SUSTAINABLE
ENGINEERING

(eISSN:3083-8231)
Article
Volume 2, Issue 2, 2025
https://doi.org/10.51200/susten.v2i2.5679

Waste Lubricant Oil Treatment by Adsorption Process Using Modified Activated Carbon

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Received: 06 June 2025, Accepted: 25 Aug 2025

Abstract: The main objective of this study was to investigate the performance of modified activated carbon (MAC) in recovering waste lubricating oil (WLO) through the adsorption process. The activated carbon (AC) was modified by nitric acid (HNO_3) to increase its adsorption capacity. The first treatment of WLO was done by mixing it with glacial acetic acid and followed by adsorption treatment using MAC. The effectiveness of the adsorption experiment in treating WLO using MAC in various parameters such as different dosages, temperatures, and contact times was studied. The treated lubricating oil (LO) underwent comprehensive physical characterization for water content, density, and viscosity. The highest water removal percentage was observed in sample C1 (4.83%). It was found out that sample C1 has a better density value with 0.799 g/m and viscosity of 88 mPa.s. The characterization of the recovered oil before and after treatment was analyzed by using Fourier transform infrared spectroscopy (FTIR) and ultraviolet-visible (UV-Vis) spectrophotometry. The absence of observable S=O and C=O bands indicates the successful purification of the treated LO samples. In UV-Vis analysis, the lowest concentration absorbance values were observed in sample C1 (0.291) and sample C2 (0.328) at wavelength 200 nm, indicating that the most intense treatment may cause the decomposition of these absorbing species or the formation of new compounds with lower absorption in this range. To conclude, this study gives an insightful method for treating LO through the adsorption process by using unmodified AC and MAC.

Keywords: waste lubricant oil; adsorption; modified activated carbon; nitric acid

1. Introduction

Waste lubricating oil (WLO) contains various impurities, including heavy metals, organic compounds, and contaminants, which can be harmful if not properly managed [1]. Therefore, recovering base oil from WLO has the potential to bring economic benefits by lowering the cost of fresh oil production. WLO segregation is critical for increasing regeneration rates and obtaining high-quality base stock [2]. Therefore, proper management aims to reduce the environmental impact of spent oil and change its nature so that it can be reused as fresh oil.

Adsorption refers to the physical or chemical bonding of material molecules with effective sites on a surface via the weak Van der Waals force or by forming chemical bonds with those sites [3]. Adsorption is a chemical process in which the solid is referred to as the adsorbent and the solute as the adsorbate. This method is extremely effective at removing heavy metals, organic compounds, and other pollutants from WLO [4]. Up to 99% of the heavy metals present in the WLO were successfully deposited through the adsorption/extraction method [5]. The adsorption process uses an adsorbent that will be adsorbed and trap the contaminants from WLO. Table 1 provides types of adsorbents for the adsorption process in WLO recovery.

Table 1. Types of adsorbent for adsorption process for WLO recovery

Adsorbent	Description	Result	References
Butanol and kaolin	Investigation of the chemical and adsorption methods utilizing kaolin and butanol on the metal concentration in WLO.	The WLO after treatment has metal contents of 0.17, 9.85, 34.07, 78.22, and 1.20 ppm for Ca, Mg, Pb, Fe, and Cr, respectively.	[6]
Sawdust	The modification of sawdust with sodium hydroxide and triethanolamine to improve surface properties	The removal percentages for Si, Al, Fe, and Cu were increased from 2.54%, 20.34%, 16.55%, and 0.26% to 15.37%, 21.99%, 45.37%, and 4.88%, respectively, whereas those for oxidation, aromatics, sulphation, soot, and water were increased by 4.33, 4.69, 0.76, 1.20, and 1.28 times at 80 °C and 1000 rpm for 12 hours.	[7]
Acid clay	Clays, such as bentonite and montmorillonite, are natural adsorbents with a layered structure that facilitates adsorption through ion exchange and surface interactions.	The test results showed that viscosity increased from 38.3 cst for WLO to 85 cst for acid/clay treatment, flash point increased from 178 °C to 238 °C, density fell from 912 to 896 kg/m^3, pour point increased from 6 to 13.2 °C, and color decreased from 0.53 to 0.12.	[8]
Zeolite	A study focused on the purification of recovered base oil by using a zeolite to remove heavy metals.	The results showed that zinc removal was greater than magnesium removal. The best results for magnesium and zinc removal were 50 and 62%, respectively.	[9]

Furthermore, it has been discovered that without the use of a suitable adsorbent material, the adsorption process is not always complete. Not all adsorbents are equally effective at removing all types of contaminants. The study found that the modified activated carbon (MAC) had a higher adsorption capacity for petroleum hydrocarbons compared to bentonite [10]. The selection of the suitable adsorbent material is important to the adsorption process's performance. Hence, activated carbon (AC) was chosen as an adsorbent since it is a well-known and widely used adsorbent for treating WLO and various chemical contaminants. Commercial AC is the adsorbents that will be used in the treatment of WLO. It can be made from a variety of carbonaceous materials, including coal, bamboo, agricultural waste, and old tires [11].

