Synthesis of Biodiesel from Waste Cooking Oil by Using Seashells as Catalyst

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Summary: The basic aim of this research is to study the suitable conditions of the biodiesel by using waste refined cooking oil as a preliminary material with heat-treated seashells at 500℃, 800℃, and 1000℃ as heterogeneous catalysts. It recorded at 800 ℃ a high level of CaO as catalyst 90.89 % under the following reaction conditions: catalyst is 0.5 g, temperature at 80 ℃, reaction duration is 180 minutes, and the oil-to-alcohol ratio is 10:20%. The impact of energizing temperature on transesterification processes in the range of 500℃–1000℃ proved that the calcination temperature at 800 ℃ of seashell-derived material raised biodiesel to 97% due to active sites on catalyst surfaces, converting rate linearly in comparison to other catalysts, and this tends to reduce the cost of purification procedures of biodiesel. It has been proved that the catalyst at 800℃ possesses a density at 40℃ of 0.850 g /cm^3, an acid value of 0.6 mg KOH/g ^{oil}, a viscosity of 4.9 mm^2/s, and a flash point of 165°C. It also contains high catalytic activity in the transesterification process of wasted cooking oil (WCO) for optimizing biodiesel characteristics in the future. As a result, the catalyst demonstrated high action in the transesterification effect. Scanning Electron Microscopy (SEM) illustrates calcined waste shells are irregular in shape, which means small, minute grains of pores accumulate, providing a large area of surface. Furthermore, the physical characteristics of CaO catalysts at 800 ℃ showed Brunauer- Emmett-Teller (BET) a significant surface area of 76.1888 m^2/g and pore volume of 2.6539 cm^3/g is considerably greater than other catalysts at temperatures of 500 ℃–1000 ℃. X-ray diffraction (XRD) data analysis demonstrate the main constituent of seashells is $CaCO_3$, indicated by the diffraction peak at 2θ approximately 28.48°. However, carbon dioxide CO_2 changes when the calcination temperature at 800 ℃ it transforms $CaCO_3$ completely into CaO, which is the main component of the calcined catalyst. The CaO catalyst's well-crystallized structure is defined by the narrow, high-intensity peaks of catalyst. Eventually, the heterogeneous catalyst led to a significant rise in the active sites.

Keyword: Biodiesel, Seashell, Esterification, CaO Catalyst.

Introduction:

The growth of industry and population are the main causes of the ongoing increase energy intake [1]. Excessive use during the coming period will steer to a decreasing in the amount of energy produced from hydrocarbon fuels [2]. Fossil fuels have a detrimental effect on the environmental which linked to price instability [3]. In addition, it has resulted in climate change, environmental deterioration, and a rise in the health risks associated with pollution for the global population [4]. By focusing on these factors, it is imperative that renewable energy sources can be found quickly. Among these resources are hydro, solar, and wind energy, as well as biofuels, which could aid in addressing current global issues [5]. Researchers are looking for sustainable and alternative energy sources in response to these issues. Among an eco-friendly and renewable resource, biodiesel holds a great advantage as a substitute fuel, compared to petroleum diesel, as it emits fewer hydrocarbons and particulates non-toxic and biodegradable [6,7]. One of the advantages of biodiesel, it does not contribute to increasing carbon dioxide in the atmosphere, which leads to reducing the severity of global warming, meaning it is considered better than petroleum diesel fuel.[8].Vegetable and animal fats are examples of natural and renewable resources from which biodiesel is derived.[9].It is ensures a long life for engines due to its high viscosity compared to petroleum diesel [10].Seashells are a rich source of calcium that is used to extract calcium oxide which considered as a great source of heterogeneous catalysts[11]. Calcium oxide (CaO) has a promising place among the heterogeneous catalysts used in transesterification, a numerous publication on CaO-catalyzed transesterification utilizing laboratory-grade materials have been reported. It comes at a low cost, which is widely seen in nature (as limestone), its comes from some renewable resources waste material made of calcium carbonate, or $CaCO_3$ [12]. A good catalyst can increase the solubility of alcohol, which increases the rate of reaction and indirectly the amount of biodiesel produced [13]. Furthermore, the environmental impact of using petroleum-based fuels for industrial and transportation purposes is frequently overlooked in

