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# OBTAINING FATTY ACIDS FROM COTTON TAR BY EXTRACTION IN A ROTARY PULSATION APPARATUS

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***ABSTRACT***

The process of separation of fatty acids intensification from a saponified and neutralized cotton tar mixture's components is considered in this article as the aim of the study. The process is used for their purification from impurities and further use for the synthesis of a nonionic demulsifier - reagent based on them for the oil's preparation in field conditions. It is shown that vacuum fatty acid tars of cotton oil are a valuable raw material for free fatty acid production after appropriate processing. One of the possible and promising options for the extraction of free fatty acids from the saponified fraction is a method based on gasoline processing of a wet mixture of cotton tar components in a rotary-pulsation apparatus (RPA). It has been established that the technology of obtaining fatty acids from cotton tar in a rotary-pulsation apparatus using an effective solvent - extraction gasoline is resource-saving The influence of some design parameters of RPA working elements on the degree of extraction was investigated. By means of a multistage experimental-technological work, it was possible to optimize the extraction process and achieve results that make it possible to recommend it for use in industrial conditions for separating fatty acids from associated impurities with the following technological regime in the RPA apparatus: the ratio of the mixture: gasoline - 1: 5; the duration of the extraction is 10 minutes; temperature - 50 - 60 ° C. With this technological mode in RPA it is possible to extract free fatty acids from tar with a yield of 86-87%.

**Keywords:** tar; apparatus; extraction; demulsifier; reagent; fatty acids, demulsification; petrol; saponification; refining.

## *INTRODUCTION*

When processing cottonseed oil and its subsequent refining, depending on the technological scheme and methods of separation of the main products, a lot of secondary products and waste are formed, including free fatty acids, which are ballast components and deteriorate the oil quality. Refining is the process of purifying oil from their accompanying impurities, which consists in processing crude oil with solutions - alkali (chemical refining). As a result, salts and soaps insoluble in neutral fat are formed, the aqueous solutions of which, due to their higher density, are easily separated from the raffinate, that is, triglycerides. This soap mass is called soap stock [1].

The resulting soaps, possessing a high stabilizing and absorption capacity, carry away from fat a significant part of impurities: phosphatides, proteins, mucus, dyes and others, and alkali solutions destroy dyes. Thus, the soap stock of cotton oil refining contains neutral fat, fatty acids, gossypol and its conversion products (up to 3.5% of the total mass), in particular, the products of oxidation with air oxygen, interaction with proteins, phosphatides and fatty acids. Further, soap stock, after preparation, is subjected to vacuum distillation in order to obtain distilled fatty acids, which are used to obtain laundry soap. Residue of vacuum distillation of fatty acids - tar, which contains from 40 to 69% free fatty acids [2-4].

We have previously shown that saponification of tar with an alkali solution can produce a saponifiable fraction of fatty acids salts and gossypol, which are released in free form when treated with sulfuric acid [5,6].

The resulting mixture of fatty acids, gossypol and its derivatives was used in the initial exploratory experiments for the oxyethylation processes in the preparation of surfactants. The resulting composition during testing showed the demulsifier's properties [7]. However, more stringent requirements are imposed on modern demulsifying reagents. They must have the highest possible activity, be readily biodegradable, do not deteriorate the commercial performance of oil, be cheap and affordable. Should not have bactericidal activity (on which the effectiveness of biological wastewater treatment depends) and should not contribute to the corrosion of oilfield equipment. Therefore, the need to investigate how the purity of the feedstock affects the quality of the target product, to develop an optimal method for separating fatty acids from a mixture of tar components, and to select the conditions for the process of separating fatty acids was arose.

The aim of the study was to intensify the process of separating fatty acids from a mixture of saponified components and neutralized tar, purifying them from impurities and further use for the synthesis of a nonionic demulsifier based on them.

## *MATERIALS AND METHODS*

Cotton tar produced by "Shymkentmai" JSC was used in the process of work. As part of this task, we carried out a set of studies on the extraction of fatty acids from the obtained saponified mixture of tar components in a rotary-pulsation apparatus (RPA). Work of separating fatty acids from gossypol, its derivatives and other impurities, was carried out since in the future these impurities can have an ambiguous effect on the target product's properties. Extraction with the help of a rotary-pulsating apparatus is based on the circulation of the processed medium at different multiplicity of the solid and liquid phases. RPA refers to devices that implement the method of multifactor impact on chemical-technological processes, the low energy intensity of which is due to the fact that the processed medium is both a source and an object of hydromechanical vibrations, due to which the efficiency of the device increases [8].

