European Journal of Science, Innovation and Technology

ISSN 2786-4936

Vol. 1 No. 6 (2021)

Comparison of Parameters of Two Lubricating Oils Treated with Activated Clays of Karaqeva Region, Kosovo

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Abstract. The use of clays is well known in the treatment and purification of water discharges and the recycling of hydrocarbon products. Various branches of industry, agriculture and transport, use significant amounts of lubricating oils in the various machinery and vehicles they use. The quality of lubricating oils plays an important role in the safety and longevity of machinery, equipment and vehicles. The selection of lubricating oils with the appropriate physico-chemical parameters directly affects the work efficiency of vehicles, machinery and industrial equipment. Adherence to the standard of lubricating oils affects the continuity of work, prevention of breakdowns, carries significant economic costs and environmental effects. Some of the main parameters for evaluating the quality of lubricating oils are: density, sulfur content, viscosity, viscosity index, pour point, ignition temperature and flame temperature, etc. Since the amount of lubricating oils consumed is considerable, the need arises to treat and recycle them, applying methods with a low cost and environmentally friendly. The purpose of this paper is the recycling of used lubricating oils (after the realization of 15,000-20,000 km and 40,000-50,000 km), through their treatment with clays (bentonite), activated by three different methods (acid, base and combined) of the Karaqeva region, Kosovo.

Treatment and purification of lubricating oils used with activated clay (bentonite) is an optimal method. This process consists of removing harmful components from used lubricating oils and rehabilitating them for reuse as a base oil or as a fuel for vehicles and / or industrial machinery.

Keywords: lubricating oils, recycling, activated clays, physico-chemical parameters

Introduction

Economic and industrial development is associated not only with an increase in the number of vehicles and machinery, but also with an increase in the consumption of lubricating oils and the improvement of their quality parameters (Sychra et al., 1981). The service life and safety at work of various vehicles, machinery and equipment depends on the standard of safety and quality of the lubricating oils they use (Low No. 9870/2008). It is already known the importance of using lubricating oils to minimize the frictional forces of moving objects (internal combustion engines, gears, etc.), reduce heat transfer, movement, corrosion protection, etc. (Wills, 1980). Important sectors of industry, transport, agriculture, are widely used by internal combustion engines and their work efficiency is affected not only by the quality of fuel and lubricating oils they use (Mang et al., 2007). Due to the development of the economy and the increase in the number of engines with fuel, the demand for fuels and lubricating oils has also increased. The categories of lubricating oils include several types: engine oil, vehicle gear oil, industrial oil, lubricating oil, etc. (Sariyerli et al., 2018). This paper deals with the evaluation of physico-chemical parameters of lubricating oils used before and after the recycling process with activated clays, referring to the

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parameters of two categories of lubricating oils: SA 5W-30 and SN 90 (Lorefice al., 2017). Clay is a group of substances (SiO_2, Al_2O_3, Fe_2O_3, MgO, CaO, SO_3) derived from the degradation of feldspars (aluminosilicates) which in the dry state appear as crumbly soil, while in the wet state they form a plastic mass (Velde, 1995). Clay has absorbent properties and contains cations of alkaline and alkaline-earth metals, which can be exchanged with other cations, during contact with different fluids (ASTM C114). The clays are hydrated alumosilocates with a particle structure with dimensions of about 2-4 μ and with a large specific surface area of about 23,000 cm^2/g. The clays present chemical and mechanical stability, have a layered structure and high ion-exchange ability. Clay is a material with good absorbent properties and is used in the construction industry, metallurgical industry, chemical industry, pharmaceutical and cosmetics industry, water treatment plants, etc. (Raussell-Colom et al., 1987). The process of adsorption through clays (bentonites) is a well-known technique used to remove heavy metals from water discharges and lubricating oils used, because it has low cost, high efficiency and ease of application (Steudel et., al 2009). During the adsorption process the contaminants are adsorbed by the clay, leaving the solution in which they are located. This process of absorption through clays minimizes contamination by fluids and is an important part of Green Chemistry (Christidis et al., 1997; Babel et al., 2003). Natural and modified clays (with acid, base and acid-base combination) are successfully used as absorbents for the removal of heavy metals from the lubricating oils used (Bailey et al., 1999; Kamel et al., 2004; Jovanovic et al., 1991; Shaqour et al., 2017). In the paper is made a comparative assessment regarding the values of physico-chemical parameters of recycled lubricating oils (after the realization of 15,000-20,000 km and 40,000-50,000 km) through their treatment with clays (bentonite), Karaqeva region, Kosovo. These clays have been pre-treated with three methods three different methods (acid, base and combined) (acid, base and combined). The evaluation of physico-chemical parameters of lubricating oils was performed before and after the process of their treatment with activated clays. From the evaluation of the obtained results, it is noticed that the lubricating oils produced from the recycling of used motor oils, have physico-chemical parameters within the standard and can be reused again for the same purposes in industrial machinery and / or transport vehicles (Jafari et al., 2015; Markova et al., 2010).

