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SYNTHESIS OF CALCIUM OXIDE@MAGNESIUM OXIDE NANOCATALYST FOR TRANSESTERIFICATION OF WASTE COOKING OIL

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Abstract : Environmental concerns of fossil-based fuel sources has driven progress towards several sustainable alternatives, such as methyl esters, also known as '*biodiesel*'. The synthesis of biodiesel is done by transesterification, assisted by either homogenous or heterogeneous catalysis. Biodiesel obtained from homogenous catalysis requires purification procedures which yields a significant amount of waste water and the catalyst cannot be reused, thereby driving research towards a reusable alternative in heterogeneous catalysis. This study aims to synthesize calcium oxide and magnesium oxide nanocomposite (CaO@MgO) using microwave-assisted solution combustion technique for heterogeneous catalysis. Sodium hydroxide and potassium hydroxide were used for homogeneous catalysis, to aid the study. The synthesis of biodiesel from both types of catalysis was carried out via transesterification of waste cooking oil. The biodiesel yield was 96.5% for heterogeneous catalysis with a reaction time of 4 h and 3 wt. % catalyst, while the yield was 96.0% for homogenous catalysis with a reaction time of 1 h and 1 wt. % catalyst, and it was found to be easier to separate the former catalyst from the products, utilizing less water in the process. The density, viscosity, and calorific value were similar for biodiesel synthesized via both types of catalysis; however, the cloud point and turbidity were higher for biodiesel synthesized via CaO@MgO nano catalyst. This was however taken care of by precipitation using citric acid and centrifugation. Subjecting this catalyst to ethanol washing and ultra-sonication rendered it reusable up to three times, thereby justifying its use as a sustainable alternative.

***Index Terms* - Transesterification, Biodiesel, Heterogenous Catalysis, CaO@MgO, Nano catalyst.**

I. INTRODUCTION

The availability of energy resources is considered a crucial factor influencing a country's socio-economic development. The excessive utilization of petroleum-based resources over the years for various industrial and transportation processes is leading to their depletion, resulting in the increment of their prices associated with glitches such as increasing levels of environmental hazardous and greenhouse gases in the atmosphere [1]. It is necessary to find alternative energy sources that provide similar results in terms of fuel efficiency and must also be eco-friendly and cost-effective. The domain of biodiesel has gained much attention as a potential alternative to these conventional fuels [2].

Biodiesels are methyl esters formed when triglycerides in oils and fat are reacted with alcohol in a process called transesterification [3]. The main advantages of biodiesel compared to diesel fuel are eco-friendliness, renewability, high flash point, biodegradability and non-toxicity [4]. Biodiesel has similar properties to petroleum diesel and lower emissions, so that it can be used in the transport sector as an alternate to diesel fuel, or as a blend [5, 6]. With the increase in use, biodiesel could reduce the pollutants and movable carcinogens [7]. Different sources of feedstocks, like vegetable oils, algal oils, animal fats, microbial oils, and waste oils, can be used to produce biodiesel [8].

Energy consumption in India is more than 4% of global energy, and it is the fifth largest country in energy consumption. According to a forecast, India will be the third largest oil consumer by 2030 [9]. India's aspirational biodiesel blend goal for onroad use is B5 by 2030. Domestically sourced waste cooking oil was identified as a feedstock with an enormous, untapped potential for biodiesel production [10]. Indian cooking extensively utilizes various vegetable oils; in 2021, over 22 million metric tons of vegetable oil were consumed [11]. Used oil generated in large quantities in restaurants and college dining halls cannot be discarded as such. Therefore, these oils are readily available to serve as a source, making the process carbon neutral. Using waste cooking oil for biofuels is a cost-effective alternative to both, fossil fuel-based sources, and other sources of oils and fats [1]. Biodiesel synthesis requires a catalyst for transesterification; this catalyst can be homogenous, heterogeneous, or can be a biocatalyst [12]. An exhaustive amount of work has been carried out in synthesizing biodiesel by using potassium hydroxide (KOH) and sodium hydroxide (NaOH) in the field of homogenous catalysis due to their ease of availability and cost-effectivity. However, heterogeneous catalysis with solid catalysts has been found to offer various advantages such as reduction in soaping problems, easier separability, low sensitivity to free fatty acids (FFA), fewer side reactions, non-requirement of high temperature and pressure, and cost-effectivity [12, 13]. Little work has been done comparing the biodiesel obtained from different catalysts.

