

# One-pot synthesis of biodiesel from high fatty acid *Jatropha curcas* oil using bio-based basic ionic liquid as a catalyst

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**This article describes a facile one-pot synthesis of biodiesel from non-treated *Jatropha* oil (*J* oil) with high acid value (ca. 17 mg KOH/g). Choline hydroxide exhibited superior catalytic activity than choline imidazole in the transesterification that yields  $95 \pm 1\%$  of biodiesel, when the reaction was carried out with 9 : 1 molar ratio of methanol to *J* oil and 4 wt% catalysts at 60°C for 4 h. Further study showed that the catalyst exhibited almost constant activity for four successive trials after being recycled. The main fuel properties of the final product meets the ASTM standards. Moreover, the properties of biodiesel are identical to those obtained by conventional method (US 7,666,234 B2) and can be used as fuel in the existing diesel engines without any modification.**

**Keywords:** Biodiesel, *Jatropha curcas*, ionic liquid, transesterification.

BIODIESEL is an attractive and alternative fuel synthesized from renewable sources<sup>1,2</sup>. It possesses significant importance due to biodegradability, less pollutant emitting and non-toxic nature, displaying similar activity like conventional diesel<sup>3</sup>. Biodiesel is used in diesel engines with little or no engine modification<sup>4</sup>. Biodiesel is basically fatty acid methyl and/or ethyl ester produced from vegetable oil and animal fat<sup>4-6</sup>. Predominantly, *Jatropha* biodiesel is produced by transesterification process, which involves the reaction of triglycerides and free fatty acids of *Jatropha* oil (*J* oil) with excess alcohol, similar to low cost methanol in the presence of suitable catalyst<sup>5,6</sup>. Generally, transesterification can be catalysed by both basic and acidic catalysts<sup>7</sup>. Traditional alkali catalysts NaOH and KOH are not preferred because of serious saponification, slow reaction time and expensive purification<sup>8</sup>. Traditional acid catalysts like sulphuric acid and *p*-toluenesulphonic acid result in rigorous equipment corrosion and require long reaction time for high activity and cannot be recovered easily<sup>9</sup>. The solid catalyst faces

low activity, low reaction rate, low stability, requires high temperature and easy deactivation<sup>10</sup>. Transesterification of *J* oil with different ionic liquids (ILs) has been reported in the literature, but it requires higher temperature and results in low biodiesel yields<sup>11</sup>.

Ionic liquids are now considered as green solvent which has attractive properties like low vapour pressure, negligible volatility, high conductivity, better catalytic activity, strong dissolution ability and potential for reusability<sup>12-16</sup>. However, use of acidic ionic liquids needs high temperatures (>180°C) to obtain high activities, resulting in an energy-consuming and expensive process<sup>17,18</sup>. This directed efforts to explore basic ionic liquids for the synthesis of biodiesel which revealed that the basic ionic liquid transesterification is time saving and offers potential for reusability than the acidic ionic liquid transesterification<sup>19,20</sup>.

The purpose of the present work is to develop a facile one-pot process for the production of superior quality biodiesel from *J* oil through transesterification method using environment-friendly choline-based basic ionic liquid catalyst without any soap formation. Reaction conditions like temperature, time, molar ratio and catalyst dosage are optimized for obtaining the greatest conversion yields.

## Experimental section

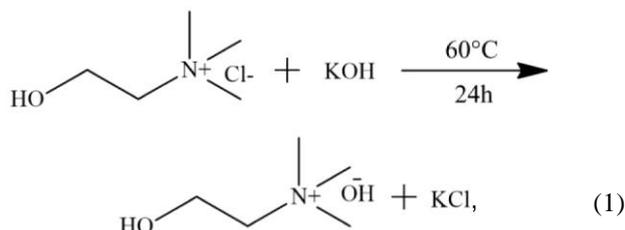
### Materials

*J* oil was used as expelled in our institute (CSIR-CSMCRI, Bhavnagar), without any free treatment. Choline chloride was purchased from TCI Chemicals, Tokyo, Japan. Potassium hydroxide, sodium hydroxide, imidazole and methanol were purchased from SD Fine Chemicals Ltd, Mumbai. All chemicals were used as received. Two different bio-ionic liquid catalysts, namely choline hydroxide (ChOH) and choline imidazole (ChIM) were prepared in the laboratory (as indicated below in eqs (1) and (2) respectively) and were used in this study.

