Using Jordan Zeolite as a Heterogeneous Catalyst for Synthesize the Used Frying Oil into Biodiesel

Falah Fahed Bani Hani and Said Alrabadi

***Abstract*—In this research, biodiesel was prepared from used frying oil (UFO) by transesterification reaction in the presence of impregnation of Jordan zeolite (JOZ) with an aqueous solution of sodium hydroxide . The transesterification process is optimized by modify the JOZ catalyst concentration and the conversion of UFO to biodiesel exceeds 95% when using 1:6 molar ratio of oil to methanol at 600C, time of reaction 3hr and 5.5% solid catalyst.. The biodiesel is analyzed by Gas Chromatography Mass Spectrometry and the result are confirm by FTIR spectral, which evidence the being of linoleic acid as the main constituent . The physical and chemical characteristic of biodiesel were analyzed to guarantee that the product meets the standards of fuel characteristic. The standard utilized was ASTM D 6751 and was used to measure the whole specific properties of biofeul. The properties of biodiesel obtained were within the range of specified limitations. The research appear that biodiesel daraived from UFO was of good quality and could be used as a diesel fuel which count as renewable energy and environmental recycling process from west oil after frying.**

***Index Terms*—Transesterification, used frying oil, biodiesel, fuel characterization, zeolite, catalyst.**

I. Introduction

Due to the increasing concerns about polluting the environmental and notwithstanding the consumption of non-renewable energy sources besides the soaring cost of petroleum products, these variables incited significant research to identify alternative fuel sources. Biofuel was one of options among the alternatives resources that have recently attracted the attention of researchers in different countries all over the world. The reason behind going after this fuel source is because it's renewable has better gas emissions and the biodegradability of the fuel product. It has been estimated that biodiesel, and more specifically bio-ethanol, could supplant roughly 10% of the diesel fuel consumed within European continent total fuel demand [1]-[3]. The alternative fuel that will replace part or all of the petroleum diesel fuel must be in fact attainable, economically competitive, ecologically satisfactory, and promptly accessible [1], [4], [5]. A considerable lot of these essentials are met by vegetable oils, or by and large, by triglycerides. Without a doubt, vegetable oils are broadly accessible from an assortment of assets, and they are inexhaustible. Furthermore, these fuels are biodegradable, have minimum sulfur content and there is no problem in transport and storage or to the environment or to the living creatures. Therefore, these items can be viewed as reasonable options for diesel fuel, in spite of the price issue thy still utilized as a diesel fuel substitute. Substantial amounts of utilized frying oil are accessible all through the world. In Jordan the amount of UFO range 320 million liters/year and in the US alone the amount of UFO ranges from 5 billion to 12 billion liters a year [2]-[4]. in some places, frying oil used in the manufacturing of soap also as an additive for fodder preparation. In few restaurants, the vegetable oil, after the primary utilize, is gone through an oil recycler and after that reused for cooking. After the second utilize, the oil is gathered and sent for the soap manufacturing [6], [7]. Large portions of UFO are illegally dumped into rivers and wastelands, causing environmental pollution. The use of UFO as a fuel for diesel engines may reduce or eliminate an environmental pollution causing factor [8], [9] Biodiesel can be processed utilizing different mechanisms; homogeneous catalytic transesterification is the most common process. In this process an ester compound is replaced with an alcohol in the alkyl group. Thus, biodiesel can be defined as fatty acid methyl esters (FAME) derived from the transesterification of triglycerides (vegetable oils or animal fats) with alcohol and suitable catalyst [2].

Yet, compared to homogeneous catalysts, heterogeneous catalytic processes are able to produce biodiesel more cost-effectively while greatly reduces formation of soap by-products [10] and increases the quality of FAME and glycerol. In fact, the development of heterogeneous catalyst systems can minimize the costs of catalyst and product separation and purification which make biodiesel economically viable and capable for competing with commercial petroleum-based diesel fuel [11], [12]. To date, many solid heterogeneous catalysts have been employed for biodiesel production, such as metal oxide catalysts (CaO, MgO, ZnO_2, TiO_2, SnO_2, and ZrO_2), zeolites, carbon group catalysts and waste material basedcatalysts [13]-[16].

The objective of this study is to synthesize biodiesel from used frying oil during transesterification reaction of UFO with methanol using NaOH/Jordan zeolite as catalyst. FT-IR spectroscopy was used to monitor biodiesel quality. investigate biodiesel physicochemical properties to be utilized as a fuel. The properties investigated such as kinematic viscosity, density, higher heating value, iodine value, flash point, cloud point, pour point, total acid number (TAN) and cetane index. The analysis of those properties refers to the standard test methods.

