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POTENTIAL OF MAGNETIC SAND AS CATALYST FOR THE HETEROGENEOUS TRANSESTERIFICATION OF *Ziziphus abyssinica* SEED OIL TO BIODIESEL

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ABSTRACT

Biodiesel is produced through esterification and transesterification reactions of vegetable oil and animal fats with an alcohol. Methanol or ethanol is usually the alcohol for biodiesel preparation. The reaction is facilitated with either homogeneous or heterogeneous catalyst. The quantity of free fatty acid is important in order to select the appropriate catalyst Today biodiesel compared with petroleum is considered an environmentally friendly fuel due to low carbon dioxide emissions, biodegradable fuel, high cetane number, high combustion efficiency, lower sulphur and aromatic content in comparison to petroleum diesel, making the biodiesel a competitive fuel in the market. Biodiesel production aims to get good qualities and quantities by choosing suitable and cheap feedstock such as virgin vegetable oils, used cook oils and animals' fats. In this study, *Ziziphus abyssinica* oil is utilized in the manufacturing of biodiesel. Magnetic sand from Lagos Street River in Maiduguri was used as a potential heterogeneous catalyst. The study revealed that the viscosity, flash point, cloud point, pour point, pH and density are in good agreement with those parameters earlier carried out using K_2CO_3, Na_2CO_3, MgO, CaO, mixed oxides of zinc and aluminium etc as catalysts. FTIR analysis of the biodiesel shows intense peaks at 2862.46 cm^{-1} and 2916.47cm^{-1}, indicating the presence of C – H, 1743.971cm^{-1}, signifying the presence of C = O, 1427.37cm^{-1}, implying the presence of a methyl ester group – $COOCH_3$. A closely and neatly packed spherical granule was observed in the magnetic sand from the SEM analysis.

KEYWORDS: Biodiesel, *Ziziphus abyssinica*, Magnetic sand, Heterogeneous catalyst, Transesterification.

INTRODUCTION

Biodiesel is produced through esterification and vegetable oil transesterification reactions and animal fats with an alcohol. Methanol or ethanol is usually the alcohol for biodiesel preparation. The reaction is facilitated with a suitable catalyst either homogeneous or heterogeneous. The quantity of free fatty acid is important in order to select the appropriate catalyst [1]. Today, biodiesel compared with petroleum is considered an environmentally friendly fuel due to low carbon dioxide

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emissions, biodegradable fuel, high cetane number, high combustion efficiency, lower sulphur and aromatic content in comparison to petroleum diesel, making the biodiesel a competitive fuel in the market [2]. Biodiesel production aims to get good qualities and quantities by choosing suitable and cheap feedstock such as virgin vegetable oils, used cook oils and animals fats. In this study, *Ziziphus abyssinica* oil is utilized to produce biodiesel. A variety of catalysts, including ion exchange resins, sugars, lipases, zeolites, homogeneous and heterogeneous acids and bases, and others, can be utilized to produce biodiesel.

The transesterification reaction using homogenous basic catalysts like sodium (or potassium) hydroxide or methoxide is typically quicker, less costly, and more comprehensive with these materials than with acid catalysts [3]. Sodium methoxide is now used in the biodiesel business, since water cannot be formed by methoxide when reaction with hydroxides and alcohol, for example, methanol [3]. The homogenous base-catalyzed transesterification reaction is about 4,000 times faster compared to the analogous acid-catalyzed procedure [4]. Additionally, base-catalyzed reactions are less harmful to industrial equipment and are often carried out at lower temperatures, pressures, and reaction periods than