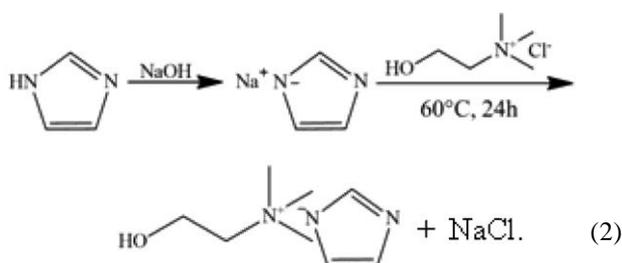
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### Preparation of catalyst

The preparation of choline hydroxide is shown in eq. (1), using a slightly modified process as described in the literature<sup>20</sup>. In brief, equimolar amount of choline chloride and KOH was dissolved in methanol separately and was charged into a round-bottom flask under stirring at ambient temperature. Then the reaction mixture was stirred at 60°C for 24 h followed by cooling to room temperature.



The preparation of choline imidazole is shown in eq. (2). Equimolar amount of choline chloride and sodium imidazole was dispersed in diethyl ether at ambient temperature. Then the reaction mixture was refluxed at 60°C with continuous stirring followed by cooling to room temperature.



### Transesterification

For biodiesel synthesis, a known amount of *J* oil, methanol and catalysts was added one by one into a round-bottom flask assembled with thermostat, condenser and mechanical stirrer. The stoichiometry for the reactions is 6 : 1, 9 : 1, 12 : 1, 15 : 1 and 18 : 1 ratios of methanol to *J* oil (MeOH/*J* oil). Methanol and *J* oil in the above-mentioned molar ratios were added into a round-bottom flask assembled with thermostat, condenser and mechanical stirrer by addition of known weight of basic ionic liquid (ChOH or ChIM) catalyst (from 1.0 to 5 wt%) at varying reaction temperatures (from 40°C to 70°C), and the reaction was allowed to proceed for 1–5 h to complete the transesterification. Reaction parameters were optimized on the basis of the greatest conversion of *J* oil into biodiesel. Similar sets of experiment were carried out under microwave irradiation conditions at varying temperatures from 40°C to 70°C to examine the efficiency of microwave heating. At least five runs were conducted for reproducibility of biodiesel.

After completion, the reaction mixture was allowed to cool to room temperature and transferred into a separating funnel. The mixture was allowed to settle down for 10 min after which it became biphasic – the upper phase having biodiesel and lower phase containing mixture of glycerol, methanol and catalyst was separated by discarding. The biodiesel layer was washed with distilled water to remove the trace amount of glycerol. Finally, trace amount of water was removed by purging of air or evaporation to obtain biodiesel products.

### Recovery of catalyst

To recover the catalyst, excess methanol was first removed from the bottom mixtures by simple distillation or evaporation. Solvent extraction method was employed to separate the ionic liquid from glycerol, as described in the literature<sup>20</sup>. In brief, the extraction process involves the addition of butanol and water, so that they form a biphasic mixture. Ionic liquid was removed from the butanol layer, while glycerol was separated through simple distillation from the water layer due to high boiling point (290°C) of glycerol.