II. Material & Methods

A. Catalyst Preparation

Natural zeolite was selected as a catalyst for our

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application. There is an abundance of sediment beds in east Jordan. The samples were taken from the north-east region and, the purity was about 95%. For activation, it was calcined at 400˚C for 3 hours [17]. Catalyst specifications and compositions are given in Table I. In this research, the catalyst was prepared based on the procedure described elsewhere [18] and by impregnation of Jordan zeolite (JOZ) with an aqueous solution of sodium hydroxide. The impregnation method involved loading 30 ml of sodium hydroxide onto 18 g of Jordan zeolite, followed by drying in atmospheric dryer. The catalyst was finally calcined in a muffle furnace at 1800C in air for 3 hours.

TABLE I: CATALYST SPECIFICATION & COMPOSITION

Catalyst specification		Catalyst composition, %			
Pore diameter (A˚)	33.12	SiO_2	65.42	MgO	1.09
Particle diameter (mm)	Dp >1.6	Al_2O_3	24.21	CaO	2.95
Surface area (m2 g-1)	69.12	TiO_2	00.02	H_2O	12.1
Density (kg m-3)	2150	Fe_2O_3	01.38		

B. Biomass

The UFO, was collected from local restaurants, it is originally a mixture of palm oil and sunflower oil, the UFO samples were mixed together and heated to 115 ℃ and removes the water molecules then filter by 20μm fabric to isolate wastes and suspended materials. By and large, biodiesel production is unequivocally influenced by free fatty acid (FFA) content of the UFO and the amount of catalyst had impact of conversion of esters amid the transesterification process. The FFA of the UFO was tested and observed more than 2%; along these lines, a tow-step transesterification process was utilized for biodiesel production from the collected samples of used frying oil. The qualities of the UFO mixed samples are shown in Table II.

TABLE II: PHYSICAL PROPERTIES OF UFO MIXD SAMPLE & DIESEL

Property	Unit	Mixed sample	diesel
Flash point	^oC	244	86
Could point	^oC	18.7	0
Density	g/cm^3	0.91	0.83
Viscosity at 40^{o}C	mm^2/s	32.5	3.43
Acid number	mg KOH/g	3.38	0.13
Saponification number	mg KOH/g	252.12	-
Iodine number	mg KOH/g	111.4	-

C. Transestrification

The data of Table I show that UFO sample has 3.38 mg KOH/g of acid value which is not perfect match property for the alkali catalysed transesterification reaction. Owing to its high free fatty acids, a two-step transesterification process was taken to convert the biodiesel from utilized frying oil. First step, as catalyst, sulfuric acid was utilized to esterify the UFO. It was described as below:

$$RCOOH+CH_3OH\text{--catalyst--}RCOOCH_3+H_2O.$$

The reaction procedure was as follows: a three-neck flask associated with a condenser was loaded with 200 ml of UFO, 4 ml of H_2SO_4 and 40 ml of CH_3OH. The blend was mixed for 1.5 h at 60 ℃. After reaction, the blend was filtered and the unreacted methanol was isolated from the fluid stage by distillation. The pretreated oil is washed three times with sodium chloride solution and after that dried using anhydrous sodium sulfate. After the pretreatment, the free unsaturated fats estimation of the pretreated oil was lesser than that of utilized frying oil [16], [19]- [21].

D. Transestrification of UFO with NaOH/Catalyst JOZ at 600C and Oil to Methanol Molar Ratio 6:1

According to our previous experimental work [3], the optimum factors utilized were ethanol/oil molar ratio 6:1, reaction temperature 60 ℃ and reaction time 3hr, were fixed as basic parameters in these experiments. A series of parallel reactions were carried out using the same method to obtain the maximum yield of biodiesel. The reaction procedure was as follows: a three neck round bottom flask used as a batch reactor connected with a condenser and was placed in a water bath to perform the reaction at the desired temperature. The process reaction performed at molar ratio of oil to methanol 1:6. First, the catalyst JOZ in amount 3%, 5%, 7%, 9%and 12% was dispersed in methanol under magnetic stirring. Then, 100gr of pretreated oil was added into the mixture and heated for 3 hr to temperature 60 ^{0}C. In the end, the excess methanol was distilled off under vacuum. After the products were centrifugated, it formed three phases, the upper layer was biodiesel, the middle layer was glycerol, and the lower layer was a mixture of solid zeolite and a small amount of glycerol. After that, it was washed three times by distilled water and was dried by distillation under vacuum and collected for analysis.

