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NEW METHODS OF BENTONITE ACTIVATION AND THEIR EFFICIENCY IN USED MOTOR OILS REGENERATION

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ABSTRACT:

NEW TECHNIQUES OF BENTONITE ACTIVATION AND THEIR EFFICIENCY IN USED MOTOR OIL (UMO) REGENERATION HAVE BEEN ASSESSED. FOUR METHODS OF RECYCLING USED LUBRICATING OIL (HAVING RUN ABOUT 15 000-20 000 KM) HAVE BEEN EMPLOYED, SUCH AS HEAT/BENTONITE TREATMENT, Na_2CO_3 /BENTONITE COMBINED WITH MICROWAVE TREATMENT, ACID/BENTONITE COMBINED WITH MICROWAVE TREATMENT, AND $NaOH$ /BENTONITE COMBINED WITH MICROWAVE TREATMENT. VARIOUS TESTS HAVE BEEN CARRIED OUT ON THE RECYCLED USED LUBRICATING OIL, INCLUDING THOSE FOR DENSITY, KINEMATIC VISCOSITY, VISCOSITY INDEX, POUR POINT, COLOR, AND SULFUR CONTENT. THE RESULTS SHOWED THAT ACID-ACTIVATED BENTONITE FOLLOWED BY MICROWAVE TREATMENT WAS MORE EFFICIENT FOR UMO REGENERATION COMPARED TO THE OTHER METHODS.

KEY WORDS: BENTONITE ACTIVATION, MICROWAVE TREATMENT, PHYSIC-CHEMICAL PARAMETERS, USED LUBRICATING OIL REGENERATION

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INTRODUCTION

Lubricating oils are complex mixtures of hydrocarbon molecules used to reduce friction between two moving surfaces in contact with each other. The presence of heavy metals in used lubricating oils, as well as the pollution caused by their disposal, is a major source of concern [1].

Heavy metals such as copper, zinc, chromium, lead, nickel, cadmium, mercury, and arsenic, in their soluble form, can be converted very easily into water and daily consumption products, which may cause serious problems in human health [2]. Indeed, many studies have been made about recycling the used motor oil (UMO), including filtration, ion exchange, reverse osmosis, precipitation, and the adsorption process [3-6].

The adsorption process is one of the best methods for cleaning used lubricating oil/UMO since it is possible to remove heavy metals with high efficiency and low cost.

This research paper focuses on producing high-quality lubricating oil from used motor oil that has run between 15 000 and 20 000 km using an adsorption process involving activated bentonites.

Bentonite is a clay mineral mostly composed of montmorillonite [7]. Due to its unique adsorptive properties, attributed to its structure, bentonite has been known since ancient times for cleaning cotton or for the production of soaps. They were used in small amounts for medical purposes until the nineteenth century [8]. However, the use of bentonite increased rapidly during the twentieth century [9]. Since natural clays have poorer regenerating activity, acid and alkaline activation methods are employed to improve their properties, such as porosity or specific surface area [10, 11].

Thermal treatment of bentonite affects the amount of "free" or adsorbed water, which depends on the temperature and time of treatment [12, 13]. Thermal treatment of clays (bentonite), according to Zuzana *et al.*, results in phase transformation with a decrease in specific surface area and total pore volume [14, 15].

Nevertheless, Sarikaya *et al.*, showed a "zig-zag" variation of the specific surface area and total pore volume up to 700°C and a rapid decrease above this temperature [16]. The main advantage of using microwaves is to increase the rate and selectivity of certain types of reactions [14]. This is due to the direct interaction of the microwave field with the material being irradiated and cannot be achieved during ordinary heating [17-19].

Acid activation of bentonite is a process during which bentonite is partially dissolved using acidic solutions in order to remove Ca^{2+} ions, thereby increasing the overall negative charge. This partial dissolution causes the specific surface area, porosity, and acidity of the surface to increase [20, 21]. However, the solubility of bentonite can vary based on its composition. Because of the solubility of their oxides, magnesium and iron are more soluble in bentonite with high magnesium and iron content [22]. The most commonly used procedure and the most inexpensive method of recycling used lubricating oil is the acid/clay method [23].

For better results, the acid or base treatment is followed by heat treatment with microwaves. Due to their properties and easy access, bentonites have been used to remove heavy metals from used lubricant oils [24, 25].

