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Production of Karanja Methyl Ester from Crude Karanja Oil Using *Meretrix Lyrata* Synthesised Active Cao Catalyst

D. Harreh[1]*, A. A. Saleh[1], A. N. R. Reddy[1], S. Hamdan[1] and K. Charyulu[2]

[1]Faculty of Mechanical Engineering, Universiti Malaysia Sarawak,
94300 Kota Samarahan, Sarawak, Malaysia
*Email: dewiharreh@gmail.com
Phone: +6082583299
[2]Malla Reddy College of Engineering and Technology, Maisammaguda,
Dullapally, Hyderabad, Telangana 500043, India

ABSTRACT

Active calcium oxide catalyst was synthesised from *Meretrix Lyrata (M.Lyrata)* following calcination-hydration-dehydration technique. The catalytic feasibility of synthesised CaO was investigated in the production of Karanja methyl ester (KME) from crude Karanja oil (CKO). KME was synthesised through esterification using H_2SO_4 followed by transesterification utilising CaO in a two-step reaction process of CKO and methanol. The *M.Lyrata* shells were calcined at 900 °C and the catalyst samples were characterised using FTIR, SEM, PSA, and BET-BJH spectrographic techniques. A maximum fatty acid methyl ester (FAME) conversion of 97.3 % was obtained at optimum reaction conditions including methanol-to-oil ratio of 12:1, catalyst concentration of 2 wt.%, reaction temperature of 58 °C and reaction time of 2 hrs. In a comparative study with commercial CaO, *M.Lyrata* showed a higher catalytic activity. The catalyst reusability experiments ascertaining reusability of CaO up to four reuse cycles had shown good efficiency. The economic comparative study confirms that CaO derived from *M.Lyrata* can be used as an alternative and feasible catalyst for biodiesel production. The KME fuel properties complied to EN-14214 biodiesel fuel standards.

Keywords: Seashells; heterogeneous catalyst; transesterification; Karanja biodiesel; spectroscopy.

INTRODUCTION

Biodiesel, also known as fatty acid methyl ester (FAME) or fatty acid ethyl ester (FAEE) can be obtained from vegetable oils or animal fats [1, 2]. The rapid depletion of fossil fuel, demand for engine fuels that keeps growing tremendously and its unpredictability in future availability, lead to the crucial factor to explore on alternative renewable fuel sources that will be able to supplement fossil fuels [3-6]. In recent years, biodiesel had shown exciting potential and received worldwide attention being recognised as a substitute for petro-based diesel fuel. Biodiesel is known for its sustainability, non-toxicity, biodegradability, reduced gaseous pollutant emissions and easy usability as fuel to be used in conventional diesel engines without major modifications as compared to petroleum diesel [7, 8]. Production of biodiesel globally was over 5 billion gallons in 2010. Transesterification is considered to be the most reliable method for biodiesel production using vegetable oil feedstocks [9, 10]. There are several types of catalysts used for biodiesel production via transesterification reaction such as homogenous catalyst, heterogeneous catalyst and certain enzymes [11-13]. Usually KOH or NaOH are frequently being used as base homogenous catalyst, since the rate of reaction is relatively fast [14-16]. Unfortunately, the use of homogenous catalyst causes a high volume of wastewater discharged during the purification or washing process of biodiesel production in order to remove the residual catalyst present. This results in water contamination as well as increase the cost of water treatment plants. The used catalyst from the reaction cannot be recovered and reutilised backed into the process. Severe problems might arise such as formation of soap during transesterification which is caused by the presence of large amount of FFA content in vegetable oil. This will cause a decrease in catalytic efficiency [17-19]. Contrary to homogenous catalyst, heterogeneous catalyst has a number of benefits; for instance, utilisation of heterogeneous catalyst which is easily separable from biodiesel product and recyclable. This leads to an economical way for biodiesel production by reducing the consumption of wastewaters and can be reused for next transesterification [20].

