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Reutilization of Real Waste Calcium Carbonate ($CaCO_3$) from the Palm Oil Industry (POI) for Palm Kernel and Shell Separation Through Clay Bath Systems

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Abstract: The palm oil industry (POI) generates significant amounts of waste, including calcium carbonate ($CaCO_3$) from the clay bath system used for the separation of palm kernels from shells. This $CaCO_3$ waste is often discarded, leading to environmental issues. However, the $CaCO_3$ can potentially be reused in the clay bath separation process to improve efficiency and reduce waste. To obtain PKO, the kernel is separated from the palm shell using a clay bath unit, where natural $CaCO_3$ acts as a decanting agent and adsorbent. This wet separation method, involving a mixture of water and $CaCO_3$ with a density of 1.12 g/mL, generates substantial amounts of saturated $CaCO_3$ waste that is often discarded into the environment. Therefore, this research aimed to regenerate oil-bound $CaCO_3$ waste for reuse as a decanter and adsorbent. Three treatments were tested, with $CaCO_3$ waste-to-water ratios of 1:1, 1:3, and 1:6, under varying pH levels (8, 10, 11, 12) and temperatures (28 °C, 80 °C, 100 °C). The regeneration process was conducted in an open reactor at 450 rpm with a volume of 0.0054 m^3, followed by drying and grinding the waste for analysis. The results showed approximately 75.75% oil removal and $CaCO_3$ regeneration rates between 94.50% and 99.26%, with an increase in density from 1.687 g/mL to 2.467 g/mL. The efficiency of reusing regenerated $CaCO_3$ waste is 96.87%. When mixed with 25% natural $CaCO_3$, the efficiency increases to 99.24%. Additionally, a mixture of 50% regenerated $CaCO_3$ waste and 50% natural $CaCO_3$ achieves an average efficiency of 99.46% over five consecutive feed additions. This showed that the reuse of $CaCO_3$ waste regeneration results for the separation of palm shells and kernels has a high potential for application. These findings suggest that regenerated $CaCO_3$ waste can be effectively reused, offering a sustainable solution for palm oil mills.

Keywords: $CaCO_3$ waste; clay bath; decanting agent; palm oil; palm kernel shell; regeneration

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1. Introduction

Since 2019, Indonesia has emerged as the world's largest producer of palm oil and palm kernel oil, with an annual production of approximately 58 million tons [1]. The province of Aceh plays an important role in this industry, especially in the production of crude palm oil (CPO). Notably, the regencies of Aceh Tamiang and Aceh Timur have the highest number of palm oil mills in the province [2–4]. In 2020, Indonesia processed 49 million tons of palm oil, resulting in approximately 19.6 million tons of waste biomass. This significant volume of waste poses considerable disposal challenges and necessitates proper treatment to mitigate environmental threats [5–7]. In general, the processing of palm oil from palm fruits generates substantial amounts of palm kernel shell waste, which is occur in various applications that otherwise would have a low carbon footprint [8,9].

A palm oil mill converts fresh fruit bunches (FFBs) into CPO and palm kernels. In several African countries, such as Nigeria and Ghana, kernels and shells are separated manually, as reported by Aderinlewo et al. [10,11], Akusu et al. [12], and Alade et al. Conversely, in Indonesia, this separation typically occurs at the Nut and Kernel Recovery Station (NKRS) using a wet process [13,14]. Research on efficient kernel separation methods continues to focus on achieving high recovery rates through both dry and wet techniques, each of which presents advantages and limitations. For instance, the dry method requires significant energy to generate high-pressure air and demands particle uniformity for effective separation, along with creating noise issues [15–17]. Meanwhile, the wet method involves extensive use of clay and water, resulting in substantial clay waste [18–20]. Considering these factors, this research evaluated palm kernel shell separation using the wet method.

Previous research explored various materials for separating kernels and shells using wet methods, including empty fruit bunch ash [21], CO_2 gas [22], saline water [23,24], and clay [21,25,26]. The findings showed that for soil and biomass ash, a high separation efficiency can be achieved of approximately 98% [17]. However, this method is limited by restricted ash sources, reusability challenges, and the need for an additional combustion process to obtain ash, which is energy intensive. Separation methods carried out using saltwater or saline water also show a good efficiency and have advantages over traditional clay methods [23], such as the abundant availability of seawater. However, they require significant energy for drying the kernels and shells. The traditional clay separation method results in a 1.728% loss of palm kernels, which meets company requirements, but sourcing suitable clay is difficult. Additionally, the process is time-consuming and unhygienic, taking approximately 10 min and leading to high moisture adsorption in the kernels [27]. Natural $CaCO_3$ is widely used as a separation agent in the palm kernel shelling process due to its high efficiency and cost-effectiveness, stemming from its abundant availability, although it has also been reported that the process generates a substantial amount of waste [28].