E. Analysis

Different physic-chemical propertied of UFO and got biodiesel were estimated, The analytical techniques used to decide the attributes of the obtained biodiesel are essentially those prescribed by the ASTM standard methods, including density (D1298), kinematic viscosity at 40 ℃ (D445), flash and Combustion point (D93), cloud point (D2500), pour point (D97) , cetane index (D976) , acid value (D664) and high heating value (Parr-1351 bomb calorimeter, according to ISO 1928) and iodine value [16]. Unsaturated fatty acid quantitative was determined by utilizing a Hitachi G-5000A gas chromatography, analysis is performed for identifying the hydrocarbon compounds, for example, unsaturated fats and methyl esters. The separation is carried out by using capillary column Rtx-5MS with helium as a carrier gas. FT-IR spectrometer (Perkin Elmer GX system with a horizontal ATR accessory and flow cell) was used to identify the main functional groups presence at both the optimum produced biodiesel sample and its parent used frying oil.

III. RESULTS & DISCUTION

A. Heterogeneous Catalyst/UFO Mass Ratio

Different catalyst amounts (*NaOH/JOZ):* 3%, 5%, 7% , 9% and 12%, relative to the UFO weight (100 g) were employed to obtained the maximum biodiesel yield with

optimum value of catalyst. From Fig. 1, when the transesterification equilibrium shifted to right for increasing catalyst amounts ratio the optimum biodiesel yield reached 95.1% with optimum value of catalyst, 7% . However, further increase in catalyst amounts resulted in slight reduction in biodiesel yield because, may be, of phase separation problems.

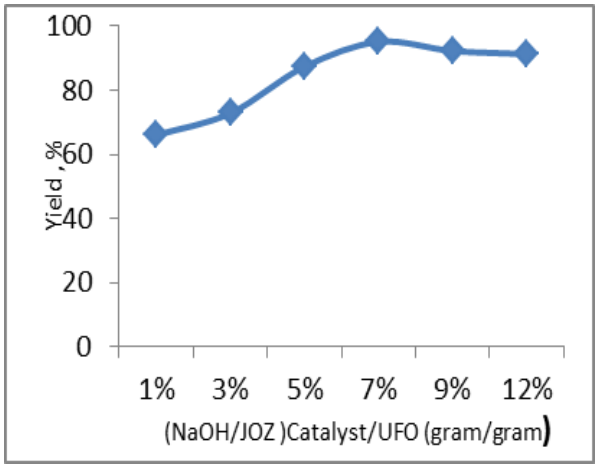

Fig. 1. Biodiesel yield as a function of (NaOH/JOZ) Catalyst/UFO mass ratio. Methanol/UFO molar ratio 6:1, Reaction time 3 hr, Reaction temperature 60°C.

The physiochemical properties of UFO as said in Table I was decide by the officially settled techniques to check their properties after frying. All the palatable oils are constituted fundamentally of fatty triglyceride esters with various substitution patterns, lengths and degree of saturation of the chains and of other minor components [22], [23]. Considering the unsaturated fat structure of the UFO utilized as a part of this investigation, it comprised for the most part of methyl esters of linoleic (C18:2) > oleic (C18:1) > palmitic (C16:0) > stearic (C18:0) acids. The substance of the other confirm acids were around 1 mass % or less. Such a composition reflects the sunflower and palm oil origin of the UFO [24]. The iodine value of the feedstock, which is a measure of the level of unsaturation, was roughly 120 mg I2/100 g [25], [26]. The ascertained iodine value were higher than the tentatively decided in around 4%. In any case, the utilized oils have an assortment of characteristics as an outcome of the diverse frying conditions and the period of utilization .

Gas chromatography (GC) analysis is present for identifying the hydrocarbon compounds like fatty acids and methyl esters. The isolation is completed by using gas chromatography with flame ionization detector. Major fatty acid for obtaind oil is Linoleic acid with Retention Time, (min) 981802 took after by Oleic acid with Retention Time, (min) 524447, Linolenic acid with Retention Time, (min) 1255 and Palmitic acid with Retention Time, (min) 184101. The fatty acid syntheses by transesterification of UFO with NaOH/catalyst JOZ at 60°C and oil to methanol molar ratio 6:1 is shown in Table III.

The original used frying oil presents the following syntheses of fatty acids (in wt. %): palmitic (11.2), oleic (22.4), linoleic (41.62), linolenic (14.57) , stearic (5.10), and Eicosenic (1.81). It is related to blend oil from palm and sunflower oils which have a similar structure [27]-[30].

B. Investigation of Biodiesel Quality FT-IR Spectra

FT-IR spectroscopy was the analytical technique employed in this work to identify the main functional groups presence at both the optimum produced biodiesel sample and its parent used frying oil. As shown in Fig. 2 and Fig. 3.

