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Performance Evaluation of Waste Chicken Eggshell and Waste Seashell as a Heterogeneous Catalyst for Biodiesel Production

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***Abstract*— *Due to their reusability, heterogeneous catalysts for biodiesel production have gained popularity. If the commercial-grade heterogeneous catalyst could be replaced with CaO generated from eggshells (which is a waste product) and seashells (which is abundantly available in Sri Lanka) would be beneficial for the sustainability of the process and circular economy. This work em-phasizes and summarizes the derivation of CaO from eggshell and seashell, the investigation of the produced catalyst using a scanning electron microscope, and the comparison of the two by powder X-ray diffraction. CaO produced from eggshell, and seashell exhibited slight differences in its surface structure. However, there are larger voids in the seashell-derived catalyst due to the release of CO_2 during calcination. The derived catalyst and commercial CaO were both employed to produce biodiesel under identical operating circumstances, including a 12: 1 Methanol-to-oil molar ratio, 60 °C operating temperature, 120min reaction time, 600 rpm, and a 5 wt.% catalyst concentration. The biodiesel yield was computed and compared for all three scenarios involving catalyst recycling. Commercial grade CaO produced the highest yield, followed by seashell- and eggshell-derived CaO. Both derived catalysts are less pure than commercial CaO, as they were produced with $CaCO_3$ in addition to CaO.*

***Keywords*— *Biodiesel, Catalyst, CaO, Eggshell, Hetergenous, Renewable energy, Seashell, Transesterification, Waste cooking oil.*

I. INTRODUCTION

As a consequence of industrialization and population growth, energy supply instability has been a challenge for several decades. Due to the alarming rates of global warming and environmental pollution, the worldwide community has identified the development of sustainable energy sources as a top research priority [1]. Multiple causes are re-sponsible for environmental pollution such as industrialization, globalization, vehicle emission, population [2-4]. The combustion of fossil fuels for power generation and transportation accounts for a greater proportion of air pollution [5,6]. The research of worldwide energy consumption patterns indicated that automobiles fueled by fossil fuels account for more than 70 % of all CO emissions and 19 % of all CO_2 emissions [7]. There-fore, Biodiesel production and implementation widely discussing worldwide to reduce the fossil fuel combustion and to confirm the energy security. Biodiesel has multiple benefits such as, high combustion efficiency, biodegradable nature, lubricity, less environmental pollution, and clean sustainable energy source [8]. Biodiesel is produced by the reaction of triglycerides with simple alcohols such as Methanol, Ethanol, Propanol and Butanol and with the presence of catalyst [9]. The overall reaction of the biodiesel production is transesterification reaction (Fig. 1).

The most widely used catalyst for biodiesel production is homogeneous alkaline catalyst such as potassium hydroxide or sodium hydroxide due to its availability and ease of operation [11]. These catalysts are present in same phase during the reaction [12, 13]. However, heterogeneous catalyst has also gain broader attention recently due to reusability of the catalyst and the availability [14, 15].

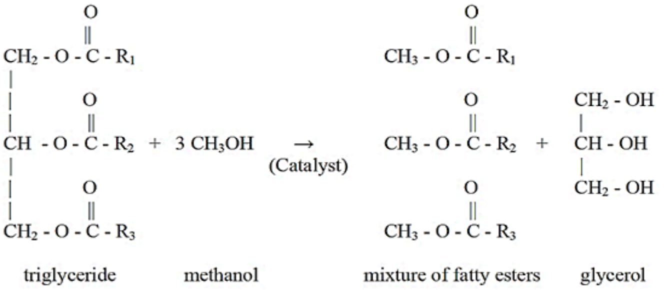

Fig. 1. A schematic representation of the Transesterification Reaction [10]

By considering the advantages of heterogeneous catalysts, they are easily separated from the product by filtration, and they can be recycled several rounds for the biodiesel production without depleting the yield significantly [16]. As an examples for heterogeneous catalysts, calcium oxide (CaO), magnesium oxide (MgO), barium oxide (BaO), zinc oxide (ZnO), barium hydroxide ($Ba(OH)_2$) can be considered [12, 17]. CaO is one of the most popular catalysts because to its abundance in nature, high activity, and inexpensive cost [18]. Further, CaO base catalyst impregnated with metal oxide will boost reactivity by overcoming separation difficulties when CaO is used alone in the reaction media [15, 16]. numerous studies have been conducted to determine the efficiency of CaO catalyst obtained from eggshell waste and CaO derived from seashells. Some of the researchers were generating eggshell derived CaO catalysts [16, 19-25] to optimize the biodiesel process. Several studies have also used a CaO catalyst generated from seashells to reduce the cost of the catalyst and increase the biodiesel yield [26-29].