When using RPA, the operations of extraction and dispersion are combined. Thus, the apparatus makes it possible to intensify the process of extracting target fatty acids from raw materials. A setup was used, the diagram of which is shown in the Fig. 1.

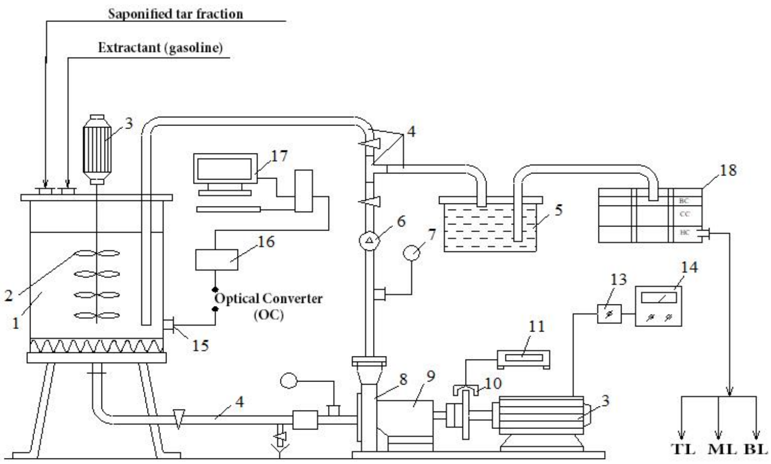


Fig. 1. Schematic of a semi-industrial plant for the extraction of fatty acids:
1 - extractor, 2 - paddle mixer, 3 - electric motors, 4 - piping system with taps and tee, 5 - reserve tank, 6 - flow meter, 7 - pressure gauge, 8 - RPA working part, 9 - coupling, 10 - optical sensor, 11 - tachometer, 12 - vacuum gauge, 13 - thyristor converter unit, 14 - wattmeter, 15 - sampling fitting, 16 - scanning device, 17 - computer, 18 - sump. (TL - top layer, ML - middle layer, BL - bottom layer).

## *RESULTS AND DISCUSSION*

Currently, the development of resource-saving technologies using more efficient extractants and equipment is relevant. One of the possible and promising options for extraction may be a method based on gasoline processing of a wet mixture of cotton tar components in RPA.

The semi-industrial RPA installation (Fig. 1) is equipped with measuring instruments to determine the technological characteristics. At the initial stage of the work, the influence of the width of the rotor slots on the efficiency of the extraction process was established, the width of the rotor slots was investigated in four values - 4, 6, 8 and 10 mm; the width of the stator slots was fixed at 10mm; the gap between the rotor and the stator was 2mm. Preliminary studies of the extraction process were carried out with the following parameters: the ratio of the mixture: gasoline - 1: 8, the duration of the extraction - 10 min. It is known that the extraction process in RPA consists in the impact on the working mixture of hydromechanical factors number, and their influence will be determined by the peculiarity of the structural elements of the rotor and stator, depending on the required technological result [9]. Increasing the efficiency of the extraction process for rheologically complex systems under study is a hard task. As follows from the data in Fig. 2, with an increase in the width of the rotor slots, an increase in the degree of extraction of fatty acids and side impurities is observed up to values of a width of 10 mm, with a width of the rotor slots of 12 mm for the FA output, the indicators do not change, and the amount of extracted impurities increases.

