

Original Research Article

Synthesis and characterization of CaO catalyst obtained from *Achatina achatina* and its application in biodiesel production

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ARTICLE INFORMATION

Received: 07 April 2018

Received in revised: 12 May 2018

Accepted: 20 May 2019

Available online: 2 June 2019

DOI: 10.48309/JMNC.2019.3.2

KEYWORDS

Achatina achatina

Calcium oxide

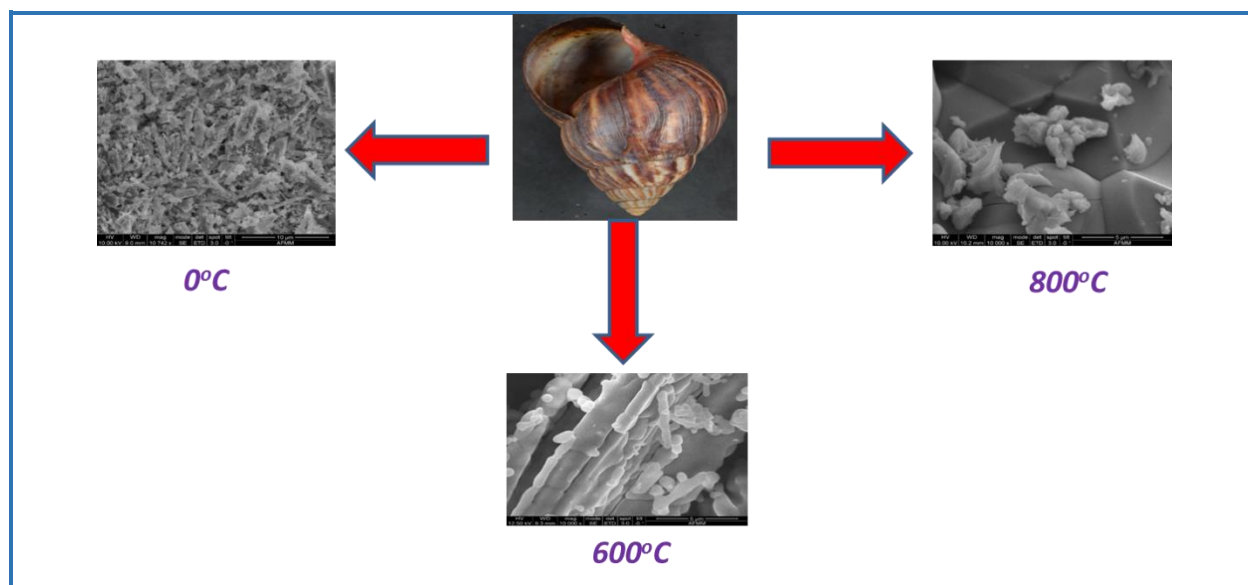
Heterogeneous catalyst

Biodiesel

ABSTRACT

In this research study, *Achatina achatina* shells was used as the source of raw material to produce calcium oxide which was used as a catalyst in the production of biodiesel. The main aim of this study was to investigate the effect of varying temperatures on the calcium oxide formed using *A. achatina* during the calcination process for their possible use as a heterogeneous catalyst in the production of biodiesel. The shells were first grinded and then calcinated at different temperatures ranging from 0 °C to 1000 °C. After calcination, the CaCO₃ present in the *A. achatina* shell was converted to calcium oxide. The obtained calcium oxide was characterized using Fourier transform infrared spectroscopy (FT-IR). The asymmetric stretching of the CO₃²⁻ (cm⁻¹) absorption was not proportional with the increasing temperature as it was observed over the plane vibrational modes of CO₃²⁻ (cm⁻¹). Also, the O-H stretching band (cm⁻¹) at 100 °C and 800 °C had similar absorption values. Pearson correlation revealed both negative and positive relationship between the absorption rate and the temperature, disclosed a significant difference at p<0.05. The calcined structure was examined using scanning electron microscope (SEM) with a magnification of 10,000X. A more stabilized, uniform structure, and agglomerate into lumps were observed at 800 °C and 1000 °C. The results showed that, the *A. achatina* shell is a suitable catalyst in the production of Biodiesel because it is readily available and has no adverse effect on the environment.