In the present work, transesterification was used to produce biodiesel from the waste cooking oil and methanol using heterogeneous CaO@MgO nano catalyst. The nano catalyst was obtained by microwave-assisted solution combustion technique and conventional homogeneous catalysts such as NaOH and KOH were used as procured. The objective was to optimize the process for synthesis of heterogeneous catalyst, biodiesel yield, and subsequent catalyst recovery and regeneration.

II. METHODS

2.1 Materials

Potassium Hydroxide (KOH), Sodium Hydroxide (NaOH), Methanol, Activated charcoal, Calcium Nitrate Tetrahydrate ($Ca(NO_3)_2.4H_2O$), Magnesium Nitrate Hexahydrate ($Mg(NO_3)_2.6H_2O$), Urea (NH_2CONH_2). All the chemicals were used as available without further purification. All the chemicals were AR grade with a purity of 99.9%. Deionized water (DI) was used in the catalyst preparation. The waste cooking oil was sourced from a local restaurant.

2.2 Methods

2.2.1 Pre-treatment of waste cooking oil

Phan et. al [14] worked on the production of biodiesel successfully from waste cooking oil using homogenous catalysis reporting a yield of about 90%. In addition, H. Hadiyanto et al [15], Patil et al [16], and Sahar et al [17] reported a similar yield of 88% thus further highlighting waste cooking oil as a viable source. Iván Dario et al [18] have shown filtration, water removal, and acid-catalyzed esterification as the pre-treatment steps to be used for the waste cooking oils to finally obtain oil with, 1% of moisture, 0.25% of solids suspended and 1.38 mg KOH/g oil of acid value. Raqeeb et. al [19] have also highlighted similar pre- treatment steps being successful for making the oil viable to be used for transesterification. The oil was first passed through a fine-mesh strainer and then through a V60 filter paper to remove tiny bits of sediments [14, 18]. It was then preheated for 2 hours at 110ºC to remove water from the discarded cooking oil. The acid value of oil was determined by directly titrating the oil in ethanol against a standard sodium hydroxide solution; phenolphthalein was used as an indicator. The acid value of sample 1 was found to be 1.31 mg of NaOH per gram of oil, which is ideal, and less than the acceptable value of <2 [19]. Waste cooking oil was then esterified with MeOH and H_2SO_4 as a homogeneous acid catalyst in a 0.5-liter conical flask attached to a thermometer, with magnetic stirring using a magnetic bar stirring at the speed of 900 rpm. The opening of the conical flask was covered to prevent the loss of MeOH by evaporation [14-21].

The esterification was carried out as per the conditions mentioned in table 1. The reaction product was then moved to a decanter and was allowed to settle for 24 hours to separate into the oil phase and an aqueous phase, where the latter was removed from the oil phase as per the difference in density. The oil phase was then washed with hot water to remove the catalyst and the alcohol. This process was carried out at 70ºC two times. The washed oil phase was dried by heating for 90 min at 110ºC [14-20]. The acid value test was performed on the esterified oil and was found to be 1.63 mg NaOH/g Oil which met the required standard for biodiesel synthesis.

Table 1: Conditions for esterification for pre-treatment of oil

Temperature	% Catalyst	Molar ratio	Reaction Time
70 °C	1 w/w%	Cooking oil: MeOH 1:4	2 hours

2.2.2 Synthesis of CaO@MgO nano catalyst

The Microwave-assisted solution combustion method was used to synthesize the nano catalyst, which involves the following steps: Selection of calcium nitrate tetrahydrate and magnesium nitrate hexahydrate and urea, as the oxidizers and the fuel respectively. The proportional (3:1) quantity of the fuel to the oxidizer was taken as per the stoichiometric equation. Then the solid mixture was dissolved with a minimum quantity of distilled water and slightly mixed. This mixture was then subjected to heating in a wire mesh. After a short interval of time, once the gelation was observed, the gel was transferred into the microwave oven at a set power of 800 W for 10 minutes for the chemical reaction to occur. A vigorous reaction occurred and underwent dehydration by giving a white puffy mass of CaO-MgO nanoparticles [22, 23]. This solid was ground into a fine powder using a pestle and mortar and stored in an air tight bottle.

