Original article

Transesterification of waste cooking oil using biogenic calcium oxide nano catalyst

Transesterificación de aceite de cocina usado utilizando nanocatalizador de óxido de calcio biogénico

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ABSTRACT

Introduction: With advent of sustainable development and green chemistry, valorisation of waste is highly desirable. In this context, conversion of waste cooking oil into biodiesel through transesterification has been proposed.

Objective: This study intends to sustainably transform waste cooking oil into biodiesel using biosynthesized calcium oxide nanoparticles as nano-catalysts during the transesterification process.

Methods: The nano-catalyst was obtained from waste egg shells and utilized further in transesterification of waste cooking oil to obtain biodiesel. The one-pot transesterification was conceded to study the impact of changing reaction parameters on the reaction yield

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including temperature, concentration of nano-catalyst, methanol-oil molar ratio, and contact time.

Results: Fourier Transform Infrared Spectroscopy (FTIR) analysis revealed the presence of various functional groups while Gas chromatography mass spectrometry (GCMS) analyses identified the methyl ester groups extant in the biodiesel. The optimized yield of biodiesel (94.35%) was obtained after 4 hours at a temperature of 60 ºC utilizing the 1.0% nano- catalyst and methanol to oil ratio of 11:1.

Conclusion: The study provides a sustainable and eco-friendly approach for effective transesterification of waste cooking oil using calcium oxide nano-catalyst.

Keywords: nanotechnology; calcium oxide; egg shell; biosynthesis; waste cooking oil.

RESUMEN

Introducción: Con la llegada del desarrollo sostenible y la química verde, la valorización de los residuos es muy deseable. En este contexto, se ha propuesto la conversión del aceite de cocina usado en biodiesel mediante transesterificación.

Objetivo: Transformar de manera sostenible el aceite de cocina usado en biodiesel con el uso de nanopartículas de óxido de calcio biosintetizadas como nanocatalizadores durante su proceso de transesterificación.

Métodos: El nanocatalizador obtenido a partir de cáscaras de huevo de desecho se utilizó posteriormente en la transesterificación de aceite de cocina de desecho para obtener biodiesel. La transesterificación se realizó en un solo recipiente para estudiar el impacto del cambio de los parámetros y rendimiento de la reacción, en la que se incluyeron la temperatura, la concentración del nanocatalizador, la relación molar metanol-aceite y el tiempo de contacto.

Resultados: El análisis de espectroscopía infrarroja transformada de Fourier reveló la presencia de varios grupos funcionales, mientras que los análisis de espectrometría de masas por cromatografía de gases identificaron los grupos éster metílico existentes en el biodiesel. El rendimiento optimizado de biodiesel (94.35 %) se obtuvo después de 4 h a una

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temperatura de 60 oC utilizando el nanocatalizador al 1.0 % y una proporción de metanol a aceite de 11:1.

Conclusiones: El estudio proporciona un enfoque sostenible y ecológico para realizar la transesterificación efectiva del aceite de cocina usado con el uso de nanocatalizador de óxido de calcio.

Palabras clave: nanotecnología; óxido de calcio; cáscara de huevo; biosíntesis; aceite de cocinar usado.

Received: 16/01/2023

Accepted: 29/06/2024

Introduction

Due to their widespread use of fossil fuels, rising energy demands as well as home consumption have raised questions about sustainable development. Both domestically and internationally, this is the case of major concern.(1) Fossil fuel combustion provides a multitude of risks to environmental and public health issues, some of which may have widespread and permanent consequences on global warming.(2) As a direct result, concerns about the consequences on the environment have increased, which has led to research into alternative sources of energy. Among the many types of renewable energy include biomass, solar energy, wind power, hydropower, and biofuels.(3) The contribution of each of these resources is essential due to both economic and environmental issues, and alternative fuel might be one of the answers. Because of both the diminishing availability of petroleum and the detrimental effects that emissions from motor vehicles have on the surrounding environment, alternative fuels are rising in significance.(4) Producing alternative fuels through the production of biodiesel from waste materials is a process that is superior to others in terms of both its cost-effectiveness and its efficiency.(5)

Biodiesel is an alternative fuel that is biodegradable, eco-friendly, sustainable, and safe to use for the effective control of greenhouse emissions.(6) Many researchers have considered biodiesel as an additional energy source to petroleum fuel. The findings suggest that biodiesel is a very promising future fuel with its numerous feedstock options and low

