INVESTIGATION OF THE PHYSICOCHEMICAL PROPERTIES OF RECOVERED OIL BY DETERMINING THE OPTIMAL AMOUNTS OF ACIDS AND ADSORBENTS IN THE USED OIL RECYCLING PROCESS

T.E. Juraev[1*], O.Y. Ismailov[1], Y.Y. Yakubov[1], A.U. Auesbaev[1,2,3], D.N. Isamatova[2], D.P. Radjiboev[4], M.J. Boltaboeva[1]

[1]Institute of General and Inorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan, 77a Mirzo-Ulugbek ave., Tashkent, Uzbekistan
[2]Branch of Gubkin Russian State University of Oil and Gas (NRU) in Tashkent
[3]Karakalpak State University named after Berdakh, 1 Ch. Abdirov str., Tashkent, Karakalpakstan, Uzbekistan
[4]Tashkent State Technical University named after Islam Karimov, University Street 2, Tashkent, Uzbekistan
**e-mail: tolqinjonjorayev15@gmail.com*

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Abstract. *This study explores the purification methods of used lubricating oils from internal combustion engine lubrication systems using hydrochloric acid (HCl), nitric acid (HNO_3), sulfuric acid (H_2SO_4), acetic acid (CH_3COOH), and trichloroacetic acid (CCl_3COOH). The research focuses on the physical and physicochemical properties of the treated oils, including kinematic viscosity, viscosity index, density, flash and pour points, water content, total acid number, and infrared (IR) spectroscopy analysis. The results indicate that treatment with acetic and trichloroacetic acids improved the physical and physicochemical properties of the oil, achieving characteristics comparable to new lubricants. Treatment with sulfuric and nitric acids allowed for 70–80% restoration of the oil's properties; however, an increase in the acid content in the adsorbent composition was observed when used to enhance color improvement. In the case of hydrochloric acid treatment, the oil's properties were partially restored, and it was found that unactivated bentonite could yield positive results for improving the color. The findings suggest that the proposed oil regeneration process can partially meet the demand for fresh lubricants and contribute to reducing the overall cost of oil production.*
Keywords: *used oil, bentonite, regenerated oil, acids, viscosity, density, flash point, pour point, acid number.*
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1. Introduction

Engine oil plays a crucial role in global industry, transportation, energy, and various other sectors. Different types of oil samples are used across industrial enterprises, transportation, and the energy sector. This leads to an increasing demand for oil year by year [1]. The growing demand for oil cannot be entirely met by the lubricants derived from petroleum refining. Due to the high demand, the capacity to proportionally increase oil production is very limited.

Various types of lubricants, based on different compositions, are widely used to reduce friction coefficients in mechanical systems. During operation, engine oil becomes contaminated with metal particles (iron, copper, lead, zinc, barium, and cadmium), sulfur compounds, and water. This contamination reduces the lubricating properties of the oil and increases its abrasive characteristics [2]. Additionally, the organic components of lubricating oil undergo changes over time due to operational conditions.

The presence of such contaminants in used oil makes its disposal through open combustion or direct environmental discharge highly hazardous to the environment. Considering this, it is necessary to develop and implement new methods for recycling used engine oil [3-4].

The recycling of used oil began as early as 1930, and since then, different countries have developed their own approaches based on their needs and technological capabilities [5]. Several methods for recycling used oils exist, including traditional acid adsorbent treatment [6-7], solvent extraction, vacuum distillation, and membrane filtration methods [8].

Among these, the acid adsorbent method is particularly noteworthy due to its low cost, simple equipment requirements, and minimal need for specialized expertise [9-10]. Considering these advantages, our study focuses on purifying used oil through the acid-adsorbent method [11-12].

The objective of this study was enhancement of the physicochemical properties and operational efficiency of purified oil by determining the optimal amounts of acids and adsorbents in the used oil recycling process. Improving the technology for obtaining base oil from waste lubricants significantly reduces the production cost of the final product. At the same time, it helps to eliminate environmental problems associated with the improper disposal of used oils, thereby minimizing their negative impact on the environment, particularly on atmospheric air.

