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Research Article

Continuous Production of Biodiesel from Rubber Seed Oil Using a Packed Bed Reactor with BaCl₂ Impregnated CaO as Catalyst

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Abstract

The goal of this research was to test barium chloride (BaCl₂) impregnated calcined razor clam shell as a solid catalyst for transesterification of rubber seed oil (RSO) in a packed bed reactor (PBR). The waste razor clam shells were crushed, ground, and calcined at 900 °C in a furnace for 2 h to derive calcium oxide (CaO) particles. Subsequently, the calcined shells were impregnated with BaCl₂ by wet impregnation method and recalcined at 300 °C for 2 h. The synthesized catalyst was characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), energy dispersive spectrometer (EDS), Brunauer-Emmett-Teller (BET) surface area, and basic strength measurements. The effects of various parameters such as residence time, reaction temperature, methanol/oil molar ratio, and catalyst bed length on the yield of fatty acid methyl ester (FAME) were determined. The BaCl₂/CaO catalyst exhibited much higher catalytic activity and stability than CaO catalyst influenced by the basicity of the doped catalyst. The maximum fatty acid methyl ester yield was 98.7 % under optimum conditions (residence time 2.0 h, reaction temperature 60 °C, methanol/oil molar ratio 12:1, and catalyst bed length 200 mm). After 6 consecutive reactions without any treatment, fatty acid methyl ester yield reduced to 83.1 %. The option of using waste razor clam shell for the production of transesterification catalysts could have economic benefits to the aquaculture and food industries. Copyright © 2018 BCREC Group. All rights reserved.

Keywords: BaCl₂ Impregnated CaO; Biodiesel; Rubber Seed Oil; Transesterification; Waste Razor Clam Shell

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1. Introduction

The increasing energy demand, depleting fossil fuels and environmental problem resulted

from climate change by continues consumption of petroleum derived fuel, make challenges for the scientific community and the researchers worldwide today [1]. Among the options explored for alternative energy sources, biodiesel is one of the attractive alternatives due to its renewability, biodegradability, and non-toxicity

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[2]. It is usually produced via esterification or transesterification using acidic or basic catalysts. The most common process is to facilitate a reaction between triglycerides (TGs) and alcohol in the presence of a homogeneous catalyst [3]. However, existing biodiesel processes suffer from some serious problems with the use of homogeneous catalysts, such as equipment corrosion, waste effluent treatment, soap formation and catalyst removal, leading to severe economic and environmental penalties. Therefore, exploring heterogeneous catalysts is becoming more important in chemical and life science industry [4].

Among the heterogeneous catalysts, calcium oxide (CaO) is a potential candidate for its low cost, high basic strength and catalytic activity, low methanol solubility, and green material. Calcium nitrate (Ca(NO₃)₂), calcium carbonate (CaCO₃), β -tricalcium phosphate (β -Ca₃(PO₄)₂), and calcium hydroxide (Ca(OH)₂) are raw materials to produce CaO. Generally, CaO could be derived from CaCO₃, which is the major constituent in many natural sources and wastes [5]. Catalysts derived from waste shells were proposed for biodiesel production and it was concluded that these catalysts were environment friendly as they are mainly prepared from natural materials [6]. Moreover, different kinds of CaO catalyst, such as gel-combusted CaO catalyst, CaO extracted from biowaste and supported CaO catalyst, had been investigated aiming at improving the catalytic activity [7].

Doping of metal oxides increases basic strength. Barium ions (Ba²⁺) being highly basic, when added to the calcium ions (Ca^{2+}), become a competitive base catalyst with high conversions of TGs to biodiesel at lower reaction conditions [6,8]. Based on these findings, we hypothesize that impregnating calcined razor clam shell surfaces with barium chloride (BaCl₂) will enhance the catalytic activity of waste shells towards transesterification due to synergistic effects of a basic element deposited on a naturally basic solid support. Studies in which calcined razor clam shells have also been reported but to the best of our knowledge investigations have not been carried out to enhance their catalytic activity by impregnating it with Ba²⁺.

