Production of biodiesel from chicken fat using NiFe$_2$O$_4$/GO magnetic catalyst and combining it with diesel in order to improve fuel properties

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ABSTRACT: Biodiesel is one of the sources of renewable fuel. Due to increasing environmental pollution, global warming caused by fossil fuels and limited fossil fuel resources, its production has significantly increased during the last decades. In addition, low price renewable sources have been wisely used to produce biodiesel. Here in, biodiesel was produced using chicken fat in the presence of Nickel ferrite nanoenabled graphene oxide nano-catalyst. Also the effect of various parameters like temperature, reaction time, catalyst amount and methanol to oil ratio was investigated on the biodiesel production. The results showed that the best conditions for biodiesel production were obtained such as the temperature of 65 °C, methanol to oil ratio of 1:9, the catalyst amount of 1 wt.% and reaction time of 5 h which in these conditions the biodiesel efficiency was determined 95%. The produced biodiesel is mixed with different ratios of petroleum diesel to improve the physical properties of the produced biodiesel (B25, B50 and B75), such as flash point, kinematic viscosity, density, cloud point and pour point. The results showed that the mixture ratio of B75 and B100 had density and viscosity in the range of standard. Additionally, this fuel should not be used in cold weather since its pour point is greater than zero.

Keywords: *Biodiesel, Chicken fat, Nickel ferrite nanoenabled graphene oxide nanocatalyst, Transesterification*

INTRODUCTION

Manufacturers of petrochemical products face multiple demands for fossil fuels. According to reports, the average annual increase in the consumption of fossil fuels has reached 1.5%, which has led to an increase in the price of biodiesel[1]. Recently, the use of renewable sources such as biodiesel to replace fossil fuels has been proposed. Biodiesel is an environmentally-friendly non-toxic fuel that emits very small amounts of harmful compounds upon burning. Biodiesel is produced via transesterification process in the presence of a catalyst[2]. Various sources of feedstock for bio-diesel production include herbal oils, waste edible or cooking oils, and animal fat[3]. Direct competition in the supply of foodstuff for humans, herbal oils increase the overall cost of biodiesel generation[4]. This has made the animal fats and waste edible oil (WEO) more affordable for biodiesel production. Among animal fats, chicken fat[5], lamb fat and goat fat have been used as oil sources for biodiesel production[6].

Various catalysts have been used to produce biodiesel, including, acidic, alkaline, and enzymatic catalysts. Previous studies have indicated high yield of biodiesel production using alkaline catalysts[7]. The homogeneous catalysts produce high biodiesel yield at

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mild temperature and less time; however, they have some drawbacks such as reusability, catalyst separation, and production of excess wastewater during the transesterification process. To solve these problems, heterogeneous catalysts are preferred because they reduce the cost of purification and separation[8].

Different heterogeneous alkaline catalysts have been applied for biodiesel production, including graphene oxide/TiO_2, MgO, CaO/$CuFe_2O_4$, AC/$CuFe_2O_4$@CaO, magnetic charcoal, etc[9, 10]. Nowadays, magnetic catalysts and nanocatalysts have received special attention due to their unique properties[11].

Graphene oxide (GO) have received much attention because of its easy accessibility and compatibility with different materials. GO is relatively inexpensive, mechanically strong, and chemically stable because of the high strength of CeC bond in its structure. Therefore, its stable structure protects the active sites of the catalyst. Also, GO-supported catalysts can be easily dispersed in the mixture because of their light weight[12]. Moreover, GO has a high specific surface area. In addition, the production of GO composite makes the catalyst more stable. Different studies have been done on the application of GO in the biodiesel production process[13, 14]. The magnetic catalysts such as $ZnFe_2O_4$, $CoFe_2O_4$, and $NiFe_2O_4$ extremely increase the biodiesel production in comparison to conventional acidic and alkaline catalysts[15]. Magnetic catalysts can be easily recovered and reused in several cycles for producing biodiesel because of their magnetic properties. Prominent characteristics of magnetic catalysts like low cost and non-toxicity, eco-friendly, and easy separation using an external magnetic field have made them useful catalysts for biodiesel production[16]. The most common magnetic materials like Fe, Fe_2O_4, g-Fe_2O_3, and Fe_3O_4 after functionalization and modification, not only preserve the magnetic features but can be easily separated from the product using an external magnetic field. Also, they can maintain their catalytic strength and reusability[17]. Moreover, magnetic nano-particles have a high specific surface area and rich functional groups and is easily dispersed in the solvent. Previous studies have indicated that the catalytic activity of magnetic catalysts in biodiesel production is higher than homogeneous catalysts, which is due to the magnetic properties between the

particles, resulting in high activity and stability[18].