However, commercial ACs still suffers limitations such as variable quality and performance. Modifying the AC by treatment with nitric acid (HNO_3) enhances properties like porosity and surface chemistry, increasing adsorption capacity and affinity for contaminants. The modification of AC with HNO_3 can improve its adsorption properties by increasing its surface area and introducing new functional groups that can attract impurities from the WLO [12-14]. The utilization of HNO_3 induced several changes in the AC's structure to make a porous structure and expansive surface area to optimize the interaction between the oil and the adsorbent, hence increasing the effectiveness of the adsorption process [12].

The main objective of this study was to investigate the performance of MAC in recovering and enhanced the quality of WLO through the adsorption process. The effectiveness of the adsorption experiment in treating WLO using MAC in various parameters, such as different dosages, temperatures, and contact times, was assessed. The physical characterization for the performance of the adsorbents AC and MAC in recovering the WLO was determined. The characterization of the WLO after treatment was analyzed by using Fourier transform infrared (FTIR) spectroscopy and Ultraviolet-Visible (UV-Vis) spectrophotometry.

2. Materials and Methods

Raw material

The WLO was collected from local car oil-change shops. The WLO was Castrol GTX 15W-40 SN/CF mineral engine oil. Charcoal AC (DARCO) was purchased from Sigma-Aldrich. The charcoal AC was in granular form with mesh particle sizes ranging from 20 to 40. The charcoal AC has a total surface area of 650 m^2/g, ash content of ≤0.40%, and a moisture content of ≤12%. HNO_3 with a concentration of 63% was obtained from ChemAR, SYSTERM. The essential laboratory apparatus and equipment used in this study were an electronic measuring balance, measuring cylinder, beaker, oven, hot plate, centrifuge, dropper, magnetic stirrer, reagent bottle, thermometer, Fourier transform infrared spectroscopy (FTIR) (Bruker INVENIO®), and UV-Vis spectrophotometry (Perkin Elmer LAMBDA™ 365).

Preparation of MAC

10 g of AC was cleaned with deionized water to eliminate tiny AC powder. The sample was poured into a round-bottom flask and placed on a heating mantle. Then, 100 mL of HNO_3 (at a 1:1 acid-to-water ratio) was introduced to a round-bottom flask at room temperature for 2 hours and left for 24 hours. Finally, AC was continuously washed with distilled water until the pH was stable, then dried overnight at 120 °C [15].

Dehydration of WLO

The oil was heated to 250 °C in an oven for approximately 1 hour to undergo the dehydration process [10]. Subsequently, the oil was allowed to cool to room temperature before proceeding to the filtration stage.

Acid treatment

A mixture of 10 mL of filtered oil and 1 mL of glacial acetic acid was heated and stirred for about 1 hour at room temperature using a hot plate [16]. Following this, the mixture was left to settle at room temperature for 24 hours. The final stage of the acid treatment involves centrifuging at 300 rpm for an hour to separate the base oil from impurities.

Adsorption process

A mixture of 2 g, 4 g, and 6 g of AC was combined with 10 mL of the treated oil and left for 30 min, 1 hour, and 1 hour 30 min at 40, 50, and 60 °C, respectively. The same procedure was repeated using MAC. Subsequently, the oil was centrifuged for 30 minutes at 3000, 4000 rpm, and 6000 rpm to obtain the final recovered oil, separating the oil from the sludge. A total of 6 samples were prepared for conducting adsorption experiments; 3 samples (A1, B1, and C1) were prepared using MAC treated with HNO_3 and the remaining 3 samples (A2, B2, and C2) were prepared using unmodified AC. Table 2 provides the description of process parameters for each sample. The recovered lubricating oil (LO) samples from the treatment underwent property analyses using FTIR and UV-Vis spectrophotometry.