2. Experimental part

2.1. Materials. As the object of the study, used automotive oil samples were collected from oil depots and oil change stations. The collected used oils were stored in a dark place at room temperature for a certain period to allow the settling of larger particles before further processing.

The subject of the study included the use of certified acids such as hydrochloric (HCl), nitric (HNO_3), sulfuric (H_2SO_4), acetic (CH_3COOH), and trichloroacetic (CCl_3COOH) acids, as well as unactivated bentonite and an aqueous solution of liquefied alkali. For the separation of larger particles in the oil, a “Labnet International” Z-206-A centrifuge was used. The spectral analysis of waste and regenerated oils was performed using a PerkinElmer infrared (IR) spectrometer.

To determine the physical properties of the purified oil, the following equipment was used: Viscosity was measured using the "Haake Viscotester 1 Plus" by Rheology Solutions; density was analyzed with the "DMA 501" by Anton Paar; flash point was determined using the "TVZ-LAB-12" by LOIP; pour point was measured with the "ATP-LAB-12" device; and magnetic stirrers were also utilized during the study.

2.2. Experimental Methodology

Sedimentation of Particles Using a Centrifuge

To separate metal shavings, sand particles, and other relatively large contaminants present in used oil, a "Labnet International" Z-206-A centrifuge was used. Four 25 ml containers were filled with used oil samples and centrifuged at 2000 rpm for 20 minutes. This method was used to prepare the used oil samples for further processing.

Methodology for Determining Water Content in Used Oil

To determine the water content in used oil, 100 ml of each sample was distilled in a laboratory flask. The percentage of water content in the oil was calculated using the following formula:

$$H_2O = \frac{Volume\ of\ Dehydrated\ Oil}{Initial\ Volume\ of\ Oil} \times 100$$

Acid Treatment Methodology

Used oil samples that had been centrifuged were measured and prepared by adding 100 ml of used oil into a 500 ml beaker. For acid treatment, 15 ml of hydrochloric acid (31.5%), 12 ml of nitric acid (66.47%), and 10 ml each of sulfuric acid (93.6%), acetic acid (99.8%), and trichloroacetic acid (99.5%) were measured separately in 50 ml beakers. The used oil samples were placed on a magnetic stirrer and gradually mixed with the prepared hydrochloric acid at 50 rpm and 40–45°C. The stirring process continued for 20 minutes. The same procedure was applied for the other acids in separate treatments.

Sedimentation and Decantation Process

To enhance the efficiency of oil separation after acid treatment, sedimentation and decantation processes were performed. At the final stage of acid treatment, the acid-treated oils were transferred into separatory funnels.

Hydrochloric and nitric acid-treated oils were sedimented at room temperature for 48 hours. Sulfuric, acetic, and trichloroacetic acid-treated oils were sedimented for 24 hours.

Preparation of Adsorbent for Oil Property Enhancement

For the adsorbent preparation, bentonite clay obtained from the "Bolghali" quarry was cleaned of sand and stone particles. A homogeneous mixture was prepared by adding 85 ml of distilled water to 200 g of bentonite. This mixture was dried in a muffle furnace at temperatures up to 1000°C. To improve the color and properties of the used oil, the purified bentonite was used. The process involved heating 100 ml of used oil to 110°C using a magnetic stirrer, gradually adding 15 g of bentonite while continuously stirring for 20 minutes; at the end of the adsorption process, the oil was neutralized.

Neutralization Process Using Sodium Hydroxide

During the neutralization stage, a 10% sodium hydroxide (NaOH) solution was added to the purified oil to compensate for the increased acid content. The oil was continuously stirred for 10 minutes to ensure thorough neutralization. After the neutralization process, the oil was left at room temperature for 24 hours in a separatory funnel to undergo sedimentation. As a result, the oil separated into two layers: The lower layer (sediment) was removed; the clear upper oil layer was subjected to filtration.

To determine the total acid number (TAN), the following steps were performed: 10 ml of lubricating oil was measured into a conical flask; A few drops of phenolphthalein indicator were added; NaOH solution was added from a burette and titrated until a pink color appeared.

