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Article

Synthesis, Characterization and Catalytic Application of Magnetic Iron Nanoparticles (Fe_3O_4) in Biodiesel Production from Mahogany (Khaya Senegalensis) Seed Oil

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Abstract: Magnetic iron nanoparticles (Fe_3O_4) were synthesized and characterized using Fourier Transformed Infrared ((FT-IR), UV-Visible spectrophotometer, Scanned Electron Microscopy (SEM) equipped with an Energy Dispersive X-ray spectrometer (EDX), and X-ray Diffraction (XRD). The synthesized nano catalyst was used in the transesterification of mahogany seed oil with methanol. The optimized reaction conditions gave a reaction yield of 88% at a catalyst concentration of 1.5% wt., a volume ratio of methanol to oil of 5:1, a reaction temperature of 60 °C, and a reaction time of 120 minutes. The Fe_3O_4 nanoparticles was regenerated from the mixture and reused for various circles by applying the optimum conditions obtained during the present study. The results showed that the biodiesel yield decreased by increasing the number of cycles when the regenerated catalyst was used. However, good conversion (81.9%) was obtained up to the 5th cycles. The elemental analysis of the synthesized magnetic iron nanoparticles Fe_3O_4) revealed the highest proportion of iron with 64.37 and 74.40% for atomic and weight concentration respectively, followed by oxygen with 34.27 and 24.50% for atomic and weight concentrations respectively. It could be concluded that the synthesized nano catalyst would serve as an excellent catalyst for the transesterification of vegetable oils.

Keywords: Nanoparticles, Optimization, Biodiesel, Magnetic Iron

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1. Introduction

Catalysis is one of the applications of nanoparticles [1]. Various elements and materials like CuO, aluminium, iron, titanium dioxide, clays, and silica all have been used as catalysts in nanoscale for many years [2-4]. Nano catalyst is a material from nanotechnology with the size ranging between 1-100nm [5]. Some researchers have studied about the production of biodiesel by using Nano catalyst [6-10]. It has been recently discovered that, Nano catalyst has become the focus for an efficient biodiesel production because of high surface area, high catalytic efficiency and resistance to saponification along with good rigidity [11-14]. The particle size of the catalyst that is used in biodiesel production is one of the most important factors for its catalytic activity [15]. Some studies have used Nano catalyst in biodiesel production because of it has a high surface area [16]. Many studies have confirmed that catalyst with lower particle size and higher surface area accelerate reaction rates due to an increased number of molecules that have minimum required energy for the reaction to occur [17-21].

The resources of petroleum as fuel are dwindling day by day and due to increasing demand for fuels, as well as an increase in stringent regulations on the use of petro diesel pose a challenge to science and technology [22]. Therefore, the need to provide an alternative fuel, keep the environment clean and reduction of any form of pollutant has become a global concern. Petro diesel is non-renewable and pollutes the environment by emitting greenhouse gases [23] and this calls for investigations into a renewable and non (minimal) polluting form of diesel known as biodiesel.

2. Experimental

2.1. Materials

The main materials used in this work were $FeCl_3$, $FeCl_2$, NH_3 25%, HCl, doubly distilled water, all of which were of analytical grade and were purchased from Sigma Aldrich.

2.2. Method

2.2.1. Synthesis of magnetic iron nanoparticles (Fe_3O_4)

The nanoparticles were synthesized by coprecipitation technique as reported [24] as follows: 4.24 g of $FeCl_3$ and 1.52 g of $FeCl_2$ were dissolved in 50 ml of double distilled water and was heated with stirring at 50 – 60 °C for 30 minutes for complete dissolution, then 30 ml of 25% NH_3 was added drop wise to the solution with vigorous stirring at 50 – 60°*C*, for 30 min. A black precipitate formed was separated using a magnet. The precipitate was washed two times using double-distilled water. Next, 0.01 M HCl was added until a pH of 7 was obtained indicating the neutralization of anionic charges on the nanoparticles. The precipitate was separated again using a magnet and then dried in air.