Generally, there are four major types of feedstock available for the biodiesel production including oil seed (vegetable oil), animal fats, algae and different low-quality material such as waste cooking oil (WCO), greases and soap stock [9]. Non-edible plant oils have been found to be promising crude oils for the production of biodiesel. The use of non-edible oils when com-

pared with edible oils is very significant in developing countries because of the tremendous demand for edible oils as food, and they are far too expensive to be used as fuel at present [10]. Rubber seeds are the important by-product of the rubber trees and treated as a waste. These seeds contain 40-50 % oil and still underutilized in Thailand. Thus, oil extracted from the rubber seeds can be used as a prominent feedstock for the production of biodiesel in developing countries, where almost 85 % of the crude oil imports from the other countries [11]. In the recent years, there are several works reported on biodiesel production using rubber seed oil (RSO) which has higher potential to be used as alternative diesel fuel because it is a non-edible oil that can produce sufficient amount of oil for the industry [12]. Hence, in the present study, RSO is used as a feedstock for the synthesis of biodiesel

Batch reactors are widely employed in the early research of biodiesel production. Compared with batch reactors, continuous reactors can reduce production cost, provide a uniform quality of the final product and are more conducive to large-scale industrial production. Among all reactors, packed bed reactors (PBR) are the most commonly employed reactors for biodiesel industrial production [13]. There are lots of studies focused on using a PBR to improve the yields of biodiesel from vegetable oil. However, the RSO-derived biodiesel production using a PBR and novel catalyst BaCl₂/CaO derived from waste razor clam shell has seldom been addressed. The present research aims to exploit BaCl₂ impregnated CaO as a packed bed catalyst for the continuous production of biodiesel from RSO. The effects of residence time, reaction temperature, methanol/oil molar ratio, and catalyst bed length are systematically studied. Moreover, the reusability of the catalysts derived from the natural calcium materials was investigated.

2. Materials and Methods

2.1 Materials

The razor clam shell was collected as waste from Tae-lea Thai market, Phetchaburi Province, Thailand. The waste shell was rinsed with running water (H₂O) to eliminate dust and impurities, and was then dried in an oven at 60 °C for 24 h. BaCl₂ (AR grade) and all necessary solvents were procured from Ajax Finechem Pty Limited, Australia and were used as received without further purification. The rubber seed was collected from the Northeastern provinces of Thailand as an oil source for biodiesel production. The RSO was extracted using solvent extraction technique. It extracted with hexane is a clear yellow liquid. The molecular weight and density of the yellow oil were measured to be 875 g/mole and 0.887 g/cm³, respectively.

2.2 Preparation of catalyst

The waste razor clam shell was repeatedly washed to remove any organic impurities attached to it and then dried in an oven. The dried shell was crushed and sieved to pass 60-200 mesh screens (75-250 mm). The waste shell was calcined at a high temperature of 900 °C for 2 h in an air atmosphere with a heating rate of 10 °C/min similar to the procedure described by Buasri et al. [5]. The calcined sample was obtained as white powder. Subsequently the CaO was impregnated with 2 M of BaCl₂ as described by Mahesh et al. [8]. BaCl₂/CaO catalyst was prepared by the wet impregnation method, and was then dried in the vacuum oven at 60 °C for 24 h and recalcined at 300 °C for 2 h. The product was kept in the closed vessel to avoid the reaction with carbon dioxide (CO_2) and humidity in the air before used.

2.3 Characterization of catalyst

X-ray diffraction (XRD) patterns of the samples were recorded on a LabX XRD-6100 analyzer (Shimadzu, Japan) operating at 30 kV and 20 mA with a Cu anode and a graphite monochromator (l = 1.5405 Å), an angle of scanning range 10-70° (20), a scan step size of 0.04° and a scan rate of 4°/min. The XRD phases were identified using the Powder Diffraction File (PDF) database created by International Centre for Diffraction Data (ICDD). The sample morphology and elemental chemical analysis were also characterized at room temperature by a Hitachi TM3030 (USA) scanning electron microscopy (SEM) system equipped with an energy dispersive spectroscopy (EDS) detector. The material was coated with gold (Au) using a Sputter Coater for protecting the induction of the electric current. The accelerating voltage was 15 kV and working distance was 14 -15 mm. The surface area and pore distribution of the synthesized catalysts were determined by the Brunauer-Emmett-Teller (BET) method using a nitrogen (N₂) adsorption/desorption analyzer (Autosorb-1 Model No. ASIMP.VP4, USA). Prior to the analysis, the catalysts were degassed at 300 °C for 4 h to remove moisture and foreign gases on the surface. Adsorption and desorption process of N_2 on the catalyst surfaces were examined in a vacuum chamber at -196 °C. The basic strength of the synthesized catalysts was characterized using a Hammett indicators method as described by Boro *et al.* [6].