In this research, chicken fat was applied to produce biodiesel using $NiFe_2O_4$/graphene oxide (GO) as a novel catalyst. The aim of this research is to investigate the effect of different parameters such as temperature, reaction time, catalyst amount, and methanol to oil ratio on biodiesel production using chicken fat oil in the presence of calcium oxide nanocatalyst. The produced biodiesel under optimal conditions was then mixed with diesel at different mixing ratios and their characteristics (flash point, cloud point, pour point, viscosity and density) were determined. The characteristics were then compared to those under standard conditions to come with the best mixing ratio between biodiesel and diesel, so as to obtain the best properties of the mixed fuel.

MATERIALS AND METHOD

Chemicals used for the synthesis of nanocatalyst and reaction
Sodium hydroxide (purity 98%) was purchased from Merck Company. In addition, H_2O_2, $NaNO_3$, and $KNiO_4$ were purchased from Merck Company. Furthermore, H_2SO_4 and HCl were purchased from Merck Company with a purity of 98% and 37%, respectively.

Preparing the $NiFe_2O_4$/GO catalyst
The Hummers' method was used to produce GO from oxidation of graphite powder. To this end, firstly, graphite and $NaNO_3$ with concentrations of 1 and 0.5 g were placed into a 500-mL Erlenmeyer. Afterwards, 46 mL of sulfuric acid (H_2SO_4) was added to the Erlenmeyer in the ice bath at 0 C, and the mix was vigorously stirred using a magnetic stirrer for 30 min. Subsequently, 6 g (37.97 mmol) of potassium permanganate was gradually added to the reaction vessel while the temperature remained below 20 °C. The ice bath was then removed, and the resultant mixture was stirred at 35 °C for 2 h. Then, 92 mL of distilled water was added to it and the mix temperature was increased to 98 °C. At the next step, 280 mL of distilled water and 5 mL of hydrogen peroxide were gradually added to the vessel. The mixture was filtered and washed several times with 10% hydrochloric acid (HCl) and

water to be neutralized completely. The obtained powder was fully dehydrated in the oven at ambient temperature under vacuum conditions[19, 20].

NiFe$_2$O$_4$/GO was synthesized using the chemical deposition method. To do so, nickel chloride (NiCl$_2$) and iron chloride salts (FeCl$_3$.6H$_2$O) were mixed at a molar ratio of 2:1 (0.63 g of NiCl$_2$ and 0.68 g of FeCl$_3$.6H$_2$O) to obtain a solution containing manganese and iron (NiFe$_2$O$_4$). Then, 0.25 g of the GO produced in the previous step was added to the aforementioned solution (50 mL) and the mix was stirred using ultrasonication for 50 min. Once thoroughly mixed, the solution was introduced by 30 mL of NaOH (3M) in a dropwise fashion and the solution was blended on a magnetic stirrer at 85 C for 1 h. Subsequently, the synthesized magnetic nanocomposite was removed from the aqueous solution using the magnet before being washed with distilled water to achieve a pH value between 6 and 7. Afterwards, the product was dehydrated in a vacuum oven at ambient temperature.

Characterization of the catalyst
Crystalline phases in the NiFe$_2$O$_4$/GO catalyst were specified using XRD analysis (Siemens, D-5000 model, Germany) within a 2q range of 5-80. Also, SEM analysis (TESCAN MIRA3) was employed to specify the catalyst morphology. The elemental compositions of GO and NiFe$_2$O$_4$@GO were investigated by using XPS. FTIR analysis (Brucker TENSOR 27, Czech Republic) was also conducted to specify the functional groups within the catalyst structure at 400-4000 cm^{-1}.