Graphical Abstract



Introduction

The ever-increasing population of human being and growing the industrialization is a significant factor increased energy consumption. Several sources of energy have been exploited by the man driving to several consequences ranging from environmental pollution to other adverse effects of exploitation of natural energy sources. The desire for a cleaner, safer, and renewable energy source has led to the use of biofuel as an alternative source of fuel [1–3]. Biofuel is a breakthrough in the quest for sustainable energy source leading to a reduction of global warming [4]. Biodiesel has been studied and accepted as a notable option for complementing conventional fuels [2, 5]. Its production from renewable biological sources such as vegetable oils and animal fats makes it an easily assessable and readily available source of alternate energy. Other renewable energy sources such as solar energy, wind energy, hydro energy, and energy from biomass and waste (Biofuel including bioethanol and biodiesel) materials are the major sources of

energy, developed and used by different nations to limit the use of fossil fuels.

Catalysts are the major components of several reactions. Catalyst is known to aid the fast completion of reaction thereby increasing the rate of reaction even though they do not act as major reactant because they are separated out after the completion of the reaction, leading to formation of the target molecules. The catalysts used for transesterification reaction are homogeneous, heterogeneous or enzymatic catalysts [6].

Homogeneous catalysts exist in the same phase with the reactants while heterogeneous catalysts exist in a different phase from the reactants making them easy to separate out on completion of reaction unlike the former [7]. Sodium hydroxide and potassium hydroxide are the commonly used base homogeneous catalyst for the transesterification process for biodiesel production [8]. The benefits of using a traditional homogeneous catalyst (Basic or acidic) are due to their high activity (i.e., complete conversion within 1 hour) and mild reaction condition (i.e., from 40 °C to 65 °C and

atmospheric pressure), however the major concern faced is the tendency of homogeneous catalysts to form soap [9]. Heterogeneous catalysts, on the other hand, are preferable due to the fact that they can improve the transesterification process by eliminating the neutralization salt formed in the glycerol, leading to some difficulties in the separation process because the catalyst needs to be reused after the reaction which may be difficult to achieve with homogeneous catalyst [10]. Among the commonly used heterogeneous base catalysts such as Na_2CO_3 , K_2CO_3 , CaCO_3 , CaO , MgO , SrO , BaO , and ZnO , calcium-containing catalysts have gained high reputation due to their wide use and major advantage as they have a higher tendency to be reused and having less environmental impact [11, 12]. CaO has been reported to show high basicity and high activity compared to other heterogeneous catalyst, also its catalytic ability does not depend only on the calcination temperature but also on the source [13].

The choice of *A. achatina* as a source of CaO in this study presents an environmentally friendly synthesis of calcium oxide from readily available and less expensive source. *A. achatina* is known and commonly referred to as African giant snail. It is widely distributed in West Africa and sometimes kept as pets. This species is abundant in nutrient hence it is edible. Therefore, this justifies the abundance and availability of their shells found in commercial places. The CaO obtained from *A. achatina* in this work was characterized for their possible use as a heterogeneous catalyst.

Experimental

Sample collection

Achatina achatina shells (Figure 1) were collected from Ogbomosho, Oyo State, Nigeria which is geographically located on the Latitude

8°04'23.52" N and Longitude 4°14'11.69" E. These shells were gotten from commercial places including, restaurants and residential sites.



Figure 1. a) The picture of a living *A. achatina*



Figure 1. b) showing the image of *A. achatina* shell before grinding

Catalyst preparation and characterization

The *A. achatina* shells were washed with the distilled water to remove the impurities, then dried. The dried shells were ground to fine powder and sieved to obtain a uniform particle size. They were later calcinated in an oven at a different temperature to obtain calcium oxide. The calcination was done at 0 °C, 200 °C, 400 °C, 600 °C, 800 °C, and 1000 °C to form the calcium oxide. The powdered calcium oxide was characterized using the Fourier transformed infrared spectroscopy (FT-IR) and scanning electron microscope (SEM). We further

conducted a correlation test using the Pearson (r) to statistically establish a relationship between the FT-IR results and the temperature.

Results and Discussion

FT-IR results

The details of the FT-IR analysis are presented in Table 1, and Figure 2 shows the percentage absorption at different temperatures. Although, the *A. achatina* shells that calcined at 200 °C, it showed major absorption at 1463.44 cm⁻¹ which is the asymmetric stretching for CO₃²⁻ that is present in the *A. achatina*. This is within the absorption rate (1427cm⁻¹) reported by Aldes et al. [14].