2.4 Continuous production of biodiesel

Continuous transesterification was performed in a PBR at an atmospheric pressure similar to the procedure described by Buasri and Loryuenyong [14] and Gui et al. [4]. The reactor was composed of a water-jacketed stainless steel column with an external diameter of 60 mm, an internal diameter of 40 mm, and a length of 345 mm. The column was packed with BaCl₂/CaO catalyst. The scheme of the continuous production of biodiesel from RSO and methanol is shown in Figure 1. RSO and methanol were charged into the system using a plunger pump. The reactants were mixed and preheated in a mixing column with random packing. The reaction temperature was controlled by a heater to keep a constant temperature of the reactor wall with an error of ±1.0 °C. The temperature difference between the inlet and the outlet was below 1.0 °C during all of the runs. Temperature and pressure of the system were monitored by a temperature indicator and pressure gauges. The effects of residence time (0.5 to 2.5 h), reaction temperature (50 to 70 °C), methanol/oil molar ratio (6:1 to 18:1), catalyst bed length (100 to 300 mm), and repeated operation of PBR (1-8 cycles) on the % yield of fatty acid methyl ester (FAME) were investigated. All experiments were repeated 3 times and the standard deviation was never higher than 7 % for any point.

The content of FAME in biodiesel samples was analyzed by the gas chromatography mass spectrometry (GC-MS, QP2010 Plus, Shimadzu Corporation, Japan) equipped with a flame ionization detector and a capillary GC column (DB-WAX, Carbowax 20M, 30 m × 0.32 mm × 0.25 µm) using inner standard method as described by Buasri *et al.* [15]. The yield of biodiesel was calculated by dividing the weight of biodiesel with the weight of oil and multiplying the resulting number by 100 (Equation (1)).

$$Yield (\%) = \frac{weight \ of \ biodiesel}{weight \ of \ raw \ oil} x100\%$$
(1)

The physical and chemical properties of FAME were analyzed according to ASTM and EN methods such as kinematic viscosity, density, flash point, cloud point, pour point, acid value, moisture content, ester content, free glycerine, total glycerine, iodine number, Bulletin of Chemical Reaction Engineering & Catalysis, 13 (2), 2018, 323

cetane number, and calorific value of the biodiesel. The obtained values were then compared with the United States biodiesel standard (ASTM D-6751) and European biodiesel standard (EN 14214).

3. Results and Discussion

3.1 Catalyst characterization

XRD patterns of natural razor clam shell, calcined shell and synthesized catalyst are given in Figure 2. The waste shell mainly consisted of CaCO3 phase, and after calcined at 900 °C for 2 h, this CaCO₃ was completely converted to CaO by evolving the CO₂. The formation of the sharp peak indicates the formation of highly crystalline materials [16]. The catalyst shows the presence of CaO, Ca(OH)₂, and BaCl₂ with prominent 2θ values at 18.08° , 29.36°, 34.06° and 47.12° is also seen. Doping with barium (Ba) brings some shifting in the XRD pattern of the parent CaO. The nonvisibility of the peaks in the doped samples might have arisen due to the fact that the d value of diffraction peaks of tetragonal phase barium oxide (BaO) is very close to the cubic phase CaO. It might be that the diffraction peaks of both the phases overlapped almost completely [6]. The XRD analyzes confirmed the presence of BaCl₂ on the shell surface.

The morphology of natural razor clam shell, calcined shell and the synthesized catalyst was detected by SEM (4000x magnification) and results were showed in Figure 3. From the figure, we can see that the morphology of natural shell displayed a typical layered architecture with macro-pores and irregular shapes of particles of various sizes. When the calcined temperature was 900 °C, the particle shapes become

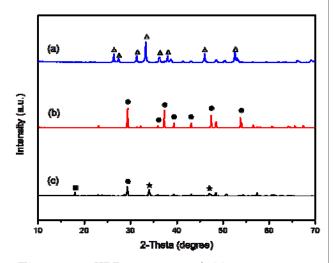


Figure 2. XRD patterns of (a) waste razor clam shell (b) calcined shell and (c) novel catalyst (Δ : CaCO₃, • : CaO, • : Ca(OH)₂, and ★ : BaCl₂)