In Indonesia, the palm oil industry (POI) primarily utilizes a wet process for separating palm kernel shells, with nearly all mills using a $CaCO_3$ suspension in the clay bath unit [29]. The usage of $CaCO_3$ varies from 15 to 25 tons per month for processing 30 tons of FFB per hour, depending on the volume of FFB processed. Before use, natural $CaCO_3$ is ground to a mesh size of 60 to 270 [30]. The separation process uses a $CaCO_3$ solution with a density of approximately 1.12 g/mL, creating a density gradient that allows the lighter kernel to float and the heavier shell to sink, thereby minimizing kernel loss. The wet palm kernel has a specific gravity of 1.07 g/mL, while the shell has a specific gravity between 1.15 and 1.20 g/mL. To separate the core from the shell, a $CaCO_3$ solution with a density of 1.12 g/mL is prepared, allowing the core to float and the shell to sink. The effective density of the $CaCO_3$ solution varies from 1.11 to 1.13 g/mL [14].

Over time, the $CaCO_3$ solution used in the clay bath unit becomes saturated with oil, leading to its disposal as waste without further processing [26]. This waste poses significant environmental challenges if not managed properly [30,31]. This research examined the potential for reusing the $CaCO_3$ waste for palm kernel and shell separation. $CaCO_3$ can be regenerated, by utilizing water [32–36] with high temperatures. This investigation focused on the importance of the mixture temperature and solution alkalinity as important factors in operating conditions. Previous research has shown that hot water can effectively remove oil and regenerate adsorbents [37–39]. However, the regeneration of $CaCO_3$ waste from palm oil processing has not been extensively developed, primarily due to limited interest in the POI. Therefore, this research aimed to investigate the reuse of $CaCO_3$ waste through regeneration to enhance efficiency and reduce environmental pollution.

The results of the literature review indicate that research on the regeneration of $CaCO_3$ waste generated from the separation of palm kernels and shells in palm oil processing plants has not been previously explored. Additionally, the use of natural $CaCO_3$ in the palm kernel and shell separation process has not been widely explored. Research on palm oil processing has not attracted significant attention from researchers in developed countries; instead, most investigations on palm oil are conducted by researchers in palm-oil-producing and developing countries. For the reasons outlined above, this research aimed to investigate the reuse of $CaCO_3$ waste through a regeneration process, with the goals of enhancing cost efficiency and reducing environmental pollution in the surrounding area.

Specifically, this research investigated the regeneration of saturated $CaCO_3$ waste from palm oil mills' clay bath units by washing with hot water at 80–100 °C, creating alkaline conditions. This investigation examined the effectiveness of utilizing hot water under specific pH (8, 10, 11, and 12) and temperature conditions (room temperature, 80 °C, and 100 °C), comparing different $CaCO_3$ waste-to-water ratios (1:1; 1:3, and 1:6), and also investigated its performance in the separation palm kernels and shells [12]. This research was conducted in batch processes through an open reactor and assessed the impacts of pH, temperature, and the process time on the density, oil content, and regeneration yield of the treated waste.

2. Results and Discussion

2.1. Influence of $CaCO_3$ Waste-to-Water Ratio, pH, and Temperature on the Oil Content and the Density of Regenerated $CaCO_3$ Waste

Three distinct treatments were used to regenerate $CaCO_3$ and assess the reduction in oil content in the waste. The treatments involved $CaCO_3$ waste samples with pH values of 6.48 (1:1 ratio), 6.63 (1:3 ratio), and 6.59 (1:6 ratio). Each treatment was conducted at room temperature, 80 °C, and 100 °C, with four operational points per treatment. The analysis showed that all treatments significantly reduced the oil content, though the extent varied across different conditions and treatments, as shown in Figure 1.