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developing countries [14,15]. On the other hand, the use of waste materials as heterogeneous catalysts has garnered attention recently in the quest for a sustainable procedure [16]. These shells might also be useful in other significant organic reactions that are catalyzed by bases, adding value to the waste that is produced [17]. There are many positive aspects of biodiesel very well-liked renewable energy source. But at the moment, its output cannot keep up with the rising demand. Extreme parameters production conditions are needed to generate biodiesel without the use of catalysts; these includes longer reaction times, higher expenditures, higher energy requirements, and higher temperatures. Moreover, low conversion efficiency, reaction speed, and product yield are linked to the manufacture of biodiesel without catalysts [18,19]. Homogeneous catalysts do have certain disadvantages including the inability to be recycled production of low-quality glycerol, and a significant volume of wastewater [20,21]. For example, a variety of heterogeneous alkali catalysts, particularly those based on CaO, have been extensively employed in the biodiesel production because of their high basicity, high catalytic activity, abundance, and affordability [22-24]. Dolomite rocks and other natural dump sources that include calcium carbonate $CaCO_3$ are good sources of CaO [25]. Including waste eggshells [26], leftover of mollusk, clamshells [27]. Waste cooking oil can be utilized as a feedstock for affordable, sustainable manufacturing of biodiesel, as opposed to being disposed off into the eco-environment, which may pollute water and soil [28].

Experimental

Materials

Chemical structure of Biodiesel

Its known that, alkali catalyzed reactions are greater 4000 times than acid catalyzed reactions [27]. As shown figure (1), esterification reaction produce by 1 mole of triglycerides and 3 moles of alcohol react to create one mole of glycerol and three moles of mono-alkyl ester biodiesel. Biodiesel is composed of methyl and ethyl ester. Its chemical formula is generally R - COOR1 where R1 is the methyl or ethyl radical attached to the acid radical Fat - COOR, noting that R is a saturated, unsaturated chain containing one or more double bonds (28).

Preparation of seashell catalyst

Seashells were collected, then clean, wash, transport to the drying oven and were heat treated at different temperatures (500°C, 800°C, 1000°C).

Preparation of waste cooking oil

Waste cooking oil is collected from restaurants, then purified and filtered from suspended impurities before using it.

Preparation of Biodiesel from waste cooking oil Trans-esterification

- The heat-treated seashells are ground well and weighted by using a sensitive scale.
- Take the required volume of 20% ethanol and 10% cooking oil using a grade tube tester measure its weight and record it, and 1 gram shell catalyst in a water bath with a magnet for mixing on the electric heater for 3 hours.

Place the resulting products in a separating funnel and leave it for 24 hours. Its notice that, separated two layers, an upper layer is biodiesel and a lower layer is glycerol as a byproduct, then used the electronic pipette for extracting biodiesel only which placed on the electric heater for 10 minutes to get rid of the alcohol residue, then percentage of the amount of biodiesel is taken calculate for percentage of biodiesel. According to the equation below (1):

$$\text{Yield}(\%) = \frac{\text{Weight of biodiesel produced}}{\text{weight of sample oil used}}\, x\, 100$$

- Take the bottom layer of glycerol and set it aside for later use [29].

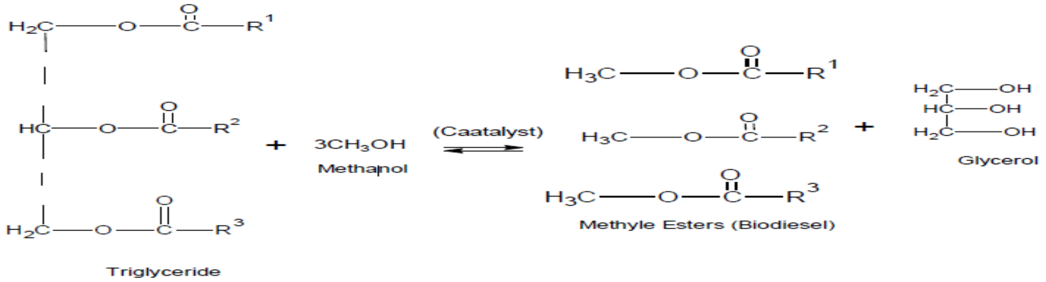

Fig. 1: Scheme of Chemical reaction to produce biodiesel.