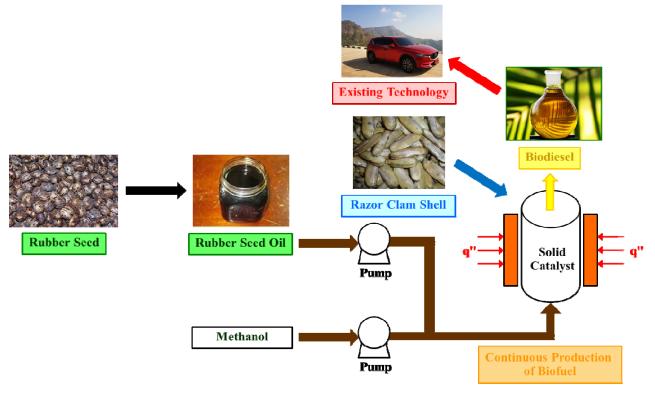
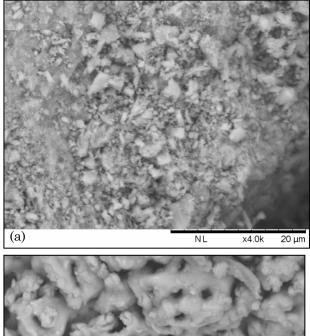


Figure 1. Scheme of the continuous production of biodiesel from RSO and methanol with a PBR

more regular. The SEM images of CaO showed a cluster of well-developed cubic crystal with obvious edges [17]. It was observed that the particles of the catalyst have a spherical structure and are agglomerated into lumps. This is because the sample is in the oxide form. The isolated particles were reasonably isotropic [2,8].

Elemental analysis of the synthesized catalyst was characterized by EDS which is well known analytical technique to determine the chemical or elemental characterization of a material [18]. Figure 4 represents the EDS of BaCl₂/CaO derived from waste razor clam shell. Here, the positions of specific elements emitting characteristic X-rays within an inspection field can be indicated by unique color. The weight percent (norm. wt.%) of calcium (Ca), oxygen (O), barium (Ba), and chlorine (Cl) on a particular area at the surface of catalyst were determined to be 78.08 %, 18.04 %, 1.16 % and 2.72 %, respectively. The weight ratio between



(b)

barium and calcium (Ba:Ca) was 1.49, while the weight ratio between chlorine and calcium (Cl:Ca) was 3.48, indicating that $BaCl_2$ was well loaded onto CaO. Furthermore, the surface of the catalyst has strong basic character and therefore holds tendency to absorb moisture. H₂O adsorbed within the pores on the basic catalyst surface has an influence on O:Ca. Hydrogen was not quantified by EDS because of small orbital diameter (atomic number = 1), which reduces catch probability for spectroscopy to almost zero [5].

The BET specific surface area (S_{BET}), average pore diameter (D_p) and total pore volume (V_p) of the calcined shell and synthesized catalyst are summarized in Table 1. It indicated that the CaO catalyst (calcined shell) possessed a surface area of 18.78 m²/g, a pore diameter of 9.77 nm and a pore volume of 0.053 cm³/g. The results of BaCl₂/CaO catalyst showed gradually reduced of surface area and pore volume upon increasing the Ba species concentration. The significant reduction in the S_{BET} and V_p for synthesized catalyst was due to the excess of active metals embedded into the channel of parent materials and incorporated into the pore of CaO [1]. The average pore size of the novel catalyst was 1.16 time bigger than that of the CaO catalyst, suggesting that the particles of the calcined and treated razor clam shell were packed much closer.

