



## Biodiesel production over a catalyst prepared from biomass-derived waste date pits

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### ABSTRACT

Date palms are predominately produced in arid regions and the date pits, or seeds, produced from them are sometimes considered to be a waste. Date pits, ground to powder following an oil extraction, were used to synthesize a renewable heterogeneous catalyst. The green carbon catalyst was modified by an alkaline earth metal oxide (CaO). The oil extracted from date pits was transformed into biodiesel. The biodiesel process was optimized and the optimal yield was 98.2 wt% at a reaction temperature of 70 °C, reaction time ~120 min, methanol to oil molar ratio of 12 and catalyst loading of 4.5 wt%. The quality of the produced biodiesel meets the standard limits set by regulating agencies (ASTM, EU) which indicates its suitability to be used as a fuel. Thus, it can be concluded that the green carbon catalyst synthesized from waste date pits has a high potential for biodiesel production.

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### 1. Introduction

Currently, fossil fuels are the major contributor fulfilling the total world energy demand; and they remain the main source of the global energy supply scenario [1–3]. Due to the industrial revolution in the last century, which increased economic activity, energy demand and consumption [4], fossil-based carbonaceous fuel was a major source for energy production and transportation purposes [5]. However, the demand for fossil-based fuel increased drastically owing to a rapid increase in industrialization and human population. This rapid increase in oil consumption increased attention towards the availability and environmental issues due to the burning of fossil-based fuels. Hence, renewable and sustainable resources such as wind, biomass, and solar energy can decrease some concerns related to fossil fuels [6]. Moreover, among all alternative energy resources mentioned, biomass is the most suitable alternative based on the fact that it is an enriched

source of carbon besides fossil-based resources, which leads to its uniqueness.

Utilization of biomass as an energy source is one medium to reduce concerns related to fossil-based fuels usage and can also help to reduce the energy crises faced worldwide. Biomass has been used from ancient ages on a small-scale for domestic usage based on its positive environmental effects and high energy density and its applications for biofuels, chemicals, and materials [7]. This requires special focus from different sectors across the world such as industrial, academic, national laboratories, non-profit organization and governing authorities so as to increase the awareness regarding the usefulness of biomass in different aspects.

Date palm is considered the main crop in Oman and occupies 54% of total agricultural land in the country [8,9]. Oman has great potential to supply and utilize waste from dates as biomass for biofuel production. This source of biomass has excellent potential as an alternative energy resource as it avoids the use of the edible parts of the plant and does not compete with the food supplies for biofuel production. The by-product of date fruit processing is the date pits which are largely available and cheap. They can, therefore, be used as a source for producing biofuel. The residues of date palm tree and spoiled dates comprise economic and environmental burden to be managed. Interestingly, date pits contain volatile

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compounds that make up 15–20 % of the material and can serve as a potential source for biofuel production.

The use of heterogeneous catalysts for the production of biodiesel is an important aspect to be taken into account for the economics of the commercial production of biodiesel [10–12]. Heterogeneous catalyst use for the production of biodiesel may yield a high-quality biodiesel and simplify downstream separation steps associated with glycerol which is obtained as by-product and eliminate the need for an aqueous quench [13–15]. Heterogeneous catalysts are easily separable from the reaction mixture and can be reused which makes the biodiesel process more economical [16,17]. The template material is of high importance for synthesizing heterogeneous catalysts and can affect production cost of associated with the process [18]. A number of supports have been studied such as  $\text{Al}_2\text{O}_3$ , CaO, MgO etc. due their high porosity and stability [19–21]. However, biodiesel production can be more eco-friendly and economical if the green catalyst is utilized. Catalysts synthesized from biomass can be suitable alternatives as compared to conventional catalysts for producing economical and eco-friendly biodiesel. Generally, the biomass-derived carbon material is known to be porous in nature and possess a high surface area after small modification and can successfully be used for the transesterification of plant-based oils [22]. Baroutian et al. reported efficient biodiesel production in the presence of activated carbon catalysts from biomass modified by KOH [10]. Dhawane et al. used Flamboyant pods to synthesize the green carbon catalysts and observed it was a highly active catalyst after alkali modification for the transesterification of *Hevea brasiliensis* oil [23].

