

The Adsorptive Purification Of Low-Quality Glycerin By Biocarbon Adsorbents

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Abstract

This study investigates the purification of low-quality glycerin using biocarbon adsorbents derived from cherry pits. The carbon adsorbent produced (ACCP) demonstrated superior efficacy in reducing glycerin color, decreasing it from 10 mg I₂/100 cm³ to 5 mg I₂/100 cm³, outperforming the imported coconut carbon adsorbent (ACC), which only achieved a reduction to 6 mg I₂/100 cm³. It has also been established that a carbon adsorbent with a particle size of 2-3 mm is considered optimal, as smaller particles hinder filtration, thereby increasing ash content due to the migration of fine carbon particles into the filtrate. A reduction in the acid number by 5-6 times allows for an improvement in the quality of glycerin and extends its shelf life. It has been determined that the carbon adsorbent derived from cherry pits selectively reduces the amount of free fatty acids. Initial glycerin parameters corresponded to grade T-88 specifications; however, after treatment with the synthesized adsorbents, the parameters improved to meet the D-98 standard requirements. These findings suggest that cherry pit-based adsorbents are a promising alternative for purifying glycerin in various industrial applications.

Keywords: Glycerin; adsorbent; adsorption; impurities; carbonaceous materials; purification; biocarbon.

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1. Introduction

Currently, the adsorption method of cleaning liquids, including glycerin, is recognized as a promising approach because it enables the removal of small amounts of harmful impurities. As a result, it is widely used for the fine purification of liquids intended for medical, pharmaceutical, and other critical military-strategic purposes.

Glycerin is a trihydroxy alcohol that serves as the basis for plant lipids, such as triglycerides. It is widely used in medicine, explosives manufacturing, cosmetology, rubber technology, pharmaceuticals, the automotive industry, and other fields. In the oil and fat industry, crude glycerin is obtained from fats through non-reactive breakdown of fat and soap stocks.

From low-quality technical fats, it is usually impossible to obtain purified glycerin that meets standard requirements for non-volatile organic residues, color, saponification value (complex esters), and other parameters. The adsorption cleaning method for glycerin in a mechanical stirrer apparatus has comparatively low efficiency and does not always lead to a product with the necessary quality level. It should be noted that during the purification process, the quantity and quality of impurities in the initial glycerin are often overlooked, and there is no theoretical justification for selecting a specific type of adsorbent. As a result, many enterprises are compelled to perform repeated distillation to produce a product that meets standards, leading to substantial losses of valuable glycerin. [1,2]

Significant challenges arise in the production of purified glycerin when a large amount of low-quality technical fats is subject to hydrolysis, containing up to 5% of undesirable impurities—such as non-saponifiable substances, insoluble in ether, proteins, phosphatides, and other components that end up in the glycerin.

Currently, glycerin with a high content of undesirable impurities is used much less frequently compared to distilled glycerin. During distillation, volatile impurities carry over with the glycerin into the distillate, while mineral substances predominantly remain in the glycerin residue. Extended storage of distilled glycerin containing

amino acids results in its darkening, which is associated with the formation of colored compounds from amino acids and other organic substances. [3]

There is very little information in the literature regarding impurities in distilled glycerin. A partial understanding of the composition of these impurities can be gained by studying those found in both crude glycerin and glycerin waters. During the evaporation process, impurities present in glycerin waters are transferred to raw glycerin. Carbonyl-containing compounds and products of their interaction with phosphatides and amino compounds negatively affect the color of glycerin, often resulting in intense coloration.

In glycerin water obtained from technical fats, carbonyl-containing compounds have been found in amounts ranging from 0.05% to 0.079%. M.V. Irodov and N.S. Eliseeva conducted comprehensive studies on impurities in 86% glycerin, determining the quantitative content of fatty acids and glycerides. [4-7]

Impurities from technical fats are transferred to glycerin waters during non-reactive hydrolysis, which contain substantial amounts of accompanying substances. To mitigate the presence of undesirable impurities, glycerin waters undergo various purification methods.

The impurities in glycerin water derived from natural fats exhibit significant diversity and are chemically analogous to the original raw materials. Key impurities include phosphatides, fatty acids, glycoproteins, glycolipids, lipoproteins, sterols, sterides, hydrocarbons, waxes, vitamins, as well as chromophores and products of their thermo-hydrolytic degradation [8,9]. The presence of these impurities is undesirable, as it markedly increases the loss of glycerin during industrial processing, particularly in conjunction with sludge (e.g., calcium sludge). Moreover, these impurities adversely impact the quality of the final purified glycerin.

Authors [10,11] demonstrated that mineral impurities in initial glycerin solutions significantly influence glycerin losses during distillation. These ash-related substances are derived from both the raw materials and the technical water utilized in the hydrolysis process. Furthermore, additional impurities may be introduced during

purification processes. Undesirable compounds can form during the neutralization with lime milk, resulting from the dissolution of gypsum, hydrated calcium oxide, and other associated compounds generated in the industrial production of lime.

Through spectral analysis, Zaits and Zhalyud identified several impurities in glycerin water, including ash components such as Fe, Na, Si, and Mg at concentrations of 0.4% to 0.5%, aluminum in minor percentages, and trace elements of copper, zinc, lead, manganese, and cadmium. Gravimetric analysis revealed the following compounds present in an average ash sample: SO_2 at 22.03%, Cl^- at 3.9%, SiO_2 at 21.81%, P_2O_5 at 20.98%, NiO at 1.91%, and combined MgO and CaO totaling approximately 0.5% [12].

