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Regeneration Of Perlite After Winterization Of Sunflower Oil

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Abstract: This article studies methods of regeneration and their impact on the filtering capabilities of perlite. It was found that increasing alkali concentration up to 40% at various ratios enhances the purification of vegetable oil; however, with further regeneration, the effectiveness decreases. Increasing acid concentration raises the amount of distilled water required, leading to higher costs and inefficient water usage. Regenerated perlite after winterization filters saturated fatty acids because, at lower temperatures, waxy substances and saturated fatty acids begin to crystallize and precipitate with the filtering agent. As a result, the content of unsaturated fatty acids increases in the system.

Keywords: Perlite, refining, vegetable oil, winterization, vermiculite, calcination, expansion, adsorbents, waxy substances, acid value, peroxide value.

Introduction: One of the modern trends in the development of the oil and fat industry worldwide is the production of competitive functional food products with enhanced biological value that are safe for consumption. Therefore, developing and improving methods for growing and processing vegetable oils to produce high-quality oil and fat products is a key objective [1-3].

In Uzbekistan, new equipment and technologies are being actively introduced for producing light vegetable oils from sunflower, soybean, and safflower, which differ in composition and properties from cottonseed oil. For example, light oils contain waxy substances (sterols, waxes, etc.) that are relatively rare in cottonseed oil. In contrast, cottonseed oil contains a significant amount of gossypol and its derivatives, as well as various phospholipids [4].

Waxy and high-melting substances are removed from refined or deodorized oil using winterization to obtain salad oil, i.e., a more purified product ready for sale as winterized commercial oil [5,6].

The appearance of vegetable oil is also a key quality criterion. However, in recent years, due to a shortage of oil-bearing crops, the industry has struggled to produce clear oils because imported filtering materials are expensive. Known waxy substances, with melting points from 32 to 98 °C, form a fine and stable suspension of crystals in the oil upon cooling — often referred to as a “network.” This greatly deteriorates the oil's appearance and quality [7]. None of the oil refining stages (hydration, alkali neutralization, bleaching, deodorization) significantly remove waxy substances.

Waxy substances in oil (0.02% to 0.3%) not only affect product quality but also complicate processing and storage. They create problems during polishing filtration and can negatively impact hydrogenation catalysts [8].

The experimental part

There are several methods for removing waxes from vegetable oils. One known method uses freezing, where oil is cooled with added special filter powders that serve as crystallization centers. After cooling, the sediment is filtered out. A drawback of this method is that the auxiliary filtering powders (e.g., zeolites, filter perlite) contain pores and capillaries that get filled with wax and neutral oil, leading to high oil losses and increased filter consumption. These powders are single-use, and the wax adsorbed on them is not reclaimed [8].

Another method involves adding a crystallization initiator to the oil and separating the resulting crystals using wax substances extracted from vegetable oils. In this case, the used filtering powder from oil freezing acts as the wax source. However, this also introduces oxidation products (peroxides, anisidines, dienes, trienes) into the oil from the used filtering powders, degrading oil quality.

In Uzbekistan, filtering materials used in production accumulate as waste after use, creating environmental issues. Therefore, this study explores regeneration methods.

Regeneration Method

Regeneration was performed using various concentrations of alkalis (NaOH, KOH) as follows: samples of used perlite were mixed with alkali at various ratios and concentrations, stirred continuously at 40–45 °C for 30 minutes, then neutralized with distilled water to pH 7. The neutralized material was dried and ground.

The results of the research are presented in Table 1.

Table 1.

Effect of Perlite Treatment with Alkali (KOH) on the Degree of Wax Removal

Alkali Concentration (%)	Alkali-to-Perlite Ratio	Degree of Vegetable Oil Purification After Regeneration, %		
		1	2	3
10	1:0.5	73.5	68.7	62.4
	1:1	78.4	75.1	72.4
	1:1.5	85.0	81.4	77.2
	1:2	89.1	84.4	77.9
20	1:0.5	75.6	72.7	68.4
	1:1	82.6	79.6	74.5
	1:1.5	88.4	85.2	80.1
	1:2	91.2	88.6	81.4
30	1:0.5	76.9	72.8	70.6
	1:1	79.7	76.8	73.8
	1:1.5	87.8	84.3	81.3
	1:2	93.4	90.7	87.8
40	1:0.5	77.2	68.7	62.4

	1:1	81.6	78.4	76.2
	1:1.5	89.4	84.6	81.6
	1:2	95.7	92.3	89.4

As seen from Table 1, increasing the alkali concentration up to 40% at various ratios enhances the degree of vegetable oil purification; however, with further regeneration, the purification efficiency decreases. An increase in acid concentration leads to greater use of distilled water, which in turn raises production costs and results in inefficient water usage—an especially pressing issue in the Republic, where water scarcity is a significant concern.

Based on the above table, it can be concluded that a 20% alkali solution with a 1:2 alkali-to-perlite ratio is sufficient for regeneration, achieving a purification efficiency of 91.2% on the first use.