Herein we report the use of date pits powder following oil extraction as a source of a carbon catalyst which was modified by an alkaline metal oxide (CaO) and to produce biodiesel. The synthesized carbon catalyst was characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), Brunauer-Emmett-Teller analysis (BET) and X-Ray Photoelectron Spectroscopy (XPS). Date pits oil is used as source oil for transesterification in the presence synthesized catalysts for producing biodiesel. Complete characterization of the produced biodiesel was carried out to prove its feasibility as fuel.

## 2. Experimental

### 2.1. Materials

Waste date pits were collected from an industrial firm in the city of Muscat in Oman. All the chemicals and reagents were of analytical grade and were purchased from Merck, Germany. Oil was extracted from date pits using a Soxhlet extractor in a 16.5 wt% yield. Hexane was used as a solvent for extraction and process continued for 7 h followed by solvent separation using a rotary evaporator (Buchi, Switzerland) [24]. The fatty acid profile of the date pit oil was determined using a Perkin Elmer Clarus 600 gas chromatograph mass spectrometer (GC-MS) equipped with a DB-Wax column.

### 2.2. Catalysts synthesis

The date pit powder remaining after extraction was washed thoroughly with distilled water to remove impurities and chemicals and dried at ambient temperature for 5 h followed by oven drying at 110 °C for 12 h to remove the moisture content. Subsequently, a carbonization process was carried out in a muffle furnace under a nitrogen atmosphere at 500 °C for 4 h. After cooling the carbon material at room temperature, it was impregnated with an aqueous solution of calcium nitrate tetrahydrate at different concentrations of Ca (2 wt%, 4 wt%, and

6 wt%) on the carbon material. The resultant material was calcined in a furnace at 450 °C for 4 h with a ramping rate of 3 °C/min. The resultant catalysts were termed as C1 (carbon with 2 wt% CaO), C2 (carbon with 4 wt% CaO), C3 (carbon with 6 wt% CaO) and C for pristine carbon respectively.

### 2.3. Catalysts characterization

X-ray powder diffraction (XRD) patterns were measured using a PANalytical, Xpert PRO instrument, USA equipped with rotating anode and  $\text{CuK}\alpha$  radiations. The measurements were conducted in continuous  $\theta/2\theta$  scan refraction mode. A JEOL JSM-7800F instrument, Japan, scanning electron microscope (SEM) attached with energy dispersive X-ray spectroscopy (EDS, Oxford instrument, UK) was used to determine the surface morphology and surface elemental composition. The BET surface area, pore size and pore volume of samples were measured using a Micromeritics Instrument Inc. ASAP 2020 (Norcross, GA, USA) using  $\text{N}_2$  gas (99.995% pure). X-ray photoelectron spectroscopy (XPS, Omicron Nanotechnology, Germany) was utilized to determine the chemical composition of the sample surface. Al  $\text{K}\alpha$  with the energy of 1486.6 eV was used as the monochromatic X-ray. A pass energy of 50 eV was used for the survey scan and 20 eV for the high-resolution scan. All of the obtained binding energies were calibrated with respect to the intrinsic carbon peak which has a binding energy of 284.6 eV.

### 2.4. Biodiesel production and quality analysis

The biodiesel was produced through a transesterification reaction carried out in two-neck flat-bottom flask. In one neck, cooling water line was attached to use as a condenser; in the second neck, a thermocouple was attached through which heating was controlled using a programmed hot plate on which the reaction flask was placed. The transesterification was carried out on oil extracted from date pits in methanol solvent catalyzed by the synthesized heterogeneous catalyst. The process parameters, which include reaction temperature (55–75 °C), methanol to oil molar ratio (6–18), time (30–150 min) and catalyst amount (1.5–7.5 wt%) were varied to optimize the biodiesel yield. The yield of biodiesel was determined using Eq. (1)

$$\text{Yield}(\%) = \frac{\text{weight of biodiesel}}{\text{weight of oil}} \quad (1)$$

The pits oil was heated, and then a calculated amount of methanol and catalyst was added to the heated oil. Prior to the addition of a catalyst in the reaction, the mixture was stirred with methanol for 20 min, and then the reaction mixture was stirred at 600 rpm and ran at the specified conditions. Once the reaction was completed, it was allowed to cool down to room temperature (~25 °C). The water condenser was removed from flask along with the thermocouple controlling temperature and the mixture was centrifuging at 5000 rpm for 30 min to remove the solid catalyst. The reaction mixture, after catalyst separation, was poured into a separating funnel and the mixture was allowed to settle for 8 h. It was observed that two liquid phases formed whereby the bottom layer was glycerol and the upper layer was biodiesel. The bottom layer of glycerol was removed and the upper layer was placed in a funnel and washed with warm deionized water to remove any kind of impurities from biodiesel. Then rotary evaporation was used to remove methanol and water left was biodiesel. To remove any residual moisture from the biodiesel, anhydrous sodium sulfate was added and subsequently filtered.