The acid value was calculated using the following formula [18]:

$$Acid\ Value = \frac{Volume\ of\ NaOH\ (ml) \times\ Normality\ of NaOH\ \times\ 40}{Massa\ of\ Oil\ (g)}$$

Filtration Process. The final stage of the process is filtration, which was carried out using a Büchner funnel and filter paper. The filtration was performed under vacuum conditions using a vacuum pump with a flow rate of 0.9 s^{-1} to ensure the effective removal of impurities.

3. Results and discussion

3.1. IR Spectroscopy Results of Primarily Settled Used Oil. To determine the functional groups present in the used oil, an infrared (IR) spectroscopic analysis was conducted using an IR-Spectrometer. Initially, the IR spectrum of the untreated used oil was analyzed. The obtained results are presented in Fig. 1.

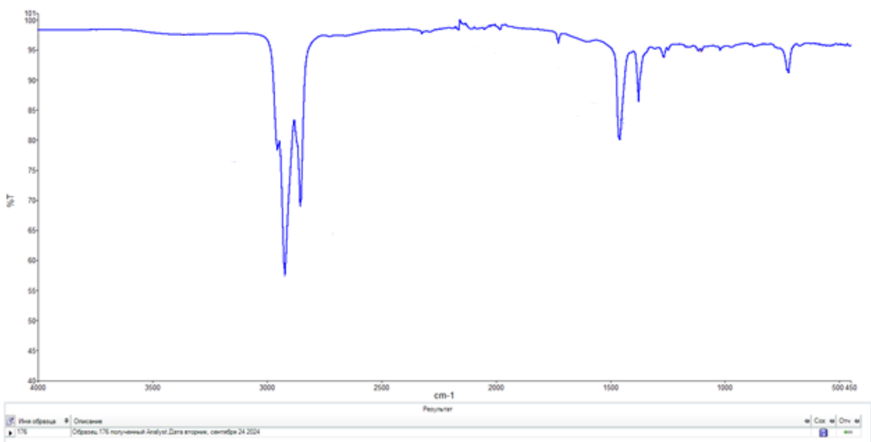

Fig. 1. Infrared Spectroscopy analysis of used oil

The 1727.2 cm^{-1} peak in Fig. 1 indicates the presence of a carbonyl (C=O) functional group, which is typically found in esters, ketones, or carboxylic acids. This peak in the oil composition suggests the presence of fatty acids or esters. The 2622.03 cm^{-1} peak is associated with carboxyl (-COOH) functional groups, indicating the presence of free fatty acids in the used oil. The identified peak at 3600 cm^{-1} corresponds to O-H stretching vibrations, which suggests the presence of alcohols or carboxylic acids in the oil. This IR spectroscopic analysis confirms that the used oil contains several additional functional groups, indicating the presence of a certain amount of water, the existence of free fatty acids, and a high degree of oxidation in the oil.

3.2. IR Spectroscopy Results of Oil Regenerated Using Nitric Acid. The IR spectroscopic analysis results of the oil regenerated using nitric acid are presented in Fig. 2.