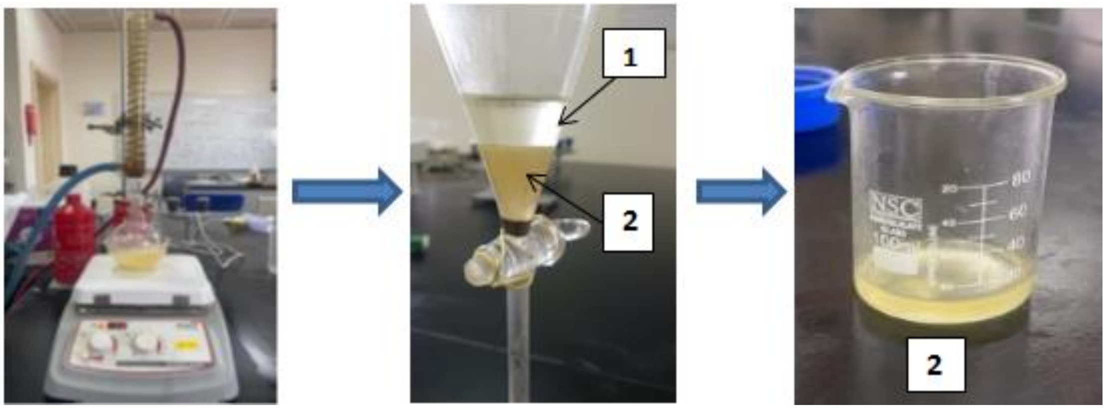

Fig. 2: Biodiesel (1) and glycerol (2) separation after 24 h.

Results and Discussion

Characteristics of waste cooking oil

The results are displayed in Table-1. The biodiesel layer was filtered and separated, and the density, acid value, and free fatty acid value of waste cooking oil were assessed.

Table-1: Physical and chemical characteristics of used cooking oil (WCO).

Property	Unit	Measured value
viscosity at 40°C	**Cst**	**0. 35**
Acid value	**mg KOH/g ^{oil}**	**1.50**
Flash point	**°C**	**263**

Preparation of the catalyst

As shown in figures (3&4). Seashells that were from the sea after treatment in warm water were gathered, cleaned several times with tap water, and then rinsed with distilled water to remove any remaining toxins. The seashells were then oven-dried at 120°C for 16 hours. After drying, it grounded, and the powders were discarded. Seashells were undergoing calcination in a muffle furnace for a duration of 3 hours, temperatures of (500°C - 1000°C).

Fig. 3: Catalyst preparation using waste seashells.

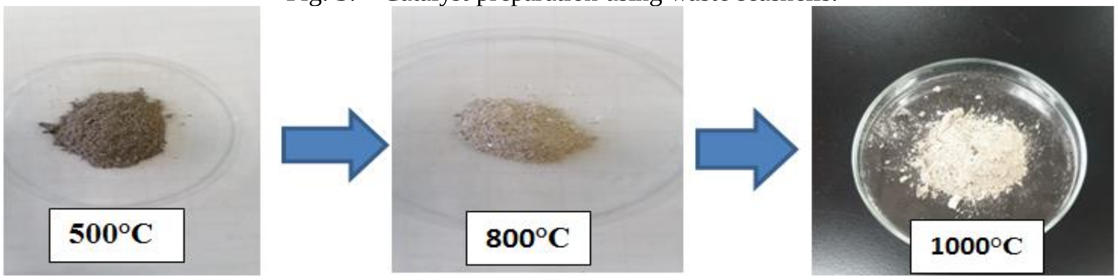

Fig. 4: Treated seashells at different temperatures (500°C,800°C &1000°C), calcination and grinding.

Characteristics of biodiesel

Findings are shown in Table-2. The assessment of density, acid value, viscosity, and flash point of the biodiesel layer.

Table-2: Characteristics of biodiesel produced under optimal conditions [29].

Property	Unit	Measured value
Density	g/c^3	0.850
Acid value	mg KOH/ g ^{oil}	0.6
Viscosity @40°C	mm^2/s	4.9
Flash point	°C	165

Characterizations of calcined seashells catalyst

Scanning Electron Microscopy (SEM) analysis

SEM was used to analyze the morphology of waste seashells [30] that were calcined at various temperatures of (500°C, 800°C, and 1000°C. Figure (5) provides a summary of the CaO catalyst's physical characteristics and showed a significant surface area of 85.70 m^2/g and pore volume of (0.120 cm^3/g at 800°C with consistent pore size. Multiple layers are visible in the natural shell [31]. The microstructures of natural shells undergo a considerable transition from multilayered to porous structures when the calcination temperature is raised from 500°C -800 °C It was found that the calcined waste shells are irregular in shape [1-2], means small, minute grains of pores accumulate, providing a large area of surface. The catalyst broken at 1000 °C and active site turns into inactive due to high temperature [3]. Notice at 500°C -1000°C, there is a decrease in pore volume (0.119-102 cm^3/g) and surface area (57.91-33.9 m^2/g) respectively. Evidently, the heterogeneous catalyst led to a significant rise in the active sites [32].