Materials and Methods

Materials

The evaluation of the quality of lubricating oils is done referring to European standards, which include: density, sulfur content, viscosity, viscosity index, pour point, ignition temperature and ignition. The main lubricating oils are: engine oil, vehicle gear oil, industrial oil, lubricating oil, etc. (Mang et al., 2007; Wills, 1980). Recycling of used lubricating oils is very important not only for the high economic cost they carry, but on the other hand they constitute an important source of pollution for the environment (Jafari et al., 2015). The development of technology is associated with an increase in the level of processing of internal combustion machinery, equipment or engines and at the same time with an increase in quality and a reduction in consumption over a period of time. During use, over time the lubricating oil is denatured by high temperatures and enriched with metal particles produced by the friction of metal surfaces, solid impurities (soot, coke, and corrosion products), moisture, dust and other gases. Meanwhile, the oxidation and nitration process are the main factors that contribute to the degradation of the oil of transport machinery / vehicles (Mang et al., 2007; Jafari et al., 2015). The total surface area and volume of clay pores increases in relation to the acid-based and combined (acid-base) treatment method (Temuujin et al., 2004; Yildiz et al., 2002; Griffiths, 1990).

Methods

Acid activation of clay

Acid activation of dry and crushed clay in the region of Karaqeva, Kosovo was carried out at an average temperature of 100°C with sulfuric acid with respective concentrations of 10%, 30% and 50% and the mixture of sulfuric acid - acetic acid 25% to 25%. This acid activation process took about 2 hours. During acid activation, a partial loss of calcium, magnesium, iron and aluminum from the clay layers occurs, which increases the porosity of the clays.

Clay-based activation

Activation based on dry, crushed and homogenized clay of the Karaqeva region, Kosovo was realized at an average temperature of 25°C with sodium carbonate with respective concentrations of 10%, 30% and 50% and the mixture of sodium carbonate – sodium hydroxide 25% with 25%. This activation process lasted 30 days.

Combined activation

Another alternative that was used is combined activation (acid-base). During this process initially the clay was activated with acid.

Table 1. Clay (bentonite) composition of Karaqeva, Kosovo

Karaqeva bentonite (non activated)	Quartz %	Anorthite %	Clinochlore %	Talc %	Illite %	Beidellite %	Muscovite %	Albite %
-	65.85	4.72	-	-	2.58	2.26	2.06	11.15

The clay was then rinsed with distilled water at 70°C to remove acid. The clay was then activated with the base, as above. The ASTM C114 standard was used to determine the chemical composition of untreated and activated H_2SO_4 clay in Karaqeva clay at concentrations of 10%, 30%, and 50%.

