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RESEARCH ARTICLE

SYNTHESIS, CHARACTERIZATION AND APPLICATION OF Ni /SiO2 HETEROGENEOUS NANOCATALYST IN OPTIMIZATION OF METHYL ESTER FROM Khaya senegalensis SEED OIL

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ABSTRACT

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Ni / SiO_2 heterogeneous nanocatalyst was synthesized and characterized using different analytical tools including FT-IR, UV spectrophotometer, SEM equipped with an energy dispersive X-ray spectrometer (EDX), and XRD. Subjecting the synthesized heterogeneous nanoparticle into catalysis, Biodiesel was produced from mahogany seed oil and methanol using the synthesized nanocatalyst. The reaction conditions were optimized. The yield of 85% was achieved when the reaction was carried out using Ni / SiO2 with 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. The Ni / SiO2 nanocatalyst was regenerated from the mixture and was reused for various circles by applying the optimum conditions obtained during the present study. The results showed that the methyl ester yield decreased exponentially by increasing the cycle number when the regenerated catalyst was used. However, good conversion (>72%) was obtained up to the 4th cycles. All the biodiesel quality parameters determined agreed with the specifications for biodiesel quality standards, hence, Ni / SiO_2 nanocatalyst is catalytically active.

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INTRODUCTION

Nano catalyst is a type of nanomaterial with the size range of 1-100nm [1]. Recently, Nano catalysts have become the focus as efficient biodiesel production because of high surface area, high catalytic efficiency and resistance to saponification along with good rigidity [2]. The particle size of the catalyst that is used in biodiesel production is one of the most important factors for its catalytic activity [3]. Many studies have confirmed that catalyst with a lower particle size and higher surface area accelerate reaction rates due to an

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increased number of molecules that have minimum required energy for the reaction to occur. Generally speaking, Heterogeneous catalysis is a type of catalysis in which the catalyst occupies a different phase than the reaction mixture. But running a reaction under heterogeneous catalytic conditions has several advantages compared to other catalytic processes; easy handling, good selectivity (having multiple binding sites), safe to store, long life time, good thermal stability, rigid structure avoids the aggregation of active catalyst, insoluble in organic solvents and finally easy and inexpensive removal from reaction mixture by filtration or centrifugation. In another word, heterogeneous catalysts are catalysts that are in different phase with the reactants (the catalyst is usually in solid phase while the reactant is in liquid phase).They can be separated from final product by filtration and reused. This causes less consumption of both chemicals and time of course. Heterogeneous catalysts are noncorrosive. They have a high selectivity and can be easily separated from the products. The disadvantages of utilization of homogeneous catalysts in biodiesel production are saponification reaction and regeneration issue after the transesterification process is completed as well as producing toxic wastewater [4-5]. Biodiesel is one of the promising options for the alternative renewable fuels that can be used in existing engines [6] and nanoparticles are therefore used as catalysts for production of biodiesel. Nanoparticles of most transition metals have been synthesized and applied either as a catalyst, a reducing agent or as an antibacterial agent [7-11]. Nanoparticles can be synthesized either as monometallic or bimetallic nano particles [12-16]. In this research, the choice of Ni/SiO_2 nanocatalyst over others was conceived out of the fact that Ni/SiO_2 demonstrates special properties including lower particle size, higher surface area, multiple binding sites as well as good thermal stability. Hence, accelerates reaction rates [17].

EXPERIMENTAL SECTION

Materials

The main materials used during this work were Nickel (ii) nitratehexahydrate, silica gel, doubly distilled water, all of which were of analytical grade and were supplied from Sigma Aldrich.

Synthesis of Ni / SiO_2 as heterogeneous Nano catalyst

The catalyst was prepared by the impregnation method with slight modifications as reported by the literature [17]. This was achieved by dissolving 2.51g nickel (II) nitrate hexahydrate in 20ml of distilled water and adding it to 5.0g silica gel and stirring for 2 h using a magnetic stirrer at room temperature (20 ±1 ^oC). This was aged at room temperature overnight. The excess water was removed by heating the mixture on water bath and a Rota vapor was used under vacuum to evaporate water. The catalyst material was dried in an oven at 100–120 ^oC for 12 h. Figure 1 shows the synthesized Ni / SiO_2.

