Experimental study of highly alkaline motor oils in express diagnostics by drop test method

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Abstract. In order to maintain the reliability and durability of the internal combustion engine during the operation of machines, systematic and operational monitoring of the state and level of technical fluids is necessary, where the most informative indicator is the change in the state of motor oils. The drop test method has been used for express diagnostics of motor oil for many years. Grades of oils with high base number, which are mainly used for high-speed internal combustion engines when diagnosing the state of these oils using the drop test on the surface of the filter used for analysis (Blue Tape filter paper), do not form zones in the form of rings, the change in diameter of which allows evaluating the operational properties of the operating motor oil. The study established that when analyzing the preservation of the properties of strongly alkaline motor oils (assessment of the contamination coefficient, dispersing properties), a spot of dirty oil is formed on the chromatogram of filter paper thus showing the filter structure. Theoretical studies revealed that the segregation into zones during the analysis of motor oils with strongly base number does not occur due to the action of electrical forces leading to the repulsion of contaminant particles. Electrical forces arise due to changes in molecules and alkali when a drop of oil flows through the surface of the filter, this interaction continues, and therefore, to analyze strongly alkaline motor oils, the base number must be neutralized, and a correction factor for the neutralizer must be introduced. The paper presents the results of experimental studies to determine the effective neutralizer of the alkalinity of a working motor oil in the process of diagnosing oil by the drop test, which made it possible to obtain concentric zones on the surface of filter paper to calculate the dispersing ability and other estimated oil indicators.

1. Introduction

The durability of the internal combustion engine (ICE) is directly related to the quality of technical fluids (motor oils, cooling fluids). The reliable operation of friction pairs in the internal combustion engine is only ensured by motor oil with regulatory operational properties [1, 2, 3]. Previously used to analyze the properties of motor oil in ICE, the simplest drop test method, which is sufficiently detailed and available in the technical literature, cannot be used to diagnose and analyze modern strongly alkaline grades of oils. During the research the authors considered the issue of impossibility of oil drop segregation into zones during the analysis of modern oils [4, 5]. This is an irreparable drawback that makes the drop test unsuitable for diagnosing strongly alkaline oils, or the formation and manifestation of concentric zones on filter paper is a consequence of the influence of some factors, for example, electrical repulsion forces formed due to dissociation of alkali molecules into ions [6]. Repulsion
forces when the droplet spreads over the surface of the filter paper prevent coagulation of carbon black particles, resinous oil compounds and, as a result, a continuous dark spot is formed on the filter [7].

Theoretical conclusions were confirmed by experimental studies, for this the authors developed the methods of some experiments and compiled an experiment scheme. Motor oils of Neste Tyrbo LXE SAE 15W-40 and BP Vanellus C6 Global (U) 15W-40 were used as the analyzed brands of motor oil.

2. Materials and methods
The experimental scheme and methodology included the following:
1. Sampling of strongly alkaline motor oils from tractors operating in the farms of Omsk Region;
2. Benchmark survey for the selection of effective solution for neutralization of alkalinity and its optimal concentration;
3. Study of the influence of total contamination and base number of an oil sample on the formation of oil segregation zones on a Blue Tape filter.

The samples were taken during routine maintenance in a farm, the sampling frequency corresponded to 250 motor hours.

Thus, the methods of observation in the operating conditions of machines, methods of mathematical modeling and statistical processing of information were used as research methods.

3. Research results
When analyzing a motor oil with high base number using the drop test, a black or grey (depending on the contamination of the sample) chromatogram (Fig. 1) displaying filter pores is formed on the filter, and this makes it impossible to determine the functional properties of the analyzed oil.

![Fig. 1. Image of oil chromatograms (a) – BP Vanellus C6 Global (U) SAE 15W-40; (b) – Neste Tyrbo LXE SAE 15W-40](image)

During the experiment to determine a suitable type of oil alkalinity neutralizer, the following acid solutions were tested: sulfuric, hydrochloric, phosphoric, nitric, and acetic.

The effect of acids on the formation of concentric zones on the filter was assessed by the percentage of acids in the solution for filter impregnation. The influence of the variable concentration was evaluated visually according to the accepted patterns [8]. To conduct the experiment, the authors adopted patterns that ranged from “absolutely black body” to various degrees of gray color. The examples of sample patterns are presented in Figure 2.

The variable (acid percentage) was varied by 10 values ranging from one percent to ten. The result of the experiment was the production of the most suitable sulfuric acid from the tested acids, which, in a wide range of concentrations, ensured the separation of the oil drop on the filter, as a result, a chromatogram was obtained corresponding to the drop test method used to analyze the operating motor oil.

Subsequently, a rapid coagulation threshold was determined to evaluate the coagulation capacity of sulfuric acid, for which purpose a settling method was used. The test tube was filled with a 100 ml oil sample and a different volume of sulfuric acid was added according to the sample numbers as a base number neutralizer, which corresponded to the percentage of the concentrated acid.
When mixing acid with a sample of oil, the contents of the tubes were settled, then the intensity of sludge of resinous substances and carbon black was recorded over various times, the range was selected every 30 seconds.