In recent times, research studies on the use of calcium oxide (CaO) as a heterogeneous catalyst have been carried out owing to its low cost [21, 22] and high basicity [23]. Additionally, CaO is readily availability in nature, and can be derived from natural waste materials such as seashell, rice husk, egg shell, mussel shell and chicken bones making the production of biodiesel more cost-effective, added further by its low stability, non-toxicity and high basic properties [24-26]. This present research focuses on synthesis of active CaO catalyst using locally available waste seashells as raw material. An economically feasible laboratory scale synthesis technique, calcination-hydration-dehydration is adopted in the CaO synthesis. Newly synthesised catalyst samples were characterised using FTIR, SEM, PSA, and BET-BJH spectrographic techniques. Karanja methyl ester (KME) production was experimented with newly synthesised active CaO using a two-step transesterification protocol.

EXPERIMENTAL SET UP

Materials

The crude Karanja oil (CKO) was extracted from their seeds using soxhlet apparatus and mechanical expeller. According to Mahanta and Shrivastava (2004), soxhlet apparatus yielded better quality and higher amount of vegetable oil [27]. Experimental data revealed acid value and saponification value for CKO as 5.06 mg KOH/mg and 187 mg KOH/mg, respectively [28]. The laboratory grade chemicals that include methanol (MeOH; >99% pure), H_2SO_4(95-97%), calcium oxide, distilled water and NaOH were used for experimental protocols. The biodiesel production was carried out at the Energy Laboratory of the Faculty of Engineering, Universiti Malaysia Sarawak (UNIMAS) Sarawak, Malaysia.

Catalyst Synthesis

Initially, the *M.Lyrata* shells were collected from a local market situated at Samarahan, Sarawak, Malaysia. The shells were then rinsed simultaneously using tap, hot and distilled water to remove any dirt and stain as well as impurities on the shells surface [29]. *M.Lyrata* shells were dried overnight in a hot-air oven at 105 °C [30]. The dried shells were then grinded to fine powder form using a commercial blender and sieved through an 80µm mesh. The CaO derived from *M.Lyrata* was calcined at 900 °C for 2 hrs in a muffle furnace (KSL-1700X-A4, MTI Corporation, USA), then the shell powders were refluxed in distilled water for 6 hrs at 60 °C. The samples were filtered and dried overnight at 120 °C for 3 hrs as well as recalcined for 3 hrs at 600 °C to produce fine calcium oxide. The CaO samples were dried in a hot air oven at 100 °C for 1 hr and finally stored in an air-tight sample bottle [29]. Thus, the CaO was successfully synthesised as an active heterogeneous CaO catalyst synthesis from *M.Lyrata* by 'calcination-hydration-dehydration' technique.

CaO Catalyst Characterisation

The chemical composition of *M.Lyrata* shells synthesised CaO was evaluated over the wavelength region of 600 cm^{-1} to 4000 cm^{-1} by employing Fourier Transform Infrared Spectrophotometer (FTIR) (Perkin Elmer Model 100 series). A scanning electron microscope (SEM) (Hitachi TM3030) was utilised to study the particle morphologies and elemental analysis of both calcined CaO and commercial CaO. A particle size analyser (PSA) (CILAS-1090) was used to analyse particle size distribution of *M.Lyrata* shells synthesised catalyst. The surface area of calcined *M.Lyrata* seashell and commercial CaO were estimated using Brunauer-Emmett-Teller (BET) equation while pore diameter was determined using Barrett-Joyner-Halenda (BJH) [31, 32].

M.Lyrata CaO Catalyst Economic Synthesis Study

M.Lyrata are available abundantly in the state of Sarawak, Malaysia and largely consumed as a food source. Thus utilisation of *M.Lyrata* waste shells as a renewable source is substantially potential. Further, shell-based wastes were utilised as renewable sources for synthesising heterogeneous catalysts for producing biodiesels.

Experimental Set Up for Biodiesel Production

The free fatty acid (FFA) contents of CKO range over 20% which results in lowering biodiesel yield especially in transesterification of vegetable oils using heterogeneous solid catalyst [29, 33]. Since the FFA of CKO is higher than the required value (>1%), KME derived from CKO is achieved by a two-step process [33]. According to Ahmad et.al, transesterification process transforms crude oil to fatty acid methyl ester [34]. The KME was produced by performing pre-treatment (esterification) followed by alkaline (base catalysed) transesterification [9].