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toxicity.(7) Additionally, it produces fewer pollutants when burnt than petroleum diesel and does not contribute to global warming as it has a closed carbon cycle.(8) Perhaps the most surprising part of this fuel is that normal diesel engines can run on biodiesel with little to no additional work and only a slight drop in efficiency. The majority of experiments found that as compared to diesel fuel, the exhaust emissions of oxides, hydrocarbons, particulate matter, and smoke were significantly reduced in biodiesel due to the availability of sufficient oxygen.(9) It is plausible to attribute this behaviour to complete combustion and lower emissions of biodiesel with sufficient oxygen content.(10)

Biodiesel can be obtained by transesterification of animal as well as vegetable fats and oils.(11) Many researchers have used vegetable oils extracted from edible and non-edible crops as feedstock for the biodiesel.(12) Waste cooking oil is typically generated in enormous quantities during the cooking process in a variety of food outlets.(13) The majority of hotels, restaurants, and other food-related enterprises either dump used cooking oil on neighbouring land or discharge it directly into rivers close by. Due to the fact that it is causing environmental contamination and disposal issues, its efficient management is still a serious challenge.(14) Waste cooking oil can be utilized as a source of raw materials for the production of biodiesel in place of virgin vegetable oil.(15) Despite the presence of moisture and other impurities, waste cooking oil has been extensively used to obtain biodiesel after pretreatment. It has been revealed that producing biodiesel from waste cooking oil is an economically viable procedure and presents a workable option for the acquisition of energy resources.(16)

The process of transesterification can be conceded using either homogeneous or heterogenous catalysis to obtain a concoction of fatty acid methyl esters (FAME).(17) Whilst synthesis using homogeneous base catalysis is capable of providing high FAME yields, this method is afflicted by a number of drawbacks, including the inability to regenerate homogenous catalyst as the catalyst is mixed along with products in the course of the reaction.(18) The separation of catalysts is a challenging process that requires the purchase of new equipment, which ultimately results in an increase in the cost of manufacturing.(19) As a result of these shortcomings, the utilization of heterogeneous catalysts for the

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manufacture of biodiesel has been preferred, as it enables biodiesel to be easily isolated and prevents undesirable saponification reactions during the course of reaction(20) The utilization of solid catalysts results in a further reduction in manufacturing costs due to the fact that the catalyst may be recycled and utilized again.(21)

Literature reports use of various heterogeneous catalysts for transesterification reactions including $TiO_{2,}$(22) $SnO_{2,}$(23) cuprospinel nanoparticles,(24) copper oxide,(25) MgO and because of their moisture tolerance, reusability, and immiscibility with methanol. One of the more beneficial catalysts utilized in the biodiesel synthesis process is calcium oxide nanoparticles that have been widely studied for the transesterification of raw(13) and used cooking oil.(14) This may be as a result of properties, and affordability because it can be produced using eco-friendly methods. In addition, this can be related to the fact that it can be recycled.(26,27) Calcium oxide has proven to be an effective additive in a variety of settings, but particularly in those in which the FFA level of the feedstock is low; for example, in the case of low-quality waste or used oils.

This work is based on effective utilization of two types of wastes i.e. waste cooking oil and waste chicken egg shells that are generally discarded as such. Waste chicken eggshells have been used to produce calcium oxide nanoparticles, that have been used as a nano-catalyst for transesterification of waste cooking oil. The process has been optimized to obtain maximum yield of biodiesel. This study intends to sustainably transform waste cooking oil into biodiesel using biosynthesized calcium oxide nanoparticles as nano-catalysts during the transesterification process.

Materials and methods

Materials

Egg shells and leftover cooking oil were procured from the local restaurant. The oil was filtered to remove insoluble components and heated to 120 °C to eliminate moisture content. All chemicals were used as such after being purchased from Merck.

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Preparation of nano-catalyst

After being cleansed with tap water followed by distilled water, the egg shells were dried and later on powdered. In order to eliminate the CO_2 created during the transformation of $CaCO_3$ to CaO, the powdered eggshells were calcined at 800 ºC for 12 hours. The calcined product was thereafter reserved in a desiccator for further use.(21)

Transesterification reaction

Methanol was used to pre-treat the nano-catalyst before it was used in the transesterification procedure. In a round bottom reactor (1000 ml) equipped with an overhead mechanical stirrer, a reflux condenser, and a thermostat, the oil sample underwent pretreatment with anhydrous sodium sulphate.

