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Production of Biodiesel from Waste Cooking Oil using Calcium Oxide Nanocatalyst

Md. Saiful Islam[1]*, Md. Moynul Islam[1], Afia Mariam[2] and Sadi Muhammad[2]

[1]*Department of Chemistry, Bangladesh Army University of Engineering and Technology, Qadirabad Cantonment, Natore-6431, Bangladesh*
[2]*Department of Civil Engineering, Bangladesh Army University of Engineering and Technology, Qadirabad Cantonment, Natore-6431, Bangladesh*

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Abstract

The production of biodiesel from waste cooking oil (WCO) represents a sustainable and environmentally friendly approach to waste management and renewable energy generation. This study explores the use of calcium oxide (CaO) nanocatalyst in the transesterification process of converting WCO into biodiesel. CaO nanocatalyst is favored for its high catalytic activity, low cost, and environmental compatibility. In this study, calcium oxide (CaO)-based nano-catalyst was prepared through calcination process using local waste eggshells to produce biodiesel. The nano-CaO catalysis impact was investigated in a two-step transesterification chemical reaction from waste cooking oil (WCO) to biodiesel. Transmission electron microscope (TEM), Fourier transform infrared (FT-IR) spectroscopy and X-ray diffraction (XRD) techniques were utilized to confirm the CaO nano-catalyst. It was discovered that the CaO catalyst made from eggshell waste was an efficient, stable, and reasonably priced heterogeneous catalyst to be highly effective for the manufacture of biodiesel. The production of biodiesel was confirmed through FTIR analysis. Moreover, the biodiesel properties such as calorific value, specific gravity, acid value, density, flash point, kinematic viscosity, ash content, water content and cetane number were investigated and found to be similar with the EN 14214 biodiesel standard. It is expected that producing biodiesel using eggshell as a heterogeneous catalyst offers an economical and sustainable method of producing green fuel. This study underscores the potential of CaO nanocatalyst in enhancing the efficiency and sustainability of biodiesel production, offering a viable solution for waste management and energy production.

Keywords: *Biodiesel; Waste Cooking Oil; Transesterification; Fourier-Transform Infrared Spectrometer; Sustainable Energy.*

Introduction

The rising demand for sustainable and environmentally friendly energy sources has intensified the search for alternative fuels to replace conventional fossil fuels. Biodiesel, derived from renewable biological resources, has emerged as a promising candidate due to its biodegradability, lower emissions, and compatibility with existing diesel engines. Fossil fuel consumption is significantly reducing its supply and producing high concentrations of air pollutants (Wan *et al.,* 2015). Due to the lack of fossil fuels and the rising global need for fuel, research on renewable fuels is becoming more and more interesting. A number of alternative fuel types have recently been taken into consideration for production and quality enhancement.

Given that it is renewable, biodegradable, complies with environmental regulations, and has combustion characteristics like those of fossil diesel fuel, biodiesel fuel is a promising substitute fuel. It displays a flash point that is higher than that of

*Corresponding author e-mail: mdsaifulislam16@bauet.ac.bd regular diesel. Hence, it has been demonstrated to be a green fuel with nearly low CO_2 emissions (Mansir *et al.,* 2016). Fatty acid methyl esters (FAMES), which are the building blocks of biodiesel, are typically made from animal and vegetable fats. Since the cost of the feedstock adds to the whole project cost, the primary obstacle to the commercialization of biodiesel is the high production costs associated with all edible vegetable oils. Low-cost vegetable oils, like waste and nonedible oil, are used as feedstocks; nevertheless, because of their restricted output, there are worries about their sustainability (Amani *et al.,* 2014). Additionally, waste oils and fats, or leftover cooking oils, are useful as feedstock and may be purchased for around half as much as virgin oil (Mahesh *et al*., 2015). Furthermore, the biodiesel plant's use of waste fried cooking oil (WFCO) as a reactant aid in the disposal of this waste and lowers the cost of producing feedstock (Amani *et al*., 2014). Therefore, the basic goal of engineering designers is to create an efficient process that yields a product of commercial quality at the best possible pace of production (Boey *et al*., 2011).