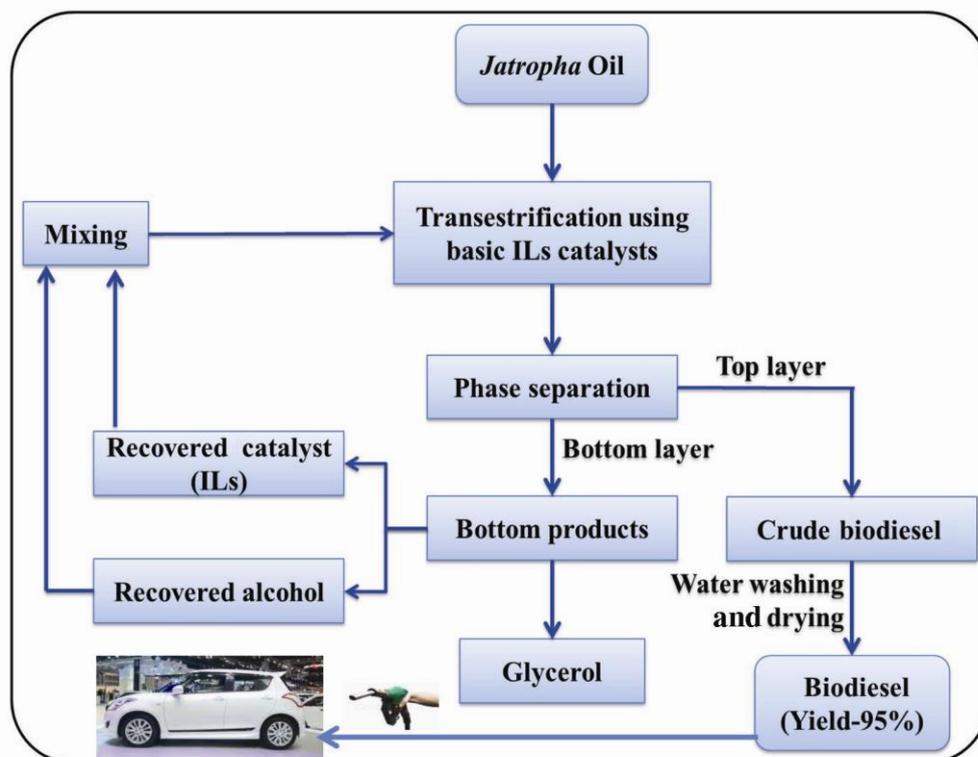
### Characterization of methyl ester

FTIR spectra were recorded on a Perkin-Elmer FTIR machine (Spectrum GX, USA). Methyl esters were characterized by GC-MS analysis of biodiesel (Shimadzu GCMS QP 2010, Japan, using RTX5 column with dimensions 30 m × 0.25 mm × 0.25 m). Moisture content (MC) was measured by Karl-Fisher method using Radiometer TIM 880 Titration Manager, France. The free fatty acid in the oil was determined by titrating it against potassium hydroxide (KOH) using phenolphthalein as indicator<sup>21</sup>. For this 2 g of oil was dissolved in 50 ml of methanol in a 250 ml conical flask; then 3 to 5 drops of phenolphthalein indicator were added and titrated against 0.1 N KOH. The content was constantly stirred until a pink colour which persisted for 15 s was obtained. Total glycerol in the final biodiesel sample was determined utilizing a standard procedure (ASTM D7637). Density bottle was used to determine the density of oil. A clean dry bottle of 25 ml was weighed ( $w_0$ ) and then filled with the oil; a stopper was inserted and then reweighed ( $w_1$ ). The oil was substituted with water after washing and drying and weighed again ( $w_2$ )<sup>21</sup>. The specific gravity was determined as follows

$$\text{Specific gravity} = w_1 - w_0 / w_2 - w_0.$$

### Determination of flash point, caloric value and cloud point

The cloud point (CP) was measured using American Oil Chemists (AOCS) official method Cc 6–25 for



**Figure 1.** Schematic representation of the synthesis of biodiesel from *Jatropha* oil using basic ionic liquids catalyst.

commercial oils and fats ([www.aocs.org/methods/search&buymethods](http://www.aocs.org/methods/search&buymethods)). The set-up contained a thermally insulated oil bottle of size 115 ml immersed in a water bath, the temperature of which can be controlled. The temperature was observed using a thermometer in the temperature range  $-50^{\circ}\text{C}$  to  $200^{\circ}\text{C}$ , with  $1^{\circ}\text{C}$  sensitivity. Initially the sample was heated to  $130^{\circ}\text{C}$  to destroy any crystal nuclei present. The sample was collected into the bottle so that upper level of the sample in the bottle was the same with water level in the bath. The sample was cooled on stirring so that the temperature of the sample was uniform. The temperature at which portion of the thermometer in the sample was no longer visible when viewed horizontally through the bottle and sample was recorded as CP. There was good repeatability of values and the deviation was not more than  $1^{\circ}\text{C}$ . Flash point was measured by Pensky Martin Flash point method in a flash point measuring instrument (Eie Instrument Pvt Ltd, Ahmedabad). Caloric value was measured using Toshiba<sup>TM</sup> digital bomb calorimeter (Toshiba India, Delhi).