Characterization of Ni / SiO_2 heterogeneous Nano catalyst

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

Transesterification Process of Khaya senegalensis Seed Oil Using Ni / SiO_2 Heterogeneous Nano catalyst

Modified method of Balaji *et al.*, 2018 [18] was adopted as follows:

Mahogany seed oil was used as a` reactant in the biodiesel transesterification process. First the mahogany seed oil was boiled at 50°C. The reaction was carried out in a 250ml three necked flask. A Liebig condenser was attached onto the middle neck; a thermometer was inserted through the second neck; while the third neck was used for sample introduction and withdrawal. The catalyst was first dissolved in methanol. The measured amount of the oil and a stirring bar were introduced into the three necked flask. The flask and its content were heated on a hot plate equipped with a magnetic stirrer. Volume ratio of mahogany seed oil to methanol varied from 1:1 to 6:1. The Ni/ SiO_2 nanocatalyst concentration was 0.5wt% to 3.0wt%. The reaction temperature was varied from 10ºC to 80°C under standard conditions. The reaction was allowed to run for various reaction times, from 30 min to 180 min, to study the transesterification process. The sample was allowed to settle by gravity for 12 h after collection with a separation funnel, and it settled into a clear, liquid layer of biodiesel on top of a glycerol layer. The glycerol was drained from the bottom of the separation funnel, and the remaining crude biodiesel was washed four times with millipore water to remove the catalyst. To ensure the high purity of the biodiesel, a rotary evaporator was employed to remove excess methanol from the biodiesel. The high-purity biodiesel was dried with anhydrous sodium sulfate and the percentage of biodiesel yield was calculated. The standard ASTM D6751-02 methods were used in determining the properties of the biodiesel produced.

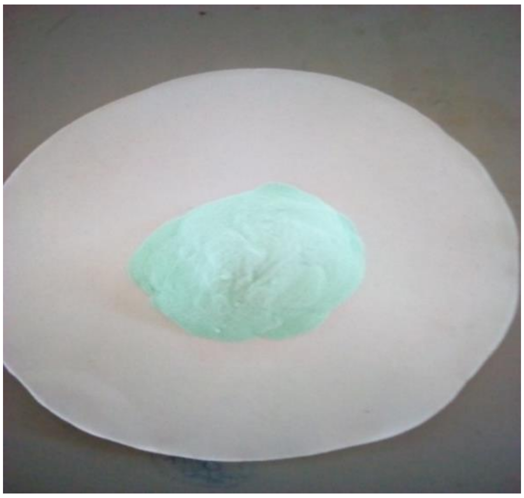


Figure 1. The synthesized Ni / SiO_2 heterogeneous nanocatalyst

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

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

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

Regeneration and Reusability of the Catalyst

Regeneration of the resulting heterogeneous nanocatalysts was performed by filtration method, as reported by the literature [19]. The residue which is the catalyst was washed with a methanol so as to remove biodiesel, glycerol and the oil molecules attached to the catalyst surface [19]. After the regeneration, the regenerated catalyst was tested for transesterification of the mahogany seed oil with methanol using the optimal conditions obtained during the present study.

Catalysts Reusability Test

The reusability of the catalysts was performed under the optimized reaction conditions of 1.5wt% catalyst loading, 60ºC reaction temperature, 5:1 methanol to oil ratio and 120 min reaction time. After completion of each run, the used catalyst was separated from the reaction mixture and was reused without further regeneration treatment.