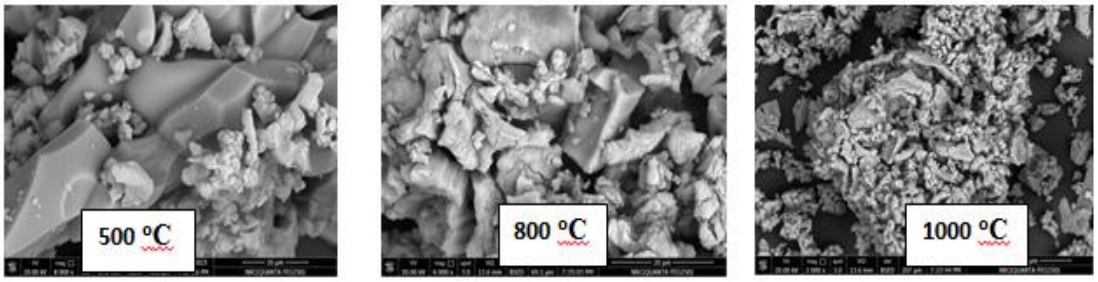

Fig. 5: SEM image of sea shell calcined (500 °C,800 °C &1000 °C.

Brunauer- Emmett-Teller (BET) analysis

Table (3), showed that, the catalysts were calcined at 800°C showed a higher pore volume and BET surface area of (76.1888 m^2/g). It's also presented the catalyst exhibited high catalytic activity in the transesterification of cooking oil for the manufacture of biodiesel. Whereas at 500°C &1000°C showed less pore volume and BET surface area (24.9506 m^2/g & 12.7217 m^2/g) respectively.

Table-3: BET analysis of seashells calcined catalyst at different temperatures.

No	Surface area (m^2/g)	Pore Size (nm)	PoreVolume (cm^3/g)
500 °C	24.9506	2.3677	0.23432
800 °C	76.1888	2.6539	0.091243
1000°C	12.7217	2.3952	0.12214

X-ray diffraction (XRD) analysis

Based on the XRD data displayed in figure (6), the main constituent of seashells is $CaCO_3$, as indicated by the diffraction peak at 2θ approximately 28.48°. However, carbon dioxide (CO_2) changes when the calcination temperature rises, $CaCO_3$ completely transforms into CaO. At temperatures 800°C calcium oxide CaO is formed which is the main component of the calcined catalyst. The CaO catalyst's well-crystallized structure is defined by the narrow, high-intensity peaks of catalyst [33], which suggests that calcined material was well-crystallized during the heat treatment procedure [34].

Analysis of mineral salts in seashells catalyst at (500 °C -800°C)

Mineral salts (Na, Mg, P, K, Cao and oxygen) have been measured in heat treated seashells used as catalyst at (500°C -800°C). It was demonstrated that both (800 °C –90.89%) and (500°C–62.82%) had elevated calcium levels and moderate level of sodium (800 °C –20.98%) and (500 °C –10.32%). As shown in table (4&5) figure (7&8) respectively.

Table-4: Measurement of mineral salts of waste shells at 800°C –derived catalyst.

Element	Weight %	Atomic %	Net Int	Error %
Na	**20.89**	**3..43**	**2.23**	**46.23**
Mg	**0.6**	**0.68**	**0.88**	**76.63**
P	**1.48**	**1.3**	**3.34**	**41.15**
K	**0.74**	**0.51**	**1.83**	**65.06**
Cao	**90.89**	**44.09**	**106.48**	**3.58**
Oxygen	**29.38**	**49.99**	**0**	**0**

Element	Weight %	Atomic %	Net Int	Error %
Na	**15.32**	**5.05**	**4.4**	**30.34**
Mg	**1.13**	**1.25**	**2.15**	**69.62**
P	**1.8**	**1.56**	**5.31**	**33.58**
K	**0.23**	**0.16**	**0.75**	**72.98**
Cao	**62. 82**	**42.11**	**136.22**	**3.43**
Oxygen	**29.7**	**49.87**	**0**	**0**

Table-5: Measurement of mineral salts of treated seashells at 500°C.