Table 2. Description of process parameters for each sample

Samples	Description
A1	2g MAC + 40 °C + 30 min + 3000 rpm
A2	2g AC + 40 °C + 30 min + 3000 rpm
B1	4g MAC + 50 °C + 1 hour + 4000 rpm
B2	4g AC + 50 °C + 1 hour + 4000 rpm
C1	6g MAC + 60 °C + 1 hour 30 min + 6000 rpm
C2	6g AC + 60 °C + 1 hour 30 min + 6000 rpm

Water content removal

This study used several steps to identify the water content in WLO. First, the WLO was filtered to remove tiny particles. An empty 250 mL beaker was weighed using an electronic balance, and 100 mL of WLO was measured and placed into the beaker. The WLO was then heated to 250 °C in an oven for an hour to remove any remaining water. After cooling to room temperature, the dehydrated WLO was weighed again. The final weight was recorded, and the traditional oven-drying process equation was used to determine the water content removal [17].

$$\textbf{Water content removal}, \% = \frac{\textbf{intial mass (g)} - \textbf{final mass (g)}}{\textbf{initial mass (g)}} \times \mathbf{100} \qquad (1)$$

Density

Densities of the oil samples at room temperature were calculated using equation 2. A density container with a known mass and volume was properly filled with each oil sample. After the masses of the oil and the bottle were determined, the mass of the oil was computed using the bottle's known volume. The density was then computed using the mass and volume that were reported in the following way:

$$\textbf{Density} = \frac{\textbf{Mass of the sample oil, g}}{\textbf{Volume of the sample oil, mL}} \qquad (2)$$

Viscosity

The viscosity of the regenerated oil was measured using a DV-E viscometer. The procedure involved selecting a suitable spindle and adding enough oil to a beaker. The spindle was placed in the oil up to a marked line. All samples were kept at the same temperature, spindle size, and speed for consistency. The viscosity in centipoise (cP) was displayed on the instrument, and the most prominent reading was recorded for analysis.

FTIR analysis

With FTIR, the oil's chemical structure and functional groups were to be determined. The FTIR employed in this study has a wavelength range of 400–4000 cm^{-1} [18]. 1 drop of modified oil samples was placed onto the surface of the attenuated total reflectance (ATR) accessory. The ATR crystal, often made of diamond or another IR-transparent material, facilitates the interaction between the infrared light and the sample. The FTIR spectra of AC before and after modification were used to identify changes in the material's functional groups, which can be crucial for understanding its behavior in various processes.

UV-Vis spectrophotometry analysis

UV-Vis spectrophotometry was used to analyze the recovered oil to measure the amounts and UV wavelengths that were absorbed. In order to facilitate comparisons between samples, the color of the treated LO was absorbed from the instrument and measured at peak 510.02 nm for each sample [18]. The modified oil samples (1 mL) were first diluted with 10 mL of kerosene. This method allows for the characterization of the oil and the detection of any changes in its properties.

Adsorption capacity and removal efficiency before and after modification

Adsorption capacity (Q_e) and removal efficiency (R) before and after modification were calculated using equations 3 and 4, respectively.

$$\text{Adsorption capacity } (Q_e), mg/g = \frac{(C_0 - C_e)V}{m} \qquad (3)$$

where, C_0 is the initial concentration of the contaminant; C_e is the final concentration of the contaminant; V is the volume of the solution, m is the mass of the adsorbent.

$$\text{Removal efficiency } (R), \% = \frac{(C_0 - C_e)}{C_0} \times 100 \qquad (4)$$

3. Results and Discussion

3.1.1. Characterizations for physical and chemical properties of treated LO

3.1.1. Water content removal of WLO

Figure 1 illustrates the percentage of water content removal from WLO after treatment with unmodified AC and MAC.

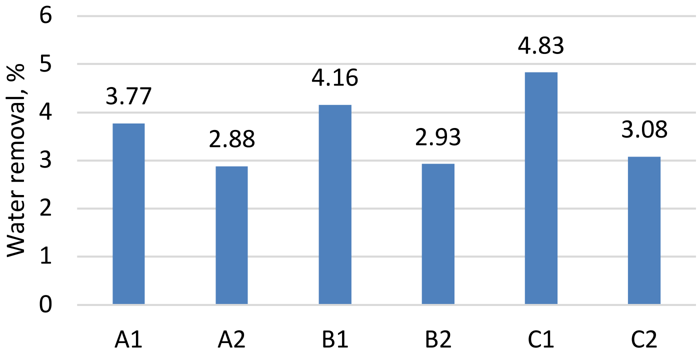


Figure 1. Water content removal from the WLO samples

Based on Figure 1, all the WLO samples clearly contained water. In this investigation, 6 WLO samples were tested for water content, and all of them included water. The highest water removal percentage was observed in sample C1 (4.83%). The lowest water removal percentage was observed in sample A2 (2.88%). The differences in moisture content removal percentages can be due to a variety of variables. The surface area of AC determines its ability to adsorb moisture, with larger surface areas often allowing for more effective moisture uptake. [19,20] These parameters can influence the efficiency of water removal from WLO using MAC. Increasing the temperature can improve water removal efficiency. The analysis of water removal percentages from WLO treated with MAC highlights the complex interplay of factors influencing the efficiency of water removal. The surface area and pore structure of AC play a crucial role, with a larger surface area and well-defined pore structure enhancing its ability to adsorb water effectively.