Biodiesel's commercialization and market competitiveness are hampered primarily by its high production cost and the high price of its raw materials [30-33]. Compared to fossil diesel, biodiesel produced from regular crops is more expensive; therefore, it is essential to search for inexpensive and readily available feedstocks, alcohols, and catalysts in order to address this issue. Biodiesel manufacturing from wastes such as waste cooking oil (WCO) and eggshell-derived CaO is favorable for both economically and environmentally sustainable industry. [31-38].

Eggshell debris and seashells include approximately 95 % calcium carbonate ($CaCO_3$), calcium phosphate, and organic materials [39, 40]. Additionally, seashells contain a trace quantity of magnesium carbonate.

Several previous works have reported on the utilization of CaO material as a catalyst in the transesterification reaction from different sources. CaO catalyst is prepared from natural sources, mostly from different waste shells. As stated by Rezaei et al. a biodiesel yield of 94.1 % was obtained from soybean oil using CaO catalyst prepared from mussel shell with pronounced purity at a specified experimental setting [41]. Likewise, Chen et al. also reported a 93% biodiesel yield from palm oil using CaO catalyst made from ostrich eggshell prepared through an ultrasonic technique [42]. Moreover, Chouhan et al. achieved a biodiesel yield of more than 97 % from soybean oil using an actual CaO catalyst pre-pared from chicken waste eggshell at optimal experimental conditions. They demonstrated the practicality of waste chicken eggshell based CaO catalyst for high yield and good quality biodiesel production [43]. Similarly, Gupta et al. also reported a biodiesel yield of 96 % from soybean oil using snail eggshell based CaO catalyst at a particular experimental condition [44]. Yoosuk et al. [45] observed that hydrated CaO obtained through decomposition exhibited higher catalytic activity, particularly at shorter reaction times, compared to CaO obtained through direct calcination of $CaCO_3$ under mild conditions. The preparation of hydrated CaO involved subjecting $CaCO_3$ to thermal decomposition at a temperature of 800 ℃ for a duration of 3 hours. The resulting solid product, CaO, was further processed by refluxing it in water at a temperature of 60 ℃ for 6 hours, followed by filtration and subsequent heating at a temperature of 120 ℃ overnight. Prior to application, the product was subjected to calcination at a temperature of 600 ℃ for a duration of 3 hours, in order to convert the hydroxide form to the oxide form, which is known to be the active form for transesterification reactions. The solid product that was obtained as a result of this series of procedures was referred to as hydrated CaO. The authors achieved a biodiesel yield of 93.9 % with an oil-to-methanol molar ratio of 1:15, a 7 % by weight catalyst loading, a reaction temperature of 60 ℃, and a reaction time of one hour. Notably, the use of hydrated CaO catalysts resulted in a significant biodiesel yield within a short reaction time, primarily attributed to the enhanced catalytic activity. [45].

Consequently, by subjecting $CaCO_3$ to a high-temperature calcination process, it can be converted into CaO. Calcium oxide is known to be an active metal oxide catalyst that can be used for transesterification reactions, which is a chemical reaction used for the conversion of esters into other chemicals. Therefore, the conversion of $CaCO_3$ to CaO through the calcination process can provide a useful material for catalyzing such chemical reactions. [45, 46]. This research presents a novel approach to sustainable and cost-effective catalyst development by utilizing waste eggshells and seashells as a precursor for CaO production. This method offers an eco-friendly and low-cost alternative to commercially available CaO, which is currently derived from non-renewable sources. Furthermore, the potential of waste eggshell and seashell derived CaO as a heterogeneous catalyst has not been extensively explored in literature. Hence, this study aims to contribute to the scientific community's understanding of the catalytic activity of waste derived CaO and its potential as a commercially viable alternative to traditionally sourced catalysts.

II. MATERIAL AND METHOD

2.1 Catalyst preparation

Eggshells and seashells were used to prepare CaO samples, whereas CaO of commercial grade was procured from Loba Chemie (Pvt) Ltd. In Colombo, Sri Lanka, eggshell debris was gathered from numerous restaurants, while seashells were gathered from the neighboring beach. The highly active CaO catalyst from eggshells and seashells was prepared by the three steps, such as calcination, hydration, and dehydration treatment, which are reported in the literature [19, 29, 39]. This treatment was carried out in order to get the desired results. The empty shells were properly washed three times with hot water to eliminate any undesired substances that had clung to their surface, and then rinsed with distilled water. The egg and seashells were placed in a 105 °C hot air oven for 24 hours. For further processing, the dry shells were ground into small bits with a blender and sorted with 60 µm sieve mesh. Raw eggshells (Fig. 2a) and seashells (Fig. 2c) were calcined for 3 hours at 900 °C in a muffle furnace under static air conditions. CaCO3 of the shells was degraded into CaO at temperatures above 800 °C, as shown in reaction (1).

$$CaCO_{3(s)} \xrightarrow{900^{o}C} CaO_{(s)} + CO_2 \qquad (1)$$

CaO was refluxed at 60 °C for 6 hours, after which solid particles were filtered by vacuum filtration and dried overnight in a 105 °C hot air oven. The solid particles were further crushed, dehydrated by calcining at 600 °C for 3 hours to convert the hydroxide form to oxide form, and then placed in a desiccator to prevent moisture absorption. By calcination-hydration-dehydration treatment, pure CaO catalysts derived from eggshells (Fig. 2b) and seashells (Fig. 2d) were produced. CaO generated after treatments are designated as Eggshell-CaO-900-600 and Seashell-CaO-900-600 for identification and explanation purposes.