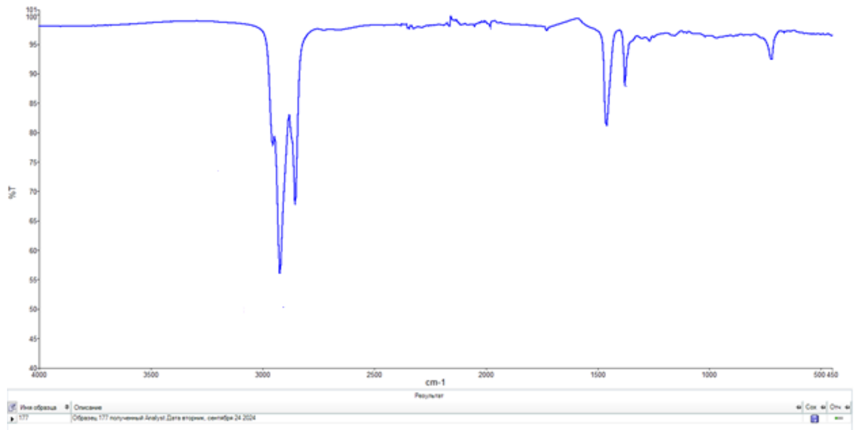

Fig. 2. IR Spectrum of oil regenerated using nitric acid

In Fig. 2, the IR spectrum of oil regenerated using nitric acid (HNO_3) shows a 1727.3 cm^{-1} peak, similar to the 1727.2 cm^{-1} peak observed in the unprocessed oil (Fig. 1). In both cases, this indicates the presence of a carbonyl (C=O) functional group, typically found in esters, ketones, or carboxylic acids. However, in the purified oil, this peak appears slightly weaker, suggesting a partial removal of free fatty acids or oxidation by-products. Additionally, in the unprocessed oil (Fig. 1), a 3600 cm^{-1} peak was observed, which corresponds to hydroxyl (-OH) groups. This peak is absent in the regenerated oil (Fig. 2), indicating the removal of hydroxyl and water molecules during the purification process.

3.3. IR Spectroscopy results of oil regenerated using trichloroacetic acid. In the next stage of experiments, the used oil was purified using trichloroacetic acid (CCl_3COOH). The IR spectroscopic analysis results of the oil regenerated using trichloroacetic acid are presented in Fig. 3.

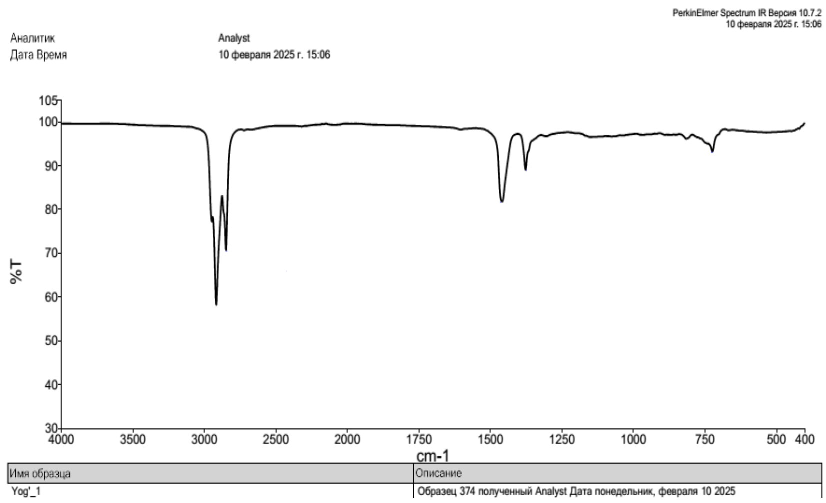

Fig. 3. IR Spectrum of oil regenerated using trichloroacetic acid

In the IR spectrum of the used oil (Fig. 1), a strong peak at 1727.2 cm^{-1} is observed, corresponding to the carbonyl (C=O) group. This indicates the presence of free fatty acids and oxidation products in the oil. From the results shown in Fig. 3, it is evident that trichloroacetic acid (CCl_3COOH) treatment effectively removes free fatty acids and oxidation products from the oil. In the used oil spectrum (Fig. 1), strong peaks at 2954.5 cm^{-1} and 2853.33 cm^{-1} indicate the presence of hydrocarbon chains. After treatment with trichloroacetic acid, the 2853.37 cm^{-1} peak remains, but its intensity is significantly reduced, suggesting the removal of some contaminants or additives (Fig. 3). Additionally, in the used oil spectrum (Fig. 1), a 3600 cm^{-1} peak is present, indicating the presence of hydroxyl (-OH) groups or water. In the trichloroacetic acid-treated oil, this peak disappears, confirming the removal of hydroxyl groups and water residues. The IR spectroscopy analysis confirms that oil treated with trichloroacetic acid shows significant purification from oxidation products, free fatty acids, and water. This enhances the oil's properties, making it more comparable to fresh lubricating oil.

3.4. Analysis of the physical properties of oils. The kinematic viscosity, viscosity index, density, flash point, pour point, water content, cloud point, total acid number, and other physical and operational properties of the regenerated oils were determined through experimental analysis using the acid-adsorption purification method. The physical and operational characteristics of oils reprocessed with different acids are presented in Table 1.