Esterification: Initially, CKO was heated up to 100 °C for 30 min. to remove moisture, then acid pre-treatment process was applied to reduce the FFA content by using 0.8 %H_2SO_4 (v/v) added to oil, and next mixed with 6:1 (methanol-to-oil ratio) molar ratio. The solution was heated to 50 °C and stirred for 90 min. Subsequently, the solution was transferred to a separating funnel which was then allowed to settle for 2 hrs. The methyl ester was washed three times using distilled water and the layers of oil from methanol and H_2SO_4 were separated. The pre-treated oil was heated up to 100 °C to remove excess water content [34-39].

Transesterification: This is the second step of biodiesel production process. The pretreated CKO was mixed with methanol at a molar ratio of 12:1 and CaO catalyst concentration of 2 wt.% at 60 °C. The reaction then proceeded with stirring at 250 rpm for 2 hrs using a magnetic stirrer. The reaction mixture was transferred to a separating funnel for separation of 2 layers where the upper layer consists of KME while the lower layer consists of glycerol [40].

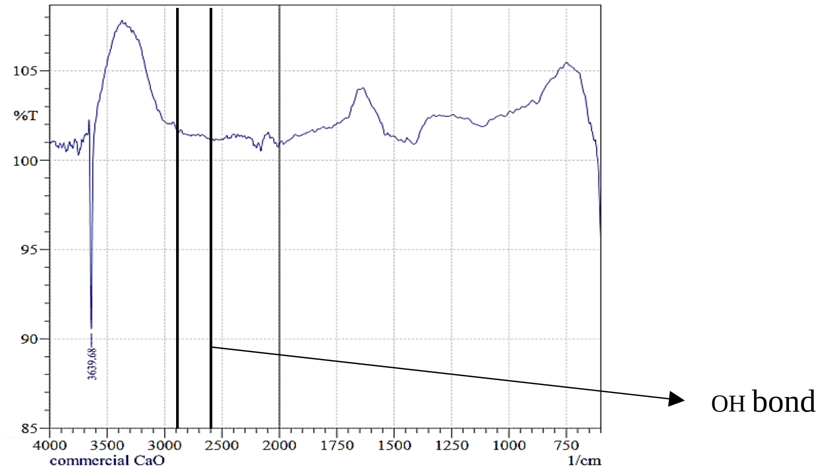
Catalyst Reusability Study

Karanja biodiesel and other mixtures were centrifuged at 3000 rpm for 1 hr to recover *M.Lyrata* shells CaO catalyst and then washed using n-hexane for three times to remove all dissolved impurities. The sample was then dried in an oven for 24 hrs. Then, the recovered catalyst recalcined again before being used for the next transesterification of CKO for 3 hrs at 600 °C.