In our research work, a combination of alkaline-microwave curing, acid-microwave curing, and the calcination method for bentonite activation is employed, and its effect on used motor oil (UMO, having run about 15 000–20 000 km) regeneration is assessed.

MATERIALS AND METHODS

Materials:

The following materials were used in the recovery of the used lubricating oil: H_2SO_4 (98%) with density $d = 1.835 \text{ g/cm}^3$, NaOH anhydrous pellets with $\geq 97.0\%$ purity, Barium chloride ($BaCl_2$), and Na_2CO_3 powder ($\geq 99.5\%$, ACS reagent), supplied from Merck. As a raw material, bentonite from the Goshica region of Kosovo is employed. The UMO used in this research had been running for approximately 15 000–20 000 km and was obtained from a local petrol station. The reference oils are a new oil of type SAE 5W-30 with additives and a new oil of type SN 90 without additives.

The apparatus and equipment used for the synthesis include the following: a 3-nozzle balloon with a round bottom (2000 ml), a thermometer, a heating magnetic stirrer, a reflux condenser, a measuring cylinder, a beaker, a vacuum pump, an Erlenmeyer flask, a filtering flask, a Buchner funnel, a porcelain pot, microwave, and a muffle furnace.

Lubricating oil quality is assessed using European standards, which include density, viscosity, viscosity index, pour point, sulfur content, and color.

Methods:

Heat activation of bentonite

The required amount of the ground and sieved Goshica's clay is placed in a porcelain pot and carried out for 1 hour at 700°C in a muffle furnace. The calcined bentonite is cooled down at room temperature for 24 hours.

Heat activation of bentonites with Na_2CO_3

The Goshica's bentonite is mixed with powdered Na_2CO_3 in a 4:1 (w/w) ratio and homogenized in a porcelain mortar. Subsequently, the mixture is placed in a muffle furnace for 1 hour at 700°C and then cooled at room temperature for 24 hours.

Activation of bentonite with H_2SO_4 (10%) and microwave treatment

Goshica's bentonite and 10% sulfuric acid were mixed in a 2-liter flask with a round bottom in a 9:1 (w/w) ratio. The flask was filled to about 1/3 with double-distilled water. For the activation process, the flask was placed on a heated magnetic stirrer at 150 rpm and $99\text{--}103^\circ\text{C}$ for 2 hours. To clean the clay from the acid, it was washed with distilled water, heated at 70°C , and controlled with a $BaCl_2$ indicator until no more sulfate ions appeared. The remaining amount of the activated clay was dried at 105°C , grounded, and put in the microwave for 30 minutes at 600W.

Activation of bentonite with NaOH and microwave treatment

Goshica's bentonite was mixed with pearl anhydrous sodium hydroxide in a 4:1 clay/NaOH ratio until a fine homogeneous mixture is obtained. Consequently, the mixture was microwaved for 30 minutes at 600W.

Regeneration of used motor oil (UMO)

The same manner of oil recycling is employed with each of the activated clays as follows: 100 ml of UMO are added to a round-bottomed flask and stirred at 200 rpm until the temperature reaches 90°C . In the instant moment, 10g of activated bentonite was added. The regeneration process proceeded by stirring thermally at 200 rpm for 60 min. The solution was left at room temperature for 24 hours and then filtered in a filtration flask.

For each recycled oil, the following physicochemical parameters were determined: Density at 15⁰C according to (EN ISO 3675) g/cm³, Kinematic Viscosity at 40⁰C and 100⁰C (EN ISO 3104), Viscosity Index (ISO 2909), Pour Point (ISO 3016), Sulfur in % (m/m), and Color (EN ISO 2049).

RESULTS AND DISCUSSION

Used motor oil (UMO), having run about 15 000 to 20 000 km, is tested to determine its degradation level. Various tests, including oil density at 15⁰C, pour point, sulfur content, kinematic viscosity at 40⁰C and 100⁰C, viscosity index, and color are performed. Tables 1, 2, and 3 summarize the results from the relevant analyses performed to characterize the experimental samples.

Table 1. Used motor oil (UMO) characterization results and its comparison with reference new oils (new oil with and without additives).

