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Cu-Doped chicken eggshells: An eco-friendly catalyst for the production of green biodiesel with *Azadirachta indica* seed oil to promote the circular economy

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In this study, waste eggshell (WES) has been used to synthesise a heterogeneous Cu-CaO catalyst for *Azadirachta indica* seed oil straight transesterification. The catalyst is made *via* wet impregnation, which produced a crystalline structure having an area on the surface of 4.8893 m²/g and on average, the size of the pores 275.826 Å, as measured by XRD and BET measurements. The composition of the Cu-CaO catalyst is further verified by SEM-EDS analysis. The direct transesterification process has been optimized by modifying the catalyst dosage (%), reaction temperature (°C), ethanol mole ratio, and time (min). At a catalyst quantity of 9% wt/wt, 3 h response duration, 12:1 Ethanol to oil mole proportion, and 65°C temperature, the maximum yield of Methyl Ester of Fatty Acid (90%) is obtained. In addition, a number of characteristics of the produced biodiesel are measured, including cloud point 9°C, pour point 4°C, higher heating value 38.86 MJ/kg, acid value 0.32 mg KOH/g, density 0.8553 g/cm^3, kinematic viscosity 5.10 cSt, and moisture content 0.03. After six cycles, the Cu-CaO catalyst's stability and reusability are established. These results show that eggshell waste-derived Cu-CaO catalyst has the potential to be an affordable, sustainable and environmentally friendly catalyst for the synthesis of biodiesel.

Keywords: *Azadirachta indica,* Biodiesel**,** Circular economy, Heterogeneous catalyst, Waste chicken egg shell

Introduction

Biodiesel, Methyl Ester of Fatty Acid (FAME), is synthesized through the *via* transesterification reaction of triglycerides found in different oils, both edible and non-edible, animal fats, used cooking oil, including algal biomass. Biodiesel boasts intrinsic advantages, including biodegradability, non-toxicity, a minimal sulphur content, maximum cetane number, excellent flash point, natural lubrication, and elevated combustion efficiency[1-2]. However, issues with supply and demand and high production costs make it difficult to synthesize biodiesel using refined edible oil. The cost of producing biodiesel depends on a number of variables, such as the availability of feedstock, the location, varieties of crops throughout different seasons, and the price of crude oil. In order to address concerns with food security and reduce production costs, oils that are not edible, including castor, neem, jatropha, even karanja oil, among others, appear to be promising substitutes[3]. Other feedstocks include pig lard, beef tallow, and yellow grease[4]. The production of biodiesel through transesterification processes frequently depends on homogeneous, heterogeneous, or biocatalysts (sometimes referred to as enzyme catalysts). Heterogeneous catalysts, which have a large number of basic or acid active sites, have great selectivity and activity[5]. According to research, chemically generated heterogeneous solid catalysts have exceptional catalytic activity and can completely convert oil into biodiesel[6-7]. Nevertheless, heterogeneous catalysts may have disadvantages including limited active site density, mass transfer restrictions, and possible leaching[8]. Among the modern heterogenous catalysts calcium oxide (CaO) obtained from egg shell is emerging as a highly potential heterogeneous base catalyst for the production of biodiesel. Furthermore, a variety of waste shells, including the shells of golden apple snails, cockles, mussels, and chicken eggs, can be easily used as sources of calcium for making CaO catalysts[9]. Research by Kaewdaeng *et al*.[10], has shown that CaO obtained from discarded shells is a good heterogeneous catalyst for the synthesis of biodiesel. However, the limited surface area of CaO restricts the number of active basic sites that can reside there and it is vulnerable to calcium species leaching during the transesterification process. These elements considerably reduce catalytic effectiveness of CaO, which is necessary to produce the highest possible amount of biodiesel. Considerable work has gone into altering the CaO catalyst to improve its physicochemical characteristics, such as catalytic activity, stability, and basic strength, in order to solve these issues. To improve the catalyst's effectiveness in the synthesis of biodiesel, for instance, doping CaO with particular elements such as copper oxide can increase its basicity, increase its surface area, and reduce the size of its particles.

In order to improve the catalytic efficiency of calcium oxide (CaO) obtained from eggshells, this work proposes a novel process that involves moisture impregnation *via* transition metal doping. Stability, catalytic activity, and basic strength are the main physicochemical features of these mixed metal oxide catalysts such as CuO, have properties including varied oxidation states, the ability to form ion complexes, and high catalytic effectiveness that make these excellent co-catalysts [11]. As far as we are aware, no one has ever attempted to synthesize these unique mixed metal oxides to be used in neem oil transesterification by adding CuO to calcium oxide-based catalysts made from leftover eggshells. Further, the objective of the research is to look into the impacts of several parameters, including dopant loading proportions, kinds, and calcination temperatures, in order to design an efficient catalyst that produces biodiesel. In this work, a novel technique for improving Cu-CaO catalysts made from eggshells by adding transition metal Cu by a straightforward wetness impregnation process.