Fig. 2. Waste shells before and after the calcination-hydration-dehydration treatment. (a) waste chicken eggshell before the treatment; (b) waste chicken eggshell after the treatment; (c) waste seashell before the treatment; (d) waste seashell after the treatment

2.2. Catalyst Characterization

2.2.1. Scanning Electron Microscope (SEM) Analysis

The morphological analysis of the chicken eggshell derived CaO catalysts and seashell derived CaO were obtained using a Scanning Electron Microscope (SEM) with ZEISS EVO LS 15 Variable Pressure Environmental Scanning Electron Microscope. A forced beam of electrons was used in the instrument to captures the images of sample. While the electron interacts with atoms in the sample, various signals generate based on the topography information and composition details.

2.2.2. Powder X-Ray Diffractometer (XRD) Analysis

Powder X-ray diffraction (XRD) patterns of CaO catalysts produced from chicken eggshells and seashells were collected using a Bruker D8 Advanced Eco XRD equipment. Data were obtained across a range of 0 to 90° in two dimensions. Based on the XRD image, crystalline phase in the samples, crystalline size of the samples based on the generated peaks were analyzed. According to the identified peaks generate in the pattern was compared with the standard diffractogram of a calcium compound which exists in the Joint Committee on Powder Diffraction Standards (JCPDS) system.

2.3. Biodiesel production

2.3.1 Pre-treatment

The waste cooking oil (WCO) was collected from a Colombo, Sri Lanka, market as it was being prepared for disposal along with the rest of the city's garbage. Obtaining used cooking oil was followed by filtering it via a filter cloth to remove any suspended particles. The moisture content of feedstock was removed by preheating filtered cooking oil to 110 °C.

2.3.2 Transesterification

The procedure of transesterification was executed following the information disclosed by Miyuranga et al. [15] For transesterification, a 250 mL two-necked flask equipped with a thermometer was utilized. In the presence of a catalyst weighing 5 % by weight, 100 g of WCO with the molecular mass of 679.3 gmol-1 was mixed with methanol at a molar ratio of 1: 12. The mixture was agitated at 600 rpm for 120 minutes, and the reaction was carried out at 60 °C. After the reaction time was completed, the catalyst was isolated by centrifugation. The reaction mixture was centrifuged at 6000 rpm for 5 minutes, followed by the separation of glycerol and biodiesel in a funnel. Then, to boost the biodiesel's purity, it was placed on a hot plate at 110 °C for 20 minutes under magnetic stirring at 600 rpm to remove excess methanol. Using the formula outlined in equation, the biodiesel yield was calculated (2).

$$Biodiesel\ yield(\%) = \frac{Dry\ weight\ of\ biodiesel}{Dry\ weight\ of\ WCO} \times 100 \quad (2)$$

2.4. Catalyst regeneration

After transesterification was completed, the spent catalyst was extracted from the reaction mixture. To recover the spent catalyst, a centrifuge at 6000 rpm was used for 5 minutes. Recovered catalyst was refined by mixing it with methanol and heating it at 90 °C with a low-speed mixer (100 rpm) until the methanol evaporated completely.

2.5. Impact of co-solvent

As described by Miyuranga et al. [53], to achieve single phase formation, 20 vol.% acetone and WCO were pre-mixed for 3 minutes at 200 rpm on a magnetic stirrer. All other variables remained consistent during the period of the study. The reaction parameters were maintained at 12: 1 molar ratio of methanol to oil, 50 °C reaction temperature, 600 rpm, 120 minutes of reaction time, and 5 % catalyst loading.

2.6. Physiochemical properties of biodiesel

The laboratory analyzed the biodiesel's physical properties, including its flash point, density, kinematic viscosity, and chemical characteristic of acid value (ASTM D664-0). All samples were analyzed under identical conditions using the ASTM analytical procedure.

2.7. Statistical analysis

In reusability studies, the mean value of biodiesel generation from various CaO sources were examined statistically using Minitab 2016 software and one-way ANOVA. The level of confidence utilized was 95 %. As the null hypothesis, it was stated that the mean values of each data set differ significantly, whereas the alternative hypothesis indicated that there are no such disparities.

III. RESULTS AND DISCUSSION

3.1. SEM analysis

Fig. 3 and Fig. 4 display SEM images of CaO catalysts produced from eggshell and seashell, respectively. Fig. 4 depicts granular and irregular particles, showing the production of CaO particles comparable to those described in the literature [19, 25].