After the regeneration of perlite, winterization of various light vegetable oils was carried out, and their organoleptic and physicochemical properties were studied. The data are presented in Table 2.

As seen from Table 2, all indicators after winterization meet the requirements of GOST standards; however, these results correspond to the first stage after regeneration. As shown in Table 1, regenerated perlite begins to lose its effectiveness after the second or third stage, which is why other regeneration methods will be explored further.

Table 2

Fatty acid composition (FAC) before and after freezing with regenerated pearlite

Indicators	Vegetable oils			
	Refined sunflower oil Freeze-dried		Refined soybean oil	
	Pre-freezing	Post-wetting	Pre-freezing	Post-wetting
Acid number, mg KOH/g	0,4	0,36	1,6	0,09
Peroxide number, ½ mmol O/kg	4,6	3,4	5,4	4,6
Transparency	Muddy	transparent	Light clouding	transparent
Smell and taste	Specific odor and taste	Typical according to GOST for this type	Specific odor and taste	Typical according to GOST for this type

Further, the fatty acid composition of vegetable oils (sunflower oil, soybean oil) freeze-dried with regenerated perlite was studied.

Table 3.

Fatty acid composition (FAC) before and after freezing with regenerated pearlite

Name of sunflower oil fatty acids	Initial LCS of sunflower oil, %	LCS after freezing of refined sunflower oil with regenerated pearlite, %	Initial soybean oil GLC, %	LCS after freezing of refined soybean oil with regenerated pearlite, %
Stearic acid ($C_{18}H_{36}O_2$)	4,1	2,9	2,5	1,8
Palmitic acid ($C_{16}H_{32}O_2$)	6,4	3,4	9,3	6,6
Myristic acid ($C_{14}H_{28}O_2$)	0,07	-	0,2	-
Arachinic acid ($C_{20}H_{44}O_2$)	0,25	-	0,17	2,4

Oleic acid ($C_{18}H_{34}O_2$)	19,1	22,5	21,5	27,2
Linoleic acid ($C_{18}H_{32}O_2$)	67,09	71,2	52,9	61,4
Other housing estates	2,99	100,0	13,43	99,4

As can be seen from Table 3. regenerated perlite after freezing filters saturated fatty acids, because at temperature decrease with waxy substances, also saturated fatty acids begin to crystallize and to feed with a filtering agent in a precipitate. This increases the unsaturated fatty acids in the system.

For comparison of the initial and regenerated pearlite a comparative analysis was carried out, the data of which are summarized in Fig. 1.

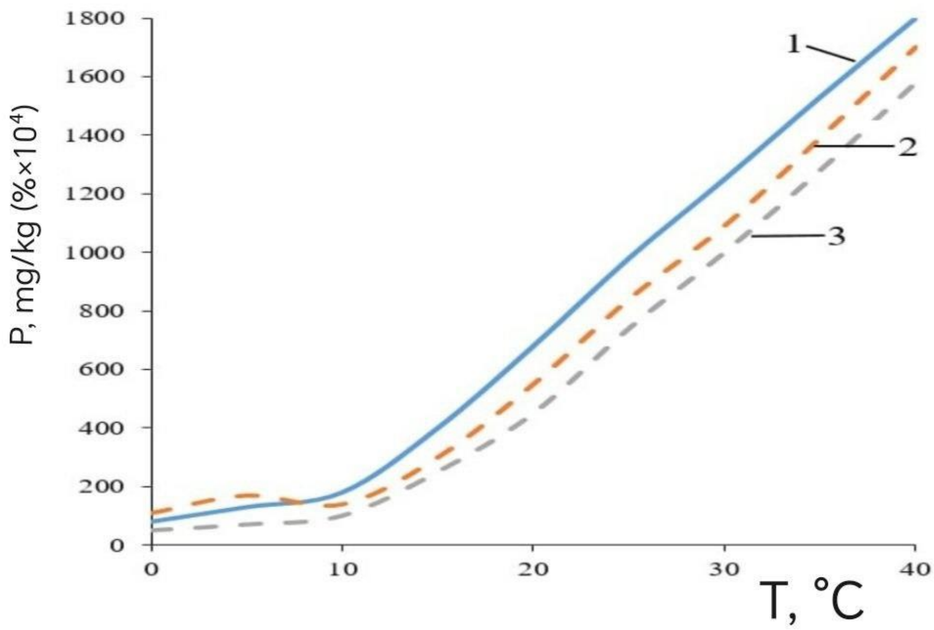


Figure 1. Effect of temperature on the solubility of waxes

1 -perlite regenerated after the 2nd stage; 2- regenerated after the 1st stage; 3- initial perlite.