acid-catalyzed methods. Thus, in the case of the base-catalyzed transesterification technique, biodiesel manufacturing facilities incur lower capital and operating expenses[5]. However, the homogenous acid-catalyzed reaction holds an important advantage over the base-catalyzed method in that the performance of acid catalysts is not adversely influenced by the presence of FFA. In fact, acids can simultaneously catalyze both esterification and transesterification [6]. A mixed oxide of zinc and aluminum has been reported to be the catalyst used in the recent industrial-scale biodiesel synthesis process utilizing heterogeneous catalysts [7]. Heterogeneous catalysts are environmentally friendly, economical and have high chance for heat integration [8]. At the industrial level, heterogeneous catalysts for the synthesis of biodiesel have not yet been extensively utilized. Lewis or Brönsted catalysts could be used to describe heterogeneous acid and basic catalysts. The transesterification reaction rate is determined by this catalyst's characteristics. It has been determined that catalyst performance in the transesterification reaction is enhanced by higher basicity and, consequently, by the existence of more active sites. Compared to homogeneous catalysts, heterogeneous catalysts are less corrosive and can be employed in fixed-bed reactors, making operations safer, less expensive, and more environmentally friendly. They also require fewer separation steps. The heterogeneous catalysts are likely to be kept in the reactor by filtering since they do not leave neutralizing salts in the glycerol [9].

Heterogeneous basic catalysts that are most frequently researched are alkaline metal carbonates (Na_2CO_3, K_2CO_3), alkaline earth metal

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Factors Influencing the Biodiesel Production Process

Water and Free Fatty Acid (FFA) Contents' Effects

Regarding the transesterification process the amounts of water and fatty acids that are free (FFA) are crucial. Water-free and low acid value (<1) raw materials are needed for the base-catalyzed transesterification reaction in order to produce biodiesel [5]. More alkali catalyst is needed in the reaction to neutralize the FFA if the oil samples contain a high FFA level (more than 1%). Since water can result in soap production and foaming, which can raise viscosity, its presence has a more detrimental effect than that of FFAs. Furthermore, the separation of glycerol from biodiesel is impeded by the development of gels and foams [5]. During transesterification, free fatty acid and water always result in unfavourable outcomes, causing soap to develop and consuming the used catalyst for biodiesel production is sodium hydroxide (NaOH) or potassium hydroxide (KOH). However, Freedman *et al*., 1984 [13] found that the sodium methoxide would be more effective because mixing of sodium hydroxide with methanol produces little amount of water which inhibit the formation of end product (biodiesel) due to the hydrolysis reaction [18] This is one of the reasons for mixing of catalyst with methanol first and then added to the oil or fats. In addition to this when the concentration of catalyst is increases with oil samples, the conversion of triglycerides into biodiesel is also increases. On the other hand, insufficient amount of catalyst leads to the incomplete conversion of triglycerides into fatty acid esters [16]. However, optimal product yield (biodiesel) was achieved when the concentration of NaOH reaches 1.5 wt. % at the same time further increase of catalyst concentration proved to have negative impact on end product yield. Because addition of excess amount of alkali catalyst reacts with triglycerides to form more soap [16].

MATERIALS AND METHODS

5 mL of *Ziziphus abyssinica* oil was purchased from a commercial oil producer in Maiduguri metropolis of Borno State. Methanol 99% purity of analytical grade was obtained from Northern scientific (Nigeria). Magnetic sand was collected directly from its deposit with the aid of a magnet bar to minimize impurities. The sample was packed into a plastic container prior to analysis.

Catalyst Characterization

X-Ray Diffraction

The X-ray diffraction (XRD) characterization of magnetic sand was performed on a Empyrean (Panalytical model) based generator X-ray diffractometer. The analyzed material was finely ground to pass through 63 microns, homogenized, and average bulk composition was determined. The powdered sample was then prepared using the sample preparation block and compressed in the flat sample holder to create a flat, smooth surface that was later

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mounted on the sample stage in the XRD cabinet.

The sample was analyzed using the reflection-transmission spinner stage using the Theta-Theta settings. 2θ starting position was 0.00483 and ends at 75.000 with a 2θ step of 0.026 at 3.57 seconds per step. Tube current was 40mA and the tension was 45VA. Fixed Divergent Slit size of 1° was used and the goinometer radius was 240mm.