The quality of biodiesel was analyzed by measuring its properties. Density was measured using an Anton Paar instrument (DMA 4500M, USA) according to ASTM D-4052 and a standard

method and the viscosity by Cannon Ubbelohde viscometer along with test instrument (Tamson TVB445, ISL, France) at 40 °C by ASTM D445. Low-temperature properties such as cloud point and pour point were measured by using the CPP 5G's analyzer by ASTM D 2500 and ASTM D97 methods. Cold filter plugging point was measured using FPP 5Gs analyzer according to ASTM D6371. The acid value was measuring using the ASTM D664 method and flash point by standard method ASTM D93 using a semi-automatic Cleveland flash point analyzer (SETA, UK). The cetane number was measured by the ASTM D613 standard method in a Shatox Octane meter.

### 3. Results and discussion

#### 3.1. Catalysts characterization

XRD patterns of the synthesized catalysts are shown in Fig. 1. All of the calcium oxide modified carbon catalyst shows similar trends. The wide broad peak between 24°–26° can be attributed to pristine carbon due to its amorphous nature [25]. However, there are sharp peaks present at 2θ values of 17.1° and 34.2° which can be attributed to the impregnated material, CaO. Similarly assigned peaks have been reported before showing the presence of CaO [26,27].

Fig. 2a–h show the surface morphology and elemental composition of pristine carbon and the synthesized catalysts (CaO impregnated carbon). EDS was taken on various sample areas in order to observe the dispersion of CaO on pristine carbon. The average wt% of Ca has found to be ±0.3 which is consistent with our experimental value. Therefore, it can be concluded that CaO is well dispersed on the surface of pristine carbon and the difference between various CaO concentrations impregnated can be attributed to the increment of the dispersed particles as shown in Fig. 2. Based on SEM and EDS results, CaO is well dispersed on pristine carbon material. Moreover, the surface morphology of pristine carbon was unchanged after CaO impregnation. Furthermore, XPS was used to characterize the surface of the catalysts and the O 1s peaks are shown in Fig. 3a and b. Binding energies were observed at 530.2 eV, 530.4 eV, 530.6 eV and 530.8 eV for C (pure carbon), C1 (carbon with 2 wt% CaO), C2 (carbon with 4 wt% CaO) and C3 (carbon with 6 wt% CaO). Due to the impregnation of CaO in the catalyst, there is a binding energy shift of ca. 0.2 eV that was

observed at a higher value from 530.2 eV for C (pure carbon) to 530.4 eV C1 (carbon with 2 wt% CaO). When the CaO concentration was further increased, a greater binding energy shift was observed which might be due to the bond formation of Ca–O. Table 1 shows the oxygen concentration based on XPS analysis indicating higher concentrations of oxygen observed in the C2 sample. Thus, the greater oxygen content gave rise to more basic sites for C2 sample.

Table 2 shows the physiochemical properties of prepared catalysts. The pristine carbon material has a high surface area along with a large pore volume and diameter. However, after impregnation resulting in different wt% of CaO, the surface area along with the pore volume and diameter of resultant material decreased. Thus, the decrease in the surface area is due to impregnation of CaO which occupy the pore space of base carbon material and reduces the pore volume of resultant material. It has been reported that the particle size of triglyceride is 5.8 nm. The pore diameters of the catalyst should be higher than 5.8 nm in order to provide better channelling within the catalyst for reactants [28]. Therefore, based on BET results it can be concluded that the synthesized catalyst is suitable for transesterification for triglyceride for biodiesel production.

#### 3.2. Catalyst evaluation and parametric study

Table 3 shows the chemical composition of date pit oil which was determined prior to biodiesel production. It can be seen that unsaturated fatty acids are an in excess amount as compared to saturated components. The higher degree of unsaturation provides better low-temperature properties. Transesterification of date pits oil was conducted in presence of all three synthesized catalysts to evaluate the best catalyst as shown in Fig. 4. The transesterification was run in presence of different catalysts at similar operation conditions (temperature ~60 °C, time 60 min, methanol to oil molar ratio 6 and catalyst loading 2 wt%) revealed that best catalyst is C2 (carbon with 4 wt% CaO). Moreover, the performance of C2 can be related to the XPS results which shows a shift in O1s, in which it was observed that C2 possess a maximum quantity of normalized oxygen as shown in Table 1, which can be attributed to the fact that it possesses the maximum number of basic sites. Thus C2 was used for further optimization of biodiesel production.