Analysis of enterprise operations indicates that the distillation method for purification does not yield glycerin of adequate quality when low-quality technical fats are used. Consequently, it is recommended to employ the adsorption method at the final stage of purified glycerin production, which necessitates the development of specialized modified carbon adsorbents and an appropriate technological scheme for their application.

This underscores the need for developing a technology for producing modified carbon adsorbents based on production waste.

Waste from canned food production, specifically cherry pits, represents a promising raw material for carbon adsorbents. Methods and technologies for obtaining carbon adsorbents are detailed in the works of [13,14].

2. The Experimental Part

The next phase of the research focused on determining the adsorption properties of the developed carbon adsorbents for glycerin purification [15]. To this end, samples of distilled glycerin from JSC "Uchkurgan YOG" were selected, as they did not meet the color and ash content requirements set by GOST [16]. According to the method described in [17], the purification of the selected glycerin samples was achieved by adsorption. The results are presented in Table 1.

1. Results and Discussion

According to the method described in [17], the purification of the selected glycerin samples was achieved by adsorption. The results are presented in Table 1.

Table 1

Indicators of adsorption purification of distilled glycerin using obtained carbon adsorbents

Glycerin Sample	Color Number (mg $\text{I}_2/100 \text{ cm}^3$)	Ash Mass Fraction (%)	Light Refraction (%)	Density ($\rho, \text{kg/m}^3$)	Mass Ratio Based on Density (%)
Initial Glycerin	10	0.22	90	1.230	89
Glycerin Purified with Activated Carbon from Cherry Pits (ACCP)	5	0.01	98	1.260	98

Glycerin Purified with Imported Activated "Coconut" Carbon (AUC)	6	0.02	97	1.255	96
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As shown in Table 1, after purification with activated carbon adsorbent, the color of glycerin decreased from 10 to 5 mg I₂/100 cm³, and the ash mass fraction reduced from 0.22% to 0.02%. This is corroborated by the data on light refraction and density. Based on the initial glycerin data, its properties align with those of grade T-88; however, after purification with the developed adsorbents, these values improved to those of grade D-98. The results indicate that the purification of distilled glycerin with the obtained adsorbents significantly

reduces harmful impurities, thereby upgrading its quality to meet D-98 requirements.

The particle size of the carbon also affects other performance indicators, such as filterability, oil retention capacity of the adsorbent, and yield. We investigated the influence of dispersity on glycerin filterability, as presented in Table 2. For this analysis, distilled glycerin with an ash content of 0.14% was purified. The ash content plays a crucial role in assessing the quality of the glycerin. Taking this into account, the purification was

Table 2
Changes in Filterability and Ash Content of Glycerin Depending on Carbon Particle Size

Carbon Particle Size (mm)	Filterability (ml/5 sec)	Ash Content of Purified Glycerin (%)
1-2	4.8	0.18
2-3	8.6	0.02
3-4	10.3	0.06
4-5	13.6	0.09
5-10	16.5	0.11

As observed in Table 2, the use of carbon adsorbents with particle sizes of 1-2 mm complicates the filtration process. Therefore, the optimal particle size for the carbon adsorbent has been determined to be 2-3 mm. While increasing the particle size enhances filterability, it negatively impacts the quality of the purified glycerin. The high ash content in the purified glycerin obtained with 1-2 mm carbon can be attributed to smaller carbon

particles passing easily through the filter and remaining in the purified glycerin.

It is known that a certain amount of free fatty acids, resulting from the cleavage of triglycerides, is introduced into the glycerin during its production. Their presence in glycerin is undesirable, as they adversely affect processes such as the production of explosives, pharmaceuticals, and other applications.

Consequently, when evaluating the selectivity of carbon adsorbents, their adsorption characteristics regarding free fatty acid content are considered. In practice, the acid number is used as an indirect measure of free fatty acid content in oils, fats, and glycerin [15], calculated using the following formula:

$$K_q = 5,11 \cdot V/a; \quad (1)$$

where: V- volume (in mg) of 0.1 N alcoholic KOH solution used for titration;

a- weight of the substance (in g).

Using this parameter, we examined the selectivity of the carbon adsorbents developed under laboratory conditions. As a control, Indian activated carbon commonly used in glycerin production was also employed.

Table 3 presents the results of the study on the selective sorption of free fatty acids using the developed carbon adsorbents.

Table 3
Changes in acid number (an) of glycerin depending on the type and temperature of pyrolysis of carbon adsorbent

Type of carbon adsorbent	Free fatty acid content in glycerin (an) (mg koh/g)	Before purification	After purification
Glycerin Purified with Activated Carbon from Cherry Pits (AUCP)	3.6	3.6	0.5
Glycerin Purified with Imported Activated "Coconut" Carbon (ACC)	3.6	3.6	0.65

As shown in Table 3, the proposed adsorbent, activated carbon derived from cherry pits, demonstrates selective sorption of free fatty acids from glycerin that is comparable to the more expensive imported "Coconut" carbon.

3. CONCLUSION

Thus, it has been established that the carbon adsorbent obtained from cherry pits exhibits similar sorption properties to the imported adsorbent (AUC). It is also noted that a particle size of 2-3 mm for the carbon adsorbent is optimal, as smaller particles hinder filtration, consequently increasing ash content due to the migration of fine carbon particles into the filtrate.

Furthermore, the reduction in free fatty acids using the proposed carbon adsorbents enhances the quality, stability, and flavor characteristics of glycerin.

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