The nanocatalyst in presence of methanol was added and the transesterification was carried out with variations in the reaction temperature (50-65 ºC), reaction duration (3-4.5 hours), catalyst concentration (0.5-2%), and the molar ratio of methanol to oil (3:1-15:1). The nano-catalyst was removed, splashed with n-hexane, and dried for two hours at 110 °C before being returned to the system.

After extracting the glycerol and eliminating the unreacted methanol, biodiesel was obtained. The standard approach was used to calculate the biodiesel's final yield and analyse the physico-chemical characteristics.(21) Biodiesel was examined using GCMS (Model THERMO Scientific Trace 1300GC) and an FT-IR Spectrometer (Model Perkin Elmer Spectrum 400).

Results

It is necessary to characterize the physico-chemical properties of the biodiesel to consider its suitability as an alternative fuel.(21)

Table 1 illustrates the physico-chemical properties of the obtained biodiesel.

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Table 1- Physicochemical properties of the biodiesel

Property	**Value**
Density (at 15 °C)	891 kg/m^3
Acid value	0.428
Iodine value (per100 g of sample)	115 g iodine
Flash point	191 °C
Pour point	-2.98°C
Cloud point	7.88°C
Kinematic viscosity	5.295

Effect of the operating conditions on the synthesis of biodiesel

The transesterification reaction was carried out with a variety of operating conditions, and the impact those conditions had on the amount of biodiesel produced was investigated so as to identify the operating conditions that produce the highest possible amount of biodiesel. The variation impact of the methanol-to-oil molar ratio (Fig.1a), time (Fig. 1b), temperature (Fig. 1c), and wt% of catalyst (Fig. 1d) on the production of biodiesel was observed.

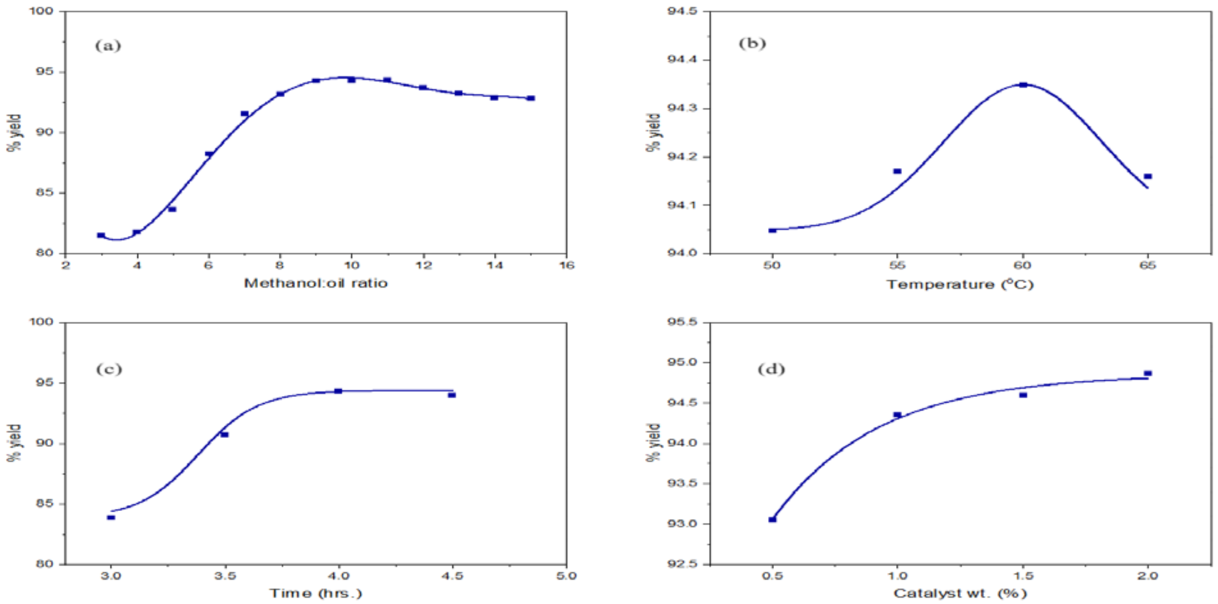

Fig. 1- Variation of % yield with (a) methanol to oil ratio, (b) time, (c) temperature, (d) wt% of catalyst

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Analysis of Biodiesel

The obtained biodiesel has been characterized by a variety of spectrochemical analyses, as reported ahead.

