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Regeneration of Used Industrial Oils Using Coke

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Abstract

Mitigating environmental pollution caused by industrial waste and hazardous by-products remains one of the key priorities of modern environmental protection. Addressing this challenge requires not only the improvement of existing purification and regeneration technologies but also the development of efficient, cost-effective sorbent materials with enhanced adsorption performance. Used industrial lubricating oils represent a significant category of hazardous waste due to their toxicity, fire risk, and explosion hazard. Consequently, their environmentally sound management and regeneration are essential to minimize adverse environmental impacts and recover valuable resources. Regeneration technologies enable the production of reclaimed oils that satisfy quality requirements while simultaneously reducing the consumption of virgin natural resources, promoting waste valorization, and providing industries with economically viable alternatives to scarce lubricating oils.

Keywords: Used industrial oils, regeneration, petroleum coke, adsorption, adsorbent, activated petroleum coke, thermochemical activation, acid activation, porous structure, micro- and mesopores, oil purification.

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1. Introduction

Under high-temperature conditions, hydrocarbons in oils undergo oxidation, thermal decomposition, polymerization, and condensation reactions in the presence of atmospheric oxygen [1–8]. In addition, during operation, oils are continuously exposed to wear products from metal friction surfaces and become contaminated with road dust and water. As a result, various impurities accumulate in the oil, including tar and asphaltic agglomerates (sludge), colloidal coke and soot particles, salts, organic acids and other acidic compounds, fuel residues, metallic dust and chips, mineral particulates, fibrous materials, and water.

During machine operation as well as storage and transportation, lubricating oils are in constant contact with atmospheric oxygen. This interaction represents the primary factor responsible for chemical degradation processes in oils, particularly oxidation.

According to N.I. Chernozhukov and S.E. Crane [4], aromatic hydrocarbons in petroleum-based oils exhibit the highest resistance to oxidation, while naphthenic hydrocarbons show intermediate stability, and paraffinic hydrocarbons are the most susceptible to oxidative degradation at elevated temperatures. During oil processing, a small fraction of resins remains, which can act as natural antioxidants. Oxidation of resinous substances leads to the formation of insoluble condensation products such as asphaltenes and carbenes.

At temperatures up to 20–30 °C and atmospheric pressure, the oxidation process proceeds slowly;

however, its rate increases significantly with rising temperature [5–12]. At temperatures of 270–300 °C and above, oxidation is accompanied by intensive thermal cracking of hydrocarbons, resulting in the formation of sulfur dioxide (SO_2), water, and carbonaceous residues.

2. Methods

The rate of hydrocarbon decomposition increases with rising temperature and, within a specific range (400–450 °C), follows the Van't Hoff rule, according to which the reaction rate approximately doubles for every 10 °C increase in temperature.

At temperatures up to approximately 425 °C, paraffinic molecules typically undergo cleavage into nearly equal fragments. At higher temperatures (around 600 °C and above), the decomposition pathways shift toward the formation of high-molecular-weight unsaturated alicyclic hydrocarbons as well as lighter kerosene-range products.

Iso-paraffinic hydrocarbons exhibit a higher susceptibility to thermal degradation compared to normal paraffins, with fragmentation primarily occurring at side chains.

In contrast, saturated aliphatic hydrocarbons demonstrate relatively higher thermal stability. Under severe thermal conditions, these compounds may undergo polymerization, followed by secondary decomposition of the resulting products, ultimately leading to the formation of unsaturated hydrocarbons through successive reaction stages.

$$C_nH_{2n} + C_mH_{2m} \rightarrow C_{n+m}H_{(n+m)}$$
$$\downarrow$$
$$C_{n+m-k}H_{2(m+n-k)} + C_kH_{2k}.$$

The thermal decomposition of aromatic hydrocarbons primarily involves the cleavage of their side chains, which subsequently undergo further breakdown in a manner similar to that observed for kerosene-range hydrocarbons.

The conventional decomposition of peroxides formed during oxidation proceeds under thermal influence, resulting in the generation of alcohols and carbonyl compounds. In this process, primary hydroperoxides typically yield aldehydes, whereas secondary and tertiary hydroperoxides lead to the formation of ketones and alcohols.

