Green Technology for Biodiesel Production From *Mesua Ferrea* L. Seed Oil

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Abstract

In this paper, the non-edible *Mesua ferrea* L. (MFL) seed oil with an acid value 18.8 mg KOH/g of oil, was tranesterified in the presence 1 MPa hydrogen pressure and also without the application of external pressure at 250–275 °C for 1 hour. The heterogeneous 1–5 wt.% *Musa balbisiana* Colla underground stem (MBCUS) ash catalyst was used during transesterification. The catalyst has a nano-metric dimension and has a versatile composition that consist of several alkali metal oxides, alkaline earth metal oxides, non-transition metal oxides and nonmetal oxides. The resultant biodiesel was separated using rotary vacuum evaporator and purified using molecular sieve and carbon columns. The biodiesel so obtained from MFL seed oil have been characterized as per available methods and found that the fuel properties are in conformity to ASTM and EN standards. 5 wt.% catalyst was found more effective as compared to other composition during ethanolysis. The effect of hydrogen pressure upon tranesterification was found negligible. Ethanol is a renewable liquid because it is made from renewable precursor such as biomass. It is expected that the use of whole renewable precursor during the process will fulfil the demand of 100% Green Technology for biodiesel production.

Keywords: biodiesel, tranesterification, ethanolysis, *Mesua ferrea L.*, green technology

1. Introduction

Due to the limited resources of fossil fuel, increasing prices of crude oil and environmental concerns over the past couple of decades, biodiesel has recently gained significant attention (Lukic, Krstic, Jovanovic, & Skala, 2009). Biodiesel refers to a vegetable oil or animal fat-based diesel fuel consisting of alkyl (methyl, propyl, or ethyl) esters obtained by chemical reaction of the lipids (vegetable oil, animal fat) with an alcohol. Chemically, biodiesel consists of alkyl esters instead of the alkanes and aromatic hydrocarbons of petroleum derived diesel (Adebayo, Ameen, & Abass, 2001). More than 350 oil-bearing crops have been identified, among which mainly jatropha, sunflower, safflower, soybean, cottonseed, rapeseed, and peanut oils are considered to be a potential alternative fuels for diesel engines. Nonetheless, other unknown oleaginous crops, which are being grown in less-favoured countries, could perform well as an adequate fuel with chemical and physical properties similar to those of diesel fuels (Dorado, Ballesteros, Lopez, & Mittelbach, 2004). As India is deficient in edible oils, non-edible oils become the primary choice for biodiesel. Generally, a blend of 5% to 20% is proposed in India (Dwivedi, Jain, & Sharma, 2011).

Mesua ferrea L. (MFL) is a timber plant that grows naturally in the northeastern parts of the Himalayan regions of India and in Srilanka. The oil seed contains 55–57 wt.% nonedible, reddish-brown-colored oil (the shelled kernel contains > 75 wt.% oil), which had been traditionally used as fuels (Sarma & Konwer, 2005). It is used in folk medicine for the treatment of fever, dyspepsis and renal diseases (Dennis, Kumar, & Srimannarayana, 1988). Biodiesel (i.e., the fatty acid alkyl ester) can be processed through the conventional tranesterification process. However the most commonly used methanolysis cannot be regarded as a green technology because methanol is mostly derived from the fossil fuel sources. Under such conditions, vegetable oils can be ethanolysed under high pressure, temperature conditions using some renewable catalyst (Zhou, Konar, & Boocock, 2003).
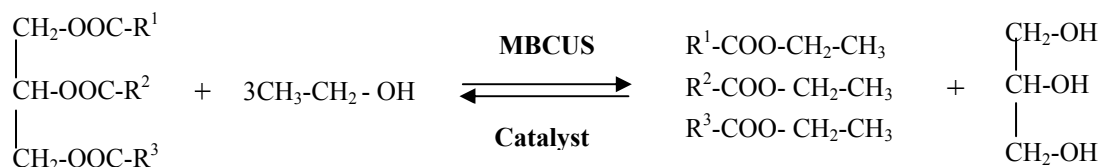
$$
\begin{array}{l}
CH_2\text{-OOC-R}^1 \\
| \\
CH\text{-OOC-R}^2 \quad + \quad 3CH_3\text{-}CH_2\text{ - OH} \\
| \\
CH_2\text{-OOC-R}^3
\end{array}
\quad
\begin{array}{c}
\textbf{MBCUS} \\
\rightleftharpoons \\
\textbf{Catalyst}
\end{array}
\quad
\begin{array}{l}
R^1\text{-COO-}CH_2\text{-}CH_3 \\
R^2\text{-COO- }CH_2\text{-}CH_3 \quad + \\
R^3\text{-COO- }CH_2\text{-}CH_3
\end{array}
\quad
\begin{array}{l}
CH_2\text{-OH} \\
| \\
CH\text{-OH} \\
| \\
CH_2\text{-OH}
\end{array}
$$

Figure 1. General tranesterification reaction of MFL to FAEE with MBCUS nano-catalyst

The commonly used homogeneous base catalysts are NaOH, KOH and their alkoxides. Homogeneous alkali-catalyzed transesterification is much faster than acid-catalyzed transesterification (Vicente, Martınez, & Aracil, 2005). However, a large amount of water is needed to transfer the catalysts from the organic phase to a water phase after the reaction. So, it is considerably more costly to separate the catalyst from the produced solution (Ebiura, Echizen, Ishikawa, Murai, & Baba, 2005; Kim et al., 2004; Dorado, Ballesteros, Lopez, & Mittelbach, 2004).

