Musa Champa Fruit Peel Ash: A Renewable And Highly Effective Heterogeneous Base Catalyst For Biodiesel Production From Linum Usitatissimum (Linn.) (Flaxseed) Oil As A Source

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ABSTRACT

The use of a biomass-derived catalyst for bio-diesel production has gained attention today. The heterogeneous catalyst derived from the fruit peel of Musa champa fruit, a waste biomass material, was a highly efficacious catalyst for biodiesel manufacture. The present work reveals catalytic activities of varying catalyst loading; prepared from the fruit peel of Musa champa and applied in biodiesel production from Linum usitatissimum (Linn.) oil, commonly known as flax seed. The ash of Musa champa (MCA) fruit peel, which has a good content of potassium, demonstratedthe greatest level of catalytic activity. The catalyst has been characterized by FTIR, FESEM, and TGA analysis. Conversion of oil to bio-diesel was confirmed by NMR and GC-MS techniques. This low-cost and environmentally benign catalyst claims a novel procedure for the manufacture of bio-diesel from Linum usitatissimum (Linn.) oil.

Keywords: Linum usitatissimum (Linn.) oil, bio-diesel, heterogeneous catalyst, Musa champa.

1. INTRODUCTION

The intensifying demand coupled with the fading supply and environmental pollution associated with the usage of fossil fuels has directed the search for possible eco-friendly sources of energy. A huge percentage of the world energy consumption and the energy required in variegated sectors rely on the fossil fuels whose consumption stands responsible for severe environmental pollution by increasing the concentration of CO_2 concentration in the atmosphere leading finally to global warming [1]. About 80 % of air pollutants get released due to the combustion of fossil fuel and coal which generates polycyclic aromatic hydrocarbons, particulate matters, carbon dioxide, sulfur oxides (SOx) and nitrogen oxide (NOx) [2-3]. Also, the world's accessible petroleum reservoirs are undergoing depletion because of the escalated rate of consumption. Therefore, the scenario's current need is to satisfy the rising energy demands and the elevated CO_2 emissions, which raise concerns about how to use renewable resources to limit the rate of global warming in a sustainable and friendly manner. Despite this, there are numerous more renewable energy sources that could meet energy needs and reduce greenhouse gas emissions. But in view of the existing infrastructures, biodiesel grabs the position as one of the most promising and easily implementable alternatives to conventional diesel [1]. Biodiesel is a transesterification product, which possesses the potentiality of being a sustainable, renewable, biodegradable, renewable, low toxic and low sulfur content alternative renewable source of energy. It holds similar functional properties as that of petro-diesel and also emits lower pollutant gases which can be blended with fossil diesel for its miscibility [4].

Amongst the various methods available for the synthesis of biodiesel including dilution, transesterification, micro emulsion, thermal cracking [5], transesterification is the simplest and best process for getting a better specification of biodiesel from various different edible and non-edible feedstocks, including fat and oil from algae, bacteria, fungi, etc [6]. Biodiesel synthesis, because of its

several special properties and the escalated demand of alternative energy source is expected to rise in the upcoming years or decades. This entails the assurance of the biodiesel quality, degradation of which will lead to severe consequences such as engine malfunction, regardless of the renewable sources. Thus, the validation of the properties of the source oil becomes necessary before selecting it for the synthesis of biodiesel.

Catalysts play a significant role in this reaction under an explicit alcohol to oil ratio and temperature in order to produce an optimum yield of biodiesel [7]. Therefore, developing a suitable low-cost, eco-friendly and recyclable solid catalyst is one of the most vital factors in terms of the low-cost production of biodiesel on a commercial scale [8].

Because of their wide accessibility, higher basic attributes,and growing use in the manufacture of green biodiesel, a diversity of inexpensive well-grounded catalysts have emerged from agro-waste materials in the past decade. Some of the agro-wastes sources which have been used as catalysts in biodiesel production are Heteropanax fragrans [9], cupuaçu (Theobroma grandiflorum) [10], Lemna perpusilla [11], Carica papaya stem [12], orange peel [13] and many others.

As the second-largest fruit in the world and a prominent member of the Musaceae family, bananas serve an important role in horticulture. Bananas are grown in around most of the nations and are the fourth most prime food hervest in the world. Bananas have several species and over 75 species of wild banana are native to regions ranging from humid tropical extending from India to countries in the Pacific [14]. India holds the position as one of the largest producers of banana, thereby contributing about 27 % of the world's banana production [8]. Renowned for their high potassium and fiber content, bananas also provide a number of health advantages, such as preventing colon cancer, relieving constipation, and treating ailments including high blood pressure and muscle cramps. Also, bananas possess characteristics of controlling conditions like muscle cramps and high blood pressure [15]. In addition to their substantial nutritional value, bananas produce a large amount of post-harvest waste, which is gradually recycled into products with added value. The after-harvest portion of the musa plant, which comprises the rhizome, fruit peel, peduncle, and banana leaf sheath, produces a significant amount of organic leftovers that make up roughly a dominant portion of fresh plant mass. These waste residues comprise of natural fibers, which can then be utilized as the raw materials for the production of paper [16]. The banana residues also finds application for the production of bioethanol [17], biochar [18], biomethane [19], and wastewater treatment [20]. Materials produced from banana leftovers have demonstrated their effectiveness in the creation and use of promising and efficient catalysts in the biodiesel synthesis process. A variety of banana species used in Biodiesel production which have been reported include Musa paradisiacal peel [21], Musa acuminata peduncle [22], Musa spp “Pisang Awak” peduncle [23], Musa ‘Gross Michel’ peel [24], Musa acuminata peel [25], Musa balbisiana [26-28], and Musa chinensis [5].

