\omega = 50\text{-}60\ rad \cdot s^{-1}$, after which the curves acquire a linear growth pattern with their maximum values, respectively, $\upsilon z = 0{,}55\ m \cdot s^{-1}$, $az = 65\ m \cdot s^{-2}$ under steady-state operation.

Based on the above experimental data, it can be concluded that the intensive processing of raw glycerine is observed under conditions of stabilization of the components of the amplitude of the vibrations of the container in the range of the angular velocity of the drive shaft $\omega = 68\text{-}118\ rad \cdot s^{-1}$. According to these parameters, the energy consumption of the developed machine is consumed, in the mode of integrated vibrocentric clearing make up 1,4 kWh (Fig. 3, d). While in the mode of only centrifugal filtering, power consumption is 1,1 kWh.

Further research was based on the determination of quality indicators of purified glycerin, depending on the operating modes of the vibrocentric machine and its operational parameters.

Among the main factors influencing the process of purifying raw glycerin, one can note the temperature of the raw material and the time it is processed. Variation of these factors may lead to a substantive change in the physical and mechanical properties of the purified raw material.

For the experiment, raw glycerin of the third grade was used, the density of which at T = 20 °C is $\rho = 1{,}22\text{-}1{,}23\ g \cdot cm^{-3}$, the mass fraction of ash $X_{ash} = 8{,}5\%$, the mass fraction of the non-remaining organic residue $X_{organic\ residue} = 4.3\%$, mass fraction of pure glycerol $X_{p.\ gl.} = 76.8\%$.

We conducted a series of experimental studies on the change in the quality of raw glycerine quality during its purification on a developed vibrocentric machine. Experimental research was implemented under different processing regimes. In particular, with centrifugal filtering, vibration protection and integrated vibrocentric purification.

Moreover, the influence of processing time t on the quality of glycerine purification was investigated at a temperature T = 20 °C. Whereas, the effect of temperature T on the quality of glycerine purification was investigated under conditions of processing time t = 60 s.

The obtained graphic dependencies show that the most effective is the application of integrated vibrocentric purification. It is also worth noting that the quality of glycerine increases with increasing time and temperature of its processing.

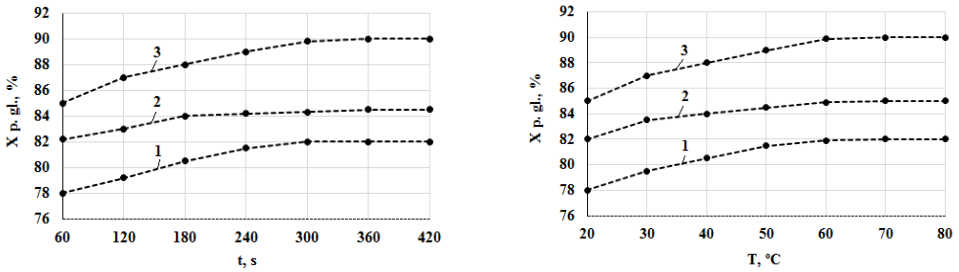


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Fig. 4. Amounts of mass fraction of pure glycerin depending on the time and temperature of its processing: 1 – under vibration protection; 2 – at centrifugal filtration; 3 – with integrated vibrocentric cleaning

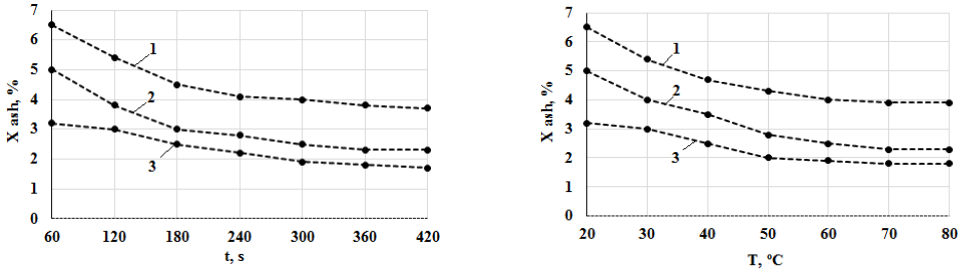

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Fig. 5. Percentage of the mass fraction of ash depending on the time of treatment of glycerine and its temperature: 1 – in vibration protection; 2 – at vibrocentric filtration; 3 – with integrated vibrocentric purification