Heterogeneous base catalysts have many advantages: they are noncorrosive, environmentally benign and present fewer disposal problems. Meanwhile, they are much more easily separated from the liquid products and can be designed to give higher activity, selectivity and longer catalyst lifetimes (Gryglewicz, 1999; Tanabe & Holderich, 1999).

Musa balbisiana Colla underground stem (MBCUS) derived ash has been employed as a heterogeneous catalyst in this work for the first time for ethanolysis of MFL seed oil, which was also covered recently for methanolysis of Jatropha oil having high percentage of free fatty acids (Sarma, Kumar, Aslam, & Chouhan, 2014).

Thus a single step transesterification process with ethanol in a high pressure- high temperature (HPHT) batch process under various temperature ranges has been refined and discussed in this study. Moreover, the product obtained has been analysed for different fuel properties as envisaged for biodiesel as per ASTM standards. The details of the catalyst characterization and use during transesterification of Mesua Ferrea L oil having high percentage of free fatty acids have been expounded in the articles.

2. Experimental

2.1 Materials

Mesua ferrea Linn. (A national tree of Sri Lanka) seeds was procured from the Assam (India) and the seeds were transported to the Institute for physico-chemical analysis and further studies.

MBCUS was employed as catalysts. Petroleum ether -60–80 °C AR (Loba Chemie),Chloroform (Merck, 99.8%), Ethanol (Merck, 99.9%), Methanol extra pure (Loba Chemie, 99.5%), Iodine mono chloride/Wijs Solution (Merck), Phenolphthalein (Himedia, 98%), Potassium hydroxide pellets GR (Merck, 85% minimum), Sodium thio sulphate penta hydrate GR (Merck, 100%), Potassium iodide AR (Himedia, 99.8%), Sodium sulphate anhydrous AR (Himedia, 99%), Starch soluble AR (Himedia, 99%), Hexane GC grade (Sigma Aldrich), Heptane GC grade (Sigma Aldrich, 99.5%), Methanol GC grade (Sigma Aldrich), Methyl heptadecanoate GC grade (Merck), Molecular sieve etc. were used as AR grade chemicals in this study.

2.2 Feedstock Characterization

Mesua ferrea L. (MFL) seed kernels were ground, using a mechanical grinder, and extracted in a soxhlet apparatus, using petroleum ether (boiling point 60–80 °C). The extracted lipid was obtained by filtering the solvent-lipid mixture to get rid of the solid from solvent before the petroleum ether was removed using rotary evaporator 45–50 °C and 2.25×10^3 Pa. The extracted seed oil was stored in freezer at -2 °C for subsequent physicochemical analysis. The fatty acid profile of the oil was determined from GC analysis at PBTI, Mohali, India; an NABL accredited laboratory as shown in Table 1. The morphology of MFL seed oil was represented in the Figure 2.

Table 1. Fatty acid profile of *Mesua ferrea* L. seed oil

Saturated Fatty Acids

S. No.	Common Name of Fatty Acids	IUPAC/Systematic Name	Lipid No. (C:D)	Molecular Formula	Fatty Acid (%)
1	Caproic Acid	Hexanoic acid	6:0	$C_6H_{12}O_2$	0.022
2	Myristic Acid	Tetradecanoic acid	14:0	$C_{14}H_{28}O_2$	0.047
3	Palmitic Acid	Hexadecanoic acid	16:0	$C_{16}H_{32}O_2$	13.635
4	Heptadecanoic Acid	Heptadecanoic acid	17:0	$C_{17}H_{34}O_2$	0.046
5	Stearic Acid	Octadecanoic acid	18:0	$C_{18}H_{36}O_2$	13.646
6	Arachidic Acid	Icosanoic acid	20:0	$C_{20}H_{40}O_2$	1.3
7	Behenic Acid	Docosanoic acid	22:0	$C_{22}H_{44}O_2$	0.351
8	Lignoceric Acid	Tetracosanoic acid	24:0	$C_{24}H_{48}O_2$	0.186

Mono Unsaturated Fatty Acids

S. No.	Common Name of Fatty Acids	IUPAC/Systematic Name	Lipid No. (C:D)	Molecular Formula	Fatty Acid (%)
9	Palmetoleic Acid	Hexadec-9-enoic acid	16:1	$C_{16}H_{30}O_2$	0.087
10	Oleic Acid	(9Z)-Octadec-9-enoic acid	18:1	$C_{18}H_{34}O_2$	50.713
11	Cis-11-Eicosenoic Acid	(Z)-Eicos-11-enoic acid	20:1	$C_{20}H_{38}O_2$	0.18