Fig. 2 demonstrates the FTIR spectrum of UFO. The majority of the pinnacles of the spectrum are related to the specific functional groups. As appeared, the spectrum of utilized frying oil was portrayed with lopsided and symmetric strong extending vibrations of carboxyl group at 2671.9 cm^{-1}, along with the O-H extending of the hydroxyl bonded with alcohol at 3458.3 cm^{-1} and Aldehyde. The C=O group of triglycerides coming about an extending vibration at 1752.1 cm^{-1}. The groups at 1462.4 cm^{-1} coming from the bending vibrations of CH_2 and CH_3 aliphatic groups, the bandes at 2931.1 cm^{-1} and 2862.4 cm^{-1}, relegated to (C-H) symmetrical and asymmetrical extending of the saturated carbon-carbon bond. The outcome which demonstrates the groups at 1243.8, 1161.6, some of them could be allotted to the extending vibrations of the (C-O) esters group.

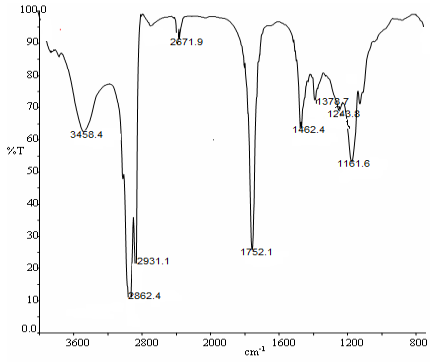

Fig. 2. FTIR Specter for used frying oil.

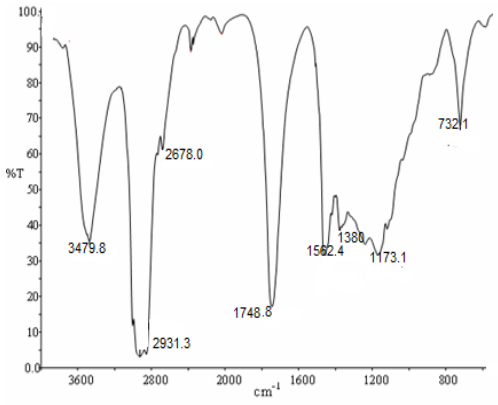

Fig. 3. FTIR Spector for obtained biodiesel.

Fig. 3 demonstrates the FTIR spectrum of obtained biodiesel. As appeared, the range of got biodiesel by using impregnation of Jordan zeolite (JOZ) with an aqueous solution of sodium hydroxide as catalyst was symmetric strong extending vibrations of carboxyl group at 2931.3-2678.0 cm^{-1}, alongside the O-H extending of the hydroxyl fortified with alcohol at 3481.1-3200 cm^{-1} (wide) with alcohols, Phenols groups. Aldehyde, Ketones (C=O) group alongside carboxylic group, Ester group found at 1470-1670 cm^{-1}. C-O amass joined with carboxylic group, Ester assemble extended at1000-1173.1 cm^{-1}. At last, Alkanes (C-H) bunches extended at 1462.4-1380.0 cm^{-1}.

Unsaturated fatty acid substance are the significant markers of the properties of biodiesel. Duplicate samples were utilized to decide the unsaturated fat substance of the biodiesel product. Table III demonstrates the summary of unsaturated fat substance in the utilized frying oil and in biodiesel got from it by Transesterification with NaOH/catalyst JOZ. From Gas Chromatography analysis, it was discovered that they got biodiesel from UFO using catalyst JOZ/NaOH contains oleic acid (59.7%) trailed by linoleic acid (19.31%), linolenic acid (9.57%), palmitic acid (6.17%), stearic acid (2.6%), ecosenoic acid (1.21%) and rest the others (2.66%). Gotten biodiesel contained the highest amount of oleic acid among other unsaturated fats in the product.

TABLE III: FATTY ACID COMPOSITION OF UFO & PRODUCED BIODIESEL

Type of fatty acids	Carbon Chain	UF oil, %	Derived Biodiesel,%
Myristic	C14:0	0.43	0.61
Palmitic	C16:0	11.22	6.17
Stearic	C18:0	5.10	2.61
Oleic	C18:1	22.40	59.70
Linoleic	C18:2	41.62	19.31
Linolenic	C18:3	14.57	9.57
Arachidic	C20:0	0.22	0.60
Eicosenic	C20:1	1.81	1.21
Behenic	C22:0	0.03	0.08
Erucic	C22:1	0.61	-