Fig. 5a shows the parametric study for biodiesel production by changing process variables which include temperature, time,

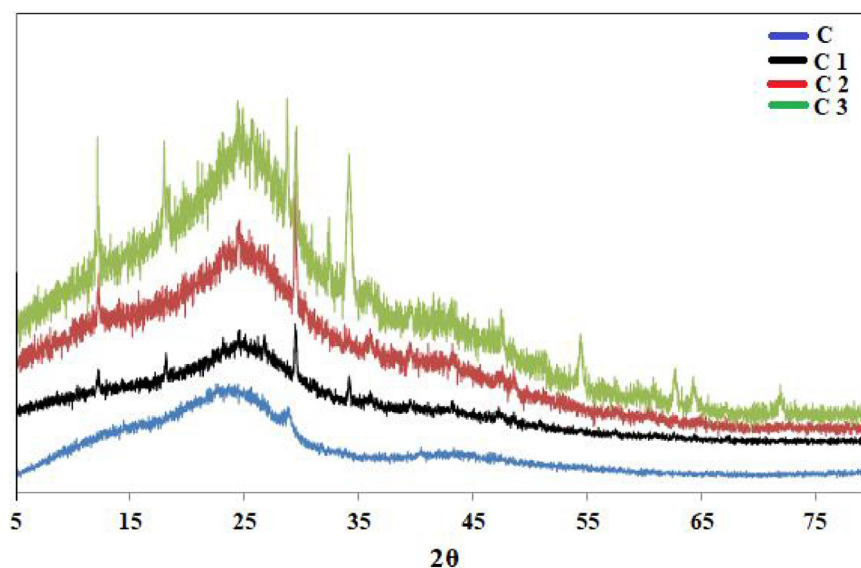
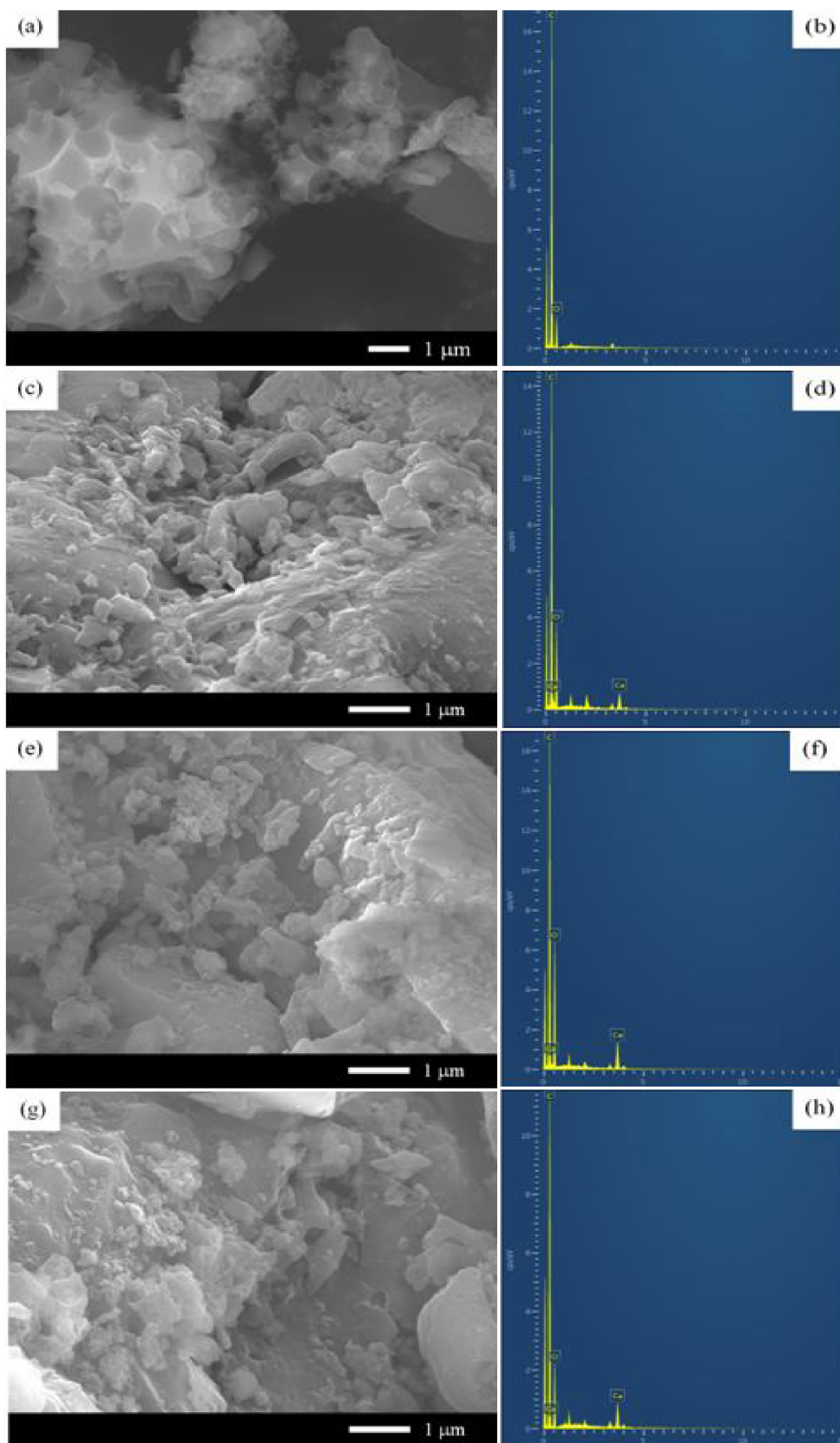
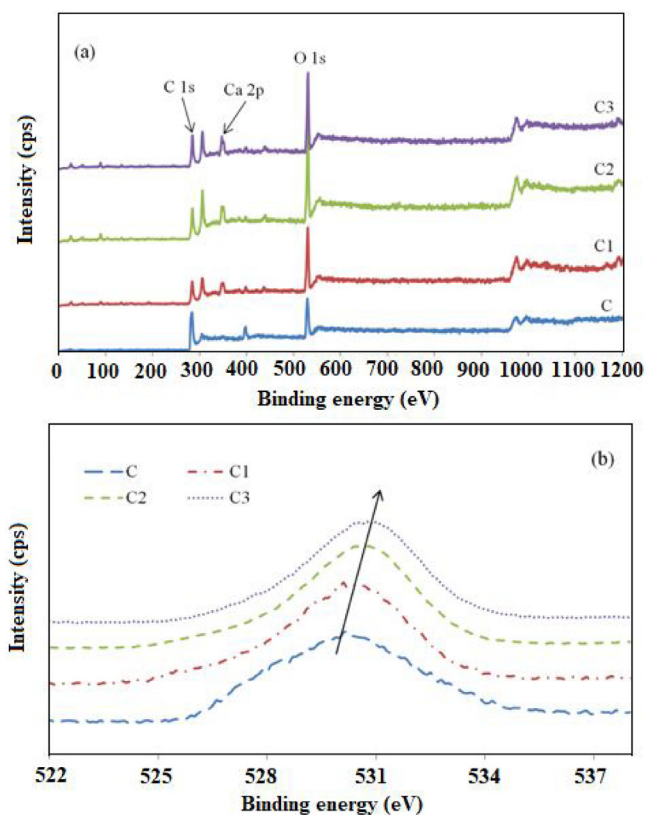