3.1.2. Density of WLO

Figure 2 illustrates the densities of WLO after treatment with unmodified AC and MAC.

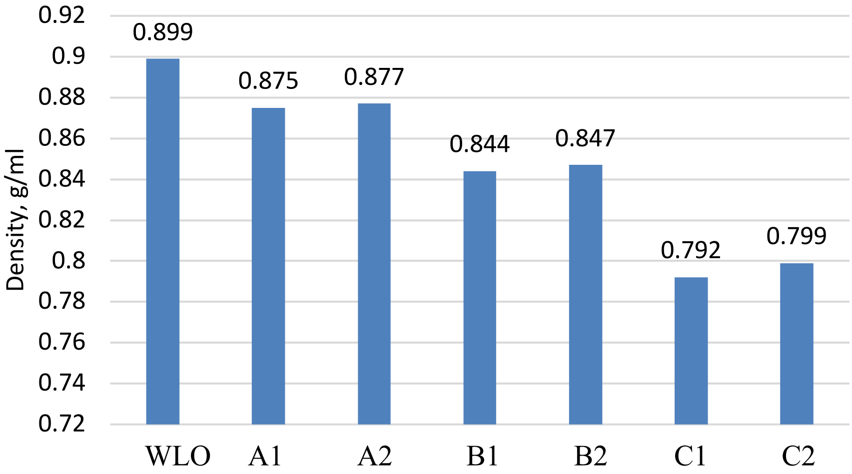


Figure 2. Densities of the treated WLO samples

Based on Figure 2, each sample shows a marked decrease in density after the treatment process. Based on the data density above, the value density of the WLO obtained shows a higher which 0.899 g/mL. This is due to the presence of sludge and a greater percentage of sulphur compounds [21]. The density of the WLO shows a higher density (0.899 g/mL) compared to all the treated samples. The higher density of the WLO is due to the amount of solids that is present in the oil. According to Hamawand et al. [16], the density of the WLO increases due to the metal, contamination, and oxidation products that are formed while the LO is in use. Thus, the purpose of the recovery of WLO was to reclaim its density property of the oil. The density in sample A1 (0.875 g/mL) and sample A2 (0.877 g/mL) show a slight density reduction compared to the untreated oil. Next, the density in samples B1 (0.844 g/ mL) and B2 (0.847 g/ mL), which were treated under moderate conditions shows higher density reduction. The greater density reduction indicates a significant alteration in the oil's composition, possibly by removing heavier contaminants or breaking down larger molecules into lighter fractions. Lastly, the density in sample C1 (0.792 g/ mL) and C2 (0.799 g/ mL) which were treated under the most severe conditions, these samples show the lowest densities. The lower density of C1 compared to C2 implies that MAC treatment at these conditions leads to a more profound change in the oil's composition than AC treatment, possibly due to better impurity removal or more extensive breakdown of the oil's components. According to the recorded results, the WLO has a higher density than the existing treated lubricant oil and all treated samples. According to Khan et al. [22], the virgin lube oil shows 0.882 g/ mL. Therefore, the result on Figure 2 shows valued density below this virgin lube oil decreasing indicating that the treatment effectively modifies the lubricant oil's composition by removing heavier contaminants and breaking down larger molecules.