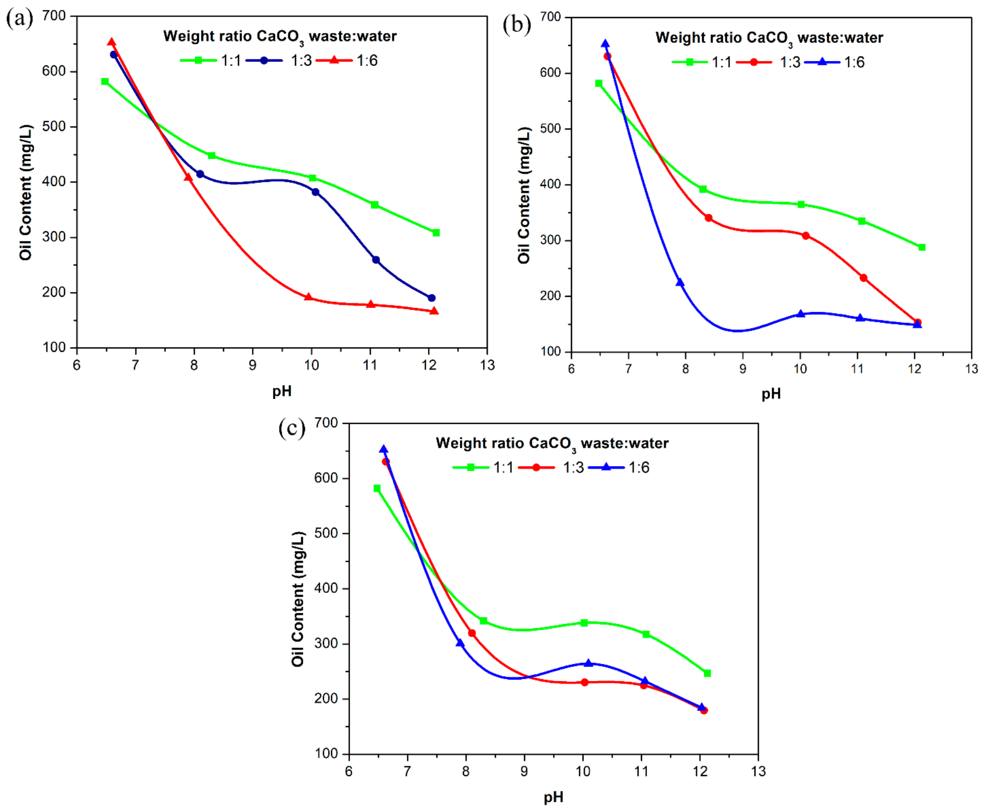

Figure 1. Influence of pH on the oil content (**a**) regeneration at room temperature; (**b**) regeneration at 80 °C, and (**c**) regeneration at 100 °C.

The analysis of pH influence showed that at room temperature, the oil content decreased from 581.82 mg/L to 448.02 mg/L with a 1:1 ratio, and further to 165.96 mg/L with a 1:6 ratio. At 80 °C, the reduction ranged from 581.82 mg/L to 364.91 mg/L with a 1:1 ratio, and to 148.78 mg/L with a 1:6 ratio. At 100 °C, the oil content decreased from 581.82 mg/L to 342.12 mg/L with a 1:1 ratio, and from 630.78 mg/L to 179.36 mg/L with a 1:3 ratio. The most significant reduction, to 148.78 mg/L, was observed at a 1:6 ratio under actual conditions of 81.5 °C and pH 12.06.

The density of regenerated $CaCO_3$ is influenced by pH (Figure 2), with variations depending on the ratio and conditions of use. At a 1:1 ratio, the density is 1.553 g/mL, while at a 1:3 ratio, the density is 1.730 g/mL, and at a 1:6 ratio, it is 1.687 g/mL. These differences are due to the amount of oil bound to the $CaCO_3$, which is released during regeneration, causing the density to increase and approach its original value. The lowest density of regenerated $CaCO_3$ waste at a 1:1 ratio is 1.720 g/mL, achieved at 79.8 °C and pH 8.3, while the highest density is 2.467 g/mL at a 1:6 ratio under 28.1 °C and pH 12.09. These findings underscore the importance of monitoring and optimizing density values during the regeneration process, which is essential for evaluating the efficiency of oil removal under varying pH and temperature conditions.

