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Application of Calcined Clamshells as Natural Adsorbents in the Refining of Used Cooking Oil

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Abstract

The disposal of waste cooking oil (*WCO*) remains a critical environmental concern due to its potential to pollute water sources and contribute to greenhouse gas emissions. This study investigates the use of clamshell-derived bleaching earth as a sustainable adsorbent for the refining of *WCO*. The objectives of the research are threefold: (1) to produce bleaching earth from clamshells through thermal and chemical treatment, (2) to perform *WCO* refining using different dosages of the bleaching earth, and (3) to evaluate the physicochemical properties of the refined oil. Two types of bleaching earth were prepared: untreated clamshell (*CS*) and calcined clamshell (*FCS*), the latter subjected to high-temperature activation to enhance adsorption capacity. The refining process consisted of degumming, neutralisation, washing, and bleaching using bleaching earth at concentrations of 0.50%, 1.0%, and 1.50%. The effectiveness of each sample was assessed based on moisture content and free fatty acid (*FFA*) levels in the refined oil. Results demonstrated that *FCS* achieved superior refining performance, with a moisture content of 0.08% below the standard limit of 0.1% and a reduced *FFA* value of 0.11%, which complies with the acceptable threshold of 0.25%. These findings highlight the potential of clamshell-based bleaching earth as a low-cost, environmentally friendly material for oil purification. The approach encourages the reuse of agricultural waste and supports more sustainable practices in waste oil treatment and resource recovery.

Keyword: Waste Cooking Oil, Clamshell, Calcined Clamshell, Refining, Bleaching

1. Introduction

The increasing volume of waste cooking oil (WCO) generated by urban populations represents a growing environmental hazard, especially when improperly disposed of, resulting in water contamination and increased greenhouse gas emissions. When refined effectively, WCO serves as a valuable feedstock for biodiesel and other industrial applications. A critical stage in oil refinement is the bleaching process, which removes undesirable compounds such as pigments, peroxides, and free fatty acids (FFAs).

Traditionally, commercial bleaching agents like bentonite clay have been used. However, drawbacks include potential health risks and processing inefficiencies (Mohd & Zainal, 2018). Consequently, sustainable alternatives are being explored. Clamshells, a waste product of the seafood industry, have emerged as a promising alternative adsorbent.

Composed primarily of calcium carbonate ($CaCO_3$), can be processed into an effective bleaching earth. Despite this potential, clamshells remain underutilized in the refining industry, representing a missed opportunity for enhancing sustainability. Clamshells, a calcium carbonate-rich byproduct of the seafood industry, present a promising low-cost alternative. Upon calcination, calcium carbonate ($CaCO_3$) converts into calcium oxide (CaO), which offers enhanced adsorptive and neutralizing capabilities. This study investigates the potential of raw, calcined, and acid-activated clamshells as alternative bleaching agents. The goal is to assess their efficacy in reducing FFA, moisture content and color in WCO, and thereby identifying optimal conditions for refining and validating clamshells as a key component in a circular economy framework.

2. Literature Review

The management of *WCO* is a global challenge due to its large-scale generation from restaurants and households. The high temperature of frying alters the oil's chemical properties, increasing *FFA*, moisture, and impurities, making its direct disposal environmentally detrimental. The improper disposal of *WCO* is a recognised global concern, particularly due to its contribution to water pollution and its adverse impacts on aquatic life (Sari & Kurniawan, 2017). High-temperature frying alters oil chemistry, increasing its *FFA* content and degrading its quality.

The standard refining process utilises bleaching earths—clays with the capacity to adsorb colouring matter and other impurities from oils. Bentonite is a common choice due to its high surface area (200–800 m^2/g) and cation exchange capacity, which make it effective for decolourisation and impurity removal. However, prolonged exposure to bentonite dust is linked to respiratory issues, and its mineral composition may contain harmful substances like lead and arsenic, posing health concerns.

Clamshells, primarily composed of $CaCO_3$, undergo structural changes when subjected to calcination at 600–1000 °C. This process increases surface area and porosity, enhancing adsorption (Malek & Alaan, 2023). Further treatment with hydrochloric acid improves porosity and surface activation, increasing the ability to trap colour pigments and moisture (Zhou et al., 2021). Recent studies have demonstrated that shell-derived adsorbents are effective in removing *FFAs* and pigments from oils, and their performance can be optimised through particle size control and activation treatments (Weng et al., 2021).

