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Production and Characterization of Biodiesel Produced from Seeds Oil of Sunflower (*Helianthus annuus*) Plants

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Abstract:

The aim of this work was the extraction of oil from the seeds of two types of Sunflower (Helianthus annuus) plants, (Bazian and Shahrazour).Two solvent extractions (N-hexane and Methanol) and two degrees for moisture (4 and 5%) were used for oil extraction.The results showed that the use of N-hexane was the most acceptable in terms of oil production amounted to oil ratio of 49.1% and 40.4% from Bazian and Shahrazour seeds respectively. The best moisture content in the Sunflower seeds was 4% with Bazian type and 5% with Shahrazour type. Also in this work studied the activity of CaO and KOH as a catalyst in biodiesel production by transesterification of oil with methanol. The data of methanolysis showed that the best result occurred when CaO at 1% w/w used and molar ratio of oil to Methanol was 1:6, at 65°C for 3 hours. In addition several parameters were tested of oil and biodiesel. The results were similar to the ASTM standards.Interestingly, the biodiesel produced from Bazian oil was the best in yield and matching the standard properties.

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Key words: Sunflower; extraction; oil; moisture; hexane; biodiesel; methanolysis ; CaO ; KOH.

1. INTRODUCTION

In recent years, because of the world energy crisis, reduction of the world's petroleum reserves and on the other hand increasing energy requirement and also environmental concerns, there is a great demand for alternative sources of energy. Therefore, many countries have started to perform a series of investigation on new fuel resources (Leung *et al.,* 2010). Among many possible sources, non-toxic, biodegradable and renewable biofuel which can be obtained from bio-resources such as vegetable oils or animal fats which is called "biodiesel", has attracted much attention as a promising alternative for fossil diesel fuels (Al-yozbaki *et al*.,2015). Biodiesel is very attractive due to its environmental benefits and especially the renewable resources. Significantly, it has lower emissions in compare with fossil diesel. In addition, biodiesel is better than diesel fuel because of no sulfur content and aromatic content, high flash point, and biodegradability (Patil and Deng, 2009). It doesn't raise the level of carbon dioxide in the atmosphere and leads to minimize the intensity of greenhouse effect (Gashaw and Lakachew, 2014). Biodiesel is usually prepared in the presence of homogeneous base or acid catalysts. With homogeneous base catalysts (sodium and potassium hydroxides, carbonates, sodium and potassium alkoxides, principally) the reaction is faster than with acid catalysts (sulfuric acid, phosphoric acid, hydrochloric and sulfonic acid principally) (Ma & Hanna, 1999). On the other hand, heterogeneous catalysts could improve the synthesis methods by eliminating the neutralization salts in the glycerol and therefore the number of separation steps can be reduced. Also, heterogeneous catalysts

can be used in a fixed-bed reactor, leading to safer, cheaper and more environment-friendly operation (Romero *et al*., 2011).

The sunflower is one of the four most important oilseed crops in the world, and it was first domesticated in the Americas.Oil-type sunflower seeds contain 38–50% oil and about 20% protein(FAO, 2010). Sunflower oil, extracted from the seeds, besides the use of sunflower oil in the human diet, this oil can have a wide variety of uses in different industries, such as illumination oil, soaps, cosmetics, pharmaceuticals, emulsifiers, lubricants and greases, drying and semi-drying oils in paints, varnishes and other coatings, plastics and polymers, synthetic rubber manufacture, fat liquors for the leather industry, or produce biodiesel fuel (Škorič *et al*.,2015).

In this study, two extraction solvents (Methanol and Hexane) and two degrees for moisture (4 and 5%) for each plant seeds (Shahrazour and Bazian) were used. Two catalysts (KOH and CaO) were used for the biofuel production (biodiesel), and many parameters were investigated for oil and biodiesel.

2. MATERIAL AND METHODS

The seeds obtained from sunflower plants (*Helianthus annuus*) which grow in the Shahrazour and Bazian region in the province of Sulaymaniya, Iraq for oil extraction.

2.1 Preparation of seeds

The choice of seeds quality and the exclusion of seeds affected as well as the impurities associated with seeds, and then broke the seeds using a ceramic mortar and the coat were removed from them. The seeds crushed to a very fine powder using electric seed grinder then transferred to the oven at 105 ºC for 2-5 hours to freeze the protein and make oil extraction from seeds easier, taking into consideration the moisture content of the seeds.