Triglyceride feedstock and methanol undergo a catalytic transesterification reaction to make biodiesel. For the manufacturing of biofuel, several oils, including maize oil, soybean oil, sunflower oil, coconut oil, tobacco seed oil, palm oil, sugarcane oil, and animal fats, have been utilized extensively for a time (Kouzu and Hidaka, 2012). An alcohol molecule is employed to shift the equilibrium to the FAMEs side. This excess encourages the transesterification reaction and makes it easier for products to desorb from the catalyst surface and replenish the active catalytic sites (Kumar *et al*., 2014). From the perspective of economics and available resources, methanol is found to be the most suitable alcohol for this operation. Additionally, methanol offers a faster rate of reaction than both propanol and ethanol (Lam *et al*., 2010). The advantages of the catalytic transesterification reaction over the non-catalytic approach have been demonstrated by the application and evaluation of several catalysts in the biodiesel synthesis process (Yaakob *et al*., 2012). There are two types of catalysts used in transesterification: homogeneous and heterogeneous. The simplest procedure is homogeneous transesterification, however because the reaction mixture (reactant, catalyst, and product) is homogenous, it includes the complexities of product separation and purification. Additionally, it tends to produce emulsions in combination and is more prone to form soap, which requires more washing. High production costs are the outcome of all these process intricacies. Due to their ability to address certain limitations inherent in homogeneous catalyst processes, heterogeneous catalysts are currently preferred for biodiesel production. These catalysts offer extended lifetimes, high selectivity, significant resistance to water and free fatty acids, and robust catalytic activity (Gui *et al*., 2008).

Different acidic and basic solid catalysts have been developed over the past ten years for use in biodiesel products (Amani *et al*., 2014; Gui *et al*., 2008; Guana and Kusakabe, 2012). Because of its great activity, affordable production costs, and accessibility, CaO-based catalyst is one of the biodiesel production methods that has received the most research (Tan *et al*., 2010). Furthermore, waste materials containing $CaCO_3$, such as eggshells, mollusks, and cockles, can be used to produce calcium oxide, a high basic catalyst that is noncorrosive. Using CaO as a catalyst not only makes producing biodiesel more affordable, but it also provides an excellent means of recycling natural mineral resources in a way that complies with environmental rules, turning the catalytic biodiesel process into an environmentally friendly fuel production method (Piker *et al*., 2016; Viola et al., 2012). Tan et al.'s study used a 12:01 methanol to oil ratio, a 3-hour reaction period, and 8% of CaO per oil mass to manufacture a CaO catalyst from ostrich eggshells and apply it to the transesterification of soybean oil (Tan *et al*., 2010). With the calcined shells, they were able to attain a 96% yield. Several research have examined the use of CaO as a catalyst and have shown successful production in the past ten years. In a similar procedure using fresh soybean oil, Piker *et al.* produced 97% of FAMEs at a molar ratio of 1:6 and 5.8% of CaO catalyst (Piker *et al*., 2016). Transesterification of WFCO was carried out by Viola *et al*. using three distinct catalysts, CaO, SrO, and K_3PO_4 (Viola *et al*., 2012). Results included 92% in three hours, 5% CaO per oil (m/m) at 65 °C, and a methanol to oil molar ratio of 6:1. The transesterification of WCO was investigated by Li *et al*. utilizing calcined scallop shells as a catalyst at 65 °C and a loading amount of 5 wt%. They produced an 86% yield (Li *et al*., 2018). Pandit and Fulekar reported an 86.41% yield by trans esterifying dry biomass into biodiesel using leftover chicken eggshells as a CaO nanocatalyst (Pandit and Fulekar, 2019). In order to generate biodiesel, Teo *et al*. created a nanocatalyst from Gallus eggshell waste and used a neural network model to forecast a 97% yield at 60 °C and five hours of reaction time (Hwa *et al*., 2016).