Results and Discussion

Acid activation of clays is a method that is applied to increase their absorption properties. After the modification of the clays a change occurs in their surface, in porosity and in the type and concentration of exchangeable ions. Used lubricating oils have a very low level of biodegradation in nature and cause environmental pollution. The standard test methods applied for the evaluation of oil parameters are according to EN ISO / IEC 17025. The tested used oil is wholly or partly composed of mineral or synthetic oil. In this paper, two types of used motor oils (mineral oil) are examined, respectively with 15,000 to 20,000 km realized and 40,000 to 50,000 km realized. After treatment of the oils with activated clays, physico-chemical parameters were determined, such as: density, kinematic viscosity (40°C and 100°C), viscosity index, sulfur, color and pour point. After the performed tests, the characteristics of the recycled lubricating oil result in compliance with the requirements of the ASTM D4485 standard. Viscosity and viscosity index are important factors that determine the quality of lubricating oils. The viscosity index determines the temperature range in which the viscosity ensures the normal operation of the engine. The higher the viscosity index, the better the quality of the lubricant. Viscosity is characterized by two indicators: kinematic viscosity and dynamic viscosity. Kinematic viscosity: characterizes oil fluidity in the temperature range from 40°C-100°C. This depends on the density of the lubricating oil and the thinner it is, the smaller this indicator is and vice versa. Dynamic viscosity: This indicator does not depend on the density of the lubricating oil. It represents the resistance force that occurs when two layers of oil move, placed at a distance of 10 mm from each other, at a speed of 1 cm/sec. The surface of each layer is 1 cm^2. The methods for determining the dynamic viscosity are: low temperature viscosity and high temperature viscosity, which is determined under conditions similar to those operated by the internal combustion engine. Pour point: Indicates the moment when the oil tends to "thicken" and does not flow freely. Ignition point: Indicates the minimum temperature at which lubricant vapors mixed with air and heated in a standard laboratory container ignite when approaching a flame. This parameter is useful, as it serves as an indicator to indicate possible fuel pollution. The clay was first washed with distilled water to remove unnecessary components contained in it, such as sand, through the process of decantation, filtration, and dried first at a temperature of about 100°C and then at room temperature and finally ground in powder form. Physico-chemical parameters determined for engine oil include: density at 15°C (EN ISO 3675) g/cm^3, kinematic viscosity at 100°C (EN ISO 3104) cst, kinematic viscosity at 40°C (EN ISO 3104) cst, viscosity index (ISO 2909), sulfur in% (m/m), pour point (ISO 3016) and color (EN ISO 2049). Table 2 shows the parameter values of New Motor Oil with additives of type (SAE 5W-30), New Basic Oil (called Base Oil without additives of type (SN90) and the parameters of tested motor oil used with 40,000-50,000 km performed, before and after treatment with Karaqeva Benton activated with acid, base and combined method (acid and base), at a temperature of 120°C for 90 min. physico-chemical parameters after the treatment of used oil with activated clay (bentonite) of the Karaqeva region. Referring to the parameters, we can say that this oil is promising to be reused as a lubricating oil for vehicle engines.

Table 2. Tested parameters of motor oil used before and after treatment with Karateva Benton, activated with acid, base and with the combined method (acid and base), at a temperature of 120°C for 90 min, with 40,000-50,000 km done

Nr	Sample name	Density at 15 °C (EN ISO 3675) g/cm^3	Kinematic viscosity at 100 °C (EN ISO 3104) cst	Kinematic viscosity at 40 °C (EN ISO 3104) cst	Viscosity index (ISO 2909)	Sulphur content % (m/m)	Pour point (EN ISO 3016)
1	**New oil with additives (SAE 5W-30) (reference) reported by producer**	(0.840-0.860) 0.834	(11.3-12.3) 12.1	(7.9-9.5) 72.5	(min 165) min.165	(max 0.5) 0.12	(max.-33) -30
2	**New oil without additives (SN90) (reference) reported by producer**	Raportim 0.843	(min 5.80) 6.2	(min. 31.5) 35	(> 120) 126	(<0.03) ne testim	(-26 máx) -18
3	**Used untreated oil (not filtered)**	0.8673	9.17	50.23	167	0.186	-40
4	**Used oil with not activated clay at 120 °C, 90 min (A.L. 40,000-50,000 km)**	0.8477	11.2	68.21	157	0.106	-37
5	**Used oil with activated clay 10% H_2SO_4 120 °C, 90 min**	0.8726	11.07	67.4	157	0.135	-39
6	**Used oil with activated clay 30% H_2SO_4 120 °C, 90 min**	0.8725	10.78	68.6	147	0.145	-38
7	**Used oil with activated clay 50% H_2SO_4 120 °C, 90 min**	0.8525	10.14	50.11	196	0.112	-39