3.1.3. Viscosity of treated lubricant oil

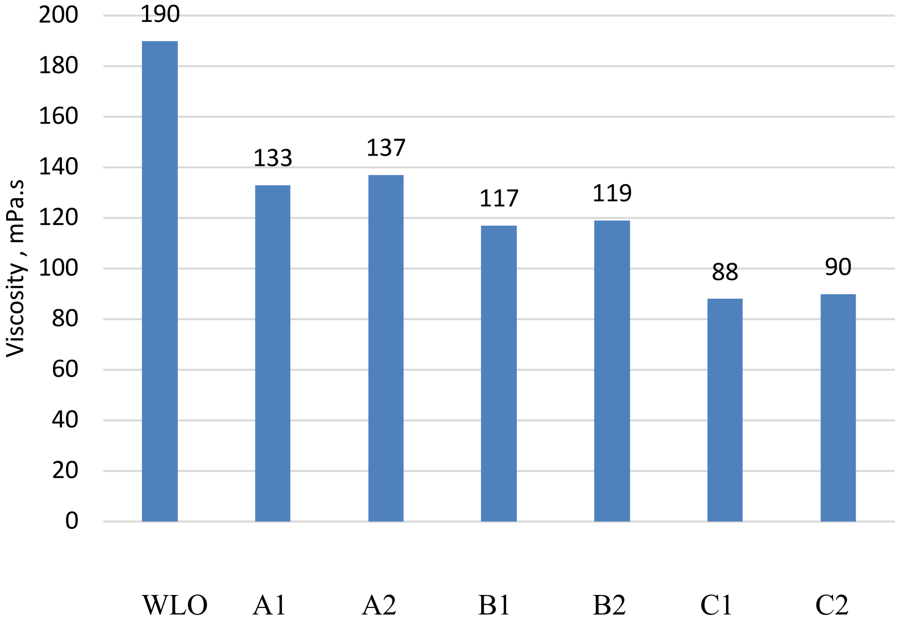


Figure 3. Viscosity of the WLO and treated WLO samples

Figure 3 presents the viscosity of the WLO and treated WLO samples. As shown in Figure 3 that WLO has the highest viscosity at 190 mPa.s, serving as the baseline for comparison. The comparison of viscosity can be made from [5] where the WLO gave the highest viscosity at 180 cP. This high viscosity indicates the presence of contaminants in the used oil. Next, A1 (133 mPa.s) and A2 (137 mPa.s) show a significant reduction in viscosity. This reduction suggests effective removal of contaminants through treatment with activated carbon or modified activated carbon. Besides that, B1 (117 mPa.s) and B2 (119 mPa.s) exhibit further reduction in viscosity. Lastly, C1 (88 mPa.s) and C2 (90 mPa.s) show the lowest viscosities among the treated samples. This trend suggests that the treatment with MAC may have a slightly different effect on viscosity compared to AC. Viscosity reduces with increasing temperature, and vice versa [23]. Due to the molecular collision occurring during the momentum transfer between the layers, the liquid lubricant's viscosity will drop as the temperature rises. As a result, as temperature rises, momentum transfers quickly, lowering the viscosity level [24,25]. The decrease in viscosity following treatment signifies the successful elimination of contaminants and enhancement of the lubricant oil's flow characteristics. Therefore, optimizing the viscosity is maximizing the adsorption of pollutants [26]. Hence, the adsorption performance of the material can be enhanced, leading to more efficient removal of contaminants from the LO.

3.2. Analysis performances in treated WLO

3.2.1. FTIR analysis

The FTIR approach was utilized to analyze the oxidation of lubricant oil and determine oil quality changes [26,27]. By comparing the FTIR spectra of WLO and treated LO, researchers studied the chemical structures of these oils. The analysis involved assessing individual band levels in used oils through differential spectra, subtracting used oil spectra from fresh oil spectra. Figure 4 shows the FTIR analysis of WLO, and Table 3 represents the wavelength of the component that was found in the LO treated using unmodified AC and MAC.