The aim of this study is to synthesize a highly active heterogeneous CaO nano-catalyst with low particle size and high surface area, optimizing its application in biodiesel production from waste cooking oil. To achieve this, fresh eggshells were calcined to produce CaO nano-catalyst, enhancing the transesterification reaction rate (Yee *et al*., 2011; Noshadi *et al*., 2012; Pradhan *et al*., 2012). The study focuses on optimizing key reaction parameters, including catalyst concentration, reaction time, and methanol ratio, to maximize biodiesel yield. Additionally, the catalytic and morphological properties of the synthesized CaO nano-catalyst were characterized using appropriate analytical techniques. The produced biodiesel was then evaluated against conventional diesel standards to ensure high-quality fuel properties.

Materials and Methods

Materials

In Dayrampur, Natore, Bangladesh, waste eggshells and waste cooking oil (soybean oil) were gathered from neighborhood restaurants (shown in figure 1). A reference catalyst was created using laboratory-grade ingredients, such as calcium oxide (UNI-CHEM chemical reagents, CAS.NO.1305-78-8, M.W.56.08), KOH, methanol (MeOH) >99% pure, double distilled deionized water, H_2SO_4 95-97%, and KOH.

Preparation CaO nano-catalyst

To create the CaO nano-catalyst, the eggshells were calcined, hydrated, and then dehydrated to produce a fine powder. In short, about 1 kg of eggshells were properly cleaned to eliminate all contaminants and the edible portion. They were then rinsed with distilled water and dried in a hot air oven at 105°C for a full day. After being blended into a fine powder, the eggshells were sieved using an 80μm mesh screen. After that, the eggshell powder in the micron size was calcined for 2.5 hours at 900 °C in a chamber furnace. The powdered eggshell calcium carbonate broke down into calcium oxide and carbon dioxide at 850 °C, as seen in the reaction (Eq. 1):

After refluxing the eggshell derived CaO in water at 60 °C for six hours, the solid particles were filtered and dried for an entire night at 120 °C in a hot air oven.

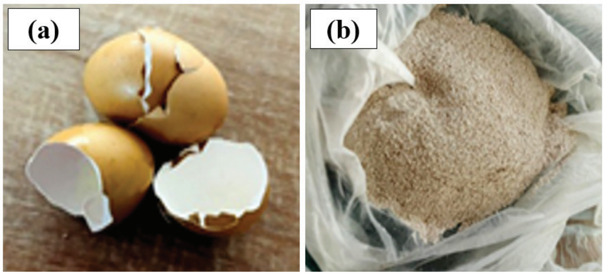

Figure 1. (a) Eggshell and (b) Powder of eggshell

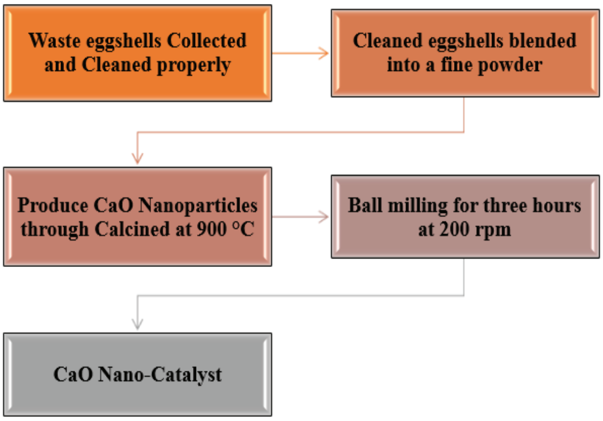

Figure 2. Flow chart of synthesis of CaO nano-catalyst

The solid powder was then processed even more in a planetary ball mill for three hours at 200 rpm. To convert the hydroxide form of the powder into oxide form, it was then calcined for three hours at 600 °C. As a result, eggshells that underwent the calcination, hydration, and dehydration process were used to create a highly active CaO nano-catalyst. The overall synthesis of CaO nano-catalyst was shown by flow chart in Figure 2.