No.	Sample name	Density at 15 ⁰ C (EN ISO 3016) g/cm ³	Pour point (EN ISO 3016) °C	Sulfur % (m/m)	Color	Kinematic Viscosity at 40 ⁰ C (EN ISO 3104) cst	Kinematic Viscosity at 100 ⁰ C (EN ISO 3104) cst	Viscosity index (ISO 2909)
1	<i>New oil without additives (SN 90, reference)</i>	Reported 0.843	(-26 max) -18	(<0.03) In test	>9	(min.31.5) 35	(min.5.80) 6.2	(>120) 126
2	<i>New oil with additives (SAE 5W-30, reference)</i>	(0.840-0.860) 0.834	(max.-33) -30	(max. 0.5) 0.12	(L 0.5) colorless	(79-95) 72.5	(11.3-12.3) 12.1	(min.165) min.165
3	<i>Untreated UMO</i>	0.8798	-24	0.542	>8	23.65	6.18	232

From the aforementioned analysis, it is evident that a major property alteration is done, compared to the fresh oil, during its application period. The increased density and viscosity index indicates possible oxidation and polymerization of products suspended and dissolved in the spent oil [26]. Furthermore, the increased sulfur content indicates the oxidation of sulfur components in the fuel used in internal combustion engines [27].

In this research work, recycling of the used motor oil was conducted based on the adsorption method by activated Goshica's bentonite. To activate the bentonite, a combination of alkaline-microwave curing, acid-microwave curing, and the calcination method was employed.

Tables 2 and 3 present the data from the tested treated and untreated UMOs, as well as the data from the reference oils (new motor oil with additives, type SAE 5W-30, and new motor oil without additives, type SN 90).

Table 2. Summary of the analysis results of UMO (15000 - 20000 km) treated with calcined and activated bentonites at 90°C for 60 minutes, and its comparison with untreated UMO and reference new oils (new oil with and without additives).

No.	Sample name	Density at 15°C (EN ISO 3016) g/cm ³	Pour point (EN ISO 3016) °C	Sulfur % (m/m)	Color	Kinematic Viscosity at 40°C (EN ISO 3104) cst	Kinematic Viscosity at 100°C (EN ISO 3104) cst	Viscosity index (ISO 2909)
1	<i>New oil without additives (SN 90, reference)</i>	Reported 0.843	(-26 max) -18	(<0.03) In test	>9	(min.31.5) 35	(min.5.80) 6.2	(>120) 126
2	<i>New oil with additives (SAE 5W-30, reference)</i>	(0.840-0.860) 0.834	(max.-33) -30	(max. 0.5) 0.12	(L 0.5) colorless	(79-95) 72.5	(11.3-12.3) 12.1	(min.165) min.165
3	<i>Untreated UMO</i>	0.8798	-24	0.542	>8	23.65	6.18	232
4	<i>UMO treated with calcined bentonite (UMO+CB)</i>	0.8797	-23	0.534	>8	20.93	4.97	175
5	<i>UMO treated with calcined bentonite and activated with Na₂CO₃ (UMO+CB-Na₂CO₃)</i>	0.8797	-25	0.535	>8	47	5.75	200

Table 3. Summary of the analysis results of UMO (15000 - 20000 km) treated with activated bentonites at 90°C for 60 minutes, and its comparison with untreated UMO and reference new oils (new oil with and without additives).

