

Characterization of Clay from Merauke Regency and Its Utilization as an Adsorbent of Free Fatty Acids in the Purification of Waste Cooking Oil

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ABSTRACT

This study investigates the characterization and application of natural clay from Merauke as an adsorbent for the purification of waste cooking oil. The clay was characterized using X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) before and after acid activation. XRD analysis revealed quartz as the dominant mineral, while acid activation caused partial dissolution of layered silicates, leading to structural modifications without affecting quartz stability. SEM images confirmed morphological changes from compact aggregates with limited pores to rougher surfaces with larger and more interconnected pores, indicating increased surface area and porosity. Adsorption experiments demonstrated that both natural and acid-activated clays reduced free fatty acids (FFA) in waste cooking oil, with the acid-activated clay consistently showing higher efficiency. Prolonged stirring times (1, 3, and 5 hours) further enhanced adsorption, though the reduction rate decreased over time as adsorption sites approached saturation. In addition to lowering FFA levels, the treatment improved the visual quality of the oil by reducing discoloration. These findings highlight that acid-activated clay from Merauke possesses promising potential as a low-cost and effective adsorbent for oil purification and broader environmental remediation applications.

Introduction

Merauke, located at the easternmost part of Indonesia, is well known for its abundant natural resources. However, several local potentials remain underutilized, including clay. To date, the use of clay in Merauke has been limited to bricks, construction materials, and handicrafts. Clay itself is a natural silicate mineral with particle sizes smaller than two micrometers, notable water absorption capacity, and relatively high cation exchange capacity. These properties make clay comparable to bentonite and highlight its potential for broader applications across different fields [1], [2].

The characteristics of clay differ depending on its origin, particularly due to variations in impurities and associated metal oxides. To optimize its function, activation is required, which can be performed either physically

through heating or chemically using acids. Chemical activation with hydrochloric acid (HCl) has been proven effective in removing impurities and metal oxides, opening the pores, and enhancing the adsorption capacity of clay. Characterization methods such as X-ray diffraction (XRD) and scanning electron microscopy (SEM) are essential to identify mineral phases and morphological changes resulting from the activation process [3], [4].

One promising application of activated clay is as an adsorbent in the purification of used cooking oil, commonly referred to as "jelantah." In Merauke, many street food vendors reuse cooking oil repeatedly, often without awareness of the potential health risks. Repeated use of cooking oil leads to an increase in free fatty acid content and initiates autoxidation, thermal oxidation, and polymerization reactions, producing harmful

free radicals. Long-term consumption of such degraded oil has been associated with fat deposition in vital organs such as the liver, heart, and arteries [5].

Adsorptive purification of used cooking oil provides an effective approach to mitigating oil degradation, as the application of activated clay reduces free fatty acid (FFA) concentrations, restores physicochemical properties similar to those of fresh oil, and extends usability, thereby enhancing food safety [6], [7]. In this context, the present study characterizes natural and hydrochloric acid-activated clay from Merauke, and evaluates its performance as an adsorbent for FFA removal in used cooking oil. Unlike previous research that primarily examined commercial clays, this study focuses on the utilization of local clay from Merauke, a resource that has not been systematically explored for advanced applications. Beyond characterization, the study further compares the adsorption performance of natural and acid-activated clays under different stirring durations, highlighting their effectiveness in reducing FFA levels and improving oil color. This integrated approach offers new insights into the potential of underutilized local resources to address practical environmental and public health issues.

Method

Material

The materials used in this study included natural clay obtained from Telaga Sari Village, Kurik District, Merauke Regency. HCl 0.25 M (Merck), AgNO₃ 0.1 M (Merck), NaOH 0.1 M (Merck), phenolphthalein indicator, Ethanol 96%, and distilled water. In addition, used cooking oil was collected from local street food vendors in Merauke, to be used as a sample for purification experiment. The instruments used were, Bruker D2 Phaser (XRD), and Phenom Desktop ProXL Scanning Electron Microscopy

Sample Preparation and Activation

A natural clay sample was collected from Telaga Sari Village, Kurik District, Merauke Regency, Indonesia. The sample, characterized by a grayish-black color, was air-dried to reduce moisture content, ground with a mortar and pestle, and sieved through a 250-mesh sieve.