RESULTS AND DISCUSSION

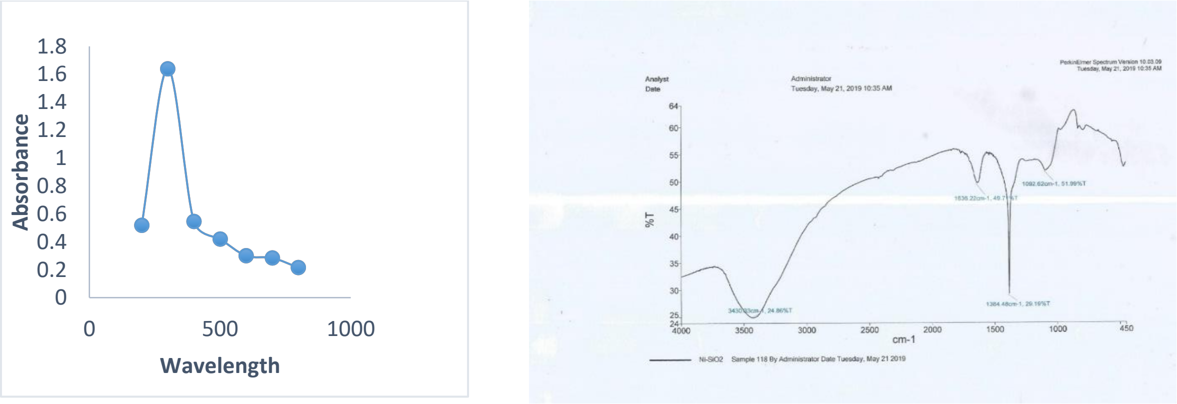
UV analysis

The wavelengths of the sample were varied from 200 – 800nm. After each run, the absorbance was recorded and the wavelength was adjusted before proceeding to the next. Figure 2 shows the graph of absorbance against wavelength for Ni / SiO_2. The highest absorbance was noticed at 300nm and then there was a decrease in the absorbance (see figure 3) which might be attributed to the reduction of Ni (+2) to Ni (+0). This trend agrees with the one reported by some researchers [19-20].

FT- IR Analysis

FT-IR is used to determine different functional groups such as alcohol, alkane, alkynes, alkenes and other such groups present in the substance which here is Ni / SiO_2.

FT-IR studies presented in Table 1 on Ni / SiO_2 catalyst shows a sharp band at 1384.48 cm^{-1} on the catalyst. This band is due to the presence of Ni-O and a broad band at 3430.33 cm^{-1} is due to N-H stretch. The absorption band in the range of 1092.62 cm^{-1} indicates the presence of C-OH group. Similarly, the bands at 802 cm^{-1} and 455cm^{-1} is due to Si-O stretching and bending vibration respectively. The FT-IR results are in good agreement with the one reported by another researcher (21-22).



(a) UV image

(b) FT-IR Spectrum

Figure 3. UV and FT-IR images for Ni / SiO_2 nanocatalyst

Table 1. FT-IR studies of Ni / SiO_2 Heterogeneous nanocatalyst

Peak(cm^{-1})	Bond	Mode	Transmittance (%)	Intensity
802	Si-O	Stretch	26.16	Strong
1384.48	Ni-O	Stretch	29.19	Strong
1636.22	C-C		49.7	Strong
1092.62	C-OH	Stretch	51.99	Strong

SEM and XRD Analysis

The morphologies of the synthesized Ni / SiO_2 catalyst was examined by scanning electron microscope (SEM) 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 Ni / SiO_2 nanoparticles. It was identified that shapes of Ni / SiO_2 nanoparticles gave a closely packed but porous and well dispersed surface morphology which is slightly different with the one reported by another researcher[22]. This slight variation is possibly due to environmental working conditions. In the same vein, XRD measurements were carried out to understand the crystalline structure of Ni / SiO_2 catalysts, and the results were presented in figure 5(b). Patterns of Ni / SiO_2 exhibited a broad and large peak around 22∘, which was attributed to amorphous silica of support. The sample showed the fcc NiO phase, with typical reflections of the (111), (200), and (220) planes at 2θ = 37°, 43°, 63°, respectively. This result is in conformity with the one reported by another researcher [23].

Energy dispersive micro analysis (EDAX)

