

Crystalline Manganese Carbonate a Green Catalyst for Biodiesel Production

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ABSTRACT

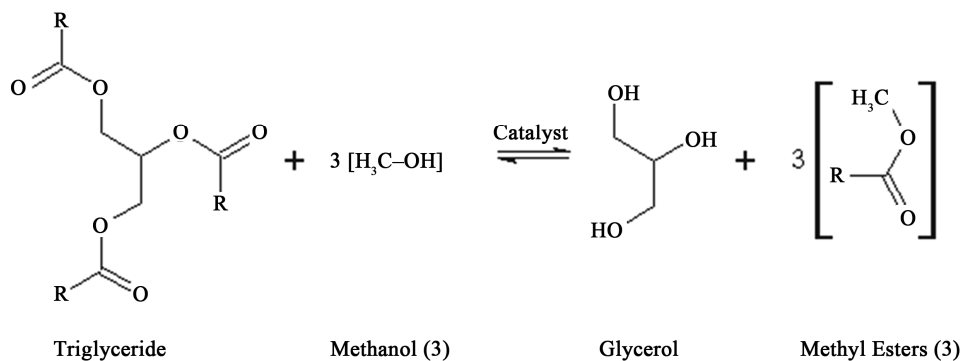
Crystalline manganese carbonate was found to be a versatile green, non corrosive and environmental friendly catalyst for transesterification of vegetable oils. Its use as catalyst in the transesterification process involving methanol and vegetable oils (palm, rapeseed, groundnut, coconut and castor oils) resulted in a conversion rate of 80% - 95% in the production of biodiesel. The chemical composition of the obtained biodiesel was studied by GC-MS analysis and showed the presence of linoleic, oleic, palmitic, and stearic acids methyl esters to be the major compounds. Manganese carbonate in comparison with other solid catalysts was found to decrease the reaction time and temperature concomitant with an increase of biodiesel yield. Finally, the effect of various parameters including methanol quantity, catalyst amount, reaction time and temperatures on the production of biodiesel was investigated.

Keywords: Biodiesel; Vegetable Oils; Manganese Carbonate; Green Catalyst; Transesterification

1. Introduction

Rising petroleum prices and continuous increase of petroleum usage may drive the world, in near future, to feel the pinch of deficiency of transportation fuels. Biodiesel as an alternative fuel for diesel engines is becoming increasingly important due to diminishing petroleum resource [1,2]. Various attempts are being made for the search of new catalysts and vegetable oil sources, in transesterification reactions, for the production of biodiesel [3-5]. Biodiesel synthesis using solid catalysts instead of homogeneous catalysts could potentially lead to cheaper production cost by enabling reuse of the catalyst. Biodiesel is produced by the transesterification of triglycerides of re-

fined or edible and non-edible oils by using alcohol and alkaline catalyst. The transesterification process consists of sequences of three consecutive reversible reactions, which includes conversion of triglycerides to diglycerides, followed by diglycerides to monoglycerides. The glycerides are converted in to glycerol and yield one ester molecule in each step [6]. The overall transesterification reaction can be represented by the following **Scheme 1**. The reaction is normally performed at reflux temperature; the glycerol and FAME (fatty acid methyl ester) are separated by settling after catalyst neutralization. The crude biodiesel and glycerol obtained are then purified. There are different transesterification processes that can be applied to



Scheme 1. Transesterification of triglycerides with alcohol.