8	**Used oil with activated clay 25% H_2SO_4 + 25% CH_3COOH 120 °C, 90 min**	0.8544	10.8	66.82	152	0.158	-41
9	**Used oil with activated clay 10% Na_2CO_3 120 °C, 90 min**	0.8726	10.82	68.29	149	0.143	-38
10	**Used oil with activated clay 30% Na_2CO_3 120 °C, 90 min**	0.8734	10.85	52.8	203	0.145	-38
11	**Used oil with activated clay 50% Na_2CO_3 120 °C, 90 min**	0.8732	12.82	69.4	188	0.147	-40
12	**Used oil with activated clay 25%Na_2CO_3 + 25%NaOH 120 °C, 90 min**	0.8736	11.4	64.1	174	0.143	-38
13	**Used oil with activated clay 50% H_2SO_4 + 50%$CaCl_2$ 120 °C, 90 min**	0.8741	11.19	66.01	163	0.147	-38
14	**Used oil with activated clay 50% H_2SO_4 + 10%Na_2CO_3 120 °C, 90 min**	0.8743	12.75	63.88	204	0.146	-40
15	**Used oil with activated clay 50% H_2SO_4 +30% Na_2CO_3 120 °C, 90 min**	0.8742	10.08	52.6	183	0.146	-40
16	**Used oil with activated clay 50% H_2SO_4 + 50% Na_2CO_3 120 °C, 90 min**	0.8744	12.66	68.9	186	0.143	-41

Density: The density of the new base oil called base reference oil without additives of the type (SN90) at 15°C is 0.843 g/cm^3. The density values at the temperature of 15°C of the oil treated with clay (bentonite) of Karaqeva activated with H_2SO_4 acid, with 10%, 30%, 50% are respectively 0.8726 g/cm^3, 0.8725 g/cm^3, 0.8525 g/cm^3. Oil treated with bentonite activated with 25% H_2SO_4 + 25% CH_3COOH has the value of density 0.8544 g/cm^3 and Oil treated with bentonite activated with 10% Na_2CO_3 has the value of density 0.8726 g/cm^3. The lowest value of density 0.8525 g/cm^3 has the oil treated with Karaqeva bentonite activated with H_2SO_4 acid, with 50%.

Viscosity: The kinematic viscosity at 40°C of the new base oil called base reference oil of the type (SN90) is 35 compared to untreated used motor oil which is 68.21. The values of kinematic viscosity at 40°C of the oil treated with Karaqeva bentonite activated with three activation modes, represent an improvement of the viscosity values. The best values are Karaqeva Bentonite treated oil activated with 50% H_2SO_4, 30% Na_2CO_3 and 50% H_2SO_4 + 30% Na_2CO_3 respectively 50.11, 52.8 and 52.6.

Base Oil Reference Base Index without additives (SN90) is 126 compared to untreated Used Motor Oil which is 167 with higher value. The values of the Viscosity Index of oil treated with Karateva Benton inactivated at a temperature of 90°C, for 90 min, with 15,000-20,000 km realized, is 157. Good results are presented by the Viscosity Index of Oil treated with Karaqeva bentonite activated with 50% H_2SO_4 + 30% Na_2CO_3 which has a value of viscosity index 204.

In the following, good results referring to the viscosity index are presented the treated clay oil (bentonite) of Karaqeva activated with 30% Na_2CO_3, which has the value of the viscosity index 203 and the oil treated with activated clay (bentonite) with 50% H_2SO_4, with a value of viscosity index 196.

Pour point: The base reference oil spill point without additives (SN90) is a maximum of -18°C, while the untreated used motor oil spill point is -40°C. The analyzes tested for the discharge point of activated Karaqeva clay-treated oils, respectively with 10% Na_2CO_3, 30% Na_2CO_3 and 25% Na_2CO_3 + 25% NaOH at a temperature of 120°C, for 90 min, are -38, where is also the best result of the pour point value.

Sulfur: The data presented in Table 2 show that the amount of sulfur present in the Basic Reference Oil without additives (SN90) is 0.005% (or 50 ppm). The amount of untreated used motor oil sulfur is 0.186%.

