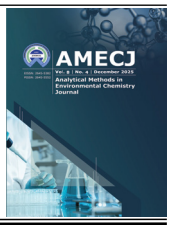
Anal. Methods Environ. Chem. J. 8 (4) (2025) 67-84

AMECJ

Research Article, Issue 4
Analytical Methods in Environmental Chemistry Journal
Journal home page: www.amecj.com/ir



Analysis and removal of free fatty acid and peroxides from waste cooking oil using Fuller's earth-China clay adsorbent by titration methods

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ARTICLE INFO:

Received 9 Aug 2025
Revised form 20 Oct 2025
Accepted 16 Nov 2025
Available online 27 Dec 2025

Keywords:
Adsorption,
Fuller's Earth-China Clay,
Waste cooking oil (WCO),
Purified cooking oil (PCO),
Free fatty acids (FFAs),
Peroxide value (PV)

ABSTRACT

The current study investigated the suitability of Fuller's Earth-China Clay (FECC) as an adsorbent for removing toxicants, including free fatty acid (FFAs) and peroxides, from waste cooking oil (WCO). Key quality indicators, such as FFAs, peroxide value (PV), and color change, were measured to evaluate the effectiveness of using indigenous FECC material. FECC was activated using sodium hydroxide (NaOH) and acetic acid to enhance its adsorption ability, surface area, and active sites. The structural and functional properties of activated FECC were characterized using techniques like Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), BET surface area analysis, and scanning electron microscopy (SEM). The FFA content in the WCO reduced from 2.98 % to 0.91 % after being treated with activated FECC, having a reduction percentage of 69.64 %, and the peroxide value reduced from 5.5 meq/1000 g to 1.0 meq/1000 g, thus, having a reduction percentage of 81.82% and RSD<2%. There was a marked increase in the oil's quality, with significant absorption capacity, with about 80-85% of the oil purified. These results show that activated FECC can be potentially used as an efficient, environmentally friendly, and economical solution for purifying waste cooking oil. This research provides a promising method to enhance oil quality and a step towards addressing food safety challenges, with the potential for broader application of oil refining in the food sector.

1. Introduction

Approximately 41 to 67 million metric tons of cooking oil are produced and consumed yearly, and 20 to 32% of this amount is in the form of Waste cooking oil (WCO) [1]. WCO is mainly generated by households, hotels, and the food industry and poses potential risks to the environment and health due to the presence of toxicants [2, 3]. For practical and financial reasons, it is common to reuse cooking oil multiple times for frying. [4]. However, with repeated frying, oxygen can enter the oil and react with unsaturated acylglycerols to form undesirable products, including dimeric and polymeric acids, dimeric acylglycerols, free fatty acids (FFAs), and peroxides. These reactions form polar compounds, which, in turn, increase the

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https://doi.org/10.24200/amecj.v8.i04.1062

viscosity of the frying oil [5]. FFAs affect the quality of the oil and can give it an undesirable flavor. In addition, they are involved in the generation of toxic oxygenated species. The breaking of the long chain of saturated FFAs into smaller fractions and the oxidation of these FFAs produce poisonous substances such as alkenes, ketones, aldehydes, and short-chain FFAs [6]. Likewise, the level of peroxide in oil can be a measure of oxidation and rancidity, which are negative factors for food quality. During heating, the peroxide content in WCO increases, further decreasing its safety and usability [7]. The health risks associated with reusing waste cooking oil (WCO) are enormous because lipid degradation increases the oil's acidity and gives it an unpleasant smell. Inadequate management of WCOs worsens environmental and structural issues, including sewer blockages, wastewater surges, damage to expensive infrastructure, pest and insect infestations, foul odors, and higher operating costs of centralized wastewater treatment plants [8]. When WCOs are disposed of in municipal waste dumps, they cause soil pollution [9]. Reports of illegal dealers who collect and resell WCOs as regular cooking oil are even more troubling, putting public health and food safety at grave risk. These practices not only affect the quality of the food available but are also very dangerous to consumers' health. One rational approach to these problems is to refine WCO into reusable, high-quality oil for non-food uses, such as biodiesel production. The majority of studies seek to identify methods to efficiently remove free fatty acids (FFAs) and achieve a minimal peroxide value (PV) in waste cooking oil (WCO). Some of the conventional techniques utilized for this purpose include turbidity-based shaking operations [10], FT-IR analysis, Raman spectroscopy [11], and high-performance liquid chromatography (HPLC) [12, 13]. While these techniques might provide the required results, applying them on a large scale is not feasible at times [14]. Their disadvantages include a slow operating rate, extensive use of solvents, dependence on qualified laboratory personnel, high costs, and relatively low effectiveness in removing toxic compounds. However, adsorption has been considered more effective and feasible compared to other methods [15]. This technique is appraised for its simplicity, easy design, relatively low cost, and satisfactory performance with minimal oil loss. Due to its operational benefits, it can be considered a viable option for tackling issues associated with the removal of FFA and PV in waste cooking oil [16, 17]. Several adsorbents have been investigated for the purification of waste cooking oil (WCO), such as TiO_2/chitosan composite fibers [18], oil palm boiler ash (OPBA) [19], ionic liquids [20], zeolite, magnesium silicate, China clay, kaolinite clay, bamboo charcoal, Fuller's earth, Cassava peels [21], Cameroonian clay [22], and rice husk ash. Other popular adsorbents, including activated carbon, silica gel, and magnetite nanocomposites, have also been employed to reduce free fatty acids (FFAs) and peroxide content (PV) in WCO. Despite the effectiveness of many of these adsorbents, their high cost often limits their practical application. This challenge has led to increased interest in low-cost alternatives derived from natural resources, agricultural waste, and industrial by-products [23]. These materials, in their natural form or after processing, provide a cost-effective and environmentally friendly solution for WCO purification. Fuller's earth and China clay (kaolinite, $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$) are among the most effective adsorbents available since they hold beneficial properties. They possess a high surface-to-volume ratio [24], and good porosity, which makes the adsorption process efficient [25]. Further, they can be altered in terms of their physical and chemical characteristics to meet specific applications [26], and they are relatively inexpensive due to their abundance. Research has shown that incorporating Fuller's earth with China clay improves the porosity of the final product, thereby increasing the adsorption of free fatty acids (FFAs), lowering the peroxide value (PV), and removing color impurities and dyes. Also, many nanoadsorbents were characterized by FTIR, SEM, and XRD for the extraction or treatment of organic materials [27, 28]. This combined method is effective for improving adsorption capacity and simultaneously eliminating more impurities. Not only is FECC cheaper and convenient to employ, but it is also reusable across multiple cycles and can remove the adsorbed impurities without compromising the framework. These qualities make FECC the most appropriate and sustainable

medium for oil purification, particularly for large-scale industrial applications. In addition, to increase the clay material's number of active adsorption sites, activation via acidic or basic treatments is necessary. Different modification methods have been studied, including the use of inorganic acids, organic acids, inorganic bases, salts, and heat treatments [29]. New acidic surface sites are readily generated by treating clay with inorganic acids such as H_2SO_4 or HCl, thereby significantly enhancing its adsorption capacity. However, this method has the disadvantage that Al_2O_3 in the clay tends to leach out, reducing the overall strength of the clay. On the other hand, organic acids offer a promising solution to the problem. They improve the adsorption capacity of the clay and maintain its skeleton structure, making them a more optimal and less destructive option for clay activation [30].