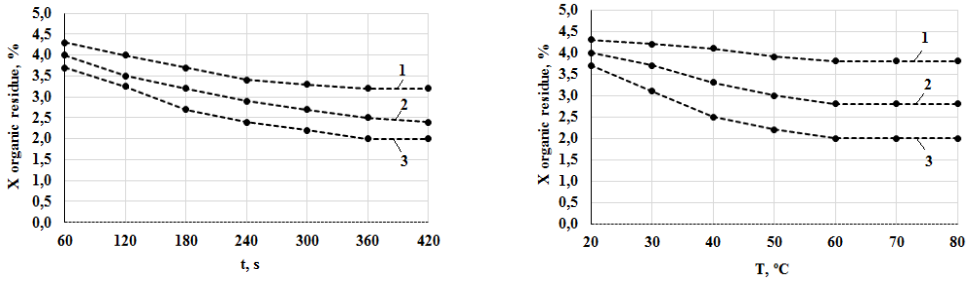

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Fig. 6. Percentage of the mass fraction of the non-volatile organic residue in glycerol depending on the processing time and its temperature: 1 – under vibration protection; 2 – at vibrocentric filtration; 3 – with integrated vibrocentric purification

Comparing the distribution of experimental data on the quality of glycerine, depending on the time and temperature of its treatment, it can be concluded that their effect on the purification process. However, it should be noted that in the energy equivalent increase in the temperature of the glycerine is a more energy-intensive process than an increase in the time of its processing.

It was found that when vibrocentric purification glycerin acquires the highest quality indices at a treatment temperature of 60-80 °C and processing time t = 300-420 s. For such parameters, the mass fraction of pure glycerin is $X_{p.gl.}$ = 88,5-89,5%, the mass fraction of X_{ash} = 1,8-1,9%, the mass fraction of the non-volatile organic residue $X_{\text{organic residue}}$ = 2,0-2,1%.

To determine the colored number of purified glycerin, we used the Lovibond method. This method is based on a comparison of the color of the purified glycerin and the spectrum of Livobond's colored plates. In fig. 7. shows the samples of purified glycerin are presented depending on the modes of its processing.

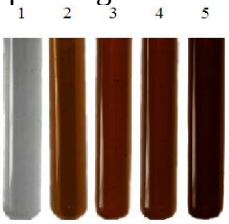

Fig. 7. Samples of glycerin depending on the treatment mode: 1 – distilled glycerol; 2 – with vibrocentric cleaning; 3 – at centrifugal filtering; 4 – with vibration protection; 5 – raw third grade glycerine

The analysis carried out in accordance with the methodology of Leibombard showed that the color of the purified glycerin with vibrocentric treatment, the angular velocity of the drive shaft vibration drive ω = 68-118 rad·s^{-1} and the processing time t = 300-420 s is 30,0 / 7,0.

Based on the results of experimental studies, quality indicators of purified glycerin were determined depending on the change of processing regimes and operational parameters of the developed machine (Tab.1).

Table 1

Indicators of quality of purified glycerin

Cleaning mode	Mass fraction of pure glycerol,%	Mass fraction of non-volatile organic residue,%	Ash mass fraction,%	Color
Centrifugal filtering	82,5-84,9	2,2-4,3	2,2-4,8	40,0/10,0
Vibration protection	78,5-82,0	3,2-4,5	3,8-6,2	60,0/30,0
Vibrocentric purification	85,0-89,5	2,1-3,6	1,8-3,1	30,0/7,0

5. Conclusions

The prototype of a vibrocentricmachine for cleaning raw glycerine has been developed. In the result of the experimental study of this machine, operating parameters are established. In particular, the angular velocity of the drive shaft is 110-120 rad·s^{-1}, the angular speed of the rotor is 120 rad·s^{-1}, the amplitude of the

vibration of the container is 4-4,5 mm. According to these parameters, the energy consumption consumed is 1,4 kWh.

It has been established that the most effective glycerin treatment mode is the vibration center mode of the machine, which combines centrifugal filtration and vibration protection of the raw material in a comprehensive manner. As a result of experimental studies it has been proved that the purification of raw glycerol is rationally carried out at a temperature of 60-80 °C and the duration of vibrocentricinfluence is 300-420 s.

The beforementioned parameters allow to obtain a product with a mass fraction of pure glycerin 88,5-89,5 %. In addition, the mass fraction of ash is 8,5-9,5% to 1,8-1,9%. While the mass fraction of the non-volatile organic residue is from 4,0-4,3% to 2,0-2,1%. Also, using the method of tonometer Lovobond, the color values of the obtained glycerin were established, which are in the range of 70,0 / 40,0-30,0 / 7,0.

On the basis of the experimental results obtained, it can be concluded that the complex use of centrifugal forces and low frequency oscillations can increase the productivity of the initial purification of glycerin by 4 times to 90-94 $kg \cdot h^{-1}$.

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