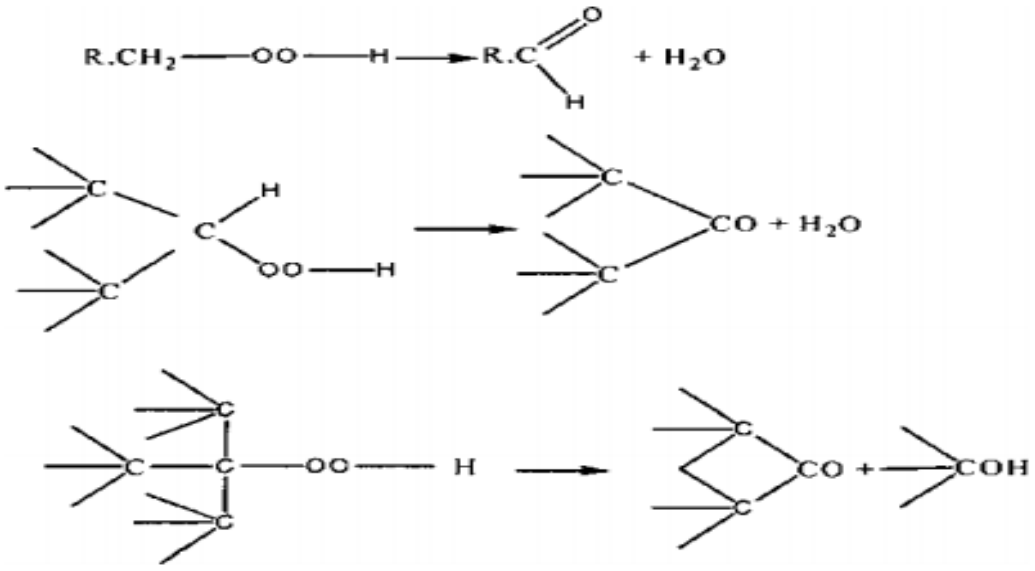

Figure 1. primary hydro peroxides are aldehydes, secondary and tertiary ketones and alcohols.

Alcohols and aldehydes are typically formed with one fewer carbon atom than the parent hydrocarbon, whereas ketones retain the same carbon skeleton as the original hydrocarbon molecule.

When lubricating oils come into contact with heated machine components, thermal cracking reactions occur, leading to the formation of both light volatile fractions and heavier degradation products. The susceptibility of mineral oils to thermal decomposition is primarily

governed by their hydrocarbon composition. In general, the more extended and structurally complex the hydrocarbon molecules are, the more readily they undergo thermal breakdown under elevated temperatures.

During operation, lubricating oils are also contaminated by various mechanical impurities, including dust, sand, metallic particles, water, resins, asphaltenes, coke, and carbonaceous matter, without altering the fundamental chemical base of the refined oil.

Among regeneration techniques, physical purification methods such as suction, centrifugation, filtration, and water washing are most widely applied. Sedimentation represents the initial and obligatory stage of the regeneration process, during which mechanical impurities and water are removed through natural settling under gravitational forces. The efficiency of sedimentation depends on particle size, density, liquid viscosity, and temperature.

As viscosity decreases with increasing temperature, the rate of particle settling improves. However, temperatures above approximately 80 °C do not significantly enhance sedimentation efficiency due to the limited further reduction in viscosity. Considering that water present in oil may begin to boil at around 100 °C, leading to foaming and process instability, the optimal sedimentation temperature range is typically 80–90 °C.

Analysis of the pore structure distribution of the sorbent indicates that it consists of 73.4% macropores, 3.9% micropores, and 22.7% mesopores. Activation significantly enhances the adsorption properties of the sorbent. Acid activation increases the specific surface area up to 462.3 m²/g (approximately 1.5 times), while thermochemical activation raises it to 416.1 m²/g (approximately 1.35 times). Correspondingly, the adsorption capacity of the natural and modified (K_2O and TX-treated, respectively) sorbents increases from 0.129 g/g to 0.229 g/g and 0.219 g/g, respectively.