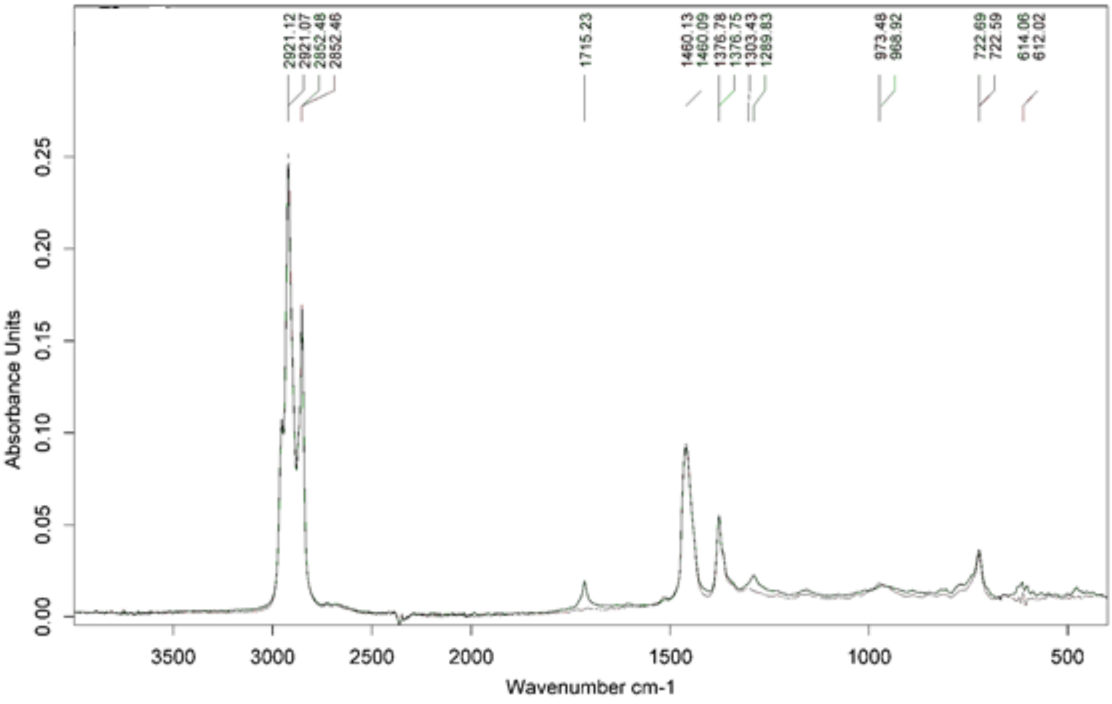


Figure 4. FTIR analysis of WLO

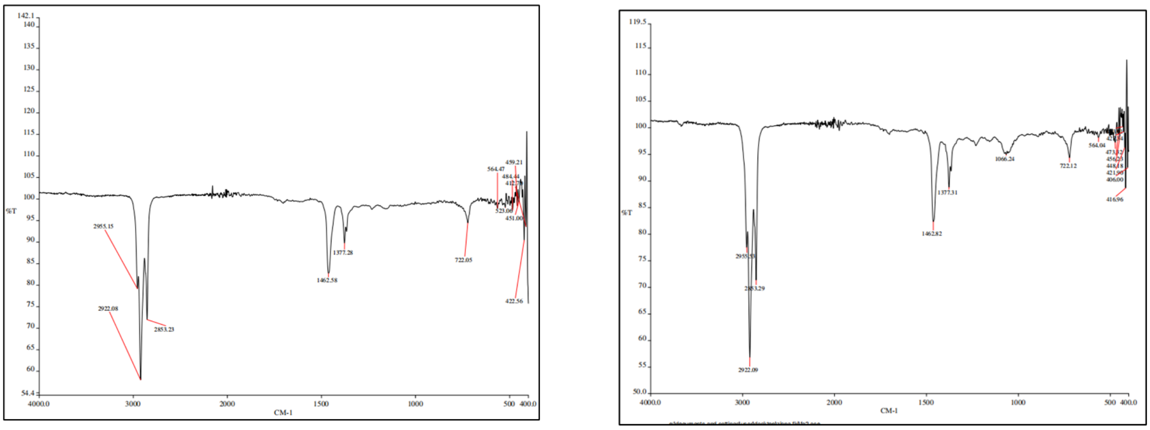
Table 3. Wavelength of component that was found in the treated LO using unmodified AC and MAC

Samples	**Wavelength (cm -1)**				
	C-H Stretching	**C-H2 Stretching**	**C=0 Stretching**	**C-S Stretching**	**S=0 Stretching**
WLO	2921.07 2852.46 1376.78	1460.13	1715.20	722.59	und
A1	2922.08 2853.23 1377.28	1460.13	und	722.05	und
A2	2922.09 2853.29 1377.31	1460.13	und	722.12	und
B1	2922.14 2853.28 1377.28	1460.13	und	721.91	und
B2	2922.13 2853.31 1377.24	1460.13	und	722.28	und
C1	2922.04 2853.18 1377.28	1460.13	und	722.13	und
C2	2922.11 2853.25 1377.26	1460.13	und	722.59	und