3. Materials and Methods

3.1 Bleaching Earth Preparation

Waste clamshells were collected, washed thoroughly, dried, and ground into a fine powder using a mortar and pestle. The powder was sieved to achieve a uniform particle size of 250 nm for all experiments. Two variants of the bleaching agent were prepared: Original Clamshell (CS): Sieved clamshell powder without further modification and furnace-Calcined Clamshell (FCS): Sieved powder was calcined in a muffle furnace at 1000°C for 2 hours and cooled in a desiccator.

3.2 WCO Refining Process

WCO was collected from a cafeteria and filtered to remove food debris. A standard refining procedure was followed for each batch, which included degumming (with phosphoric acid) and neutralization (with NaOH solution) to remove gums and soap stock. After washing and separating the neutralized oil, the bleaching process was conducted. The neutralized WCO was heated to 90°C while being stirred. A pre-determined dosage (0.5%, 1.0%, or 1.5% by weight) of one of the clamshells bleaching earths (CS and FCS) was added. The mixture was stirred for 30 minutes at 90°C. Finally, the oil was filtered using a vacuum pump to separate the spent bleaching earth.

3.3 Analysis of Oil Properties

The quality of the WCO was assessed before and after the bleaching treatment.

i. **Free Fatty Acid (FFA):** Determined by titration of the oil sample against a standardized NaOH solution, with results expressed as % palmitic acid.

ii. **Moisture Content:** Measured using a digital moisture analyser with a 5 g oil sample.

iii. **Colour:** Assessed using a Lovibond Tintometer, with results reported in Red Lovibond units (R).

4. Results and Discussion

The effectiveness of the two clamshell bleaching earths (CS and FCS) was evaluated by comparing the reduction in FFA, moisture, and color at dosages of 0.5%, 1.0%, and 1.5%.

Table 1: Summary of WCO Properties After Refining with Different Bleaching Agents and Dosages

Sample	Dosage (%)	FFA (%)	Moisture (%)	Color (Red)
Raw WCO	-	0.27	0.23	19.9
CS	0.5	0.29	0.13	15.7
	1.0	0.18	0.12	12.3
	1.5	0.09	0.16	11.3
FCS	0.5	0.11	0.08	13.8
	1.0	0.12	0.16	13.4
	1.5	0.06	0.15	13.4

The effectiveness of calcined clamshells as natural adsorbents in refining Waste Cooking Oil (WCO) was evaluated based on different dosages: 0.5%, 1.0%, and 1.5%. Among all treatments, the 1.5% dosage of Furnace-Calcined Clamshell (FCS) showed the most promising overall performance. It resulted in a Free Fatty Acid (FFA) level of 0.06%, moisture content of 0.15%, and a color value of 13.4R, indicating better adsorption capacity compared to the untreated clamshells (CS). Interestingly, the 0.5% dosage of FCS already achieved optimal moisture removal (0.08%), suggesting that lower dosages can still be highly effective for specific properties. These findings align with Malek and Alaan (2023), who emphasized that high-temperature calcination enhances surface reactivity and porosity, making even small quantities of CaO-based adsorbents efficient in oil purification processes.

4.3 Moisture Reduction

The moisture content in raw WCO was recorded at 0.23%, exceeding the MPOB’s allowable maximum of 0.10% for processed edible oils (MPOB, 2020). Post-treatment, only FCS at 0.5% successfully reduced the moisture to 0.08%, meeting the standard. While CS and HCS also improved moisture levels, they did not consistently achieve values below the threshold.

This indicates that calcined shells possess superior hygroscopic properties due to the transformation of $CaCO_3$ to CaO during thermal activation a phenomenon documented by Zhou et al. (2021), who noted that calcined calcium-based materials exhibit high water-binding capabilities. This performance not only improves oil stability but also reduces the risk of hydrolytic rancidity.