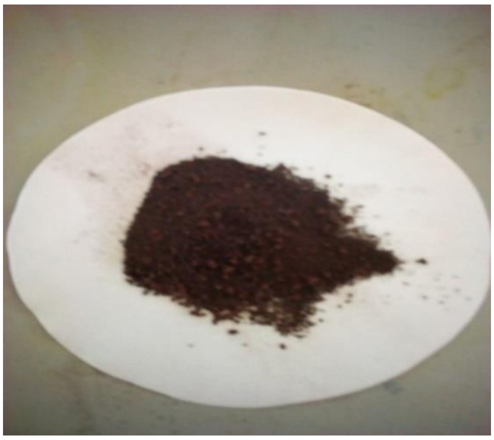

Figure 1. Synthesized magnetic iron nanoparticles (Fe_3O_4)

2.2.2. Characterization of Fe_3O_4 Nano Particles

The Nano catalyst was characterized using UV Spectroscopic technique, Fourier Transform Infra-red spectroscopy, SEM and XRD for its absorption, functional group determination, surface morphology and crystallinity respectively.

2.2.3. Transesterification Process of *Khaya senegalensis* Seed Oil Using Fe_3O_4 Nano Particles

The method adopted was that of Bolaji *et al*. [12] with slight modifications as follows:

The reaction was carried out in a 250 ml three- necked flask. A Liebig condenser was inserted into the middle neck; a thermometer was inserted into the second neck; while the third neck was used for sample introduction and withdrawal. First, the mahogany seed oil was introduced into the reaction flask and boiled at 50 °C. The catalyst was dissolved in the methanol and the mixture added to the reaction flask containing the oil. A stirring bar was introduced into the measured amount of the oil in the three- necked flask. The flask and its content were heated on a hot plate equipped with a magnetic stirrer. The volume ratio of mahogany seed oil to methanol were varied from 1:1 to 6:1. The Fe_3O_4 Nano catalyst concentration was varied from 0.5 wt% to 3.0 wt%. The reaction temperature was varied from 10°C to 80 °C. The reaction was allowed to run for various reaction times from 30 min to 180 min, for the transesterification process. The batches of the samples were withdrawn and poured into a separating funnel and allowed to settle under gravity for 12 h. Two layers were formed after settling down; a top layer containing the biodiesel and a bottom layer of glycerol. The glycerol was drained from the bottom of the separation funnel, leaving behind the crude biodiesel. To ensure the high purity of the biodiesel, a rotary evaporator was employed to remove excess methanol from the biodiesel. The biodiesel was further washed four times with distilled water to remove the catalyst. The high-purity biodiesel was dried with anhydrous sodium sulfate and the percentage of biodiesel yield was calculated.

The % yield of the methyl ester produced was calculated using the equation below:

$$\%\ \text{Yield} = \frac{\text{Volume of Biodiesel}}{\text{Volume of mahogany oil}} \text{X}\ 100\% \tag{1}$$

Figure 2. Collected sample showing the biodiesel layer on top and glycerol at the bottom

3. Results and discussions

3.1. UV Analysis

Here, the wavelengths of the sample were varied from 180 to 540nm. After each run, the absorbance was recorded and the wavelength adjusted before proceeding to the next. Figure 3 shows the graph of absorbance against wavelength for Fe_3O_4. The maximum absorbance was recorded at 360nm and then decreases gradually, which is slightly lower than those reported in the literatures which is 365nm [25]. This slight difference might be as a result of the environmental working conditions.