Crystallite Size Determination

Fityk 0.9.8 version software was used to obtain the Full Width at Half Maximum (FWHM). The average crystallite size of catalysts was calculated from the line broadening or FWHM (full width at half maximum) of corresponding peaks (the most intense peaks), using Modified Scherrer formula which decreases errors by obtaining the average value of crystallite size through all the peaks (or any number of selected peaks) using least squares method [18]. The basic Scherrer formula is written as:

$$\beta = \frac{K\lambda}{L.Cos\theta} = \frac{K\lambda}{L} \cdot \frac{1}{Cos\theta}$$

Where L is the crystallite size for (hkl) phase (nm), λ is the x-ray wavelength of radiation for Cuk_{α}, β_{hkl} is the Full Width at Half Maximum (FWHM) at (hkl) in radian and θ is the diffraction angle for (hkl) phase.

Now by making logarithm on both sides;

$$In\beta = In\frac{K\lambda}{L.Cos\theta} = In\frac{K\lambda}{L} + In\frac{1}{Cos\theta}$$

From Table 2, plotting the results of lnβ against ln(1/Cosθ), gave a straight line with a slope of around one and an intercept of about $In\,(K/L)$. The exponential of the intercept yields

$$e^{In\frac{K\lambda}{L}} = \frac{K\lambda}{L}$$

Hence the value for the crystallite size L was obtained.

Having K = 0.89 and $\lambda(\lambda Cuk\alpha 1$ = 0.15405 nm), a single value of L in nanometer was calculated as shown in Figure 2.

Biodiesel Characterization

Fuel properties such as viscosity, flash point, cloud point, specific gravity, acid value and free fatty acid were determined according to American Society for Testing and Materials (ASTM) standards. The biodiesel product was analysed by Fourier Transform Infra-Red spectrometer (Nicolet 5700).

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RESULTS AND DISCUSSIONS

Table 2: Biodiesel quality parameters

Fuel Property	**MOCZAME**	**MSCZAME**	**MOMSCZAME**	Limit (ASTMD6751)	**Specification**	
					US	**UK**
Viscosity (cst)	5.4	5.6	5.3	1.9–6.0	1.9-6.0	3.5-5.0
Flash point (◦C)	183	208	241	130 min.	315-350	360
Cloud (◦C)	8	7	10	-3 – 12	-3-12	-
Pour point (◦C)	3	4	6	-3 – 16	-15-10	-
pH value	8.9	8.7	8.9	- - - - - -	9	9

KEY: MOCZAME=Magnesium oxide Catalyzed *Ziziphus abyssinica* Methyl ester, MSCZAME=Magnetic Sand Catalyzed *Ziziphus abyssinica* Methyl ester and MOMSCZAME= MgO/Magnetic Sand Catalysed *Ziziphus abyssinica* Methyl ester

Pour Point

The pour point is the lowest temperature at which the oil will pour or flow when it is cooled, without stirring, under standard cooling conditions. In terms of gravity, the pour point is the lowest temperature at which oil may flow. One of the key low-temperature properties of high-boiling fractions is this. It was discovered that biodiesel was found to be within ASTM D6751 standard for biodiesel specification. A similar trend was obtained from the biodiesel produced from *Khaya senegalensis* seed oil by Babakura *et al*., 2019 [17]. Conversely, the viscosity and pour are higher than that reported by Danbature *et al*., 2015 [18].

Flash point

Flash point, the lowest temperature at which a liquid (usually a petroleum product) will form a vapour in the air near its surface That will flash or briefly ignite on exposure to an open flame. The flash point is a general indication of the flammability. Flash points are lower than those reported by same [19]. Although, the flash point of the oil falls within the standards but it is

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lower than those of sunflower and rape seeds methyl esters. Therefore, these biodiesels can ignite at a higher temperature than those of the other oils.