Table 1. Results of Fourier transformed infrared spectroscopy of calcined shell of *A. achatina*

Sample (°C)	Asymmetric Stretching of CO ₃ ²⁻ (cm ⁻¹)	Out of Plane Vibrational Modes of CO ₃ ²⁻ (cm ⁻¹)	In Plane Vibrational Modes of CO ₃ ²⁻ (cm ⁻¹)	O-H Stretching Band (cm ⁻¹)
C ₀ (0)	1455.19	854.35	707.83	-
C ₁ (200)	1463.44	855.78	708.27	-
C ₂ (400)	1456.02	856.77	709.41	-
C ₃ (600)	1389.05	870.20	710.56	-
C ₄ (800)	1410.44	870.16	-	3637.98
C ₅ (1000)	1410.18	872.30	-	3637.70

Furthermore, the absorptions rates for out of the plane and in-plane band vibrational modes for CO₃²⁻ were 855.78 cm⁻¹ and 708.27 cm⁻¹, respectively. Interestingly, this is in agreement with the out of the plane and in-plane band

vibrational modes absorptions (871 cm⁻¹ and 709 cm⁻¹) that were recorded by Aldes *et al.*, [14]. Also, at 400 °C, the absorption of 1456.02 cm⁻¹ is attributed to the asymmetric stretching of CO₃²⁻

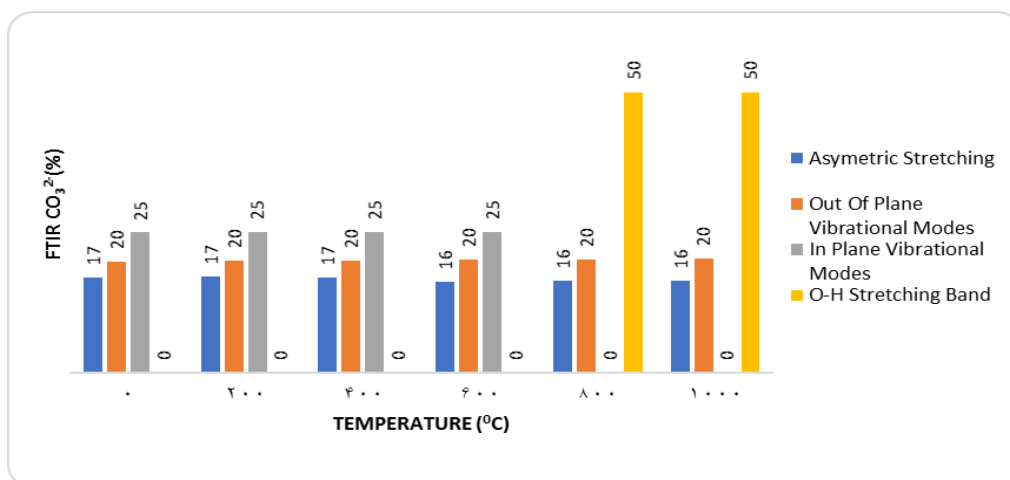


Figure 2. FT-IR results of the calcined shell of *A. achatina*

The out of the plane and in-plane bands vibrational modes for CO₃²⁻ observed at 856.77

cm⁻¹ and 709.41 cm⁻¹, respectively. Asymmetric stretching of 1389.05 cm⁻¹ is the dominant

absorption for CO_3^{2-} at 600 °C while 870.20 cm^{-1} and 710.56 cm^{-1} were recorded for out of the plane and in-plane band vibrational modes for CO_3^{2-} , respectively. Significant absorption at 1410.44 cm^{-1} , 870.16 cm^{-1} , and 708.27 cm^{-1} at 800 °C confirmed the presence of C-O stretching and bending vibrational modes of the CaCO_3 , showing that the CaCO_3 is present at this temperature. It was observed that as the temperature is increasing the calcined *A. achatina* starts to lose the carbonate ion. At 800 °C, a sharp O-H stretching band starts to appear at 3637.98 cm^{-1} and 1000 °C the O-H stretching band is also observed at 3637.70 cm^{-1} .

Also, the investigation of the significant relationship between the temperature and FT-IR analysis at 95% confident intervals and alpha = 0.05 was considered (As can be seen in Table

2). The study, however, showed a strong negative correlation between increasing the temperature and asymmetric stretching of CO_3^{2-} (-0.772). This indicated that the rate of absorption of the asymmetric stretching of CO_3^{2-} does not depend on rising the temperature. In contrast, there was a strong negative correlation between In Plane vibrational modes with temperature (-0.827) while on the out plane vibrational mode the relationship was strongly positive (0.928). Moreover, there was a strong relationship between O-H Stretching band and temperature (0.828). However, there is a significant difference in the relationship between the temperature and all the indices for absorption rates ($p < 0.05$) as shown below.