Poly Unsaturated Fatty Acids

S. No.	Common Name of Fatty Acids	IUPAC/Systematic Name	Lipid No. (C:D)	Molecular Formula	Fatty Acid (%)
12	Linoleic Acid	(9Z,12Z)-9,12-Octadecadienoic acid	18:2	$C_{18}H_{32}O_2$	19.47
13	Linolenic Acid	all-cis-6,9,12-octadecatrienoic acid	18:3	$C_{18}H_{30}O_2$	0.316

Summarized Fatty Acids Analysis

Total Saturated Fatty Acids (%)	29.233
Total Unsaturated Fatty Acids (%)	70.766
Grand Total	**99.999**
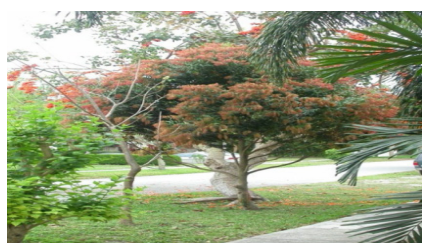
MFL Tree (National Tree of Sri Lanka)

MFL Leaves

MFL Seeds

MFL Kernels

Figure 2. Morphology of *Mesua ferrea* L. (MFL)

The acid value of the oil and biodiesel was measured as per AOCS official method Ca 5a–40 as stated in Equation (1). The chemicals required for titration were freshly prepared caustic KOH (0.25 N) and phenolphthalein indicator (1% in 95% ethanol). The percentage of free fatty acids was calculated as oleic acid using Equation (1).

$$Free\ Fatty\ Acids\ of\ Oleic\ \% = \frac{ml\ of\ Alkali \times N \times 56.1}{wt\ of\ Sample} \tag{1}$$

The acidity (AD) of the crude MFL was 9.4% with an acid value (AV) 18.8 mg KOH/g. For transesterification reaction analytical grade methanol (purity 99.5%, Loba Chemicals) and ethanol (99.9%, Merck) were used without further treatment.

The chemicals required for iodine value estimation were purchased from Merck and prepared as follows: reagent grade iodine monochloride solution, potassium iodide solution (15%), sodium thiosulphate (0.1 N), glacial acetic acid, chloroform and starch (1%). The Iodine values of the oil and biodiesel were estimated as per AOCS official method Cd 1–25 using freshly prepared Wijs solution and starch indicator from the Equation (2).

$$The\ Iodine\ Value = \frac{[(B-S) \times N \times 12.69]}{Weight\ of\ Sample} \tag{2}$$

Where B = titration of blank; S = titration of sample; N = normality of sodium thiosulphate solution.

2.3 Catalyst Preparation

MBCUS was cut into thin pieces of the dimension 1.5 cm × 15 cm and sun dried in March, 2011 for about 15 days, which were later burnt traditionally in an open environment upon a tin sheet for ash production. These ashes obtained were further processed in a furnace in the laboratory at 550 °C for one hour. For material balance, a piece of the fresh MBCUS was collected at sampling plastic bag of known weight and studied for proximate analysis. It was found that the fresh MBCUS contain 91.34% moisture at 105 °C, 7.45% volatile matter at 350 °C and 0.14% carbon at 550 °C. While the ash obtained at 550 °C was 1.07% of the fresh MBCUS (Figure 3).
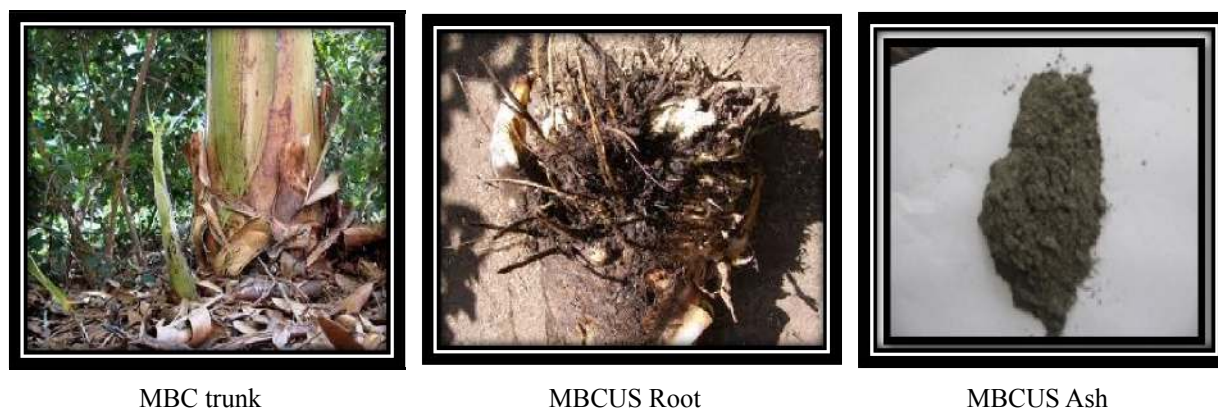