To carry out the extraction process, 80 liters of gasoline were poured into the RPA extractor (Figure 2) and the stirrer was turned on, then gasoline was heated to a temperature of 60 ° C using a thermostat, the valves were opened while the apparatus was running, and the extractant was circulated along the circuit. After establishing the temperature regime, a wet mixture of fatty acids in the amount of 16.0 kg was simultaneously loaded into the extractor. The mixture was processed for 10 minutes, during processing, samples of the suspension were taken every 30 seconds after the mixture was loaded into the apparatus. It was found that with the reverse loading mode, the degree of extraction of target fatty acids with the same parameters decreases by 5-7%. The resulting suspension was defended for 2 hours in a settling tank and after settling, the lower aqueous layer with water-soluble impurities is sent for purification. The upper layer, a solution of fatty acids in gasoline, was taken for processing (distillation of the solvent), the middle layer containing gossypol, its derivatives and other impurities that did not pass into the water layer and the gasoline layer was used to isolate technical gossypol. The selected samples of the suspension were defended in laboratory conditions, washed until the medium was neutral, and the composition of all three layers was analyzed.

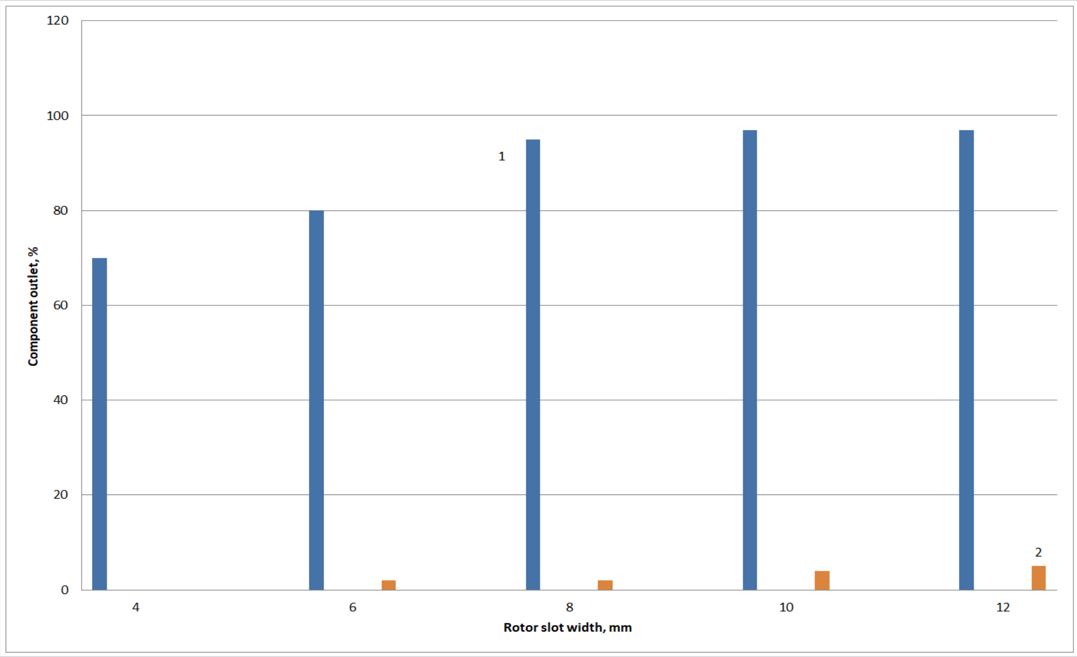


Fig. 2. Influence of the rotor slots' width on the fatty acids' extraction degree (1) and impurities (2). Conditions: rotor diameter 0.125 m, mixture ratio: gasoline - 1: 5, extraction time - 10 min, stator slot width - 10 mm; gap between rotor and stator - 2mm.

Similar dependences were obtained when studying the width of the stator slots; therefore, in the main series of experiments, the width of the stator and rotor slots was 10 mm.

RPA technical characteristics are shown in the table 1

Table 1. RPA technical characteristics

| main parameters | **The value** |
|---|---|
| Extractant circulation capacity, $m^3$ / h. | 0.96 |
| Rotor speed, revolution / s. | 40 |
| Rotor diameter, mm | 125 |
| Number of rotor cylinders, pcs. | 2 |
| Number of stator cylinders, pcs. | 2 |
| Radial clearance between rotating and stationary elements, mm. | 2 |
| The width of the rotor slots, mm. | 10 |
| Stator slot width, mm. | 10 |

Table 1 continuation.

| | |
|---|---|
| The length of the corrugation zone, mm. | 20 |
| Inlet pipe diameter, mm. | 32 |
| Outlet pipe diameter, mm. | 22 |
| Supply voltage, V. | 380 |
| Overall dimensions, mm. | 500x250x260 |
| Weight of the device, kg. | 33 |