2.2 Determination of Moisture Content of the Seeds

The seeds of 30 grams dried in oven at 105°C for 5 hours and the seeds weight measured every two hours until the stability of weight, samples were removed from the oven, placed in a desiccators for 30 min. to cool, then removed and re-weighed. The percentage of moisture is calculated from the equation below (Akpan *et al*., 2006) :

$$\% \text{ Moisture} = (W_1 - W_2) / W_2 \times 100$$

W_1 = Original weight of the sample before drying
W_2 = Weight of the sample after drying

2.3 Oil Extraction

In this study, two extraction solvents (Methanol and Hexane) and two degrees for moisture (4 and 5%) for each type of seeds (Shahrazour and Bazian) were used. The extraction of Sunflower oil was carried out in the laboratory, about 90-100 gm of the seeds powder putted in thimble and settled in the center of the extractor and 150 ml of solvent was weighed and placed on it , the seeds left immersed extraction solution for 24 hours, then added about 50ml of solvent and Soxhlet apparatus was heated at 70°C. The solvent boiled and vaporized through the vertical tube into the condenser at the top. The condensate liquid start dropping into the thimble containing the sample and the extracted oil seeped through thimble into the flask via the siphon. Extraction process lasted for two hours, until the disappearance of the yellow color from the solvent. After that the sample was transfer to rotary evaporator to remove the solvent and dried in the oven at 85°C about 2 hours (Kyari,2008).The yield percent of Sunflower oil was calculated using the following formula :

$$\% \text{ Yield} = Y_1 - Y_2 / Y_1 (100)$$

Y_1= weight of seeds before extraction
Y_2=weight of seeds after extraction

2.4 Refining of Extracted Oil

2.4.1 Degumming and Neutralization

Crude Sunflower oils were degummed by heating the oils to 80 °C, mixed with water (5% vol.) and stirred for 15 min by magnetic stirrer,and allowed to stand in the separating funnel and the aqueous layer was removed. The procedure was repeated to ensure removal of most gums. For neutralization; water-degummed oil was heated to 80 °C and was initially mixed intensively with a phosphoric acid (14 %) in amount of 0.1 % by weight of oil. After a short reaction time (for approximately 5 min), the acid is partially neutralized with 0.1N NaOH in amount of 0.3 % by weight of oil. The total reaction times were 10 minutes. The phosphatides that are hydrated by this way are removed by centrifugation for 20 min. to obtain oil with low content of phospholipids, Ca and Mg. Then transferred into a separating funnel, allowed to stand for 1 hour and the soap was separated from the oil. Hot water was added again and again to the oil solution until the soap residues was removed (Zufarov *et al.*,2008).

2.4.2 Bleaching

Sample of neutralized oil was placed in a beaker, heated to 90°C, activated clay (10% of oil weight) was added and the mixture was stirred continuously for 30 min. The temperature was allowed to rise up to 110°C for another 30 min. and mixture was filtered in an over at 70°C (AL-Harbawy and AL-Mallah, 2014).

2.5 Characterization of Sunflower Oil

In this study important physicochemical characteristics was measured which are reliable in vegetable oils used in biodiesel production.

2.5.1 Determination of Density and Specific Gravity

Density bottle of 5ml capacity was weighed (W_0), filled with oil, then stopper inserted and reweighed (W_1). The oil was substituted with water after washing and drying the bottle and weighed (W_2). The expression for specific gravity (Akpan *et al.,* 2006) and Density is:

$$\textbf{Density} = (W_1 - W_0) / 5$$

$$\textbf{Sp.gr} = (W_1 - W_0) / (W_2 - W_0)$$

= Mass of the substance / Mass of an equal volume of water

2.5.2 Determination of pH value

Samples of 2gm was each poured into 25ml glass beaker, 13ml of hot distilled water was added to each sample and stirred slowly. The mixtures were cooled in a cold-water bath to 25°C. The pH meter electrode was standardized with buffer solution and then immersed into the sample and pH value was measured (Akpan *et al.,* 2006).

2.5.3 Determination of Refractive Index

Few drops of oil samples were transferred to glass slide of the refractometer. Through the eyepiece of the refract meter, the dark portion viewed was adjusted to be in line with the intersection of the cross, In this case the pointer on the scale pointed to the refractive index and values were recorded (Akpan *et al.,* 2006).