Musa chinensisgrabs its position as an extensively grown commercial banana type in Assam, out of the 15 to 20 varieties produced in India. The banana variety Musa champa, which is a member of the Musaceae family, is also referred to as Chini champa, in local language of Assam. It is a small sized fruit with a thin and dark green peel which takes a golden color when in its fully mature form. It has a dark red flower with a slight sourness and pleasant sweetness in taste. Musa champapeel finds application for the development of a synthetic green catalyst which reported its utilization in the Claisen–Schmidt reaction for the fabrication of chalcone and flavone derivatives [30]. This study aims to explore the utilization of the ash of Musa champa peel as the efficient solid catalyst for the manufacture of biodiesel using the oil extracted from the seeds of flax as the source. In a XRD analysis, B. Basumateryet al. have observed that M. champaash has a high percentage of K_2CO_3, $CaCO_3$, CaO, SiO_2, KCl, K_2O etc; which may enhance its catalytic activity and a good choice for biodiesel synthesis [8].

2. EXPERIMENTAL METHODS

2.1. Chemicals and materials:

Chemicals used in the analysis included silica gel G for TLC, iodine, hexane, petroleum ether, phenolphthalein, aniline, bromothymol blue, ethyl acetate (purity GC ≥ 99.5%), methanol (purity GC ≥ 99.0%), acetone (purity GC ≥ 99.0%), anhydrous sodium sulfate (assay ≥ 99.0%), iodine, petroleum ether, and phenolphthalein: were purchased from Merck, India. The fruit peel of Musa champa, was gathered from Bokajan, Karbi Anglong, India, in order to prepare the catalyst. Hexane was employed as

a solvent in the Soxhlet extraction process to extract Linum usitatissimum(Linn.) oil used in this investigation.

2.2. Preparation and Characterization of the catalyst:

The ripened Musa champa fruit was gathered from the Karbi Anglong district of Assam, which is the study area. The ripe banana peel from Musa champa fruits was gathered and sliced into little pieces. After carefully washing the tiny peel fragments with distilled water, they were allowed to air dry for two to three days. After that, the peels were heated up at 70–80 °C for 24 hours. Then the oven dried banana peels were burnt to generate ash, which was ultimately ground into a powder. The resultant powder was sieved to fine ash to create MCA (Musa Champa Ash). MCA was then stored for later use in a desiccator.

2.3 Procedure for oil extraction from the seed of Linum usitatissimum (Linn.):

It was observed that the yield of oil was good in a non-polar solvent [30]. So, hexane was taken as solvent for the extraction of oil from the seed. Five grams of Linum usitatissimum (Linn.) seed, also referred to as flax seed, were first ground in a mixture grinder before being added to the Soxhlet chamber's thimble. The distillation procedure started after 50 mL of the solvent hexane was put in a round-bottom flask and put together for a Soxhlet extraction, which was heated for approximately 10 hours using a heating mantle. After completing the extraction process, the round bottom flask was removed from the apparatus and the solvent was removed by using a rotary evaporator. The oil was then dried using a vacuum pump and weighed.

2.4 Transesterification of Linum usitatissimum (Linn.) oil:

2.4.1 Optimization of Reaction Parameters:

Linum usitatissimum (Linn.) oil, commonly known as flaxseed oil, was mixed with methanol in the ratio of 1:15 in a 100ml round bottom flask attached to a condenser followed by initial stirring it for 4-5 mins. After that, 100 mg of the MCA catalyst was added, and allowed to stir. Using the TLC plate (solvent system, 1:20 v/v of ethyl acetate and petroleum ether), the reaction was regularly monitored while being continuously stirred to check for the formation of biodiesel. The reaction duration, which was approximately 30 minutes, was also recorded. Using a suction pump and Whatman no. 42 filter paper, the catalyst was filtered out once the reaction was finished. Two separate layers were visible after shaking and letting the filtrate stand in the separating funnel. The upper layer containing biodiesel was separated and collected in a conical flask, initially dried by adding anhydrous Na_2SO_4 and then vacuum dried which was then used for further analysis.

