



Waste Engine Oil Remediation Using Low Cost Natural Clay Absorbent Material

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ABSTRACT

The aim of the current research is to examine the potential of locally available natural clay mineral to act as an effective absorbent for waste engine oil treatment. The clay mineral was collected from the southern region of Saudi Arabia and was used without any kind of surface modification. The clay mineral was characterized by X-ray diffraction (XRD), X-ray fluorescence (XPS) and Brunner-Emmitt-Teller (BET) surface area analysis. XRD data showed the crystalline structure of the clay while chemical composition of the clay was determined by XRF analysis in which the main constituents were SiO₂ (51.77%), Al₂O₃ and Fe₂O₃ with 29.17% and 13.22% respectively. Other minor compounds were detected. BET surface area was found to be around 65 m²/g. Clay adsorbent amount (ranging from 5 – 20g) was added to 100 ml waste engine oil sample under different temperature conditions. Sample analysis obtained by UV-spectrophotometer indicates that the oil sample S10 which was conducted at 450°C with 5g clay showed the best correlation (the peaks and wavelength number) very close the virgin oil. Hence, suggesting these to be the optimum operating conditions.

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1. INTRODUCTION

Lubricating oils are hydrocarbon-based viscous liquids that are used for lubricating moving parts of engines and machines for friction reduction, heat dissipation, and power transmission amongst many other applications. The use of lubricants has significantly increased worldwide over the last century. Lubricants produced by crude oil refining industry represent about 1.2% of the annual consumption of petroleum. This value corresponds to over 40 million tonnes of base oil at the global level [1]. These lubricating oils consist mainly of complex mixtures of isoalkanes having slightly longer branches, monocycloalkanes and monoaromatics which have several short branches on the ring [2]. The composition of lubricating oil varies significantly depending on the types of additives and their percentages as well as other end user applications [3].

During applications, lubricating oils tend to degrade leading to deterioration of various essential features

such as rheological and film forming properties. Rheological properties are considered to be among the most important degradable properties [4]. The level of this degradation is dependent on the environment and operating conditions where the oil is used. However, during the degradation process, a point is reached where the lubricant oil would no longer be able to perform its functions. Despite this degradation, most of the base oil part in the waste oil is not exhausted [5] and may be recovered by various means.

Furthermore, the solvent to oil ratio was reported to have a more pronounced effect on the quality of oil produced after treatment [6]. The various solvents reported in literature include various hydrocarbon solvents, mainly: hexane, methyl ethyl ketone (MEK), toluene, butanol and acetone. MEK is reported to have exhibited the best performance with the lowest percent of oil losses followed by propanol and butanol [7] whereas acetone has shown the least performance in comparison to the other solvents.

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The use of sulphuric acid has practically been diminished in the oil refining processes and has been replaced by other preferential solvents. However, its efficiency (in the form of oleums) is still recognized in the manufacture of white oil [8]. The use of acid tends to be replaced by catalytic hydrotreatment which uses platinum catalyst under high pressure and low temperatures but with relatively long residence times.

Clays are known to be a permeable media [9], that usually possess high surface area and active sites making the clay as potential adsorbent. Different types of clays have proven to have the ability to act as bleaching agent, particularly bentonite [10, 11]. Natural unmodified clays tend to attract oil molecules to its solid surface by weak Van der Waal forces postulating that the adsorption mechanism is physisorption in nature. This type of adsorption is promoted by increasing pressure and temperature. In most instances, clays are treated to enhance their adsorption capabilities and surface activity by various techniques such as thermal treatment, acid treatment and alkaline treatment depending on the nature of the adsorbate. Acidic treatment of clays includes sulphuric acid, hydrochloric acid and nitric acid. However, during concentrated sulphuric acid treatment of clays, asphaltenic compounds are removed, leaving highly toxic acid compound on the spent adsorbent (known as acidic sludge). This presents significant challenges for adsorbent regeneration in addition to significant risk to the environment. The use of hydrochloric acid with thermal treatment to 650 °C has also been reported [12] which concluded that significant increase in the surface area, morphology and mesopores was achieved by activation. Thus, acid treatment improving the potential adsorption capabilities of the treated clay [13]. The adsorption mechanism of waste oil over clay adsorbent is based on its ability to selectively extract resinous and unsaturated polycyclic materials and also organic residues from waste oils. Mohammed *et al.* [14] reported improvements in fluidity, particularly viscosity using acid/clay treatment, which was possibly attributed to the conversion of contaminants by the acid and removal by the clay from the lube oil. Similar results were reported by Udonne [15] who studied the used oil treatment process using acid/clay methods.