2.2.3 Biodiesel production

2.2.3.1 Heterogenous catalysis

Biodiesel production from the waste cooking oil in the presence of CaO@MgO nano catalyst involved weighing 40 grams of oil in an Erlenmeyer flask. The operating conditions for transesterification are depicted in table 2. The measured quantity of oil was heated to 60ºC on a magnetic hot plate stirrer. Methanol and the oil were taken in a 6:1 molar ratio where firstly 1.2 grams of nano catalyst (about 3 wt.% of the oil) was weighed separately in an Erlenmeyer flask, and then calculated amount of methanol was added into it. The catalyst-alcohol mixture was agitated separately and then transferred into the heated oil and covered with an aluminum foil to keep the alcohol vapor from escaping.

The mixture was stirred at 900 rpm for about 240 minutes at a constant temperature and was then transferred to a separating funnel and then was allowed to settle overnight [22, 23]. Two distinct layers were observed, i.e., lower layer (trace glycerol and catalyst) and the upper layer (biodiesel). The biodiesel was separated and collected in a flask and then weighed. About 38 grams of biodiesel was obtained. The biodiesel yield was found to be 96.5% based on the amount of oil used initially.

Table 2: Conditions used for Transesterification

Temperature	% Catalyst	Molar ratio	Reaction Time
60 °C	3 w/w% (1.2 g)	Cooking oil: MeOH 1:6	240 minutes

2.2.3.2 Homogenous catalysis

The production of the biodiesel using homogeneous catalysis was carried out in two separate batches once with KOH and once with NaOH respectively. Firstly, 250 grams of waste cooking oil was trans esterified with MeOH and the catalyst in a 0.5- liter conical flask attached to a thermometer, with magnetic stirring using a magnetic hot plate stirrer at a speed of 900 rpm. The opening of the conical flask was covered to prevent the loss of MeOH by evaporation [24, 25]. The reaction was carried out as per the conditions mentioned in table 3.

The reaction product was moved to a decanter and settled for 24 hours to separate into biodiesel and glycerol, where the former was removed from the latter as per the difference in density [24, 25]. The removal of unreacted methanol, triglycerides, catalyst, and glycerol from the biodiesel was brought about by treatment with activated charcoal [26]. From this process, 240 grams of biodiesel were obtained, and the yield of the biodiesel was found to be 96%.

Table 3: Conditions used for Transesterification

Temperature	% Catalyst	Molar ratio	Reaction Time
60 °C	1 w/w% (2.5 g)	Cooking oil: MeOH 1:6	60 minutes

III. RESULT AND DISCUSSIONS

3.1 Characterization of CaO@MgO nano catalyst

3.1.1 X-ray diffraction

The nano catalyst synthesized using the microwave assisted solution combustion method and was characterized by X-ray diffraction (XRD) as shown in figure 1.

The peaks of Magnesium Oxide (Ref. Code - 98-005-6143) and Calcium Oxide (Ref. Code 98-005-1409) have been indicated by colored symbols. As seen by the Miller Indices, upon calculation the nature of the lattice was found to be cubic. The miller Indices were compared with the XRD results of R. Foroutan et al [23] on the characterization of calcium and magnesium oxide catalyst. The peaks seen by the nature of the XRD graph indicate that it is a crystal lattice. The materials identified by the software present in the sample were only calcium and magnesium oxide indicating no other impurities were present.