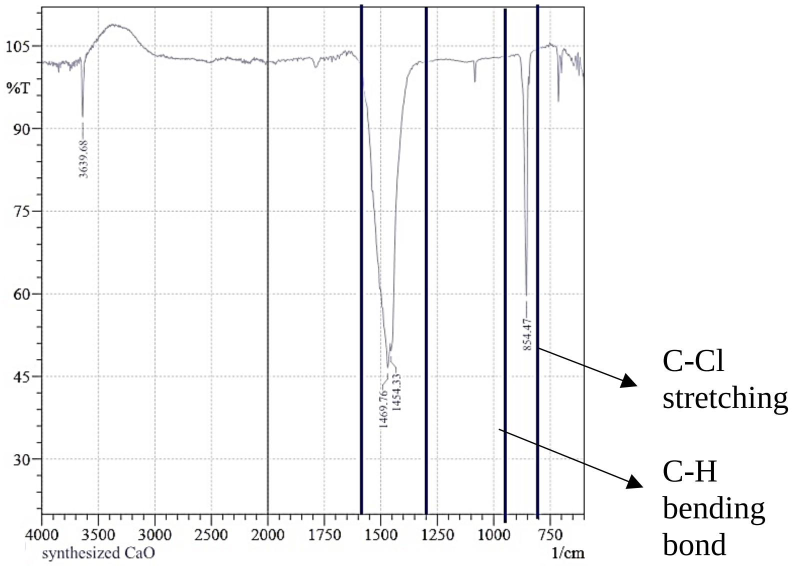
RESULTS AND DISCUSSION

Catalyst Characterisation Analysis

The infrared (IR) spectrum of CaO catalyst as synthesised from *M. Lyrata* shells is graphically shown in Figure 1. It is evident that CaO group absorptions were strongly attributed less than 700 cm^{-1} corresponding to the functional group Ca-O from calcium carbonate. The peak indicates strong as well as broad medium absorptions band signifying the Ca-O which confirms the presence of CaO. The absorption peak over 854.47 cm^{-1} corresponds to C-Cl stretching, while the absorption band over wavelength 1469.76 cm^{-1} and 1454.33 cm^{-1} corresponds to medium C-H bending bond. Furthermore, a weak absorption wavelength of more than 3700 cm^{-1} specifically can be seen at 3639.68 cm^{-1} where the absorption peaks correspond to the hydroxyl group due to the presence of calcium hydroxide.



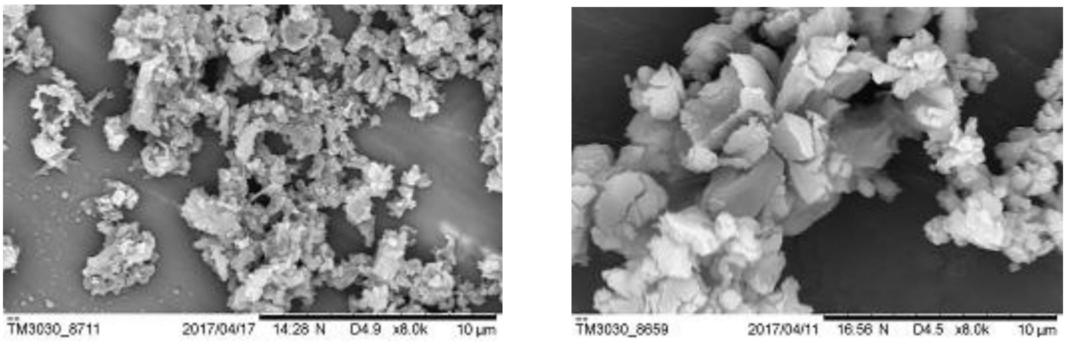
(a)



(b)

Figure 1. FTIR spectrum of (a) commercial CaO and; (b) synthesised CaO from *M. Lyrata*.

The surface morphology of calcium oxide derived from both hard-calm shell and commercial CaO was analysed using scanning electron microscopy (SEM). Referring to Figure 2(a), it can be observed clearly that SEM images of CaO derived from hard calm shell is totally different from commercial CaO. SEM images of calcium oxide synthesised from hard calm shell show a porous structure with smaller particles. According to Buasri et al., smaller sizes of grains and aggregates of *M. Lyrata* CaO could give a higher specific surface area [18, 19]. Conversely, the commercial CaO particles appear in agglomerate forms as shown in Figure 2(b).



(a) (b)

Figure 2. SEM images of (a) synthesised CaO and; (b) commercial catalyst.

Figure 3 shows the particle size distribution of CaO particles from hard clam shells where the particle distribution is in the range of 0.04 to 60 µm. A large fraction of the powder particle falls within a range of 20-40µm. The average particle diameter was 15.67 µm. The highest peak of the bimodal size distribution ranges from 30-60 µm. The BET and BJH analysis of *M. Lyrata* synthesised having a S_{BET} of 36.6 m^2/g yield pore diameters of 13.861 nm and total pore volume of 0.126 cc/g. Further, commercial CaO having a S_{BET} of 5.21 m^2/g results in pore diameters of 11.355nm and total pore volume of 0.0121 cc/g. According to Ren et al., the results S_{BET} of *M.Lyrata* is comparatively high and the pore diameter ranges are categorised within mesopores 2 nm to 50 nm consequently, resulting in high surface area along with their catalytic activity, able to store energy and suitable of adsorption [32, 41].