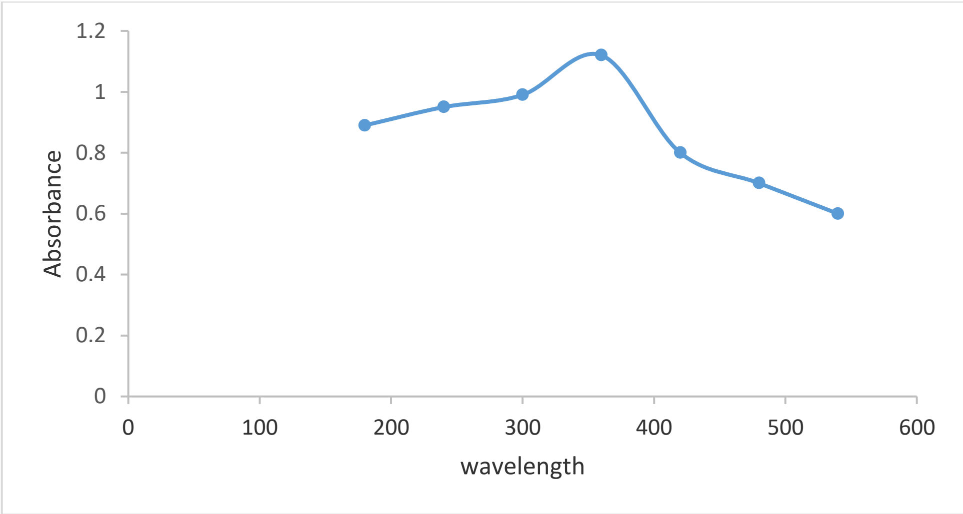

Figure 3. Graph of absorbance against wavelength for Fe_3O_4

3.2. FT- IR Analysis

FT-IR is used to determine the different functional groups such as alcohol, alkane, alkynes, alkenes and other such groups present in the substance which here is Fe_3O_4 nano catalysts as shown in Figure 4.