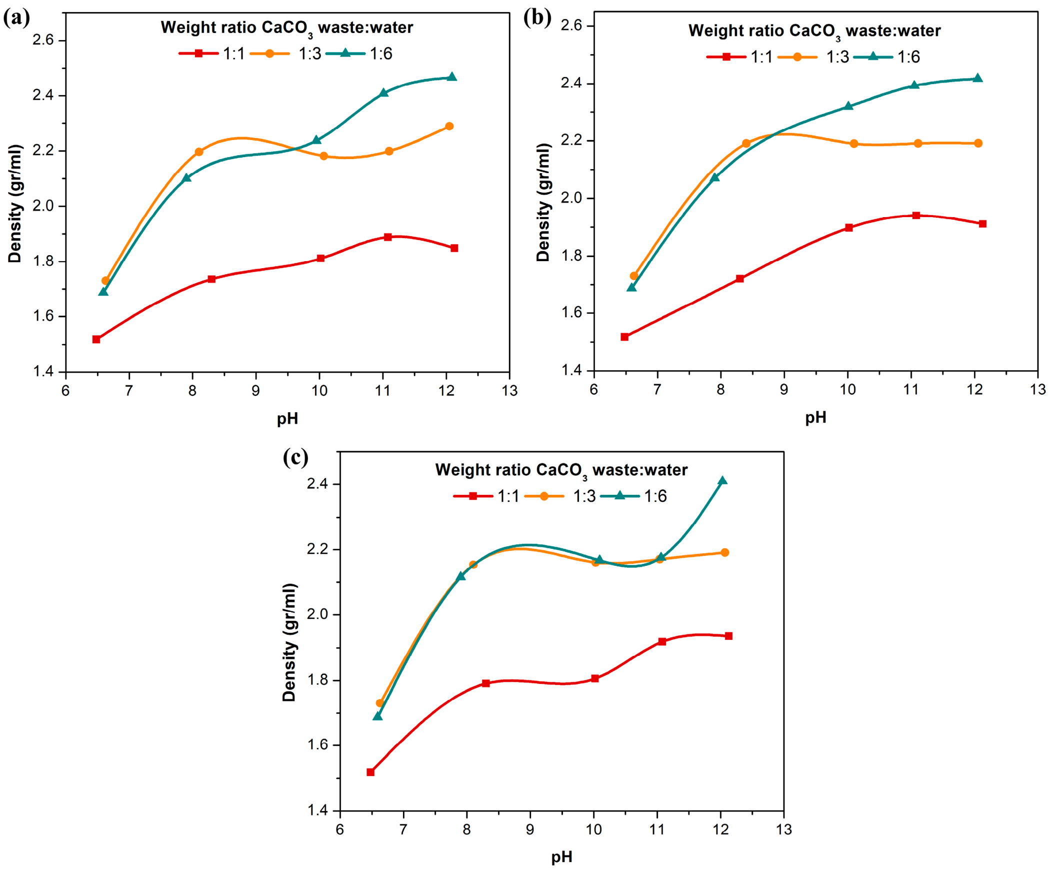

Figure 2. Influence of pH on the density of $CaCO_3$. (**a**) Regeneration process at room temperature; (**b**) regeneration process at 80 °C; and (**c**) regeneration process at 100 °C.

The efficiency of oil removal from regenerated $CaCO_3$ waste varies significantly with different treatment ratios (1:1, 1:3, and 1:6), as shown in Figure 3. At a 1:1 ratio, the lowest reduction in oil content (22.99%) was observed under room temperature, neutral pH, and a $CaCO_3$ waste-to-water ratio of 1:1. This minimal reduction was due to limited interaction between the waste and the washing medium [40,41].

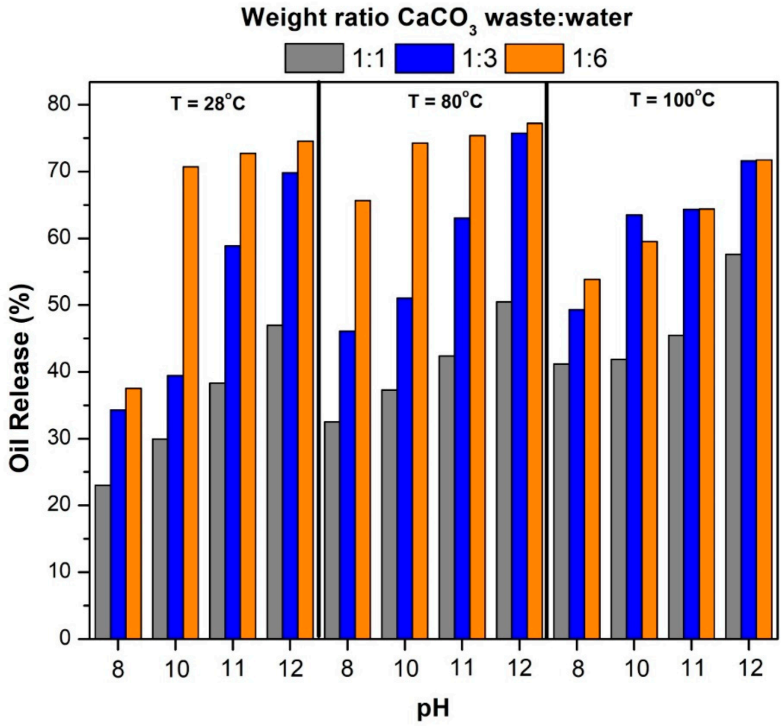

Figure 3. Percentages of oil content removal in $CaCO_3$ waste regeneration process at a ratios of $CaCO_3$ waste to water of 1:1, 1:3, and 1:6.

The highest reduction at this ratio (57.61%) was achieved at 98.2 °C and pH 12, indicating that an elevated temperature and alkaline pH significantly enhanced the oil removal efficiency. For the 1:3 ratio, the least oil content removal was 34.28% at room temperature and a neutral pH, slightly higher than the 1:1 ratio. The maximum decrease in oil content, 71.53%, occurred at an operating temperature of 96.8 °C and a pH of 12. This occurred because the oil bound to $CaCO_3$ degraded due to the high temperature and pH [42]. At a 1:6 ratio, the minimum oil content reduction was 58.75% under room temperature and a neutral pH, attributed to the increased amount of water available for washing. The highest oil content reduction, 78.15%, was recorded at a pH of 12.3 and 97.5 °C, underscoring the synergistic effect of a high pH and temperature on the liberation of oil from $CaCO_3$ waste.

Figure 4 presents the impact of temperature on the density and oil content of $CaCO_3$ during the utilization of regenerated $CaCO_3$ waste, analyzed at three treatment ratios: 1:1, 1:3, and 1:6. For the 1:1 ratio (Figure 4a), increasing temperature led to higher density values across all pH levels (8, 10, 11, and 12). The lowest density recorded was 1.736 g/mL at 28 °C, while the highest was 1.940 g/mL at 80 °C and pH 11. At pH 10 and pH 11, there was a decrease in density at a temperature of 100 °C. This occurred because, at 100 °C, the oil was reabsorbed by $CaCO_3$, resulting in a decrease in density. In addition, the 1:3 ratio (Figure 4b) showed a decrease in density with a rising temperature, with the lowest density of 2.154 g/mL at 100 °C and pH 8, and the highest of 2.289 g/mL at 28 °C and pH 12. For the 1:6 ratio (Figure 4c), density decreased at 80 °C but increased again at 100 °C, with the lowest density of 2.116 g/mL at 100 °C and pH 8, and the highest of 2.467 g/mL at 28 °C and pH 12. These findings lead us to surmise that the density of regenerated $CaCO_3$ waste is influenced by both the amount of oil bound to it and the ratio of waste regeneration to water. An increased volume of water used leads to a higher density of the regenerated $CaCO_3$ waste.