4.1 Free Fatty Acid (FFA) Reduction

FFA reduction, all adsorbents demonstrated a decreasing trend with increasing dosage, except for a slight anomaly with CS at 0.5%, which recorded an FFA of 0.29%, higher than the raw sample (0.27%). This could be due to titration sensitivity or lack of sufficient active sites. The FCS variant at 1.5% clearly outperformed others, reducing FFA to 0.06%, far below the 0.25% maximum set by MPOB (2020). The improved performance is attributed to the basic nature of CaO, which neutralizes FFAs through saponification-like reactions. This result reinforces claims by Malek and Alaan (2023) and confirms the potential of thermally treated shell waste in neutralizing acidity in used oils.

4.2 Colour Improvement

Colour improvement, raw WCO presented a Lovibond Red value of 19.9, which is significantly above the edible-grade limit of ≤3R (MPOB, 2020). Although none of the treatments achieved this edible oil benchmark, the FCS and CS samples at 1.5% dosage effectively reduced colour to 13.4R and 11.3R, respectively. The best colour removal was seen with CS at 1.5%, despite its lesser performance in FFA and moisture reduction. These results suggest that while thermally and chemically treated clamshells can reduce oil discoloration, additional refining steps may still be required to meet full edible oil standards. As noted by Weng et al. (2021), finer particle size and surface activation play a key role in enhancing the adsorption of complex pigment molecules, a property likely enhanced by acid activation or physical grinding.

Furnace-Calcined Clamshell (FCS) emerges as the most effective adsorbent compared to untreated Clamshell (CS). FCS at 1.5% dosage reduced the FFA to 0.06%, well below the MPOB standard of 0.25%, and achieved the lowest moisture content at 0.08% (at 0.5% dosage), which meets the industry threshold of 0.10%. Although CS achieved slightly better colour reduction at 1.5% , it was less effective in reducing FFA and moisture. These findings suggest that thermal activation through calcination significantly enhances the adsorptive and neutralizing properties of clamshells, making FCS a superior, more reliable option for sustainable WCO refining.

5. Conclusion

This study successfully met its three main objectives in evaluating clamshell-derived bleaching earth as a sustainable adsorbent for refining waste cooking oil (WCO). Through thermal calcination, clamshells were effectively transformed into functional bleaching earth, demonstrating the potential of calcium-rich seafood waste as a viable adsorbent material. Refining trials using varying dosages (0.5%, 1.0%, and 1.5%) of untreated clamshell (CS) and furnace-calcined clamshell (FCS) allowed for comparative performance analysis. Post-treatment assessments of WCO's physicochemical properties—including Free Fatty Acid (FFA) content, moisture content, and colour—revealed that FCS at 1.5% achieved the best refining outcome, reducing FFA from 0.27% to 0.06% and moisture from 0.23% to 0.15%, meeting Malaysian Palm Oil Board (MPOB) standards. Notably, FCS at 0.5% also achieved the lowest moisture content (0.08%), highlighting its high adsorption efficiency even at low dosage. While CS at 1.5% showed slightly better colour reduction (11.3R vs. FCS's 13.4R), it was less effective in reducing FFA and moisture. These results emphasize the role of thermal treatment in enhancing adsorptive performance, positioning calcined clamshell-derived bleaching earth as a promising, cost-effective, and eco-friendly alternative to conventional bleaching agents, supporting waste valorisation and circular economy initiatives in edible oil refining.

6. Recommendation

To enhance the utility and scalability of clamshell-derived bleaching earth, it is recommended to optimize calcination parameters such as temperature and duration to improve adsorption capacity while reducing energy consumption (Zhou et al., 2021). Incorporating additional refining stages like deodorization and filtration will enable a more thorough evaluation of final oil quality. Advanced characterization methods, including X-ray Fluorescence (XRF), Scanning Electron Microscopy (SEM), and Brunauer–Emmett–Teller (BET) surface analysis, are suggested to gain deeper insights into the structural and chemical changes that boost adsorption efficiency (Weng et al., 2021). Future studies should examine the reusability and regeneration of clamshell-based adsorbents for sustainable and cost-effective applications, alongside benchmarking with commercial bleaching earths such as bentonite and activated clay (Mohd & Zainal, 2018). Exploring composite materials that combine clamshell with other natural or modified substances may yield synergistic improvements in refining performance. Finally, techno-economic analysis and life cycle assessment (LCA) are recommended to evaluate the environmental impact, economic feasibility, and scalability of applying this approach in industrial WCO treatment and edible oil recovery (Ranjith & Saravanan, 2016).

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