synthesize biodiesel like base catalyzed transesterification, acid catalyzed transesterification and enzyme catalyzed transesterification [4]. Biodiesel is currently synthesized using homogeneous acid catalysts, the rate of reaction much slower than the base catalyzed reaction. There are several comprehensive studies of base catalyzed transesterification. The most common basic catalysts are potassium hydroxide (KOH) and sodium hydroxide (NaOH) [7,8]. Even though homogeneous catalyzed biodiesel production process are relatively fast and show high conversion with minimal side reactions, they are still not very cost effective with petrodiesel. The transesterification reaction can also be catalyzed by Bronsted acids preferably sulphonic and sulphuric acids, but the reaction rates are very low and require relatively high temperature to get the high product yield [9]. According to an acid catalyzed mechanism for esterification, carboxylic acid can be readily formed by hydrolysis of the carbocation intermediate formed upon protonation of the ester. This suggests that acid catalyzed transesterification should be carried out in the absence of the water to avoid the competitive formation of carboxylic acids and concomitant reduction in the yields of alkyl ester [10]. The most common acid catalysts used are H_2SO_4 and HCl. The scientist Freedman et al showed that the methanolysis of soybean oil, in the presence of 1 mol% of H_2SO_4 with an alcohol/oil molar ratio of 30:1 at $65^\circ C$, takes 50 hrs to reach complete conversion of the vegetable oil [11]. The transesterification of oils is also carried out by solid super acids such as tungstated zirconia-alumina (WZA), sulphated tin oxide (STA) and sulphated zirconia-alumina (SZA), of which the WZA catalyst was the most effective achieving conversion $>90\%$ at temperature above $250^\circ C$ after 20 h [12]. Current industrial catalysts for biodiesel are based on the basic catalyst mentioned above require pretreatment of the feedstock to remove impurities, water and the free fatty acids (which precludes the use of low quality feedstocks such as waste oils) [13,14]. They also lead to saponification (*i.e.* soap formation) of vegetable oil, which occur as an undesired side reaction and necessities lengthy after process separation procedures. These production steps negate the low price of the catalyst and are energy intensive. It is therefore necessary to develop new environmentally benign, inexpensive and effective catalysts which avoid the costly saponification reaction. A wide range of alternatives have been proposed but to date none are ideal. In this work, manganese carbonate ($MnCO_3$) was found to be a versatile catalyst in transesterification reaction to produce monoalkyl ester (biodiesel). In this communication manganese carbonate was found to be a versatile catalyst with 80% - 95% conversion rate in the production of biodiesel involving, vegetable oils like palm oil, rapeseed oil, groundnut oil, coconut oil and castor oil. Although palm oil and rapeseed

oil are extensively used for biodiesel production the other oils mentioned are studied for comparison purposes.

2. Experimental Details

2.1. Materials

All chemicals used in the experiments such as methanol (99.5%), chloroform, petroleum ether, sodium sulphate, sulphuric acid were of analytical reagent grade and silica gel G LR for TLC were purchased from Sarabhai Division Fine Chemicals (Mumbai, India). Refined palm, sunflower, groundnut, coconut, rapeseed and castor oils were purchased from local food store. Pure, ash colored, crystalline manganese carbonate was purchased from Chemical Corporation (Mumbai, India).

2.2. Transesterification

Transesterification process was performed in 25 ml round bottom flask with reflux condenser and a magnetic stirrer. Stirring was set at constant speed throughout the experiment. The manganese carbonate in methanol (0.5% - 3% w/v) was used for the conversion of oil to fatty acid methyl ester (FAME), other oil to methanol ratio varied from 5:1 to 12:1 v/v. A known amount of catalyst was added in the required amount of methanol and was heated separately at desired temperature. This methanolic catalyst was added to the preheated oil and the reaction was kept under reflux condition. Formation of methyl ester from vegetable oil was monitored by thin layer chromatography. After reaction completion, the formed methyl esters mixture was extracted with chloroform dried over sodium sulphate. Chloroform was evaporated using a rotary evaporator and the obtained methyl esters mixture was then subjected to GC-MS analysis.