The sieved clay underwent triple washing with distilled water, followed by filtration and oven drying at 80-90 °C. The dried material was ground to a fine powder and stored in a desiccator until use. For chemical activation, 100 g of sieved clay was combined with 500 mL of 0.25 M HCl in a 500 mL round-bottom flask at a 1:5 ratio. The mixture was refluxed on a hot plate with continuous stirring at 200 rpm for 3 hours, then filtered and washed with warm water (60–70 °C) until chloride ions were no longer detected, as confirmed by the absence of a white precipitate after the addition of 0.1 M AgNO₃ solution. The activated clay was oven-dried at 100–120 °C, then reground into a fine powder and stored in a desiccator for subsequent analysis.

Adsorption Experiment

The adsorption experiment was carried out by treating 100 mL of used cooking oil with 5 g of either natural or HCl-activated clay under continuous stirring at 60 °C for different contact times (1, 3, and 5 hours). The mixture was then filtered to separate the adsorbent from the oil, and the purified oil was subjected to further quality analysis. The free fatty acid (FFA) content of the oil was determined following the method of Asihta (2017) with slight modifications, in which 2.5 g (2.9 mL) of oil was mixed with 5 mL of 96% ethanol, heated for 1–2 minutes at a temperature not exceeding 70 °C, and allowed to cool before adding three drops of phenolphthalein indicator. The solution was titrated with 0.1 M NaOH until a stable pink color appeared for 30 seconds, and the NaOH volume consumed was recorded to calculate the FFA percentage. In addition, color analysis was conducted qualitatively using visual observation by comparing the color of untreated used cooking oil with that of oil adsorbed using non-activated clay (1 hour of stirring) and acid-activated clay at different stirring times (3 and 5 hours). The adsorption process was repeated three times.

Result and Discussion

The sample preparation of natural clay in this study involved several sequential steps. Clay samples were randomly collected from paddy field areas and air-dried to reduce

moisture content. The dry material was then finely ground and sieved using a 250-mesh sieve to decrease particle size and increase the adsorption surface area. The sieved clay was washed three times with distilled water to remove impurities, filtered, and oven-dried at a moderate temperature range of 80–90 °C for 30–60 minutes. This controlled drying process was chosen to eliminate residual water while preserving the clay's physical and chemical structure, yielding a fine, clean powder suitable for activation and characterization. The sieving and washing steps are critical in enhancing surface activity and reducing contaminants, directly improving adsorption efficiency. Additionally, moderate temperature drying is essential to prevent structural degradation—a precaution aligned with recent findings on clay activation and preservation of adsorption properties [8], [9]

Characterization of Clay

The mineral composition of the natural clay sample was analyzed using X-ray diffraction (XRD). The identification of minerals was carried out by observing the highest diffraction peaks in the XRD diffractogram and subsequently comparing them with the standard data of interplanar spacing (d , Å) and mineral types as presented in Table 1.

Table 1. Standard d (Å) Data of X-Ray Diffraction and Types of Minerals [10]

No.	d (Å)	Jenis Mineral
1.	1,31 – 1,36	<i>Kaolinite</i>
2.	1,53	<i>Vermiculite, Illite</i>
3.	1,99 – 2,00	<i>Mica, Illite, Kaolinite, Chlorite</i>
4.	2,89 – 2,90	<i>Carbonate</i>
5.	3,32 – 3,35	<i>Illite</i>
6.	3,67 – 3,78	<i>Feldspar</i>
7.	3,85 – 4,04	<i>Feldspar</i>
8.	4,26	<i>Quartz</i>
9.	4,36 – 4,46	<i>Kaolinite, Illite, Sepiolite</i>
10.	4,90 – 5,00	<i>Illite, Mica</i>
11.	6,44	<i>Attapulgite</i>

The XRD characterization of the natural clay sample prior to activation, within the 2θ region, produced the diffractogram shown below :

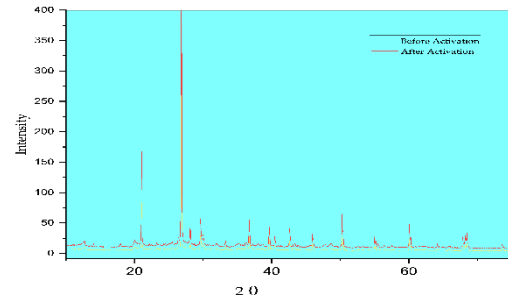


Figure 1. XRD diffractogram of clay before and after acid activation.

The diffractogram of natural clay before activation exhibited multiple sharp peaks, with the most intense at $2\theta \approx 26.6^\circ$, corresponding to quartz. Additional peaks were observed around $2\theta \approx 12\text{--}14^\circ$ and 20° , which can be attributed to montmorillonite and kaolinite. The presence of illite and feldspar was also confirmed by smaller peaks in the range of $2\theta \approx 30\text{--}40^\circ$, consistent with the standard diffraction data [11]. This indicates that the natural clay sample consists of a mixture of silicate minerals with quartz as the primary phase.