MBC trunk   MBCUS Root   MBCUS Ash

Figure 3. Morphology of MBCUS catalyst

2.4 Catalyst Characterization

The physico-chemical characteristics of the MBCUS ash (Baked at 550 °C) were analysed to estimate the catalytic activity and basic composition as per AOAC methods. The catalyst was analysed in a TGA instrument manufactured by Perkin Elmer, model STA 6000 for thermal and oxidative stability. The weight losses of the catalyst (prepared @ 550 °C), temperature range 30–1000 °C, heating rate 10 °C/min and constant flow of air and nitrogen (20 ± 0.5 ml/min) were recorded. The contents of the catalyst were ascertained from appropriate AOAC, TGA, XRF analysis and SEM EDX pattern. The basic strength of the catalysts (H-) was estimated using Hammett indicator method. The catalyst sample (100 mg) was shaken along with suitable amount of Hammett indicators diluted with methanol and left to equilibrate for about two hours upto no colour change observed. The titrate color was then recorded. The following Hammett indicators were used: neutral red (H- = 6.8), bromothymol blue (H- = 7.2), phenolphthalein (H- = 9.3), 2,4-dinitroaniline (H- = 15.0), 4-nitroaniline (H- =

18.4). The basic strength of the catalysts was defined as being stronger than the Hammett indicator if it showed a colour change but weaker if no colour changes observed.

The structural study of the catalyst was done with the XRD pattern using X-ray diffractometer (XRD). Nitrogen adsorption isotherms were obtained at -196 °C using a Quantachrome NovaWin - Data Acquisition and Reduction (Quanta chrome Instrument) system. These isotherms were used to see the BET surface area, pore volume and pore size distribution.

The SEM images and subsequent EDX analysis of the samples were carried out using FE-SEM QUANTA 200 FEG of FEI Netherlands make. The EDX pattern showed the composition and different crystalline materials present in the MBCUS ash catalyst. The transmission electron microscope images of the MBCUS samples were taken using a Tecnai G2 20 of FEI Neitherlands make transmission electron microscope (TEM).

The nature, composition and detail physicochemical properties of the MBCUS nano-catalyst have been accepted for publication in a reputed journal recently (Sarma, Kumar, Aslam, & Chouhan, 2014).

2.5 Transesterification in Batch Reactor

The tranesterification reactions were carried out in a HPHT Bench Top reactor of 1.8 L capacity (Amar equipments Company, Mumbai, India). The reactor contains to heating mantle, cooling system and pressure-temperature control and recording devices.

The average molecular weight was calculated from the triglyceride composition of MFL seed oil and was found to be 866. The oil to alcohol molar ratio in the reaction mixture was kept at 1:9 and the catalyst concentration was kept at 1%, 3% and 5% of the initial oil mass. The reaction was carried out under 250 °C and 275 °C for easy conversion of the free fatty acids to ethyl ester and also for resisting, oxidation of the reactants. This was accomplished by stripping the reaction mixture inside the reactor. All the experiments were performed with a total oil, alcohol (ethanol) and catalyst volume not exceeding 1.5 L. The initial hydrogen pressure inside the reactor was maintained at 1.0×10^5 Pa (atmospheric) which rises up to 4.4×10^6 Pa with increasing internal vapour pressure (IP) at 275 °C. The reactions were also conducted in the absence of catalyst under the same set of experimental condition. In this instance, the internal vapour pressure raised up to 6.6×10^6 Pa at 275 °C. The mixture was vigorously stirred at 650 revolutions per minutes. Samples were removed by opening the flash bottom valve after completion of the reaction. The samples collected from the reactor were allowed to settle in a ventilator before analysis. Since triglycerides (TGs), diglycerides (DGs), monoglycerides (MGs) and free fatty acids (FFAs) analysis relates only with the oily liquid phase of the sample and the results are presented as percentage of the total oily mass, the oily mass was purged of impurities that might affect the final measurements. So, the oily phase was subsequently transferred to the rotary vacuum evaporator (Heydolph, Germany Make) and the unreacted alcohol was separated in 45–50 °C and 2.25×10^3 Pa. Then, the samples were centrifuged to separate the catalyst, glycerine (GL) and oily phase. The upper layer of the centrifuged sample contained the oily phase (biodiesel), the lower one of the glycerine, while in the middle, between the oil and the glycerine phase appears the catalyst. The catalyst was later washed using petroleum ether and methanol to remove the bound ester and glycerol.

2.6 Fuel Property Analysis of the FAEE

The crude MFL seed oil was initially analyzed using GC FID detector as per AOCS Official method 1998, Ce 1–62 and Ce 2–26 at Punjab Biotechnology Incubator (Mohali) an NABL accredited laboratory of India. The fuel properties of the FAEE obtained at optimum conditions were analysed using appropriate ASTM D6751 and EN 14214 standard procedures specified for B100 biodiesel stocks.