According to the results of the derivatographic analysis, the thermogram of the natural Coke sorbent is shown in Fig. Figure 2.

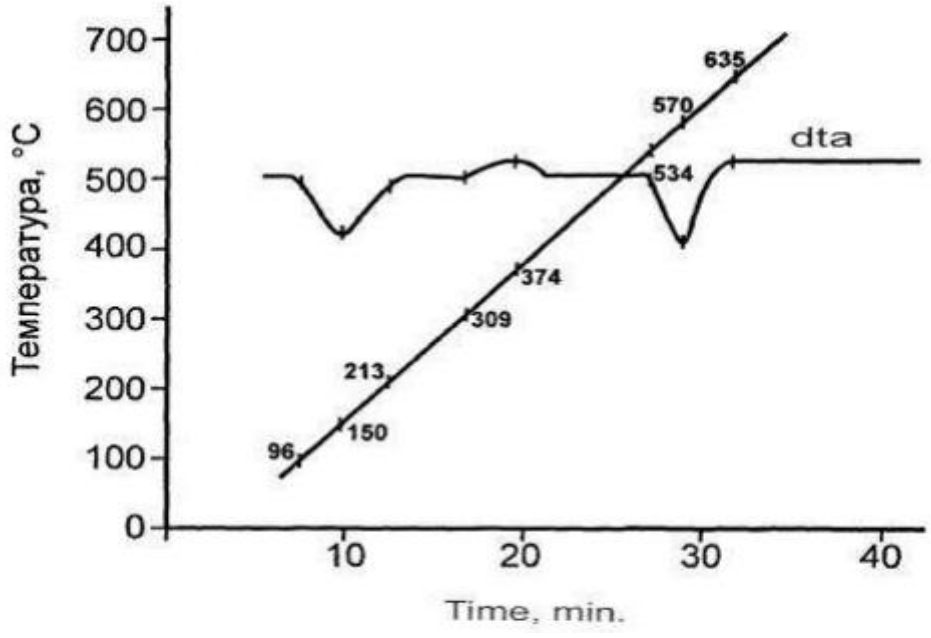

Figure 2. Sorbent thermo gram in the local position

Accordingto the results of the analysis, the thermogram contains:

- endothermic effect in a temperature range of 100-150 °C as a result of the removal of adsorbed moisture;

Endothermic effect in the range of 530-620 °C as a result of removal of crystalline hydrated moisture.

That is, when natural Coke is heated, processes occur associated with the physical removal of moisture and the

destruction of crystalline hydrates. This determines different ways of obtaining and activating sorbents. Table 1 lists the indicators of the porous structure of sorbents obtained from oil Coke. Oil Coke is obtained by heating to 200 °C and holding for 1 hour ("soft" thermal activation). Oil Coke sorbents are obtained as a result of acidic and thermochemical activation of sorbent.

Table 1 sorbents properties

№	Rename	Coke	K_{20}	TX
1	Density, g / cm^3			
	authenticity	2,38	2,30	2,42
	accuracy	1,15	1,01	1,14
2	Porosity,%	51,68	56,61	52,89
3	Relative density, g/cm^3	0,88	0,79	0,86
4	Static activity on water, g H_2O/g as	0,129	0,229	0,219

When activated, the porosity of the sorbent increases. In particular, with acid modification, porosity increases by 10.9%, with thermochemical-by 2.3%, which corresponds to the literature. Balance static activity on N2O is also growing.

Activated sorbent enters reactor 2 for regeneration, where oil also enters. Temperature 80°C. after recovery, the suspension is divided into a filter press. The oil is sent to the consumer, and the adsorbent is sent for regeneration or destruction.

3. Conclusion

The share of macro, micro and mesopores is 73.4, 3.9 and 22.7%, respectively.With acid and thermochemical activation, the specific surface increases by 1.35 and 1.5 fold, respectively, and the adsorption capacity by 1.7 and 1.78 times.The composition of micro and mesopores is also growing.Activation oil Coke significantly increases the adsorption properties of the sorbent and allows you to obtain activated charcoal with high adsorption structural properties.

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