Methods
Extraction of oil from chicken fat first step, chicken fat was washed to remove wastes and blood because

residual waste may burn by heating, thereby changing the color of the produced oil or lowering its quality. The fats were then placed in a colander at ambient temperature to drain its water content. The fats were subsequently divided into smaller parts to facilitate the oil extraction process. Fats were then placed in a large vessel and mildly heated on the flame of the oven with the vessel lid closed to have the fat texture liquefied to oil. This process took three hours to accomplish. Afterwards, the obtained oil was passed through a filter to become free of suspended and waste material. Prior to use, the obtained oil was heated at 100 °C to remove any remaining water content.

Analysis of chicken fat using gas chromatography, Fatty acids contents of the obtained oil from chicken fat were determined using gas chromatography (GC). In this investigation, Varian CP-3800 GC was utilized. The apparatus was equipped with a flame ionization detector (FID) and a capillary column of 30 m in length. Helium was used as the carrier gas. Flow rates of nitrogen, hydrogen and air were set to 30, 30 and 300 ml/min, respectively. The fatty acids content of the extracted oil from the chicken fat are presented in Table 1.

Biodiesel production method
In order to produce biodiesel from chicken fat using NiFe$_2$O$_4$/GO nanocatalyst, transesterification method was utilized. A condenser was further used to avoid methanol vaporization and better control the reaction temperature. At first, 50 g of chicken oil was poured in a two-necked flask and the flask was placed on a heater to reach the temperature 65 °C. Then, the methanol-catalyst mixture was introduced into the flask. The time at which the oil was mixed with methanol

Table 1. Fatty Acid Contents of Chicken oil using GC Analysis

Fatty acid	Molecular formula	Chemical formula	Molecular weight	Content (%)
Palmitic acid	C16:0	$C_{16}H_{32}O_2$	256.42	29
Stearic acid	C18:0	$C_{18}H_{326}O_2$	284.48	5
Oleic acid	C18:1	$C_{18}H_{34}O_2$	282.46	44
Linoleic acid	C18:2	$C_{18}H_{32}O_2$	280.45	13
Linolenic acid	C18:3	$C_{18}H_{30}O_2$	278.49	0.025
Myristic acid	C14:0	$C_{14}H_{28}O_2$	228.37	8

Table 2. Experimental Conditions for Determining the Optimum Conditions on Biodiesel Production.

Run No.	Time (h)	Temperature (°C)	Catalyst amount (wt.%)	Methanol/Oil molar ratio	Biodiesel yield (%)
1	4	65	1.5	4:1	67
2	3	65	1.5	6:1	73.5
3	4	65	1.5	9:1	92
4	4	50	1.5	12:1	88
5	3	65	1.5	15:1	76
6	4	65	0.5	9:1	81.5
7	4	65	1	9:1	91.293
8	3	65	1.5	9:1	81
9	4	65	2	9:1	84
10	4	50	3	9:1	70
11	4	50	1	9:1	82.55
12	5	55	1	9:1	86.5
13	4	60	1	9:1	85
14	4	65	1	9:1	95
15	2	65	1	9:1	71
16	3	55	1	9:1	76
17	5	65	1	9:1	91
18	5	65	1	9:1	93
19	6	65	1	9:1	80

and catalyst was recorded as the starting time of the experiment. The smaller neck of the flask was sealed with a plastic cap through which a thermometer was introduced into the solution to control the solution temperature without letting the methanol leave the flask.

Determination of optimal conditions

In the present research, the effects of parameters such as methanol to oil molar ratio, catalyst amount, reaction temperature, and reaction time were investigated on the biodiesel production. The yield of biodiesel production was used as the criterion for reporting optimal biodiesel production conditions. In order to find the optimal value of each parameter, other parameters were kept constant. Accordingly, in the first stage, reaction temperature, reaction time and catalyst amount were set to 65 °C , 4 h, and 1.5 wt.%, respectively, and different methanol to oil molar ratios (1:4, 1:16, 1:9, 1:12 and 1:15) were tested; the best results were reported with the methanol to oil ratio of 1:9. In or-

der to determine the best values of other parameters, experiments were conducted according to the details given in Table 2. Further, reported in this table is the yield of biodiesel production under these conditions. Analysis of the biodiesel produced under optimal conditions. After examining the laboratory conditions for biodiesel production, properties of the produced fuel such as viscosity, density, flash point, cloud point, and pour point were examined and analyzed according to international standard procedures (e.g. ASTM D6751 and EN 14214).