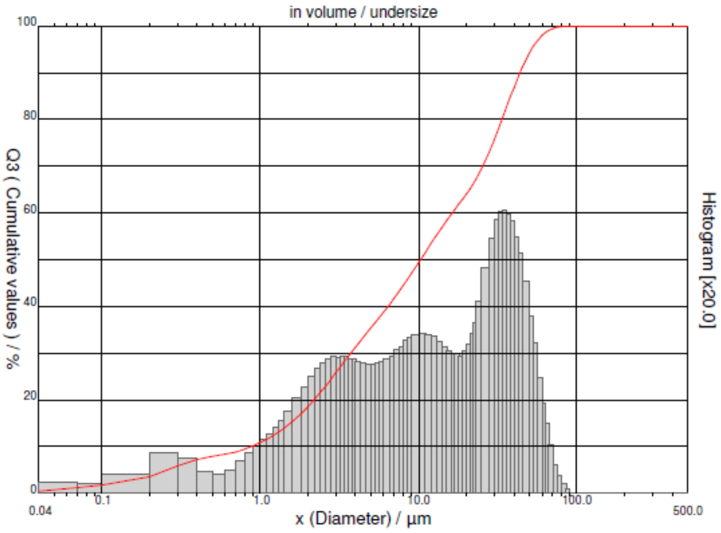


Figure 3. Particle size distribution of CaO synthesised from *M. Lyrata*.

Economic Analysis of CaO Synthesised from *M. Lyrata*

From economical point of view, attentions have been focused on making CaO as a candidate for solid base catalyst. As a major source, CaO has its own ready availability and of low cost. Moreover, CaO can be prepared at laboratory scale thus resulting in economic benefit.