The clustered form of the particles may be the result of exposure to high temperatures during calcination. In addition, Fig. 3 resembles Fig. 4 in terms of clustering and irregularities, as both Egg shell and seashell were exposed to 900 °C during the calcination procedure. During the high-temperature calcination procedure, a substantial amount of CO2 gas escaped from the sample, revealing small particles in SEM images [19]. Based on Fig. 3 and Fig. 4, both exhibited similar porosity patterns that were greater than that of shell meal. The results were consistent with earlier investigations documented in the scientific literature [19, 47].

The interconnecting irregular-shaped particles depicted in Fig. 3 and Fig. 4 resulted from the heterogeneous mechanical qualities responsible for the catalyst's activity. Both irregularities exhibited uneven forms and microscopic imbedded particles of varying sizes and shapes.

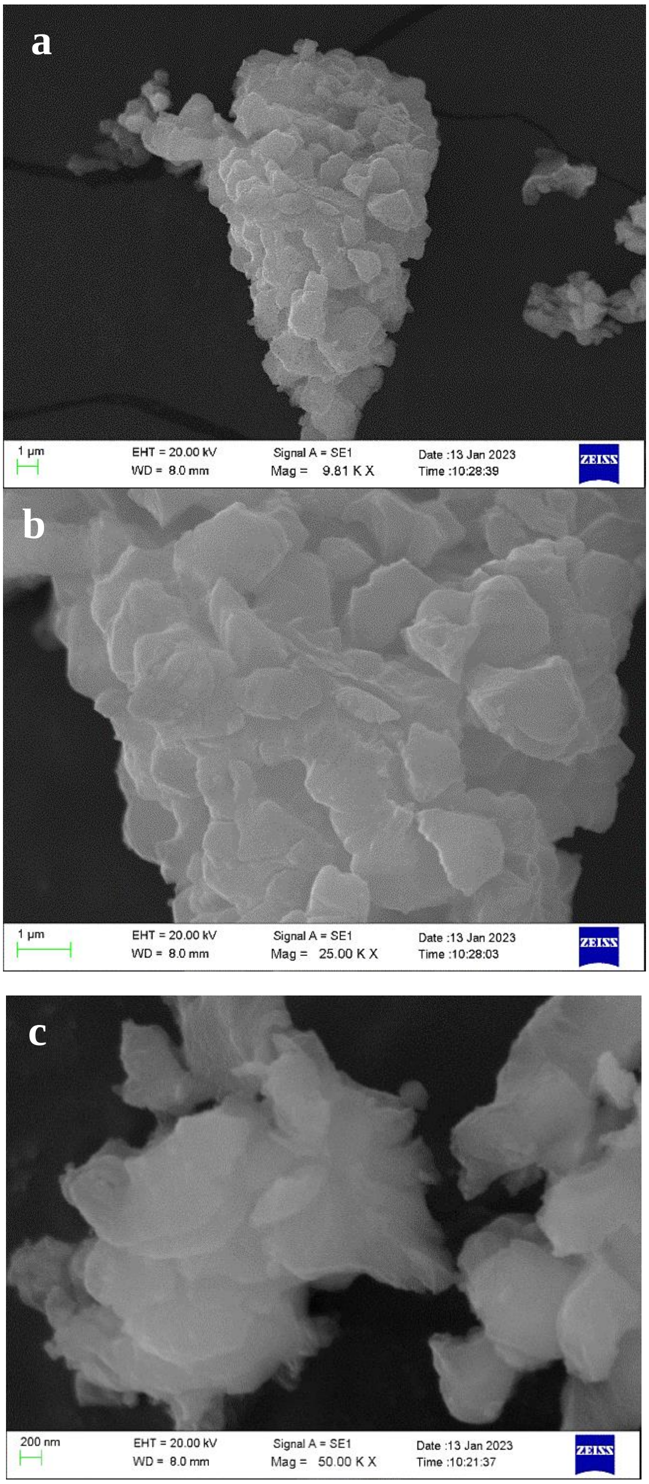

Fig. 3. SEM images of chicken eggshell derived CaO which were calcined at 900 °C, (a) Agglomerated larger particle, (b) particle with 25000 magnifications, (c) particle appearance with 50000 magnifications.