The yield of biodiesel was calculated by using the following equation (Eq. (1)) mentioned by Aleman-Ramirez et al. [37].

$$\text{Yield of biodiesel (\%)} = \frac{\text{Weight of biodiesel}}{\text{Weight of oil taken for reaction}} \times 100\ \% \text{----------(1)}$$

2.4 Characterization and Property Determination

^{1}H NMR and GC-MS studies were used to determine the formation and purity of the synthesised fatty acid methyl ester, usually known as FAME product. ^{1}H NMR analysis data for the flaxseed oil and the synthesized biodiesel was obtained on Bruker AVANCE NEO NMR SPECT. using internal reference as tetramethylsilane (TMS) and DMSO solvent at 400 MHz. To determine the methyl ester compositions of biodiesel; Perkin Elmer Claurus 680 Gas Chromatograph/600C Mass spectrometer was used. For this analysis, the starting temperature was kept at 60 ◦C for 1 min and after that it was raised to the temperature to 200 ◦C at the rate of 7 ◦C /min. After that, it was raised to 300 ◦C at a rate of 10 ◦C per minute. After maintaining the temperature at 250 °C, the sample was put in and ran. The GC column was Elite 35 MS, measuring 60.0 m × 250 micrometers, and the mass scan ranged from 50 to 600 Da. Helium was utilized as the carrier gas, splitting in a 20:1 ratio at a flow rate of 1 mL/min. 160°C was the transmission temperature, and 150°C was the source temperature. The physicochemical properties such as density, kinematic viscosity, total sulfur, water content of the synthesized biodiesel from flaxseed oil were tested at the laboratory of IOCL, Bongaigaon Refinery, India. The biodiesel's additional properties, including its cetane number, iodine value (IV), saponification number (SN), higher heating value, American Petroleum Index, Diesel Index, and aniline point, have been evaluated. Amongst the mentioned properties, Diesel index, higher heating value (HHV), API (American petroleum index) and aniline point were calculated using the ASTM D2015 standard equation [33-34].

2.5 Effect of the reaction parameters on biodiesel synthesis:

2.5.1 Reusability of Catalyst

When the catalyst's reusability was examined, 10 wt% of it was first loaded in a 1:15 oil/methanol ratio in accordance with the ideal reaction conditions, which was allowed for a 30 min reaction time. Petroleum ether and methanol were used to wash the catalyst in order to remove any contaminants that might have been present. To get rid of any leftover products, glycerol, and other contaminants, the recovered catalyst was rinsed many times with acetone. After being cleaned, the catalyst was placed on a petri dish and left to dry for four hours at 100 °C in a hot air oven. The dried catalyst was then utilised in the next reaction after being cooled in a desiccator; this procedure was followed for every cycle of the reaction. A reduced percentage of conversion of oil to FAME product was observed with the reused catalyst. After the 4th run, only approximately 40% of FAME conversion was observed (Figure 1). The reduced activity of the catalyst may be due to the leaching process of some of the elements, which may cause loss of the active site of the catalyst [1].

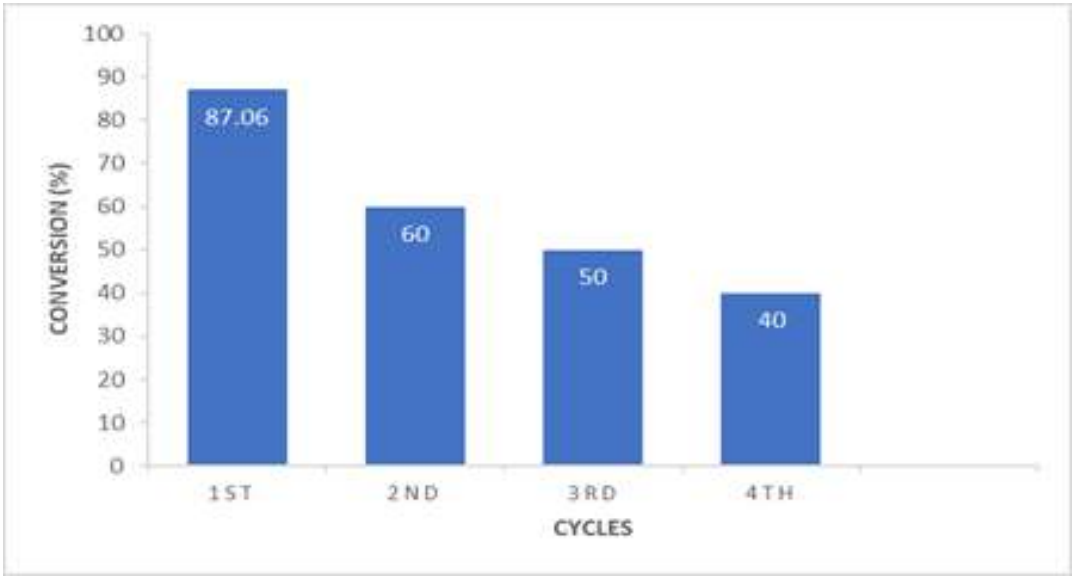

Figure 1: Reusability of the catalyst on transformation of oil to FAME

Figure 1 shows the findings of reusability investigations, which showed a progressive decline in catalytic activity for each reaction cycle. With a considerable increase in reaction time, the biodiesel production dropped from 87.06% (fresh reaction) to 40% (4th cycle).