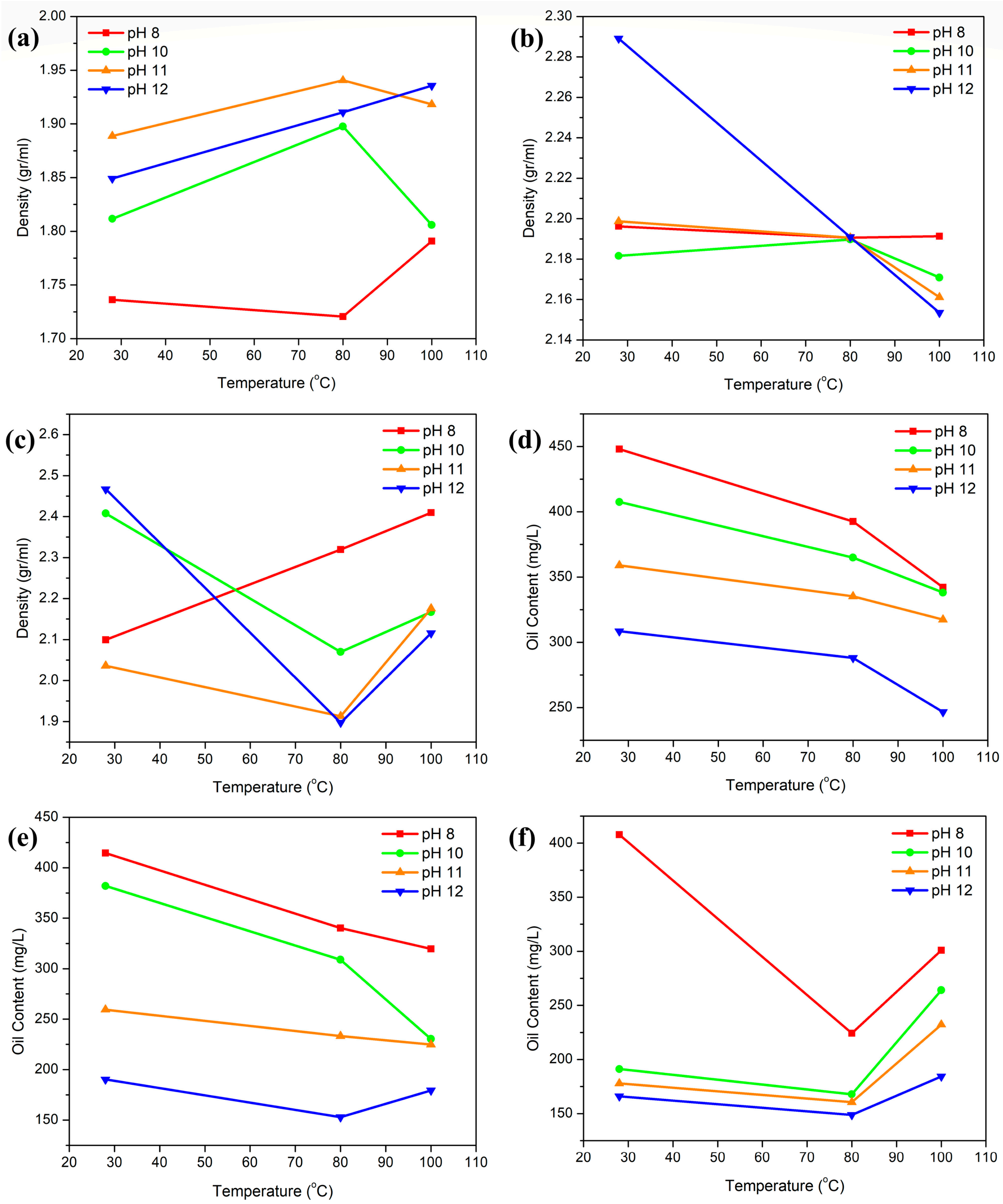

Figure 4. Influence of temperature on the density of regenerated $CaCO_3$ at waste-to-water ratios of (**a**,**d**) 1:1; (**b**,**e**) 1:3; and (**c**,**f**) 1:6.