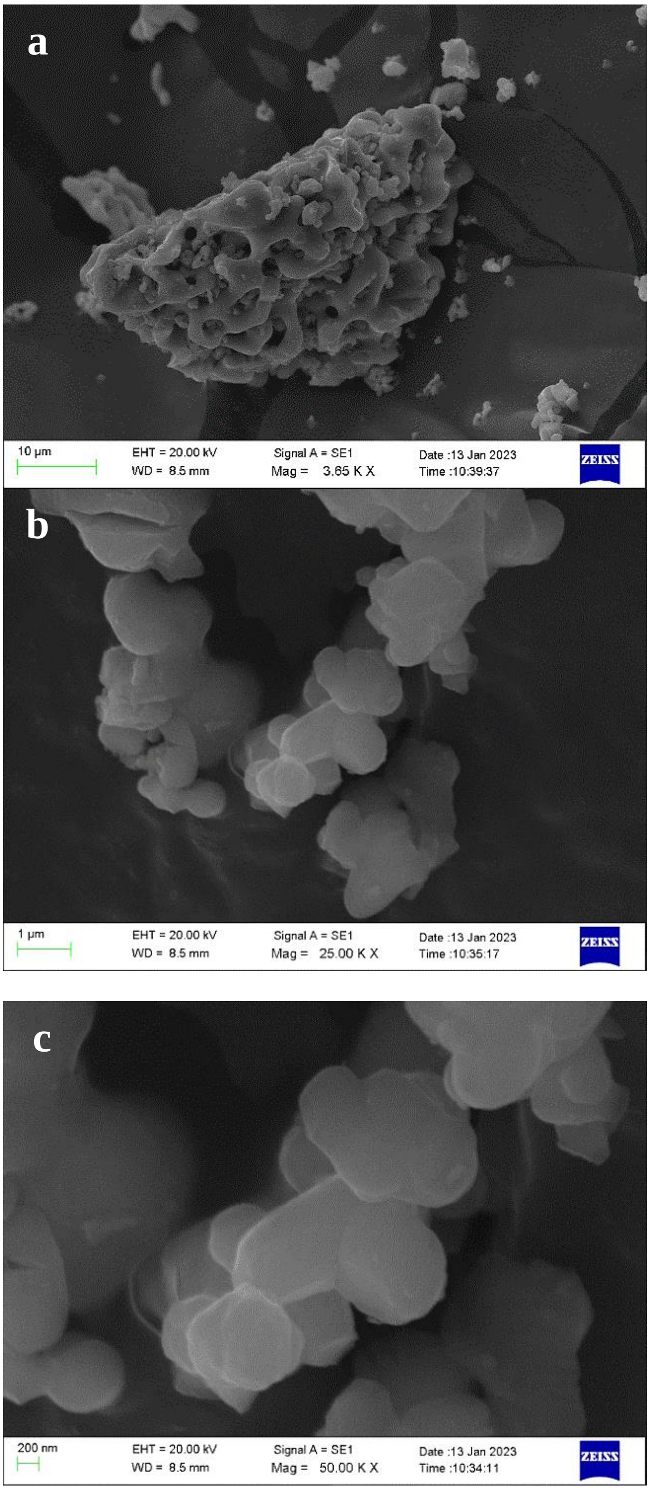

Fig. 4. SEM images of seahell derived CaO which were calcined at 900°C, (a) Agglomerated larger particle, (b) particle with 25000 magnifications, (c) particle appearance with 50000 magnifications.

Fig. 5 demonstrates the SEM analysis of CaO purchased from a chemical supplier. The SEM images depict the regular surface pattern of the CaO catalyst.

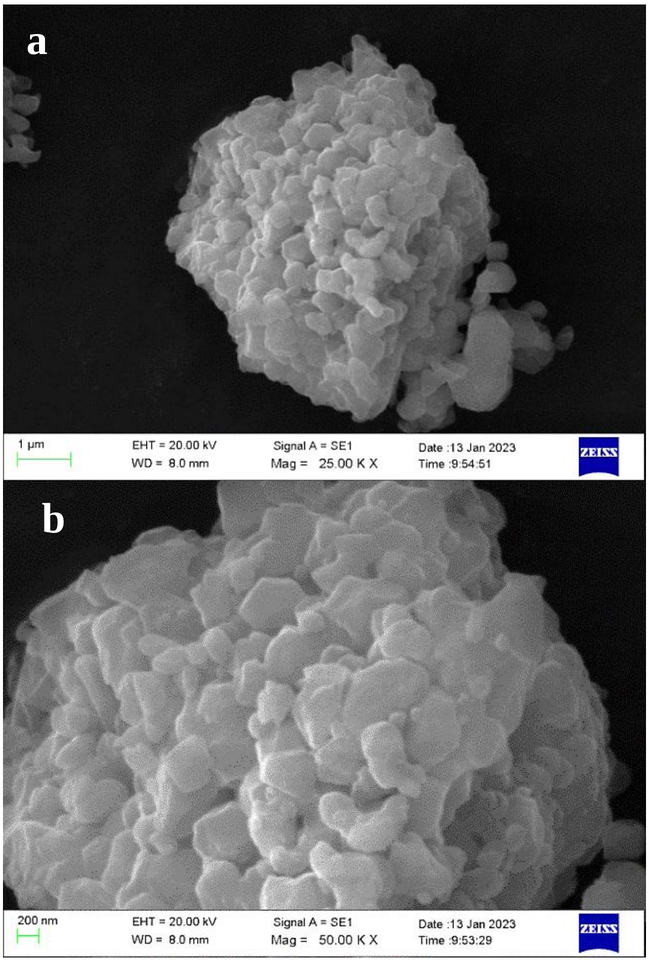

Fig. 5. SEM images of chemically produced CaO (a) Agglomerated larger particle, (b) particle appearance with 50000 magnifications.