Experimental Section

As a source of the $CaCO_3$ leftover egg shells were obtained from the CSIR-IICT canteen, Hyderabad. The Hyderabad-based local provider provided the *Azadirachta indica* seed oil. $Ca(NO_3)_2$ (laboratory grade), Ethanol (99.5%) as well as phenolphthalein were purchased from Merck India. To ensure complete removal of moisture, the feed material i.e., the vegetable oil was dried in a hot air oven with before keeping that inside a desiccator.

Table 1 — Physico-chemical and characteristics of neem seed oil chemically

Properties	ASTM standards	Value
Kinematic viscosity (40°C)	ASTM D 445	40.32cSt
Density (at 15°C, g/cm^3)	ASTM D 4052	0.93
Acid value (mg KOH/g)	AOCS cd 3d-63	1.029
Physical state at 25°C	-	Liquid
Moisture content (wt%)	AOCS cd ca 2c-25	0.02
Iodine value (g of I_2/100 g oil)	AOCS cd 1-25	102.62
Peroxide value (meq O_2/kg oil)	AOCS cd 8-53	88.0
Saponification value (mg KOH/g)	AOCS cd 3-25	185.93
Calorific value (mJ/kg)	-	39.16
Flash point (°C)	ASTM D 93	217

Neem seed oil physico-chemical characteristics

The different physicochemical characteristics of neem oil are displayed in Table 1. Details of characterization procedure for the neem oil, catalyst and fuel are given in the Supplementary Information.

Preparation of the catalyst

Doping with Cu and calcination

To get rid of dirt and organic materials, the collected waste egg shells were carefully cleansed with tap water followed by a distilled water rinse and then dried at 110°C for whole night. Agate mortar and pestle were used to ground the desiccated egg shells into powder. The egg shells were eventually calcined in a muffle furnace for 4 h at 900°C and preserved to be used later. To improve the CaO potential as an activity, Cu was doped onto it using $Cu(NO_3)_2$ solution. The wet impregnation method used in this investigation involved mixing 30 mL of distilled water with 5 g CaO, followed by the addition of 3% $Ca(NO_3)_2$. The concoction was subsequently filtered and the extract was dried for an entire night during 110°C in a hot air oven before being stored in a desiccator.

Preparation of biodiesel

In a round-bottom, three-neck, 1000 mL flask, the reaction was conducted. The process mixture's heat was determined by inserting a thermometer through the last arm before the magnetic stirrer and water-cool condenser were connected into the side arms and center neck, respectively. First, after adding a set quantity of oil (200 g), it had been heated up to the appropriate temperature. A fixed amount of Alcohol (Ethanol, 600 mL) with heterogenous catalyst (9 wt% of oil) have been added, and to keep the reaction time, temperature constant, the heating was modified and allowed for the necessary amount of time of reaction, while continuously stirring. After the response was finished, the excess ethanol was collected through distillation, and the catalyst was restored by filtration. Before the fuel is standardized, the resultant product undergoes ultracentrifugation to eliminate any impurities. The yield of the ester obtained was 90%.

Results and Discussion

X-ray diffraction (XRD)

At first, peaks linked to $CaCO_3$ were identified by XRD analysis from natural eggshells (Fig. 1). When the material was heated to 900°C, the calcined eggshells showed a clear shift from $CaCO_3$ to CaO. The calcined materials exhibited narrow and clearly defined peaks, indicating a crystalline structure. This suggests that thermal activation must occur before the catalyst is used in transesterification procedures. A significant peak at 2θ = 29.54° in the XRD pattern of dried eggshells suggested that calcite ($CaCO_3$) was the predominant primary phase in the eggshells. After 2 h of calcination at 900°C, the CaO phase emerged with peaks with values of 2θ for 32.00°, 37.18°, 53.97°, 63.42°, and 67.10° which matched well with COD entry no. 96-720-0687. These results were in good agreement with earlier studies[12]. It was further shown that calcium hydroxide was present since diffraction peaks consistently appeared at 2θ values of 17.79°, 28.46°, 33.82°, 46.83°, and 50.57° in all catalyst sequences[13]. Particularly, the XRD analysis of the calcined eggshells showed complete transformation of the $CaCO_3$ phase into CaO due to a lack of the main calcite phase peak (2θ = 29.54).

Significant peaks at 2θ values of 32.01°, 35.25°, 38.96°, 43.66°, 53.43°, 58.05°, and 61.18° were found in the Cu/eggshell catalyst. These values corresponded to the (1 1 0), (0 0 2), (1 1 1), (−2 0 2), (0 2 0), (2 0 2), as well as (−1 1 3) planes monoclinic CuO, correspondingly, and aligned perfectly with PDF no. 80-1917. In addition, peaks were visible at 2θ values of 23.23°, 29.30°, and 31.25°. These peaks most likely originated from the hexagonal $CaCO_3$ planes (0 1 2), (1 0 4), and (0 0 6) as indexed in PDF no. 05-0586. All of this can be attributed to the development of Cu/CaO, where Ca^{+2} sites have been replaced by Cu^{+2} sites.