Table 1. Results of oil regeneration

Indicator	SI Unit	New Oil (Standard)	Used Oil	Regenerated Oil + Unactivated Bentonite				
				HCl	HNO_3	H_2SO_4	CH_3COOH	CCl_3COOH
Viscosity (cSt) at 39.2°C	(cSt)	104.2	64.6	76	81	85	89.2	92.4
Viscosity (cSt) at 99.8°C	(cSt)	11.2	6.25	7.4	7.9	8.5	8.8	9
Viscosity Index		92.55	10	31.55	42.57	57.38	58.94	59.19
Density	kg/m³	0.825	0.96	0.937	0.925	0.91	0.885	0.875
Flash Point	ºC	228	180	188	196	216	221	224
Pour Point	ºC	-10	-6	-23	-19.2	-17	-15	-13.8
Water Content	(%)	<0.02	13.6	1.8	1.4	0.85	0.735	0.705
Total Acid Number		0.02	3.36	2.8	2.3	1.8	1.4	1.1

Viscosity is a state function dependent on temperature, pressure, and density and is inversely proportional to temperature. The viscosity of used engine oil is commonly measured to determine contamination levels within the oil. The presence of dissolved and suspended polymerized oxidation products in the oil leads to a reduction in viscosity. A decrease in engine oil viscosity often indicates fuel contamination in the oil composition [13].

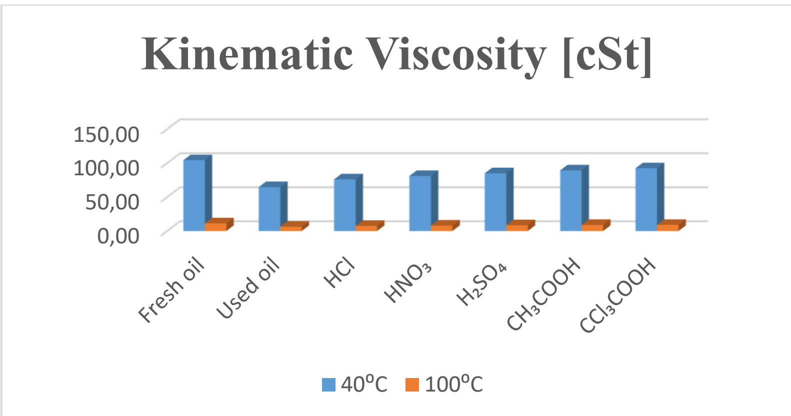

Fig. 4. Dynamics of kinematic viscosity changes

Considering these factors, the viscosity of the oils was measured using the "Haake Viscotester 1 Plus" by Rheology Solutions. The results of the kinematic viscosity analysis of the tested oils are presented in Fig. 4.

The diagram in Fig. 4 illustrates the kinematic viscosity (cSt) of new, used, and acid-treated regenerated oils. The kinematic viscosity of new oil at 40°C is 104.2 cSt, and as the temperature increases to 100°C, its viscosity decreases to 11.2 cSt. The kinematic viscosity of used oil before treatment is 64.6 cSt at 40°C, which drops to 6.25 cSt at 100°C. Among the regenerated oils, the best viscosity values were observed for oil treated with trichloroacetic acid, where the viscosity at 40°C was 92.4 cSt, decreasing to 9 cSt at 100°C.

One of the key performance indicators of oil is its viscosity index, which is an empirical parameter that quantifies the effect of temperature changes on oil viscosity. A higher viscosity index indicates that the oil experiences less viscosity change with temperature variations, which is critical for engine stability under extreme operating conditions. The viscosity index is influenced by the presence of aromatic hydrocarbons and volatile compounds in the oil. The lower the proportion of aromatic hydrocarbons and volatile compounds in the oil, the higher the viscosity index, ensuring better thermal stability and improved low-temperature fluidity [14]. The viscosity index calculations were performed using a viscosity index calculation tool [15], and the results are presented in Figure 5.