3.2 XRD analysis

Fig. 6 and Fig. 7 exhibit the XRD patterns of eggshell- and seashell-derived CaO catalysts, respectively. According to Figure 6, intense peaks of 2θ values at 18º, 28.7º, 32.2º, 34.1º, 37.4º, 47.3º, 50.8º, and 53.8º correspond to the respective crystal planes for CaO produced from eggshell. Moreover, based on Fig. 7 for seashell derived CaO, the intense peaks of 2θ values at 32.2º, 37.4º, 53.9º, 64.1º, 67.4º, and 79.7º correspond to the respective crystal planes. Both lattice parameters are in excellent agreement with the respective standard values given in JCPDS 77-9574 for a sample of calcite CaO powder [22]. It is evident that both CaO and Ca(OH)2 were present in the calcined catalyst, with CaO being the predominant phase. However, CaO intensity diminishes over time since CaO reacts with water due to its hydroscopic nature when exposed to moisture and air to produce Ca(OH)2 [48]. In addition, commercial CaO was analyzed to obtain the XRD analysis report, which is presented in Fig. 8. The commercial CaO XRD study reveals intense peaks of 2θ values at 18.0º, 32.2º, 37.4º, 53.9º, 64.1º, 67.4º, 79.7º, which correspond to the relevant crystal planes. These intense peaks are identical to the CaO intense peaks produced from seashells.

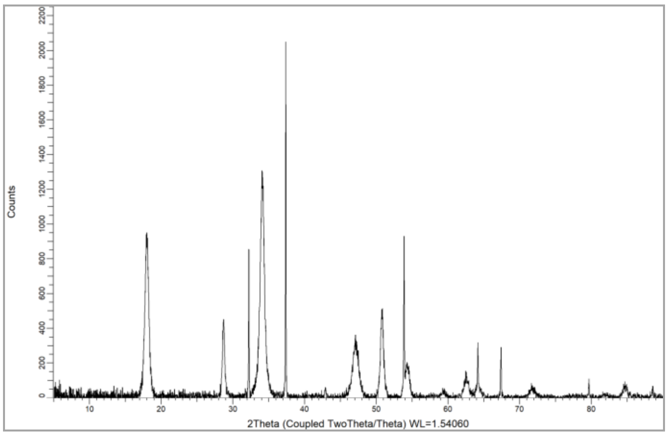

Fig. 6. XRD spectrum for CaO catalyst as prepared by waste eggshell.

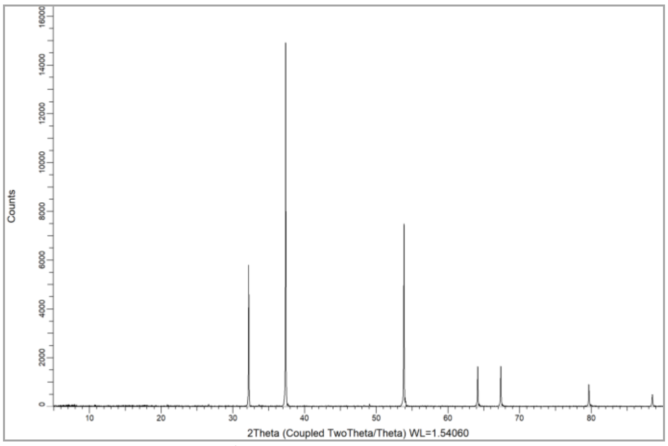

Fig. 7. XRD spectrum for CaO catalyst as prepared by waste seashell.

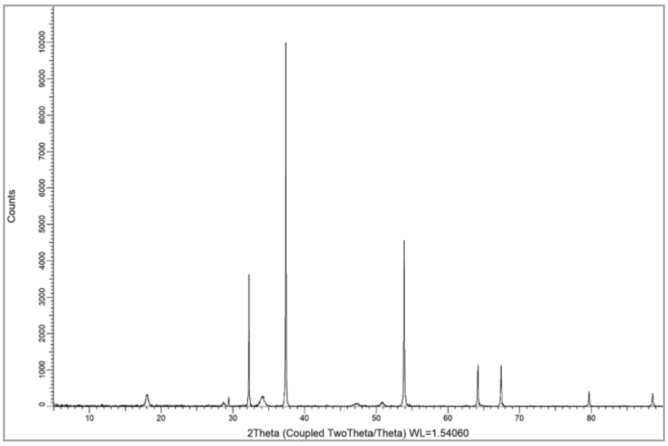

Fig. 8. XRD spectrum for commercially produced CaO

According to the XRD analyses depicted in Figures 5, 6, and 7, the shape of the sharp and strong diffraction peaks indicates that the samples are well crystallized and are in accordance with the corresponding standard values.