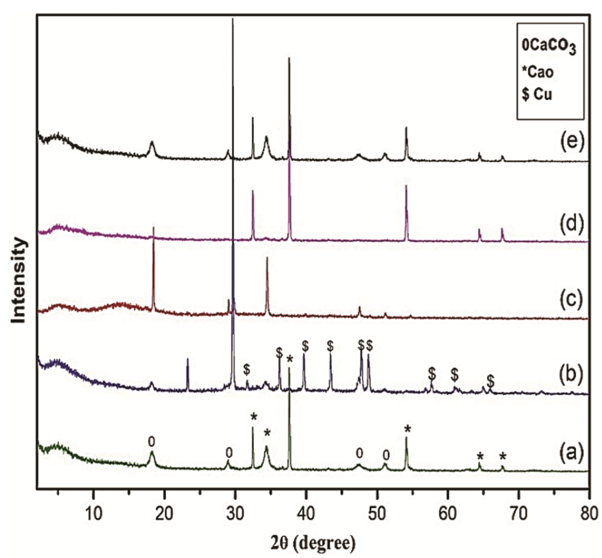

Fig. 1 — XRD pattern of eggshell at 900°C and Cu catalyst made from eggshells at various (a) 600 °C, (b) 800 °C, (c) 900 °C, and (d) 1000°C

Fourier transform infrared spectroscopy (FTIR) analysis

As shown in Fig. 2, the FTIR spectrum of CuO/egg shell catalyst shows peak at 3266 cm^{-1} (-OH stretching, caused by residual water content), 1411 cm^{-1} and 874 cm^{-1} (stretching and flexing bonds of calcium carbonates in C–O)[14], 712 cm^{-1} (Ca–O bonding)[15]. In the spectrum of calcium oxide (CaO-900) burnt eggshell, 3439 cm^{-1} [OH in $Ca(OH_2)$], 1426 cm^{-1} and 864 cm^{-1}, (OH bonds connected to hydrated CaO), indicates eggshell that has been calcined and hydrated (CaO-900 water). The in-plane and out-of-plane deformation vibrations of carbonate anions were identified as the cause of the bands at 858 cm^{-1}, which exhibited lower peak intensities in comparison to pure eggshell[16].

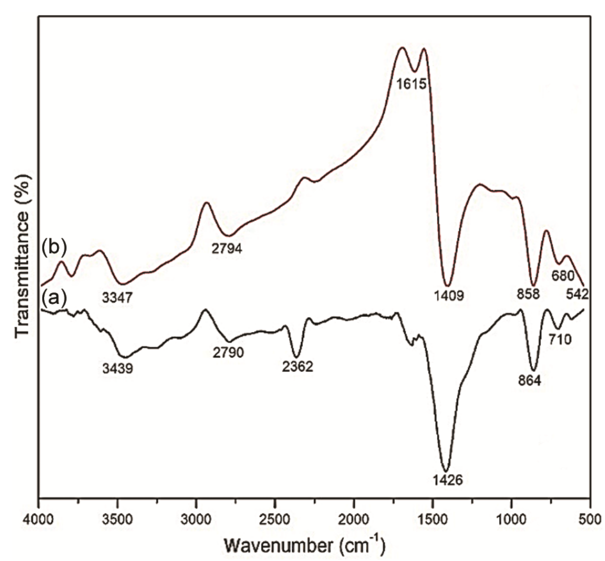

Fig. 2 — FTIR spectra of (a) eggshell calcined and (b) prepared catalyst Cu-Cao

Thermo-gravimetric analysis (TGA)

TGA study was carried out to establish the appropriate calcination for egg shell and the results are shown in Fig. 3. Slight weight loss was observed in the temperature range 50 to 550°C, which might be fully attributable to a similar chemical material and elimination of water molecules. Nonetheless, significant weight loss could be seen at temperatures between 600 and 800°C. The weight of the sample remained stable above 800°C, revealing that no further decomposition of $CaCO_3$ occurs.