This study introduces a novel, efficient, and environmentally friendly bio-adsorbent, FECC, for the purification of waste cooking oil by removing FFAs and Peroxide values. The activation process employed sodium hydroxide and acetic acid, altering the surface chemistry of FECC by adding more active sites and increasing its surface area, thereby enabling it to adsorb more impurities present in the WCO. The activated FECC displayed several improvements, including higher porosity, a more suitable surface-to-volume ratio, a more suitable pore size, and a more uniform pore distribution. The activation also enhanced FECC's adsorption capacity and demonstrated its viability as a low-cost, effective material for oil purification. To evaluate the effectiveness of FECC, advanced characterization techniques, including FTIR, XRD, BET, and FESEM, were used to assess the removal of FFAs and other impurities. The adsorption features were also investigated during the study, demonstrating the role of FECC functional groups in binding and FFA removal.

2. Materials and Methods

2.1. Materials and Reagents

Fuller's earth and China clay are the primary raw materials used in this work, procured locally in Pakistan. Waste cooking oil was obtained from the local market, Sher Shah Lahore. Acetic acid 99% (CH_3COOH, CAS Number: 64-19-7), Sodium hydroxide 87% (NaOH, CAS Number: 1310-73-2), and Ethanol 100% (C_2H_5OH, CAS Number: 64-17-5) were acquired from Sigma-Aldrich. Phenolphthalein indicator (CAS Number: 77-09-8), Starch solution (Laboratory grade, CAS Number: 9005-25-8), and Chloroform (CAS Number: 67-66-3) were obtained from Fisher Chemicals. Sodium Thiosulphate (CAS Number: 7772-98-7) was obtained from Acros Organics. All chemicals used had 99.9% purity and were kept according to MSDS instructions. Since all the compounds were of analytical variety, additional purification was unnecessary. All aqueous solutions were made using ultrapure distilled water from the Millipore System.

2.2. Synthesis of Fuller's Earth-China Clay

Four composite samples were prepared using the co-precipitation method by mixing China clay and fuller's earth in varying ratios of 2:8, 4:6, 5:5, and 8:2, respectively.

2.2.1.Preparation of base-activated adsorbent

To make the surface of the adsorbent more active for adsorption, FECC was modified with base NaOH [31]. Briefly, 4.0 g of China clay and 6g of Fuller's earth material were mixed well in 100 mL of deionized water under vigorous stirring for 30 minutes, followed by adding NaOH pellets to maintain the basic PH (13) of the sample solution. Further, the reaction was permitted to continue for 8 hours at 50 °C under vigorous stirring. The sample was purified by washing with distilled water and drying at 100 °C in an oven. Fine powder was obtained after calcining at 900 °C for 5.0 hours in a muffle furnace and stored in a bottle for further use.

2.2.2. Preparation of acid-activated adsorbent

To make the surface of the adsorbent more active for adsorption, FECC was modified with acetic acid [31]. Briefly, 4.0 g of China clay and 6.0 g of Fuller's earth material were mixed well in 100 mL of deionized water under vigorous stirring for 30 minutes, followed by adding acetic acid drop by drop to maintain an

acidic PH (4.6) of the sample solution. Further, the reaction was permitted to continue for 8 hours at 50 °C under vigorous stirring. The sample was purified by washing with distilled water and drying at 100 °C in an oven. A fine powder was obtained after calcination at 900 °C for 5 hours in a muffle furnace and stored in a bottle for further use.

2.3. Procedure for analysis of peroxide value (PV)

A 10 g sample of oil was transferred to a 500 mL Erlenmeyer flask containing 60 mL of solvent consisting of 60% acetic acid and 40% chloroform. Then, 0.5 mL of a saturated KI solution was added, and the mixture was shaken continuously before standing for 2.0 minutes. After this, 30 mL of distilled water was added, followed by 0.5 mL of a 1% starch solution. Titration of this mixture was carried out in the presence of 0.01 N $Na_2S_2O_3$ until the blue color disappeared. The peroxide content was calculated by using Equation 1 [32]. Also, the results are validated using UV-Visible Spectrophotometry. These colorimetric methods involve peroxides oxidizing Fe(II) to Fe(III), which then reacts with a dye to form a colored complex that UV-Visible Spectrophotometry measures (Schema 1).

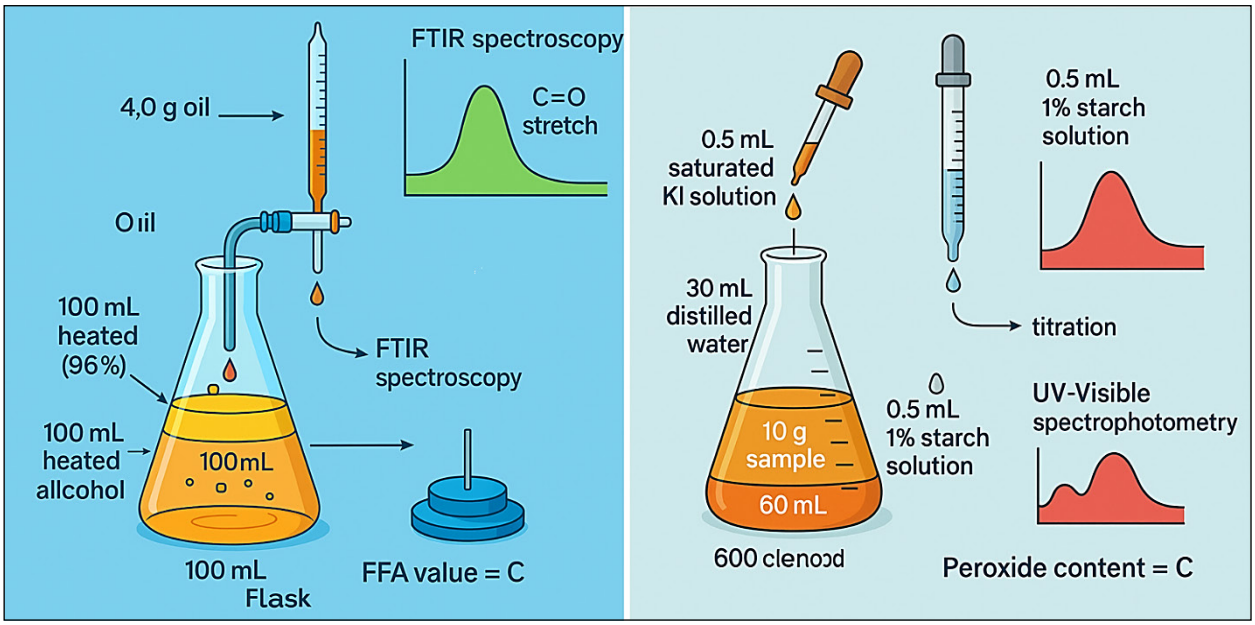
$$\text{Meq/1000 (grams)} = \frac{A\,x\,N}{G} \times 1000 \quad \text{(Eq.1)}$$