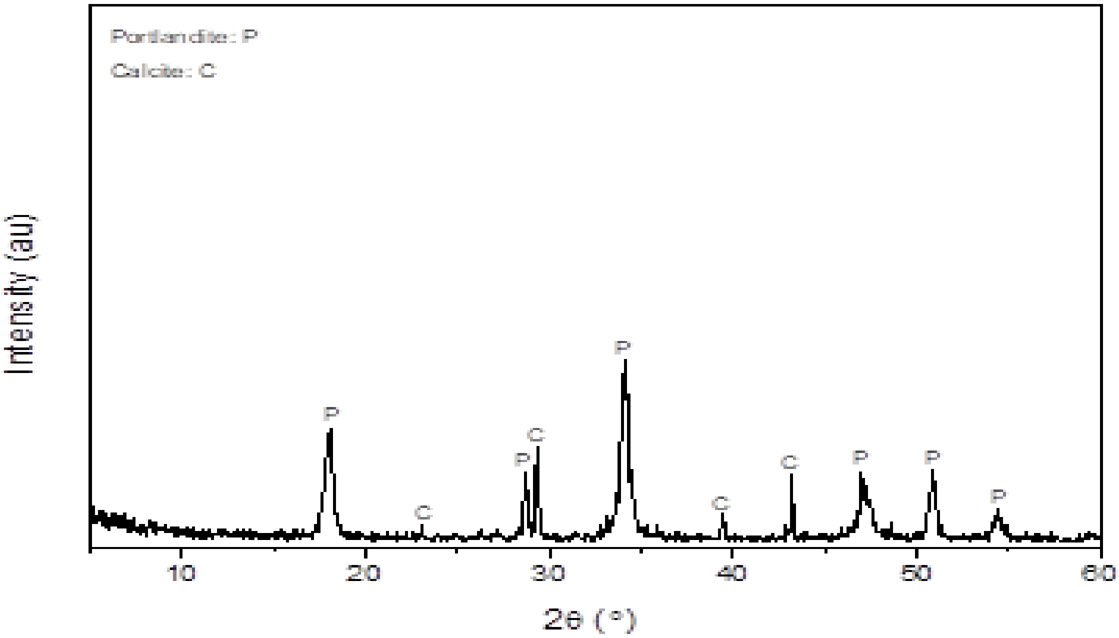

Fig. 6: Synthesized catalysts from waste seashells (hydroxyapatite) calcined at different temperature.

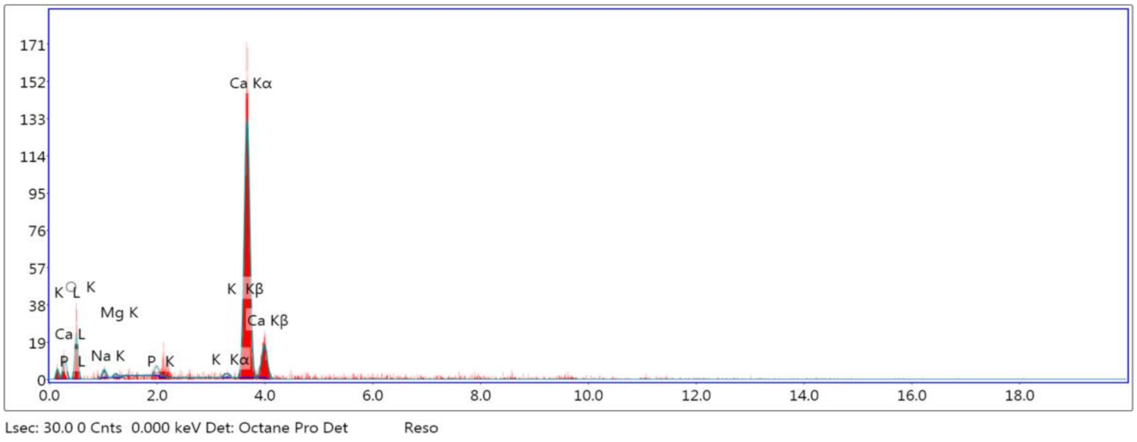

Fig. 7: Measurement of mineral salts of treated seashells at 800°C.