Fig. 1. XRD patterns of pristine carbon (C) and its modified forms (C1–C3).



**Fig. 2.** SEM and EDS images of synthesized catalysts (a and b) pristine carbon (c and d) 2% CaO impregnated on pristine carbon (e and f) 4% CaO impregnated on pristine carbon and (g and h) 6% CaO impregnated on pristine carbon.



**Fig. 3.** (a) The XPS survey spectra for pristine carbon and CaO impregnated carbon samples (C1, C2 and C3) (b) High resolution XPS spectra of O 1s peak for C (pristine carbon), C1 (carbon with 2 wt% CaO), C2 (carbon with 4 wt% CaO) and C3 (carbon with 6 wt% CaO).

**Table 1**

Oxygen concentration of prepared catalysts based on XPS analysis.

Catalyst	Normalized oxygen concentration
C	0.14
C1	0.15
C2	1.00
C3	0.78

**Table 2**

Physiochemical properties of synthesized catalysts.

Properties	Carbon	C1	C2	C3
$S_{BET}$ ( $m^2/g$ )	398.1	255.8	232.2	205.5
Pore Volume ( $cm^3/g$ )	0.25	0.21	0.17	0.13
Pore Diameter (nm)	8.62	8.01	7.19	5.68

**Table 3**

Fatty acid profile of waste date pit oil.

Fatty acid	Date seed oil (wt%)
Lauric acid (C 12:0)	16.44
Myristic acid (C 14:0)	9.96
Palmitic acid (C 16:0)	9.54
Stearic acid (C 18:0)	2.38
Oleic acid (C 18:1)	55.36
Linoleic acid (C 18:2)	6.32

methanol to oil molar ratio and catalyst loading. It can be observed that as the temperature is increased the biodiesel yield increased. The increase in biodiesel yield can be related to the fact that as temperature is increased the miscibility of reactants is increased which allow more reactants to interact to transform into product biodiesel. At high temperatures, the affinity of the carbonyl group in triglyceride increases towards the nucleophilic methanol resulting in the higher biodiesel yield [29]. Transesterification is endothermic reaction and is favored at high temperature. However, if the temperature is increased beyond 70 °C, the decrease in the yield of biodiesel was observed. Thus, this decrease in biodiesel yield at higher temperature can be attributed to the excess vaporization of methanol at a higher temperature which leads to decreased availability in the liquid phase. Moreover, it has been also reported that at higher temperatures, the polarity of methanol also decreases which leads to a decrease in biodiesel yield. Fig. 5b shows the effect of time on biodiesel yield. It can be observed that as the time is increased the biodiesel yield increased which can be attributed to the fact that both reactants require a certain time period to complete their reaction to give the product. However, it can be seen that if the reaction time increases beyond 120 min, a decrease in biodiesel yield is observed. As transesterification is a reversible reaction, the decrease in yield is due to an increase in the rate of the reverse reaction with time due to higher product concentrations.

Fig. 5c shows the effect of methanol to oil ratio on biodiesel yield. The yield at six methanol to oil ratio was low and increases until the ratio reached 12 but slightly decreased at a methanol to oil ratio of 15 and further decrease when the ratio was 18. Methanol to oil ratio is considered an important parameter as it constitutes one of the reactant amounts. Based on the stoichiometric calculation, three moles of methanol are required but it is always used in excess to avoid the reversible reaction. Thus, up to certain limit excess methanol can lead to yield increases. If methanol is in excess in a large amount, it may end up decreasing biodiesel yield due to the increased difficulty of separation of product and by-product [30].

Fig. 5d shows the trend of biodiesel yield with the catalyst loading. At minimum catalyst loading (1.5 wt%) the yield of biodiesel was low and increases as the catalyst amount increases. This can be attributed to the fact that the number of active sites were less but with increasing catalyst amount (4.5 wt%), sufficient active sites were present to give the maximum biodiesel yield. Increasing the catalyst amount beyond (4.5 wt%) led to a decrease in yield, which can be related to the fact that excess heterogeneous catalyst may cause issue with reactant interaction with active sites and this mass transfer limitation causes a decrease in biodiesel yield [29,31]. Moreover, in an excess amount of catalyst, more reactants are adsorbed on catalyst however desorption of product may decrease and this results in a decrement in biodiesel yield [32]. Thus, due to decrement in biodiesel yield by increasing catalysts amount the optimum catalyst is 4.5 wt%. Therefore, based on the parametric study, the optimal biodiesel yield obtained was 98.2 wt% at the following operating conditions: temperature 70 °C, time 120 min, methanol to oil molar ratio 12 and catalyst loading 4.5 wt%. It can be concluded that synthesized catalyst is highly active which led to high biodiesel yield at mild operating conditions.

### 3.3. Catalyst reusability

In order to consider the feasibility of long-term use of the catalyst, its reusability was examined based on the biodiesel yield at the optimal set of operating conditions. Fig. 6 shows the reusability of C2 catalyst under optimized operating conditions. After each reaction run, the catalyst was thoroughly washed with