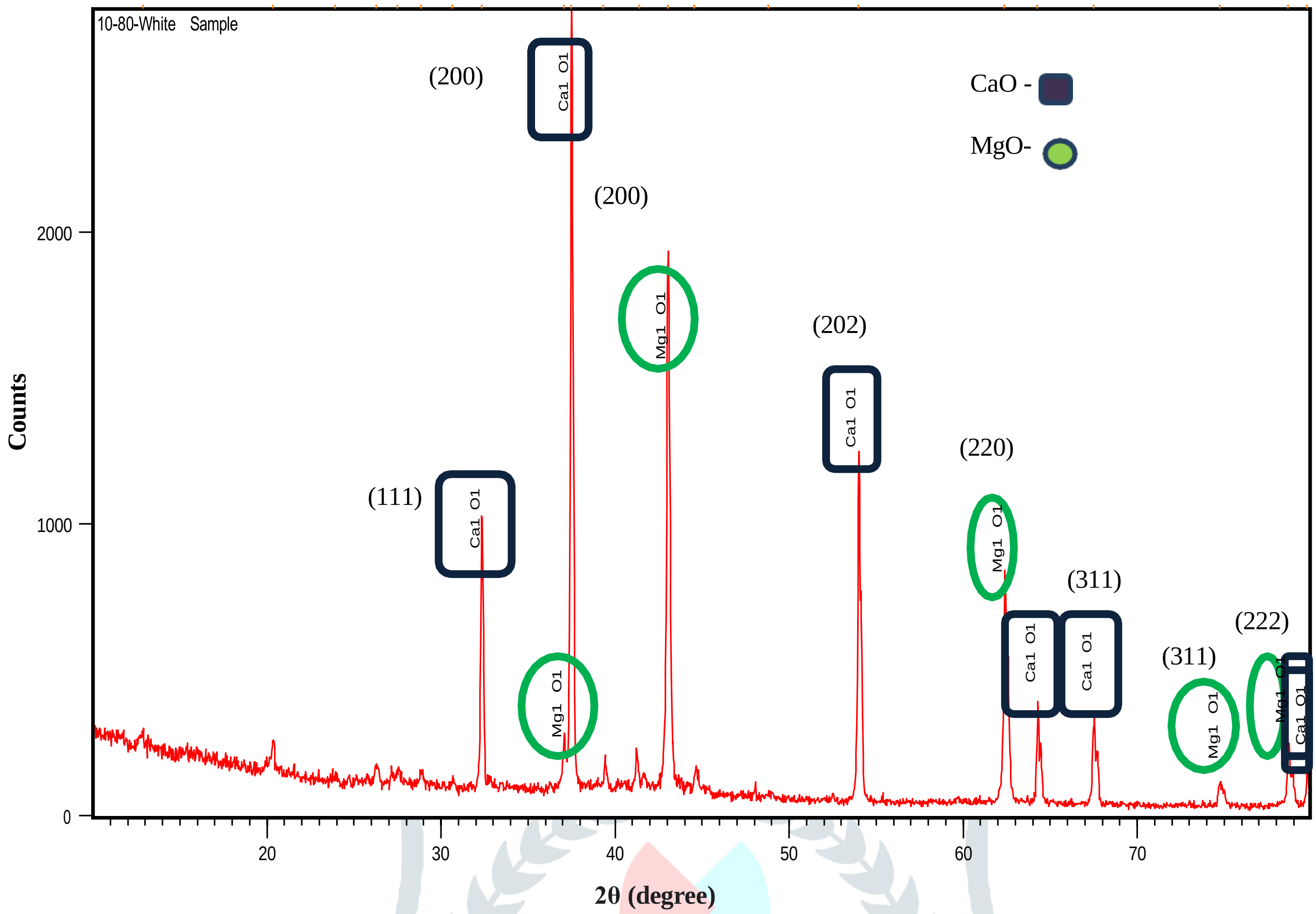

Figure 1: XRD pattern of CaO@MgO nanocomposites synthesized through micro-assisted combustion technique

3.1.2 Scanning electron microscopy

In order to study the surface morphology of CaO@MgO nano composites scanning electron microscopy **(**SEM) was done as shown in figure 2. From the SEM image it was observed that the composite had a roughly cubic structure and the average particle size was 75.08 nm.