Density

Density is an important biodiesel parameter, with impact on fuel quality. Predicting density is of major relevance for a correct formulation of an acceptable Blend of raw materials that optimize the cost of biodiesel fuel production while allowing the produced fuel to meet the requisite quality criteria. Biodiesel were found to be within ASTM D6751 standard for biodiesel specification. A similar trend was obtained from the biodiesel produced from Khaya senegalensis seed oil by Babakura *et al*., 2019 [17].

Viscosity

Viscosity is the degree of a material's resistance to flow, high viscous materials flow with great difficulty, while less viscous one's flow with ease. [20]. It causes the creation of engine deposits since it influences how a fuel atomizes when it is injected into the combustion chamber, the higher the viscosity, the greater the propensity of the fuel to cause such problems. The kinematic viscosity value of the biodiesel decreases with increase in temperature [21]. The viscosity of biodiesel was discovered to be within ASTM D6751 standard for biodiesel specification.

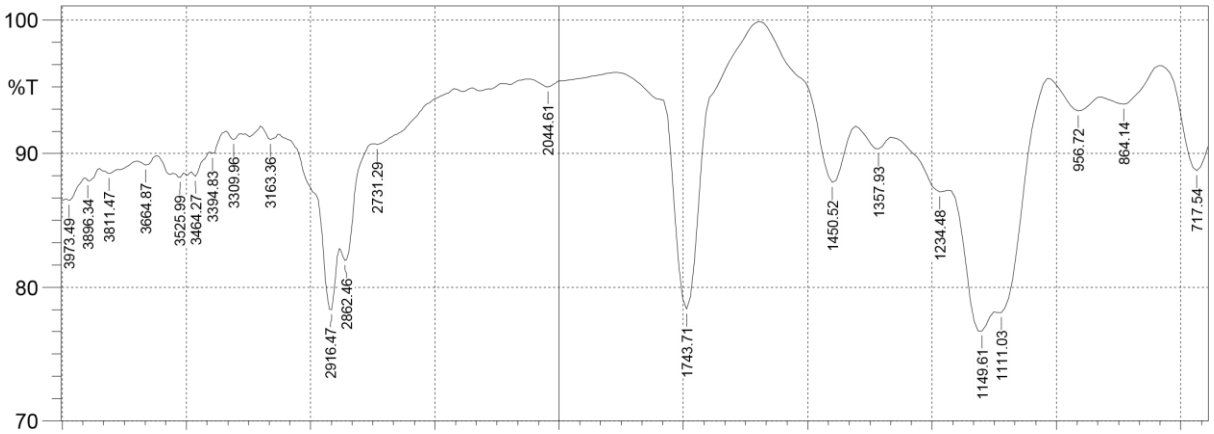

Figure I. FT-IR spectrum of the *Ziziphus abyssinica* seed oil

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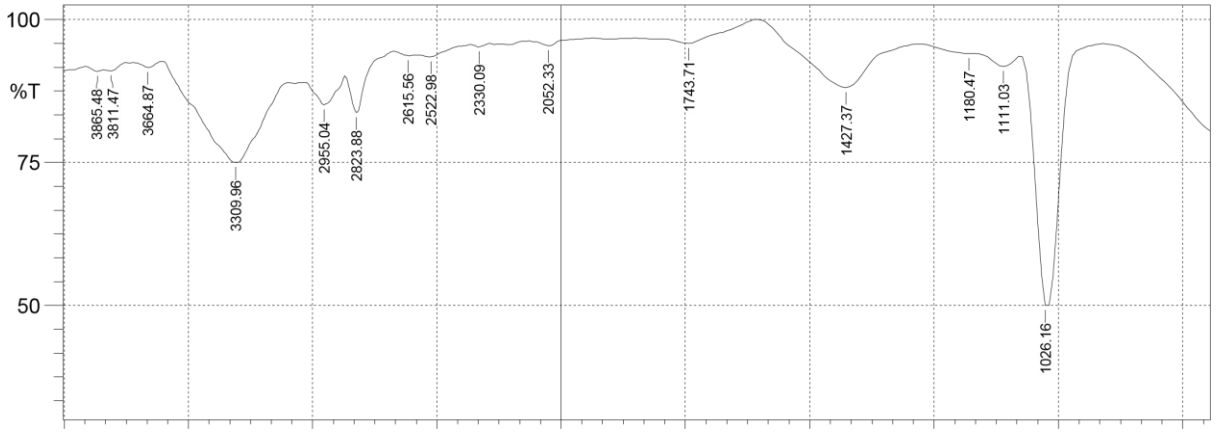