Aziz *et al.* [21] reported the use of acid-free clays, where waste oils were initially treated with natural polymers to remove carbonic materials. The oil was then subjected to vacuum distillation and clay treatment in the appropriate amount to obtain the desired product coloration [12, 16]. Besides the high costs due to the required quantity of clay, recovered oils obtained by this process still have relatively high metal percentages [17]. The use of activated carbon has also been reported in literature and the results showed highly effectiveness

waste engine oil treatment by PAH removal [18].

The objective of the current investigation was to explore the potential use of locally available natural (untreated) clay mineral from the southern of KSA to act as low-cost adsorbent on waste engine oil at various operating conditions. The quality of treated oil will then be assessed using UV spectrophotometer and the results will be compared with similar findings from other clay treatments. The clay mineral in the current research is used without any kind of surface treatment (modification) and has proven to be an effective adsorbent hence; the operational costs are expected to be lower.

In this paper, materials used and methods applied in this research are highlighted in section 2. Characterisation techniques applied on the clay, namely BET, XRD and XRF are explained in section 3. Section 4 presents the results and discussions obtained based on the studied experimental conditions. The final section of the paper presents the conclusions and findings of this paper.

2. MATERIALS AND METHODS

In this work, fresh SAE 20W-50 engine oil was used for a total of 3500km before being changed and collected for treatment and assessment. Three litres of the waste oil was filtered and left to sediment for 48 hours to remove all settleable particles prior to other treatments.

Natural red clay was collected from Aseer region, south of KSA. The clay was washed with distilled water, dried in the oven at 60°C for 24 hours then crushed and sieved with 200µm mesh. Samples of the clay were subjected to different characterization techniques; a) BET test to measure the specific surface area, total pore volume and average pore diameter. b) X-Ray Diffraction (XRD) analysis and c) XRF compositional analysis.

Different quantities of the clay (5, 10 and 20g) were added to 100ml of the waste engine oil. Each set of flasks containing oil-clay mixture was subjected to specific temperature; 100, 250, 350 and 450°C with constant mixing speed of 800 rpm for 3hours [19]. The mixtures were left to naturally cool to room temperature and to allow the clay to settle. Simple decantation was used to obtain most of the oil before the use of filtration.

Samples from the treated oil (4ml) were then diluted using kerosene (1liter) before being tested using a UV spectrophotometer. A simplified experimental process flow diagram is presented in Figure 1.

Kerosene was chosen because it is more amenable to mass spectrometric analysis as reported in [20]. Table 1 presents the numerations of tested samples at given temperatures and clays weights applied. The same

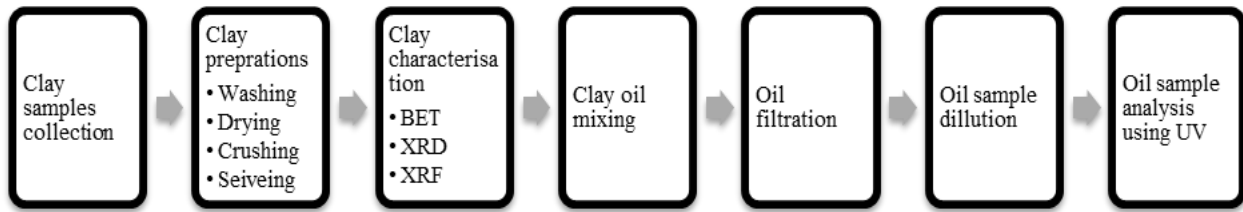


Figure 1. Experimental flow diagram

enumeration is maintained throughout the presented results. Clay treatments were carried out at four different temperatures and in each case; three different quantities of the clay were added. Each treatment condition is presented as S1, S2, etc. as shown in Table 1.

According to the Beer-Lambert relationship, the change in light intensity due to absorption is determined by the number of absorbing types. The Beer-Lambert relationship states that there is a logarithmic dependence between the transmission of light through a substance and the product of the absorption coefficient of the substance, α , and the distance the light travels through the material, i.e. the path length '1'

$$A = -\log T = \log \frac{I_0}{I} = a \cdot c \cdot b \quad (1)$$

where A is the absorbance, T is the transmittance defined as I/I_0 , I is the incident light intensity and I_0 is the transmitted light intensity, c is the concentration of the absorbing types (gm/l), b is the path length traversed by the light, a is the absorptivity of the absorbing types. The chemicals dissolved in oil can be identified by sending particular wavelengths into a compound and analyzing the intensity of the incoming light [21]. Here the spectral response, UV-visible spectroscopy is used to locate the appropriate optical technology. As waste engine oil constitutes thousands of hydrocarbons, there is a possibility that it can absorb the light at specific wavelengths in the electromagnetic spectrum. The sample absorbs energy, i.e. photons from the radiant light [19].