RESULTS AND DISCUSSION

SEM Analysis of the Nanocatalyst

Fig. 1a illustrates the characteristic layered sheet morphology of GO, whereas micrography of Fig. 1b highlights the notable difference with respect to nanoenabled NiFe$_2$O$_4$/GO. Nanoparticles of NiFe$_2$O$_4$ were homogenously distributed on a GO thin film,
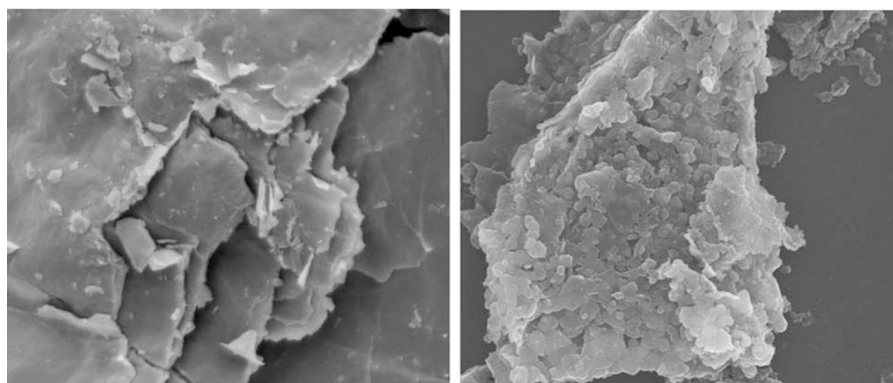
Fig. 1. Scanning electron microscopy images of a pristine GO and b nanoenabled $NiFe_2O_4$/GO nanocatalyst

displaying attachment between the components of the composite nanomaterial.

The XRD analysis

The XRD analysis of pristine GO depicts a characteristic strong peak at 2θ of 10° associated with the interlayer spacing induced by the presence of the oxygen functional group. The wide peak observed is associated with plane (002) of graphite. The nanocomposite $NiFe_2O_4$/GO photocatalyst depicts a differentiated XRD pattern from pristine GO, which allows inferring a homogeneous dispersion of $NiFe_2O_4$ on the GO sheets as observed in the SEM images (see Fig. 1). The peaks observed in $NiFe_2O_4$/GO correspond to a single-phase spinel-type structure (JCPDS 54-0964) with characteristic crystal planes identified in Fig. 2a. The crystalline structure of $NiFe_2O_4$ with atomic occupancy is described as $(Fe^{3+})_A[Ni^{2+}Fe^{3+}]_BO_4^{2-}$, where A and B denote tetrahedral and octahedral sites in the spinel structure, respectively[21]. The adsorption–desorption study allowed to de-termine a specific surface area (S_{BET}) of 76.7 m^2g^{-1}, average pore diameter of 7.76 nm, and pore volume of 0.15 cm^3g^{-1}.

Fourier-transform infrared (FT-IR) was used to investigate the surface functional groups of GO and $NiFe_2O_4$/GO. Figure 2b depicts the existence of oxygen containing function-al groups on the graphene surface, which demonstrates successful oxidation treatment. The strong peak at 3420 cm^{-1} can be associated with the stretching vibration of OH groups. Characteristic peaks associated with C=O stretching vibration at 1727 cm^{-1}, C=C stretching at 1633 cm^{-1}, O–H deformation at 1400 cm^{-1}, C–O (epoxy) stretching vibration at 1200 cm^{-1}, and C–O (alcoxy) at 1054