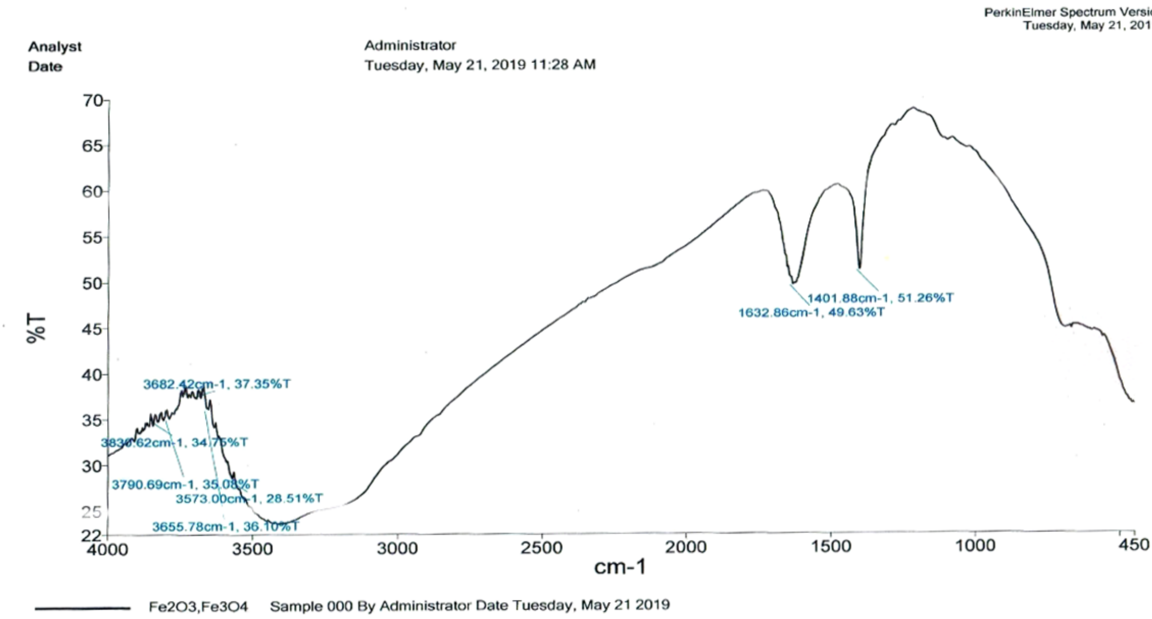

Figure 4. FT-IR Spectrum of the Magnetic iron nanoparticles

The FT-IR studies of magnetic nanoparticles (Table 1) shows that the weak and characteristic peak of carbon-carbon double bond C=C is present at 1401.88 cm-1. The sharp and medium intensity of O-H due to water is present in the range of 3790.69 cm^{-1} on the surface of magnetic iron nanoparticles. The sharp and characteristic peak of O-H stretch is presence around 3682.42 cm^{-1} in the synthesized nanoparticles. The sharp peak around 3573.00 cm^{-1} indicates the presence of N-H group. This result is in concordance with the ones reported by other researchers [26, 27]. The FT-IR results aside from being in concordance with the other researchers' findings, agree with the UV-Vis results indicating that the nanoparticles were successfully synthesized.

Table 1. FT-IR studies of Magnetic Iron Nanoparticle as a Catalyst

Peak(cm^{-1})	Functional group / Bond	V. Mode	Transmittance (%)	Intensity
3790.69	Water O-H	Stretch	35.08	Strong
3682.42	Alcohol O-H	Stretch	37.35	Sharp
3655.78	O-H	Stretch	36.10	Sharp
3573.00	N-H	Stretch	28.51	Sharp
1632.86	C = C	Alkene	49.63	Weak
1401.88	C = C	Aromatic	51.26	Weak

3.3. Scanning Electron Microscopy

The morphologies of the synthesized Fe_3O_4 catalyst were examined by Phillips Scanning Electron Microscope (SEM) XL- 30ESEM FEG, equipped with an Energy Dispersive X-ray spectrometer (EDX). The analysis of the synthesized catalyst using Scanning Electron Microscopy provided the morphology and size details of the Fe_3O_4 nanoparticles. It was identified that the shape of Fe_3O_4 nanoparticles showed a spherical and cubic morphology (Figure 5). It has a cubic inverse spinel structure consisting of a closely packed array of oxide ions which confirms the formation of iron oxide nanoparticles as reported by the previous researcher [27].

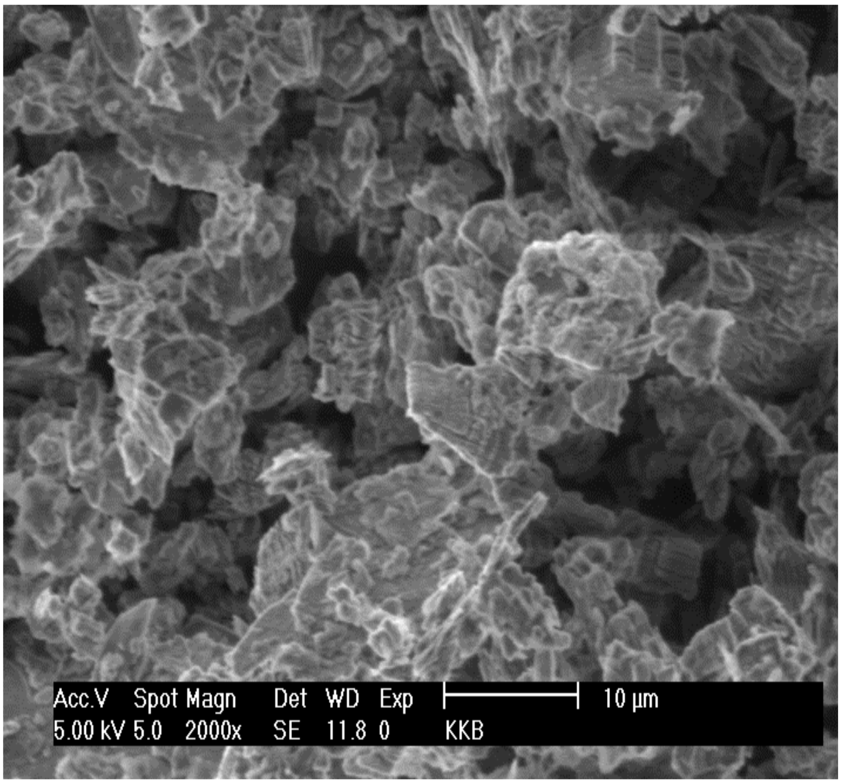

Figure 5. SEM image for Fe_3O_4 nanoparticles(FOV: 537 µm, Mode: 15kV - Image, Detector: BSD Full, Time: JUL 10 2019 15:22)