TABLE IV: FATTY ACID COMPOSITION OF UFO & PRODUCED BIODIESEL ACCORDING TO DEGREE OF UNSATURATION

Fatty Acid Composition	UFO Content,(%)m/m	Derived biodiesel Content (%), m/m
Saturated fatty acids	17.00	10.07
Monounsaturated fatty acids	24.32	61.41
Polyunsaturated fatty acids	55.19	24.13

TABLE V: CHARACTERISTICS OF PRODUCED BIODIESEL IN CONTRAST WITH STANDARD VALUE

Property	Value according (ASTM)	Derived Biodiesel	Diesel fuel
Viscosity at 40°C, mm2/s	1.9–6.0	5.69	3.43
Acid number, Mg KOH/g oil	0.8 max	0.40	0.13
Density at15 ℃, g/cm^3	0.86 – 0.90	0.88	0.83
Cloud point, ℃	-	6	3
Pour point, ℃	-	1	-7
Cetane index	47(min)	46.9	46
Flash point, ℃	130(min)	168	86
combustion point, ℃	140	171	92
Iodine value, gI_2/100g	120	97.8	111

The fatty acid structure, as per level of unsaturation is given in Table IV. For derived biodiesel, Monounsaturated (C18:1) fatty acids were found around 60%, Poly unsaturated fatty acids were observed to be roughly 29% (C18:2, C18:3) and just about10% fatty acids were saturated. For UFO, the major saturated fatty acid were found Palmitic acid and stearic acid. The quality and sort of unsaturated fat in the biodiesel are the central point which decide the viscosity of the derived biodiesel.

C. Fuel Characterization

The sample was tested to decide the fuel attributes as per ASTM Biodiesel. , different physical properties of derived biodiesel, were measured to ensure these meet the standards (ASTM D6751). Table V lists the results and also compares them with those of petroleum diesel fuel. It can be seen from this table that the biodiesel produced in this research has the required properties to be used in diesel engines.

The ASTM standard for biodiesel viscosity is 1.9-6.0 mm2/sec at 40°C, It was watched that the viscosity of UFO was 32.5mm^2/sec, while for biodiesel was observed to be 5.63 mm^2/sec, which was in the range prescribed by ASTM. The density of UFO and biodiesel was observed to be around 0, 91 g/cm^3 and 0.88 g/cm^3. It was in the range suggested by ASTM. The ASTM standard for biodiesel density is 0.86 g/cm^3. The cloud and pour points of the biodiesel got from UFO in this experiment are higher than those for customary diesel. Both cloud and pour points were 6°C and 1°C for the biodiesel while 3°C and <-7°C for regular diesel. This is because of chemical properties of the crude UFO samples, comprising of 65% saturated fatty acid alkyl chains [31]-[33]. Cetane number is the pointers of start properties of the diesel fuel. As can be seen in Table 4, the estimation of the cetane index for biodiesel got from UFO was 48.9 while a typical value for diesel fuel is 46. It was higher than those in diesel fuel in light of the fact that higheroxygen content in biodiesel contrasted with oil diesel [1], [32]. Flash and Combustion Points are exceptionally homogeneous; the flash point is a parameter to consider in the handling, storage, and safety of fuels and flammable materials. The values of the two parameters are higher than those for oil diesel (85-95 ℃), and hence, shows that there is no alcohol residue in biodiesel structure. Consequently, High value of the two pointes diminish the danger of flame. That mean compared with petroleum diesel represents a good advantage for biodiesel. The acid value of biodiesel, which is a measure of free fatty acid content, also matched ASTM standards, as shown in table Iodine value a parameter that measures the level of unsaturation of the fat/oil, in result, the biodiesel obtained from the same oil should have similar iodine values. For our situation the iodine values for UFO and for obtained biodiesel was 116 g and 97.8 g iodine I_2/100 g respectively . This scattering at standard (120 g I_2/100 g) can be inferable from the dilution of samples with ethanol or there heterogeneity [19], [32].

IV. CONCLUSION

1. Combination of utilizing the UFO as a low-cost feedstock as well as Jordan Zeolit as a heterogeneous base catalyst overcomes problems associated with homogeneous acid and alkali-catalysed biodiesel production such as saponification and purification. Moreover, this method offers a less pollutant, more environmentally friendly and

economically profitable process for biodiesel synthesis

2. The optimum biodiesel yield reached 95.1% with optimum value of catalyst 7%. The biodiesel was described for its physical and fuel properties utilizing ASTM standard methods for biodiesel fuel quality assurance. Out of 9 properties tested, 7 of them met the ASTM criteria for fuel standard.

3. Production of biodiesel from used frying oils for diesel substitute is particularly important because of the decreasing trend of economical extracted oil reserves and the ecological issues caused because of the utilization of petroleum derivative.

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