3. Results and Discussion

3.1 Structural Analysis and Porosity Analysis of MBCUS Catalyst

The XRD analysis ascertains the presence of crystalline phases the in MBCUS catalyst, which have been reported earlier (Sarma, Kumar, Aslam, & Chouhan, 2014). It has been found that the surface area of the catalyst is 38.710 m^2/g, total pore volume 4.233×10^{-8} m^3 /g for pores smaller than 796.2×10^{-11} m (Radius) at P/P_o equal to 0.98780 and pore size expressed as average pore radius is equal to 21.8681×10^{-11} m. These values are significantly better than earlier reported literature (Sharma, Khan, Puri, & Tuli, 2012; Kumar et al., 2010). All these attributes show that the catalyst chosen could be an excellent one for transesterification reaction.

3.2 SEM-EDX and TEM Analysis of MBCUS Catalyst

The SEM-EDX analysis of the catalyst were conducted (Figure 4) and confirmed the presence of C ($CaCO_3$), O (SiO_2), Mg (MgO), Al (Al_2O_3), Si (SiO_2), P (GaP), Cl (KCl), K (MAD-10 Feldspar), CA (Wollastonite), as

depicted in the Figure 4. The typical elemental composition as evaluated from Figure 4, that depicts the mass fraction composition as C (9.86%), O (44.01%), Mg (3.92%), Al (1.21%), Si (12.03%), P (1.59%), Cl (2.69%), K (16.83%), Ca (7.19%). Earlier studies of heterogeneously catalysed tranesterification reactions of vegetable oil have focused on single metal oxides, including CaO, MgO, Al_2O_3, zeolites, silicates etc.

Functionalized compounds such as calcium bentonite functionalised with potassium (Hoffmann & Conradi, 1988), porous silica and pumice granules, mesoporous silica and metallosilicate materials of SBA-15 types (Sanz et al., 2012) were also shown very good activity during tranesterification as reported. Therefore, the versatile composition, including several alkali and alkaline earth metal oxides, carbonate and silica makes MBCUS ash a perfect heterogeneous catalyst for transesterification reaction (Sarma, Kumar, Aslam, & Chouhan, 2014).
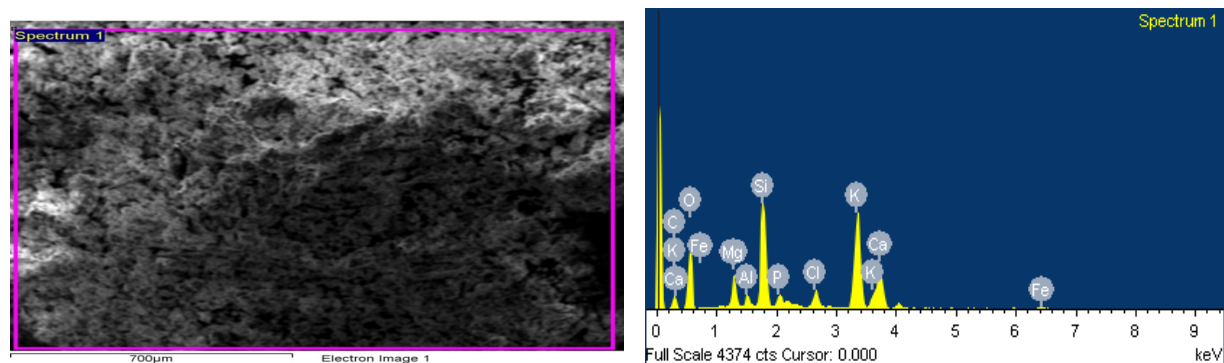
Figure 4. SEM-EDX spectrum and composition

The structural information of the catalyst was studied from the TEM images as shown in the Figure 5. It is clearly visible that the structural dimensions are similar (100 nm length) and brick like (Approx. 70 nm × 20 nm × 15 nm). The nano size structural dimension of MBCUS catalyst provided effective surfaces to their catalytic efficacy during tranesterification of triglyceride, irrespective of the free fatty acids present in the oil. Similar results were also covered by Boz, Degirmanbasi and Kalyon (2009) for the tranesterification of canola oil using nano-γ-Al_2O_3 loaded with KF that showed 97.7% ester yield with 3% catalyst loading.
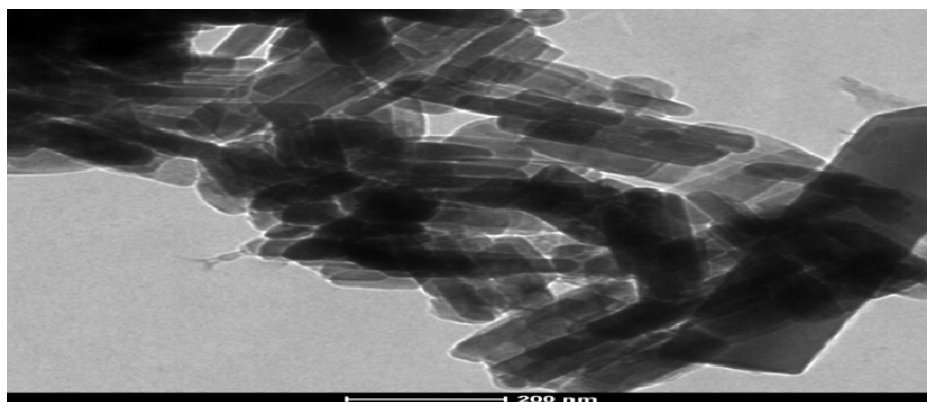