## Results and discussion

A simple, eco-friendly and green process for the preparation of superior quality biodiesel from non-pretreated *J* oil is shown in Figure 1. In this process bio-based catalyst is used for direct conversion of *J* oil into bio-

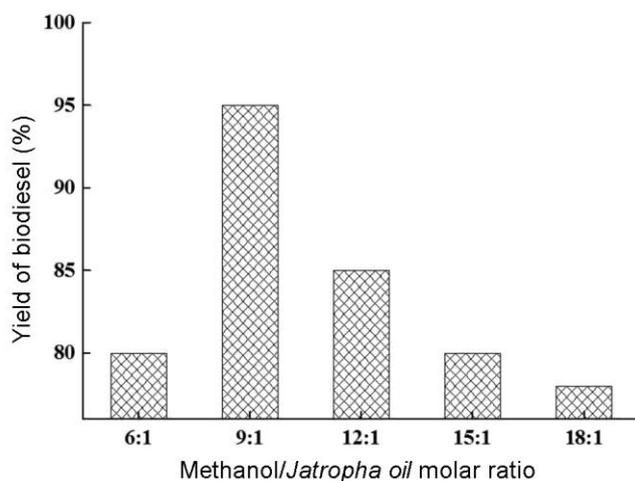
diesel without soap formation. The required parameters/properties such as density, flash point, viscosity, water content, FFA and caloric value, of the resulting biodiesel were identical to those of the German standard (DIN V 51606, 1997; Table 1), and in good agreement with those reported for biodiesel in the literature<sup>22,23</sup>. Viscosity is an important property of a fuel. It is a measure of resistance to flow of a liquid due to internal friction of one part of a fluid moving over another. The higher viscosity value may increase the tendency of the fuel to clog the engine components. However, the viscosity of the biodiesel produced in this study is 4.27 cP at  $40^{\circ}\text{C}$ , and within the range given by ASTM (1.9–6 cP) for biodiesels. Furthermore, it is also in good agreement with that reported for biodiesel product obtained from *J* oil by conventional method<sup>22</sup>. The biodiesel produced in the present study exhibits moisture content 210 mg/kg and free fatty acid 0.15%. These parameters are within the range of the standard. The specific gravity of the biodiesel produced in this study is 0.997 and in the range of the standard biodiesel sample<sup>22</sup>. Flash point and calorific value of biodiesel sample obtained in the present study are  $175^{\circ}\text{C}$  and 9173 kcal/kg, and values are identical with those ( $160^{\circ}\text{C}$ ; 9562 kcal/kg) reported for the biodiesel sample prepared by conventional method<sup>22</sup> (Table 1). The above specifications of the biodiesel prepared in this study reveal that it may be used as fuel in the existing diesel engines without any modification.

**Table 1.** Properties of *Jatropha* biodiesel

Property	Unit	DIN (V 51606, 1997) value	Value of the reference biodiesel sample <sup>22</sup>	Result/value of the present study
Density	g/ml	–	–	0.83
Viscosity at 40°C	mm <sup>2</sup> /s	3.5–5.0	4.34	4.27
Water content	mg/kg	<500	450	210
Free glycerin	% mass	<0.2	<0.02	0.01
Total glycerin	% mass	<0.25	0.04	0.11
FFA	% wt	–	–	0.15
Flash point	°C	>101	160	175
Cloud point	°C	–	–	2.5
Calorific value	kcal/kg	–	9562	9373

**Table 2.** Composition of *Jatropha* biodiesel

Sample	Biodiesel composition (methyl esters content %)					
	Methyl palmitoleate	Methyl palmitate	Methyl eicosanoate	Methyl oleate	Methyl stearate	Methyl salicylate
Reference biodiesel sample <sup>22</sup>	1.34	15.46	0.33	63.62	9.64	9.62
Biodiesel sample (prepared in the present study)	1.29	15.94	0.34	63.13	9.77	9.53

**Figure 2.** Influence of the methanol/*Jatropha* oil molar ratio on the conversion yield of biodiesel. (Optimum reaction parameters: temperature = 60°C; catalyst (ChOH) = 4 wt%, reaction duration = 4 h.)