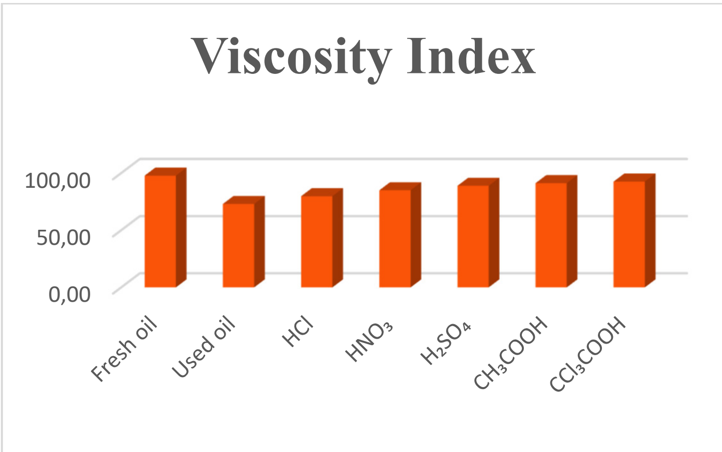

Fig. 5. Viscosity index of oils

The viscosity index of new oil is 97.8, but due to degradation and contamination, the viscosity index of used oil decreases to 73.

When used oil is treated with acids, the best viscosity index values were obtained with acetic acid treatment (58.94) and trichloroacetic acid treatment (59.19).

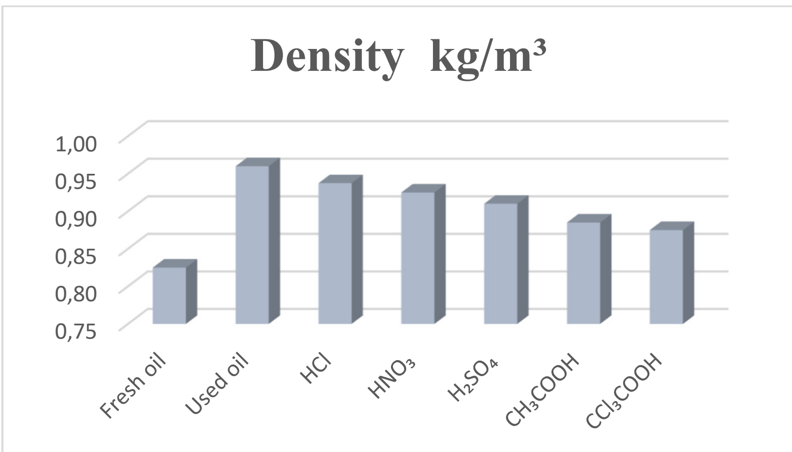

Fig. 6. Density parameters of oils

Density is a crucial parameter for thermal-physical classification of oils. It plays a significant role in determining the thermodynamic properties, viscosity, and heat transfer coefficient of the oil. The molecular interactions within the oil influence its chemical composition. An increase in aromatic compounds leads to higher relative density, whereas an increase in saturated compounds results in lower relative density [16]. To assess the effect of various acids on used oil, density measurements were conducted using the DMA 500 device. The experimental results are presented in Fig. 6.

The diagram in Fig. 6 presents the density variations of new, used, and acid-treated regenerated oils. The density of used oil (after 7000 km of engine operation) is 960 kg/m³, which is 1.16 times higher than the density of new oil (825 kg/m³). After acid treatment, the lowest density values were recorded for acetic acid treatment (885 kg/m³) and trichloroacetic acid treatment (875 kg/m³).

In lubricating oils, the flash point is the temperature at which a sufficient amount of vapor is released to form a flammable mixture with air. When the oil is heated under specific conditions, it reaches a temperature where flammable vapors ignite instantly. Since the flash point indicates the temperature at which flammable vapors are formed, it serves as a key index for fire safety potential [17-20]. To measure the flash point of new, used, and acid-treated regenerated oils, experiments were conducted using the "TVZ-LAB-12" device by LOIP. The experimental results are presented in Fig. 7.