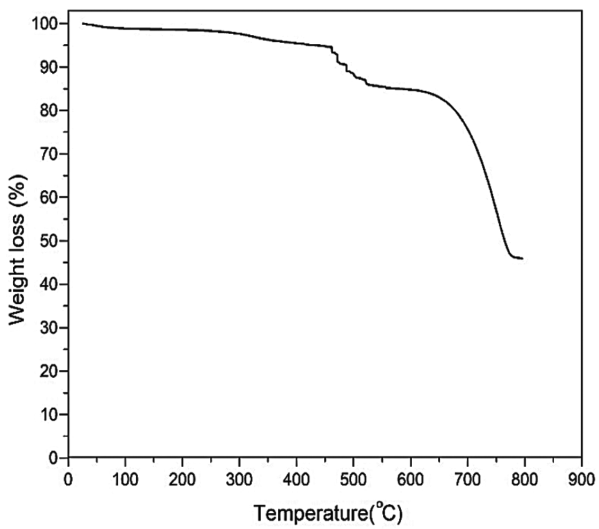

Fig. 3 — TGA curve for calcined eggshell

Scanning electron microscopy (SEM) analysis

The SEM image (Fig. 4) of calcium oxide (CaO-900) showed a regular microstructure with rod-like particles, which is consistent with earlier findings[17]. Additionally, it was observed that the calcined-hydrated eggshell (CaO-900-hydration) had porous surfaces that resembled honeycombs, which is similar to what found for treated mussel shells[18]. The modified surface morphology of the Cu-doped CaO revealed a porous structure caused by copper impregnation, whereas the clean calcined CaO showed an uneven microstructure with irregularly shaped and sized particles. The increased surface area that results from this modification in surface structure is essential for improving catalytic effectiveness. Particle aggregation during synthesis and calcination