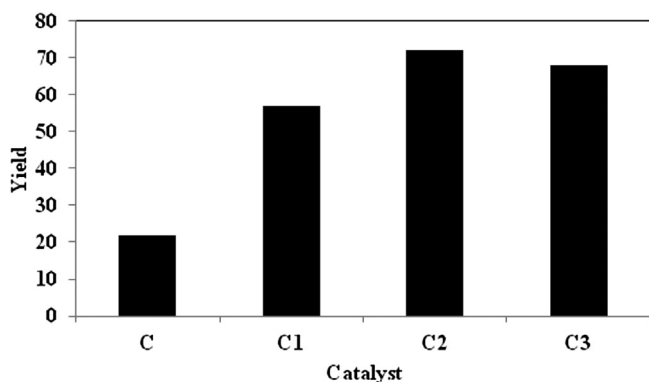


Fig. 4. Catalysts evaluation by transesterification of waste date pits oil.

absolute ethanol and dried at 100 °C for 4 h in an oven. The catalyst was found to be highly active over five repeated runs without a substantial decrease in the yield. However, in the sixth run, yield decreased as shown in Fig. 6. Thus, based on current observation, it

can be concluded that the catalyst is highly active and reusable. The decrease in biodiesel yield might be due to leaching and deactivation of active sites. The fact that the catalyst is made from a biomass-derived waste makes it attractive to use.

#### 3.4. Quality analysis of biodiesel

The quality of the synthesized biodiesel was evaluated based on ASTM and EN standards. All determined fuel properties are listed in Table 4 and compared to values provided by standards of ASTM and EN. The cetane number for produced biodiesel is 59.31 which is higher than the minimum limits set by ASTM and EN standards. The cetane number is an important property to be determined due to its direct relation to engine ignition. The cetane number is dependent on product composition. Biodiesel with a higher number of saturated components has a higher cetane number which shortens the delay in the ignition and gives better ignition. The cloud point (minimum temperature at which crystals starts appearing in liquid fuel) was determined to be 3.6 °C for produced biodiesel. Furthermore, the pour point of produced biodiesel was -2.1 °C which is reasonable for fuels

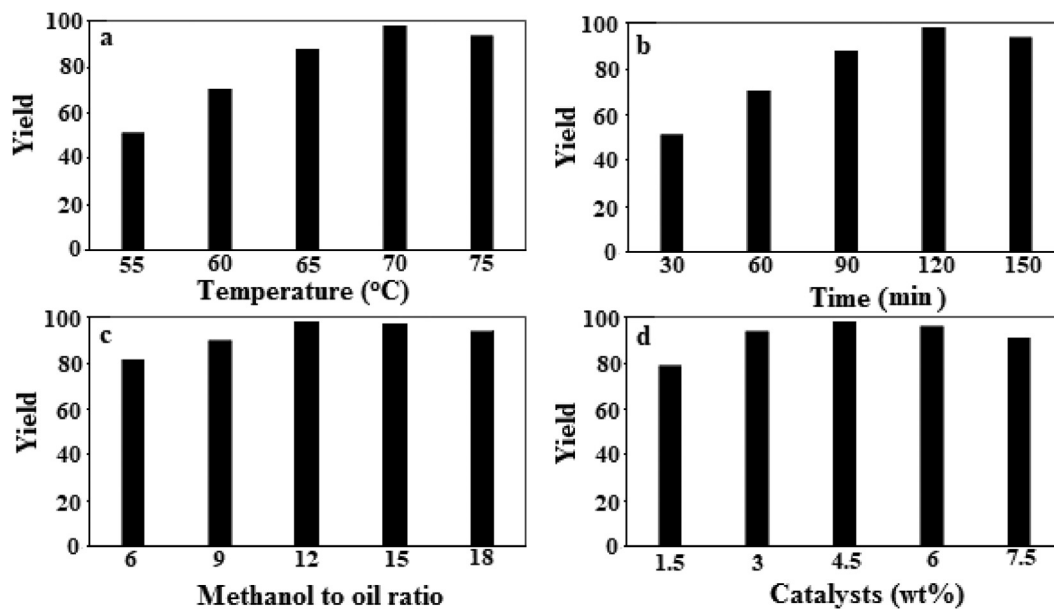


Fig. 5. Parametric study for biodiesel production through transesterification in presence of C2 catalysts by varying (a) temperature, (b) time, (c) methanol to oil ratio and (d) catalyst loading.