Figure 5. TEM images and structural information of MBCUS-catalyst

3.3 Basicity Analysis of MBCUS Catalyst

The basicity of the catalyst was determined within the range of $9.3 < H^- < 15.0$. The base strength of MBCUS is thus stronger than the weakest indicator phenolphthalein ($H^- = 9.8$) that exhibited a colour change from pink to colourless. However, it is weaker than the strongest indicator i.e. 2, 4-dinitroaniline ($H^- = 15.0$) as no colour change was observed.

3.4 Metallic & Non-Metallic Contents in MBCUS Catalyst

Nonmetallic concentration in the MBCUS ash catalyst was found out using appropriate standard methods as applicable discussed in Table 2.

Table 2. Metallic & non-metallic contents in MBCUS catalyst

S. No.	Parameters	Concentration	Specification
1	Chloride	0.60 wt.%	AOAC 915.01
2	Carbonate	ND(MDL: 2 mg/kg)	
3	Sodium	0.34 wt.%	AOAC 975.03
4	Potassium	27.70 wt.%	
5	Lead	5.19 mg/kg	
6	Cobalt	1.97 mg/kg	
7	Carbon	4. 5 wt. %	
8	Silica (various forms)	37.00 wt.%	TGA method

3.5 Product Analysis by GC

Important Physico-Chemical Characteristics of MFL seed oil have been represented in Table 3. The process for the production was optimized with regard to the conversion of the oil to ester @ 250 °C and @ 275 °C. The biodiesel was produced from MFL seed oil under the specified experimental condition and found in conformity to ASTM standards with regard to fuel property while the reaction was carried out at 275 °C. It has been observed that the acid value of MFL oil was 18.8 mg KOH/g oil (which is equivalent to about 9.4 wt.% FFA content), the acid value of the end product was 1.6 mg KOH/g oil (Figure 6). This has been observed even without externally applied hydrogen pressure. The experiment was repeated further for MFL oil at 275 °C without H_2 pressure. The tranesterification reaction was carried out under 250 °C and 275 °C for the conversion of FFAs and triglycerides to fatty acid ethyl-ester. This was accomplished by stripping the reaction mixture inside the reactor. All the experiments were performed with a total oil, alcohol (ethanol) and catalyst volume not exceeding 1.5 L. The initial hydrogen pressure inside the reactor was maintained at 1.0×10^5 Pa (atmospheric) which raises up to 4.4×10^6 Pa during the reaction along with increasing internal vapour pressure (IP) at 275 °C due to the presence of ethanol. The reactions were also conducted in the absence of catalyst under the same set of experimental condition. In this instance, the internal vapour pressure raised up to 6.6×10^6 Pa at 275 °C. This clearly demonstrated that the presence of hydrogen does not affect tranesterification.

Table 3. Physico-chemical characteristics of *Mesua ferrea* L. seed oil

Parameters	MFL Oil	MFL–FAEE (B100)
Density at 40 °C (g/cm^3)	0.90059	0.873
Specific gravity at 20 °C (g/cm^3)	0.90762	0.879
API density at 15 °C (g/cm^3)	0.91735	0.890
Distillation @ 360 °C (wt%)	--	90
Flash point (°C)	115.0	113
Calorific value (MJ kg^{-1})	36.0	35
Viscosity @ 40 °C (cSt)	34.10	5.525
Acid Value (mg KOH/g)	18.8	1.8
Free fatty acids (%) as oleic	9.40	0.8
Iodine Value (g I$_2$/100g)	131.31	113.24
Rams bottom carbon residue (wt%)	0.99	0.250
Copper strip corrosion	1a	1a