### Spectral characterization

The formation of catalysts was confirmed by NMR spectra (Figures S1 and S2 in Supplementary Information; see online). The conversion of *J* oil into fatty acid methyl esters (FAMES) was confirmed by FTIR spectra (Figure S3 in Supplementary Information; see online). The appearance of the characteristics IR peaks in the spectra of biodiesel at 1440 cm<sup>-1</sup> (methyl ester O–CH<sub>3</sub>) and 1744 cm<sup>-1</sup> (C=O ester) confirmed the transesterification of *J* oil under optimized reaction condition achieved in the present study (Figure S3 c in Supplementary Information; see online), and identical IR peaks were found for the biodiesel sample

obtained by conventional method (US 7,666,234 B2)<sup>22</sup> (Figure S3 b in Supplementary Information; see online).

The composition of FAMES in the biodiesel samples is confirmed by GC analysis (Figure S4 in Supplementary Information; see online). The GC results confirmed that transesterification process developed in this study has successfully converted *J* oil into FAMES<sup>23</sup> (Figure S4 a in Supplementary Information (see online); Table 2). GC results revealed that biodiesel consisted of six FAMES, i.e. palmitic acid (C16:0) 15.94%, palmitoleic acid (C16:1) 1.29%, stearic acid (C18:0) 9.77%, oleic acid (C18:1) 63.13%, salicylic acid (C18:2) 9.53% and eicosanoic acid (C20:0) 0.34%. The FAMES composition of biodiesel obtained in this study was identical with that prepared by conventional method in our previous study<sup>22</sup> (Figure S4 b in Supplementary Information; see online).

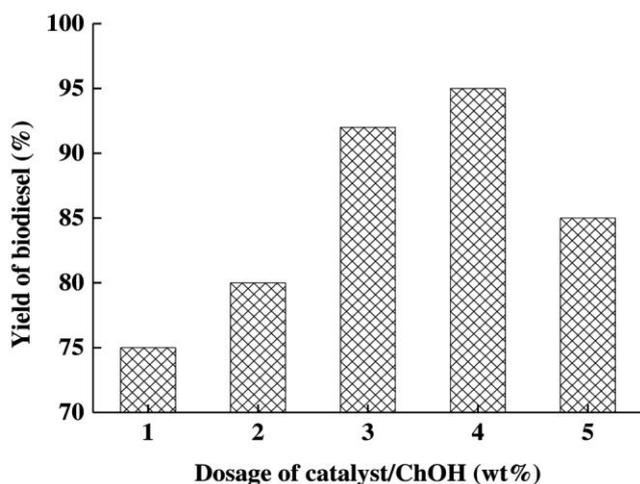
### Effect of methanol/*J* oil molar ratio

Stoichiometrically for transesterification of each mole of oil, triglyceride requires three moles of methanol<sup>24,25</sup>. Transesterification reactions were carried out using various MeOH/*J* oil molar ratios, e.g. 3:1, 6:1, 9:1, 12:1, 15:1 and 18:1, to study the effect of those ratios and the catalytic performance of choline hydroxide catalyst. Figure 2 shows the conversion efficiency of *J* oil into biodiesel with various molar ratios of MeOH/*J* oil. The yield of biodiesel significantly increases up to 9:1 molar ratio, followed by a gradual decrease beyond this value (Figure 2). As can be seen from Figure 2, yield of biodiesel was 79 ± 0.5% with 6:1 MeOH/*J* oil molar ratio, and was greatest (95 ± 1%) with 9:1 molar ratio of MeOH/*J* oil,

and sharp decrease up to 75% yield was observed up to 18 : 1 molar ratio of MeOH/*J* oil. The conversion yield increased while increasing the amount of methanol; this may be due to better dispersion of the ionic liquid catalyst and oil which can promote the catalytic activity. Further, increase in molar ratio lowers the conversion yield up to 76%, which may be due to the over-dilution of reactants *J* oil and the catalyst<sup>26</sup>. Results of the present study revealed that the optimum molar ratio of MeOH to *J* oil is 9 : 1, considering both factors of biodiesel yield and reactant methanol consumption during transesterification reaction, which is far better than 20 : 1 or 24 : 1 molar ratio for acid catalysts<sup>27,28</sup>.