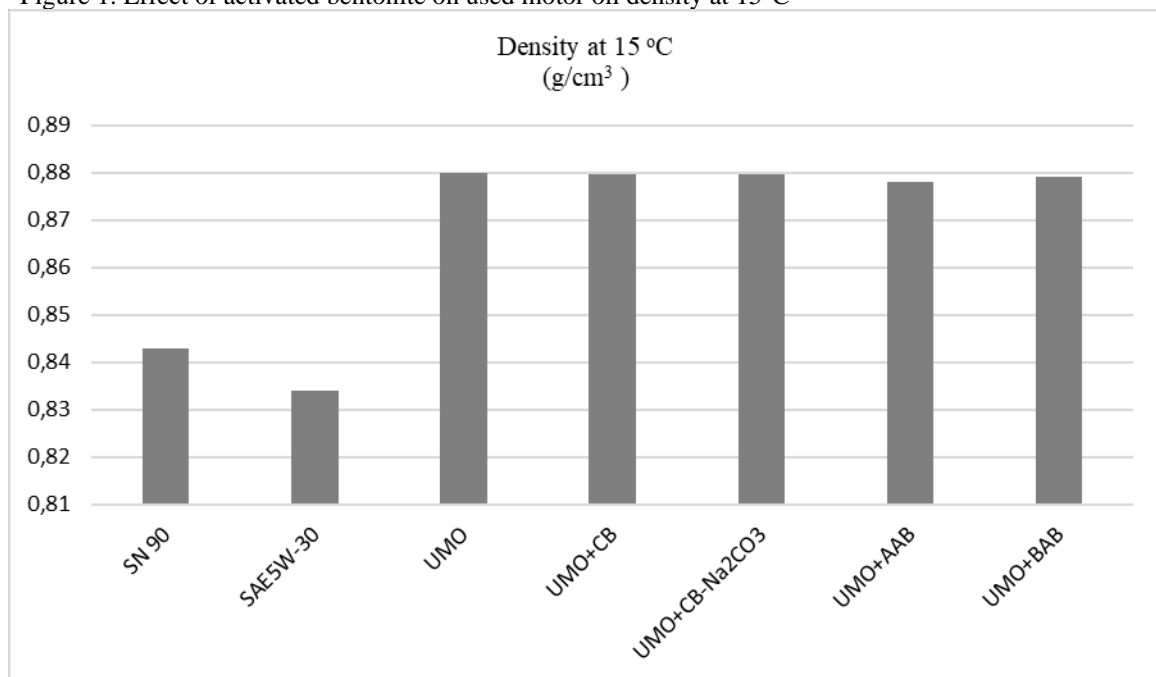
No.	Sample name	Density at 15°C (EN ISO 3016) g/cm ³	Pour point (EN ISO 3016) °C	Sulfur % (m/m)	Color	Kinematic Viscosity at 40°C (EN ISO 3104) cst	Kinematic Viscosity at 100°C (EN ISO 3104) cst	Viscosity index (ISO 2909)
1	<i>New oil without additives (SN 90, reference)</i>	Reported 0.843	(-26 max) -18	(<0.03) In test	>9	(min.31.5) 35	(min.5.80) 6.2	(>120) 126
2	<i>New oil with additives (SAE 5W-30, reference)</i>	(0.840-0.860) 0.834	(max.-33) -30	(max. 0.5) 0.12	(L 0.5) colorless	(79-95) 72.5	(11.3-12.3) 12.1	(min.165) min.165
3	<i>Untreated UMO</i>	0.8798	-24	0.542	>8	23.65	6.18	232
4	<i>UMO treated with 10% H₂SO₄ activated bentonite (UMO+AAB)</i>	0.8779	-25	0.530	>8	49	8.08	137
5	<i>UMO treated with NaOH</i>	0.8791	-27	0.540	>8	35.33	6.61	145

activated bentonite (UMO+BAB)								
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Density

The density at 15°C of the untreated used motor oil is higher than the fresh ones, based on the above results. It varies from 0.8340 g/cm³ for the new oil with additives and 0.8430 g/cm³ for the new oil without additives to 0.8798 g/cm³ for the untreated UMO. According to Tables 2 and 3, there hasn't been a significant change in the density values for UMO treated with the calcined and acid/base-activated bentonites. The lowest density value is reported for the refined oil obtained by activating bentonite with H₂SO₄ (10%) and microwave treatment, which has a density of 0.8779 g/cm³, higher than that of the reference oils. This is also evident in Figure 1.