cm^{-1} were clearly observed. The FT-IR spectra of the nanocomposite $NiFe_2O_4$/GO have bands assigned to the vibration of ions in crystal lattices, which indicate the presence of homogeneously distributed ferrite on the surface. The peak observed at 400 cm^{-1} is assigned to the octahedralmetal stretching Ni–O, while the bands at 582 cm^{-1} and 687 cm^{-1} are associated with the
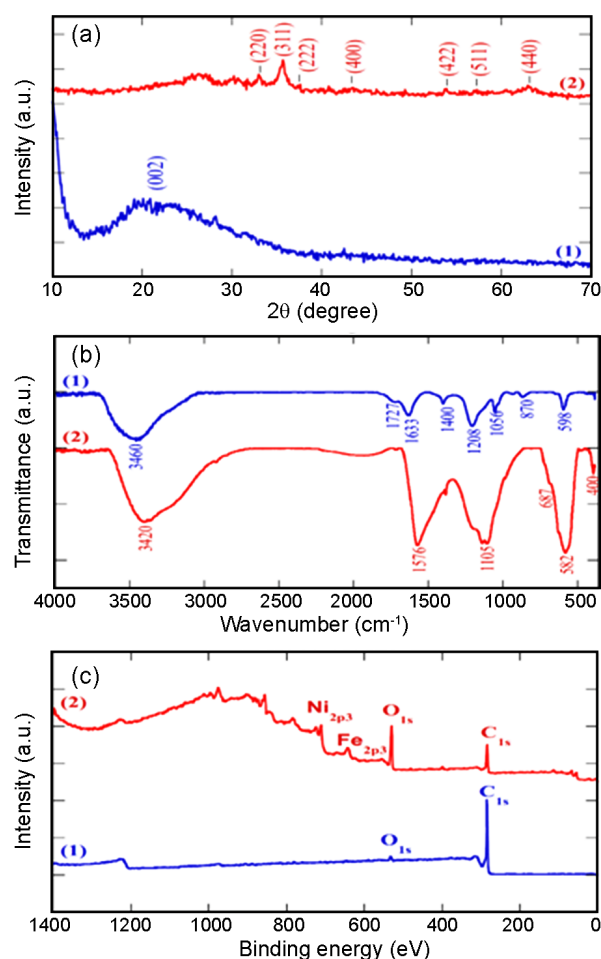

Fig. 2. Comparative a XRD, b FT-IR spectra, and c XPS spectra of 1 pristine GO and 2 $NiFe_2O_4$/GO nanocomposite.

Fe–O vibrations. The bandgap of $NiFe_2O_4$/GO nano-composites was evaluated by UV-diffuse reflectance spectros-copy. The Tauc plot denotes an energy band-gap (Eg) of 2.9 eV. The nanocomposite point-of-zero charge (PZC) pH_{PZC}= 7.2 was determined by using the pH drift method[21].

The elemental compositions of GO and $NiFe_2O_4$/GO were investigated by using XPS (Fig. 2c). The XPS spectrum of pristine GO showed only the peaks of carbon C1s at 280 eV and oxygen O1s at 526 eV, which are characteristic of graphene layers functional-ized with oxygen groups. The XPS spectrum changed when GO was nanoenabled by $NiFe_2O_4$. While the signals of carbon and oxygen remained, the intensity of O1s increased due to the higher content of oxygen associated with the metal oxide nanoparticles. More-over, small signals of nickel and ferrite were found at 849 eV (Ni_2p_3) and 703 eV (Fe_2p_3), respectively. The ele-mental composition of the $NiFe_2O_4$/GO nanocom-posite included carbon (53%), oxygen (32%), and fer-rite (8.2%), and nickel (6.7%).

Effect of Methanol to Oil Ratio on Reaction Yield
Effect of methanol to oil molar ratio (1:4, 1:16, 1:9, 1:12 and 1:15) on biodiesel production using Nickel ferrite nanoenabled graphene oxide nanocatalyst was investigated. Fig. 3 shows the effect of methanol to oil molar ratio on the yield of biodiesel production. The best methanol to oil ratio under the mentioned oper-ating conditions was found to be 1:9 as it ended up with a yield of biodiesel production from the chicken fat of 90%. With a yield of 65%, the methanol to oil ratio of 1:4 returned the lowest biodiesel production
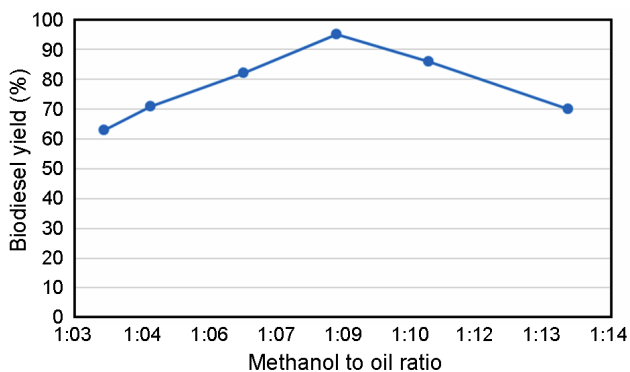
yield. Biodiesel production yield followed an increas-ing trend when methanol to oil ratio was changed from 1:4 to 1:9, while the yield changed to a decreas-ing trend for methanol to oil ratios beyond 1:9. This was because, with increasing the content of methanol, glycerin was extensively dissolved in the excessive methanol, keeping the methanol from reacting with the catalyst and hence making methanol separation from biodiesel and glycerin very difficult.