TABLE 1. Numeration of tested samples vs. operating conditions

Temp. ^o C / Clay Wt. added	5g	10g	20g
250°C	S1	S2	S3
350°C	S4	S5	S6
400°C	S7	S8	S9
450°C	S10	S11	S12

3. RESULTS AND DISCUSSION

3. 1. Clay Characterization

The characterization of the clays was performed using XRD, XRF and BET. The BET results provide information on surface areas and pore parameters analysis. As for the surface area and pore size and pore volume determination, the Brunauer-Emmett-Teller (BET) method was applied to the data obtained from the surface analyser. The clay sample was characterized by BET analysis using N₂ sorption to determine its surface area, pore size, and pore volume and the results are tabulated in Table 2.

From Table 2, the active surface area for the tested clay has shown competitive values in comparison to other clays such as bentonites that have values range from 40 to 130 m²/g [10] and that of activated bleaching earth [13]. This provides reasonable justification for this clay to be used as a natural absorbent material in the current research.

The clay sample was subjected to XRD testing to reveal the structural characteristics as shown in Figure 2 using a Shimadzu X-600 model. The XRD pattern was recorded in the 2 θ of 5-50° in a powder diffractometer with CuK α 1 radiation operated at 40 kV. The graph shows many clear, sharp peaks of different intensities, indicating the good crystalline nature of the clay sample. The XRD peaks were matched with the XRD standard database. The highest intensity peak found at 3.22 Å d-spacing corresponds to the feldspar, forming as primary minerals in clay sample studied. Another peak found at 2.04 Å d-spacing was attributed to the presence of Illite in clay. Other distinctive peaks found at 2.147, 2.497 and 2.958 Å d-spacing indicate the presence of quartz, calcite, and gypsum traces in the sample.

TABLE 2. BET analysis results for used clay

BET (m ² /g)	Total Pore Volume (cm ³ /g)	Average Pore Diameter (nm)
63.153	0.0993	6.288

XRF analysis was performed on the ZSX Primus II (Rigaku, USA) to determine the metals content in clay samples as metal oxides. Clay samples were heated to 900 °C for 8 h to convert all metals to metal oxides to avoid interaction with the platinum crucible. XRF sample was then prepared according to fusion procedure by mixing 0.3 g of the obtained metal oxides from the sample with 6 g of lithium borates flux (SPEX, USA). The mixture was then melted at 1100 °C using an automatic fusion system (Katanax, Canada) to make a glass disk in a platinum fusion disk mould. The elemental composition of the sample was finally analysed using an X-ray fluorescence spectrometer (Rigaku, USA). Table 3 summarises the XRF analysis for clay sample. It was found that of the clay sample contained the highest amount of SiO₂ (51.77%) while the other major components were Al₂O₃ and Fe₂O₃ with 29.17 and 13.22%, respectively. CaO, TiO₂ and MgO were also present in amounts of 1.53, 1.80 and 1.14%, respectively. Few other materials such as ZrO₂, V₂O₅ and K₂O were also present in trace amounts. The higher percentage of Fe₂O₃ is also demonstrated by the reddish colour of the clay.

3. 2. Waste Engine Oil Treatment

In this section, a comparative analysis between the UV spectrometer absorbance is performed for the virgin, untreated waste engine oil (S0) and treated (S1-S12) samples. The absorbance values for the oil vary significantly over a wide range of wavelengths. This is due to the presence of very complex mixtures of paraffinic, naphthenic and aromatic hydrocarbon molecules [14]. As kerosene is introduced to dilute

samples, the viscosity of oil samples becomes less. Consequently, the sample oil becomes optically more transparent [20].

Therefore, incident light passes through the sample oil and the absorbance peaks are noted as an indicator to verify the quality of different engine oil samples tested. The absorbance is presented on Y-axis in the range 0-3 and the wavelength is plotted on the X-axis in the range 200-700 nm.