2.4. Procedure for analysis of free fatty acid (FFA)

4.0 g of oil was transferred to a 500 mL Erlenmeyer flask containing 100 mL of heated (96%) alcohol. Eight drops of indicator phenolphthalein were introduced. Titration of this mixture was carried out in the presence of 0.01 N sodium hydroxide, by constant agitation, until a persistent pink color showed and remained stable for 30 seconds [33] (Schema 1). FFA value was calculated using Equation 2. In addition, Fourier Transform Infrared (FTIR) Spectroscopy measures the absorption of infrared light at specific wavelengths related to the functional groups of FFAs (C=OH band at 1710 cm^{-1}).

$$\text{Free Fatty Acid} = \frac{25.6\,x\,V\,x\,N}{W} \quad \text{(Eq.2)}$$

Where, V = Volume of NaOH solution needed in the titration (mL), N = Normality of NaOH (N), and W = Sample weight (g).



Schema 1. Procedure for analysis of free fatty acid (FFA) and of peroxide value (PV)

2.5. Characterizations

The samples were characterized using FT-IR, XRD, BET, and FESEM analysis. Fourier-transform infrared (FT-IR) spectroscopy was performed on a Thermo Nicolet 6700 spectrometer in photoacoustic mode to identify functional groups. The analysis was conducted at a resolution of 8 cm^{-1} over the wavenumber range 400–4000 cm^{-1}. The crystal structure of the material was determined using an X-ray diffraction (XRD) instrument, specifically the PANalytical X'Pert Powder Diffractometer. To evaluate the surface area and porosity of the adsorbent, Brunauer–Emmett–Teller (BET) analysis was performed using an Autosorb iQ-C-MP-AG 2 Station Viton (Quantachrome, USA). The morphology of the adsorbents was examined using Field Emission Scanning Electron Microscopy (FE-SEM) with an Apreo S microscope (Thermo Fisher Scientific, USA).

3. Result and discussion

3.1. FT-IR Analysis of Pristine Fuller's Earth and China Clay

The FT-IR analysis was conducted on pristine fuller's earth and China clay materials to identify the functional groups present in the raw materials, as illustrated in Figures 1 and 2 [34]. The prominent peaks observed at 3445 cm^{-1} and 1635.8 cm^{-1} are attributed to the O–H stretching and bending vibrations of surface-adsorbed water molecules Figure 1. A peak at 974 cm^{-1} corresponds to the stretching vibrations of tetrahedral Si–O–Si bonds [35], which also indicates inner surface Al–OH deformation, confirming the presence of aluminum hydroxyl groups on the surface [36]. Additionally, the peak at 796.6 cm^{-1} is associated with Si–O or siloxane (Si–O–Si) bonds, indicating the presence of quartz [37]. The peaks at 3618.3 cm^{-1} and 3704 cm^{-1} are attributed to O–H stretching vibrations of Al–O–H and Mg–O–H–Al groups, respectively, further supporting the presence of aluminum and magnesium hydroxyl groups in the material structure [35].

The FT-IR spectrum of raw China clay is presented in Figure 2. The absorption bands at 3619.6 cm^{-1} and 3680 cm^{-1} indicate the presence of O–H groups trapped within the crystal lattice of China clay. These O–H stretching vibrations suggest structural disorder in the material [38]. Notably, the doublet observed at 3620 cm^{-1} and 3680 cm^{-1} can sometimes appear as a single broad band around 3660 cm^{-1} [39]. The intensity ratio of the bands at 3620 cm^{-1} and 3680 cm^{-1}