2.3. Analysis of Fatty Acid Methyl Esters

Gas chromatography has been to date the most widely used method for the analysis of biodiesel due to its higher accuracy in unifying minor components [15]. The sample was analyzed with shimadzu GC-2010 gas chromatograph equipped with splitless injection system. Helium was used as carrier gas. The condition of instrument were: column oven temperature $75^\circ C$, injection temperature set at $280^\circ C$, the detection was made with a flame ionization detector (FID) at $310^\circ C$, flow control mode in linear velocity with 26.0 cm/sec, total flow 14.0 ml/min, column flow 1.0 ml/min, purge flow 3.0 ml/min, pressure 131.6 Kpa where as the split ratio 10.0. Samples were prepared for analysis by adding approximately 0.05 gm of oil phase to 5 ml of n-hexane. About 1 ml of this mixture was put in to GC auto sampler vials. Two micro liters of the sample were injected in to the column.

2.4. Recovery of Catalyst

The catalyst thoroughly washed 4 - 5 times with water for 35 hrs. After complete drying, the catalyst was re-used for transesterification reaction. The obtained results showed a catalytic efficiency of 95% and decreased after 7 times uses.

2.5. Fuel Properties of Fatty Acid Methyl Esters

The product of above transesterification was analyzed, taking into consideration specifications for biodiesel as fuel in diesel engines. The following properties of the biodiesel produced were determined: Density, Acid value, Cloud point, Pour point, Viscosity (By Redwood viscometer), Flash point (By Cleveland's open cup apparatus). Most of these parameters comply with the limits prescribed in the ASTM D6751-02 standards for biodiesel. Each experiment was conducted in triplicate and the data reported as mean.

3. Results and Discussion

3.1. Composition of Oil

The palm oil was found to give the highest oil yield with 5000 kg oil per hectare; this value is far higher than other oils which are in the range of hundreds to 2000 kg oil per hectare. The oil yield from the crops itself is always the key factor to decide the sustainability of feed stock for biodiesel production. Oil crops with higher oil yield are more preferable in the biodiesel industry because it reduces the production cost. The important criteria to determine the sustainability of oil as feedstock for biodiesel is its composition which will subsequently determine the properties of the obtained biodiesel. The composition of the palm, sunflower, groundnut, coconut, rapeseed and castor oils are well reported in literature as shown in

Table 1 The results showed that the Palm oil contains the 85% saturated acids (palmitic and stearic) and 15% unsaturated acids (linoleic, oleic). Rapeseed oil contains

86.4% unsaturated acids and only 4.4% saturated acid. Sunflower and castor oil showed high content of linoleic 73% and ricinoleic acid 89% respectively. The different results obtained in the properties of biodiesel is due to the variation of compositions of oils. The palm oil has 40% oleic and 45% palmitic acid as major components and the composition of sunflower, coconut, rapeseed, castor oils differ in the fatty acid compositions. The rapeseed oil has 64% of oleic acid as major component. This could be the reason why this is preferred oil for the production of biodiesel in Europe. In contrast castor oil has 89.5% of ricinoleic acid as major component which forms resins as effects transesterification and so is not a preferred oil for the producing biodiesel. By and large the oils studied exhibit 85% and above composition of saturated acids and facilitate the production of biodiesel in large quantity. The oil composition of palm, sunflower, groundnut, coconut, rapeseed and castor oils is shown in **Table 1**.

3.2. Analytical Results

In order to study the obtained biodiesels composition, GC-MS analysis was used for the determination of the composition of the obtained fatty acid methyl esters. The retention times in minute for palm, sunflower, coconut and rapeseed oils are 10.763 - 26.885, 10.893 - 27.300, 14.739 - 26.380 and 10.763 - 26.885 respectively. The percentage for palm, sunflower, coconut and rapeseed oils are 100.01%, 99.99%, 100%, and 100.01% respectively. The obtained qualitative and quantitative analysis and the result of methyl ester components in palm and sunflower transesterification products are presented in **Table 2**.

The **Table 2** indicates that the FAME from sunflower oil contained mainly methyl linoleate (47.43%) and methyl oleate (32.49%), which are comparable to fatty acid composition of same feedstock. While palm oil methyl esters consisted mainly of methyl palmitate (42.42%) and oleate (47.04 %).