2.5.4 Determination of Kinematic viscosity

ASTM D445, standard test method for kinematic viscosity of transparent and opaque liquids was used. The kinematic viscosity test calls for a glass capillary viscometer with a calibration constant (**c**) given in mm^2/s. The kinematic viscosity determination requires the measurement of the time (**t**) the fluid takes to go from point **A** to point **B** inside the viscometer

(ASTM D445). The kinematic viscosity (**V**) is calculated by means of the following equation:

$$V = c X t$$

2.5.5 Determination of Free Fatty Acids (FFAs)and Acid Value

FFAs were determined according to the AOCS Official Method F9a-44 (Firestone, 1994). The oil sample (5 g) was mixed with 30 mL of neutral ethyl alcohol in a conical flask and titrated against standard KOH solution using phenolphthalein as an indicator. FFA was calculated through the following formula:

$$\text{FFA (\% as Oleic acid)} = V X N X 282 X 100 / W X 1000$$

V = Volume of KOH used
N = Normality of KOH
282 = Molecular weight of Oleic acid
W = Weight of sample in gram
Then, **Acid Value = FFA X 2**

2.5.6 Determination of Saponification Value

AOCS Method Cd3-25 was used to determine the saponification value of the feedstock's (AOCS, 2010). The method includes refluxing the known amount of oil with a fixed but excess amount of alcoholic KOH. The amount of KOH remaining after hydrolysis was determined by back titrating with standardized 0.5 M HCl and the amount of KOH consumed during saponification was calculated.

$$\text{Saponification value} = (B-S) X M / W X 56.1$$

Where:
B = volume of 0.5 M HCl required to titrate blank, ml
S = volume of 0.5 M HCl required to titrate test portion, ml
M = molarity of HCl solution
W = mass of test portion in grams

2.5.7 Determination of Iodine Value

Samples of 0.4gm of oil were weighed into a conical flask and 20ml of carbon tetra chloride (CCL_4) was added to dissolve the oil. Then 25ml of Dam's reagent was added to the mixture using a safety pipette in fume chamber. Stopper was inserted and the content of the flask was vigorously swirled. The flask was placed in the dark for 2 and half hours. Then, 20ml of 10% aqueous potassium iodide (KI) and 125ml of water were added using a measuring cylinder. The solution was titrated with 0.1N Sodium thiosulphate ($Na_2S_2O_3$) solutions until the yellow color almost disappeared. Few drops of 1% starch solution indicator was added and titration continued by adding thiosulphate drop wise until blue coloration disappeared after vigorous shaking. The same procedure was used for blank test and other samples (Kyari, 2008). The iodine value (I.V) is given by the expression:

$$\textbf{Iodine Value} = (V_1 - V_2)\ X\ 126.9\ X\ N / M$$

V_1 = Volume of sodium thiosulphate used for blank
V_2 = Volume of sodium thiosulphate used for determination
126.9 = Molecular weight of I_2
N = Normality of sodium thiosulphate used
M = Mass of the sample

2.6 Biodiesel synthesis

Biodiesel was produced from Sunflower seeds oil (Bazian and Shahrazour) which extracted by hexane with moisture of 4 and 5% respectively.

A- By using KOH as catalyst

Transesterification of refined sunflower oil was carried out in a 1000 ml round bottom flask equipped with a condensation system. Then was placed in a Heating mantle with magnetic stirrer. Operational conditions were as follows: catalyst weight: 1% based on initial oil weight; methanol: oil molar ratio 6:1;

reaction temperature, 65ºC; under vigorous mixing made by heating mantle with magnetic stirrer and reaction time was 60 min (Ghanei, 2014). Oil of 500g was heated up to the reaction temperature. Afterwards, the catalyst was dissolved in methanol that was weighed before. Finally, the methanol solution was added to the reactor and reaction was started. After the mentioned reaction time, the flask was cooled immediately and reaction mixture was transferred to refine it.

B- By using CaO as catalyst

Catalyst preparation:

The CaO catalytic active form was prepared by calcination at 900ºC for 3 hours. The catalyst powders were stored in dark, well closed, glass bottles in a desiccator (Veljkovic *et al*., 2009).

Experimental procedure

The reaction was carried out in a 1000 mL round glass equipped with a condenser and heating mantle with magnetic stirrer.The reaction conditions of the sunflower oil methanolysis were as follows: catalyst weight 1% based on initial oil weight; methanol: oil molar ratio 6:1; reaction temperature 65ºC; under vigorous mixing made by Heating mantle with magnetic stirrer and reaction time was 3 hours.(Vujicic *et al*., 2010). Steps following of the reaction was similar to the interaction of sunflower oil with KOH, as shown in the above.

2.7 Refining of Biodiesel

Purification process of biodiesel which product by two methods described in above were occurred following the steps in below (Gerpen, 2005) :

1. After the finished of production process crude biodiesel cooled to the laboratory temperature ,used filter paper to get rid of the remnants of catalyst KOH.