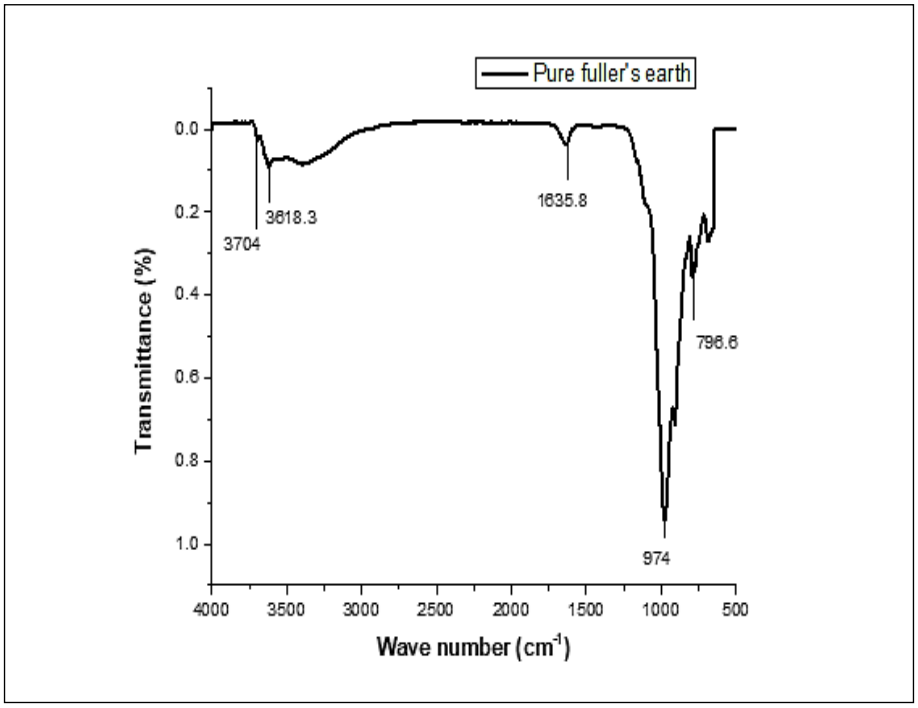


Fig. 1. FT-IR spectra of raw fuller's earth material

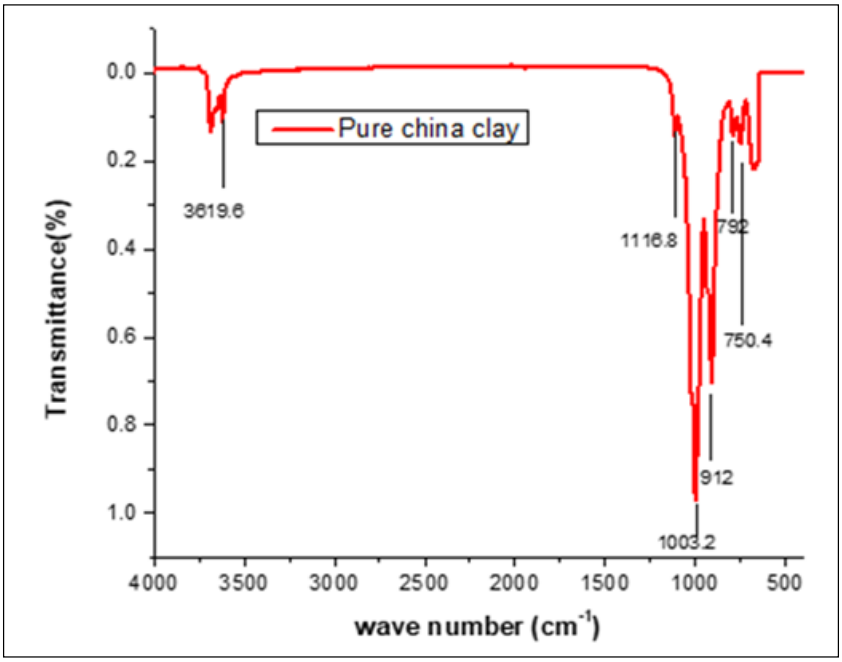


Fig. 2. FTIR of pure China clay material

serves as a quantitative measure of the degree of order or disorder in the kaolinite structure [40]. The peaks at 1003.2 cm^{-1} and 1116.8 cm^{-1} are characteristic of Si–O stretching vibrations and are indicative of amorphous silica. Additionally, the peak at 912 cm^{-1} corresponds to the O–H stretching vibrations bound to Al–O–H groups. Absorption bands observed in the range of 750.4 to 792 cm^{-1} are attributed to Al–O–Si groups, in agreement with the literature [41].

3.2. FT-IR analysis of activated Fuller's earth and China clay

The FT-IR spectra of NaOH-activated Fuller's earth-China clay (FECC) are shown in Figure 3. The spectrum shows that the bands associated with O–H groups in the raw clay completely disappear after activation [40]. A distinct peak at 1445 cm^{-1} is observed in all FECC samples prepared in the ratios of 2:8, 4:6, 5:5, and 8:2 (China clay to Fuller's earth). This peak corresponds to the Si–O–Si complex, which is critical for the interaction of the modified FECC material with free fatty acids (FFAs) in waste cooking oils (WCOs). With an increasing proportion of Fuller's earth, the formation of the Si–O–Si complex is enhanced, thereby improving the removal efficiency of FFAs from WCOs. However, optimizing the ratio of China clay to Fuller's earth is essential to achieve the best results. Among the tested ratios, the 4:6 ratio demonstrates the highest peak intensity at 1445 cm^{-1}, indicating the maximum concentration of the Si–O–Si complex. This optimized ratio was used in all subsequent experiments due to its superior adsorbent properties. Additionally, the presence of Fe^{3+}–OH–Mg groups is confirmed by absorption peaks in the range of 880.11 cm^{-1} to 1063 cm^{-1} [42]. FTIR of O-H and -COOH groups was seen in other compounds [43]. XRD and FT-IR analyses corroborate the successful conversion of Fuller's earth-China clay (FECC) into reactive meta-FECC after treatment [38].

The FT-IR spectra of acetic acid-activated Fuller's earth-China clay (FECC) are presented in Figure 4. The spectrum shows that the peak positions differ from those in the base treatment, indicating changes in the material's structure after acid activation. The peak intensities are also lower than those of raw clay, indicating that the functional groups have changed during the treatment. A distinct peak at 1443 cm^{-1} in the samples with ratios of 2:8, 4:6, 5:5, and 8:2 (China clay to Fuller's earth) confirms the formation of silanol (Si-OH) groups, which highlights the impact of the acid treatment. Furthermore, the bands observed at 881 cm^{-1} and 1066 cm^{-1} are assigned to the stretching vibrations of silanol groups [37]. Among the tested ratios, the 4:6 ratio (sample B) exhibited the

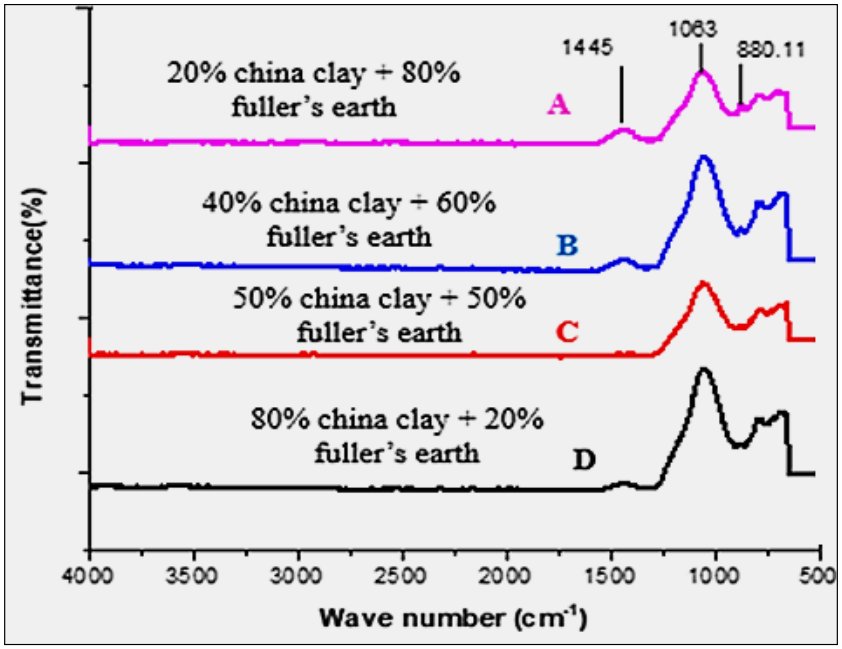


Fig. 3. FTIR spectra of base-treated Fuller's earth-China clay (FECC) material

highest peak intensity. This result corresponds with those obtained with base modification, suggesting that the 4:6 ratio is the most effective for removing free fatty acids (FFAs) from used cooking oil. Based on these findings, this ratio was selected for further experiments throughout the whole study.

3.3. XRD analysis of activated Fuller's earth and China clay

The X-ray diffraction (XRD) technique is used to determine the internal structure of materials and to identify crystalline phases or chemical components [44]. The X-ray diffraction pattern of the chemically

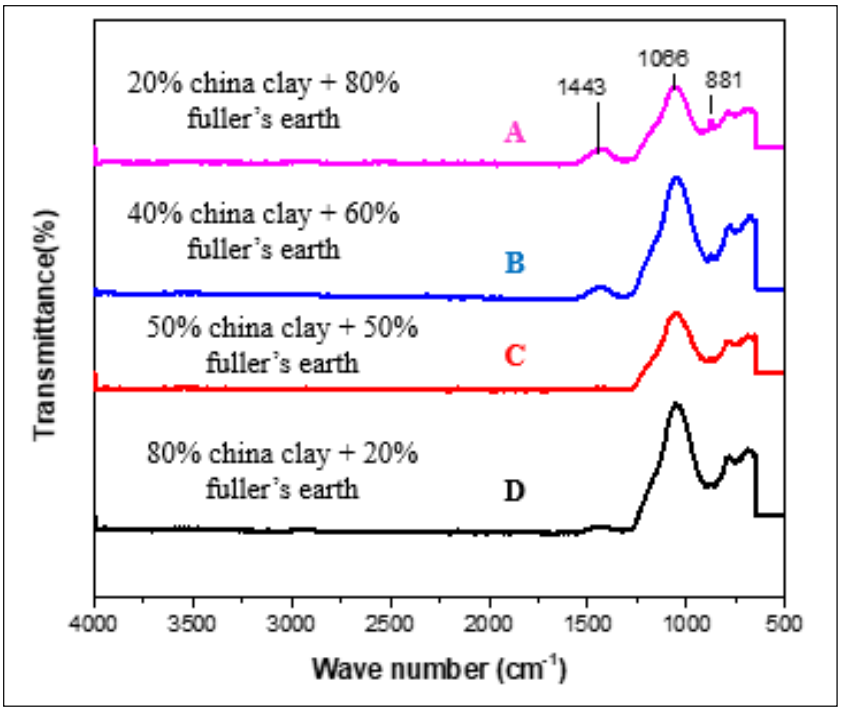


Fig. 4. FTIR spectra of acid-treated Fuller's earth-China clay (FECC) adsorbent material