Gas chromatography mass spectrometry (GCMS) analysis

The retention time (Table 2) and pattern of mass fragmentation of the GCMS analysis (Fig.2) was used to identify the chemical composition of the generated biodiesel in terms of FAME. The data shows the chemical constitution of the biodiesel that was generated under the optimum conditions.

Table 2 - Retention time and FAME composition from GCMS analysis

Peak number	Retention Time (min.)	FAME
(1)	7.571	Heptadecanoic acid, methyl ester
(2)	10.098	13-Octadecenoic acid, methyl ester
(3)	10.9333	11,14-Octadecadienoic acid, methyl ester
(4)	13.425	Methyl Heptadecanoate
(5)	14.677	Methyl dodecanoate
(6)	18.329	Methyl tetradecanoate
(7)	19.371	Hexadecanoic acid, methyl ester
(8)	23.121	Docosanoic acid, methyl ester
(9)	25.118	Tetracosanoic acid, methyl ester
(10)	26.731	9-Octadecenoic acid, methyl ester
(11)	28.811	Octadecanoic acid, 1,2,3-propanetriyl ester
(12)	31.665	Octadecanoic acid, 2,3-dihydroxypropyl ester
(13)	34.168	Heptanoic acid, docosyl ester
(14)	36.732	Tetracosanoic acid, methyl ester
(15)	39.961	Octadecanoic acid, methyl ester

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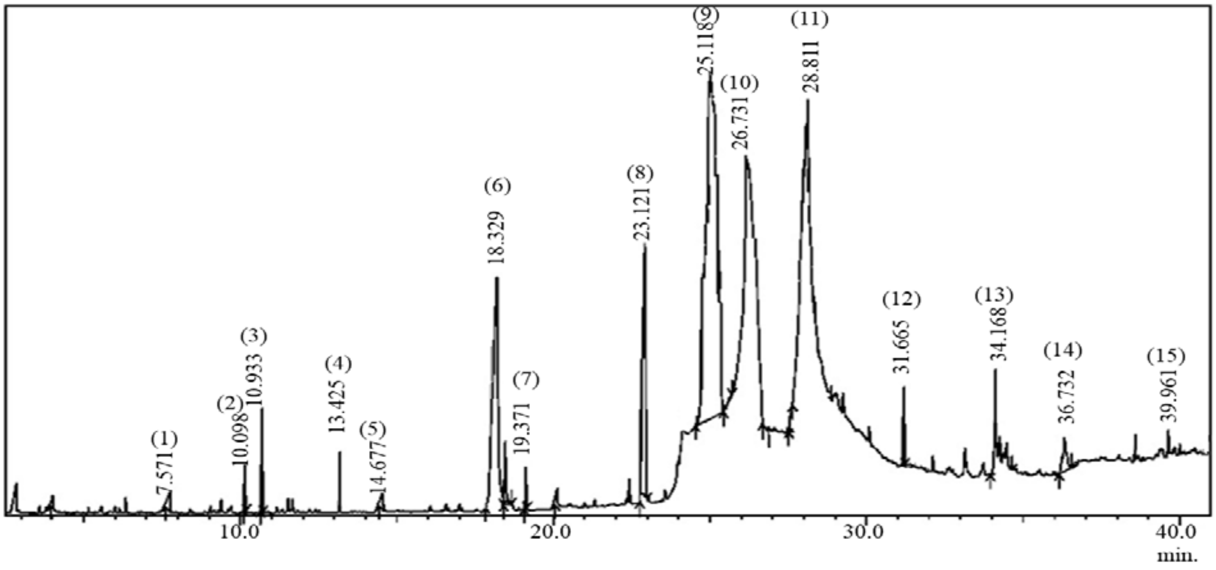

Fig. 2 - GCMS analysis of biodiesel.