Hammett indicator method was used to determine the basic strength of the catalyst according to the literature [6]. The basicity and basic strength are the most important properties regarding their application as a synthesized catalyst for the transesterification of

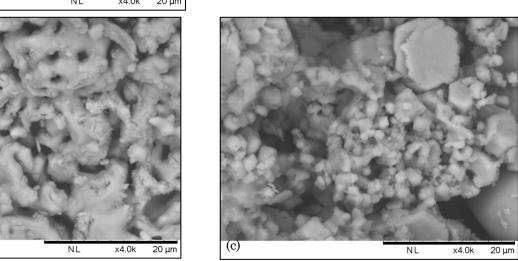


Figure 3. SEM micrographs of (a) waste razor clam shell (b) calcined shell and (c) novel catalyst. Images are magnified at 4000X, and scale bars represent 20 mm

RSO. The reaction activity depends on the number of basic sites present in the catalysts as well as on their strength [5]. The increasing Hammett indicator ($15.0 < H_{-} < 18.4$) and basic strength (0.238 mmol/g) for doped catalyst might have resulted due to the presence of BaO and CaO active species (Table 2).

3.2 Effects of transesterification process variables

The catalytic activities of the packed bed BaCl₂/CaO catalysts were studied by investigating the effects of residence time, reaction temperature, methanol/oil molar ratio, catalyst bed length, and reusability of catalyst in the transesterification. For the following experiments, the calcined and treated razor clam shell was used as a novel catalyst to catalyze the reaction of RSO and methanol in PBR.

The effect of residence time on the conversion of RSO to FAME was investigated. Figure 5 shows a gradual increase in the yield of FAME with time from 0.5 h to 2.5 h with a catalyst bed length of 200 mm and a methanol/ oil molar ratio of 12:1. The FAME yield of 98.7 % was obtained in 2.0 h at 60 °C for BaCl₂/CaO catalyst. Production of biodiesel increases with residence time until it reaches equilibrium at a residence time of 2.0 h, due to the initial transient of the reactor from the start-up to the stationary conditions [14]. It has also been estimated that for the operation beyond 2.0 h, the yield increases slightly. This is because longer residence time results in hydrolysis of esters (side reaction) and a reversible reaction between RSO and methanol [19]. In this study, the optimum residence time appears to be 2.0 h.

In order to determine the optimum reaction temperature, transesterification was carried out by varying the reaction temperature from 50 to 70 °C in a step increment of 5 °C (Figure 6). The low conversion was obtained when the reaction was carried out at low temperature of 50-55 °C which is attributed to the mass transfer and solubility limitations. At higher reaction temperature (60 °C) it is expected that the reactants may gain sufficient kinetic energy that will accelerate the mass transfer rate among the RSO-methanol catalyst phases resulting in maximum conversion. However, the

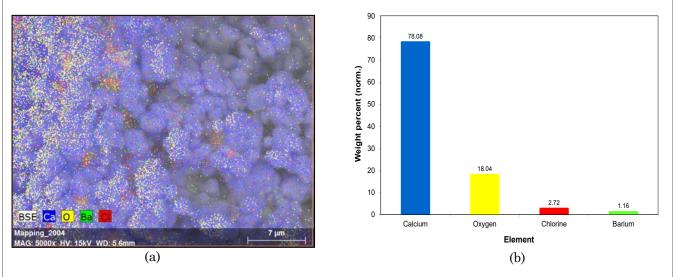


Figure 4. EDS analysis of novel catalyst: (a) chemical mapping for Ca, O, Ba, Cl, and (b) weight percent (norm. wt.%) of elements on surface

Table	1.	S_{BET} ,	D_p ,	and	V_p	of	calcined	shell	and
novel c	ata	ılyst							

Table 2. Hammett indicator and basic strength
of calcined shell and novel catalyst

Natural Material- Derived Catalyst	$S_{BET} \ ({ m m^2/g})$	D _p (nm)	$V_p \ (m cm^3/g)$	Natural Material- Derived Catalyst	Hammett Indicator (H_)	Basic Strength (mmol/g)
CaO (Calcined Shell)	18.78	9.77	0.053	CaO (Calcined Shell)	$7.2 < H_{-} < 9.8$	0.128
BaCl ₂ /CaO (Novel Catalyst)	9.26	11.29	0.034	BaCl ₂ /CaO (Novel Catalyst)	$15.0 < H_{<} 18.4$	0.238

slight loss in activity is observed when the reaction temperature is above 60 °C. This is probably due to the fact that methanol has a boiling point near to 65 °C and reaction above this temperature might cause the loss of methanol leading to low catalytic activity [6]. In addition, the catalytic activity of BaCl₂/CaO decreases due to some qualitative changes, such as loss of Ba²⁺ and Ca²⁺. Moreover, in order to save energy, it is necessary to choose the relative low temperature [20]. Therefore, the optimum reaction temperature for the transesterification of RSO to FAME is considered to be around 60 °C.