Figure 3 shows the UV spectrophotometer absorbance for a diluted sample of fresh virgin engine oil over a range between 200 to 700 nm wavelengths. Virgin oil has shown two peaks; the first peak was detected at a 248 nm wavelength with an absorbance value of 2.545, the second peak at 314 nm with an absorbance of 2.160. On the other hand, the used untreated oil (S0) has given an absorbance of 2.569 at 250nm and a second peak at 312 nm with an absorbance value of 2.196.

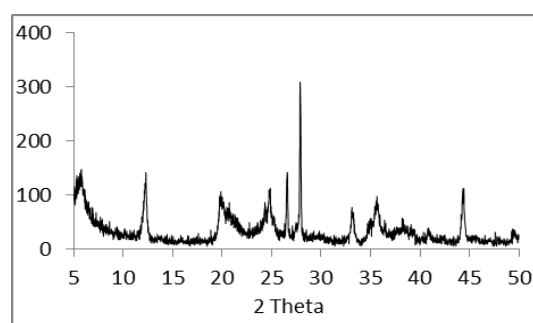


Figure 2. XRD analysis for the used red clay

TABLE 3. Clay composition using XRF analysis.

Al ₂ O ₃	CaO	Fe ₂ O ₃	K ₂ O	MgO	MnO	NiO	P ₂ O ₅	SiO ₂	SO ₃	SrO	TiO ₂	V ₂ O ₅	ZrO ₂
29.17	1.53	13.22	0.16	1.14	0.05	0.02	0.15	51.77	0.11	0.06	1.80	0.09	0.09

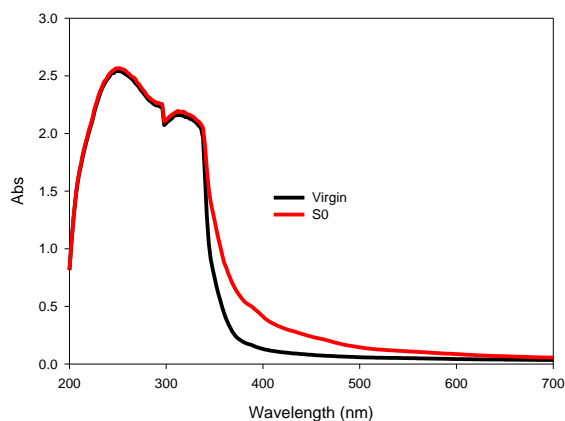


Figure 3. Absorbance of virgin vs. untreated oil (S0)

By comparing the values of these peaks, it is clear that S0 has a higher absorbance than that obtained for the virgin oil. This can be justified by the oxidation of various hydrocarbons of the oil after usage which results in darker colouring of the oil. It is also noted that the overall absorbance patterns for both samples are very similar. Moreover, below 330 nm both oils samples showed an identical pattern of absorbance. However, the untreated waste (S0) continued to maintain higher absorbance than that obtained by the virgin oil. Since both oil samples showed similar trend of absorbance throughout, only virgin data will be used as a comparative baseline with other tested samples.

As for the analysis of samples S1, S2 and S3, these were subjected to various amounts of clay; 5, 10 and 20

g at a constant temperature of 250 °C (Table 1). It has been noted that all treated oil samples are giving similar patterns of absorption to that obtained by the virgin, as shown in Figure 4. However, the treated oil exhibited higher absorbance than that of the virgin oil with peaks at 250, 252 and 250 nm for S1, S2 and S3 with absorbance values of 2.564, 2.562 and 2.564, respectively. The second peak was noted at the same wavelength of that at the virgin sample at 314 nm for both S1 and S2 and at 312 nm for S3 and the absorbance showed slight deviation among the treated samples at these wavelengths. Sample, S1 gave a value of 2.175 absorbance in comparison to 2.185 for S2 and 2.182 for S3. Comparing these values with S0 clearly indicates that oil absorbance is decreasing after the addition of clay to oil samples. This result was also confirmed when looking at the overall pattern of samples indicating shifting of absorbance towards virgin results which can conclude improving oil's quality.