Figure 2: FT-IR spectrum of Magnesium oxide Catalyzed *Ziziphus abyssinica* Methyl ester

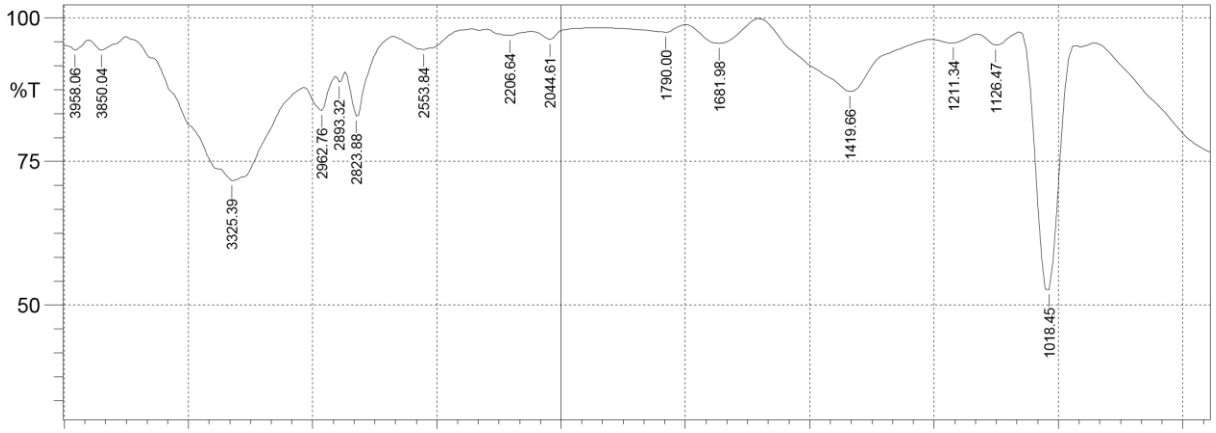

Figure 3: FT-IR spectrum of Magnetic Sand Catalyzed *Ziziphus abyssinica* Methyl ester