3.4. Energy dispersive micro analysis (EDAX)

The elemental analysis of the synthesized Fe_3O_4 Nano particles was studied using Energy Dispersive Micro Analysis (EDAX). The elemental analysis for magnetic iron nanoparticles (Fe_3O_4) revealed the highest proportion of iron with 64.37 and 74.40% for atomic and weight concentration respectively in the magnetic iron nanoparticles followed by Oxygen with 34.27and 24.50% for atomic and weight concentrations respectively followed by Chlorine with 2.13 and 1.10 for atomic and weight concentrations respectively as seen in Table 2. This result is in concord with the literature values [6, 27].

Table 2. Elemental Analysis for Fe_3O_4

Element Number	Element symbol	Element name	Atomic Concentration	Weight Concentration
26	Fe	Iron	64.37	74.40
8	O	Oxygen	34.27	24.50
17	Cl	Chlorine	2.13	1.10
13	Al	Aluminium	0.14	0.16
14	Si	Silicon	0.12	0.34

3.5. XRD Analysis

XRD measurements were carried out to analyze the crystalline structure of the magnetic iron nanoparticles (Figure 6) and the result reveals that Fe_3O_4 has the face- centered cubic spinel structure based on the 32 O^{2-} ions and closely packed along the (111) direction. The XRD results of the magnetic iron nanoparticles showed that it has a cubic inverse spinel structure that consists of a cubic closely packed array of oxide ions where all the Fe^{2+} occupy half of the octahedral sites and the Fe^{3+} are split evenly across the remaining octahedral sites. This result agrees with the one reported in the literature [28].

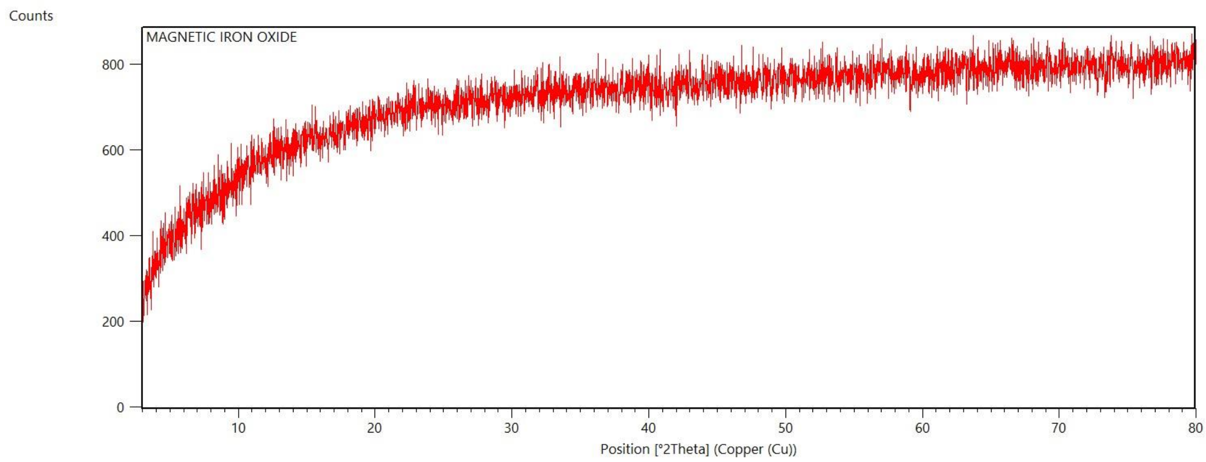

Figure 6. X-Ray Diffraction image of Magnetic iron nanoparticle

3.6. Effect of Optimization Parameters on % Yield of Methyl Ester Using Magnetic Iron nanoparticles (Fe_3O_4) as a Catalyst