activated FECC is presented in Figures 5 and 6, where the crystallinity of FECC is presented. The XRD pattern of base-modified FECC with concentration B shows the highest-intensity peaks, confirming it as the best-performing sample and consistent with the FT-IR results. The diffraction peak at 22º confirms the presence of quartz (SiO_2), while the peak at 40.7º corresponds to tetragonal MgO. Additionally, peaks at 29.48º and 26.26º indicate the face-centered cubic crystal structure of K_2O, with a characteristic crystalline

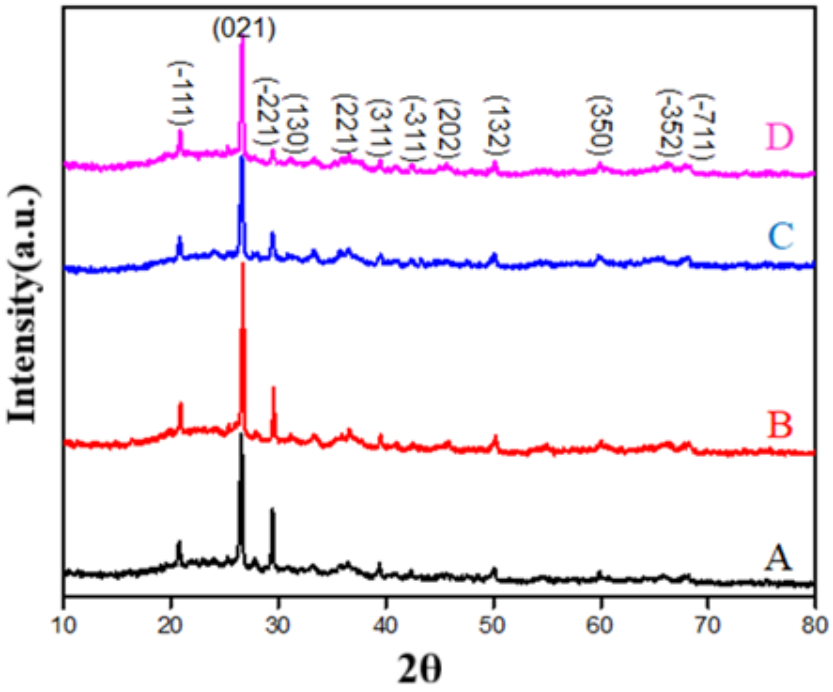


Fig. 5. XRD of base-modified (FECC) adsorbent

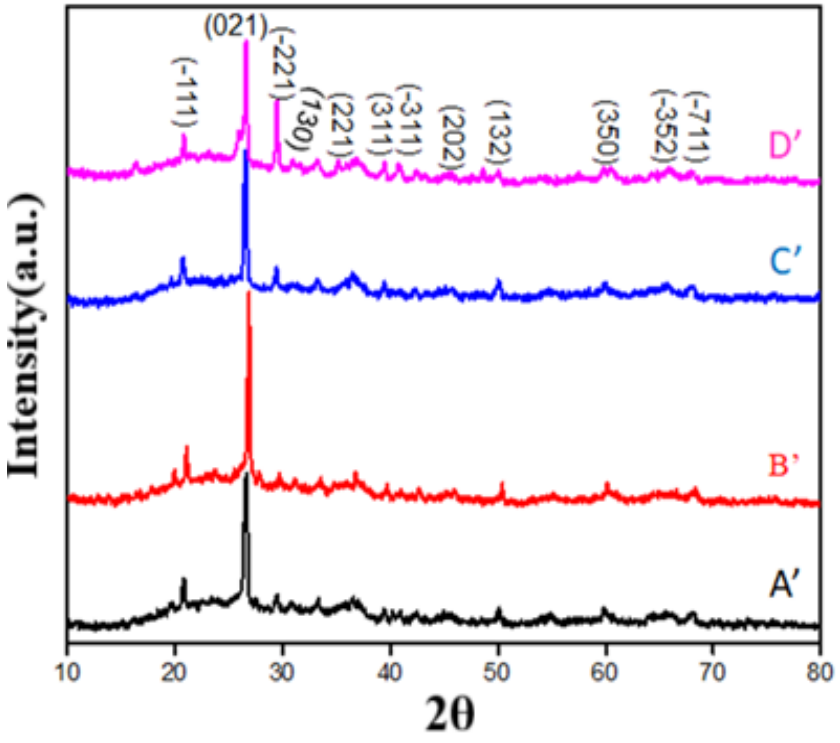


Fig. 6. XRD of acid-modified FECC adsorbent

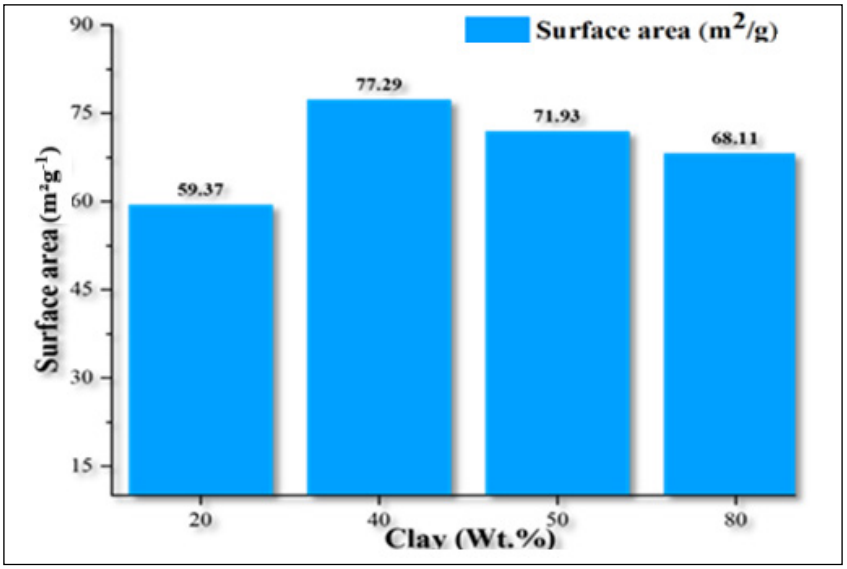


Fig. 7. BET of activated Fuller's earth-China clay (FECC) adsorbent

peak at 38.6º. After activation with NaOH, the peak intensities increased and the peaks narrowed, suggesting enhanced crystallinity. This change is attributed to an increase in crystallite size and/or a reduction in mean lattice strain, which intensifies and narrows the peaks. The observed XRD patterns are consistent with results from previous studies. This optimized concentration (B) was utilized in all subsequent experiments due to its superior performance [45-47]. The XRD spectra of FECC material modified with acetic acid are shown in Figure 6. The prominent peak observed in the region 26° < 2θ >33° corresponds to the formation of an amorphous alumino-silicate phase in the FECC material [48]. A significant presence of quartz was identified, with the primary minerals of the phyllosilicate group evident at 21.88º. Due to the severe leaching caused by strong acids, the peak intensities decreased after acid treatment. This reduction in intensity can be attributed to structural abnormalities induced by acid leaching, which negatively affect the clay's crystalline properties. The loss of crystallinity is a direct consequence of the disruption in the material's structural integrity during acid activation.