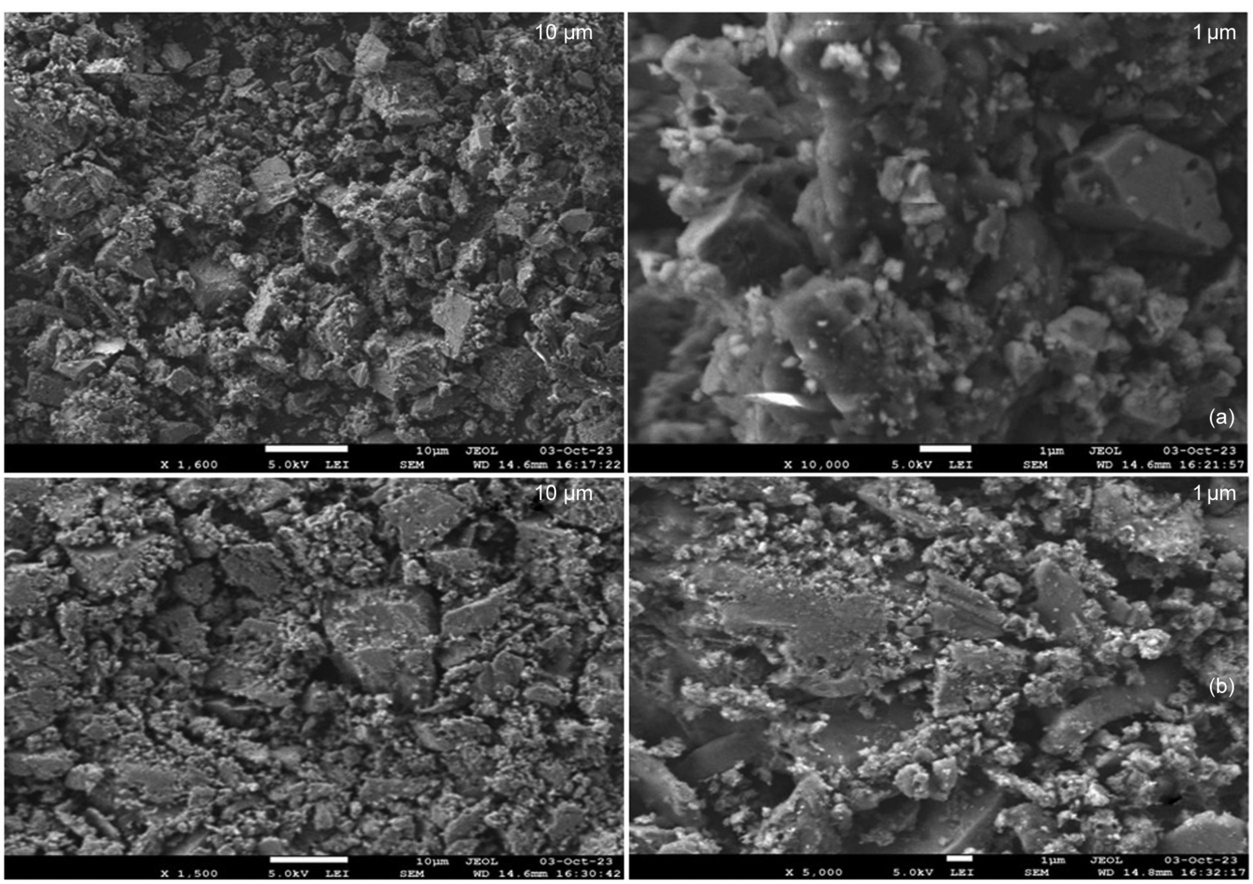

Fig. 4 — SEM micrographs of (a) eggshell and (b) Cu/CaO

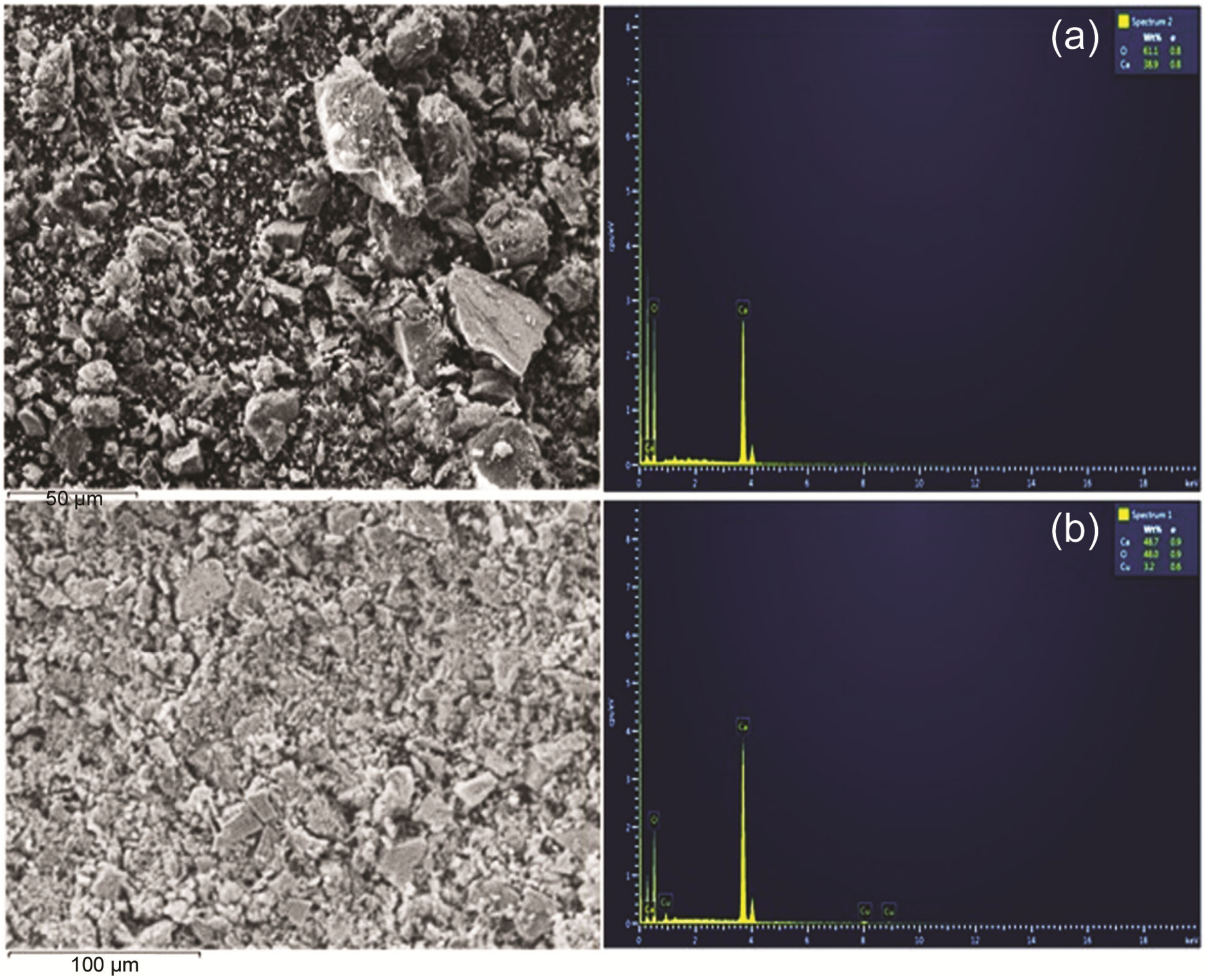

Fig. 5 — SEM images with corresponding EDS spectra of (a) chicken eggshell and (b) Cu/CaO

of Cu/CaO particles is probably the cause of the observed variance in particle. According to the prepared Cu-doped CaO, as shown in Fig. 4, there is copper (3.2 wt%), oxygen (48.0 wt%), and calcium (48.7 wt%).

Using an energy dispersive X-ray (EDX), Cu/eggshell catalyst was subjected to elemental analysis, as shown in Fig. 5. The elements that constitute the composite oxide catalyst were found to be oxygen, calcium, and copper. These components were identified on the catalyst's surface, and weight percentage was used to quantify their proportions. The quantitative study of the eggshell showed that its composition was 34.9% calcium and 41.1% oxygen. On the other hand, 41.30% oxygen, 44.7% calcium, and 3.2% copper were included in the Cu/eggshell catalyst's composition. Interestingly, the oxygen level varied throughout the catalyst from roughly 41.10% to 41.30%, suggesting the presence of oxides from the Cu metal-containing catalyst surfaces.