The iodine value decreases with increasing the catalysts percentage with respect to biodiesel production. It was observed that the unsaturation in the biodiesel decreased as iodine values decreased from 130 to 112 as shown in Figure 7. Iodine number basically represents the degree of unsaturation, the larger is the number higher is the unsaturation. While tranesterification took place some of the unsaturation present in the fatty acid chain got saturated with the proton shift that takes place in situ. This also indirectly indicated the conversion of oil to FAEE.
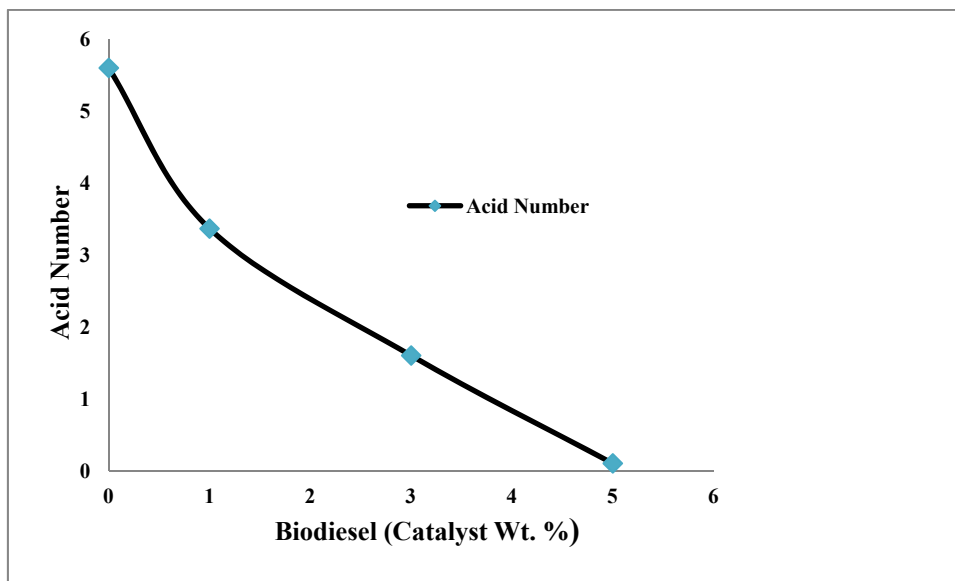
Figure 6. FFA trend using MBCUS
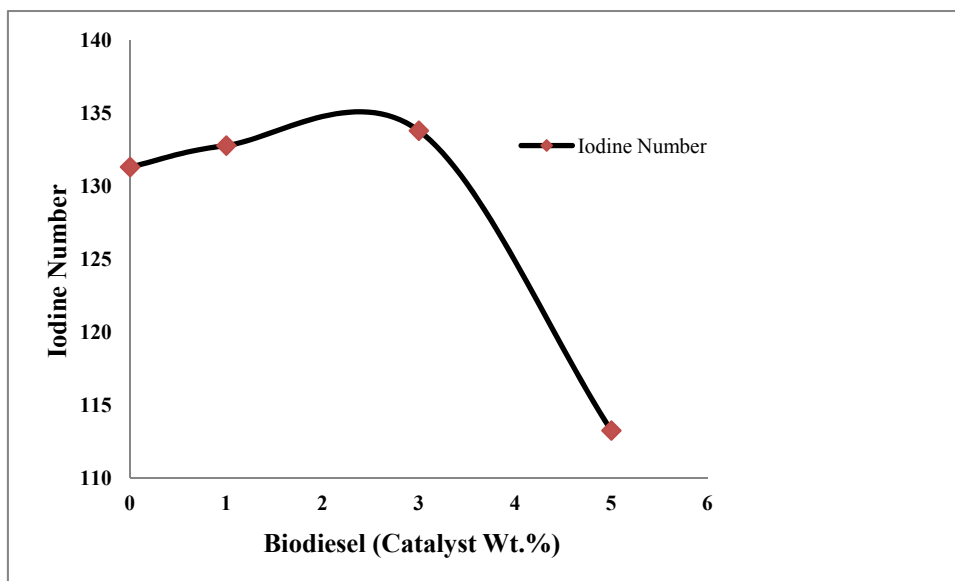
Figure 7. Iodine index variation MF oil to FAEE

It has been observed that the FFA level drastically decreased to near about 1.8 mg KOH/g from 18.8 mg KOH/g oil in 1 h. This clearly demonstrated the evidence of efficacy of the catalysts and furthers no effect of hydrogen pressure upon FFA conversion at higher temperature. MBCUS is a very good catalyst due to high reactivity and efficacy during the transesterification and the negligible cost of production. The catalyst and product separation were much easier as compared to other available methods (two step transesterification). MBCUS catalysts can be commercialized for the bio-diesel production for the development of society and economic growth of the Country as well.

The TGA profile of the FAEE was conducted under a constant flow of N_2 20 ml/min in 1 h constant heating rate and observed that over 90% of the FAEE was boiled off at 350 °C confirming the conversion of MFL to FAEE (Figure 8). The GC profile of the FAEE of MFL was recorded and found that 95% of the oil was converted to FAEE at 275 °C and one hour reaction time in a batch process in 1:9 molar ratio of oil: alcohol as specified in Figure 9 in the absence of H_2 pressure. The same was also confirmed from the vacuum distillation of the FAEE obtained and 90% recovery was found at 360 °C as per ASTM D 6751-07 b. The distillation characteristics are regarded as the most important parameter of any liquid fuel, which represents volatility during combustion and formation of proper air-fuel mixture. Both the Gas chromatograph profile and distillation characteristics, are therefore in accordance with the specified standards of the FAEE and can be regarded as a very safe fuel for our purpose.