The assessment of oil content during the use of regenerated $CaCO_3$ waste showed differences between treatment ratios (1:1, 1:3, and 1:6). The oil content was 581.82 mg/L for the 1:1 ratio, 630.78 mg/L for the 1:3 ratio, and 652.56 mg/L for the 1:6 ratio, with each treatment showing a decrease in oil content with rising temperatures. For the 1:1 ratio (Figure 4d), the oil content consistently decreased with an increasing temperature, reaching a minimum of 246.58 mg/L, a 57.62% reduction. For the 1:3 ratio (Figure 4e), the oil content decreased at 28 °C and 80 °C but increased at 100 °C, with the lowest content being 152.98 mg/L at pH 12 and 80 °C, a 75.75% reduction. For the 1:6 ratio (Figure 4f), the oil content decreased from 28 °C to 80 °C but increased at 100 °C, due to prolonged heating causing oil to rebind to the $CaCO_3$ pores [43], with the smallest reduction being 148.78 mg/L at 80 °C and pH 12, a 77.19% reduction.

To effectively separate palm kernels and shells, it is essential to measure and restore the medium density to its original value of 2.553 g/mL, which corresponds to the density of

$CaCO_3$ prior to use. The regeneration process involves increasing the pH and temperature of the washing medium (hot water) to release oil bound to $CaCO_3$. Initially, $CaCO_3$ becomes saturated with oil during separation. However, as the pH and temperature increase, the oil content in the $CaCO_3$ waste decreases. This results in a significantly lower density of the $CaCO_3$ waste compared to pure $CaCO_3$, due to the lower density of palm oil (0.909 g/mL–0.916 g/mL) relative to $CaCO_3$ (2.71 g/mL).

In a subsequent investigation evaluating the yield of regenerated $CaCO_3$ waste, the yield was determined by comparing the moisture-adjusted content of the waste with the regenerated product and then multiplying by 100%, as shown in Figure 5. For the 1:1 ratio, the lowest yield was 97.11% at 79.8 °C and pH 8.3, while the highest yield was 98.57% at 98.5 °C and pH 12.13. In the 1:3 ratio treatment, the lowest yield was 95.15% at 29.1 °C and pH 12.05, and the highest yield was 96.67% at 81.7 °C and pH 12.06. For the 1:6 ratio, the lowest yield recorded was 94.50% at 27.8 °C and pH 11.01, while the highest yield was 96.59% at 27.6 °C and pH 9.96. The variations in yield values are attributed to the different water-to-$CaCO_3$ waste ratios used, rather than changes in temperature and pH.

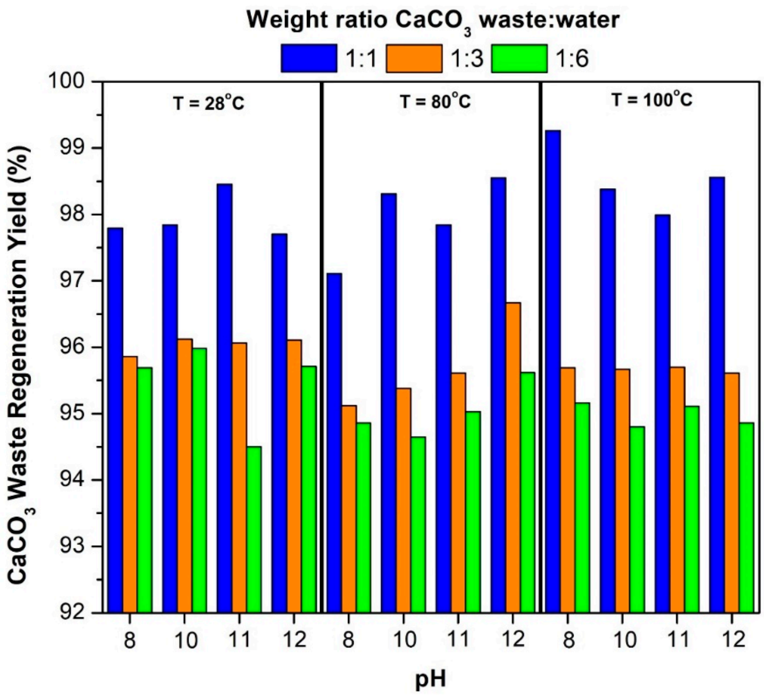

Figure 5. The percentage yield in $CaCO_3$ waste regeneration at different ratios of $CaCO_3$ waste to water (1:1, 1:3, or 1:6).

The results highlight the significance of process parameters such as temperature, pH, and the water-to-waste ratio in optimizing the removal of oil from $CaCO_3$ waste. By carefully adjusting these variables, the research shows the potential to maximize efficiency in waste regeneration processes, providing valuable information for industrial applications focused on improving both environmental sustainability and operational effectiveness. In particular, this research shows the potential for improving the reuse of $CaCO_3$ in industrial applications by achieving density values close to the original, thereby ensuring both economic and environmental benefits.

2.2. Performance of Regenerated $CaCO_3$ Waste in Separating Palm Kernels and Shells

The efficiency of the separating solution was evaluated using metrics such as kernel recovery (Kr), kernel contaminant (Kc), shell recovery (Sr), and shell contaminant (Sc). These indicators were used to measure the solution's effectiveness, with performance values recorded hourly for each treatment throughout the research period, as shown in Figure 6.