3.3. Biodiesel Production and Quality Analysis

Both derived CaO catalyst and chemical CaO were applied in the transesterification procedure used to produce biodiesel. In order to compare the yield of biodiesel derived from different sources of CaO, preparations were made under identical reaction conditions. The amount of biodiesel produced was utilized to determine and evaluate the diesel's quality in order to validate the performance of the catalyst. Table 1 details the quantity of biodiesel manufactured. According to Table1, commercial CaO yielded the highest yield compared to CaO obtained from eggshell and seashell. This is due to the fact that the quality and characteristics of the CaO produced from waste shells may differ from commercially available CaO, leading to lower biodiesel yields. Commercially available CaO is generally purer, containing fewer impurities that could disrupt the transesterification process, whereas waste shell derived CaO may contain impurities that lower the yield. Additionally, the surface area of waste shell derived CaO may be lower than that of commercially available CaO, leading to decreased catalytic activity and yield. Further-more, non-uniform particle size of eggshell derived CaO may also affect its catalytic activity and reduce the yield. However, additional research is necessary to determine the specific cause of the decreased yield. Hart and Onyeaka [49] reported that under identical calcination conditions at 900 °C, chicken eggshell-derived CaO exhibited a lower surface area (54.60 m2/g) than mussel shell-derived CaO (89.91 m2/g). The authors suggested that the lower intensity peaks observed in the XRD analysis for CaO could be attributed to a reduction in the crystallite size. Based on the XRD analysis, this study revealed that commercially available CaO possessed the smallest particle size, followed by seashell-derived and chicken eggshell-derived CaO. This finding was further supported by SEM analysis. Additionally, the XRD analysis revealed that the presence of Ca(OH)2 peaks in egg-shell-derived CaO was greater than that observed in seashell-derived CaO, indicating a lower yield for the latter.

TABLE 1. Biodiesel Yield for Eggshell derived CaO; Seashell derived CaO, Chemical CaO

Catalyst type	Biodiesel yield (%)
Eggshell CaO	65
Seashell CaO	75
Commercial CaO	84

3.4. Catalyst reusability

Several attempts were made to assess changes in biodiesel yield based on the catalyst reuse attempts.

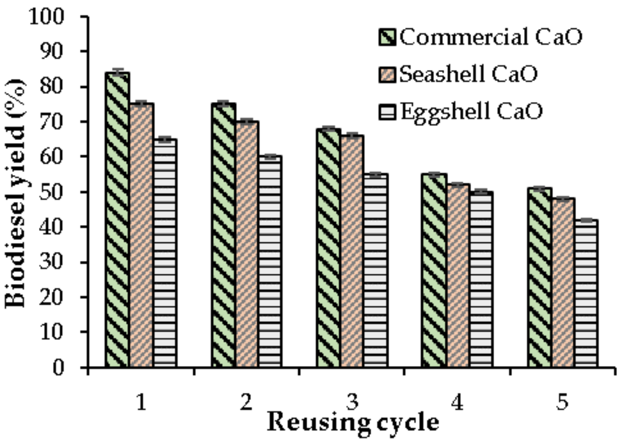

Fig. 9. Biodiesel Yield variation with reusing attempts

Based on Fig. 9, as the reuse attempts increased, the yield of biodiesel steadily de-creased from initial values of 65 %, 75 %, and 84 % for eggshell-derived CaO, sea-shell-derived CaO, and commercial CaO, respectively. Eggshell and seashell-derived CaO produced less biodiesel compared to commercial-grade CaO, primarily due to contaminants in the derived CaO, as well as factors such as surface area and particle size. Conversely, during the calcination process, CO2 leaves the material and creates surface cavities, which were identified in Fig. 4a, that reduce the crystallite size, consequently in-creasing the effective reacting contacting area for the biodiesel reaction. CaO derived from eggshells and seashells produced less biodiesel than commercial-grade CaO. The ANOVA test also verified that the biodiesel yield was lower for eggshell and seashell derived CaO compared to commercial CaO. yield for each CaO samples decrease as the number of re-use attempts increases.

Due to active metal leaching into the methanolysis solution, thereby reducing the amount of Ca2+ remaining for the second reaction by reducing the solution's basicity, as well as catalyst surface poisoning by trace elements such as glycerides, H2O, and other intermediate compounds [50], the yield of biodiesel was reduced during recycling attempts. Therefore, foreign components will cover the active surface area and inhibit the process. In addition, catalyst particles were agglomerated, and the size of the agglomeration grew as the number of recycling efforts increased [51].