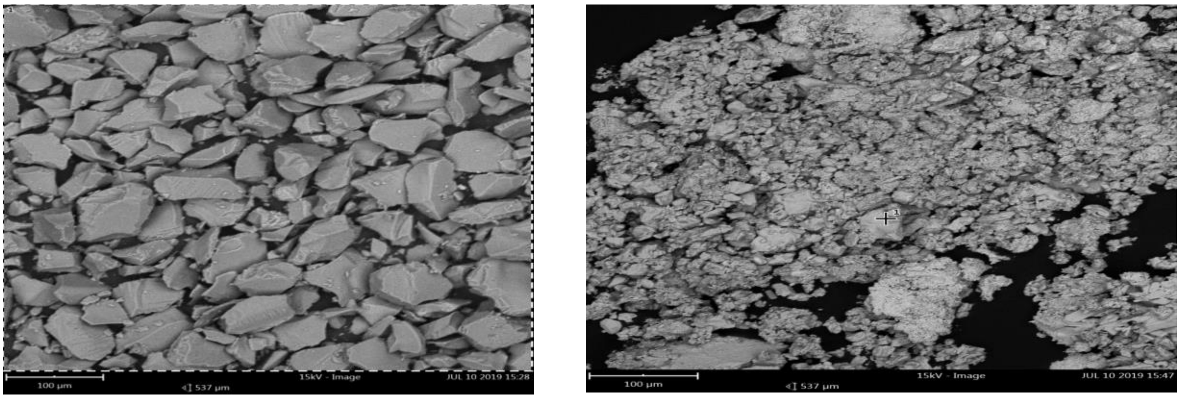
The elemental analysis of the synthesized Ni / SiO2 nanoparticles was studied using energy dispersive micro analysis (EDAX). The analysis revealed highest proportion of silicon with 78.39 and 66.91 for atomic and weight concentrations respectively in the nanoparticles followed by nickel with 15.92 and 28.40 for atomic and weight concentrations respectively. It was followed by nitrogen with 3.89 and 3.23 for atomic and weight concentrations respectively (table 2). The structure is formed by nano-sized silica particles linked together, resulting in a random arrangement of holes and cavities. The EDS spectrum of Ni / SiO2 contains signals belonging to Si and O due to the SiO2 support. In addition to O and Si signals, the EDS spectrum of Ni / SiO2 showed signs of Ni suggesting that the Ni was successfully impregnated on the silica support [22].

The effect of optimization parameters on % yield of Methyl Ester using Ni/ SiO_2 as a catalyst

The effect of the following process parameters were studied using Ni/ SiO_2 as heterogeneous nanocatalyst using optimum conditions obtained earlier during the present study

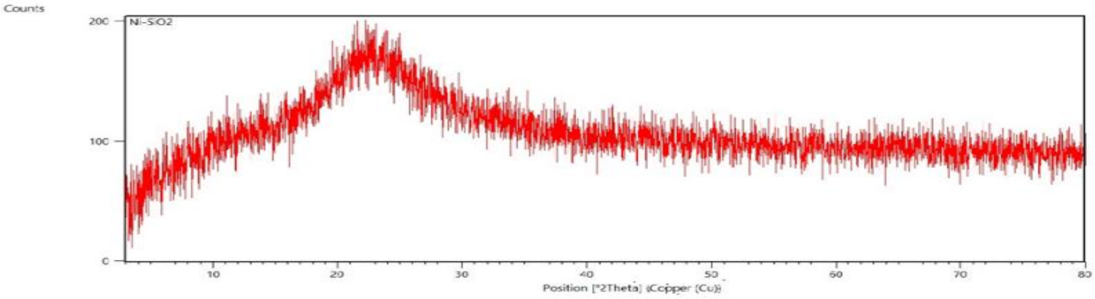
The effect of reaction temperature

Transesterification reaction was performed for 120 min at 5:1 volume ratio with 1.5 wt % of catalyst (Ni/ SiO_2) at different temperature ranges with 10°C 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. In other words, the boiling point of methanol is 64.7 °C and in order to avoid alcohol evaporation, reaction temperature has to be less than 64.7 °C. Increasing the temperature will reduce the viscosity of the oil which leads to a sufficient contact at the active site of catalyst surface between the oil and the methanol. Reaction temperature was a significant factor in biodiesel synthesis as the yield increased significantly for all the two catalysts. Increasing reaction temperature reduces the viscosity of *Khaya senegalensis seed* oil and enhances the product yield because of the inhibition of mass transfer resistance which agrees with the other researcher's report [24].