After acid activation, the overall diffractogram showed a significant reduction in the intensity of several peaks associated with layered silicates such as montmorillonite, kaolinite, and illite. This reduction implies partial destruction of the aluminosilicate framework due to leaching of Al^{3+} , Mg^{2+} , and Fe^{3+} cations from the octahedral sheets. In contrast, the dominant quartz peak remained stable and unchanged, demonstrating its resistance to acid treatment. The observed changes in the diffractogram strongly support the conclusion that acid activation selectively alters clay minerals while leaving quartz intact [12].

When comparing both diffractograms, the natural clay sample exhibited more defined peaks of layered minerals, while the acid-activated clay showed simplified patterns dominated by quartz with reduced background signals. This structural transformation is beneficial because it enhances surface porosity and increases the number of active adsorption sites. Such modifications significantly improve the performance of clay as an adsorbent material, especially for the removal of

impurities in applications such as waste oil purification [13].

XRD analysis confirmed quartz as the dominant phase, with the main peak at $2\theta \approx 26.6^\circ$. Semi-quantitative estimation using relative peak intensities suggested that quartz accounted for approximately 55–60% of the total crystalline phases, while montmorillonite, kaolinite, and illite contributed about 15–20%, 10–15%, and 5–10%, respectively. After acid activation, the intensity of layered silicate peaks (montmorillonite, kaolinite, illite) decreased significantly, indicating partial dissolution of the octahedral layers, while quartz remained stable.

In addition to XRD analysis, the clay samples were further characterized using Scanning Electron Microscopy (SEM). The SEM micrographs are presented in the figure below.

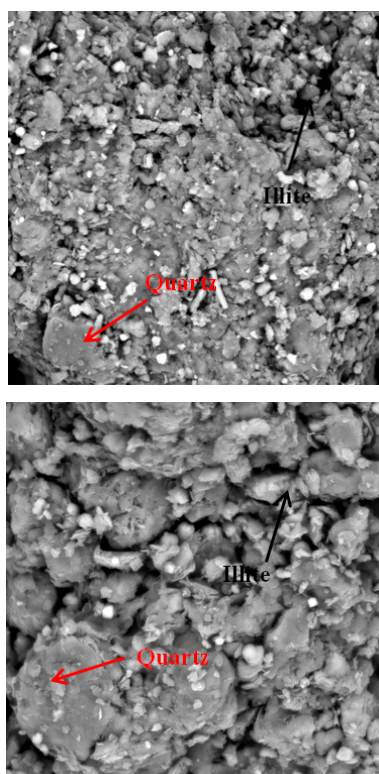


Figure 2. Scanning Electron Microscopy (SEM) images of clay samples prior to acid activation (top) and after acid activation (bottom).

The SEM micrograph of the natural clay sample before activation shows a compact and heterogeneous morphology, with agglomerated particles and irregular surfaces. The presence of dense aggregates and limited pore structures

suggests that the clay still contains impurities and unmodified mineral phases, such as feldspar and quartz, that contribute to the compact texture. Similar observations have been reported in natural clays where the surface tends to be less porous due to the presence of interparticle cementing agents and organic or inorganic contaminants [14], [15].

Following acid activation with hydrochloric acid (HCl), SEM images reveal a pronounced morphological transformation. The clay surface appears rougher and more fragmented, with larger and more interconnected pores compared to its natural state. This alteration is attributed to the partial dissolution of octahedral cations (Al^{3+} , Mg^{2+} , Fe^{3+}) and the removal of impurities during acid treatment, which collectively enhance porosity and surface roughness. Similar findings have been reported in previous studies, demonstrating that acid activation significantly improves pore accessibility and generates additional active sites for adsorption [16], [17], [18].

Adsorption Of Free Fatty Acids From Used Cooking Oil

Waste cooking oil samples were randomly collected from street food vendors (PKL) located along Parakomando Street, Merauke Regency. This sampling site was selected due to the high density of fried food vendors and the large volume of consumer activity in the area. The samples were obtained at night, coinciding with the vendors' closing time, when the oil had typically been reused for approximately five to six frying cycles.

Adsorption experiments were conducted on the collected waste cooking oil using natural clay adsorbents in two forms: untreated (raw clay) and acid-activated clay. Both adsorbents were tested under different stirring durations of 1 hour, 3 hours, and 5 hours. Each experimental condition was performed in triplicate to ensure data reliability and reproducibility.