3.4. BET analysis of raw and activated Fuller's earth-China clay

The BET surface areas of FECC materials were determined using nitrogen physisorption, as shown in Figure 7. The specific surface area of the adsorbent was calculated using the BET equation. Before forming the FECC composite, Fuller's earth exhibited a surface area of 25 m^2g^{-1}, while China clay had a surface area of 13.52 m^2g^{-1}. After formation and chemical activation of FECC, the modified adsorbent materials displayed varying surface areas, and both acidic and alkaline treatments significantly increased the surface area and pore volume of FECC. This enhancement is primarily due to the de-alumination process, which causes the material to structurally break down. Acid activation, in particular, results in the formation of finely dispersed silica oxides, the removal of cations from interlayer spaces, the creation of surface pores and cracks, and a reduction in mineral size, each contributing to increased pore volume. Similarly, the alkaline treatment promotes pore enlargement and the accumulation of magnesium and calcium hydroxides, further increasing the surface area. The increase in pore volume and surface area is especially apparent when comparing treated FECC to untreated samples. Previous studies have shown a direct relationship between clay surface area and adsorption capacity [49]. As shown in Figure 7, adsorbent A had a surface area of 59.37 m^2g^{-1}, B had 77.29 m^2g^{-1}, C had 71.93 m^2g^{-1}, and D had 68.11 m^2g^{-1}. Adsorbent B demonstrated the highest surface area of 77.29 m^2g^{-1}, reflecting the most significant impact of acid treatment. This result aligns with the FT-IR and XRD analyses, further confirming that adsorbent B has the highest capacity to adsorb toxicants from WCOs [36].

3.5. FESEM analysis

The surface morphology of Fuller's earth-China clay (FECC) was analyzed using FE-SEM, as shown in Figure 8 (a, b**)** at different angles. The micrographs reveal a small, rough, uneven, and irregular surface structure with varying pore sizes. Activation of FECC with sodium hydroxide and acetic acid significantly enhanced pore development on the material's surface. Figure 8(c) shows a highly rough, lake-like structure resembling mud in a stream, characterized by a larger surface area with numerous active sites. This structural roughening enhances adsorption affinity, making the material highly effective for the removal of toxicants, including FFAs and peroxide molecules [36]. The rough, porous surface is indicative of the impact of harsh acidic and basic treatments, which activate the material and create abundant active adsorption sites. The observed structural modifications in the FECC after chemical activation align well with findings from other characterization techniques, confirming the material's suitability for efficient adsorption processes [50]. These changes demonstrate the effectiveness of the activation treatments in enhancing the adsorption capacity of the FECC material.