### Effect of catalyst dosage

The influence of ChOH catalyst dosage on biodiesel synthesis was examined (Figure 3). Catalyst dosage represents the mass ratio of ChOH catalyst and the reactants (MeOH/*J* oil mixture), or can be represented as (ChOH: reactant w/w). Transesterification reactions were carried out using catalyst dosage from 1 to 5 wt% ChOH to study the effect of ChOH dosage on the conversion yield. The biodiesel yield steeply increased from 74% to 95% with an increase of catalyst dosage from 1 to 4 wt%, and began to decrease beyond this catalyst dosage (Figure 3). As shown in Figure 3, initial conversion yield was about 74%. This indicates that there are not enough sites for the reaction when the catalyst was 1 wt%, while biodiesel yield was 95% with 4 wt% catalyst. This may be due to the enough active sites available for the reaction. However, observed decrease in the conversion yield to 83% on further loading (with 5 wt% catalyst) is probably due to the favouring of soap formation and reverse reaction. Thus, 4 wt% catalyst dosage can be regarded as optimum



**Figure 3.** Effect of reaction time on the conversion yield of biodiesel. (Reaction conditions: Reaction temperature = 60°C; catalyst dosage = 4 wt%; MeOH/*J* oil molar ratio = 9 : 1.)

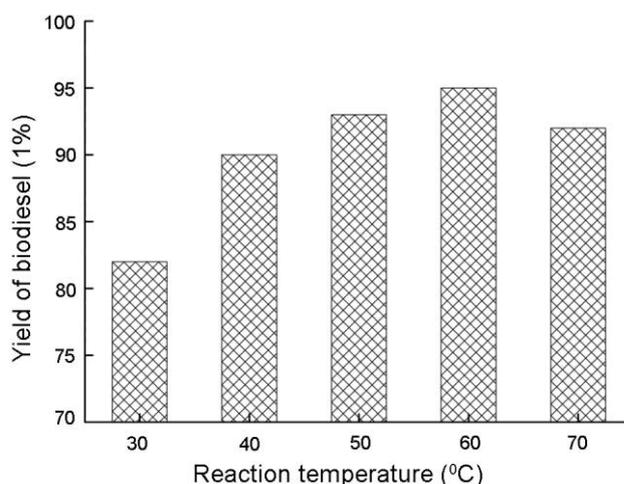
for obtaining maximum (95%) biodiesel yield in this study.

### Reaction temperature

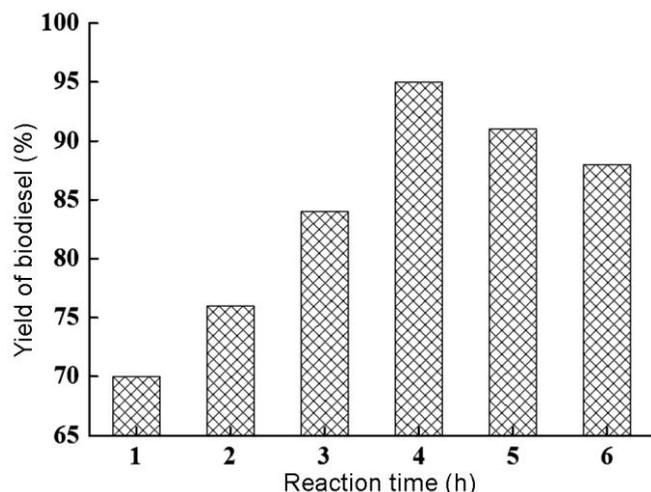
In order to determine the effect of reaction temperature on the conversion yield, the process was studied at different temperatures – 30°C, 40°C, 50°C, 60°C and 70°C. Figure 4 shows the chronological results of the study. It can be observed that the initial conversion yield of biodiesel is only 82% at room temperature. Maximum conversion yield of 95% was obtained at 60°C. Further increase of reaction temperature lowers the conversion, as it causes quick evaporation of methanol (b.p. 64.7°C) that decreases the net amount of methanol available for transesterification. Hence 60°C is to be considered as the optimum reaction temperature for the conversion yield and catalytic activity.