Table 2 shows the study results of the influence of the ratio "mixture of components - extractant" on the degree of extraction and purity of fatty acids. The obtained data indicate a significant effect of the used extraction method on the final result: the process intensifies (compared to a mixing-settling extractor), even with a mixture of components - extractant ratio equal to 1: 5, the degree of extraction and the purity of fatty acids are maximal. With an increase in the ratio "mixture of components - extractant" to 1: 6 and higher, mainly glycerides, some polyphenols and unidentified impurities, which contaminate the target product, begin to pass into the solvent (gasoline).

Table 2 - Influence of the ratio "mixture of components - extractant" on the extraction degree and fatty acids' purity *

| Component | The degree of fatty acids and impurities extraction,% | | | | |
|---|---|---|---|---|---|
| | Reaction mixture ratio: gasoline | | | | |
| | 1:1 | 1:3 | 1:5 | 1:6 | 1:8 |
| Fatty acid | 45 | 79 | 86 | 87 | 87 |
| Gossypol and its derivatives | traces | traces | 0.4 | 0.5 | 0.5 |
| Di-, triglycerides and were not transferred to the FA's extract | 31 | 23 | 11 | 9 | 8 |
| Unidentified impurities | traces | traces | 2 | 3 | 4 |

* Conditions: rotor diameter is 0.125 m, extraction time - 10 min, rotor slot width - 10 mm; width of the stator slots - 10mm; the gap between the rotor and the stator is 2mm.

In order to optimize the ratio of solvent and raw materials, the ratio “mixture of components - extractant”, equal to 1: 5, was subsequently used, which makes it possible to reduce the consumption of the extractant and, hence, subsequently to reduce the costs of its distillation and regeneration. At the same time, the content of impurities is rather high; therefore, it is necessary to investigate other factors influencing the process. Despite the great importance of the experimental studies carried out with various types of emulsions and finely dispersed suspensions described in the literature and which made it possible to design various types of RPA, the use of experimental data and confirmed regularities is possible only when solving particular problems and is impossible when RPA is working with tar, since there is no general methodology calculation of the geometric characteristics of the apparatus, which complicates its use in technological processes. Therefore, an empirical method with real objects was used in the work.

The results of experimental studies on extraction, obtained during the operation of RPA with different geometric parameters of the rotor and stator, are shown in Fig. 3.

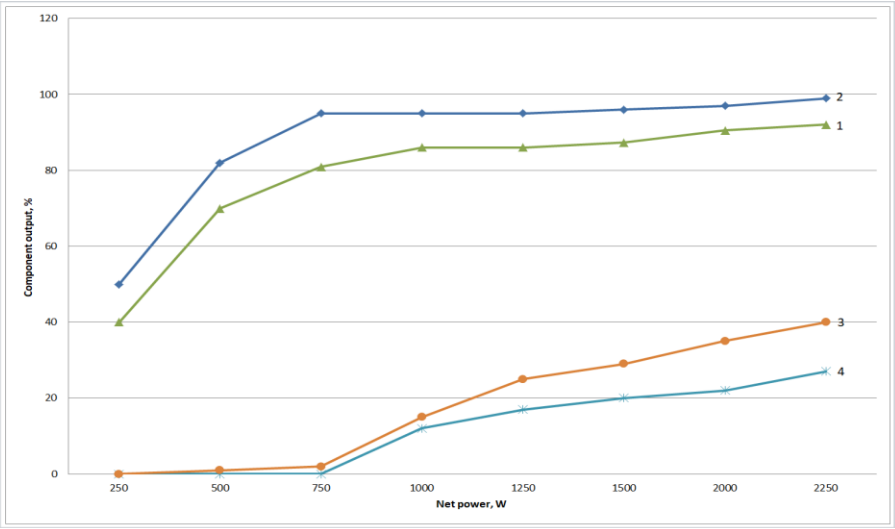


Fig. 3. Dependence of FA's extraction degree and foreign impurities on the useful power of the RPA. Conditions: rotor diameter 0.125 m, mixture ratio: gasoline - 1: 5, extraction time - 1.4 - 5 minutes; 2.4 - 10 min; width of the rotor slots –10 mm; width of the stator slots - 10mm; the gap between the rotor and the stator is 2mm.