3.6.1. Effect of Reaction Temperature

Transesterification reaction was performed for 120 min at 5:1 volume ratio with 1.5 wt% of magnetic iron nanoparticles (Fe_3O_4) at different temperature ranges with 10°C interval starting from 30 to 80°C. From the graph, it was observed that as temperature increases, reaction also increases hence the yield also increases up to 60°C and then decreases because higher reaction temperatures cause methanol to vaporize, resulting in decreased yield. This trend though similar to the one reported by the existing research, slightly varies in percentage yield as the highest conversion was recorded at a reaction temperature of 60°C for this research while for the previous research, the highest conversion was seen at a reaction temperature of 55°C [29]. This slight variation may be attributed to environmental factors.

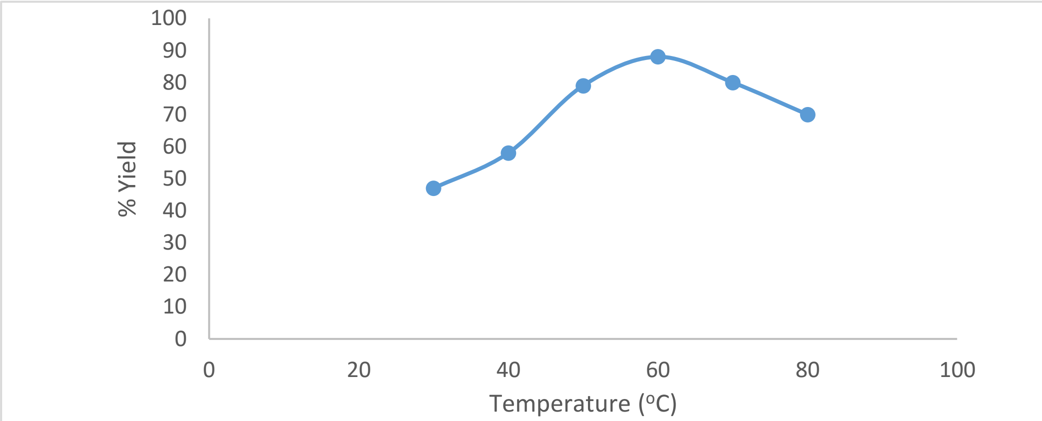

Figure 7. Effect of reaction temperature on yield with 5:1 methanol to oil ratio with 1.5wt% catalyst for 120 min for temperatures 30 to 80

3.6.2. Effect of Catalyst Concentration

Optimization was carried out using magnetic iron nanoparticles concentrations of 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0 wt% at reaction temperature of 60°C with volume ratio of 5:1 for 120 min reaction time. As the catalyst increases the yield also increases up to 1.5 wt% and then decreases gradually because addition of excess catalyst caused more triglycerides participation in the saponification reaction, resulting in increased production of soap and reduction of the methyl ester yield. This result exhibited the same trend with the one reported by another researcher's findings [30] indicating that the nano catalyst synthesized is efficacious in its catalytic activity. The trend further indicates that any increase in concentration of catalyst beyond the neutralization limit results in a decrease in methyl ester conversion. On the other hand, the more the amount of the catalyst greater than the optimum amount caused higher mass transfer resistance due to the highly viscous mixture, resulting in lower methyl ester yield which is in line with the literature report [30].