Effect of Catalyst Amount on the Yield of Biodiesel Production
In order to investigate the effect of this parameter on biodiesel production, different weight percentages of the catalyst (0.5, 1, 1.5, 2 and 3 wt.%) were tested. The obtained results are shown in Fig. 4. As can be observed in this figure, with increasing the catalyst amount from 0.5 to 1 wt.%, the amount of biodiesel production increases. However, with further increas-ing the catalyst amount from 1 to 3 wt.%, the biodiesel
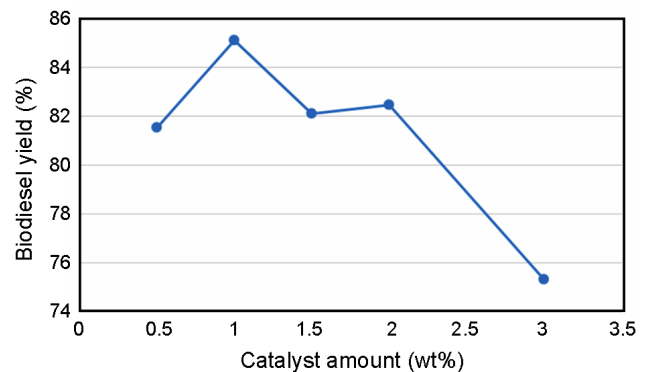

Fig. 4. Effect of catalyst amount on the biodiesel yield (con ditions: temperature 65 °C, mixing speed 1500 rpm, metha-nol to oil ratio 9:1 and contact time 4 h).
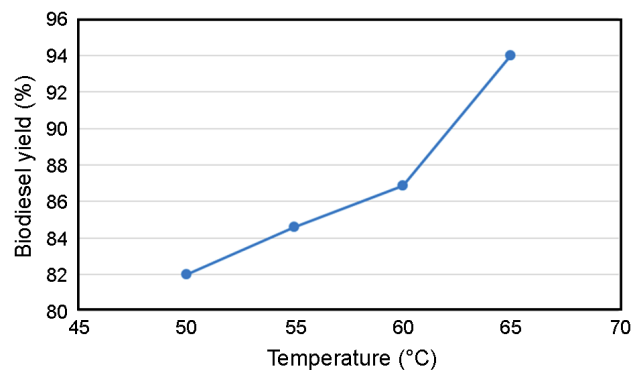

Fig. 3. Effect of methanol to oil ratio on the biodiesel yield (conditions: catalyst amount 1.75 wt.%, temperature 65 °C, mixing speed 1500 rpm a nd time 4 h).

Fig. 5. Effect of temperature on biodiesel yield (condition ns: amount of catalyst 1 wt.%, mixing speed 1500 rpm, metha-nol to oil ratio 9:1 and contact time 4 h).
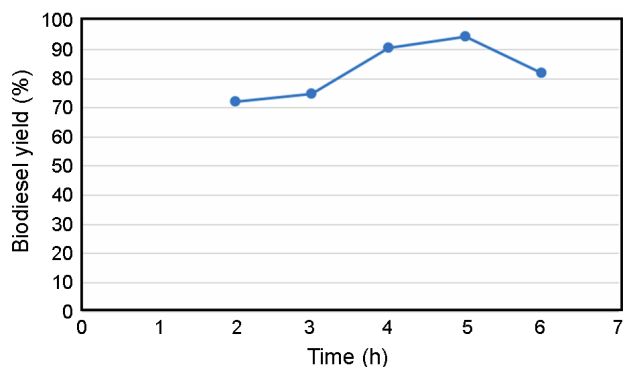
Fig. 6. Effect of time on biodiesel yield (conditions: amount of catalyst 1 wt.%, mixing speed 1500 rpm, methanol to oil ratio 9:1 and temperature 65 °C).

production yield follows a decreasing trend. This is because, with further increasing the catalyst amount, cohesion and agglomeration of the particles resulted in the reduced active surface area and increased the viscosity of the solution, thereby reducing the biodiesel production yield [22].