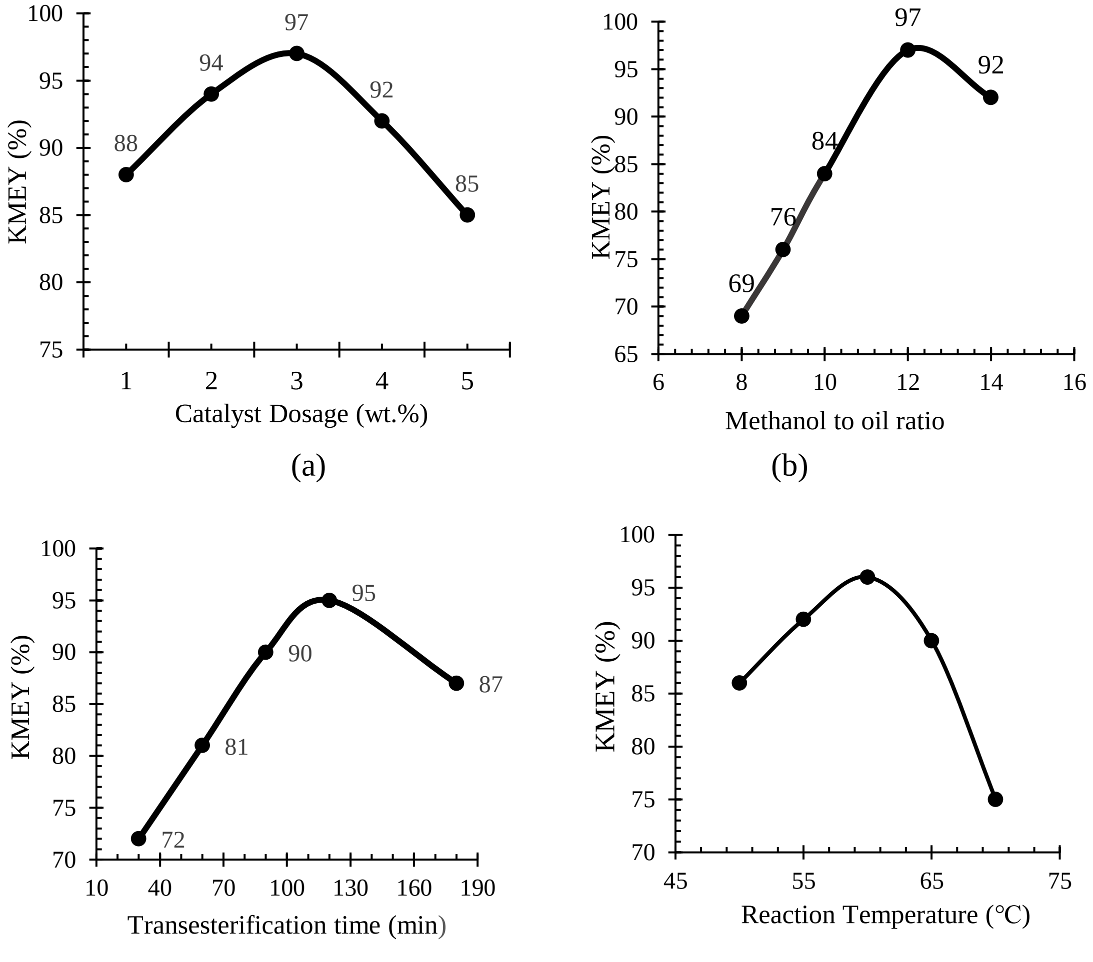
Karanja Biodiesel Production Analysis

In heterogenous transesterification, CKO was mixed with methanol and catalysed by alkaline earth oxide, CaO. The influence of reaction time, reaction temperature, catalyst concentration and methanol ratio on the performance of synthesised CaO under different transesterification conditions were investigated. Based on the results tabulated in Table 1, higher methyl ester yield for transesterification of using synthesised CaO catalyst is achieved at 97.3% in the presence of 2 (wt. %) CaO using methanol-to-oil ratio of 12:1 at 58 ℃ temperature within 2 hrs. Figure 4(a) to 4(d) present the optimal reaction parameters for KME production via a two-step transesterification.

The optimum methanol-to-oil ratio was 12:1 over the catalyst in the present study, which was double the practical methanol-to-oil ratio for homogeneous transesterification of 6:1. This higher amount methanol would cause the development of methoxy specified for the surface of CaO as well as the reaction equilibrium shifting to a forward direction [42]. However, KME yield will not increase when the methanol ratio is over 12. The reason for this is glycerol eventually dissolved in excessive methanol and at the same time constrained the reaction of methanol to the reactants and catalyst. However, further increase in methanol/oil ratio up to 18 did not promote the reaction. It was considered that the glycerol would largely dissolve in excessive methanol and subsequently inhibited the reaction of methanol to the reactants and catalyst [41, 43]. From the results, by comparing with commercial CaO, hard clam showed a higher catalytic activity. Optimum conditions of this catalyst were achieved by giving a FAME conversion of 97.3 % when the methanol-to-oil ratio of 12:1 in the presence of 2 (wt.%) catalyst at 58 °C for 2 hrs of reaction time.

Table 1. Effect on various parameters on production of Karanja methyl ester (KME)

Catalyst loading, CL		Methanol ratio, MR		Transesterification time, TT		Reaction temp., RT	
CD (wt.%)	KMEY (%)	MR (mol.)	KMEY (%)	TT (min)	KMEY (%)	RT (°C)	KMEY (%)
1	78	8	69	60	81	50	86
2	94	10	83	90	90	55	92
3	97	12	97	120	95	60	96
4	90	14	92	180	87	65	90



(a) (b)

(c) (d)

Figure 4. Optimisation of KME yield via two-step transesterification responding to (a) catalyst dosage, (b) methanol to oil ratio, (c) transesterification time and; (d) reaction temperature.

M. Lyrata Synthesised CaO Reusability Analysis

The *M.Lyrata* catalyst reusability was studied by using optimal condition of transesterification at a molar ratio of 12:1 and catalyst concentration of 2 wt.% at 58 °C for 2 hrs. After the reaction was completed, separation of catalyst from the mixture was made and proceeded to be used for a second reaction under the same conditions. From Figure 5, it is observed that the catalyst is active for three reaction cycles with yield above 90 %. It is also found out that after four reaction cycles, the biodiesel yield lowers to 83.7 %. The reason for this is catalyst deterioration occurring due to the change in surface structure causing loss of active sites in the synthesised catalyst. Moisture in the reactant caused the calcium oxide to transform into calcium hydroxide which deteriorated the catalyst activity. However, by recalcining the catalyst at 600 °C the catalyst activity could recover [8, 44]. Table 2 and Table 3 show the analysis and the test methods adopted for the determination of CKO methyl esters properties.