Table 2. Comparison of the FT-IR results

	Asymmetric Stretching of CO_3^{2-} (cm^{-1})	Out Of Plane Vibrational Modes of CO_3^{2-} (cm^{-1})	In Plane Vibrational Modes of CO_3^{2-} (cm^{-1})	O-H Stretching Band (cm^{-1})
r	-0.772	0.928	-0.827	0.828
95% CI	-0.974 to 0.107	0.473 to 0.992	-0.98 to -0.046	0.0503 to 0.981
R ²	0.596	0.862	0.683	0.686
P (one-tailed)	0.0361	0.0038	0.0212	0.0209
(alpha=0.05)	Yes	Yes	Yes	Yes

Microstructure analysis

The microstructure of the *A. achatina* powder calcined at different temperatures was observed using SEM (Figure 3a-f). At 0 °C (C_0), SEM images show irregular at 10,724X magnification with a surface area of 10 micrometres. SEM results showed that the structure of snail shell changed with calcination temperature. The SEM images revealed that calcination at 200 °C (C_1) was not uniform and it gave a cluster structure of 10,000X magnification with a particle size of 5 μm . At

400 °C (C_2), the SEM images at this temperature gave a microstructure and irregular structure. With a magnification of 10,000X at a calcination temperature of 600 °C (C_3), an irregular structure was observed. At a higher calcination temperature of 800 °C (C_4) and 1000 °C (C_5), it was found that the clustered surface was slowly varnishing and the particles with various size and shapes became visible. More regular and uniform structure and agglomerate into lumps were observed in the C_4 and C_5 specimens, respectively.

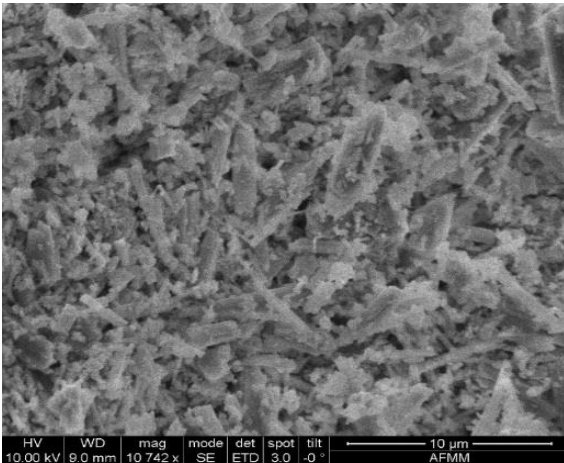


Figure 3. a) SEM at C₀ (0 °C)

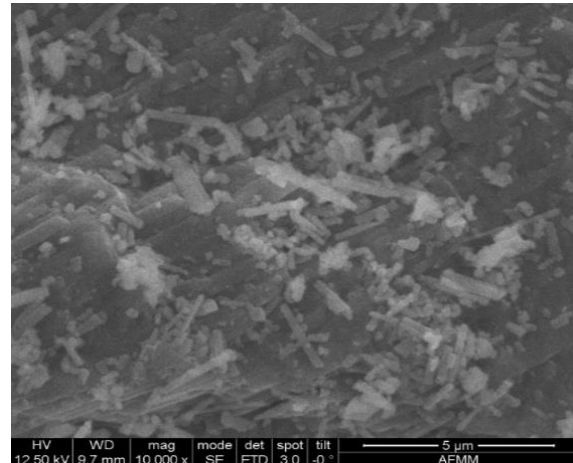


Figure 3. b) SEM at C₁ (200 °C)

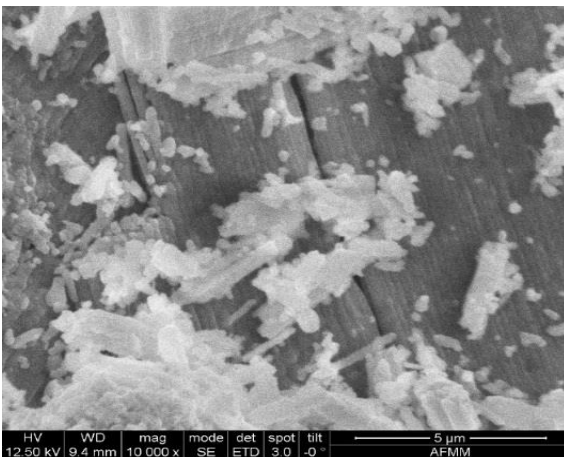


Figure 3. c) SEM at C₂ (400 °C)