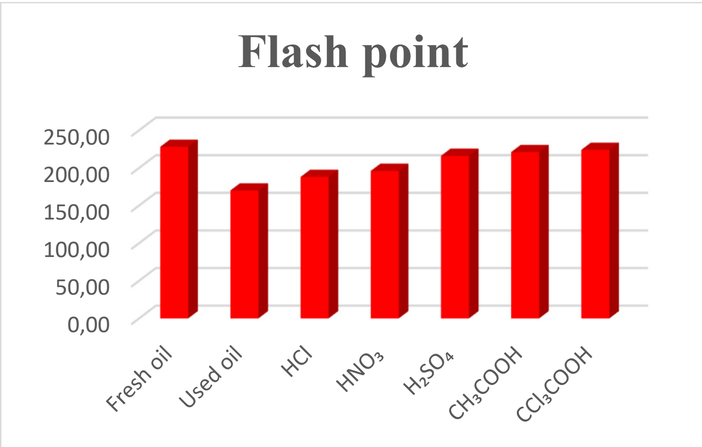

Fig. 7. Flash point of oils

Experimental results indicate that the flash point of new engine oil is 228°C. However, due to contamination and degradation, the flash point of used oil decreases to 180°C. After regenerating the contaminated oil using inorganic and organic acids, the flash point was restored from 180°C to 224°C. The best results were obtained when treating the oil with sulfuric acid, acetic acid, trichloroacetic acid, and unactivated adsorbent.

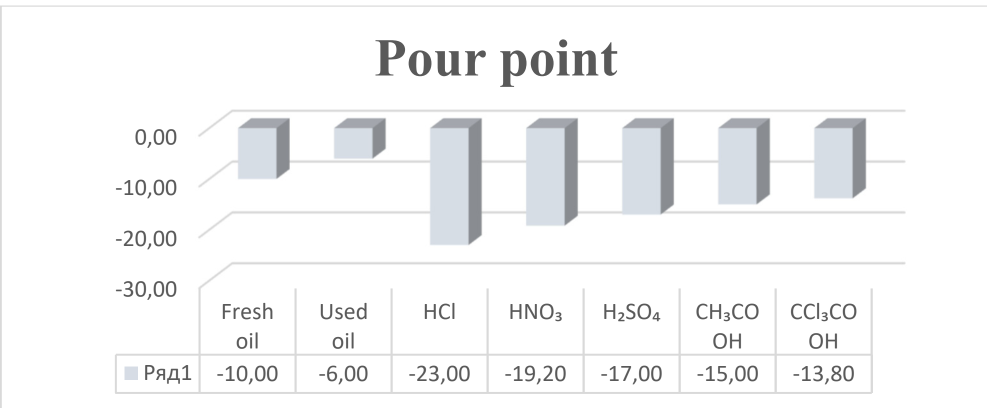

Fig. 8. Pour point of oils

The pour point is one of the critical indicators affecting the stability of machinery in cold weather conditions. The pour point represents the lowest temperature at which the oil remains fluid and operational in mechanical systems [17-22]. To determine the pour point of the oils, experiments were conducted using the "ATP-LAB-12" device. The experimental results are presented in Fig. 8.

The diagram in Fig. 8 presents the pour point temperatures of new, used, and acid-regenerated oils. The pour point of new oil is -10°C, while in used oil, this value increases to -6°C, indicating a reduction in low-temperature fluidity due to contamination. After treatment with trichloroacetic acid, the best pour point improvement was achieved, reaching -13.8°C, making the oil more stable in cold operating conditions.

During mechanical operation, the moisture content in regenerated oil increases due to technological processes within the engine and exposure to atmospheric humidity. The results of water content analysis are presented in Fig. 9.