2. Transfer to rotary evaporator at a temperature of 75ºC for the purpose of getting rid of excess methanol and re-use it.
3. Then put into a separating funnel and left for 24 hours under a lab temperature conditions. After settling two layers were separated, the upper layer represents biodiesel while the lower layer containing glycerin, gum and impurities.
4. Washing biodiesel by hot water 50ºC (20% of biodiesel weight) and blend for a few minutes and then left to be separated for a full hour. This step was repeated at least three times to remove remained catalyst and glycerol.
5. Biodiesel put inside the oven at a temperature of 70ºC for 6 hours to fully get rid of the water.

2.8 Biodiesel characterization

Used standard ways industrially, in the estimation of physicochemical properties in accordance with the US standard (ASTM, 2008) in biodiesel derived from Sunflower oil, were measured for each of the Density ,pH value, free fatty acids, acid value, kinematic viscosity at 40 ºC, iodine value and refractive index in the same steps in the above (paragraph 2.5), while other specifications have been measured as shown in the below.

2.8.1 Determination of Biodiesel yields

Calculated the weights of Sunflower oil extracted from the seeds as well as biodiesel derived from it and used in the estimation of biodiesel ratio by equation:

$$\% \text{ Biodiesel Yield} = W_0 / W_1 \text{ X } 100$$

W_0 = Weight of biodiesel (g)
W_1 = Weight of oil used (g)

2.8.2 Determination of Sulfur content

The sulfur content of the biodiesel samples was measured adoption on the method described by (Drews, 1998).

2.8.3 Determination of Ash and Sulfated ash

The ash (ASTM D482) and sulfated ash determination of biodiesel samples in this study was done following (ASTM D874).The biodiesel is ignited and burned and put in a muffle furnace and then treated with sulfuric acid to determine the percentage of sulfated ash present in the biodiesel.

Ash % w/w = wt (gm) ash / wt (gm) of sample X 100

Sulfated Ash %w/w = wt (gm)ash Sulfate / wt (gm) of sample X 100

2.8.4 Determination of water and sediment

Water and sediment tests were done as per(ASTM D2709) standard test method for water and sediment in middle distillate fuels by centrifuge.Water and sediment testing is done using 100 mL of biodiesel and centrifuging it at 1870 rpm for 11 minutes.

2.8.5 Determination of Cetane number

The measure of cetane number in biodiesel done by using formula set forth in the below (Prasad, 2000).

Cetane number = (Diesel Index X 0.72) + 10

2.8.6 Determination of Flash point and Fire point

The flash point and fire point were measured with a Pensky-Martens closed cup tester using (ASTM D93) standard test methods for flash point by Pensky-Martens closed cup tester.

2.8.7 Determination of Cloud point and Pour point

There are several tests that are commonly used to determine the low temperature operability of biodiesel. Cloud point (ASTM D2500) and pour point (ASTM D97) are two of these tests and is included as a standard in ASTM D6751.

3. RESULTS AND DISCUSSION

The data in table (1) expressed the yields of oil extracted from two types of Sunflower (*Helianthus annuus*) seeds by two solvents and two degrees for moisture. The results obtained for moisture content were varied from those recorded by the other researchers variation from the literatures (Mohamad and Al-Saade, 2010) which was between 9-10 %. The data indicate that the best degrees for moisture was 4% and 5% for Bazian and Shahrazour respectively, this differences in the best moisture to extraction may be due to the plant variety.

Table 1: Oil yield extracted from two types of Sunflower (*Helianthus annuus*) seeds using two solvents and two moisture degrees.

Solvent and moisture / Seeds type	Yields of oil (%)			
	Hexane with 4%	Hexane with 5%	Methanol with 4%	Methanol with 5%
Shahrazour	33.5	40.4	9.1	11.5
Bazian	49.1	46.7	18.2	15.6

The data in the above table indicate that the oil yield (49.1% and 40.4% for Bazian and Shahrazour respectively) fall within the standard ranges of oil content 38–50% of Sunflower seeds (FAO, 2010) depending on the variety and Bazian was the best .The best solvent for oil extraction was hexane while methanol was not suitable for oil extraction from the seeds. This result seems likely to those reported in other studies using the same plant (Samuel and Dairo, 2012 ; Pagliero *et al*., 2007).

The results in table (2) exhibit the physicochemical properties of oil extracted from two types (Shahrazour and Bazian) of Sunflower (*Helianthus annuus*). The results mentioned in the table refer to the differences among the physicochemical properties of the two types of oil.

Table.2: Physicochemical properties of oil extracted fromtwo types (Shahrazour and Bazian) of Sunflower (*Helianthus annuus*) plants.