Figure 1. Effect of activated bentonite on used motor oil density at 15°C



Pour point

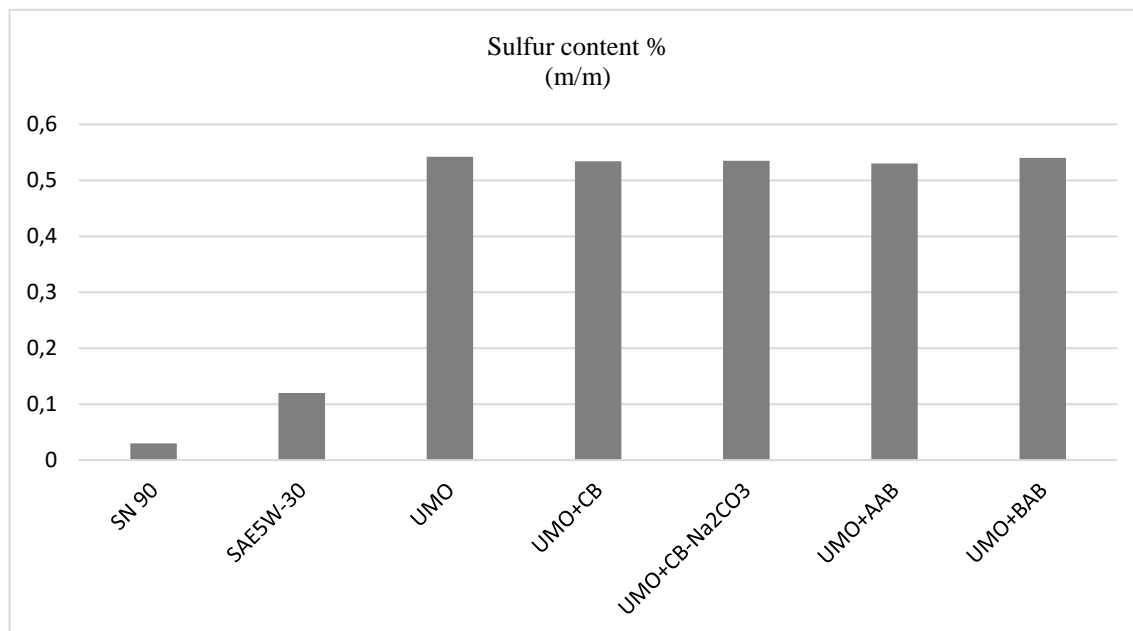
The "pour point" refers to the lowest temperature at which oil will just flow when cooled. Depending on how the lubricating oil is handled, the pour point can be lowered or raised. The results, as given in Tables 2 and 3, demonstrate that the recycled oil retained a good pour point and can be reused.

Sulfur content

The data presented in Table 1 show that the amount of sulfur in the new motor oil without additives (SN 90) is <0.03 % and in the reference oil with additives (SAE 5W-30) is 0.12%, while the amount of sulfur in UMO is 0.542%. The increase in the amount of sulfur in used motor oil is due to the presence of wear caused between moving parts. Figure 2 shows that the treatment of UMO with calcined and activated Goshica's clay at 90°C for 60 min definitely caused the sulfur content to decrease. The lower values are reached for recycled UMO with calcined bentonite at 700°C (0.534%) and recycled UMO with 10% H₂SO₄

activated bentonite followed by microwave treatment (0.530%), as presented in Tables 2 and 3.

Figure 2. Effect of activated bentonite on used motor oil sulfur content.



Kinematic Viscosity at 40°C

Viscosity can increase due to oxidation or contamination. Additionally, viscosity decreases can be caused by dilution with light fuel or contamination in the form of sludge [25]. The viscosity of lubricating oils is extremely sensitive to the operating temperature. The new oil's kinematic viscosity at 40°C with and without additives is 72.5 and 35, respectively. Meanwhile, the EN ISO 3104 method shows a decrease in the kinematic viscosity at 40°C for the untreated UMO in 23.65, and those of the recycled UMO are as follows: 20.93, 47.00, 49.00, and 35.33 with the calcined bentonite, calcined and Na₂CO₃ activated bentonite, H₂SO₄ activated bentonite, and NaOH activated bentonite, respectively.

This shows that at 40°C, the refined UMO using calcined and Na₂CO₃ activated bentonite and acid/base activated bentonite recovered the viscosity specification range.

