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Impact of primary treated sewage water on the chemical composition of safflower oil as a potential candidate for biodiesel production

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***Abstract: Biodiesel** is gaining expanded interest all over the world. It is considered a renewable source of energy. In this work, safflower was irrigated with primary treated sewage water (PTSW) without fertilization and the extracted seed oil was transesterified to yield biodiesel. Harvest index and oil yield was compared to that irrigated with fresh water (FW). Physical properties for both oils and their parent biodiesels were carried out i.e. viscosity, specific gravity, Iodine value, and cetane number for biodiesel (CN) and chemical composition using gas chromatography.A reduction in harvest index reached 47% for PTSW in comparison to FW was recorded. Also, there were many differences in physical properties (sp. gr., viscosity, and iodine value) and chemical composition (fatty acid profile) between both oils and their parent biodiesels. It was found that poly unsaturated fatty acids were increased in PTSW treatment compared to FW especially linoleic acid. The produced biodiesel complied with the standards in many aspects except CN as it was 35.2 against >54 for European standards and >55 for American standards.*
***Keywords**:biodiesel, linoleic acid, primary treated sewage water, safflower*

I. Introduction

It is worthy to mention that energy availability has a tremendous impact on society modernization[1]. Because of the limited time for fossil energy resources to be depleted, renewable energy resources were searched for. Renewable energy resources include biomass[2] and municipal solid waste[3] incineration to recover energy, hydrogen production from wastes[4], and cultivation of energy crops (biofuel crops). Also, production of bio methane from wastes is practiced in Germany[5], France[6]and Denmark[7].

The main elements for agriculture production are fertile soil and fresh water. Rain based cultivation is the dominant practice in Europe for the production of canola[8], and safflower[9] for biodiesel production, and inSpain, sugar beet is used for bioethanol production[10].

The use of rain water in the production of biofuel crops as mentioned above raised the issue of "water for food" or "water for fuel" and such act may be viewed as non-ethical as some countries is suffering or will suffer water shortage per capita and also, it is one of the causes of rising food prices around the world [11, 12].

Safflower (*Carthamustinctorius*L.) is aherbaceous plant. It belongs to the tribe cynarae of the compositae family. The seeds of safflower composed of 26-37% oil, 12-22% proteins, 5-10% moisture and 35-52% hull[13]. Safflower has been cultivated for industrial purposes for the production of varnishes because of its high linoleic acid content, preparation of textile dyes from colorful petals and animal feed from the meal resulted from oil extraction [14]. Fatty acid profile of safflower oil is an important criterion for oil quality. Also, such profile determines its best commercial uses [15].

The chemical composition of safflower oil and seed oil content percentage varied according many factors. Among these factors, are salinity[15], irrigation regime[16, 17], genotye and plant genetic variety[14, 19].

Chemical analysis of vegetable oils were carried out using different methods ranged from infrared spectrometry (near-NIR [19] and mid-MIR [20]), chromatography (HPLC, [21], and GC [22]. and hyphenated with MS [23], and luminescence methods (fluorescence [24], and chemiluminesecnce [25]).

Biodiesel is defined as fatty acid monoalkyl ester (FAME). The ester production is carried out by transesterifying vegetable oil or animal fat with methanol or ethanol in alkaline medium. Such monoalkyl esters resemble mineral diesel in carbon chain length with the advantage of possessing oxygen atoms which eases oxidation and energy release. Because of this, biodiesel may be considered a renewable and sustainable energy resource [26].

So, it is mostly encouraged to use primary treated municipal sewage in the irrigation of non-edible crops. This reduces the costs of treatment and at the same time has an added value represented by the production of wood[27], forage[28] and essential oil[29]. In the same context, marginal land[30] is used for the cultivation of biodiesel crops instead of fertile soil to save land for food and feed production.

In this work, safflower was cultivated in sandy loam soil and irrigated with primary treated sewage water (PTSW) and the chemical composition of the produced oil was investigated and compared with that produced from freshwater (FW). Also, the extracted oil from both treatments was transesterified and analyzed as biodiesel.

II. Materials and Methods

2.1 Growing Location and Crop Management

The experiment was carried out in Abdo-Basha village, Kafr El-Dawar, El Beheira governorate. Water samples were taken as follows: one from the Zohra drainagein Kafr El-Dawwar, the second water samples from freshwater irrigation canal. Water samples were analyzed according to standard methods for water and wastewater analysis [31].Safflower seeds were brought from the center of agricultural research in Giza. ¼ feddan of safflower was cultivated and irrigated with fresh water and the other ¼ feddan of safflower was irrigated with primary treated sewage without fertilization. The harvest was at June 2012.

2.2 Oil Extraction

Oil was extracted by pressing methods.The seeds heated to facilitate the extraction of the oil. Oil percent was calculated as

$$Oilpercent\% = (\text{oil weight}) \,/\, (\text{weight of seeds}) * 100)$$

2.3 Reaction Procedure of Biodiesel Production

The FAME production was carried out in a lab transesterification apparatus with a cross volume of 1 l equipped with thermostat, mechanical stirrer, heating system and vacuum. The first step: 300 ml oil was heated up to 55 C^o for 90 min at 600 rpm; then the methanol and KOH were added. On the basis of 100% vegetable oil 12%-mass of methanol and 1%-mass catalyst were used in the first transesterification step. After cooling, two phases were formed. The upper phase consisted of methyl esters, and the lower phase contained glycerol, the remaining catalyst and the soap formed during the reaction. The methyl esters formed has undergone to a new procedure (second step) using this time a concentration of 2%-mass of methanol and 0.1%-mass catalyst. After separation of the two fractions by sedimentation, the second part of glycerol was removed. The crude FAME were washed with distilled water until the pH of water used was lower than 7. The obtained biodiesel sample was dried after filtration under vacuum (approx 60 mbar absolute) at 70 C^o for 15 min[32].