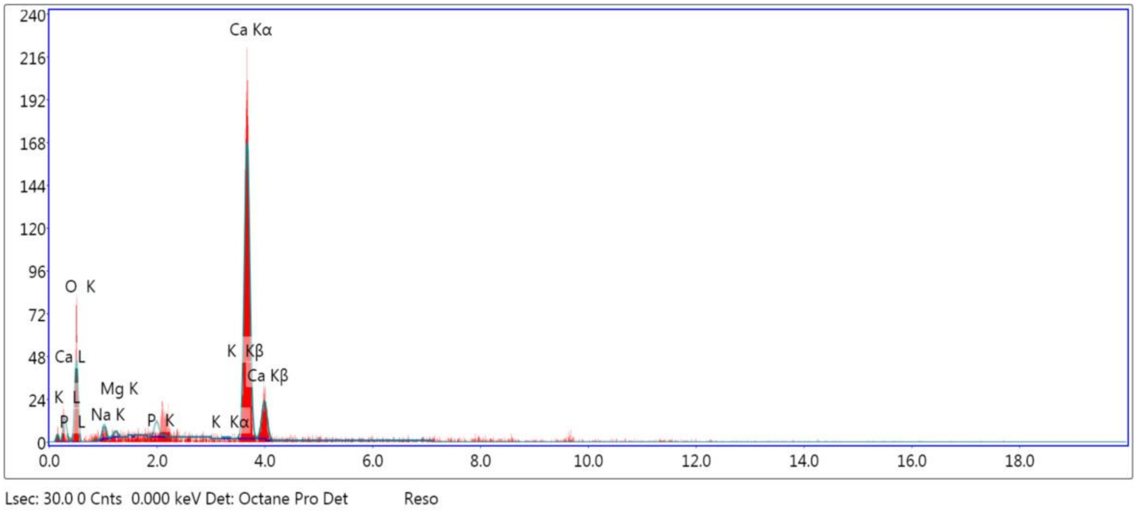

Fig. 8: Measurement of mineral salts of treated seashells at 500°C.

Reaction circumstances impact of biodiesel production and temperature of calcinations effects on catalyst activity:

As shown figure (9), the impact of temperature on transesterification activity which investigated at (500°C –1000°C). The findings showed that the biodiesel conversion increased linearly with increasing activation temperature for the CaO catalyst obtained from seashells. depending on catalyst under some reacting condition such as: loading of 4 weight percent, and the oil-to-alcohol ratio is 10:20%, a reaction time of 3 hours, and a reacting temperature of 80°C.The catalyst that was calcined at 800 °C exhibited the maximum catalytic activity (97%), owing to the presence a number of optimal active sites on its surface as displayed.

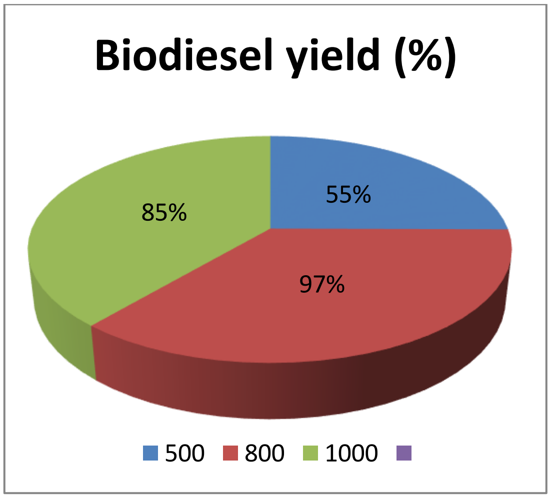

Fig. 9: Effects of calcination temperature(500°C, 800 °C &1000 °C) on seashells catalyst activity.

The effect of reaction time on biodiesel yield (%)

Three experiments were conducted at (60-120-180 m) minutes with other conditions constant. It has been noticed that 180 minutes reaction is the highest in biodiesel yield, followed by 120 minutes while less formation rate of biodiesel seen at 60 minutes, due to incomplete reaction process. We noticed that at increasing reaction time, biodiesel yield increases as shown in the following table (6) & figure (11) respectively.

Table-6: Effect of reaction time on biodiesel yield.

Percentage of biodiesel formation (%)	Time / minute
83%	60
94%	120
97%	180

The effect of temperatures on biodiesel yield

Three experiments were conducted at different temperatures (60°C -80°C -90°C). It was noted that at 60°C, the percentage of biodiesel formation was 55%, which is the lowest possible, when at 80°C the highest percentage was 97%, being the degree of complete reaction. Meanwhile at 90°C the reaction tends to decreases to 85% because in higher temperatures, ethanol lead to evaporate and reduce its presence in the reaction medium, and accordingly the percentage decreases as shown in figure(11)

The effect of catalyst amount on biodiesel yield (%)

Seashells were used as a catalyst (0.5 g - 0.75g) due it contains high level of calcium oxide (64.89-62.82) on 800-500°C respectively. We note that increasing the weight of catalyst increases the biodiesel productivity. As well as the catalyst at 0.75g leads to lower biodiesel yield due to problems in diffusion during the reaction and thus hindering the work of the catalyst, or it may be increase in the amount of catalyst which led to the formation of glycerol (by product) instead of biodiesel which is main output. As shown in figure (12).