Characterization of CaO nano-catalyst

The chemical structure of the recently produced CaO nano-catalyst was examined by evaluating powder samples using a Fourier Transform Infrared (FT-IR) Spectrophotometer (Model 100 series, Perkin Elmer) within a range of 4000 to 280 cm^{-1} region. A Transmission Electron Microscope (TEM) (Hitachi H-7100, Japan) was used to measure the shapes and sizes of the CaO nano-catalyst. The crystalline properties of the nano-catalyst were investigated using nickel-filtered Cu Kα (= 1.542 Å) radiation and an X-ray diffraction (XRD) test from a Shimadzu 6000 X-ray machine.

Biodiesel production

The newly synthesized CaO nano-catalyst was used to produce biodiesel from used cooking oil by following conventional laboratory techniques. The picture below provides a schematic representation of the step-by-step procedure. The next sections cover the two main processes in the synthesis of biodiesel: transesterification with CaO acting as a nano-catalyst and acid pretreatment.

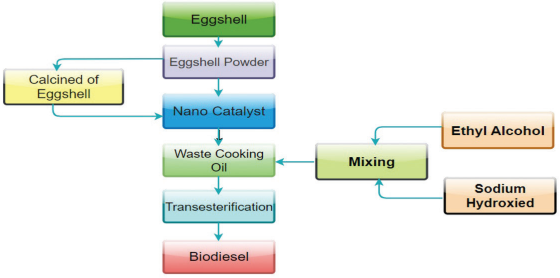

Figure 3. Flow chart of biodesel production

Acid pretreatment

Waste cooking oil pretreatment was carried out according to the methods discussed by Anr (Anr *et al.*, 2016). Using H_2SO_4, an acid pretreatment was carried out. Hotter than 100°C was used to extract the water content from filtered 25 mL of waste cooking oil (WCO). A mixture of H_2SO_4 and methanol (MeOH) was prepared by heating a three-neck flask equipped with a water-cooled condenser to 50°C. The alcohol and catalyst were added in a proportionate ratio by volume to the WCO. The WCO sample was then added to the mixture, and the liquid was heated to 50°C and stirred at 200 rpm using a magnetic stirrer. After the reaction, the contents were allowed to settle for one hour. The resulting oil was washed three times with distilled water, and then heated to evaporate any residual water.

Transesterification using CaO nano-catalyst

For catalyzed transesterification, the oil that had undergone acid pretreatment was combined with a highly active CaO nano-catalyst. The pretreated WCO was mixed with a mixture of powdered CaO nano-catalyst (0.01-0.03 w/w) and methanol (4-6 mol.) according to a ratio. The reactants were left to react for 180 minutes at 60 °C with a 500 rpm stirring speed. Using an orbital shaker set to 250 rpm and room temperature, the mixture was shaken for two hours before being left to settle for 24 hours. Glycerol was removed from biodiesel, and distilled water was used to wash the methanol-water layer. To obtain pure biodiesel, the filtered biodiesel was heated to 100 °C while being constantly swirled with a magnetic stirrer.

Biodiesel analysis

To determine the presence of FAME absorption bands in the biodiesel samples, a FTIR spectrophotometer (Model 100 series, Perkin Elmer) was used over a range of 4000 to 750 cm^{-1}. In addition, the calorific value, kinematic viscosity, density, specific gravity, flash point, ash content, acid value, water content, and cetane number of the biodiesel fuel were ascertained using standard testing equipment, such as the 6400 Automatic Isoperibol Calorimeter, Parr Instruments USA; the Fuel Integrity Test Kit (Stanhope-Seta, UK, built on ASTM D2624, ASTM D3828 standards); and others.