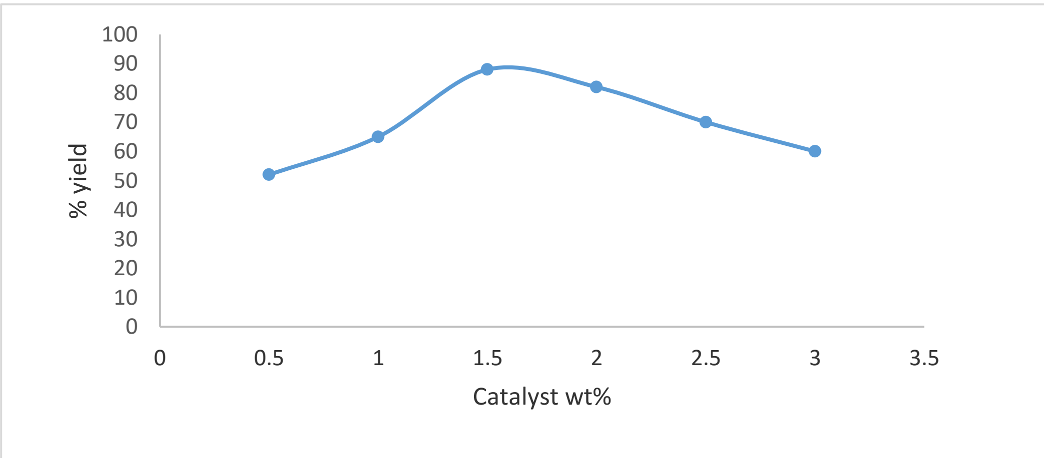

Figure 8. Effect of catalyst concentration with yield at 5:1 methanol to oil volume ratio for 120 min at 60°C for concentration 0.5 to 3.0wt

3.6.3. Effect of Reaction Time

The effect of reaction time for the transesterification process with percentage yield of biodiesel produced was also studied in this present study. The reaction time was taken from 30 to 180 Min with the interval of 30 min, keeping the optimum volume ratio 5:1, catalyst concentration of 1.5wt% with the previously found optimum reaction temperature of 60°C.The transesterification reaction proceeds quickly and around 58 % of ester was converted in 30 min. After one hour around 68 % of ester was produced, around 79% of ester was produced in 90 min, after 120 min, 88 wt% of ester was found and after that, it was observed that % yield of methyl ester was slightly reduced to 80% and 72% for 150 and 180 minutes respectively, this was as a result of the reverse reaction. This result is in conformity with the one reported by another researcher [31]. So, it can be observed from Figure 9 that the constant value of ester produced was observed after 120 minutes due to forward and reversed reactions. The summary of the effect of time on yield using magnetic iron oxide nanoparticle is given in Figure 9. So, it is concluded that 120 minutes reaction time is optimum and around 88% of ester was produced, and after that, ester production was almost constant up to 120 minutes of reaction time.

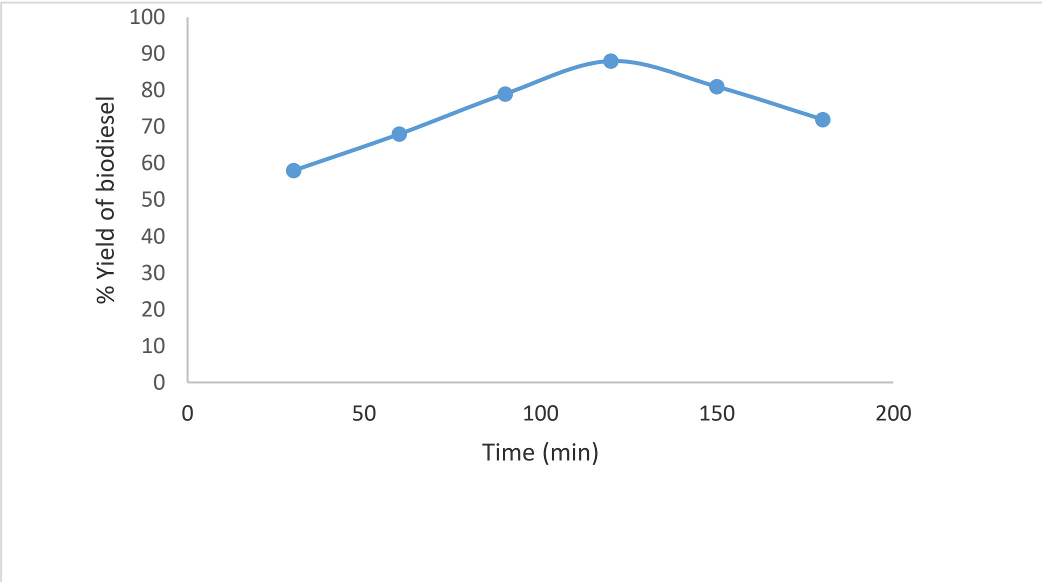

Figure 9. Effect of reaction time with yield at 5:1 methanol to oil molar ratio with 1.5wt% at 60°C with time interval of 30 min ranging from 30 to 180 minutes