X-Ray photoelectron spectroscopy (XPS) analysis

The elements Cu, O, and Ca were detected in the CuO/eggshell XPS survey spectrum Fig. 6. Lattice oxygen and surface hydroxyl groups, respectively, were identified as the primary peaks of the O 1s XPS spectum, which were observed at 531.49 eV[19]. The primary peaks in the picture, which show the existence of oxidized Ca^{2+} in calcium species, are assigned, respectively, to Ca $2p^{3/2}$ and Ca $2p^{1/2}$, at 346.08 and 349.64 eV [20]. Peaks correspond to O=C-O and C-C were found at 288.60 eV and 284.5 eV, accordingly, in the C 1s spectrum [21]. Additionally, the Cu 2p XPS spectrum showed distinctive Cu $2p^{3/2}$ peaks at 933.09 eV, indicating the existence of Cu^{2+} species and verifying the presence of CuO [22].

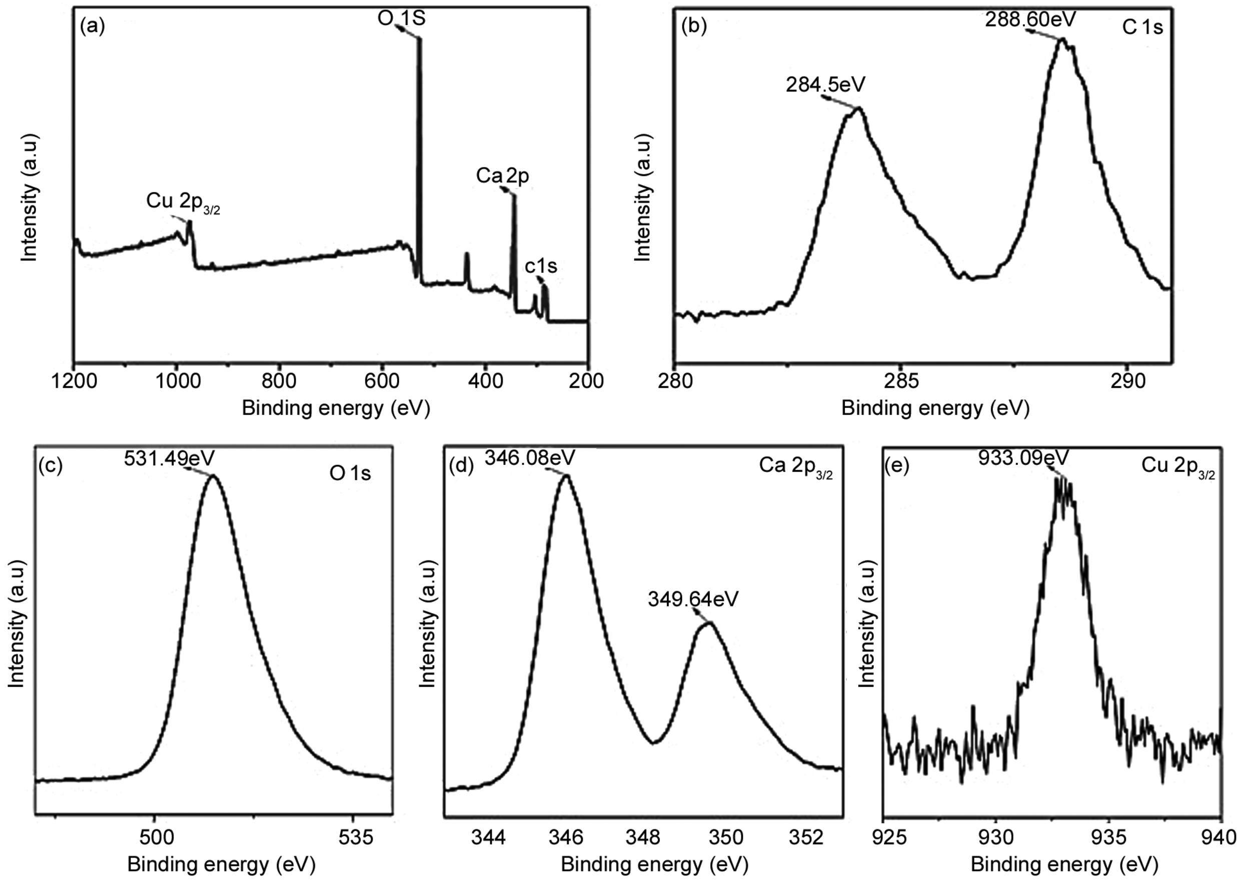

Fig. 6 — XPS spectra for (a) Cu /CaO broad scan spectrum, (b) C 1s, (c) O 1s, (d) Ca 2p and (e) Cu 2p

BET Analysis: Brunauer–Emmett–Teller

To determine the surface area of the produced catalyst, a plot was created by plotting the pressure (p/p_o) against the adsorbed nitrogen (cm^3/g) at STP. Drying of the catalyst required degassing the sample (powder) at 200℃ for 24 h prior to BET analysis. The pore size, pore volume and surface area are found to be 275.826 A^o, is 0.030571 cm^3/g, and is 4.8893 m^2/g, respectively (Fig. 7).