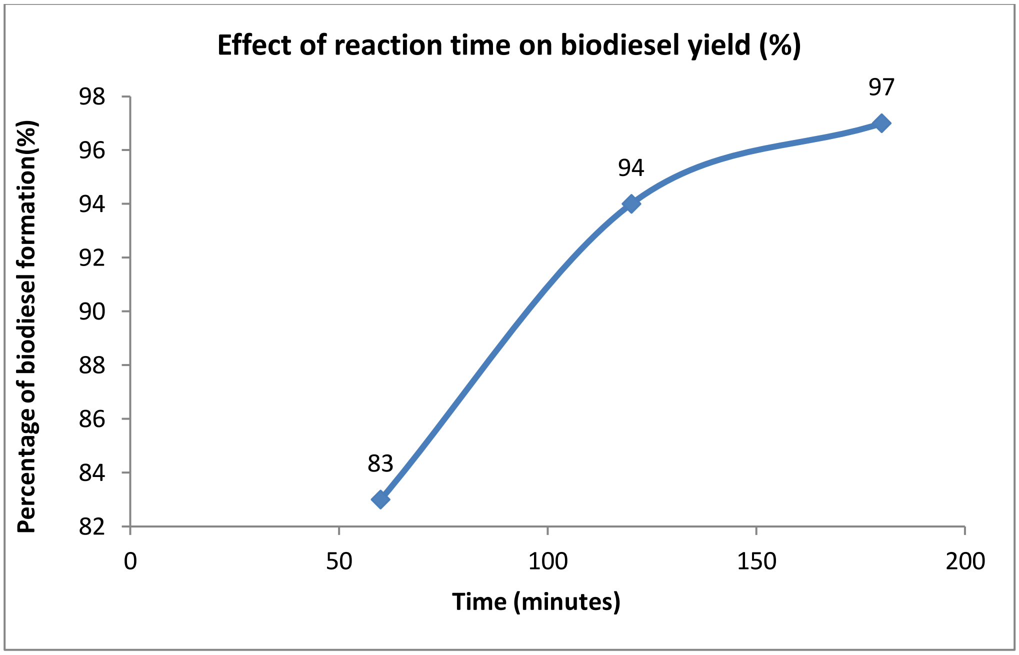

Fig. 10: Effect of reaction time on biodiesel yield(%).

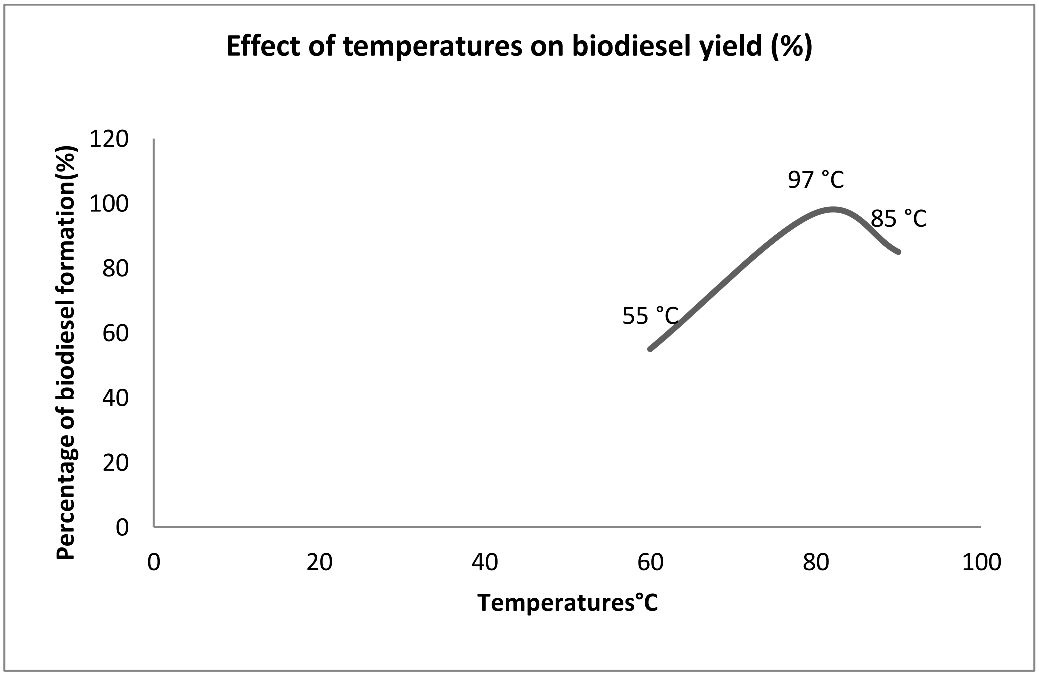

Fig. 11: Effect of reaction temperature on biodiesel yield (%).