Results and Discussion

Analysis of CaO nano-catalyst

Fourier transform infrared spectra

Together with a few faint bands at around 3640 cm^{-1}, 1445 cm^{-1}, and 877 cm^{-1}, the IR spectra of the CaO nano-catalyst sample revealed a large broad absorption band at wave numbers <600cm^{-1}, which was attributed to the Ca–O group stretching vibrations. The medium infrared band of Ca–O lattice vibrations at wave numbers near 400 cm^{-1} and the strong infrared absorption band at roughly 290 cm^{-1} are clearly indicative of CaO (Mcdevitt and Baun 1960, Zaki *et al*., 2006). But due to the hydroxyl (O-H) and carbonyl (C=O) groups' asymmetric bending vibration modes, a weak absorption band emerged at around 3640 cm^{-1} and at a medium 1445 cm^{-1} (Zaki *et al*., 2006). This is mostly caused by ambient moisture and CO_2 absorbing into the surface of the calcined catalyst, which ultimately results in the creation of $Ca(OH)_2$ and $CaCO_3$.

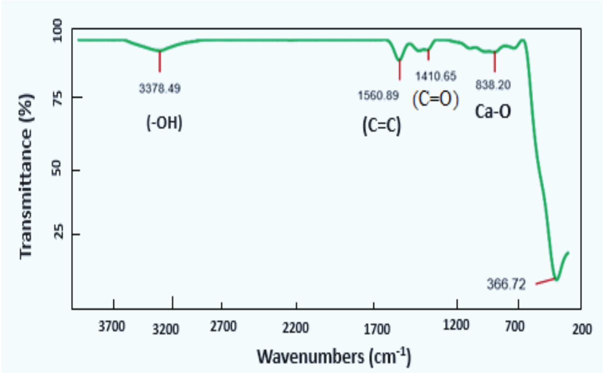

Figure 4. FTIR spectrums of CaO nano-catalyst [Amr *et al*., 2016]

Transmission electron microscopy

The crystalline nature, size and morphology of the produced CaO nano-catalyst were assessed using TEM examination. As seen in Figure 5, TEM images of the CaO nanoparticles revealed a (a) rod and (b) spherical form. The mean diameters of the spherical and rod-shaped CaO nanoparticles, as determined by TEM images, were 66.2 nm and 548.7 nm, respectively. This is consistent with findings from earlier research (Amr *et al.,* 2016). The CaO nanoparticles were discovered to have aggregated from the TEM image, most likely because of their large specific surface area and strong propensity to form aggregates.

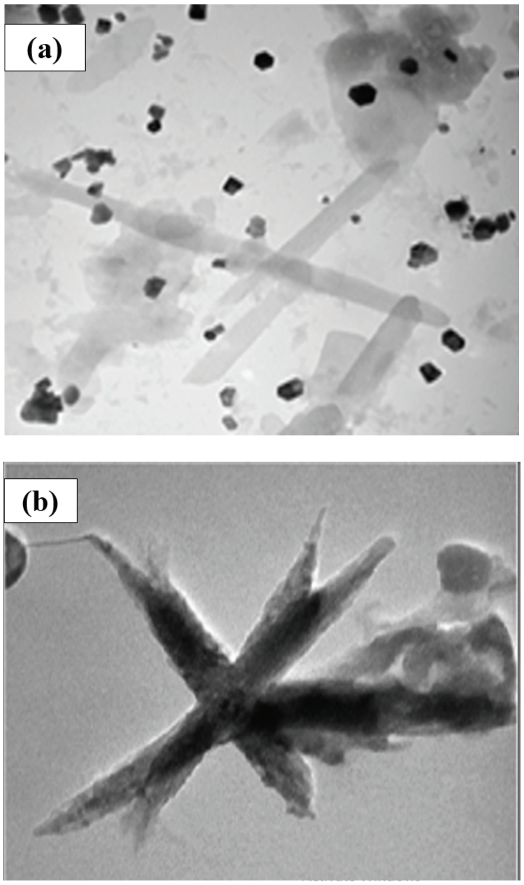

Figure 5. TEM images of CaO nano-catalyst (a) Rod shape and (b) Spherical shape (Amr *et al*., 2016)