The amount of sulfur used in motor oil treated with activated Karaqeva clay, respectively with 10% Na_2CO_3, 25% Na_2CO_3 + 25% NaOH and 50% H_2SO_4 + 50% Na_2CO_3 and at a temperature of 120°C, for 90 min, are 0.143%. Table 3 shows the tested parameters of New Engine Oil with additives of type (SAE 5W-30), New Basic Oil (called Basic Reference Oil without additives of type (SN 90) and the tested parameters of used motor oil with 15,000-20,000 km performed, before and after treatment with activated clay (bentonite) acid, base and with the combined method (acid and base) of the Karaqeva region, at a temperature of 120°C for 90 min. Comparisons of the parameters of the motor oil used before and after the treatment with clay (benton) of the Karaqeva region activated with acid, base and with the combined method (acid and base), were made with the parameters of the Basic Reference Oil without additives of the type (SN90).

Table 3. Tested parameters of used motor oil before and after treatment with Karateva Benton, activated with acid, base and combined method (acid and base), at a temperature of 120°C for 90 min, with 15,000-20,000 km done

Nr	Sample name	Density at 15 °C (EN ISO 3675) g/cm3	Kinematic viscosity at 100 °C (EN ISO 3104) cst	Kinematic viscosity at 40 °C (EN ISO 3104) cst	Viscosity index (ISO 2909)	Sulphur content % (m/m)	Pour point (EN ISO 3016)
1	**New oil with additives (SAE 5W-30) (reference) reported by producer**	(0.840-0.860) 0.834	(11.3-12.3) 12.1	(7.9-9.5) 72.5	(min 165) min.165	(max 0.5) 0.12	(max.-33) -30
2	**New oil without additives (SN90) (reference) reported by producer**	Raportim 0.843	(min 5.80) 6.2	(min. 31.5) 35	(> 120) 126	(<0.03) ne testim	(-26 máx) -18
3	**Used untreated oil (not filtered)**	0.8673	9.17	50.23	167	0.186	-40
4	**Used oil with not activated clay at 120 °C, 90 min (A.L. 40,000-50,000 km)**	0.8482	6.42	27.28	202	0.121	-32
5	**Used oil with activated clay 10% H_2SO_4 120 °C, 90 min**	0.8479	6.02	28.58	165	0.118	-34
6	**Used oil with activated clay 30% H_2SO_4 120 °C, 90 min**	0.8472	6.4	27.66	196	0.111	-33
7	**Used oil with activated clay 50% H_2SO_4 120 °C, 90 min**	0.851.4	6.09	27.45	179	0.105	-31
8	**Used oil with activated clay 25% H_2SO_4 + 25%**	0.8501	6.42	29.9	176	0.130	-36

	CH_3COOH 120 °C, 90 min						
9	**Used oil with activated clay 10% Na_2CO_3 120 °C, 90 min**	0.8499	6.92	28.32	220	0.124	-30
10	**Used oil with activated clay 30% Na_2CO_3 120 °C, 90 min**	0.8490	6.35	27.72	193	0.115	-31
11	**Used oil with activated clay 50% Na_2CO_3 120 °C, 90 min**	0.8494	6.5	28.69	192	0.126	-33
12	**Used oil with activated clay 25%Na_2CO_3 + 25%NaOH 120 °C, 90 min**	0.8503	6.13	28.42	172	0.132	-35
13	**Used oil with activated clay 50% H_2SO_4 + 50%$CaCl_2$ 120 °C, 90 min**	0.8498	6.05	28.13	170	0.117	-34
14	**Used oil with activated clay 50% H_2SO_4 + 10%Na_2CO_3 120 °C, 90 min**	0.8496	6.53	29.05	190	0.116	-34
15	**Used oil with activated clay 50% H_2SO_4 +30% Na_2CO_3 120 °C, 90 min**	0.8501	6.21	28.02	181	0.126	-34
16	**Used oil with activated clay 50% H_2SO_4 + 50% Na_2CO_3 120 °C, 90 min**	0.8502	6.29	27.88	187	0.132	-35

Density: The density of the new base oil called base reference oil without additives of the type (SN90) at 15°C is 0.843 g/cm^3. The density values at the temperature of 15°C of the oil treated with clay (bentonite) of Karaqeva activated with H_2SO_4 acid, with 10%, 30%, 50% are respectively 0.8726 g/cm^3, 0.8725 g/cm^3, 0.8525 g/cm^3. Karaqeva Benton oil treated with 25% H_2SO_4 + 25% CH_3COOH has a density value of 0.8544 g/cm^3 and Karaqeva Benton oil treated with 10% Na_2CO_3 has a density value of 0.8726 g/cm^3. The lowest value of density 0.8525 g/cm3 has the oil treated with Karaqeva bentonite activated with H_2SO_4 acid, with 50%.