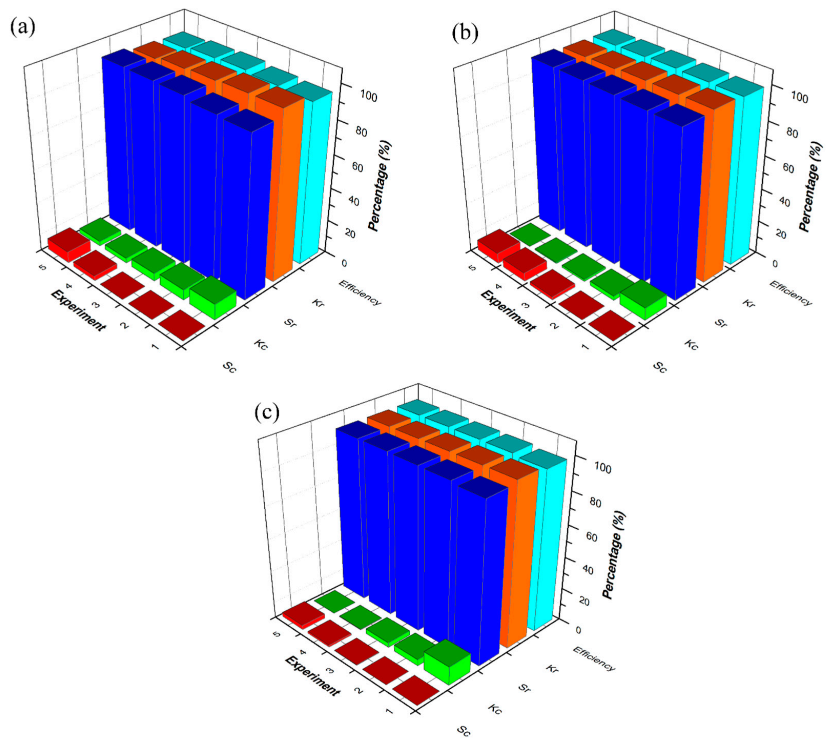

Figure 6. Performance of regenerated $CaCO_3$ waste: (**a**) 50% $CaCO_3$ waste and 50% $CaCO_3$ fresh; (**b**) 75% $CaCO_3$ waste and 25% $CaCO_3$ fresh; and (**c**) 100% $CaCO_3$ waste.

In experimental research, 1 kg of feed per batch was introduced at five distinct intervals to compare the regenerated separation solution against water at a 1:3 ratio. The solution's performance, assessed by Kr and Kc, showed that Kr remained at 100% for the first 2 h but declined from the third to the fifth hours due to a reduction in solution density. Concurrently, Kc decreased from 9.6% to 2.93%, also correlating with the decreasing density. Sr increased from 95.7% to 99.3% over the 5 h, attributed to the higher initial solution density, which facilitated the collection of shell fragments at the kernel station. Sc remained at 0% for the first 3 h, then increased to 6.77% by the fifth hour due to broken kernels and dense palm nuts. The efficiency, calculated by multiplying Kr and Sr, ranged from 95.7% to 98%, demonstrating a high efficiency with 1 kg of feed per batch in 4 L of solution.

3. Materials and Methods

3.1. Materials and Equipment

$CaCO_3$ waste was obtained from PT. Ujong Neubok Dalam (PT. UND) Palm Oil Company in Nagan Raya, Aceh, Indonesia. Before utilization, the samples underwent filtration and were analyzed to determine their initial physical properties, including pH, density, oil content, and moisture content. The solvents used, such as organic solvent S-316 (Horiba, Kyoto, Japan), NaOH (Merck, Rahway, NJ, USA), pH buffer, and distilled water, were obtained from chemical distributors and used without further purification.

The equipment utilized consisted of an open batch reactor (diameter = 0.23 m; height = 0.13 m; volume = 0.0054 m^3; equipped with a puddle impeller agitator), oil content analyzer (Horiba OCMA 350, Horiba, Kyoto, Japan), 25 mL pycnometer (Iwaki, Japan), Orion Star A329 pH meter (Thermo Fisher Scientific Inc., Waltham, MA, USA), oven (Memmert Model 100-180, Memmert, Schwabach, Germany), 1000 mL beakers (Iwaki, Japan), measuring pipettes (Iwaki, Japan), volumetric flasks (Iwaki, Japan), a stirrer motor, an electric water bath, and a temperature control unit.

3.2. Experimental Set-Up

This research used an open reactor (dimensions: diameter = 0.23 m, height = 0.13 m, and volume = 0.0054 m^3) equipped with a puddle impeller agitator. The $CaCO_3$ waste, after being analyzed for moisture content, oil content, density, and pH, was introduced into the reactor at $CaCO_3$ waste-to-water ratios of 1:1, 1:3, and 1:6. The pH of the water was then adjusted to neutral (6.5–8.5), 10, 11, and 12. The operating conditions were set at room temperature, 80 °C, and 100 °C. The regeneration process was carried out using an agitator at 450 rpm for 30 to 40 min per treatment. Subsequently, post-regeneration, the $CaCO_3$ waste was dried in an oven at 110 °C for 2 h, then ground and tested for moisture content (SNI 1965:2008), oil content, density of solid product (SNI 1965:2008), and product yield.