Parameter	Abbreviation	Unit	Oil type	
			Shahrazour	Bazian
Density	D	g/ml	0.892	0.913
Specific gravity	S.gr	----	0.894	0.915
Free fatty acid	FFA	%	0.47	1.1
Acid Value	AV	mg KOH /g	0.94	2.2
Refractive Index	RI	----	1.461	1.467
Kinematic viscosity at 40°C	KV	mm^2/s	31.1	32.2
Saponification value	SV	mg KOH /g	191.4	194.1
Iodine value	IV	$gI_2/100g$	130.6	133.2
pH	pH	----	6.9	7.1

The density, specific gravity, free fatty acid, acid value, refractive index, kinematic viscosity, saponification value, iodine value and pH values of Bazian seeds oil was higher than the values of Shahrazour type. This differences may be due to the nature of the plant variety and the nature of the land which it is grown in, which was effected on the properties of oil . It seems likely that physicochemical properties of oil produced in this study was identical to those reported in other studies using the same plant species(Vujicic *et al.,* 2010; Phani *et al.,* 2014). These oil after refining were used to production the biodiesel by using Methanol, KOH and CaO as catalysts. Fig.1 explains the steps of production of biodiesel and steps of refine this biodiesel.

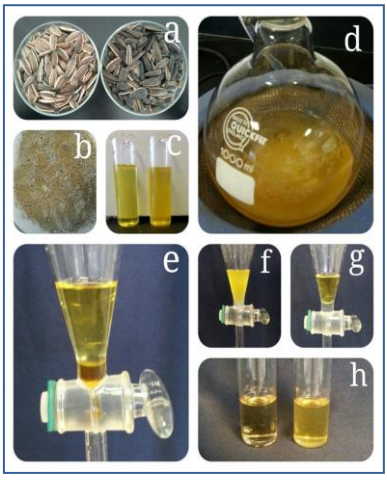

Fig. (1): Transesterification of Sunflower (*Helianthus annuus*) oil by CaO as catalysts.(a) Shahrazour (left) and Bazian (right) seeds**.(b)** Powder of seeds**. (c)** Crude oil (right) and Refin oil (left)**. (d)** Mixing of oil, methanol and CaO**. (e)** Separation of biodiesel**. (f)** Washing of biodiesel**. (g)**Separation of biodiesel after washing**. (h)** Refine biodiesel of Shahrazour (left) and Bazian (right) .

The results in table (3) explained the physicochemical properties of biodiesel produced from Sunflower oil by KOH and CaO as catalysts. Results shown that both types of catalyst used for the production of biodiesel were appropriate, but CaO was more suitable. Results obtained were similar to the results of other studies (Veljkovic *et al*., 2009 ; Ghanei, 2014).

All the qualities which have been tested for all types of biodiesel were identical to ASTM standard specifications.

Table.3: Physicochemical properties of biodiesel produced by using KOH and CaO as catalysts from two types (Shahrazour and Bazian) of Sunflower (*Helianthus annuus*) plants.

Parameters	Shahrazour Biodiesel		Bazian Biodiesel		ASTM D6751 Standard
	KOH	CaO	KOH	CaO	
Yield (%)	91.0	93.6	93.1	95.4	Report
Density (g/ml)	0.864	0.851	0.872	0.871	Report
Iodine value (gI_2/100g)	132	131	128	130	Report
Kinematic Viscosity at 40°C(mm^2/s)	4.0	4.7	4.4	4.9	1.9-6.0
Refractive Index	1.458	1.457	1.453	1.456	Report
Free fatty acid (%)	0.36	0.4	0.15	0.35	Report
Acid Value (mg KOH/g)	0.72	0.8	0.3	0.7	0.8 max
Cetane number	47.0	47.6	49.2	47.1	47 min
Flash point (°C)	175	180	170	184	130°C min
Fire point (°C)	190	198	185	192	Report
Ash(%)	0.02	0.1	0.01	0.09	0.1 max
Sulfated ash(%)	0.016	0.020	0.018	0.020	0.02 max
Sulfur content(%)	0.05	0.05	0.04	0.04	0.05 max
Water and sediment (%)	<0.05	<0.05	<0.05	<0.05	0.05 max
Cloud point (°C)	-1	0	-2	-1	Report
Pour point (°C)	-4	-3	-6	-4	-15 to 10
pH	7.0	6.8	6.9	6.7	Report

4. CONCLUSIONS

In oil extraction step: hexane was better than the methanol for oil extraction and the moisture content were (4% for Bazyan and 5% for Shahrazour) due to the high oil yield and good quality which can recommended for industrial usage. Oil obtained from Bazyan type was more than Shahrazour type, and these oil were used to production the biodiesel by KOH and CaO as catalyst. The qualities that has been tested for all types of biodiesel were identical to ASTM standard specifications .

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