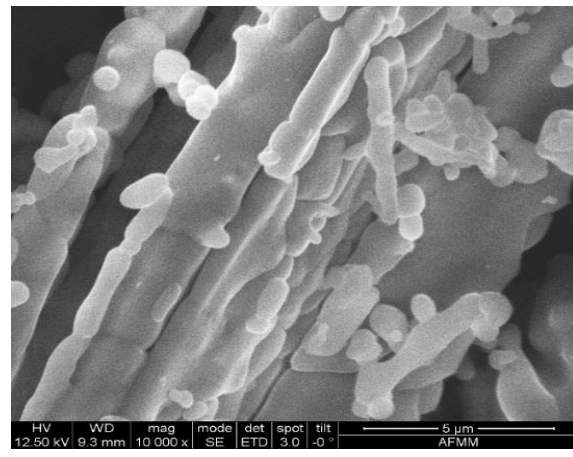


Figure 3. d) SEM at C₃ (600 °C)

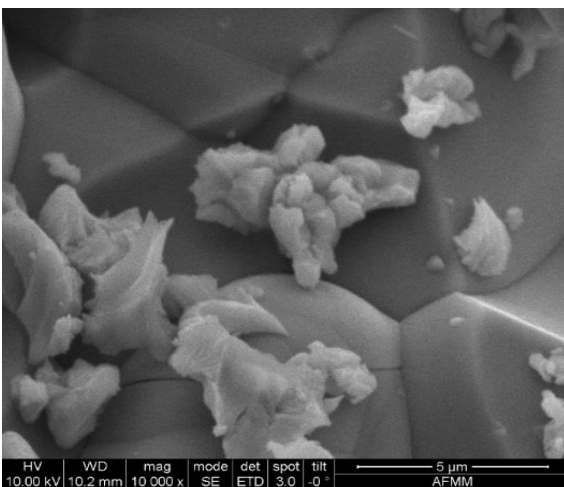


Figure 3. e) SEM at C₄ (800 °C)

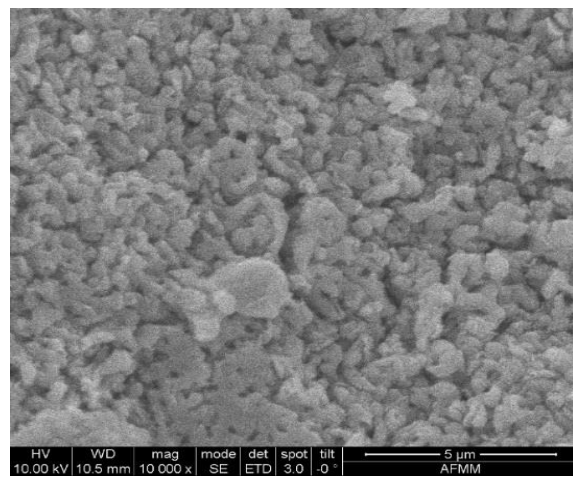


Figure 3. f) SEM at C₅ (1000 °C)

Conclusions

Biodiesel is said to be produced from vegetable oils and methanol using calcium oxide to get fatty acid methyl ester (FAME) which is a biodiesel. The homogeneously catalyzed reaction is used in producing biodiesel; however, it is unsatisfactory as it has high cost and leads to soap formation (base catalyzed). Also, the heterogeneous catalyst is of high usage because they are readily available and can be re-used. *A. achatina* shells is a major source of calcium oxide that can be used as a heterogeneous catalyst for transesterification process because they easily obtained, not expensive and can be reused.

Disclosure statement

No potential conflict of interest was reported by the authors.

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How to cite this manuscript: Oluwatobi O. Amusan*, Hitler Louis*, Adejoke T. Hamzat, Oluwatomisin F. Oyebanji , Ayodeji T. Alagbe, Thomas O. Magu. Synthesis and characterization of CaO catalyst Obtained from *achatina achatina* and its application in biodiesel Production. *Journal of Medicinal and Nanomaterials Chemistry*, 2019, 3(1), 271-277. DOI: [10.48309/JMNC.2019.3.2](https://doi.org/10.48309/JMNC.2019.3.2)