Fourier Transform Infrared Spectroscopy (FTIR) analysis

The outcomes of an FTIR study that were carried out on biodiesel in order to assess the functional group composition of the biodiesel are depicted in figure 3.

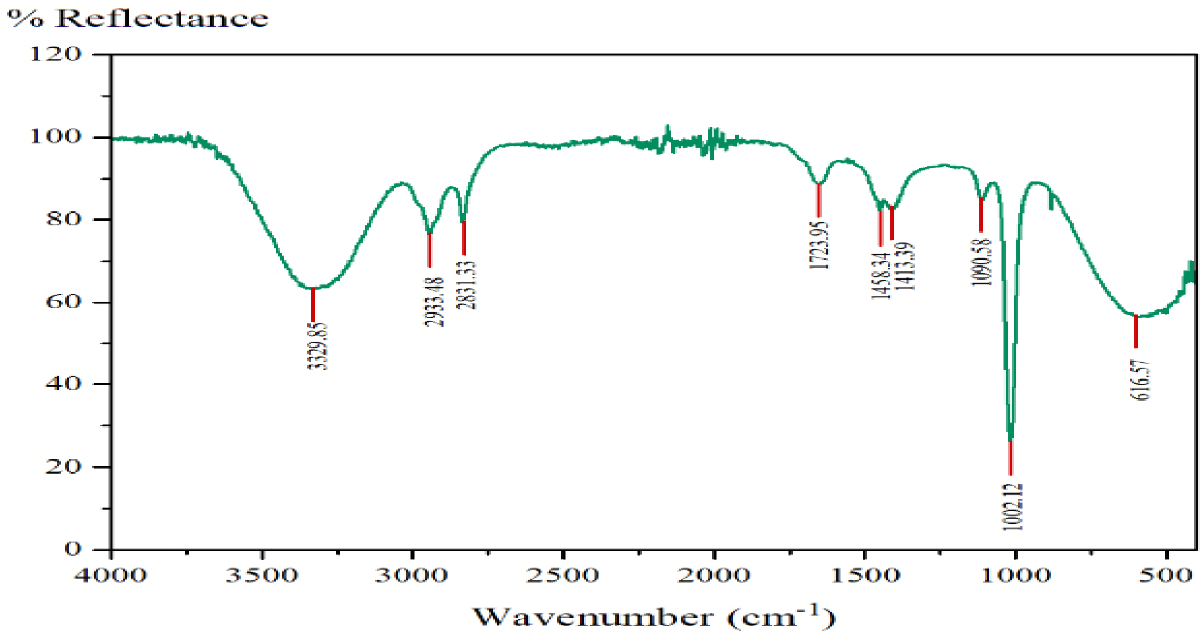

Fig. 3 - FTIR of biodiesel.

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Discussion

The physico-chemical properties of the obtained biodiesel match well with those reported in the literature. (21) The effect of the process parameters and analysis of biodiesel have been discussed ahead.

Effect of methanol: oil ratio

During the process of transesterification between the reactants, an excess quantity of methanol is introduced in order to alter the equilibrium in a way that facilitates the creation of biodiesel. This is due to the fact that in order to obtain greater yields, a significant quantity of methoxy species must be produced, which in turn fundamentally requires a significant quantity of methanol to be present. However, at a certain ratio, an excessive amount of methanol has a detrimental effect on the product.(4) It is clear from Fig. 1a that the production of biodiesel improved initially with a higher molar ratio, up to 11:1, but after 11:1 a little drop was discovered in the output. According to the studies that have been done, an excessively high concentration of methanol not only prevents the separation of glycerol because of an increase in its solubility, but it also causes the equilibrium to shift in the opposite direction, which results in a lower production rate of biodiesel.(6)

Effect of time

Because transesterification is a reversible process that requires longer time to reach equilibrium, the amount of time that reactants are in interaction with one another has a significant impact on the yield of transesterification. The prolonged reaction time increases the extent of interaction between methanol and oil at the active sites of the nano-catalyst, which ultimately results in increased catalytic activity and larger yields of biodiesel.(4) As a result, the reaction was permitted to continue for a total of 3.0 hours during the preliminary tests, and the effect of reaction time was gradually raised all the way up to 4.30 hours. An increase in contact time up to 4 hours results in a greater yield; but, after 4.0 hours, there is no discernible increase in yield (Fig. 1b). This may be because the hydrolysis of methyl esters interferes with the primary reaction after that point.(13)