2.5.2: Efficiency of Catalyst and Oil Methanol Ratio:

The reaction optimization was carried out with different catalyst loading in weight percentage of oil in order to investigate the impact of the catalyst MCA (5, 8, 11, 15 and 20 %,) (Figure 2(a)) using different methanol to oil ratio (in mL) (3:1, 6:1, 9:1, 12:1, 15:1 and 18:1) (Figure 2(b) at different temperatures (35, 45, 55, 65, and 75 ◦C) Figure 2(c). The catalytic efficacies of the burnt MCA were also investigated and reported at optimal reaction conditions (ORCs) following the attainment of the (ORCs). The reduced conversion achieved with catalyst loading of 5 and 8 weight percent of the oil may be due to poor availability of basic sites. It was found that when the reaction was run with a 15 weight percent catalyst loading, the maximum conversion of 100% was achieved; after that, the conversion stayed constant. The high density of basic sites in the material could be the reason for the high conversion [31]. A certain amount of FAME yield is increased by increasing catalyst loading, however after the ideal level of catalyst loading, the FAME yield essentially stays constant [32].

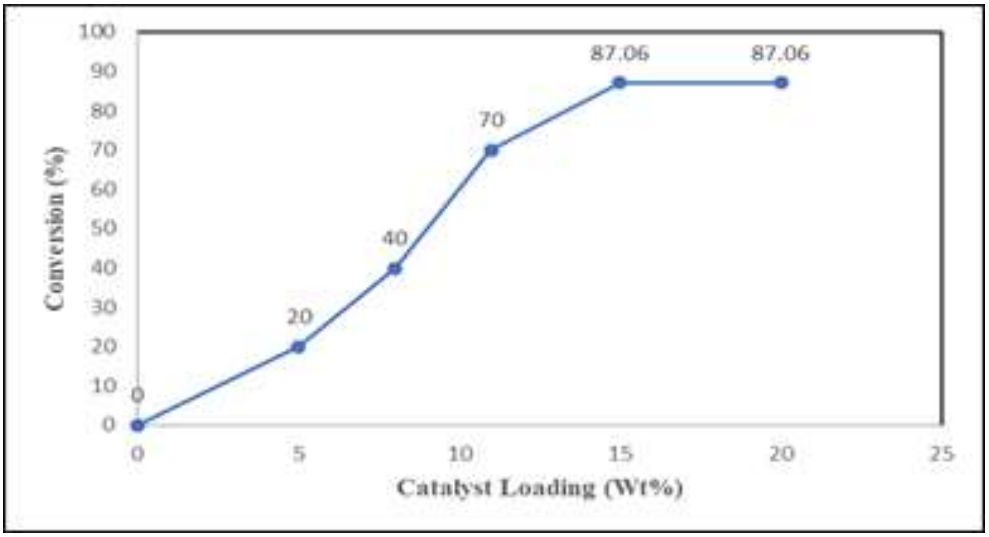

Figure 2(a) Effect of catalyst by wt% of oil

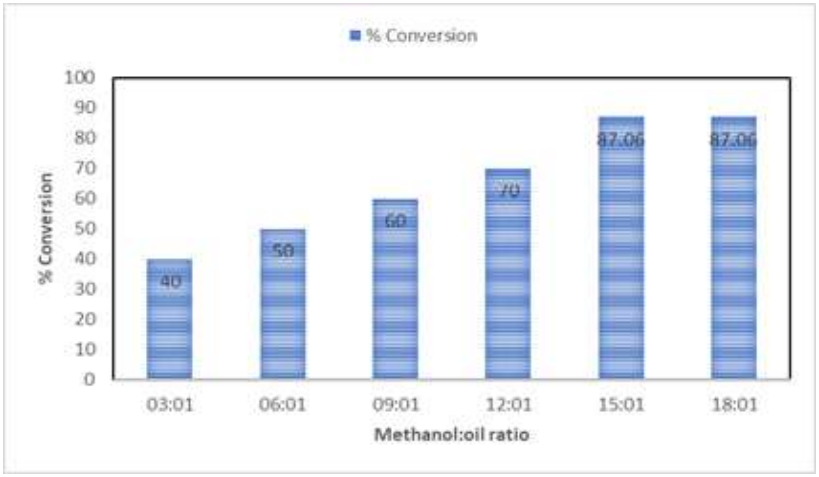

Figure 2(b) Effect of methanol to oil molar ratio on conversion of oil.