Effect of Temperature on Biodiesel Production
Rate and yield of biodiesel production in transesterification process is particularly dependent on the reaction temperature. Fig. 5 shows the effect of temperature on the yield of biodiesel production. As can be seen in this figure, with increasing the temperature, biodiesel production increases and the highest biodiesel production yield (92.6%) obtained at 65 °C.

Effect of Contact Time on Biodiesel Yield
In order to determine the effect of contact time on the yield of biodiesel production, effects of different reaction times (e.g. 2, 3, 4, 5 and 6 h) were evaluated under constant operating conditions in terms of stirring rate (1500 rpm), reaction temperature (65 °C), methanol to oil ratio (1:9), and amount of cata-

lyst (1 wt.%). The results are presented in Fig. 6. As can be seen in Fig. 5, with increasing the reaction temperature, biodiesel production increased, so that the maximum biodiesel production (95 %) occurred in 5 h. The reaction is slow due to the mixing and dispersion of methanol in oil, and the biodiesel yield rises from 2 to 5 h during the reaction time. Since the biodiesel production reaction is a reversible chemical reaction, at longer times after the formation of biodiesel, the biodiesel bonds may break up to the original reactants, and for this reason the biodiesel yield decreased after 5 h. In other words, excessive reaction time reduces product yield because of the backward reaction, resulting in a loss in esters as well as causing more fatty acids to form soaps.

Mixing Biodiesel with Diesel
After preparing biodiesel in the best conditions (temperature = 65 °C, time = 5 h, methanol to oil ratio= 1:9 and catalyst amount = 1 wt.%), to improve properties of the biodiesel as a fuel, it was mixed with diesel at different ratios (B25, B50 and B75). Also, density, viscosity, cloud point, flash point and pour point of the mixtures were determined and then compared to international standards, as reported in Table 3.

As can be observed in this table, most of the properties of the produced biodiesel and its mixture with diesel at different ratios were in the range of standard values. Meanwhile, flash points of B00, B25 and B50 were out of the standard range. Therefore, as far as the use of the produced fuel from chicken fat is concerned, mixing it with diesel as B75 or B100 fuel will end up with very good results. Furthermore, since pour point of all of the considered mixtures was above zero, such fuels are not suitable for the cold climate.

Table 3. Physical Characterization of Produced Biodiesel from Chicken Fat in the Presence of Nickel ferrite nanoenabled graphene oxide Nanocatalyst.

Test	EN-14214	EN-14214 ASTM D-6751	B00	B25	B50	B75	B100
Density (at 15 °C)	900 -860	-	830	845	862	873	881
Viscosity (at 40 °C)	-	1.9-6	3.1	3.3	3.8	4.23	4.85
Flash point (°C)	>120	>130	88	92	100	130	170
Cloud point (°C)	-	-	3	4	5	6	7
Pour point (°C)	-	-	-8	0	2	3	4

CONCLUSIONS

In the present research, biodiesel was produced using chicken fat in the presence of nano-NiFe$_2$O$_4$/GO to produce a clean, high-quality fuel in accordance with related standards. On this basis, the effect of parameters such as methanol to oil molar ratio (1:4, 1:6, 1:9, 1:12 and 1:15), NiFe$_2$O$_4$/GO nanocatalyst amount (0.5, 1, 1.5, 2, and 3 wt.%), reaction temperature (50, 55, 60 and 65 °C), and reaction time (2, 3, 4, 5 and 6 h) were investigated on the biodiesel production yield. Obtained results indicated the highest yield of biodiesel production of 94.4% under conditions in terms of reaction temperature (65 °C), reaction time (5 h), methanol to oil ratio (1:9) and catalyst amount (1 wt.%).

The produced biodiesel under optimal conditions was further mixed with diesel at different mixing ratios followed by measuring flash point, cloud point, pour point, viscosity, and density of the mixtures. According to the obtained results, among the various mixtures studied in this research, B75 and B100 exhibited better densities, viscosities, and flash points at the corresponding standard ranges. Therefore, the biodiesel produced from chicken fat and B75 and B100 mixtures can be used as alternative fuels to diesel.

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