(a) **SEM image of the unused Ni/SiO₂**

(b) **SEM image of the last regenerated Ni/SiO₂**



(c) **X-Ray Diffraction image of Ni / SiO₂**

Figure 4. SEM and XRD image for Ni / SiO2 nanocatalyst

Table 2. Elemental Analysis for Ni / SiO2 nanocatalyst

Element Number	Element symbol	Element name	Atomic Concentration	Weight Concentration
14	Si	Silicon	78.39	66.91
28	Ni	Nickel	15.92	28.40
7	N	Nitrogen	3.89	3.23
6	C	Carbon	1.03	0.73
11	Na	Sodium	0.75	0.72
8	O	Oxygen	1.9	0.83

The effect of catalyst concentration

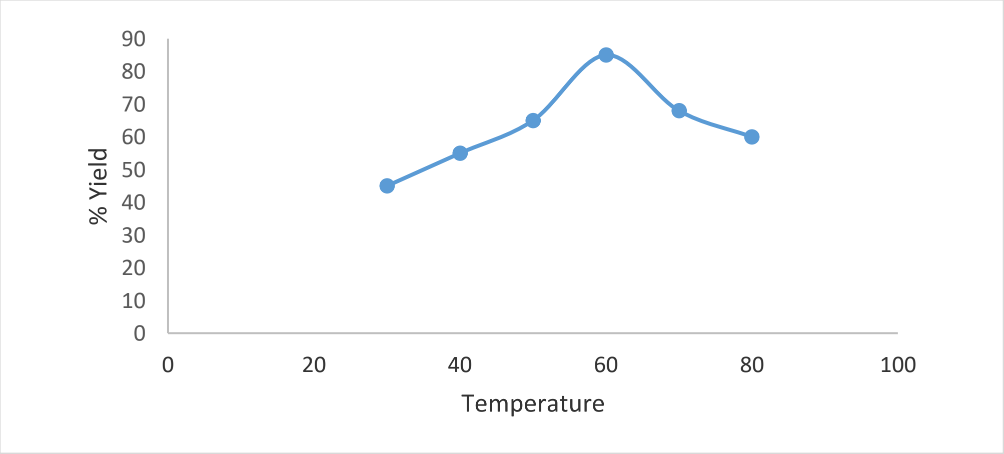
Catalyst concentration is the key variable which enhances the yield of methyl ester production. In general, as the catalyst concentration increases, the conversion of triglycerides is also increased. This is because an insufficient amount of catalyst results in an incomplete conversion of triglycerides into fatty acid esters. That is why optimization was carried out using Ni/SiO_2 concentrations of 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0wt% 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 1.5wt% and then decreases gradually because the addition of excess heterogeneous catalysts causes more triglycerides participation in the saponification reaction, resulting in increased production of soap and reduction of the methyl ester yield. So, any increase in concentration of catalyst beyond the neutralization limit results in decrease in methyl ester conversion [24].

The effect of Reaction Time

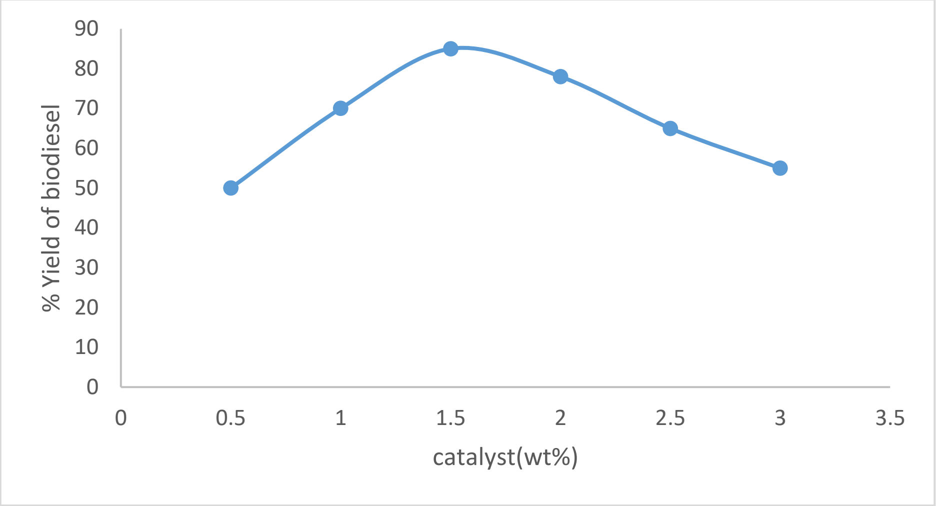
In the present study, the effect of reaction time on transesterification process with percentage yield of biodiesel produced was also evaluated. The reaction time was taken from 30 to 180 Min., with the interval of 30 min., keeping the optimum volume ratio 5:1, catalyst concentration of 1.5wt% with the previously found optimum reaction temperature 60°C.The transesterification reaction proceeds quickly and around 55 % of ester was converted in 30 min. After one hour around 70 % of ester was produced, after 90 min around 80% of ester was produced, after 120min, around 85% was found and after that slightly reduced percentage of ester was observed, this phenomenon might simply have resulted from the exhaustion of methanol due to the longer time taken. Graphically, it can be seen in figure 8, that the constant value of ester produced was observed after 120 min as a result of forward and reversed reactions. So it is concluded that around 120 min, reaction time was optimum and we got around 85% of ester production and after that, ester production decreased drastically. This result is in agreement with previous research [25].