It can be seen from Fig.1. that as the temperature increases to 40 OC, the waxy substances dissolve maximally. Solubility was determined by heating to 40 °C the oil was slowly cooled. The oil was then filtered off the precipitate, degreased with hexane cooled to 0 oC, dried and weighed. The concentration of waxes dissolved in the oil (solubility) was calculated using the formula:

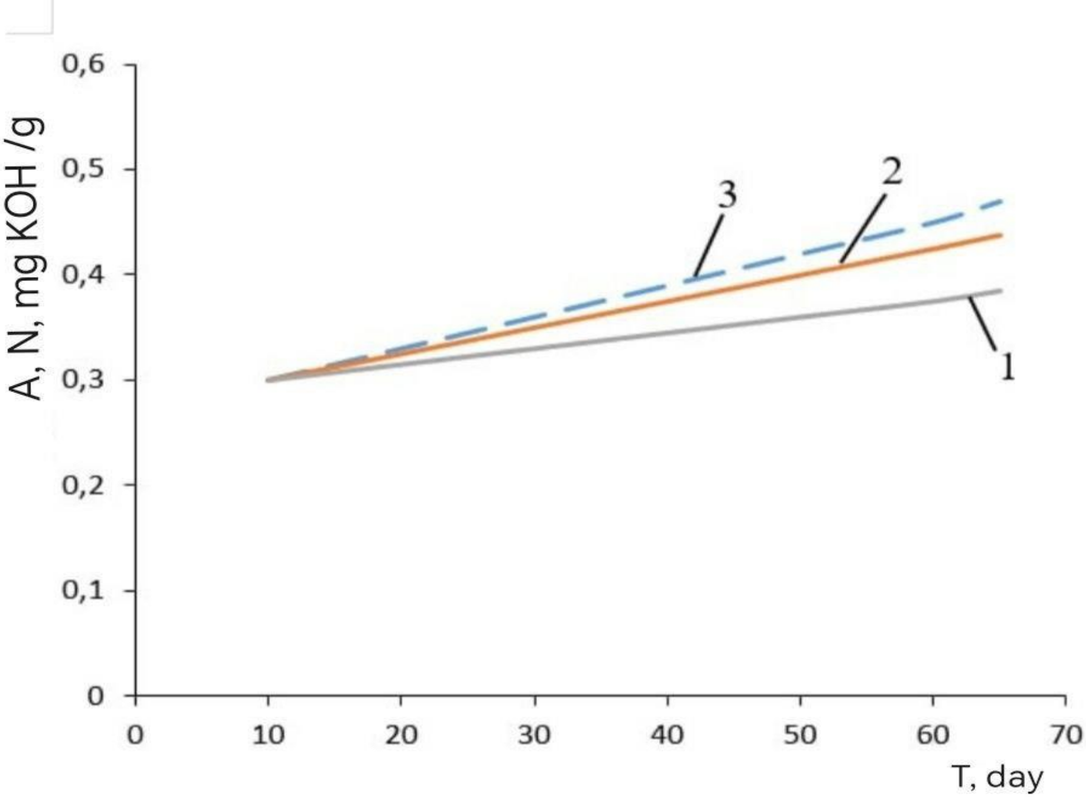
$$К_c = \frac{M_{cw.} - M_{o.}}{Mm}$$

where: Kc= concentration of waxes in oil; mg/kg; Mcw. - initial amount of waxes, mg; Mo- sediment on the filter, mg; Mm- mass of filtered oil, kg.

As can be seen from the graph the initial perlite filters better after freezing, compared to the regenerated ones. However, after each regenerated perlite its filtering capacity deteriorates. The intensity of the crystallization process proceeds in two ways, i.e., by supercooling, or by using initiators for inoculum crystallization. Increasing supersaturation accelerates the formation of nucleates, but it leads to a sharp increase in the viscosity of the system, hampering the diffusion processes of mass transfer, resulting in a decrease in the size of crystals, which hinders the deposition process. Therefore, it is rational to use an initiator for the formation of crystallization centers, which is pearlite.

Further, the influence of time on qualitative indicators of frozen oils with regenerated perlite was studied. The data of which are shown in Fig. 2.

Figure 2. Effect of storage time on the acid number of freeze-dried sunflower oils



1-Source pearlite; 2-Regenerated pearlite after the 1st stage;

3-Regenerated pearlite after the 2nd stage;

Comparative data from Fig.2. shows that freezing with initial perlite for a long time maximizes the retention of the acid number of sunflower vegetable oil. Freezing using regenerated perlite after the 1st or 2nd stage is close to the original sample. This confirms about the correctness of the regeneration process with the selected alkali. Acid number during freezing with the original perlite, increases from 0.3 mg KOH/g to 0.36 mg KOH/g after 60 days of storage. With regenerated pearlite samples, the acid number increases from 0.3 mg KOH/g to 0.45 mg KOH/g after 60 days, confirming the presence of residual alkali or other impurities in the pearlite.

Thus, regeneration with alkali will save imported filter material, which will reduce the cost of vegetable oil. It was found that with increasing the concentration of alkali up to 40% at various ratios, the degree of purification of vegetable oil increases, but it decreases with increasing the degree of regeneration. With increase in acid concentration leads to increase in distilled water which leads to increase in production cost and waste of water. It is determined that regenerated perlite after freezing filters saturated fatty acids, because when the temperature decreases with waxy substances, also saturated fatty acids begin to crystallize and feed with the filtering agent in the precipitate. This increases the unsaturated fatty acids in the system.

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