XRD analysis

The XRD pattern of the CaO nanoparticles is shown in Figure 5. At planes (0,1,2), (1,0,0), (1,0,4), (2,0,0), (2,0,0), (2,2,0), (3,1,1), and (2,2,2), it displays a clear diffraction pattern of CaO and miller indices values (h, k, l). These values correspond to 2θ values of 23.16°, 29.54°, 36.1°, 39.56°, 43.3°, 48.64°, 57.62°, and 60.84°, respectively. These patterns correspond to the standard card (JCPDS file No. 00-037-1497) of a calcite CaO powder sample. The crystalline diameters of the CaO nanoparticles were calculated and found to be 66.3 nm; similar results have been published in the literature by other researchers (Mirghiasi *et al*., 2014; Ren and Bruce, 2012).

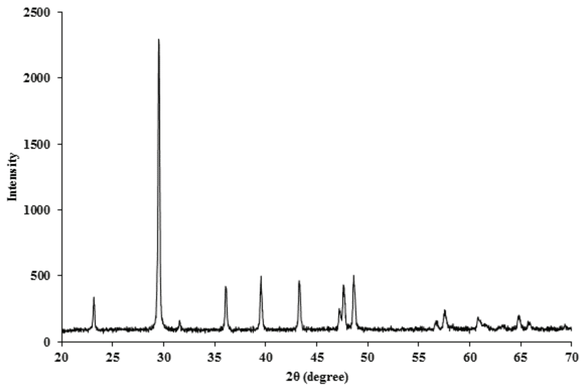

Figure 6. XRD pattern of CaO nano-catalyst as prepared from eggshells

The CaO nano-catalyst used in this work is characterized as "highly active" due to its small particle size and its derivation from eggshells. This might reduce the disadvantages of catalyst diffusion, create better flow channels, speed up reactions between the reactants, and ultimately enhance the catalytic activity of nano-CaO to improve the kinetics of the transesterification reaction.

Analysis of biodiesel

Fourier transform infra red

FTIR spectroscopy was used to monitor the transesterification processes between rice bran and sunflower oils. In all biodiesel IR spectra, the region of 1425–1447 cm^{-1} for CH_3 asymmetric bending and 1188–1200 for O-CH_3 stretching amply demonstrated the conversion of waste cooking oil into biodiesel. In the same way, the IR spectra of rice bran and sunflower oils only showed O-CH_2 groups in the glycerol (triglyceride, diglyceride, and monoglyceride moieties) in the 1370–1400 cm^{-1} range. This is in line with what other researchers has found (Dube et al., 2004; Siatis et al., 2006). For the -OH functional group, strong absorption bands were seen at 2852 cm^{-1} and 2930 cm^{-1} peak positions. The material's identity as biodiesel was confirmed by the presence of O-H absorption peaks. Moreover, the biodesel showed absorption spectra with peaks at 1000 cm^{-1} and 1320 cm^{-1} due to the C-O group (Dube et al., 2004).

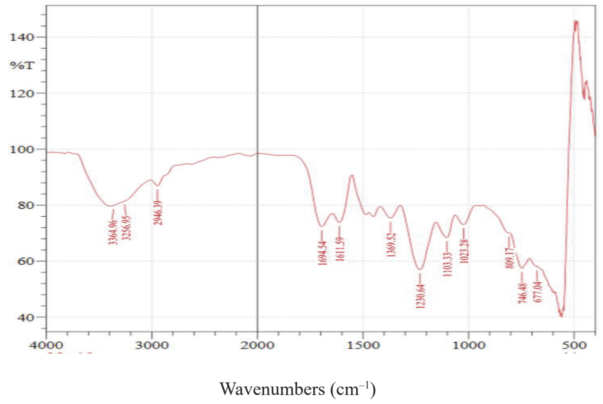

Figure 7. FTIR spectrum of biodiesel as produced.