1.2, - FA, 3.4 - impurities.

From the data in Figure 3 it can be seen that with an increase in the useful power of RPA, the degree of fatty acids' extraction and impurities increases. Experimental studies carried out in RPA with different geometric parameters of the rotor and stator make it possible to assert their influence on the degree of extraction. There is a sharp increase in net power at the final stage of extraction, when it is required to achieve the maximum degree of extraction. Recall that curves 1, 2 refer to fatty acids, curves 3,4 show the total yield of impurities obtained during extraction.

It was also found that the duration of the impact on the working mixture of hydromechanical factors also has an effect on the characteristics of the process, therefore the "time" factor was investigated more thoroughly (Figure 4). The obtained data indicate that the optimal extraction time is 10 minutes, since an increase in the duration of the hydromechanical action practically does not increase the FA yield, but worsens their purity due to the transfer of impurities from the fat mixture to the extract.

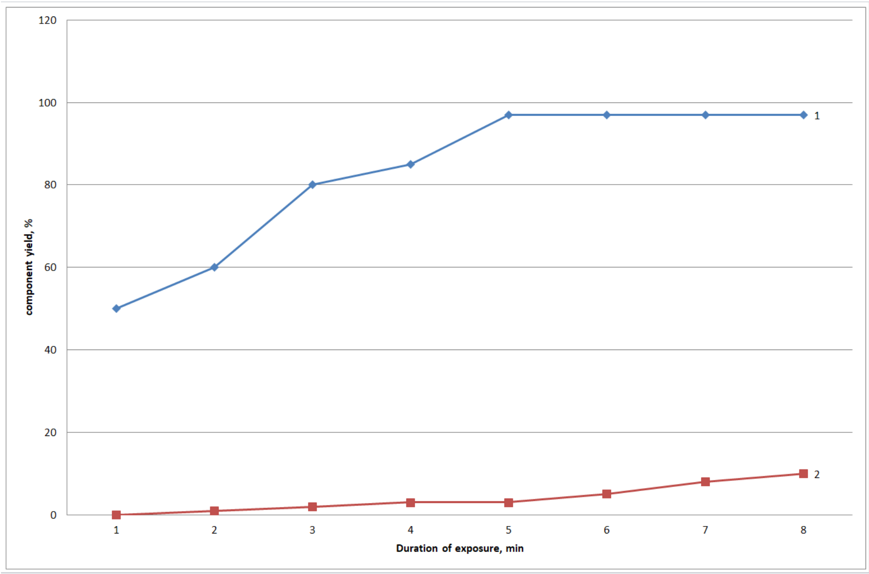


Fig. 4. Hydromechanical action's duration influence on the liquid crystals' extraction degree (1) and impurities (2). Conditions: rotor diameter is 0.125 m; the ratio of the mixture: gasoline - 1: 5; the duration of the extraction is 10 minutes; width of the stator slots - 10mm; the gap between the rotor and the stator is 2mm.

The influence of some design parameters of RPA working elements on the extraction's degree was also investigated. Fig. 5 shows the dependences of the residual content of fatty acids in the fat mixture and the impurities' extraction degree from it on the basic geometric parameters of the rotor and stator of the apparatus