The transesterification with methanol/oil molar ratio of 6:1 to 12:1 (Figure 7) indicates that the conversion of RSO to FAME increases rapidly with the mole ratio, reaching 98.7 % at the mole ratio of 12:1 catalyzed by BaCl₂/CaO. Further increasing mole ratio to 18:1, the conversion increases gradually to 99.2 %. In addition, stable emulsions may form at the higher content of methanol (18:1), leading to complicated separation and purification of biodiesel [4]. At the end of transesterification, the excess methanol was recovered by a vacuum distillatory and recycled in latter reactions.

The catalyst bed length in the PBR is associated with the residence time during continuous production of biodiesel. The RSO conversion to FAME increased rapidly from 65.4 to 81.2 % with the increase of the catalyst bed length from 100 to 150 mm (Figure 8). In particular, the yield of FAME was over 98 % when the catalyst bed length was over 150 mm. This demonstrates that a high catalyst bed length provides a lower flow rate and more active sites to promote the reaction between RSO and

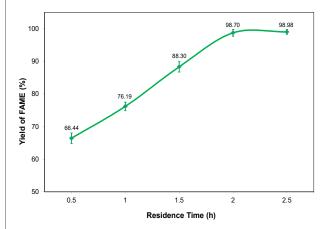


Figure 5. Effect of residence time on the %yield of FAME. Reaction conditions: reaction temperature 60 °C; methanol/oil molar ratio 12:1; and catalyst bed length 200 mm

methanol at a given residence time [14,21]. All results presented hereafter were measured using a 250 mm bed length.

The divalent metal oxide catalysts having a substantial amount of covalent character facilitates the transesterification reaction [22] as depicted in Figure 9. In general, the catalytic properties are influenced by particle size, surface area, as well as the active sites of a catalyst. Small sized catalyst with high surface area and active sites will give rise to high catalytic performance [23]. In this study, Ba has been used to increase the basicity of the CaO derived from the waste razor clam shell. The increase in the yield of FAME with Ba loading is attributed to the presence CaO and BaO which might have acted like active species during the reaction [6].

3.3 Catalyst reusability

One of the main disadvantages of homogeneous catalysts is that they cannot be recovered. So, in the present study, the catalyst was evaluated to study its efficiency and reusability. The catalyst was recycled to test its activity as well as stability. Reusability of the BaCl₂/CaO catalyst in the transesterification of RSO was checked without any further purification and activation. The solid catalyst was collected after adding fresh reactants. The catalytic activity remained 83.1 % of the fresh catalyst when the novel catalyst was employed for the 6 consecutive runs (Figure 10). It was calculated based on the conversion of RSO to FAME under the optimum condition (residence time 2.0 h; reaction temperature 60 °C; methanol/oil molar ratio of 12:1; and catalyst bed length 200 mm) in the PBR. The decay in cata-

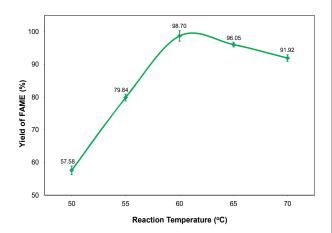


Figure 6. Effect of reaction temperature on the %yield of FAME. Reaction conditions: residence time 2.0 h; methanol/oil molar ratio 12:1; and catalyst bed length 200 mm

lyst activity could be due to the leaching of active sites to the reaction media. Notwithstanding the interaction between the Ba species and CaO surface, the loaded species are leached by the reaction media. Leaching of the active phase to the alcoholic phase can be attributed to the bond breaking and formation of Ca²⁺ and CH₃O⁻ [24,25].