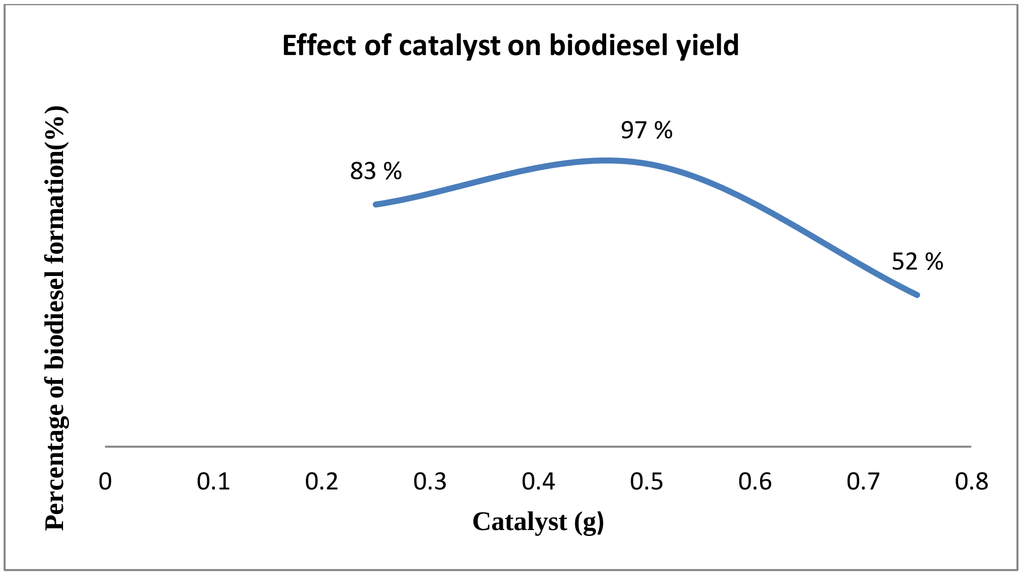

Fig. 12: Effect of catalyst on biodiesel yield (%). Reaction conditions: an oil/ethanol ratio of 1:9 reaction time 3 h, and reaction temperature of 80 °C.

Table-7: Comparison with previous studies.

Catalyst	Raw material	Catalyst Weight (%)	Biodiesel yield (%)	Reaction temp.(ºC)	Reaction Temp(h)	Stirring Rate (rpm)	Alcohol	Alcohol molar ratio	Reference
Seashells	**WCO**	**4**	**97**	**65**	**4**	**700**	**Ethanol**	**1:12**	**[35]**
		3	**90**	**120**	**4**	**700**	**methanol**	**1:9**	**[36]**
		4	**95**	**65**	**3**	**1000**	**Ethanol**	**1:9**	**[37]**
		4	**85**	**80**	**2**	**900**	**methanol**	**1:12**	**[38]**
		4	**97**	**80**	**3**	**800**	**Ethanol**	**1:9**	**This work**

Conclusion

Using a catalyst made from seashells, this study looked at waste cooking oil as a potential non-food source for the synthesis of biodiesel. The catalyst is made of $CaCO_3$, which is calcined for three hours at 800°C to produce CaO, which at 80°C has a molar ratio of methanol to oil 20:10%. In ideal circumstances, the conversion of cooking oil would be approximately 97%. All biodiesel requirements are satisfied by the fuel qualities of the produced biodiesel. It was determined that the biodiesels properties met the requirements of ASTM D6571, an American fuel standard. CaO can lower the price of biodiesel and purification processes since it is a solid catalyst. It can be applied into mass industrial production to convert waste cooking oil into methyl ester by transesterification. This strategy may make it easier to produce inexpensive biodiesel using environmentally acceptable technology. Future green energy initiatives may benefit the environment, which could eventually enhance the social and economic growth of the biodiesel industry on a global scale.

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