Table 1. Composition of various oils in %.

Fatty acid composition (%)	Molecular formula	Palm	Sunflower	Coconut	Rapeseed	Castor
Oleic	C ₁₈ H ₃₄ O ₂	40.0	16.93	0.3	64.1	3.0
Linoleic	C ₁₈ H ₃₂ O ₂	10.0	73.73	0.2	22.3	4.2
Palmitic	C ₁₆ H ₃₂ O ₂	45.0	6.8	0.2	3.5	1.0
Stearic	C ₁₈ H ₃₆ O ₂	5.0	3.26	0.2	0.9	1.0
Linolenic	C ₁₈ H ₃₀ O ₂	-	00	-	-	0.3
Eicosenoic	C ₂₀ H ₃₈ O ₂	-	-	-	-	0.3
Ricinoleic	C ₁₈ H ₃₄ O ₃	-	-	-	-	89.5
Dihydroxyteric	C ₁₈ H ₃₆ O ₄	-	-	-	-	0.7
Palmitoleic	C ₁₆ H ₃₀ O ₂	-	-	-	0.1	-
Myristic				0.3		
Others	-	-	-	-	9.1	-

Table 2. The analytical results of components of FAME from various vegetable oils using MnCO₃ catalyst.

Methyl ester	Palmoil %	Sunflower %	Coconutoil %	Rapeseed oil %
Methyl laurate	0.41	0.06	54.39	0.41
Methyl myristate	1.24	0.10	21.40	1.24
Methyl palmitate	42.42	14.96	10.58	42.42
Methyl palmitoleate	0.28	1.10	0.24	0.28
Methyl stearate	3.30	3.85	1.69	3.30
Methyl oleate	47.04	32.49	6.41	47.04
Methyl linoleate	5.32	47.43	5.29	5.32

3.3. Fuel Properties of Methyl Esters

The fuel properties of the obtained biodiesel are summarized in **Table 3** together with the relevant specifications in the ASTM biodiesel standards [16-19]. The properties of methyl esters obtained from different oils were compared with ASTM biodiesel standards. The results are shown in **Table 3**. Viscosity of the methyl esters obtained from the different studied oils was within the standard range of 3.5 - 5.0 mm²/s at 40°C. Hence no negative impact on fuel injector's system performance means produced biodiesel can be used in existing diesel engine. Flash point measures the tendency of the sample to form flammable mixture with air under controlled laboratory conditions. This is the only property that must be considered on assessing the inflammability hazard of the material. The flash point of methyl esters of studied oils is above the specification limits and is safe for transport.

In comparison with other solid catalysts such as KF/MgAl hydrotalcite, CaO-supported catalysts (Li/Cao, Na/Cao, K/Cao) was the effective achieving conversion more than 85% [12,16,20-23]. Manganese carbonate was found to be a green versatile catalyst which gives high biodiesel yield at low temperatures. Use of manganese carbonate may lead to cheaper production cost by enabling reuse of the catalyst and opportunities to operate in fixed bed continuous process. The process offers advantages in terms of mild conditions, high conversion and practical viability and may find commercial application for the biodiesel production.

3.4. Effect of Methanol to Oil Ratio

The amount of alcohol added to vegetable oil is one of the important factors that affect conversion efficiency as well as production cost of biodiesel. The conversion efficiency is defined as the yield of the process represented in terms of percentage. Generally the amount of alcohol required for transesterification reaction is analyzed in terms of volumetric ratio. Stoichiometrically, the alcohol/oil molar ratio is 3:1. Higher amount of alcohol is required to drive the reaction to completion at faster rate. It is observed that lower amount of alcohol requires longer reaction periods. The conversion efficiency of transesterification