3.4 Fuel properties of biodiesel

For biodiesel to be used in diesel engines, the fuel must meet various specifications stated in biodiesel standard, mainly United States

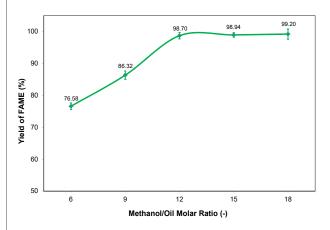


Figure 7. Effect of methanol/oil molar ratio on the %yield of FAME. Reaction conditions: residence time 2.0 h; reaction temperature 60 °C; and catalyst bed length 200 mm

biodiesel standard (ASTM D-6751) and European biodiesel standard (EN 14214) [5]. The fuel properties (kinematic viscosity, density, flash point, cloud point, pour point, acid value, moisture content, ester content, free glycerine, total glycerine, iodine number, cetane number and calorific value) of FAME obtained in this work are summarized in Table 3 along with a comparison to the recommended biodiesel international standards ASTM D-6751 and EN 14214. It can be seen that most of its properties are in the range of fuel properties as described in the latest standards for biodiesel.

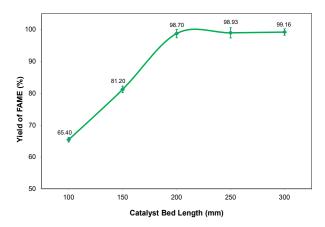
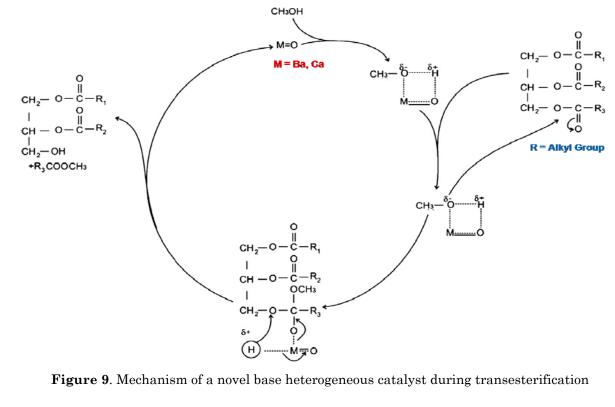


Figure 8. Effect of catalyst bed length on the %yield of FAME. Reaction conditions: residence time 2.0 h; reaction temperature 60 °C; and methanol/oil molar ratio of 12:1



4. Conclusions

The present study demonstrates the successful application of calcined and treated waste razor clam shell as the efficient heterogeneous catalysts for transesterification of RSO with methanol in PBR. The highest FAME yield of 98.7 % for BaCl₂/CaO catalyst was obtained under the optimum condition (residence time 2.0 h; reaction temperature 60 °C; methanol/oil molar ratio of 12:1; and catalyst bed length 200 mm). The reusability studies of novel catalyst showed continuous drop in catalytic activity which was attributed to the loss of active sites and deposition of product on the catalyst itself. The RSO conversions maintained higher than 80 % in 6 consecutive runs. The experimental results showed that novel

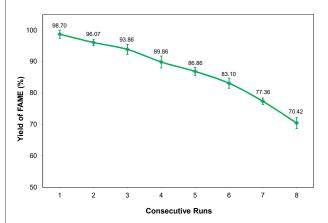


Figure 10. Effect of reusability of catalyst on the %yield of FAME. Reaction conditions: residence time 2.0 h; reaction temperature 60 °C; methanol/oil molar ratio of 12:1; and catalyst bed length 200 mm

catalysts had excellent catalytic activity, stability, and reusability during the reaction. Moreover, this new continuous biodiesel production process and apparatus have good potential for utilization of RSO with an inexpensive and easily available non-edible oil.

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Table 3. The fuel properties of biodiesel obtained in the transesterification of RSO

Fuel Property	Biodiesel in This Work	ASTM D-6751	EN 14214
Kinematic Viscosity (mm ² /s) @ 40 °C	4.8	1.9-6.0	3.50 - 5.00
Density (kg/m ³) @ 15 °C	890	860-894	860-900
Flash Point (°C)	160	>130	>120
Cloud Point (°C)	12	-3 to 12	Not Specified
Pour Point (°C)	9	-15 to 10	Not Specified
Acid Value (mg KOH/g oil)	0.42	≤ 0.5	< 0.5
Moisture Content (%)	0.02	< 0.05	< 0.05
Ester Content (%)	98.70	>96.5	>96.5
Free Glycerine (%)	0.009	< 0.02	< 0.02
Total Glycerine (%)	0.13	< 0.24	< 0.24
Iodine Number (g I ₂ /100 g oil)	115	Not Specified	<120
Cetane Number	56	>47	>51
Calorific Value (MJ/kg)	40.85	Not Specified	Not Specified

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