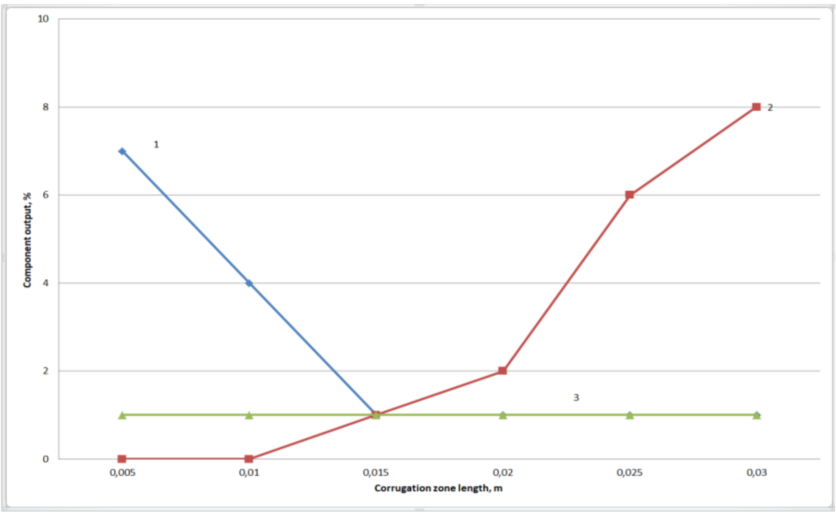


Fig. 5. Influence of the corrugation zonelength on the residual FA content in the fat mixture and the degree of impurities extraction. Conditions: rotor diameter is 0.125 m, temperature - 20 ° C, mixture ratio: gasoline - 1: 5, extraction duration - 10 min, rotor speed - 40;

1 - residual content of fatty acids in the fat mixture, 2 - degree of extraction of impurities, 3 - permissible content of components.

From the data in Fig. 5, it can be seen that with an increase in the length of the corrugation zone, there is a decrease in the residual content of target fatty acids in the fat mixture and an increase in the degree of impurities extraction. If we set the minimum permissible value of the residual content of fatty acids in the fat mixture and the degree of impurities extraction equal to 1%, then the optimal length of the corrugation zone will be from 0.015 to 0.02 m. It was found that an increase in the angular speed of rotation of the rotor by more than 50 s -1 is impractical, since this is associated with high energy input. At a rotor speed below 30 s -1 to ensure the required dispersion mode, it is necessary either to increase the number of rotor and stator protrusions, the number of which is limited by analogy with disk mills, or to increase the length of the corrugation zone, which is determined by the difference between the rotor diameter and the pump inlet pipe, by the basis of which the device is made.

It was found that an important parameter affecting the degree of separation of impurities from fatty acids is also the temperature of the extraction process. In contrast to the conditions, during extraction in a mixing-settling extractor, where the optimum temperature was a value above 60 ° C, in these conditions, the temperature of 50 ° C should be considered as the optimal indicator (Table 3). At this temperature, the degree of extraction reaches 86.2%, and the amount of impurities passing from the mixture of tar components to the extractant does not exceed 2.3%, which ultimately, after distilling off gasoline, is 0.4 - 0.6% in the composition of crude fatty acids.

Table 3. Influence of temperature on the degree of extraction and purity of FA *

| Component | Temperature, ºC | | | | | |
|---|---|---|---|---|---|---|
| | Extraction rate of fatty acids and impurities,% | | | | | |
| | 20 | 30 | 40 | 50 | 60 | 70 |
| Fatty acid | 60.6 | 74.2 | 81.7 | 86.2 | 87.5 | 87.9 |
| Di- and triglycerides triglycerides and were not transferred to the FA's extract | 30.4 | 21.0 | 14.8 | 11.0 | 10.0 | 7.6 |
| Gossypol and its derivatives | 0.1 | 0.2 | 0.3 | 0.4 | 0.5 | 0.6 |
| Unidentified impurities | 8.2 | 4.1 | 2.9 | 2.3 | 2.1 | 1.9 |

* - conditions: rotor diameter is 0.125 m, mixture ratio: gasoline - 1: 5, extraction time - 10 min, rotor speed - 40; corrugation zone length - 0.02m.

## *CONCLUSION*

Cottonseed oil vacuum fatty acid tars are a valuable raw material for obtaining free fatty acids from them after appropriate processing. One of the possible and promising options for extractions is a method based on gasoline processing of a wet mixture of cotton tar components in a rotary-pulsating apparatus. Obtaining fatty acids from cotton tar in a rotary-pulsation apparatus is a resource-saving technology using an effective solvent - extraction gasoline.

The influence of some design parameters of RPA working elements on the degree of extraction is shown. By means of a multistage experimental-technological work, it was possible to optimize the extraction process and achieve results that can be recommended for use in industrial conditions for separating fatty acids from associated impurities with the following technological regime in the RPA apparatus: the ratio of mixture: gasoline - 1: 5; the duration of the extraction is 10 minutes; temperature - 50 - 60 ° C. With this technological mode in RPA it is possible to extract free fatty acids from tar with a yield of 86-87%.

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