Kinematic Viscosity at 100°C

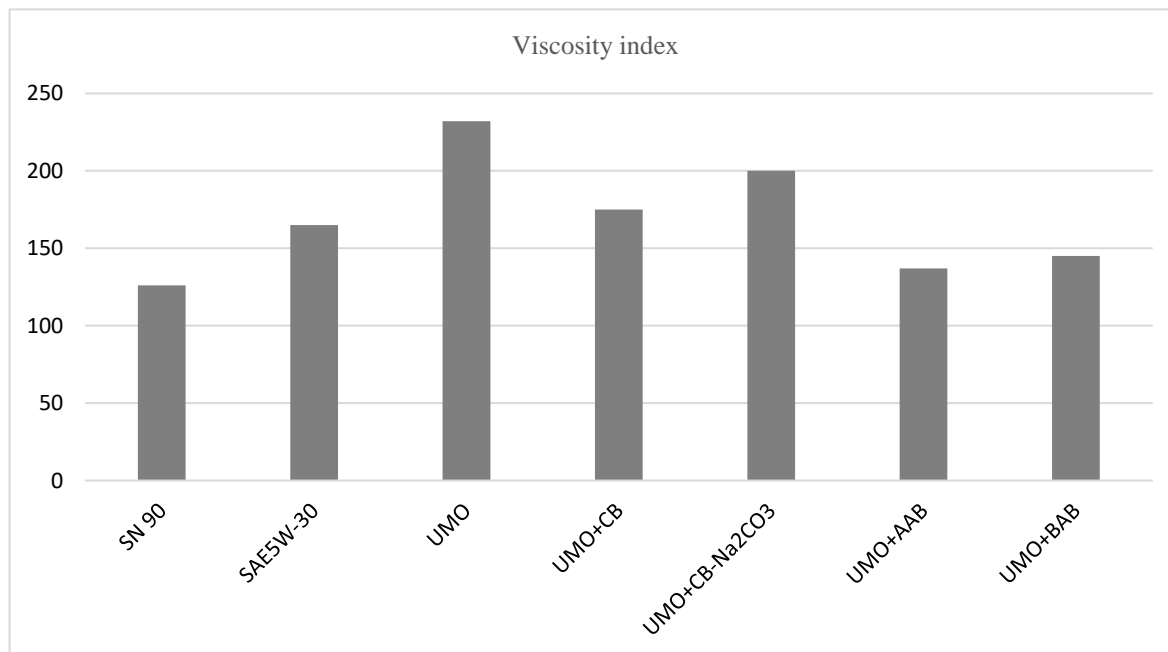
The kinematic viscosity at 100°C test results demonstrate that the viscosity of the lube oil decreases as temperature increases. Table 1 shows that the usage of the lubricating oil has not deeply affected its kinematic viscosity at 100°C. Its value of 6.18 appears to be like that of the new oil without additives (SN 90), which has a value of 6.2. Furthermore, an increase in kinematic viscosity at 100°C is shown for recycled UMO with H₂SO₄ (10%) activated bentonite and NaOH activated bentonite of 8.08 and 6.61, respectively.

Viscosity index

The viscosity index of untreated UMO after its application period was 232, higher than that of fresh oil without additives (126) and fresh oil with additives (a minimum of 165), as shown in Table 1. This might be due to oxidation or contamination with insoluble matter.

Figure 3 shows that there is an improvement in the viscosity of the UMO treated with activated bentonite with the four activation methods as compared to the viscosity of the new reference oil with and without additives. The best values are UMO treated with calcined bentonite and H₂SO₄ (10%) activated bentonite (175 and 137, respectively).

Figure 3. Effect of activated bentonite on used motor oil viscosity index



CONCLUSION

The results of this study demonstrate that activated and calcined bentonite samples can be utilized to recycle spent lubricating oil. An improvement in sulfur content and viscosity index is shown for all UMO samples treated with calcined and activated bentonites. The lowest density value of 0.8779 g/cm³ at 15°C, is reported for the refined oil obtained by activating bentonite with H₂SO₄ (10%) and microwave treatment. Regarding the pour point, the recycled oil retained a good pour point and can be reused. Treating UMO with calcined and activated Goshica's clay at 90°C for 60 min caused the sulfur content to decrease. The best kinematic viscosity values at 40°C were obtained by UMO treated with NaOH activated bentonite (35.33), similar to that of the fresh oil without additives (35), whereas the best viscosity index values were obtained for the UMO treated with calcined bentonite and H₂SO₄ (10%) activated bentonite (175 and 137, respectively). The physicochemical tests conducted for the untreated and recycled UMO, having run about 15 000–20 000 km, revealed that the best results were generally obtained for UMO treated with H₂SO₄ (10%) activated bentonite, followed by UMO treated with calcined bentonite.

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