Further analysis was therefore performed by looking at the impact of clay with the same ratio of weight to volume, but by increasing temperatures from 350 °C to 400 °C as shown in Figure 5. Similar patterns were observed as presented in Figure 5. The absorbance of treated oil was taking the same pattern for that of the virgin sample and that the increasing ratio of clay weight to volume is shifting the absorbance curve. The peaks however for the samples were not giving supporting evidence of these finding as noted for S4 at 250 nm with an absorbance of 2.579 and S5 absorbance of 2.587 at the same wavelength whereas S6 gave as specific absorbance at 248nm. The second peaks recorded were at 312 nm for both S4 and S5 with absorbance values of 2.197 and 2.191, but S6 gave an absorbance at 314 nm of 2.203. This value for S6 contradicts with expectations as indicates poor quality

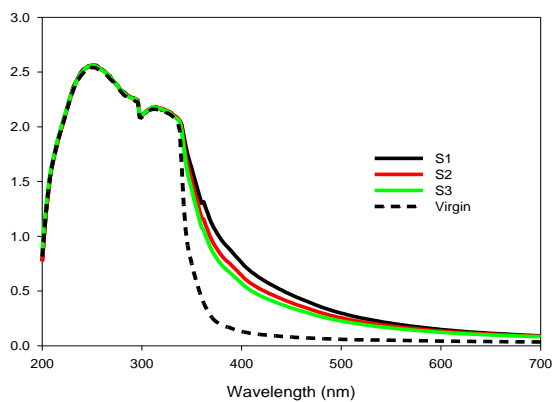


Figure 4. Absorbance of treated oil S1, S2 and S3 at 250°C

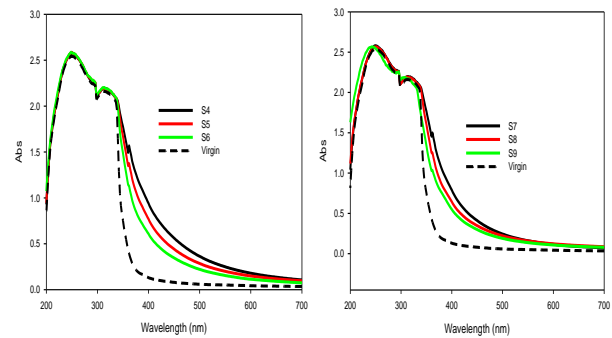


Figure 5. Absorbance of treated oil S4- S7 at 350°C and 400°C

of oil in comparison to S4 and S5. However, the overall patterns disagree with this conclusion as presented in Figure 4.

The comparison between absorbance when varying the temperature between 350 °C and 400 °C is not conspicuous, as the absorbance values for S4 is 2.579 and S7 is 2.580 both at wavelengths of 250 nm for the first peak, and 2.197 and 2.198 absorbance at 312 and 314 consecutively. Similarly, the absorbance of S5 is 2.587, S8 is 2.566 for the first peak at 250 and 252 nm and the second peaks both at 312 nm with values of 2.191 and 2.192.

At a higher temperature of 450 °C, the absorbance pattern obtained from virgin and untreated oil is no longer maintained as presented in Figure 6. It is observed that the oil absorbance first peak is at 242 nm for S10 and at 240 nm for S11 and 238nm for S12 with absorbance values of 2.584, 2.594 and 2.582 consecutively. The inconsistent absorbance noted can be justified by the breakup of a different number of acidic additives' components of the oil at this high temperature [6, 19].

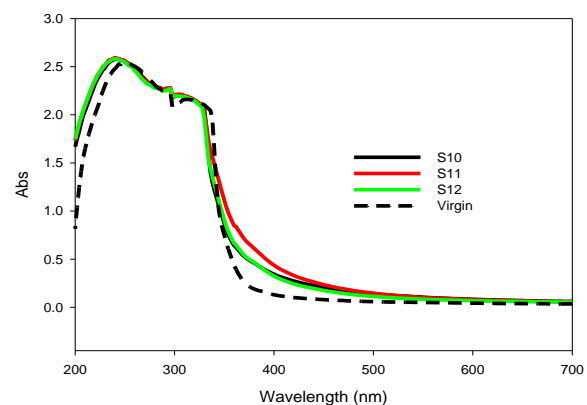


Figure 6. Absorbance of treated oil S10, S11 and S12 at 450°C

To confirm these findings, comparisons were made between these results for a given ratio of clay weight to oil volume at the investigated range of temperatures. Figure 7 presented the comparison for 5 g of clay at 250, 350, 400 and 450°C. It is noted that although the pattern of oil absorbance is similar to that of the virgin, the increase of temperature has inconsistently altered the absorbance values and peaks.

It is also noted that the pattern of oil absorbance is maintained indicating preserving overall oil quality. However, the increase in temperature has also altered the absorbance values as summarized in Table 4. This alteration can be justified by the alteration of clay morphology due to high temperature exposure as reported by other researchers [22-24].