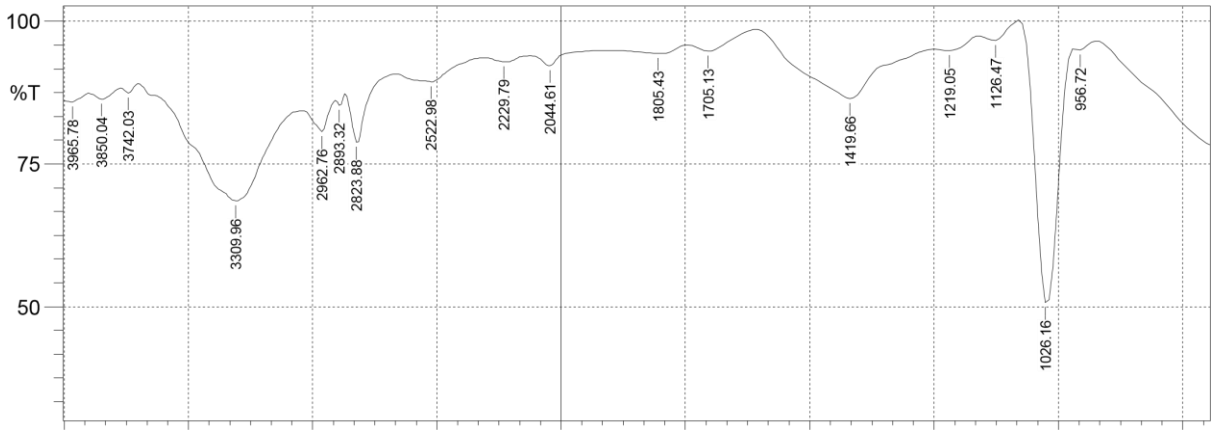

Figure 4: FT- IR Spectrum of MgO/Magnetic Sand Catalysed *Ziziphus abyssinica* Methyl ester

FT-IR Analysis

FT-IR spectrometry is a rapid and precise method for quantification of FAME. FT-IR spectrometry identifies the main functional groups presence at both the optimum produced biodiesel sample and its parent waste vegetable oil [21]. The most characteristic absorption peaks of the vegetable oil were indicated in

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Figure I. The sharp observed at 2862.46 cm^{-1} and 2916.47 cm^{-1} are characteristic of C-H stretches associated with the methane hydrogen atoms. The band at 1743.971 cm^{-1} was assigned to the C = O stretching of saturated ester [21]. The band at 1450.52 cm^{-1} corresponds to the characteristics of C-H of alkane bending

vibration and the band at 1149.61 cm^{-1} was attributed to the C-O bond stretching [21]. The absorption peak appearing at 717.54cm^{-1} is representative of-CH_2.

Figure 2 shows a dominant band at 3309.96 cm^{-1} that is due to the O-H stretching vibration, different intense peaks at such as the doublet of the - CH_3 and - CH_2 anti-symmetric stretching vibration at 2955.04 cm^{-1} and 2823.88 cm^{-1} were also detected [21]. C=O stretching vibration associated with acetate groups with a molecular vibration at 1743.71 cm^{-1} was detected and complemented by one intense peaks at 1454.61 cm^{-1} due to the -CH_3 bending vibration [13]. The peaks appearing at 1427.37 cm^{-1} which is the methyl ester group (CO-O-CH_3) and another intense peak was detected at 1026.16 cm^{-1} according to the asymmetric stretching mode of - C - O- C- of ester groups [21].

Figure III shows a dominant band at 3325.39 cm^{-1} that is due to the O-H stretching vibration, different intense peaks at such as the doublet of the - CH_3 and - CH_2 anti-symmetric stretching vibration at 2962.76 cm−1 and 2823.88 cm−1 were also detected [21]. C=O stretching vibration associated with acetate groups with a molecular vibration at 1790 cm−1 was detected and complemented by one intense peaks at 1454.61 cm−1 due to the -CH3 bending vibration [21]. The peaks appearing at 1419.66 cm−1 which is the methyl ester group (CO-O-CH_3) and another intense peak was detected at 1018.45 cm−1 according to the asymmetric stretching mode of - C - O- C- of ester groups [21].

Figure IV shows a dominant band at 3309.96 cm^{-1} that is due to the O-H stretching vibration, different intense peaks at such as the doublet of the - CH_3 and - CH_2 anti-symmetric stretching vibration at 2962.76 cm−1 and 2823.88 cm−1 were also detected [21]. C=O stretching vibration associated with acetate groups with a molecular vibration at 1705.13 cm−1 was detected and complemented by one intense peaks at 1419.66 cm−1 due to the -CH_3 bending vibration [21]. The peaks appearing at 1419.66 cm−1 which is the methyl ester group (CO-O-CH_3) and another intense peak was detected at 1026.16 cm−1 according to the asymmetric stretching mode of - C - O- C- of ester groups [21].