3.6.4. Effect of Volume Ratio

Though there are many alcohols including ethanol, propanol, isopropanol that can be used in transesterification, methanol has been considered due to its low price and highly reactive nature in this present research. Volume ratio of methanol to oil was varied from 1:1 to 6:1 at 60 ° C reaction temperature, 1.5 wt% catalyst concentrations for 120 minutes reaction time in transesterification reaction. Starting with 1:1 volume ratio the yield started increasing for the catalyst because higher alcohol volume ratio interferes with the separation of glycerol because there is an increase of solubility. Figure 100 below summarized the effect of volume ratio on % yield of methyl ester. From the graph below, it is noticed that yield of 1:1 is 45%, 2:1 is 55%, 3:1 is 65%, 4:1 is 75% and 5:1 gave 88% which is the optimum yield, then a decrease in yield was observed from 6:1 methanol to oil volume ratio. This result agrees with the already reported findings of some researchers [32, 33]. However, it is worthy to note that the increase in alcohol to oil volume ratio above 5: 1 declines the biodiesel conversion. This is due to the reversibility behavior of transesterification reaction [33].

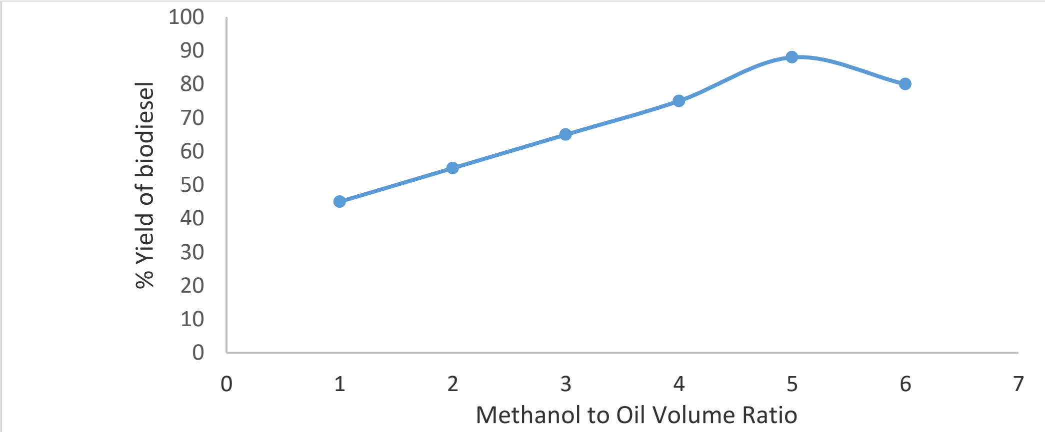

Figure 10. Effect of methanol to oil volume ratio on % yield at 60°C with 1.5wt% catalyst for 120 min

4. Conclusion

Magnetic Iron Nanoparticle was synthesized and characterized using different analytical tools / machines including FT-IR, UV spectrophotometer, SEM equipped with an energy dispersive X-ray spectrometer (EDX), and XRD. Subjecting the synthesized Nano particles into catalysis, biodiesel was produced from mahogany seed oil and methanol. The reaction conditions were optimized. The Fe_3O_4 Nano particle was efficient in the production of biodiesel of international standard. The yield of 88% was achieved as the optimum with catalyst concentration of 1.5 wt %, a volume ratio of methanol to oil of 5:1, a reaction temperature of 60 °C, and a reaction time of 120 min. All the biodiesel quality parameters determined agreed with the specifications for biodiesel quality standards, hence, Khaya senegalensis seed oil is a good feedstock for biodiesel production.

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