### Reaction time

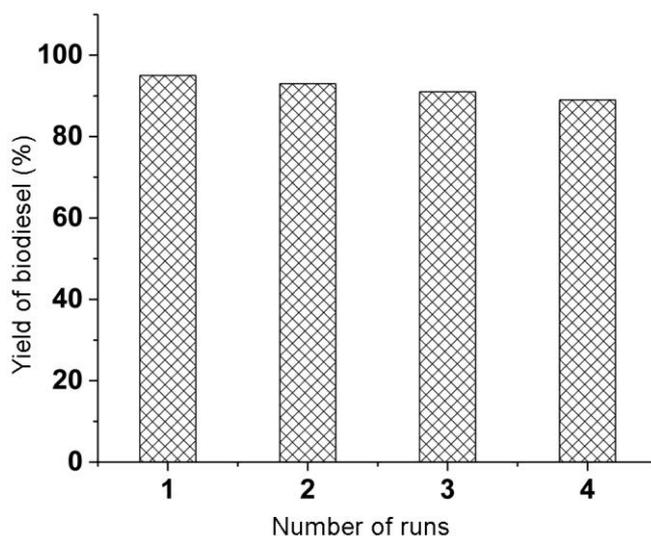
In order to observe the effect of reaction time on the conversion yield, reactions were carried out in the range 0.5–6 h. As seen from Figure 5, the biodiesel yield increased steeply when the reaction duration increased from 0.5 to 4 h, and declined beyond this reaction duration up to 6 h. Minimum 69% biodiesel yield was obtained after 0.5 h transesterification reaction, while maximum 95% biodiesel yield was obtained after 4 h transesterification reaction (Figure 5). Low biodiesel yield (69%) after 0.5 h may be due to the mixing and dispersion of methanol in *J* oil<sup>29</sup>. Moreover, steep increase in biodiesel yield was observed up to 4 h, after which there was a decline. This can be due to the favouring of backward reaction of



**Figure 4.** Effect of reaction temperature on the conversion yield of biodiesel. (Reaction conditions: catalyst dosage = 4 wt%; MeOH/*J* oil molar ratio = 9 : 1; reaction time = 4 h.)



**Figure 5.** Effect of catalyst ChOH dosage on the conversion yield of biodiesel. (Reaction conditions: reaction temperature = 60°C; MeOH/J oil molar ratio = 9 : 1, reaction duration = 4 h.)



**Figure 6.** Effect of reusability on the conversion yield of biodiesel. (Reaction conditions: reaction temperature = 60°C; catalyst dosage = 4 wt%; MeOH/J oil molar ratio = 9 : 1.)

transesterification and also probably due to the intermediate soap formation<sup>30</sup>. Conclusively, 4 h reaction time could be optimum for transesterification reaction to obtain the maximum biodiesel yield in this study, which is far better than the 18–24 h reaction time, required for acid-catalysed transesterification of *Jatropha curcas* seed oil for the production of biodiesel<sup>28,31</sup>.

### Reusability of catalyst

To study the reusability of ChOH as an environment-friendly catalyst for the basic-catalysed transesterification of *J* oil, we performed a series of recycling experiments. As mentioned above, ChOH was recovered to study the

reusability of the catalyst and was characterized by FTIR spectroscopy. The fresh transesterification experiments were done to test the reusability of ChOH catalyst, and results are shown in Figure 6. It can be seen from Figure 6 that yield of biodiesel was 93% for the second time, 91% for the third time, 89% for the fourth time, and that the ChOH catalyst still yielded 87% after four successive uses. Decreasing catalytic activity might be due to the high FFA content in oil that affects catalytic activity.

### Conclusion

The properties of biodiesel produced in the present study were identical to those of the German standard (DIN V 51606) and the reference biodiesel sample<sup>22</sup>. It may be concluded that the choline-based bio-catalyst combined with conventional heating is an effective method for the production of biodiesel from *Jatropha* oil, and it may be used as an eco-friendly fuel without modifications of existing diesel engines.

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