Impact of response conditions about the production of biodiesel using Cu-Cao catalyst

Different parameters such as temperature and the amount of catalyst loaded, reaction time and the molar ratio of ethanol to oil were studied. In order to determine the optimal molar ratio of ethanol to fuel, loading of the catalyst was adjusted between 5% and 12% in relation to the oil mass. The results showed that the conditions such as loading of catalysts 9%, a response period a molar ratio of 3 h and 12:1 of ethanol to oil were adequate to achieve the highest possible product yield of 90%.

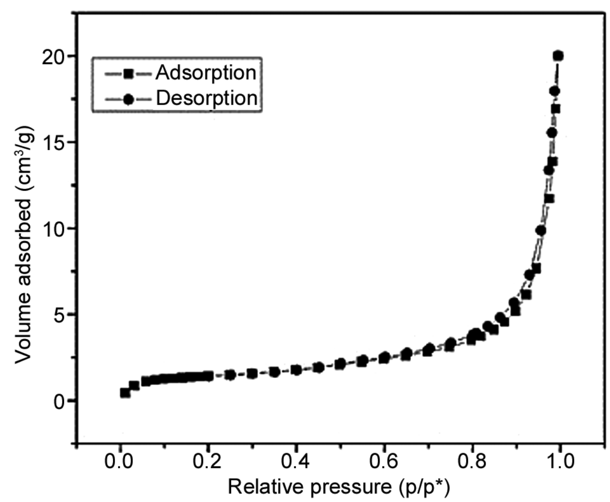

Fig. 7 — BET graph of Cu/CaO catalyst

Temperature-dependent consequences of calcining for the catalyst's performance

The impact of activation temperature on the transesterification activity of several catalysts was examined within the 600–1000°C. The catalyst Cu/CaO calcined at 900℃ had the highest level of

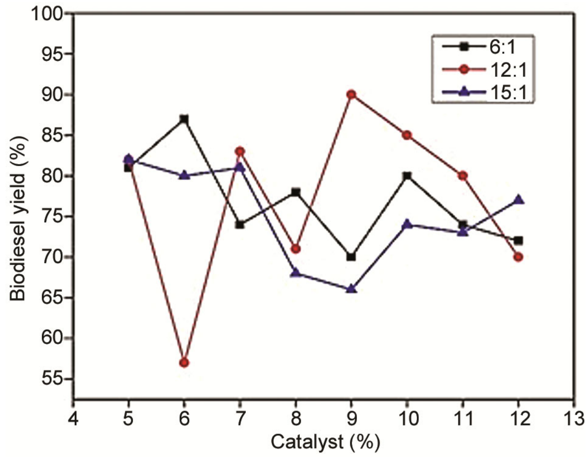

Fig. 8 — Impact of molar ratio of catalyst to oil on biodiesel conversion

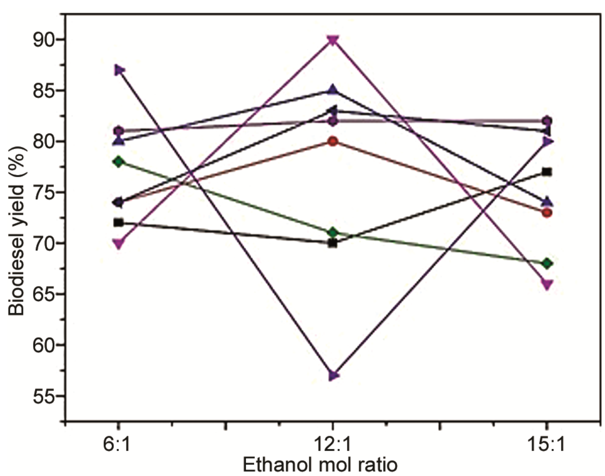

Fig. 9 — Impact of the molar ratio of ethanol to oil on biodiesel conversion

catalytic activity between various temperatures of calcination. The remarkable entire source component of the C900's catalytic reaction is the catalyst's surface having an ideal number of active sites. It was discovered that C900's pore volume and BET surface area were much higher than those of activation of catalysts at 800°C, 900°C, and 1000°C.

Effect of catalysts loadings

The experiment involved raising the catalyst's intensity loading between 5 and 12 wt%, and the conversion of biodiesel was seen from Fig. 8. However, in some cases, the biodiesel production begins to decrease when catalyst loading increases from 7 to 8 wt%. The results indicate that a catalyst concentration of 9 wt% provided the appropriate the quantity of active sites needed to generate the maximum amount of biodiesel transformation 90%.