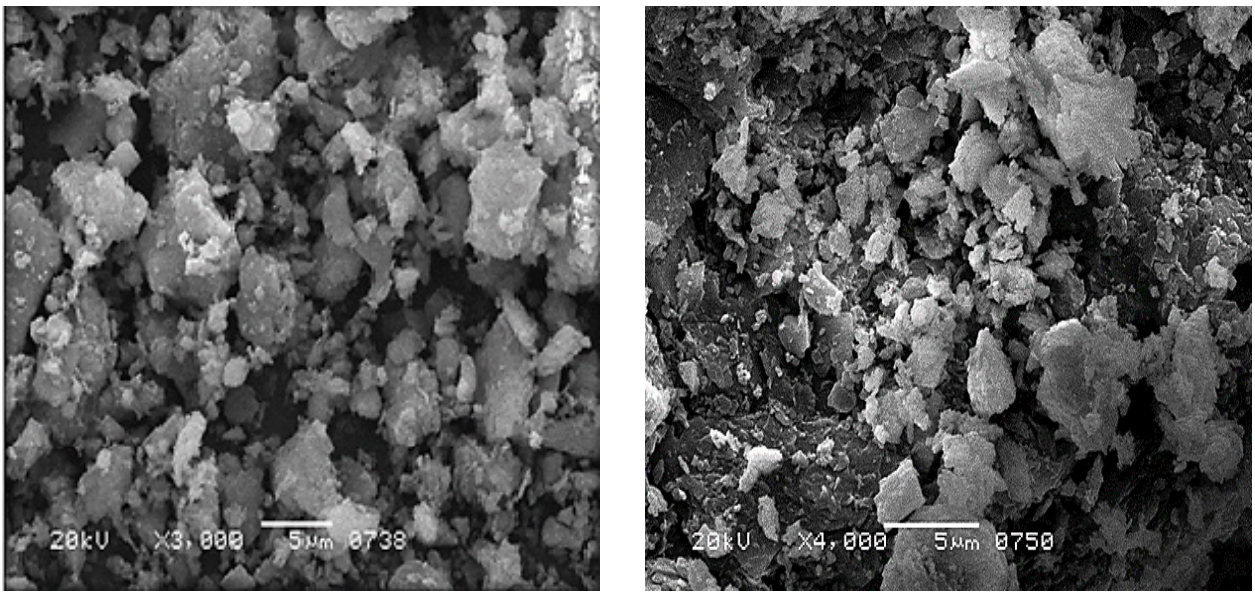


Fig. 8. FESEM of (a) and (b) Fuller's earth-China clay at different angles

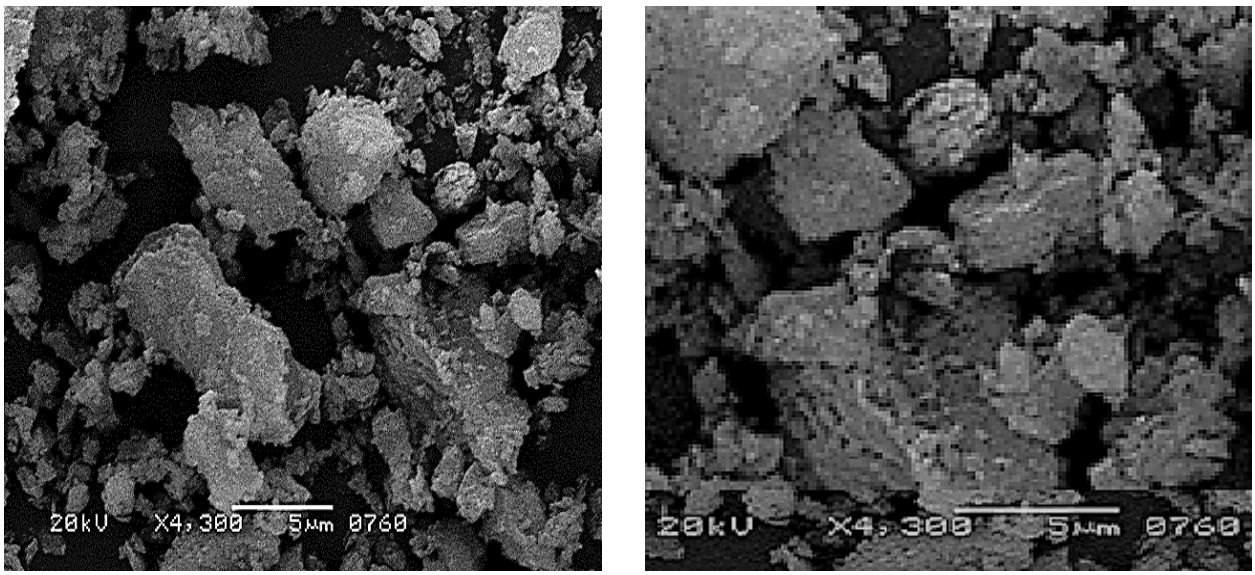


Fig. 8c. FESEM of Fuller's earth-China clay activated by base and acid

3.6. The chemistry behind FFA and PV adsorption

The chemistry behind the free fatty acid and peroxide adsorption on the surface of activated Fuller's earth-China clay (FECC) can be attributed to several factors. Fuller's earth clay is rich in magnesium, aluminum, and silicon, while China clay possesses hydrated aluminum silicate. When the composite of Fuller's earth-China clay (FECC) is treated with NaOH, it reacts with its surface, increasing its porosity and exposing more active sites by removing impurities. It also introduces negative charges (deprotonation) on the surface of silanol groups (−Si−OH) to form −Si−O^-, thereby enhancing the clay's ability to interact with cations or polar molecules [51]. In addition, the composite of Fuller's earth-China clay (FECC) is treated with acetic acid, and it protonates certain sites, balancing the charge distribution and stabilizing specific functional groups while modifying the surface properties to enhance interactions with organic molecules. The combination of these treatments results in a material with a highly porous structure, active functional groups such as −Si−OH, −Al−OH, and −Mg−OH, and increased surface area for adsorption [52].

3.6.1.Mechanism of adsorption for free fatty acids (FFAs)

Free fatty acids are polar molecules with a carboxylic acid group (-COOH) that interact with the clay surface through hydrogen bonding, electrostatic interactions, and van der Waals forces. Due to hydrogen bonding, the acidic hydroxyl groups (−COOH) of FFAs can form hydrogen bonds with the hydroxyl groups (−OH) present on the clay surface (e.g., −Si−OH, −Al−OH). These interactions can be further confirmed by FTIR analysis in Figures 3 and 4 and presented in Equation 3. Also, electrostatic interactions between deprotonated carboxylic acid groups (−COO−) and positively charged sites on the clay surface, such as $-Al^{3+}$ or $-Mg^{2+}$, form ionic bonds during activation (Equation 4). Moreover, in Van der Waals interactions, the hydrocarbon tails of fatty acids interact with the clay surface via weak dispersive forces, further stabilizing adsorption.

$$R-COOH + Si-OH \rightarrow R-COO-H\cdots O-Si$$

(Eq.3)

$$R-COO^- + Al^{3+} \rightarrow [R-COO\cdots Al]^{2+}$$

(Eq.4)

3.6.2.Mechanism of adsorption for peroxides

Peroxides in waste cooking oil are typically polar or semi-polar molecules (e.g., hydroperoxides) that interact with clay via hydrogen bonding and electrostatic interactions. Hydroxyl groups on the peroxide molecules form hydrogen bonds with the hydroxyl or silanol groups on the clay surface, as shown in Equation 5. By electrostatic interactions, peroxide compounds that contain anionic forms (e.g., −O−O−) interact with positively charged sites, such as $-Al^{3+}$. Once adsorbed, the FFAs and peroxides are immobilized on the surface of Fuller's earth-China clay (FECC), effectively removing them from waste cooking oil (WCO). Purified cooking oil (PCO) has reduced acidity and oxidative products, improving stability and quality. This process combines physical adsorption (surface interaction) and chemical adsorption (specific functional group interaction), making it highly effective for oil purification.

$$ROOH + Si-OH \rightarrow RO-H\cdots O-Si$$

(Eq.5)

3.7. Effect of Adsorbent on PV and FFA values

The peroxide value is a key measure of oil degradation resulting from initial oxidation. High peroxide levels in cooking oil cause rancidity and off-flavors, significantly reducing its quality. Table 1 shows the effect of the FECC adsorbent on WCO peroxide content, with a significant reduction at higher FECC concentrations. The peroxide value decreased from 5.5 meq kg^{-1} to 1.00 meq kg^{-1} (n=3, RSD<2%), demonstrating FECC's effectiveness in adsorbing peroxide from used cooking oil. This decrease is linked to the interaction of the −Si−OH, −Al−OH, and −Mg−OH functional groups in FECC with the free radicals in the reactive peroxide molecules. The

polarity of the peroxide groups in the oil aligns with the polar hydroxyl (-OH) groups on FECC, enabling efficient adsorption. This polarity compatibility enhances the binding of peroxide compounds to the adsorbent. Moreover, FECC contains three active -OH groups that can form bonds with adsorbate components (FFA and peroxides), thereby further facilitating adsorption. These findings highlight FECC's potential as a powerful adsorbent for substantially reducing peroxide content in used cooking oil, thereby improving its quality and stability.

The effect of adsorbent on FFA values is followed as Free fatty acids (FFA%), formed by the hydrolysis of triglycerides, which are a critical parameter for assessing the quality of edible oils. FFAs are produced during lipid deterioration processes such as oil processing, prolonged storage, and poor raw material quality [53]. According to the Codex standard for edible oils, FFA levels must not exceed 0.25%. Maintaining low FFA levels slows down fat breakdown and enhances heat transfer efficiency. Table 1 illustrates the effect of Fuller's earth-China clay (FECC) treatment on the FFA content of waste cooking oil (WCO). The untreated WCO had an FFA level of 2.98%, which decreased to 0.91% after FECC treatment, representing a 69.46% reduction. The RSD% for FFA removal is less than 2%, further demonstrating the precision of our proposed methodology. This outcome highlights FECC's efficiency as an adsorbent, attributed to its large surface area for FFA adsorption. The reduction in FFAs is primarily due to the activated structure of FECC, which contains functional groups such as −Si−OH, −Al−OH, and −Mg−OH formed through alkali and acetic acid treatment [54]. These functional groups interact with the carboxyl groups (−COOH) of free fatty acids via hydrogen bonding. This interaction chemistry enables FECC to effectively capture and retain FFAs, significantly reducing their concentration in the oil [2, 51]. These findings emphasize the potential of FECC as an affordable and efficient solution for improving the quality of used cooking oil.