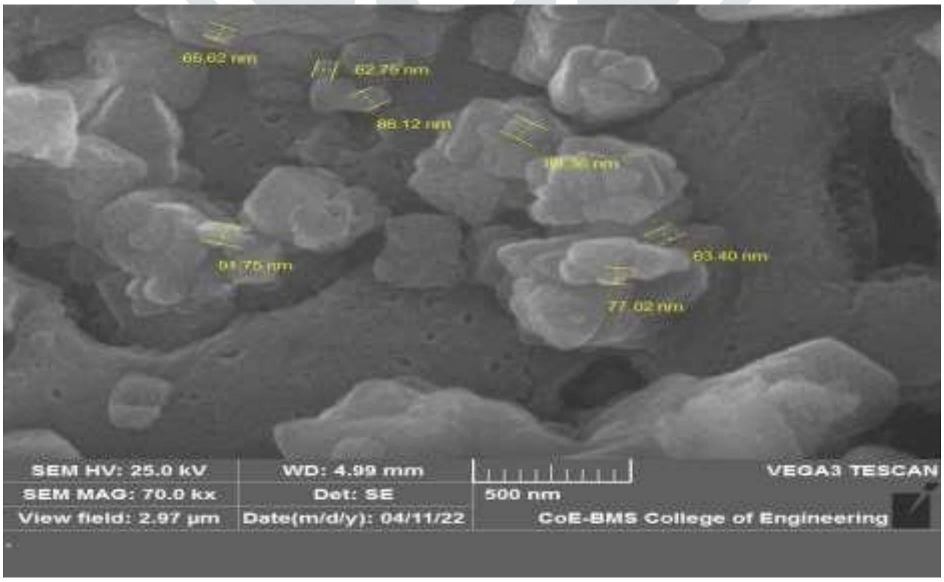

Figure 2: SEM image CaO@MgO nanocomposites

3.1.3 Normal Probability Distribution

From the normal probability distribution graph shown below, we further ascertained that a majority of the particles lie between 75 and 80 nm which shows that the particle sizes are nearly uniform and around the average particle size.