3.5. Acetone as co-solvent

During the biodiesel synthesis process with heterogeneous catalysts, a three-phase system consisting of oil, alcohol, and catalyst forms in the reaction mixture. In general, the reaction rate in the presence of a heterogeneous catalyst is slower than in the presence of a homogeneous catalyst due to the mass transfer constraint that arises when using a heterogeneous catalyst, as mass transfer between the phases becomes a crucial factor in deter-mining the reaction rate [52]. Consequently, it may take longer than normal to complete the response. Therefore, the influence of the co-solvent in a heterogeneous catalytic reaction system is an essential area of study, as co-solvents can overcome mass transfer resistance by facilitating a single-phase system rather than a three-phase system [53]. Acetone was employed as a co-solvent in this study to analyze the behavior of CaO catalysts in terms of biodiesel synthesis over a certain time period.

Fig. 10 depicts the increase in biodiesel yield in the presence of acetone for all CaO catalyst types compared to non-co-solvent systems. It was confirmed that, regardless of the type of CaO-derived source, there was a mass transfer resistance in transesterification that was overcome by the addition of acetone.

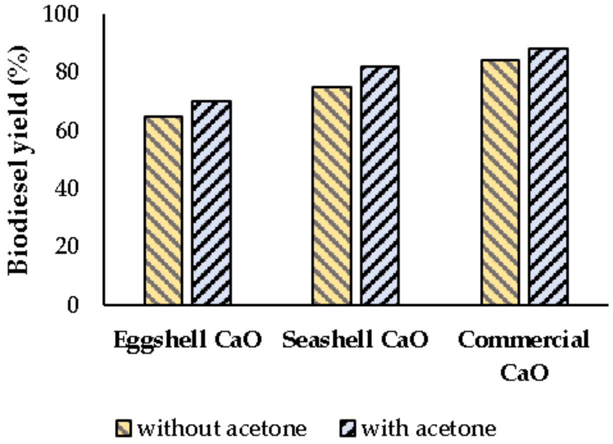

Fig. 10. Biodiesel Yield variation with acetone

3.6. Quality of the biodiesel

To determine the efficacy of the catalyst for biodiesel generation, the biodiesel quality of each of the three cases was examined. Using the ASTM standard values, the density, kinematic viscosity, flash point, calorific value, free fatty acid content, and cetane index were evaluated and compared. These values are compiled and shown in Table 2.

TABLE 2. Analysis of physical and chemical properties of biodiesel and petroleum diesel

Parameter	Test Method	Eggshell derived CaO	Seashell derived CaO	Commercial CaO	Petroleum diesel [a]	EN14214 [b]
Density (15ºC, kg/m3)	ASTM D 1298- 99	880	884	888	NA	860-900
Flash Point (ºC)	ASTM D 3278-96	115	118	109	>65	>101
Kinematic Viscosity at (40ºC, cSt)	ASTM D 445 - 01	3.792	3.798	3.794	3.0 - 8.0	3.5 - 5.0
Moisture content (wt. %)	ASTM D 4377 - 00	0.08	0.06	0.04	NA	<0.05
Gross calorific value moisture free basis (MJ/kg)	ASTM D 240 - 06	38.7	39.2	39.6	41.8	NA
Total acid number (mg KOH/g)	ASTM D 664/18	0.22	0.26	0.20	<0.1	<0.5
Cetane index	ASTM D - 91	39.1	38.8	39.2	>49	>51

[a] Tested by the Petroleum and Lubricant Testing Laboratory, Industrial Technology Institute (ITI), Colombo 07, Sri Lanka
[b] [54]

The biodiesel's physical qualities are consistent with the standard biodiesel parameters. Both eggshell and seashell-derived catalysts can be used to create methyl ester via transesterification. As their flash point and viscosity are within the specified range, these biodiesels can be blended with fossil diesel for use in power generation and automobiles.

IV. CONCLUSION

In this study, the feasibility of creating a CaO heterogeneous catalyst from waste chicken eggshells and waste seashells was examined. It was determined that both waste chicken eggshell and seashell have a greater likelihood of producing CaO with excellent catalytic activity for waste cooking oil methanolysis. SEM and XRD analysis confirmed that both waste chicken eggshell and waste seashell contained CaO after 3 hours of calcination at 900 °C. To evaluate waste based CaO activity on transesterification, commercial CaO was used and biodiesel output under identical reaction conditions was examined. Commercial CaO has the highest biodiesel yield, followed by CaO derived from waste chicken eggshells and waste seashells. Under identical reaction conditions, commercial CaO was able to produce 84 % biodiesel while waste chicken eggshell derived CaO and waste seashell derived CaO produced 65% and 75% respectively. The transesterification was carried out at 600°C with a stirring speed of 600 rpm. The molar ratio of methanol to oil was maintained at 12 to 1, and the reaction time was set at 120 minutes with a 5 wt.% catalyst concentration. In addition to investigating their catalytic activity under fresh conditions, five attempts were made to determine their reusability. It was determined that the subsequent ability decreases as the number of attempts increases in the same manner as the initial findings. Adding acetone as a co-solvent increased the biodiesel yield for all CaO catalysts, regardless of their source material, according to the findings of the study. In addition, the physiochemical parameters of the biodiesel produced from each source con-firmed that the values met the ASTM requirement.

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