3.8. Effect of Color

To evaluate the bleaching effectiveness of the three materials, a series of tests was performed under different conditions. In this process, 18 g of waste cooking oil (WCO) was passed through filter paper containing a specific amount of activated FECC material. The filtered oil was then collected in a 30 mL glass beaker, and the color of the treated oil was assessed to determine the success of the bleaching process [55]. The color reduction was quantified using the following Equation 6.

$$\text{Color reduction (\%)} = \frac{\text{Color of WCO} - \text{Color of PCO}}{\text{Color of WCO}} \times 100 \quad \text{(Eq.6)}$$

After filtration, the waste cooking oil (WCO) transformed from a dark brownish-black color to a lighter yellow, indicating successful purification and the removal of impurities.

3.9. Discussion

This study explored the potential of reprocessing and purifying WCO by reducing its free fatty acid (FFA) content using Fuller's earth-China clay (FECC) as an adsorbent. The functionalities and structural characteristics of FECC were analyzed using XRD and FTIR, confirming its suitability for adsorption.

Table 1. The color of used cooking oil before and after treatment with FECC is illustrated, along with the corresponding changes in free fatty acid content and peroxide value

No	Compounds	Raw, Waste Cooking Oil	FECC treated Oil	Reduction (%)
1	FFA content (mg KOH/g)	2.98± 0.11	0.91± 0.04	69.46
2	Peroxide content (meq O_2/kg)	5.5± 0.016	1.0 ± 0.05	81.82
3	Color Fresh cooking oil	2.00 ± 0.06	----	----
4	Color waste cooking oil (WCO)	4.00 ± 0.12	2.00 ± 0.09	----

[a]Adsorbents were used at a concentration of 15% (w/w) relative to the weight of the oil.
[b] Results are presented as mean ± standard deviation (SD) based on three independent measurements.

The purification process demonstrated FECC's high effectiveness in significantly reducing FFAs and improving the overall quality of WCO under various conditions. Specifically, FECC achieved a 69.64% reduction in FFAs and an 81.82% decrease in peroxide values, resulting in notable improvements in the oil's overall oxidation stability and quality. The comparison of the adsorption capacity for free fatty acids (FFA) and peroxide values between the proposed adsorbent material and those reported in the literature is illustrated in the supporting information file (Table 2)**.** References 56-62 are provided in the supplementary file (ESM)**.** The hydroxyl (silanol) groups and numerous reactive sites on FECC's surface were instrumental in adsorbing FFAs and peroxides, as outlined in Table 1. The more pronounced reduction in peroxide content relative to FFAs demonstrates that FECC exhibits a stronger affinity for hydroperoxide because of its polarity. However, FECC does not modify the chemical structure of glycerides or eliminate heat-induced polymers, restricting its capacity to restore WCO for edible purposes. In conclusion, the recycled WCO is appropriate for applications other than food usage, particularly in producing biodiesel. Although FECC substantially boosts the quality of WCO, it's not possible to fully return oil to its initial composition. Consequently, strict compliance with oil processing regulations is essential to safeguard public health and ensure food safety. These regulations define acceptable contaminant thresholds, labeling criteria, and quality parameters, emphasizing the need for compliance to minimize the risks posed by residual free fatty acids and other toxic substances. This study features FECC as an eco-friendly, economical material for macroscale purification of WCO. The method can be effortlessly integrated with existing frying systems with minimal modifications, providing an adaptable approach to improving oil quality while maintaining food security. Nonetheless, the research acknowledges certain limitations of our work. Laboratory conditions cannot entirely replicate the intricacies of industrial environments, and the prolonged impact of successive purification remains uninvestigated. While free fatty acids and peroxides were measured using traditional methods in this study, future research could explore advanced derivatization techniques to improve detection sensitivity and reliability. In conclusion, this work provides a solid background for the sustainable purification of waste cooking oil (WCO). It establishes the feasibility of using Fuller's earth-China clay (FECC) for industrial purposes. Subsequent research could address, improve, and eliminate the deficiencies and constraints observed in the current study when applied to different operational contexts.

4. Conclusions

This study introduces an innovative method for synthesizing a Fuller's Earth-China Clay (FECC) composite using a straightforward co-precipitation process, followed by activation with both base and acid. The approach effectively removes toxicants from waste cooking oils (WCOs). Among the

Table 2. Comparison of the adsorption capacity for free fatty acids (FFA) and peroxide values between the proposed adsorbent material and those reported in the literature

No	Material used	Oil Type	FFA Reduction (%)	Peroxide Reduction (%)	Ref.
1	FECC	Vegetable oil	69.46	81.82	This work
2	Sugarcane bagasse	Sunflower oil	48.06	76.19	[56]
3	AHMSNPs	Soybean, sunflower oil	85.08–96.82	-----	[57]
4	PTA304	Palm oil	95.00	-----	[58]
5	Cassava peel	Waste cooking oil	80.59	-----	[59]
6	$NiFe_2O_4$	Vegetable oil	80.67	-----	[60]
7	IER-A26OH	Peanut oil	25.50	-----	[61]
8	Al-MOF, Zn MOF	Sunflower oil	58.70, 59.80	27.5, 28.3	[62]

AHMSNPs: Ammoniated hollow mesoporous silica NP
PTA304: Pall Tec PTA304 resin
IER-A26OH: Ion exchange resin A26OH
*****56-62 references in supplementary

different composites tested, the 4:6 ratio (referred to as sample B, made from China Clay and Fuller's Earth) showed the most notable improvements in both structure and composition. The activated FECC, a widely available and cost-effective adsorbent, was modified using sodium hydroxide and acetic acid. Using characterization techniques like FT-IR, XRD, and FESEM, the study confirmed the successful synthesis of the activated FECC. FESEM analysis revealed a rough and porous surface, suggesting excellent adsorption capabilities. BET analysis further supported the enhancement of surface area after activation. When tested under optimized conditions for toxicant removal, the activated FECC demonstrated impressive results, reducing free fatty acids (FFAs) from 2.98% to 0.91% (69.46% recovery) and lowering the peroxide value from 5.5 meq/1000 g to 1.0 meq/1000 g (81.82% recovery). In addition to significantly reducing free fatty acids and peroxide levels, the adsorbent was also effective at removing the dark brown and black coloration from waste cooking oil (WCO), restoring 80-85% of the oil to a state suitable for cooking and other uses. These results highlight the strong potential of activated FECC as a cost-effective and sustainable solution for refining WCO in the food industry. The material's reusability further enhances its economic feasibility, making it a viable option for large-scale applications. Looking ahead, future research should aim to optimize the material to boost its adsorption capacity, yield, and reusability, while also evaluating its performance against a broader range of contaminants. By adopting this innovative approach, it is possible to address environmental issues related to improper disposal of WCO, thereby protecting the environment and improving food safety on a larger industrial scale.

5. Acknowledgment

We would like to express gratitude to Dr. Dilshad Hussain, an Assistant Professor at the HEJ Research Institute of Chemistry, ICCBS, University of Karachi, for his invaluable assistance with the SEM and BET sample characterizations. The authors declare no conflict of interest.

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