The effect of volume ratio

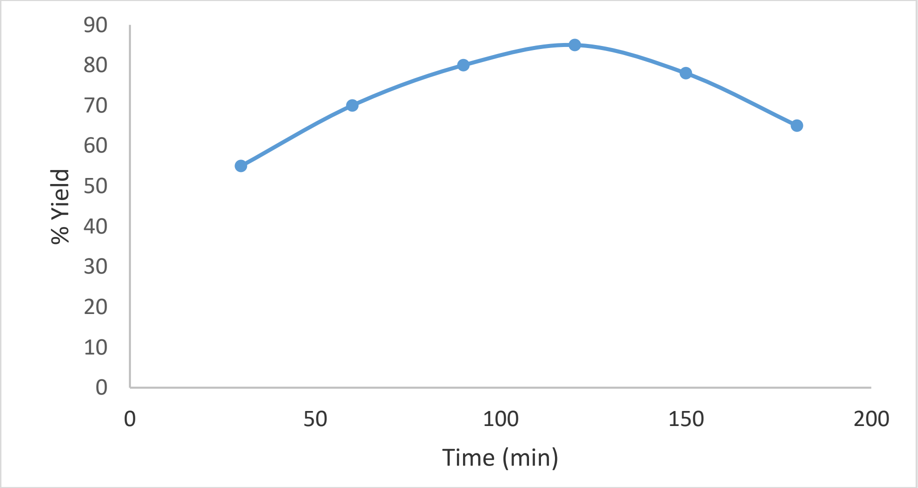
The stoichiometry of the reaction generally, requires 3 moles of methanol per mole of triglycerides to yield 3 moles of methyl ester and 1 mole of glycerol. The alcohol may be methanol, ethanol, isobutanol and isopropanol. In this present study, methanol has been considered due to its low price and highly reactive nature. Volume ratio of methanol to oil was varied from 1:1 to 6:1 at 60^o C reaction temperature of 1.5wt% catalyst concentrations for 120 min 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. From the graph it is noticed that yield of 1:1 is 40%, 2:1 is 50%, 3:1 is 65%, 4:1 is 73% and 5:1 gave 85% which is the optimum yield, then a decrease in yield was observed from 6:1 methanol to oil volume ratio which although exhibited the same trend, yet slightly differs from the earlier research [25].



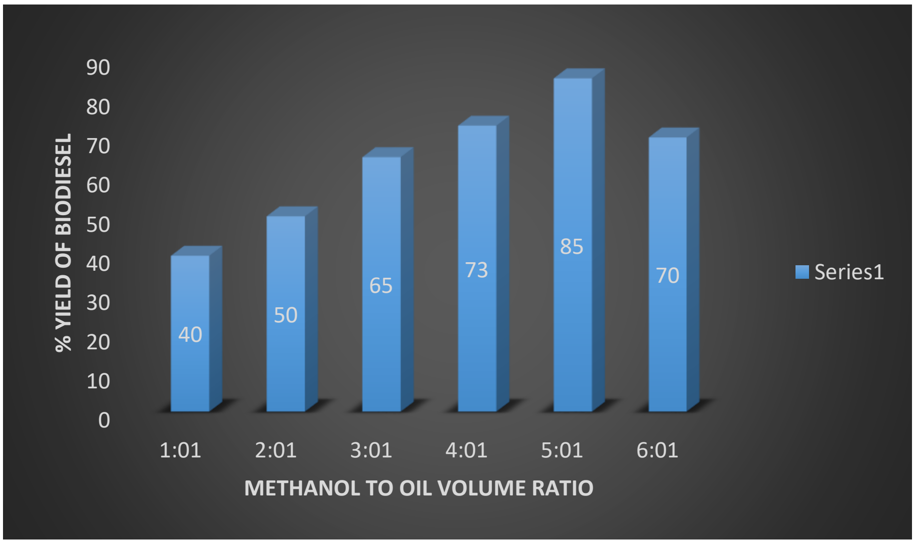
(a) Effect of Temperature



(b) Effect of Catalyst



(c) Effect of Time



(d) Effect of Methanol to Oil Volume Ratio

Figure 5. The effect of Temperature, Catalyst, Time and Methanol to oil Volume ratio respectively on the % yield