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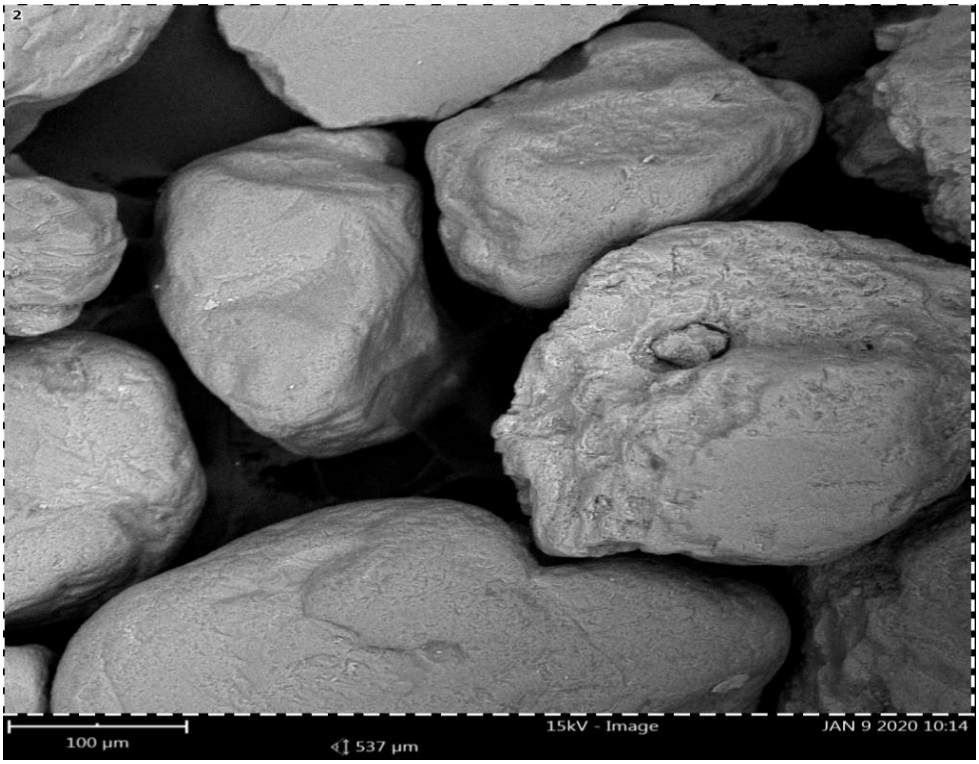

Figure 5: SEM of Magnetic Sand

Figure 6: SEM of MgO

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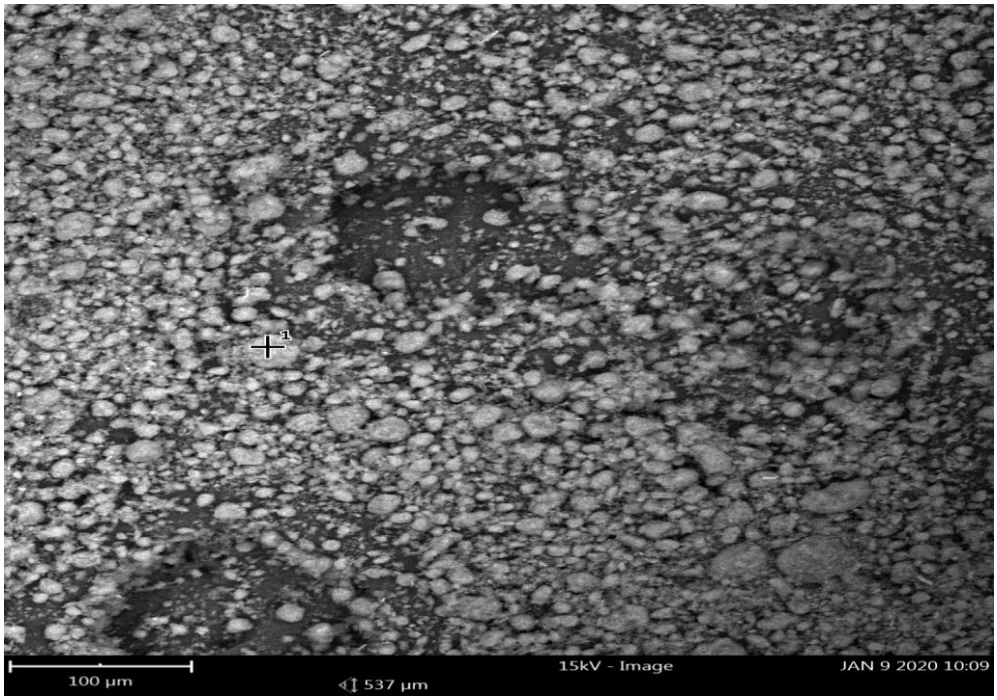