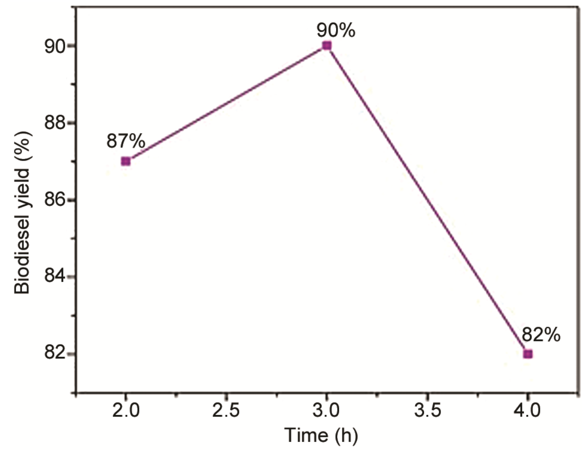

Fig. 10 — Impact of reaction time on the production of biodiesel

Impact of ethanol to oil ratio

The greatest yield ever reported was 90%, and the production of biodiesel rapidly decreases as the percentage of ethanol compared to oil increases (15:1) as shown in Fig. 9. When the ethanol oil ratio exceeded the recommended level of 15:1, the biodiesel yield decreased. Ethanol's polar hydroxy group also serves as an emulsifier, making it harder to separate the biodiesel product from the glycerol[23], which reduces the synthesis of ethyl ester. Because of this, it was decided that the best molar ratio for ethanol and oil in this study was 15:1.

Impact of reaction temperature

The impact of reaction temperature (between 55 and 70°C) on *Azadirachta indica* seed oil-derived biodiesel output was examined. The highest impact was observed at 65°C on ethyl ester conversion process; following this, the biodiesel conversions dropped. High temperatures induce reactant molecules to collide more frequently, which results in miscibility and mass transfer[24]. The amount of biodiesel produced dramatically dropped once the temperature of the reaction exceeded 65°C. The high temperatures at which the ethanol vaporized and remained in the reactor may have decreased the amount of ethanol available for the reaction[25], resulting in a drop in the biodiesel conversion.

Impact of response time

The production of biodiesel with reaction time is shown in Fig. 10. The highest production is observed at 3 h and when the transesterification reaction was allowed to continue for more than three hours, the amount of biodiesel produced essentially dropped. Accordingly, the catalyst with a loading for 9 wt%

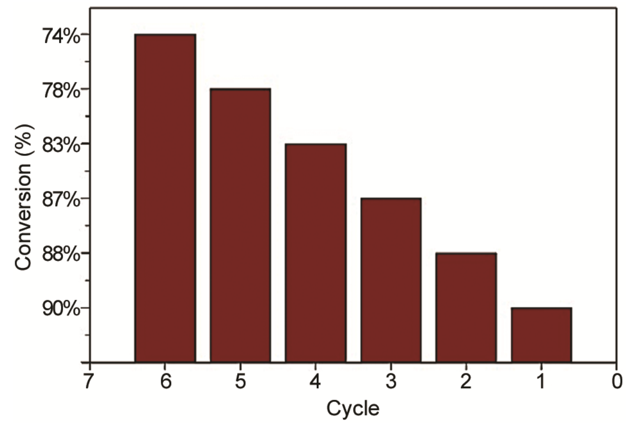

Fig. 11 — Reusability of the catalyst in the transesterification process of biodiesel production

was found to be the result of the reaction value optimization experiments, to generate 90% biodiesel, the following conditions must be met: ethanol to oil molar ratio of 12:1, reaction temperature of 65°C, and reaction duration of 3 h.

Reusability test

Fig. 11 illustrates the ideal response circumstances for the recycling test among the C900 the catalyst, this was carried out using a fresh reactant. The utilized catalyst was removed from the reaction mixture by filtering it after each run, followed by a three-hour oven drying period at 110°C and an n-hexane wash to get rid of any absorbed components. The finding indicated that up to seven runs might provide a yield of at least 75%. But, after of seven times, the yield began to decline. Organic contaminants covering the catalyst's surface may oxidize, resulting in a decline in transesterification efficiency[26-27].

Characteristics of the catalyst, fuel, spectral and chromatographic characterization of the biodiesel, Contribution of the study towards Green chemistry, Process mass intensity, comparison of CaO catalysts to other heterogenous catalysts and Future outlook based on the study are discussed under Supplementary information.

Conclusion

The potential of neem seeds as a feasible and sustainable way to produce a low cost biodiesel using copper doped chicken eggshells was successfully explored. The C900 catalyst functioned admirably and generated a maximum yield of 90% under optimal reaction circumstances of ethanol to oil molar ratio of 12:1, reaction temperature of 65°C, and reaction duration of 3 h. The green catalyst offers a number of benefits, including being inexpensive, easily obtainable, because it is environmentally benign, readily prepared, and biodegradable, it is a good choice for large-scale biodiesel production.

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Conflict of interest

The authors declare no conflict of interest.

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