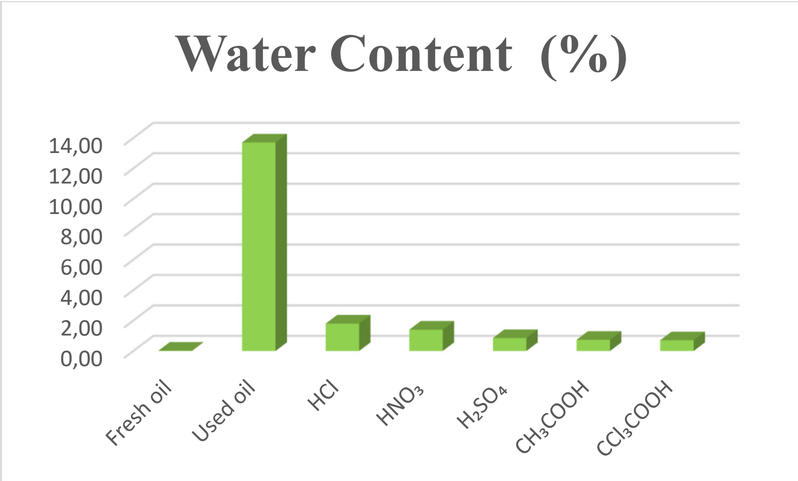

Fig. 9. Water content in oils (%)

The water content analysis results presented in Fig. 9 indicate that, according to standards, the water content in new oil should be below 0.02%. In used oil, the water content significantly increases to 13.6%, primarily due to contamination and exposure to moisture during engine operation. Among the proposed purification methods, the best result was achieved with trichloroacetic acid treatment, reducing the water content to 0.705%.

The presence of acidic and basic components in oil is measured by the Total Acid Number (TAN). TAN is one of the key indicators used to determine the oxidation level of used lubricating oil [1]. The experimental results of TAN measurements are presented in Fig. 10.

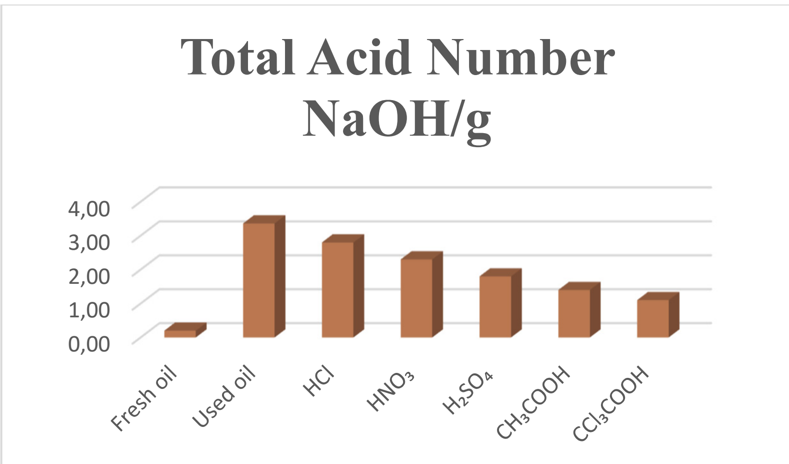

Fig. 10. Total Acid Number in oils

The diagram in Fig. 10 shows the total acid number variations in new, used, and regenerated oils. New oil has an acid content of 0.2, while in used oil, the acid content increases significantly to 3.36 due to oxidation and contamination. After acid treatment, the best reduction in acid content was achieved, bringing it down to 1.1.

4. Conclusion

In conclusion, it can be stated that the regeneration of used lubricating oils from internal combustion engines using hydrochloric, nitric, sulfuric, acetic, and trichloroacetic acids, along with unactivated bentonite as an acid-adsorbent, resulted in improvements in the oil's properties. Specifically, the viscosity increased by 1.43 times, the viscosity index improved by 5.92 times, the density decreased by 1.09 times, the flash point increased by 44°C, the pour point improved by 2.3 times, the water content was reduced by 19.29 times, and the acid content was lowered by 2.26 times. The experimental results allowed for a comparison of the regenerated oils treated with acetic and trichloroacetic acids to the properties of fresh lubricating oil. When treating used oil with hydrochloric acid, only partial restoration of its properties was observed, making it unsuitable for use as a base oil. Treatment with nitric and sulfuric acids restored 70-80% of the oil's properties; however, the high acid concentration in the sediment residue presents a minor environmental concern. The results obtained from the regeneration of waste oil using acetic and trichloroacetic acids indicate that the regenerated oil closely resembles the properties of base oil. The efficiency of organic acid treatment was found to be up to 75-80%. Consequently, the cost of base oil production is reduced, and the amount of toxic substances released into the environment due to improper waste oil disposal is minimized.

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