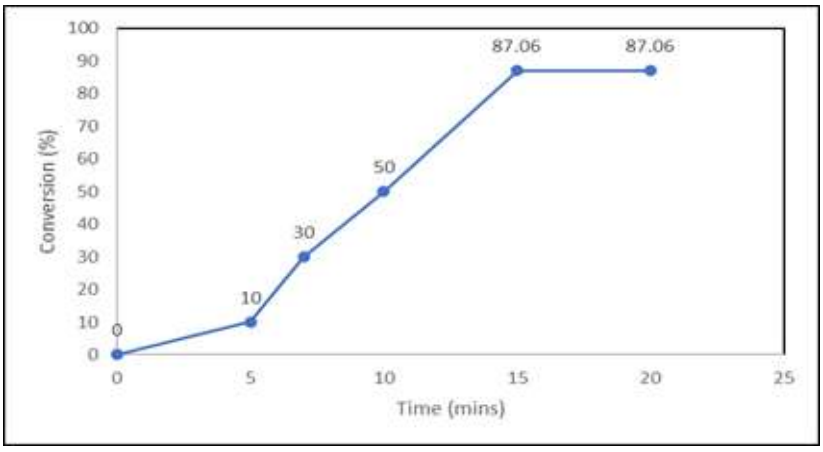

Figure 2(c): Effect of reaction time on conversion.

3. RESULTS AND DISCUSSION

3.1 Characterization of the Synthesized Ash Catalyst:

Characterization of the fabricated ash catalyst was accomplished using IR, SEM, and TGA techniques. KBr pellet in a Shimadzu FTIR-8400S spectrophotometer was used to record the IR spectrum of the catalyst in the range of 4000–400 cm^{-1}. FESEM studies(using Carl Zeiss FESEM instrument) was employed to reveal the surface profile of the ash catalyst. The weight loss(in percentage) and the thermal stability of the ash was determined through TGA analysis using a thermogravimetric analyzer (Make: Mettler Toledo,TGA2).

3.1. Characterization of Catalyst

3.1.1. Thermogravimetric Analysis

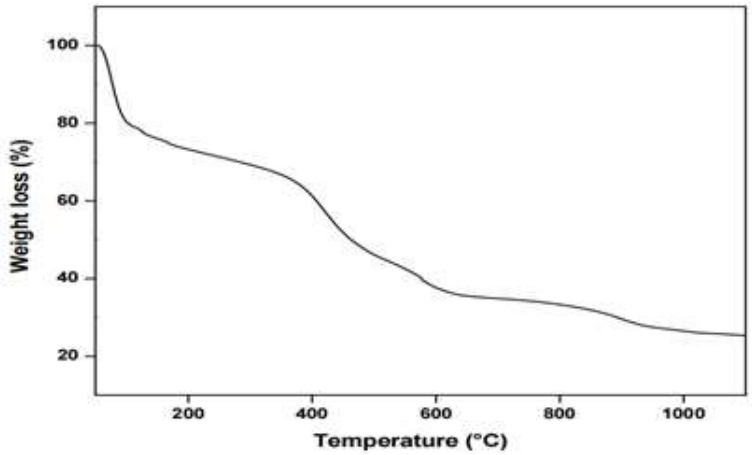

Figure3: TGA thermograms of MCA

Thermogravimetric (TGA) analyses of the ash of MCA catalysts which was obtained from the TGA curve as shown in Figure 3. During the heat treatment as measured from the curve, the gradual weight losses were observed at three phases. The heating upto the range of 150-180 degree celsius, showed the loss of weight which can be attributed to the evaporation of the adsorbed moisture on the catalyst's surface. The carbonaceous materials when decomposes releases CO and CO_2, which was evident from the another weight loss observed at the temperature beyond 450 °C. More concretely, the metal carbonates presence in the material show up their decomposition commencing from this temperature.