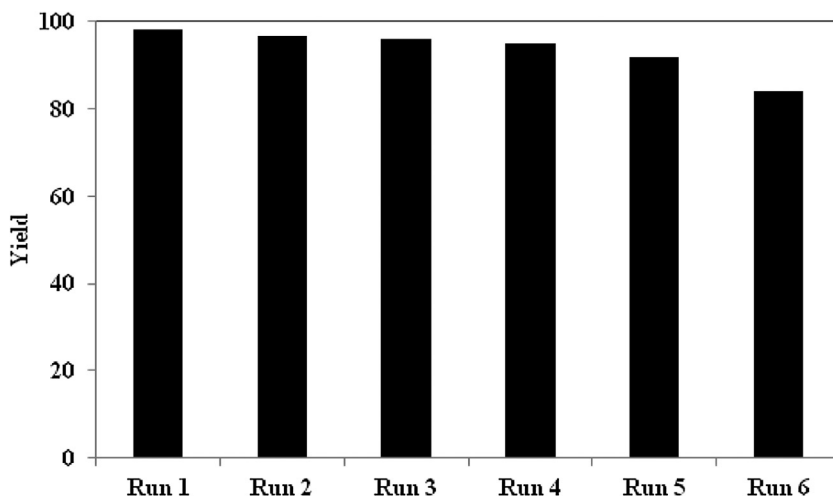


Fig. 6. Catalyst reusability test by transesterification.

**Table 4**  
Fuel properties of produced biodiesel from waste date pit oil by transesterification.

Property	Biodiesel	EN 14214	ASTM 6751
Cetane number	59.31	51 min	47 min
Cloud point (°C)	3.6	– <sup>a</sup>	–
Pour point (°C)	–2.1	– <sup>a</sup>	– <sup>b</sup>
Cold Filter Plugging Point (°C)	–0.79	– <sup>a</sup>	– <sup>b</sup>
Flashpoint (°C)	145	120 min	93 min
Gross Calorific Value (MJ/kg)	44.19	– <sup>b</sup>	– <sup>b</sup>
Viscosity (mm <sup>2</sup> .s <sup>–1</sup> ) at 40 °C	3.98	3.5–5.0	1.9–6.0
Density (kg. m <sup>–3</sup> ) at 25 °C	892	860–900	–
Acid Value (mg KOH/g)	0.26	0.50 max	0.80 max
Free Glycerin (%)	0.011	0.020 max	0.020 max
Total Glycerin (%)	0.210	0.250 max	0.240 max
Sulphur content	0.006	10 mg/kg max	15 mg/kg max

prepared from plant-based oil. The pour point refers to the minimum temperature at which liquid is still pourable. Further on, the cold filter plugging point of produced biodiesel is –0.79 °C. Thus, based on low-temperature performance profile, the produced biodiesel can be used as a fuel. The flashpoint of the produced biodiesel is 145 °C which is higher than the minimum standard limits defined by ASTM and EN. The larger flash point indicates better storage capability of biodiesel. Gross calorific value for produced biodiesel as mentioned in Table 4 is 44.19 MJ/kg. This value reflects the amount of energy produced after the complete combustion of produced biodiesel. The viscosity is an important property to be determined for fuel as it affects the atomization of the fuel. The viscosity of the produced biodiesel is 3.98 mm<sup>2</sup>/s which within the standard range defined by the ASTM and EN. The density of produced biodiesel was measured to be 892 kg/m<sup>3</sup> which is according to the range defined by EN standard. It is an intrinsic property of biodiesel and it has its direct influence on the breakup of fuel when it is injected into the engine for combustion. The acid value of produced biodiesel is 0.26 mg KOH/g which is less than the standard limit defined by ASTM and EN. The acid value is an important parameter to be determined to evaluate the quality of biodiesel, as biodiesel with higher acid value can cause engine corrosion. Moreover, the acid value predicts the storage stability because biodiesel with high acid values corrodes tanks. Moreover, the glycerin content of the produced biodiesel is within the standard limits and the produced biodiesel has a low sulfur content. Thus, based on all fuel properties it can be concluded that waste date pits can be used to produce high-quality biodiesel.

#### 4. Conclusions

A comprehensive study was performed about the value added to waste date pits to synthesize the green carbon catalyst and its utilization in biodiesel production from date pits oil. It was found that carbon catalyst modified with CaO possessed the suitable properties for transesterification of date pits oil. Analysis revealed that the most suitable catalyst, C2 (4 wt% CaO@Carbon), has suitable pore diameter 7.19 nm which can support the easy diffusion and pore channelling of the triglyceride molecules to transform into the biodiesel. The optimal yield of biodiesel of 98.2 wt% is quite high and confirms the suitability of the catalyst for transesterification reaction.

#### Conflict of interest

None.

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