reaction with increasing amount of alcohol obtained during present study is shown in **Figure 1**. The transesterification reaction was studied for different molar ratios. The methanol to oil molar ratio varied from oil to oil. The maximum ester conversion for palm oil was found to be 90% - 95% with a methanol to oil ratio of 5:1. In the case of sunflower and rapeseed oil maximum yield of 90% was obtained at 7:1 methanol oil ratio. The groundnut oil requires 9:1 ratio for the maximum conversion, while in case of coconut and castor yield achieved was 75% at 12:1 methanol to oil ratio which gives highest conversion of oil to methyl ester. **Figure 1** depicts the effect of alcohol to oil molar ratio on the conversion of oil to methyl ester.

3.5. Effect of Catalyst Amount

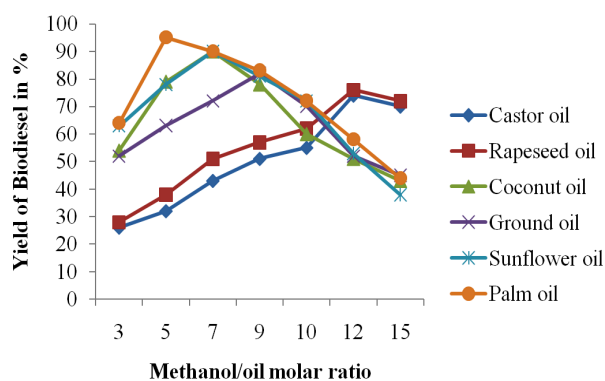
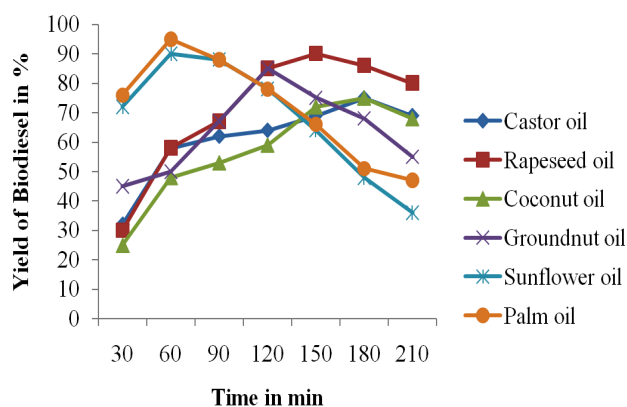
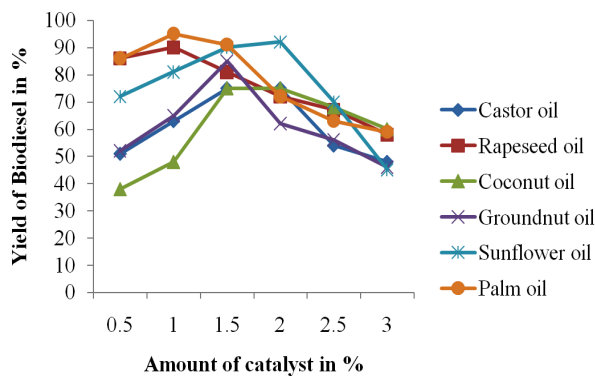
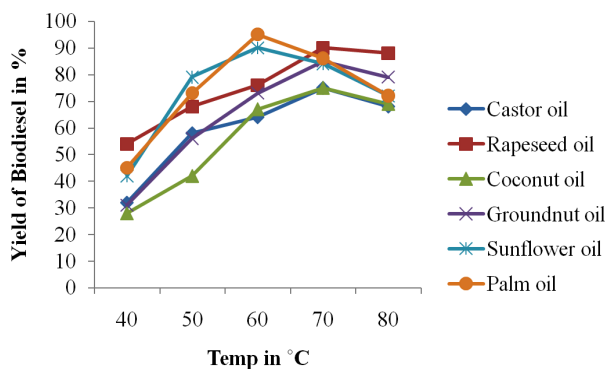
The catalyst amount in the range 0.5% - 3.0% (Weight of catalyst/volume of oil) is used in the present experimental analysis. The effect of catalyst amount on conversion efficiency is shown in **Figure 2**. The obtained results showed that the yield of palm and rapeseed oil was 95% and 90% obtained with a 1% of catalyst amount while sunflower and groundnut oil gave better yields, 90% and 85% respectively with 1.5% catalyst amount. Finally, the yield of 75% was obtained in the case of castor and coconut oils with 1.5% and 2.0% catalyst amount - respectively.