To evaluate the efficiency of regenerated $CaCO_3$ waste in separating the palm kernel from the shell, the fine powder of the regenerated waste was first dissolved in water at a 1:3 ratio to create a separation solution. Specifically, 1 kg of regenerated $CaCO_3$ waste was combined with 3 kg of water in a reactor. The separation process involved processing a 1 kg/batch mixture of palm kernels and shells for approximately 20 min in a stirred reactor. After the solution was homogenized, the mixture was poured from the top, allowing the low-density palm kernels to float while the shells settled at the bottom. The separated palm kernels and shells were then manually collected using a sieve, washed, air-dried, and finally oven-dried at 105 °C for 2 h. To determine Kr, Kc, Sr, Sc, and efficiency, the dried palm kernels and shells were weighed, following an equation from the previously published literature [17]. The schematic process of $CaCO_3$ waste regeneration in the palm kernel shell separation is shown in Figure 7.

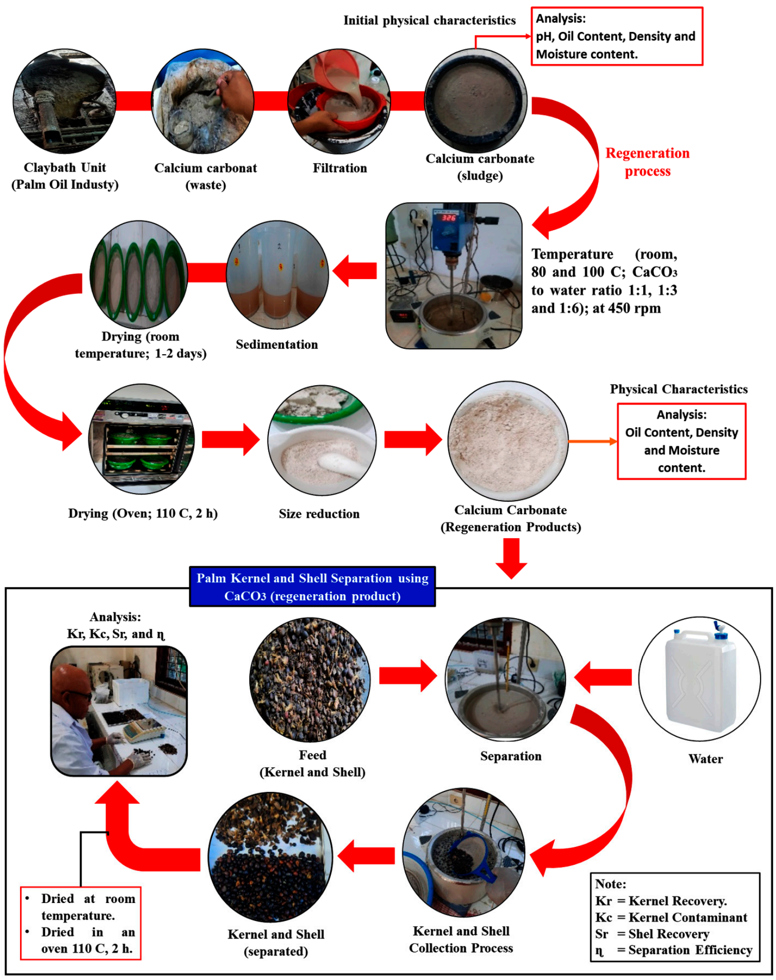

Figure 7. Schematic process of $CaCO_3$ waste regeneration in the palm kernel shell separation.

4. Conclusions

In conclusion, research on regenerating $CaCO_3$ waste from the clay bath unit at a palm oil processing plant has shown promising results. The removal of oil content from the waste was achieved at a remarkable 75.75%. The density of the regenerated $CaCO_3$ waste was measured at 2.467 g/mL, closely matching the fresh $CaCO_3$ density of 2.553 g/mL. The yield of regenerated $CaCO_3$ waste varied between 94.50% and 99.26%, this regenerated waste is anticipated to be reusable for separating palm kernels and shells. The substantial reduction in oil content, near-restoration of the original density, and high yield percentages underscore the potential for sustainable recycling of $CaCO_3$ in industrial applications, providing both economic and environmental advantages. The efficiency value of the reuse of $CaCO_3$ waste regeneration results was 96.87%, the mixture of 75% $CaCO_3$ waste regeneration results with 25% natural $CaCO_3$ had an efficiency value of 99.24%, and the results of 50% $CaCO_3$ waste regeneration with 50% natural $CaCO_3$ had an average efficiency value over five continuous feed additions of 99.46%. This indicated that reusing regenerated $CaCO_3$ waste for the separation of palm shells and kernels is highly feasible. These findings also provide a valuable reference for future research on the repurposing of regenerated $CaCO_3$ waste.

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