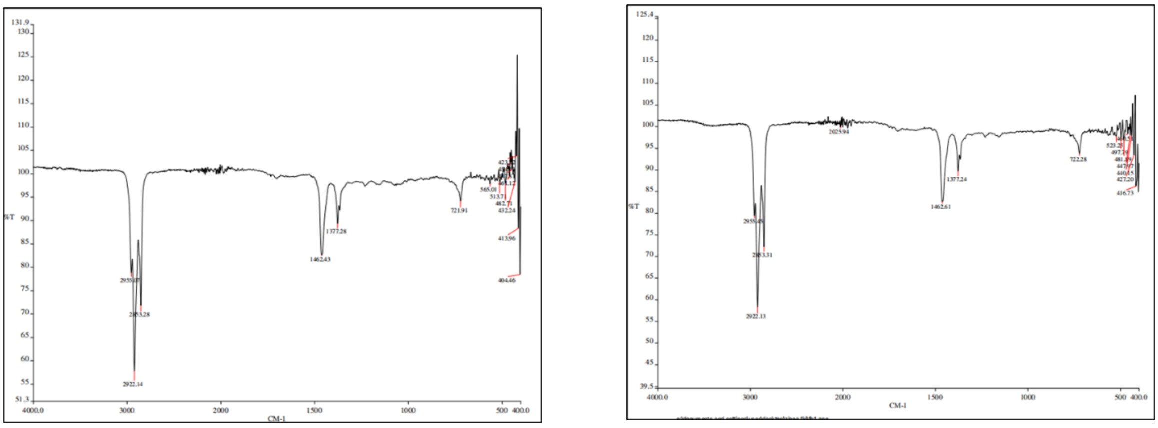
und = undefined

FTIR analysis is valuable for identifying various functional groups such as C-H stretching, C-H2 bending, C=O (esters), C-S, and S=O stretching in LO samples. In the observations from the Figure 6 and Table 3, it was shows that WLO exhibited C-H stretching at wavelengths of 2921.07 cm^{-1}, 2852.46 cm^{-1}, and 1376.78 cm^{-1}, with C-H bending also detected at 1460.13 cm^{-1} [28]. The C-S group was identified at a wavelength of 722.63 cm-1. However, the presence of a carbonyl group at a wavelength of 1715.20 cm^{-1} with higher absorbance units indicated contamination of the LO due to oxidation [5]. According to Ashirov [29], the LO consists of long chain hydrocarbons and in FTIR spectrum, long C-H stretching and bending at 2800-3000 cm^{-1} and strong CH$_2$ bending peaks at 1460 cm^{-1} can be seen.

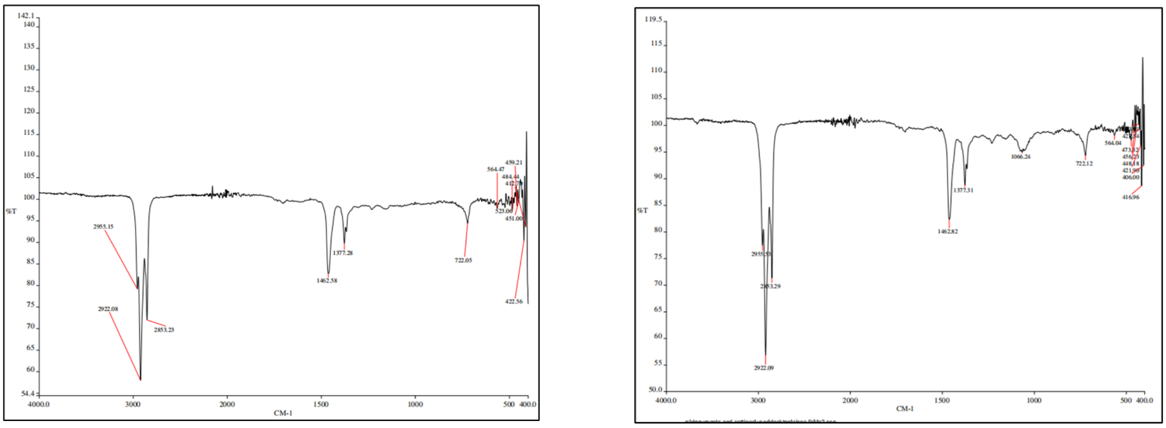
Figure 5 shows the FTIR analysis of treated LO using unmodified AC and MAC



(a) Sample A1 **(b)** Sample A2



(c) Sample B1 **(d)** Sample B2



(e) Sample C1 **(f)** Sample C2

Figure 5. FTIR analysis for six samples

The results for all samples showed the absence of carbonyl groups resulting from oxidation, suggesting that these compounds may have been adsorbed or degraded during the treatment process (Table 3). Notably, the absence of detectable S=O stretching vibrations indicates that sulphur oxides or sulfone compounds are not present. The lack of measurable C=O vibrations indicates a significant decrease or absence of ester compounds in the lubricating oil. Furthermore, the absence of observable S=O and C=O bands indicates the successful purification of the LO, highlighting the removal or reduction of sulfur oxides, sulfone compounds, and ester compounds from the samples.

3.2.2. UV-Vis spectrophotometry

The UV-Vis spectroscopic analysis of treated lubricant oils can provide information about the oxidation state and chemical changes in the oil. Based on Figure 6, the UV-Vis absorbance data presented involve measurements at 3 specific wavelengths, 200 nm, 400 nm, and 665 nm, for six different samples [30].

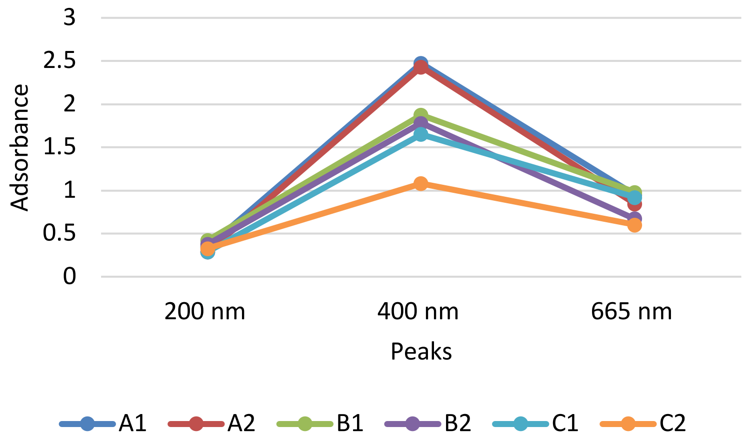


Figure 6. Adsorbance analysis on UV-Vis spectrophotometry for treated LO

The absorbance values at 200 nm provide valuable insights into the treated samples' characteristics. Samples B1 and B2 exhibit the absorbance values (0.422 and 0.376, respectively). In contrast, samples A1 and A2 show lower absorbance values (0.345 and 0.289, respectively), indicating a lower presence of UV-absorbing species. The lowest absorbance values are observed in C1 (0.291) and C2 (0.328), indicating that the most intense treatment may cause the decomposition of these absorbing species or the formation of new compounds with lower absorption in this range.