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Effect of temperature

The effect that shifting the temperature of the reaction from 50 to 65 ºC has on the amount of biodiesel produced is quite evident. The incorporation of the heterogeneous catalyst into the reaction results in the formation of a three-phase system consisting of oil, methanol, and the catalyst, with the system's interface being able to accommodate the reaction(4) The temperature at which the reaction takes place is essential to the manufacture of biodiesel because it lowers the oil's viscosity and increases the rate of biodiesel production by lowering the amount of mass transfer resistance. On the other hand, if the reaction temperature is higher than 60 ºC, the methanol will evaporate, which will result in a lower biodiesel yield.(13) The effect was observed in our study with increase of yield till 60 ºC followed by a decrease afterwards (Fig.1c). Thus, 60 ºC was considered as the optimal reaction temperature.

Effect of dosage of nanocatalyst

The concentration of the catalyst has a significant impact on the production of biodiesel due to the fact that an increase in the number of active basic sites leads to an increase in the amount of biodiesel production.(13) The nano-catalyst possesses active basic sites, and these sites are credited with the conversion of methanol into the methoxide group, which is then used to attack the carbonyl portion of oil. The generation of biodiesel is affected when there is an increase in the concentration of the catalyst (Fig. 1d). The inclusion of the catalyst, which ranged from 0.5-2.0%, resulted in an increase in the biodiesel output. However, in order to achieve the best possible conversion, the amount of catalyst used was set at 1.0%. It was shown that increasing the amount of catalyst above 1.5% (w/w) did not have a significant impact on the yield. This is presumably due to the fact that the viscosity of the reaction mixture increases and active sites are loaded with products.(6) At a nano-catalyst concentration of 1.0% (w/w), the optimal methanol: oil molar ratio was determined to be 11:1, which was validated by an optimal yield of 94.35% at 60 ºC. In addition, it was

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feasible to reuse the catalyst for a maximum of five different transesterification cycles, which is in line with the findings of the earlier research.(28)

Analysis of Biodiesel

The findings of GCMS in terms of composition were found to be consistent with the findings that had previously been reported indicating successful completion of the transesterification process.(27)

The values of FTIR analysis are also consistent with those observed in the literature for the synthesis of biodiesel. The O-H stretching vibrations were observed as strong bands at 3329.85 cm.$^{-1}$(20) Absorption bands at 2933.48 and 2831.33 cm^{-1} demonstrated the axial deformation of methylene groups.(13) The CO stretching in esters was identified as the source of the peak with the maximum intensity at 1723.95 cm.$^{-1}$(18) The bands in the biodiesel spectrum that correspond to the asymmetric bending modes of CH_3 groups of esters were observed at 1458.34 and 1413.39 cm,$^{-1}$ while the bands for stretching vibrations of methoxy groups were obtained at 1090.58 and 1002.12 cm.$^{-1}$(19) The band at 616.57 cm^{-1} was found to match to the bending vibrations of CH_2 and CH_3 groups.(27) Thus, the waste egg shells can be efficiently used for the transesterification of waste cooking oil to obtain biodiesel.

Conclusions

Egg shells and used cooking oil are two examples of typical waste items, and the current research demonstrates a simple and cost-effective method for repurposing these materials in the production of biodiesel, a value-added product. In this scenario, waste egg shells were processed to obtain calcium oxide nanoparticles.

The following points can be concluded

- Through careful optimization of the reaction parameters, it was possible to utilize these nanoparticles to their full potential as a heterogeneous nano-catalyst in the transesterification process.

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- According to the findings of the study, an optimal yield of 94.35% was achieved under optimized reaction conditions consisting of a temperature of 60 ºC, a ratio of 11:1 as methanol to oil after 4 hours, and a catalyst concentration of 1.0%.
- In terms of the nano-effectiveness, the biosynthesized catalyst was found reusable after recycling.
- The research reveals a method by which waste products can be utilized in an efficient manner to acquire biodiesel that can serve as an alternative to the depletion of natural petroleum supplies.

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Conflict of interests

The authors report no conflict of interest.

Authors' contribution

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Writing - review & editing: Rajni Garg, Nnabuk Okon Eddy.

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