3.1.2 FESEM Analysis

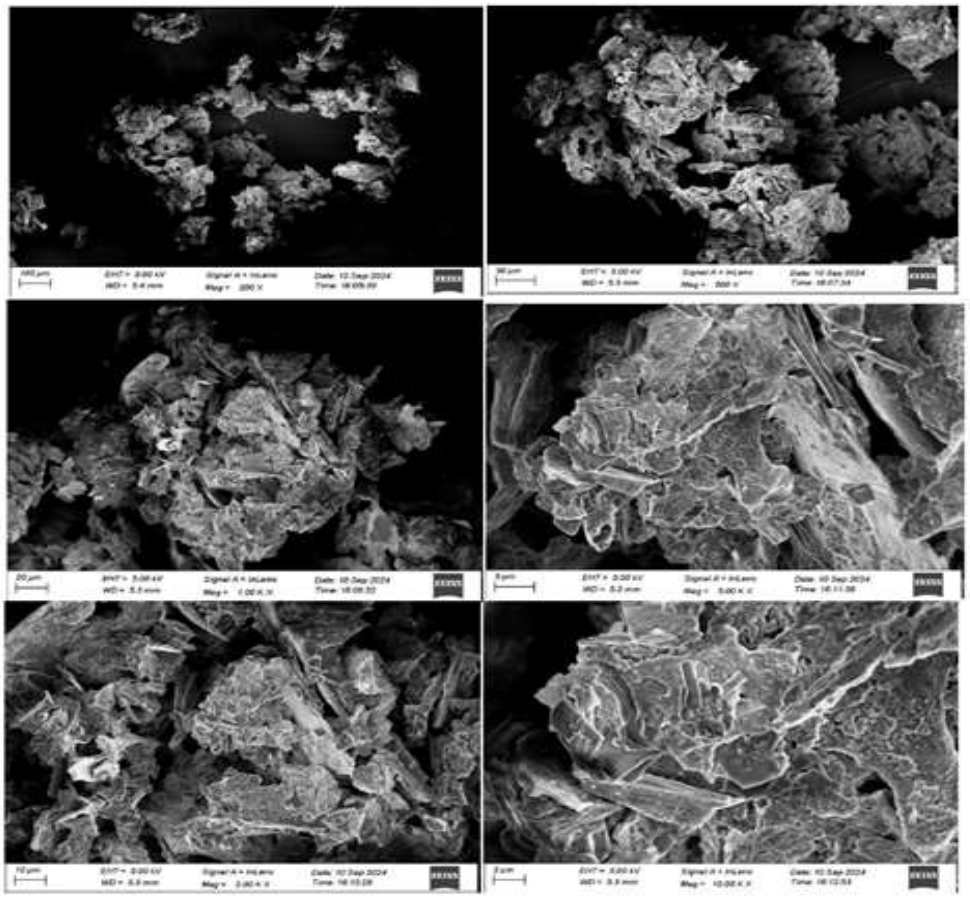

Figure 4: FESEM images of the catalyst

The external morphology of the catalyst was assessed through the FESEM analysis as in Figure 4. The images as observed in variegated magnification revealed the several agglomerated shapes found in non-uniform and irregular polygon-like agglomerated structures along with the long cylindrical shaped structures. Structures in cuboidal and hexagonal geometry were also observed in the ash catalyst. Several spongy cluster-like shapes were evident from the image taken at 100 μm magnification.

3.1.3 IR Spectrum of the Catalyst

Figure 5 displays the IR image of the catalyst used. From the IR spectrum, the peak observed at 3403 cm^{-1} arises due to the moisture adsorption on the catalyst's surface. Absorption peaks observed in the myriad of 1650–1050 cm^{-1}, could be attributed to the stretching and bending vibrations of the C–O bond present in the carbonate ions of metal carbonates, the production of which might be due to the adsorption of CO_2 available in the atmosphere, on the surface of the metal oxide. The presence of these peaks also unveiled the existence of K, Ca and various other carbonates of the metal available in the ash catalyst. The appearance of the peak at 1122 cm^{-1} could be assigned to the existence of the Si–O–Si bonds present in the ash catalyst.

FT-IR Spectrum

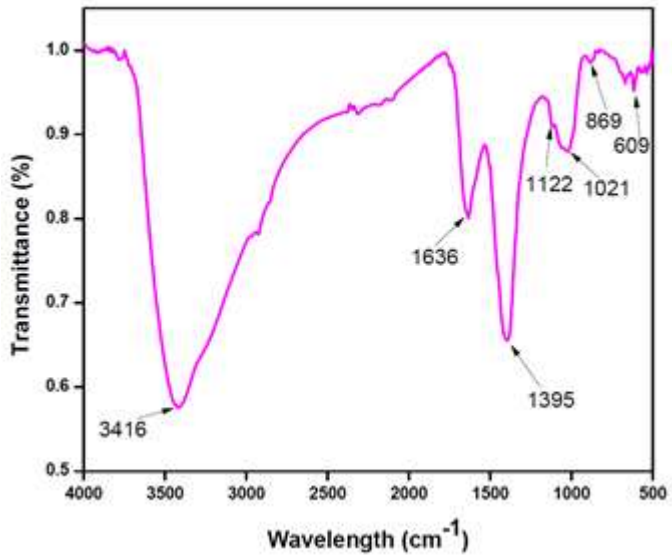

Figure 5: IR spectrum of the Catalyst

3.2 Biodiesel Analysis:

3.2.1 FAME Aharacterisation using GC-MS Analysis:

A GC-MS investigation was performed to unveil the chemical profile of the synthesised FAME product. This study allows the quantification of the distinct constituents found in the sample of the product. Figure 6 displays the chromatogram of the biodiesel sample that was produced by gas chromatography. The quantitative determination of the FAME component was assessed by contrasting the FAME peak areas with the peak area of the internal standard peak (methyl heptadecanoate, C17:0). A summary of the gas chromatogram data was provided in Table 1.