Summary of these results is presented in Table 4. A range of wavelength from 200 to 350 nm was defined and the area under the curve was noted for all tested samples. The overall values of these areas were insufficient in reaching a similar conclusion to those obtained from the overall patters presented in Figures 4-

7. It is, therefore, to be concluded that both absorbance peaks and areas under the curve are unable to provide a detailed understanding of overall oil samples qualities.

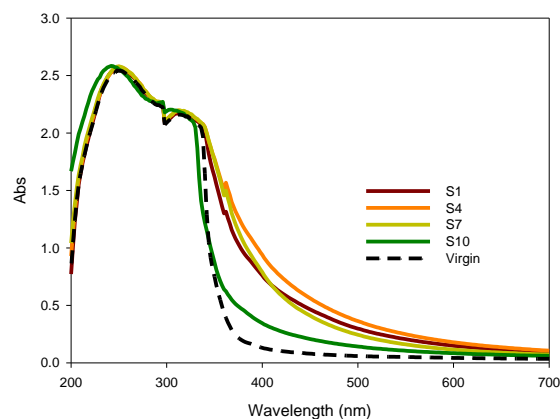


Figure 7. Absorbance of treated oil S1, S4, S7 and S10

TABLE 4. Absorbance Peaks of different samples and wavelength

Sample	Peak I		Area Under Curve from 200- 350 nm	Peak II	
	Wavelength(nm)	Abs		Wavelength(nm)	Abs
Virgin	248.00	2.545	223.20	314.00	2.160
S0	252.00	2.569	225.40	312.00	2.196
S1	250.00	2.564	224.66	314.00	2.175
S2	252.00	2.562	224.08	314.00	2.185
S3	250.00	2.564	225.21	312.00	2.182
S4	250.00	2.579	226.89	312.00	2.197
S5	250.00	2.587	227.51	312.00	2.191
S6	248.00	2.587	229.40	314.00	2.203
S7	250.00	2.580	228.39	314.00	2.198
S8	252.00	2.566	228.32	312.00	2.192
S9	242.00	2.566	235.62	306.00	2.186
S10	242.00	2.584	237.52	No peak detected	No peak detected
S11	240.00	2.594	239.38	No peak detected	No peak detected
S12	238.00	2.582	238.56	No peak detected	No peak detected

4. CONCLUSIONS

In this work, natural clay from the southern region of KSA has been used in treating waste engine oil. From the results it has been concluded that the investigated clay exhibits good adsorption capabilities for decolourization enhancement of the waste engine oil. This clay mineral is a potential candidate for possible bleaching earth as an effective adsorbent, readily available, economical and capable of removing various

contaminants from various pollutants of different sources. The decolourization was evaluated and assessed by using UV- spectrophotometer.

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Adsorption

UV-Spectrophotometer

هدف از پژوهش حاضر، بررسی پتانسیل مواد معدنی خاک رس طبیعی است که به عنوان یک جاذب موثر برای تصفیه روغن موتور سوخته عمل می کنند. این مواد معدنی رس از منطقه جنوبی عربستان سعودی جمع آوری و بدون هیچ گونه اصلاح سطح مورد استفاده قرار گرفت. مواد معدنی رس توسط آنالیز پراش پرتوی (XRD)، فلورسانس اشعه X (XPS) و Brauner-Emitt-Teller (BET) تجزیه و تحلیل سطح مشخص گردید. داده های XRD ساختار کریستالی رس را نشان می دهد در حالی که ترکیب شیمیایی خاک رس توسط آنالیز XRF تعیین شده است که در آن اجزای اصلی (SiO₂ ۷۰/۵۱ درصد)، Al₂O₃ و Fe₂O₃ با ۲۹،۱۷ درصد و ۱۳/۲۲ درصد تشکیل شده است. سایر ترکیبات جزئی نیز شناسایی شده اند. سطح BET حدود ۶۵ متر مربع بر گرم بود. مقدار جاذب خشت (بین ۲۰-۲۰ گرم) به ۱۰۰ میلی لیتر روغن موتور سوخته تحت شرایط دمایی مختلف اضافه شد. تجزیه و تحلیل نمونه به دست آمده توسط دستگاه اشعه ماوراء بنفش نشان می دهد که نمونه روغن S10 که در دمای ۴۵۰ درجه سانتیگراد با ۵ گرم خاک رس انجام گردید، بهترین همبستگی (قله ها و طول موج) را بسیار نزدیک روغن اصلی نشان داد. از این رو، پیشنهاد می شود که اینها شرایط بهینه کار عملی گردند.

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