Recoverability and reusability of the synthesized heterogeneous nanocatalysts

The most important characteristics of the heterogeneous nanocatalysts are their recoverability and reusability. Based on this fact, the utilized catalysts were regenerated and examined for transesterification of mahogany seed oil with methanol for further cycles by applying the optimum conditions obtained during the present study as shown in figure 10. The results pointed out that the methyl ester yield decreased exponentially by increasing the cycle number when the regenerated catalyst was used. However, good conversion (>72%) was obtained up to the 4th cycle for Ni /SiO_2 nanocatalyst. The drop in biodiesel yield in the fourth cycle was due to deactivation of catalyst sample, which might be as a result of adsorption of fatty acid, glycerol or water molecule on the active sites of the catalyst [26]. In another words, the reduction in the methyl ester yield with increasing the regeneration runs could be attributed to the leaching of active metals from the support surface. As a result, the number of the active sites available for the reaction will be less in number. These achievements are in concord with the previously reported data by other researchers [27]. So it can be seen that, the catalyst in question was regenerated and reused producing good yield up to a good number of cycles (four cycles) which explains why the choice of this catalyst over other catalysts is quite paramount.

CONCLUSION

Ni / SiO_2 heterogeneous nanocatalyst was synthesized using the method outlined earlier 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 heterogeneous nanocatalyst into catalysis, biodiesel was produced from mahogany seed oil and methanol using the synthesized nanocatalyst. The reaction conditions were optimized. The Ni / SiO_2 nanocatalyst was efficient and reusable in the production of biodiesel of international standard. The yield of 85% was achieved as the optimum with 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. The nanocatalyst was regenerated from the mixture and reused for various circles by applying the optimum conditions obtained during the present study as shown in figure 11. The results pointed out that the methyl ester yield decreased exponentially by increasing the cycle number when the regenerated catalyst was used. However, good conversion (>72%) was obtained up to the 4th cycles. All the biodiesel quality parameters determined agreed with the specifications for biodiesel quality standards, hence, Khaya senegalensis seed oil is a good biodiesel feedstock.

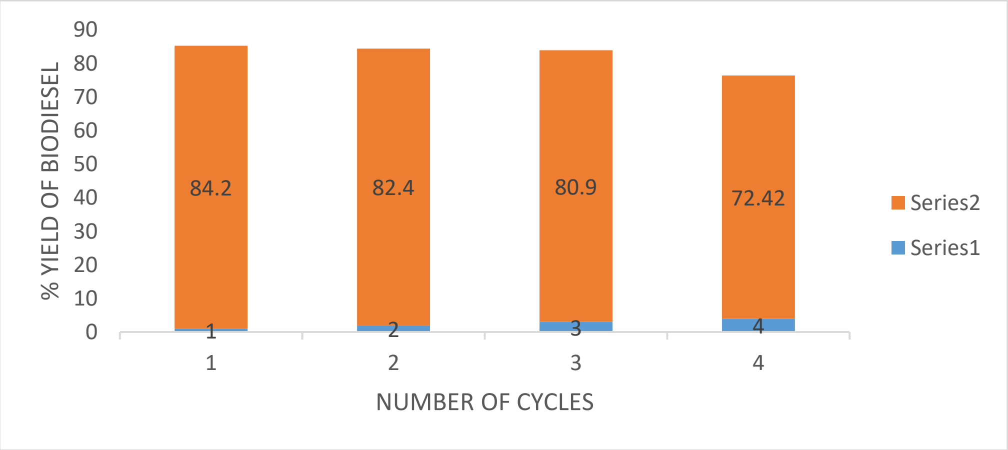


Figure 6: The influence of catalysts (Ni / SiO_2) reusability on % yield of biodiesel

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