The absorbance at 400 nm reveals interesting trends. Samples A1 (2.472) and A2 (2.432) exhibit the highest absorbance values, suggesting a high concentration of oxidation products or contaminants that absorb in the UV range. As the treatment conditions intensify, the absorbance values decrease. This trend is evident in samples B1 (1.873) and B2 (1.782), with further reductions in C1 (1.649) and C2 (1.079). This reduction indicates that more severe treatment conditions effectively reduce the concentration or breakdown of the oil components. Notably, MAC samples consistently show higher absorbance than AC samples under the same conditions, indicating potential differences in their response to treatment.

The absorbance at 665 nm, which is generally lower than at 400 nm, reveals insights into visible-range absorbing compounds. Samples A1 (0.949) and A2 (0.847) again display higher absorbance values, suggesting the presence of specific additives or degradation products that absorb in the visible range. The decrease in absorbance with more intense treatment continues, with B1 (0.979) and B2 (0.671) showing intermediate values, and C1 (0.920) and C2 (0.603) displaying the lowest values. This pattern indicates that intense treatment conditions lead to the breakdown or depletion of these visible-absorbing compounds. The notably lower absorbance of AC samples compared to MAC samples suggests a greater degradation or loss of these compounds in AC under similar conditions.

3.2.3. Adsorption capacity and removal efficiency before and after modification

Table 4 presents the summary of adsorption capacity and removal efficiency.

Table 4. Summary of adsorption capacity and removal efficiency

Samples	Adsorption capacity, mg/g	Removal efficiency, %
A1	2.7	15.2
A2	1.8	8.9
B1	3.5	17.7
B2	2.3	11.4
C1	4.2	20.3
C2	3.5	13.9

The comparison of unmodified and MAC reveals the significant effect of modification on adsorption capacity and removal efficiency. The data reveal that MAC samples consistently have greater removal efficiencies and adsorption capacities than their unmodified equivalents. Sample C1 has the maximum removal efficiency of 20.3% and the highest adsorption capacity (4.2 mg/g) among all the treated WLO samples. In contrast, unmodified samples show lower performance metrics, with C2 being the best among them but still falling short compared to the MAC samples. The results indicate that while unmodified AC can be effective, its performance is significantly improved by modification, which enhances its ability to adsorb oil molecules. Several reasons contribute to this increased efficiency. A larger amount of adsorbent increases the surface area available for adsorption, resulting in more efficient oil removal from the solution. With more AC present, the oil molecules are more effectively captured, leading to higher removal efficiency. Finally, MAC with HNO_3 may have been designed to have better adsorption capabilities than conventional AC.

4. Conclusions

This study has proved that the treatment of WLO using MAC through an adsorption process has yielded significant findings, demonstrating the effectiveness of MAC in recovering WLO. The study systematically assessed the performance of MAC under various conditions, including dosage, temperature, and contact time, revealing that both unmodified AC and MAC exhibited substantial adsorption capacities for removing impurities from oil samples. The analysis of physical properties indicated that MAC outperformed in recovering WLO, with notable improvements in water removal, density, and viscosity. The highest water content removal for treated LO was observed at 4.83%. It was found out that sample C1 using MAC has a better density value with 0.799 g/m and viscosity values is 88 mPa.s. The functional group obtained from the treated LO exhibits the presence of aliphatic hydrocarbons, methylene, and ester whose value is within the range from the virgin oil in FTIR analysis. In UV-Vis analysis, the lowest concentration absorbance values were observed in sample C1 (0.291) and sample C2 (0.328) at wavelength 200 nm. The UV-Vis spectrophotometry analysis at 200 nm, 400 nm, and 665 nm offers a comprehensive understanding of the chemical changes in treated LO. The higher absorbance values observed in samples treated with MAC compared to samples treated with AC alone indicate that MAC improves the removal of certain compounds from the LO. Overall, this study successfully met its objectives, highlighting that the MAC was successfully done and could enhance the adsorbent performance for the treatment of WLO.

Acknowledgements

The authors pay their sincere gratitude to Universiti Malaysia Sabah (UMS) for providing necessary research facilities to accomplish this study.

Conflicts of Interest

The authors declare no conflict of interest.

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