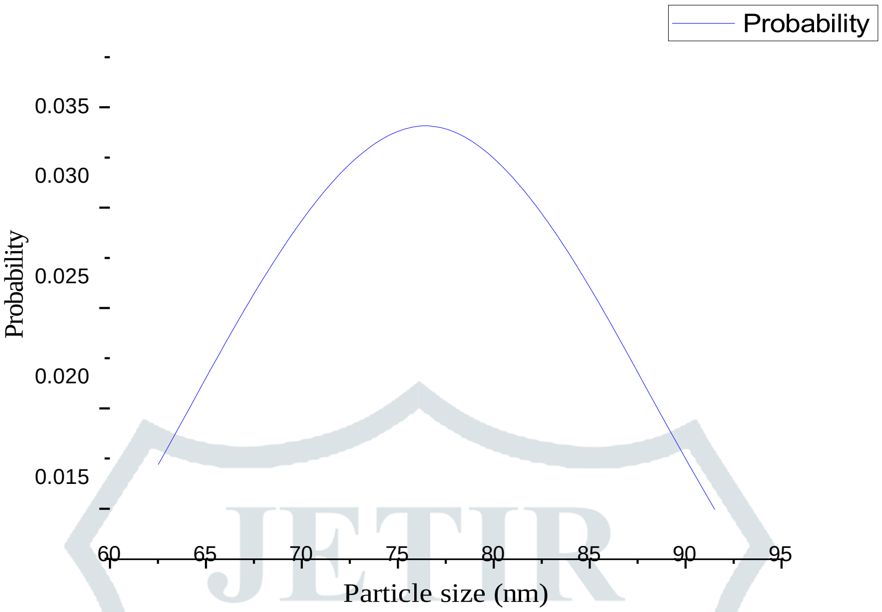

Figure 3: Probability Distribution for CaO@MgO nanocomposites

3.2 Biodiesel purification, and Catalyst reuse and regeneration studies

The biodiesel synthesis was carried out with 1.2 grams of CaO@MgO nano catalyst (3 wt.% of oil), waste cooking oil and methanol (taken in the 6:1 molar ratio) under the aforementioned reaction conditions in order to study the effects of catalyst reuse on biodiesel yield as shown in table 4. For each run the catalyst was regenerated with the following procedure, catalyst was first separated from the biodiesel catalyst mixture by filtration. Ethanol was added to the separated catalyst to dissolve biodiesel and activate the basic sites of the catalyst. The mixture was then filtered, and ethanol was added again, and the mixture was then subjected to ultra-sonication to promote better interaction between the catalyst and ethanol. The sample was then calcined at 500ºC for 30 minutes [27].

The observations showed that the catalyst can be reused up to two times before a significant deterioration in yield is noticed, making this catalyst more economic than its homogenous counterparts.

Table 4: Biodiesel yield obtained upon reuse of catalyst in successive batches

Batch no.	Total oil taken (gm)	Biodiesel produced (gm)	Yield of biodiesel
1	160	154	96.25
2	30	25.6	85.33
3	7.5	3.68	46.25

3.3 Characterization of biodiesel samples

The properties of biodiesel, such as kinematic viscosity, density, cloud point, turbidity, specific gravity, and calorific value, were determined using different types of equipment/experimental setups elucidated as follows.

3.3.1 Density

The density was determined using a specific gravity flask of 100 ml. The weight of the empty flask was noted, and our sample was poured in. The weight of the biodiesel was calculated and divided by the volume to give density. It can be seen from table 5 that the density of all biodiesel samples is about the same value.

Table 5: Comparison of Density of biodiesel at 25^{0}C

Catalyst used for Biodiesel synthesis	**Density (g/cc)**
CaO@MgO	0.864
KOH	0.867
NaOH	0.869

3.3.2 Turbidity

The Turbidity was found using Nephelo-Turbidometer [28]. The instrument was first calibrated for 0 and 100 NTU on a scale of 100, then the samples were inserted and readings were noted down and the results are tabulated below in table 6. It can however be seen that the biodiesel obtained from CaO@MgO nano catalyst has rather high turbidity as per the ASTM standards.

Table 6: Comparison of Turbidity of biodiesels obtained

Catalyst used for Biodiesel synthesis	**Turbidity (NTU)**
CaO@MgO	2.4
KOH	0.5
NaOH	0.6

3.3.3 Calorific Value

The measure of the energy contained in the fuel is determined by the heat produced upon the complete combustion of a specified amount of fuel and is usually expressed in J/Kg. The bomb calorimeter was used to determine the amount of heat released. The sample was taken in a holder, the fuse wire (nichrome wire) was attached to the fuse, and a cotton thread was dipped into the sample and attached to the nichrome wire. The entire setup was taken in the bomb enclosure with 1.5 liters of distilled water. The rise temperature was set to 0 on the digital display, and the charge was given. The temperature rise was noted. The following equation 1 was used for calculating the specific heat capacity of the fuel (u_f) [28, 29],

$$u_f = \frac{[(mc_p)_b + (mc_p)_w]\Delta T - (mu)_c - (mu)_{fw}}{m_f} \quad (1)$$

Where the energy equivalent factor of the calorimeter (the sum of specific heats of the bomb and water), $[(mc_p)_b + (mc_p)_w]$ = 2402 cal/^{0}C, heat of combustion of the fuse wire $(u)_{fw}$ = 1400 cal/g, and heat of combustion of the gelatin capsule $(u)_c$ = 4600 cal/g were known beforehand. The mass of 12 cm of the fuse wire $(m)_{fw}$ taken was found to be 0.014 g, mass of the fuel which is represented by $(m)_f$ was also found for each trial.

In table 7 it can be the seen that the biodiesel obtained from CaO@MgO nano catalyst has a comparable calorific value with the biodiesel obtained by homogenous catalysis.