Figure 7: SEM of mixed Magnetic Sand and MgO

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Morphological analysis

The SEM image of sand, MgO and an/MgO particles are shown in Fig. 5. One excellent technique for displaying the surface morphologies of solid.

The sample a, b and c observed to have a definite morphology with a crystal structure (aligning with XRD pattern obtained), and the magnitude of the

crystalline was measured to be 100µm with a uniform distribution. It can be observed that there was a good dispersion from all the images from the samples. A closely and neatly packed spherical granule was found in the sand (a). It is observed that the b) MgO were highly agglomerated and porous in nature with uniform size distribution. Sample b and c formed a group of non-uniform crystalline particles. c) shows the sand and magnesium's structural shift from MgO to sand-MgO. It has been observed that the mixture of the two samples caused a substantial decrease in particle size, which may obviously account for the combined nature of the crystalline.

Figures 11, 12, 13 and 14 show *v*ariation of Oil Molar Ratio to Methanol, Variation of Reaction Time on Methyl ester Yield and Catalyst Dosage Variation. A common process for producing biodiesel is the transesterification reaction, which can be catalyzed by either homogeneous or heterogeneous catalysts. The homogeneous alkaline catalyst is the most well-

the finding of this study shown that the Pour point, cloud point, flash point, density and the known catalyst utilized in the production of biodiesel. The choice of catalysts because of their increased kinetic rates of reaction. However, the development of different heterogeneous catalysts is currently underway due to the high cost of refined feedstocks and the challenges of using homogeneous alkaline catalysts to transesterify low quality feedstocks for the synthesis of biodiesel.

Heterogeneous catalysts are environmentally friendly, economical and have high chance for heat integration [8]. Because transesterification is a reversible reaction that results in the loss of esters and the creation of soap, a longer reaction time reduces the final product (biodiesel) [15]. Because oils become less viscous at higher reaction temperatures, the rate of reaction increases and the reaction time is decreased. Leung and Guo (2006) [16], however. insufficient amount of catalyst leads to the partial conversion of triglycerides into fatty acid esters.

Triglycerides are not fully converted into fatty acid esters when there is not enough catalyst present [16]. Triglycerides are converted into biodiesel at higher rates when the catalyst concentration is raised with oil samples is also increases. catalyst concentration above 5% the conversion starts within a short time and then stabilizes. The best results were obtained when 5% [23].

viscosity of the biodiesel were found to be within ASTM D6751 standard for

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CONCLUSION

Characterization of *Ziziphus abyssinica* oil methyl ester using FT-IR technique have been studied. The results indicated that biodiesel could be produced via transesterification of *Ziziphus abyssinica* using a crude magnetic sand solid catalyst as heterogenous catalyst. Soap formation was not observed in this research, which reduces the cost of manufacturing of biodiesel.

The 5 wt% catalyst amounts of crude Fe_2O_3 was sufficient for producing methyl ester content with promising yield. The 5 hours of reaction time gave the highest methyl ester yield and conversion of oil to biodiesel at 60^{o}C using 15:1 methanol/oil molar ratio.

The results are consistent with those of other researchers and agree with international standards.

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