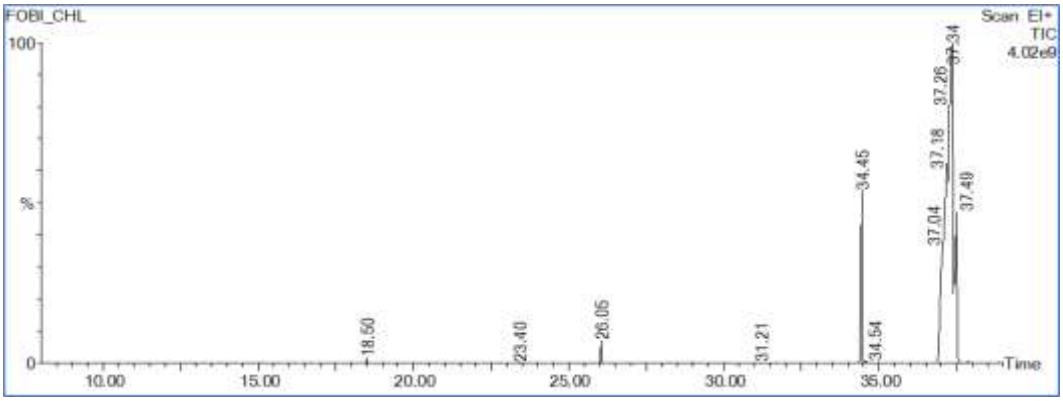

Figure 6: GC-MS of the biodiesel obtained from Linum usitatissimum (Linn.) oil.

Table 1 lists the identification of the FAME and the percentage composition of the GC-chromatogram, along with the retention time abbreviated as R.T. Ascertainment of the Linum usitatissimum (Linn.) biodiesel, the composition of the product showed that it consists of methyl linolenate as the prime constituent (78.54%). The components found subsequently were identified as methyl stearate (12.28%), methyl palmitate (8.27%), methyl isostearate (0.705%), methyl myristate (0.14%), 13,16-octadecadiyoic acid methyl ester (0.041%), methyl oleate (0.019%), etc.

Table1: Chemical compositions of the biodiesel obtained from Linumusitatissimum (Linn.) biodiesel (FAME

Serial No.	**Retention Time (in mins)**	**Component recognized**	**Corresponding acid**	**% composition**
1	18.50	methyl myristate	$C_{15}H_{30}O_2$(C14:0)	0.14%
2	23.40	13,16-octadecadiyoic acid methyl ester	$C_{19}H_{30}O_2$(C18:2)	0.041%
3	26.05	methyl isostearate	$C_{19}H_{38}O_2$ (C18:0)	0.705%
4	31.21	methyl oleate	$C_{19}H_{36}O_2$(C18:1)	0.019%
5	34.45	methyl palmitate	$C_{17}H_{34}O_2$(C17:0)	8.27%
6	37.34	methyl linolenate	$C_{19}H_{32}O_2$(C18:3)	78.54%
7	37.49	methyl stearate	$C_{19}H_{38}O_2$(C18:0)	12.28%

3.2.2 FAME Characterization employing ^{1}H NMR studies:

Formation of FAME in our synthesised biodiesel was confirmed by the development of a methoxy proton appearing as a singlet at 3.6 ppm (Figure: 7(b)) and the evanescence of glyceridic peak closeby at 4-4.3 ppm and the methine proton at 2.30 ppm appearing as a distinct triplet in the ^{1}H NMR spectra (Figure 7(a)) of the oil.

Quantification of the transition from oil to FAME, in percentage, was made using the formula mentioned below where the assessment is based on the comparison made between the integration values of the methoxy protons present in methyl ester and the protons of the α-methylene [26].

$$C = 100 \times \frac{2A_{Me}}{3A_{CH_2}}$$

where, C designates the triglyceride to FAME conversion percentage

A_{Me} gives the quantified value of the integration of the methoxy protons of FAME (2.22 ppm)

A_{CH_2} designates the value of integration value made for α-CH_2 protons (1.70 ppm).

As per the assessment made using the given equation the transition percentage from flaxseed oil to the corresponding synthesized methyl ester was found to be 87.06%.