3.6. Effect of Reaction Temperature

Although the reflux temperature was in general 60°C - 80°C, the reactions temperature and the obtained results are shown in **Figure 3** and showed that the temperature required for the complete conversion of oil to fatty acid methyl esters varied from oil to oil. In the case of palm and sunflower oils, highest yield of 95% and 90% were obtained at 60°C respectively, while for groundnut, coconut, rapeseed and castor oils, the optimum yields obtained were 85%, 75%, 90% and 75% respectively and were obtained at 70°C. It was observed that for reaction temperature below 60°C, saponification of glycerides occurs very fast.

Table 3. Fuel properties of produced fatty acid methyl esters.

Properties	Palm oil	Sunflower	Coconut	Rapeseed	Castor	ASTM biodiesel standard
Viscosity mm ² /s at 40°C	4.42	3.26	3.9	3.51	5.0	3.5 - 5.0
flash point (°C)	162	180	171	150	190	130 min
Cloud point (°C)	5	4	6	-4	-3	-3 - 12
Pour point (°C)	10	9	9	-12	-8	-15 - 10
Density Kg/m ³	0.92	0.92	0.91	0.91	0.96	0.87- 0.90
Acid value mg KOH/g	0.1	0.15	0.3	0.89	2.0	0.5

**Figure 1. Effect of methanol/oil molar ratio.****Figure 4. Effect of reaction time.****Figure 2. Effect of amount of catalyst.****Figure 3. Effect of reaction temperature.**

3.7. Effect of Reaction Time

In order to study the effect of reaction of reaction time, experiments were carried out between 30 to 210 min for the studied oils and the obtained results are depicted in **Figure 4**. The obtained results showed that palm and sunflower oils achieved highest yield of 95% and 90% respectively at 60 min under reflux conditions. Groundnut and rapeseed oil gives 85% and 90% yield at 120 and 150 min respectively; while the transesterification yield of 75% was obtained in case of coconut and castor oils maximum time of 180 minutes was sufficient for the completion of reaction with highest yield of biodiesel.

3.8. Recovery and Reuse of Catalyst

Recovery of catalyst from the reaction mixture is an important operational parameter. From a financial economic point of view, the use of heterogeneous catalyst in biodiesel production could reduce its price becoming competitive with diesel. The used catalyst was recovered and reused for further transesterification reactions. The obtained results showed the recovered catalyst was still efficient even after seven successive uses. This suggests that the use of manganese carbonate could be a fruitful approach for biodiesel production.

4. Conclusion

In this paper, we have evaluated the catalytic activity of crystalline MnCO_3 as green catalyst since MnCO_3 is non corrosive, easy to handle and environmentally friendly and an alternative low cost catalyst. MnCO_3 efficiently promote the transesterification in methanol solutions and in the presence of vegetable oils. The high yields achievable under mild reaction conditions are comparable to those obtained with a common acid catalyst such as acid and base. Therefore it is demonstrated that, MnCO_3 is a potential catalyst for the production of biodiesel which is a low-cost raw materials. The advantage of this protocol is the use of an available low-cost catalyst, which is easy to manipulate and potentially less corrosive. The results of this work suggest MnCO_3 as a promising alternative catalyst for the production of biodiesel. As a final remark, we believe that the application of MnCO_3 potentially promote a reduction of the costs related to biodiesel production.

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