The MBCUS nano - catalyst could be reused for three times with the reduction of conversion efficacy of oil to FAEE by about 20%, which was reported in our previous publication during tranesterification of Jatropha oil to FAME (Sarma, Kumar, Aslam, & Chouhan, 2014).
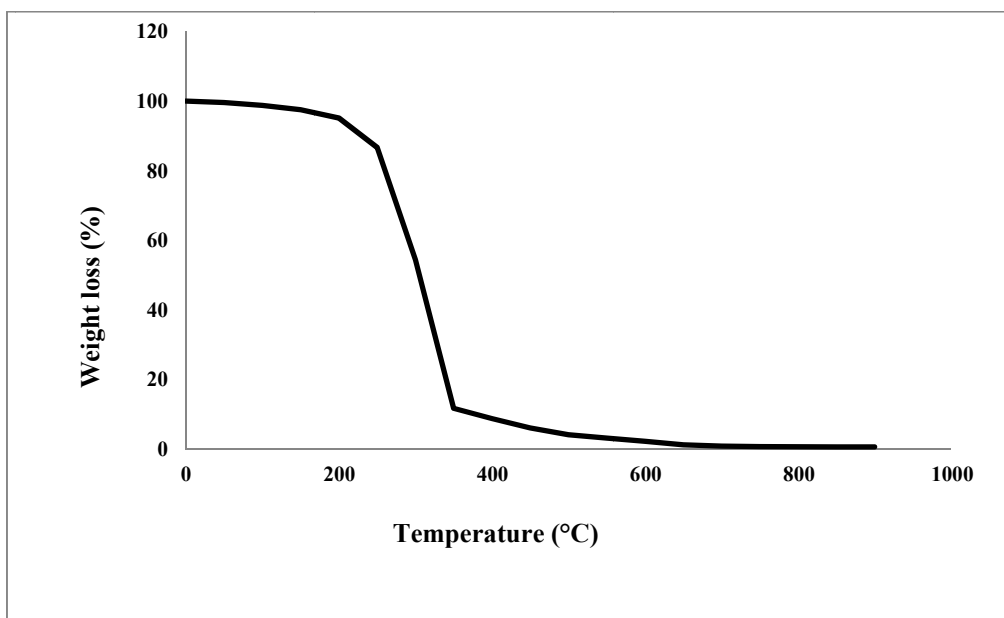

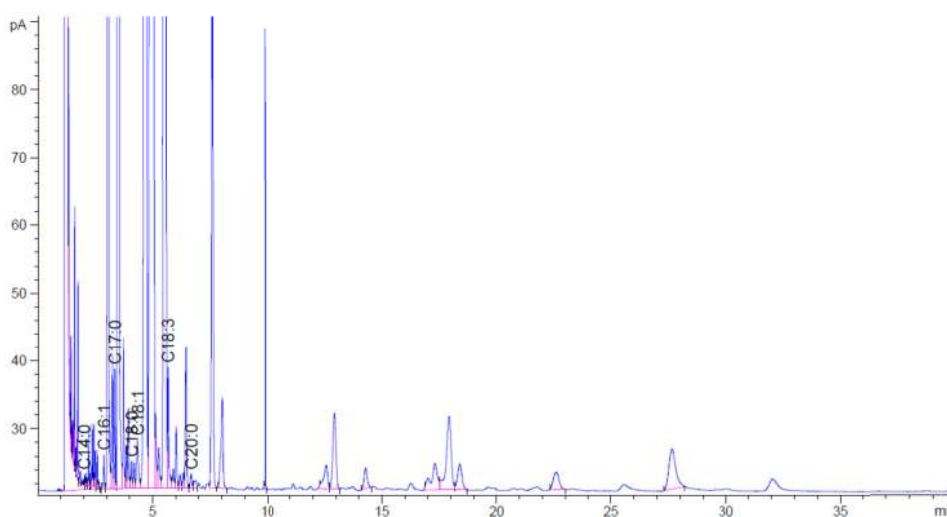Figure 8. TGA profile of the MF FAEE obtained

Figure 9. GC Profile of the MF FAEE obtained

4. Conclusion

High free fatty acid containing MFL seed oil could be transesterified with ethanol (1:9 M ratio) in a single batch process using 5 wt.% MBCUS and l with or without external pressure in closed reactors.The vapour pressure inside the reactor increased upto 3.8 MPa even without external pressure. The experiment was not conducted beyond 300 °C because at that stage poly unsaturated fatty acids would have been cracked when the reaction time exceeds 15 min. The MBCUS nano-catalyst performed well during the transesterification reaction with good reactivity and reusability. This technology is applicable to most types of oil having high percentage of free fatty acids. The industrialist may take an interest in using such materials while setting up biodiesel production, refinery in the near future for all types of high free fatty acid containing oil.

Acknowledgements

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