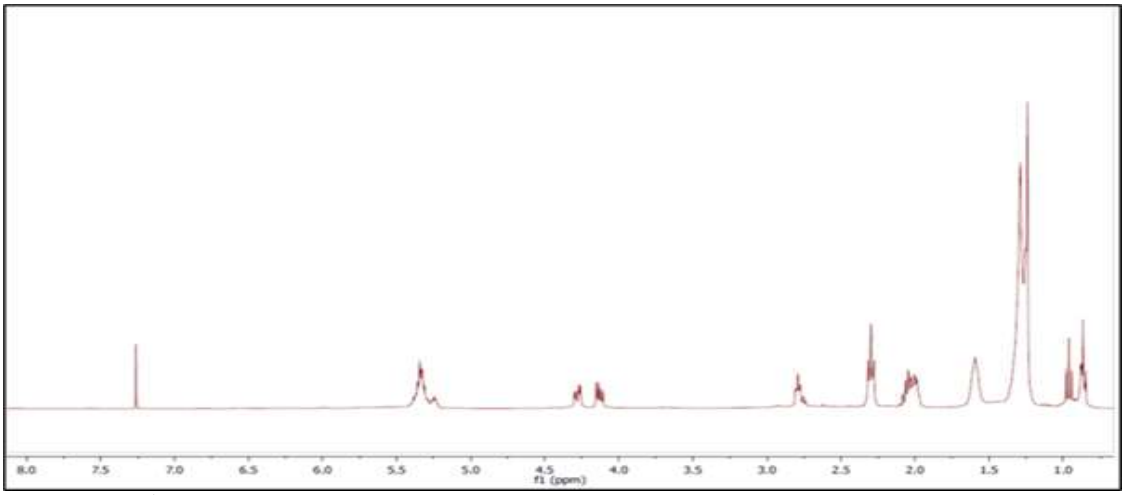

Figure 7(a) ^{1}H NMR spectra of Linum usitatissimum (Linn.) oil

Figure 7(b) ^{1}H NMR spectra FAME or biodiesel produced from Linum usitatissimum (Linn.) oil

3.2.3 Biodiesel Properties:

The physico-chemical attributes of the synthesized flaxseed biodiesel were ascertained employing standard methods as displayed in Table 2. The density of the synthesized biodiesel is found as 904.7 kg/m^3. The kinematic viscosity of the present biodiesel as observed at a temperature of 40 ◦C was 4.7226 cSt. The kinematic viscosity reveals the fuel behavior in cold conditions. The study showed the saponification value of the shaped biodiesel to be 202 mg KOH/gm, falling within the ASTM D5558-95 limit of maximum 370 mg/KOH. The SN obtained revealed the satisfactory stipulations of biodiesel owing to its display of normal components of fatty acids. It is because of the fact that the fabricated biodiesel with a high SN has a higher percentage of low molecular weight fatty acids [34-35]. An essential specification for biodiesel is the iodine value which indicates its unsaturation level. A biodiesel with a low iodine value is normally advantageous[36]. Computation of the cetane number for the synthesized biodiesel quantified it as 54.98, which falls in between the agreeable [9] (minimum 47 and 51) reference values. The calculated HHV of the present biodiesel was assessed as 39.93 MJ/kg, implying the adequate load which is integral for the liberation of heat for the complete combustion of a kg of biodiesel. Another fuel property, diesel index was determined to be 62.47.

Table 2: Physiochemical properties of synthesized biodiesel

Parameters	**Result**	**ASTM method**	**EN standard value**
Density(g/cm^3)	0.904 (slightly higher)	0.870–0.890	0.860–0.900
Kinematic viscosity (cSt)	4.7226	1.9–6.0	3.5–5.0
Saponification number (SN) (mgKOH/gm)	202	-	-
Iodine value (g I_2/100g)	81.45	NS	-
Cetane number	54.98	48 – 65	51
Higher heating value (HHV) (MJ/kg)	39.93	NS	-
Diesel Index (DI)	62.47	50.4	-

NS: Not specified

4. CONCLUSION:

To conclude, we described the synthesis of biodiesel using Linum usitatissimum (Linn.) oil successfully with the application of MCA as a sustainable catalyst. In this method, the catalyst used was a renewable, inexpensive, easily derivable, labor-efficient, and eco-friendly solid catalyst for the production of biodiesel. The preparation method was also a promising and simple one which did not demand any kind of functionalization, elevated temperature, chemical treatment. The reaction was brought off at room temperature, maintaining a molar ratio of Linum usitatissimum (Linn.) oil to methanol maintained at 1:15, a timeframe of 30 mins, and the catalyst loading of 10 wt % where the transition from oil to biodiesel reached 87.06 %.

CREDIT AUTHORSHIP CONTRIBUTION STATEMENT:

Trisha Dutta: Writing-original draft, Conceptualization, review & editing, Monmi Saikia: Writing - original review & editing, Validation, Conceptualization, Raju Narzary: Writing-original draft, Conceptualization, Swapna Nandi: Methodology, Investigation, Validation

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Conflict of Interest: The authors declare no conflict of interest for the manuscript.

Abbreviations:

FT-IR	Fourier transform infrared spectrometer
MCA	Musa champa ash
HHV	higher heating value
API	American petroleum index
GC–MS	Gas chromatography-mass spectrometry
FAME	Fatty acid methyl ester
HRTEM	High resolution transmission electron microscope
XRD	X-ray diffractometer
IV	Iodine value
NMR	Nuclear magnetic resonance
ORCs	optimum reaction conditions
SN	saponification number
TGA	thermogravimetric analysis
TLC	thin layer chromatography
FESEM	Field emission scanning electron microscope
DMSO	dimethyl sulfoxide
TMS	tetramethylsilane

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