When processing the results of settling the test tubes with oil and sulfuric acid, the authors obtained the following dependence [9]

\[
Q = 98.01 \left( 1 - \frac{1}{e^{\frac{\tau}{60}}} \right) \left( 1 - 1^{\frac{1}{1+\alpha}} \right),
\]

where \(\tau\) – settling time of an oil sample with acid, s;
\(\alpha\) – percentage of acid, %.

The calculation of oil settling is presented in Table 1, according to these data, graphs of experimental determination of oil sample settling, shown in Figure 3, were built.

Table 1. Results of study and calculation of carbon black and resinous substances deposition in the studied acid solution and oil sample

| Time \(\tau\), sec | 1    | 2    | 3    | 4    | 5    | 6    | 7    | 8    | 9    | 10   |
|------------------|------|------|------|------|------|------|------|------|------|------|
| 30               | 19.5 | 30.9 | 34.8 | 36.4 | 37.1 | 37.7 | 37.9 | 38.8 | 38.9 | 38.7 |
| 60               | 30.7 | 49.4 | 55.9 | 58.4 | 59.8 | 60.7 | 60.9 | 60.9 | 61.2 | 61.4 |
| 90               | 38.8 | 61.9 | 68.5 | 71.6 | 73.2 | 74.2 | 74.6 | 74.9 | 75.2 | 75.3 |
| 120              | 42.3 | 67.7 | 76.2 | 79.7 | 81.4 | 82.4 | 83.5 | 83.4 | 83.7 | 84.7 |
| 150              | 70.3 | 85.9 | 91.6 | 95.4 | 97.5 | 98.7 | 98.6 | 99.2 | 99.2 | 99.5 |
| 180              | 46.5 | 74.44| 83.8 | 87.6 | 89.5 | 90.6 | 91.2 | 91.8 | 91.9 | 92.2 |

Graphs of the dependence of the coagulation \(Q\) on the acid content in the solution \(\alpha\), the settling time \(\tau\) made it possible to clearly determine the dependence of the deposition of carbon black and resinous substances in motor oil (Fig. 3).

The analysis of the obtained dependencies made it possible to establish that with an increase in the deposition time and the amount of acid in the oil sample, the deposition intensity increases. With the same acid content in the solution and an increase in time, the amount of settled carbon black grows, but the deposition rate slows down [10, 11]. Thus, with one percent acid content and a deposition time of 30 seconds, 19.5% carbon black is settled, with an increase in deposition time at the next fixed stage (60 seconds), 30.7% is deposited, the increase is 11.7%, at the next stage – 120 seconds and the amount reaches 42.3%, and at 180 seconds the content of settled carbon black increases slightly and amounts to 46.5%. The deposition increases by 4.2% as the deposition time increases from 120 to 180 seconds. The obtained data may be associated with the lack of the amount of acid introduced to
completely neutralize the resulting repulsion forces. The increase in the deposition of particles at other values of introduced acid is similar to the increase given above, however, the percentage of residual impurities depending on the acid content in the test tube increases in direct relation [12].

![Figure 3. Dependence of Q deposition on the percentage of acid in solution $\alpha$ and the settling time $\tau$](image)

The change in the amount of deposition depending on the amount of introduced sulfuric acid has power dependence. As the amount of acid in the oil changes, the deposition rate changes [13, 14]. At the same time, an increase in the deposition rate was observed up to 5% acid in the solution. With an increase in the amount of sulfuric acid above 5%, the deposition rate slows down, which may be explained by a decrease in the ions of non-neutralized alkali in oil. The reduction of alkali ions leads to the reduction of repulsion forces, which may indicate complete deposition of resinous substances and carbon black in the sample [15]. As a result of the analysis of the obtained dependencies, it can be concluded that the optimal acid content in the solution is in the range of 5-6%. A further increase in acid is not feasible due to a decrease in the deposition rate. As the amount of acid increases, the filter paper used for the analysis loses its properties and becomes unsuitable for analysis.

4. Conclusion
Scientific research was carried out using the equipment of the collective use center of Omsk SAU “Additive Technologies and Material Processing”. As a result of the study conducted to determine the most effective alkalinity neutralizer in the diagnosis of motor oils by the drop test method, the following conclusions can be drawn:

- an effective neutralizer of the base number of the oil drop was selected during its analysis – sulfuric acid, which allows dividing the analyzed sample into annular zones to determine the properties of oil.

- the optimal acid content of the solution, which is $\alpha=5-6\%$ for impregnation of filter paper, was established, which allows the maximum deposition of the components of carbon black and resinous substances contained in the oil sample, as well as preservation of the strength properties of the filter paper after treatment with the acid solution.
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