Viscosity: The kinematic viscosity at 40°C of the new base oil called baseless reference oil of type (SN90) is 35 compared to untreated used motor oil which is 50.23. The kinematic viscosity values at 40°C of the Karaqeva bentonite treated oil activated in three ways, show an improvement of the viscosity values. The best values are Karaqeva Bentonite treated oil activated with 50% H_2SO_4, 30% Na_2CO_3 and 50% H_2SO_4 + 30% Na_2CO_3 respectively 50.11, 52.8 and 52.6. Base Oil Reference Base Index without additives (SN90) is 126 compared to untreated Used Motor Oil which is 167 with higher value. The values of the Viscosity Index of Oil treated with Benton of Karaqeva inactivated at a temperature of 120°C, for 90 min, with 40,000-50,000 km realized, is 157. Good results are presented by the Viscosity Index of Oil treated with Karaqeva bentonite activated with 50% H_2SO_4 + 10% Na_2CO_3, which has a value of viscosity index 204. In the following, good results referring to the viscosity index are presented the treated clay oil (bentonite) of Karaqeva activated with 30% Na_2CO_3, which has the value of the viscosity index 203 and the oil treated with activated clay (bentonite) with 50% H_2SO_4, with a value of viscosity index 196.

Pour point: The base reference oil spill point without additives (SN90) is a maximum of -18°C, while the untreated used motor oil spill point is -40°C. The analyzes tested for the discharge point of activated Karaqeva clay-treated oils, respectively with 10% Na_2CO_3, 30% Na_2CO_3, 30% H_2SO_4 and 25% Na_2CO_3 + 25% NaOH at 120 °C, for 90 min, are -38, where is the best result of the pour point value.

Sulfur: The data presented in Table 3 show that the amount of sulfur present in the Basic Reference Oil without additives (SN90) is 0.005% (or 50 ppm). The amount of untreated used motor oil sulfur is 0.186%.

The amount of sulfur used in motor oil treated with activated Karaqeva clay, respectively with 10% Na_2CO_3, 25% Na_2CO_3 + 25% NaOH and 50% H_2SO_4 + 50% Na_2CO_3 and at a temperature of 120°C, for 90 min, are 0.143%. While the minimum value of sulfur content is 0.112% in the case of clays treated with 50% H_2SO_4. By comparing the parameters of used oils with 15,000-20,000 km realized, treated with Karaqeva bentonite activated with acid, base and with the combined method (acid and base), at a temperature of 120°C for 90 min, you can we say that Karaqeva bentonite activated acids and with the combined method (in the entirety of the values of the measurements performed) is more promising for the cleaning and recycling of used motor oils.

After treatment (cleaning and recycling) of used oil, we can say that it can be used as a product to be reused again as a base oil or as an additional fuel for transport vehicles or various industrial machinery.

Conclusions

The treatment of used motor oils with activated bentonites from the Karaqeva region results in improved quality parameters. Activated bentonites from the Karaqeva region can be used for the recycling of used motor oils and as an environmentally friendly alternative. The technique applied for the treatment of oils used by the treatment with Karaqeva clays activated with three different methods (acid, base and with the combined acid-base method), is an efficient method, which does not denature (degrade), but protects the structure of the recycled oil undamaged. This technique removes contaminants, such as solid particles (metallic and non-metallic), soot, odors, heavy metals, reduces the amount of sulfur. This technique preserves the quality of additives, density, viscosity parameters, viscosity index, flow point, etc. The best density values were achieved for the used oil with 15,000-20,000 km treated with acid-activated bentonite and the combined method (acid and base). The value of viscosity and viscosity index shows that a chemical treatment of used oils has been performed, without degrading its additives, because these affect the longevity of the use of lubricating oil.

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