The results of adsorption on the free fatty acid content can be seen in the graph below:

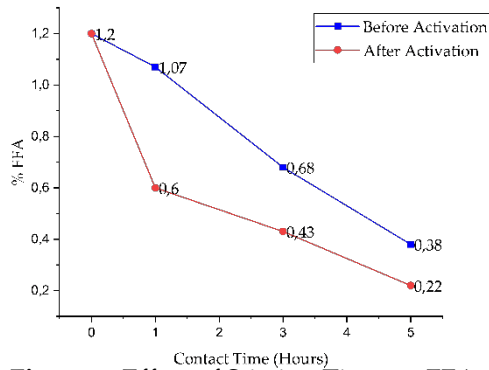


Figure 3. Effect of Stirring Time on FFA Reduction

At the initial stage of the adsorption experiments, both natural and acid-activated clays exhibited relatively high free fatty acid (FFA) values, ranging from approximately 1.1% to 1.2%. After one hour of stirring, a marked reduction in FFA content was observed, with the acid-activated clay showing a greater decrease than the natural clay. This superior performance can be attributed to the acid activation process, which enhances adsorption capacity by increasing porosity and surface activity through the removal of impurities and the partial leaching of structural cations. Previous studies have demonstrated that acid treatment modifies the surface area and adsorption characteristics of clay minerals, thereby improving their ability to adsorb various organic compounds [19], [20].

As the stirring duration was extended to 3 and 5 hours, both adsorbents continued to reduce FFA concentrations; however, the rate of reduction declined over time, indicating that the adsorption sites were approaching saturation. This observation aligns with established adsorption kinetics, in which a rapid initial uptake is followed by a slower phase as the number of available active sites decreases [21]. At all tested durations, acid-activated clay consistently exhibited lower FFA values than natural clay, confirming its greater adsorption capacity. The structural modifications induced by acid activation are known to increase the availability of active sites, thereby facilitating more effective interactions between the clay surface and free fatty acids as well as other impurities present in waste cooking oil [22].

These results are consistent with broader findings on the performance of chemically

activated clays, which have repeatedly been shown to possess superior adsorption properties compared to untreated clays. Acid activation not only increases the specific surface area but also expands the number of accessible adsorption sites, significantly enhancing contaminant removal efficiency [20], [23]. Furthermore, recent studies have confirmed that acid treatment improves clay porosity and alters surface chemistry, which positively affects both adsorption kinetics and thermodynamic behavior.

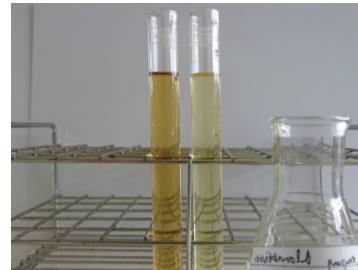


Figure 4. Color changes of waste cooking oil before (left) and after (right) the adsorption process.

Figure 3 shows the color changes in waste cooking oil before and after adsorption using clay adsorbents. The untreated oil exhibits a darker brownish-yellow color, indicating the presence of free fatty acids and degradation products, while the treated oil appears lighter and clearer. This visual improvement is consistent with the SEM results, which revealed enhanced porosity and surface roughness of acid-activated clay, leading to increased adsorption capacity. The observed decolorization also aligns with the reduction in FFA values, confirming the effectiveness of acid-activated clay in improving both the chemical quality and physical appearance of waste cooking oil.

Several studies have characterized clays from Java, Sumatra, and Sulawesi, revealing diverse mineral compositions and applications ranging from ceramics to geotechnical and industrial uses. However, clays from Merauke, Papua, remain underexplored despite the region's unique geological setting and abundant natural resources. This study addresses that gap by characterizing Merauke clay using XRD and SEM, and by evaluating its potential as a low-cost adsorbent for the purification of used cooking oil, thereby linking

local resource utilization to practical environmental and public health solutions [24], [25], [26].

Conclusion

This study demonstrates that natural clay from Merauke, when activated with hydrochloric acid, undergoes significant structural and functional improvements that enhance its adsorption capacity. XRD analysis revealed that acid activation partially modified layered silicate minerals while maintaining quartz stability, while SEM observations confirmed the development of a rougher surface with more open and interconnected pores. These structural modifications translated into improved adsorption performance, as evidenced by the substantial reduction in free fatty acid (FFA) levels, particularly when acid-activated clay was applied under extended stirring times. Furthermore, the visual improvement in oil color after adsorption highlights the practical effectiveness of acid-activated clay in enhancing both the chemical quality and physical appearance of waste cooking oil.

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