Biodiesel properties

The test findings for several biodiesel fuel parameters, including water content, cetane number, density, ash content, flash point, kinematic viscosity, and density, are summarized in Table 1. The parameters were measured and compared against the EN 14214 biodiesel standard to ensure compliance with established quality benchmarks. The experimental results demonstrate that the biodiesel samples meet this standard, confirming their suitability for use as a renewable fuel source. The measured water content in the samples was within acceptable limits, ensuring the fuel's stability and longevity. Water content in biodiesel is a critical factor, as excessive moisture can lead to microbial growth and corrosion within the fuel system. The cetane number, which indicates the ignition quality of the fuel, was also within the required range, suggesting good combustion performance and engine startability. Density and specific gravity are important for understanding the fuel's mass-to-volume ratio, which influences energy content and combustion efficiency. The biodiesel samples exhibited appropriate density values, aligning with the EN 14214 standards. Ash content, indicative of inorganic residue after combustion, was minimal, highlighting the cleanliness of the fuel and its potential to reduce engine wear and emissions

Table 1. Fuel properties of synthesized biodiesel

Properties	Yield of Biodiesel	EN-14214 Biodiesel Standard Limit
Density (kg/m³)	895	860 - 900
Specific gravity (g/mL)	0.882	0.86 - 0.90
Kinematic viscosity (mm²/s)	4.4	3.5 - 5.0
Calorific value (MJ/kg)	35.35	-
Acid value (Mg KOH/g)	0.28	< 0.5
Ash content (g/100g)	< 0.01	< 0.01
Water content (Wt%)	0.046	< 0.05
Cetane number	51	> 47
Flash point (°C)	167	> 101

The flash point of the biodiesel samples was significantly high, indicating safety in handling and storage by reducing the risk of accidental ignition. Kinematic viscosity, which affects fuel flow and atomization during injection, was measured to be within the optimal range, ensuring efficient combustion and engine performance. The fuel properties of the synthesized biodiesel samples, as indicated by the test results, confirm that biodiesel is a viable alternative to conventional diesel. Its compliance with the EN 14214 biodiesel standard underscores its potential as a renewable fuel source. The positive test findings support the suitability of biodiesel for future energy needs, contributing to the reduction of dependence on fossil fuels and promoting environmental sustainability.

Conclusions

This study demonstrates that natural resources can be effectively converted into biodiesel, offering an environmentally friendly alternative to traditional diesel fuel. The potential for biodiesel to serve as a substitute during a fossil fuel shortage highlights its importance for future energy security. Utilizing local eggshells as a CaO nano-catalyst for the transesterification of waste frying soybean oil has been proven to be a successful and sustainable approach. The eggshell powders were rigorously analyzed using FTIR, TEM, and XRD to determine optimal calcination parameters, which were verified to be at 900 °C for three hours. The resulting catalyst showed exceptional efficiency in converting WCO, containing 96.22% fatty acids, into biodiesel without the need for esterification pretreatment. Despite a slight decrease in biodiesel yield after multiple uses of the catalyst due to side reactions and product adsorption, the CaO catalyst remained effective. The fuel properties of the produced biodiesel closely matched the standards outlined in EN 14103, confirming its quality and suitability as a renewable fuel source. Therefore, CaO derived from eggshell waste is an efficient, stable, and cost-effective heterogeneous catalyst for biodiesel production. Future research should focus on improving the reusability and regeneration of the CaO catalyst to enhance yield consistency over multiple cycles. Additionally, scaling up the production process and conducting comprehensive environmental impact assessments will help determine the feasibility and benefits of large-scale biodiesel production using eggshell-derived catalysts. Exploring the use of other local waste materials for catalyst production could further diversify and optimize the biodiesel production process.

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Declaration

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. The authors also declare no conflict of interest.

Author's credit

Md. Saiful Islam: Conceptualization, supervision and funding acquisition. Md. Moynul Islam: Project administration and review & editing. Afia Mariam: Investigation, data curation and writing original draft. Sadi Muhammad: Formal analysis and writing original draft.

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