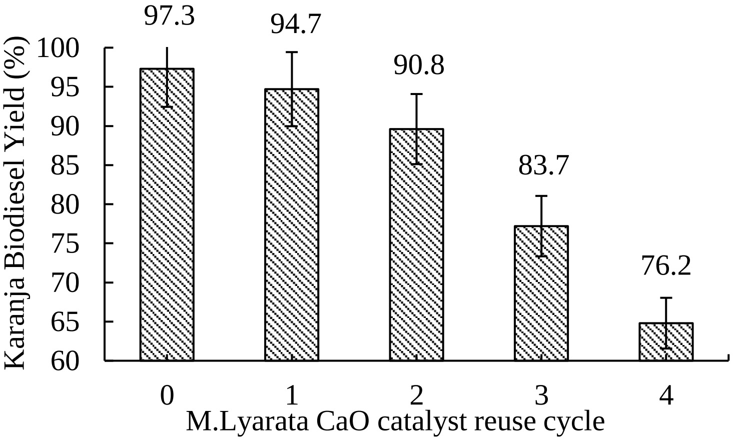


Figure 5. *M.Lyarata* CaO catalyst recycle and reusability experimentation.

Table 2. Properties of KME and test methods.

Property	KME	Limits	EN 14214/ASTM
Density at15 °C (g cm^{-3})	0.883	0.860-0.900	EN ISO 3675
Pour point (°C)	3	NS[a]	ASTM D 97
Water content (%)	0.038	0.05 max	EN ISO 12937
Acid value, Koh, (mg g^{-1})	0.17	0.5 max	EN 14104
Kinematic viscosity at 40 °C (mm^2/s)	4.66 ± 0.02	3.50-5.00	EN ISO 3104
Flash point (°C)	212	120 min	EN22719
Cloud point (°C)	6	NS[a]	EN14214
Free glycerin (wt.%)	0.0064	0.02	ASTM D6584
Total glycerin (wt.%)	0.082	0.240	ASTM D6584
Monoglyceride content (wt.%)	2.63	<0.8	EN4214
Phosphorus (mg/kg)	0.04	<4-10	ASTM D4951
Triglyceride content (wt.%)	0.06	<0.2	EN4214
Diglyceride content (wt.%)	0.78	<0.2	EN4214
Group 1 metals, Na + K (mg/kg)	-	0.5	EN14214

Table 2. Properties of CKO before transesterification and test methods.

Properties	CKO	EN 14214/ASTM
Density at 15 ℃ (g/ml)	0.924	EN ISO 3675
Water content (%)	10.20	EN ISO 12937
Saponification number (mg/g)	186.4	ASTM D5558
Acid number (mg KOH/g oil)	31.24	EN 14104
FFA (%)	15.62	ASTM D5555
Kinematic viscosity at 40 ℃ (mm^2/s)	27.82	EN ISO 3104

CONCLUSION

In this study, CaO catalyst was successfully synthesised from *M.Lyrata* seashells by following a calcination – hydration – dehydration technique. The characterisation of newly synthesised CaO was carried out using FTIR, SEM, PSA, BET and BJH. BET and BJH are spectro-photographic techniques. The characterisation results revealed that the synthesised CaO catalyst had S_{BET} of 36.6 m^2/g, pore diameters of 13.861nm and total pore volume of 0.126 cc/g. Under optimum transesterification reaction conditions of methanol-to-oil ratio of 12:1 in the presence of 2 wt.% catalyst at 58 ℃ for 2 hrs of reaction time, a KME yield of 97.3 % was obtained. It was observed that the catalyst was active for 3 reaction cycles with yield above 90 %. The produced KME fuel properties were tested according to EN 14214 biodiesel standards.

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