Table 7: Comparison of Calorific values

Catalyst used for Biodiesel synthesis	**m1**	**m2**	**ΔM**	**ΔT**	**Specific heat capacity (cal/g)**	**Specific heat capacity (MJ/Kg)**
KOH	4.21	2.45	1.76	7	9542	40
NaOH	4.75	1.82	2.93	8.53	6986	29
CaO@MgO	4.57	2.56	2.01	7.5	8953	37

Here m1 is the mass of the fuel before combustion, m2 is the mass of the fuel after combustion, ΔM is the change in mass of the fuel and ΔT is the change in the temperature during combustion.

3.3.4 Viscosity

The viscosity of a fluid is the measure of its resistance to deformation at a given rate. For estimating the viscosity, we used the Brookfield viscometer, a beaker of 300 ml capacity filled with the sample set at 50 RPM. The viscosity readings were then noted at Room temperature (30^{o}C) [30]. The results tabulated in table 8 show that all the biodiesel samples have roughly the same viscosity.

Table 8: Comparison of Viscosity at 30^{o}C

Catalyst used for Biodiesel synthesis	**Viscosity (cP)**
CaO-MgO	1.56
KOH	1.44
NaOH	1.32

3.3.5 Cloud Point

The cloud point is the temperature where the fluid starts to solidify under normal pressure conditions. For checking the cloud point of our biodiesel, an ice bath was used in which the test tube filled with the sample was inserted along with a thermometer to check the temperature. The point at which the sample started to get cloudy was noted.

The advantage of a lower cloud point is that the fuel takes a lower temperature to “gel up” or form small crystals, thereby reducing the chances of clogging in the engine and ensuring proper supply of the fuel for combustion. The lower cloud point also ensures that the fuel will be able to run in a colder climate since it would not solidify as quickly as other alternative biofuels. It was however found out that the cloud point of the biodiesel obtained from CaO@MgO nano catalyst was rather high as shown in Table 9.

Table 9: Comparison of Cloud Point

Catalyst used for Biodiesel synthesis	**Cloud Point (^{o}C)**
CaO-MgO	18
KOH	1.5
NaOH	12

3.4 Purification of biodiesel obtained from CaO@MgO:

In order to enhance the properties of the biodiesel obtained from CaO@MgO nano catalyst the following procedure was adapted. 1:1 molar ratio of citric acid to CaO@MgO was taken and subjected to stirring at 45^{o}C for 30 min on a temperature-controlled magnetic stirrer. The citric acid coagulated with calcium and magnesium ions as citrates and was separated from the biodiesel by centrifuging it at 4000 RPM for 25 minutes [31]. It was found that the procedure gave desirable improvements in the quality of the biodiesel as depicted in table 10 and table 11 for turbidity and cloud point respectively.

Table 10: Comparison of turbidity before and after treatment

Biodiesel sample	Turbidity (NTU)
Before treatment with citric acid	2.4
After treatment with citric acid	1.5

Table 11: Comparison of Cloud Point before and after treatment

Biodiesel sample	Cloud point (ºC)
Before treatment with citric acid	18
After treatment with citric acid	4

3.5 Blends

For testing the biodiesel for practical functionality of our biodiesel obtained from CaO@MgO nano catalyst, we created a blend in the 25:75 ratio with commercial diesel. The blend was then tested in a generator and a tractor where in both applications gave promising results.

IV. CONCLUSION

Through the comparison between the use of NaOH and KOH to obtain the biodiesel by homogenous catalysis, and the use of the inhouse CaO@MgO for the synthesis of the biodiesel via heterogeneous catalysis, it was found that the yield was higher in the latter case by 0.5%. The properties of the biodiesel obtained were also comparable for both the catalysis methods barring the cloud point and turbidity of the biodiesel initially obtained by CaO@MgO nano catalyst. These were then lowered by effective methods to a comparable ASTM standard. Therefore, the final comparable properties of the biodiesel enhanced with reutilization aspect of the heterogenous catalyst seen in this study, highlights the effectiveness of this method over homogenous catalysis as a more sustainable route to meet the rising energy demands through biodiesels.

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