2.4 Physical and Chemical Characteristics

The physical and chemical properties of the extracted oil and the parent biodiesel were analyzed according [33]. The analyzed parameters are density (specific gravity), viscosity, iodine value (IV), and cetane number (CN).

2.5 Fatty Acid Composition

Fatty acid composition of seed oil was determined using agilent 6890 series gas chromatography (GC) equipped with flame ionization detector and capillary column (30m×0.25mm×0.25mm). About 0.1 ml oil was converted to methyl ester using 1ml sodium methoxide (NaOMe) (1 M) in 1ml hexane before being injected into the GC. The detector temperature was programmed at 240ºC with flow rate of 0.8 ml/min. The injector temperature was set at 240ºC. Hydrogen was used as the carrier gas. The identification of the peaks was achieved by retention times by means of comparing them with authentic standards analyzed under the same conditions[22].

III. Results and discussion

3.1 Water Analysis

Irrigation water (FW and PTSW)used in this study was analyzed to investigate its physical and chemical characteristics. Results were tabulated in table 1.

Table 1 Physical and chemical characteristics of irrigation water

Parameters	Unit	PTSW	FW	*permissible limits of irrigation water		
				Weak	moderate	Strong
pH	-	7.36	7.15	6.5-8.4		
Ec	**(mmhos/cm)**	2.21	0.87			
TDS	ppm	1414.0	557.0	<450	450-2000	>2000
Na^+	**$Meql^{-1}$**	7.9	3.2	<3	3-9	>9
Ca^{++}		8.8	3.6	-	-	-
Mg^{++}		2.6	1.2	-	-	-
K^+		1.49	0.15	-	-	-
HCO_3^-		8.9	3.8	<1.5	1.5-8.5	>8.5

Cl^-		9.4	3.9	<4	4-10	>10
SO_4^{--}		2.49	0.45	-	-	-
SAR	-	3.32	2.06	-	-	-
$N(NH_4^+)$	mgL^{-1}	5.78	4.76	-	5	>5
P		0.863	0.238	-	-	-
Cu		0.004	0.001	-	0.2	>0.2
Fe		0.119	0.011	-	5	>5
Mn		0.211	0.007	-	0.2	>0.2
Zn		0.264	0.020	-	2	>2
Pb		0.003	0.010	-	5	>5

* Permissible limit for irrigation water according to [34]

From the results obtained it can be concluded that the type of water used (PTSW) is of moderate type[34]. This is because it has many parameters that fulfill such type, such as pH, TDS, bicarbonate and chlorides.

3.2 Safflower Oil

Safflower oil composition in some countries (with a multitude of studied parameters) was tabulated in table 2. It is the % of linoleic acid (C18:2) that determines the quality of safflower oil. Also, the standard composition of safflower oil was mentioned [35]. From the obtained results, safflower oil extracted from FW irrigated plants was of low linoleic acid type as it has 27.9 against 71-75% for the standard. Also, it can be considered of high oleic and palmitic acid content as it records 32.6 and 22.6% for both acids in comparison to the standard which has 16-20% and 6-8% oleic and palmitic acid respectively.

3.3 Yield Parameters

It was found that irrigation with PTSW has a great effect on yield parameters. In regards to harvest index, Kg fed^{-1} (weight of seeds per feddan), it was found to be 880 and 1640 Kg fed^{-1} for PTSW and FW respectively. This reveals that there is 47% reduction in yield. Such decrease was reflected on the reduction of oil production (280 L against 528 L for PTSW and FW respectively). Oil % for both treatments was not significantly affected as it was 32.1 and 31.8% for FW and PTSW respectively. Such decrease in harvest index and oil production was reported by some authors. It was reported by [36] that canola irrigated with sewage showed a decreased yield. Also,[37] recorded that sunflower yield and harvest index were affected negatively by using municipal sewage. At the same time, [38] reported a slight decrease in yield and harvest index for castor and sunflower plants irrigated with municipal wastewater.

3.4 Fatty Acids Profile

PTSW safflower oil havesomechanges in fatty acid profile in comparison to FW safflower oil. The ratios of fatty acidsi.e.Lauric, myristic, palmitic, stearic, linoleic, arachidic and behenicacids were changed.Oleic acid increased significantly (32.67 % against 57.39 % for FW and PTSW respectively).

In regards to the percentage of saturated fatty acid and unsaturated fatty acids, unsaturated fatty acidswere significantly increased (66.04 % against 83.36 % for FW and PTSW respectively).

Another important notice appeared from fatty acids analysis is the percentage of saturated and unsaturated fatty acids. GC analysis report showed that the percentage of saturated fatty acids were high in oil extracted from plants irrigated with FWwhile those irrigated with PTSW have high content of unsaturated fatty acid. The change in these ratios affected physical and chemical properties.

3.5 Density of Oil and Its Parent Biodiesel

Density of safflower oil extracted from plants irrigated with FWis lower than that irrigated with PTSW (0.962 g cm^{-3} against 0.966 g cm^{-3}). Results were in agreement with that reported by [22] where they studied jatropha oil properties and reported that density of oil decreased with molecular weight and increased with increasing unsaturated acids level. As shown in table 2,the oil extracted from plants irrigated with PTSW had high unsaturated degree (mono unsaturation and ploy unsaturation) as compared with those irrigated with FW.After esterification, safflower biodiesel extracted from plants irrigated with FW has a density of 0.880 g cm^{-3} while the biodieselextracted from PTSW hasa density of 0.890 g cm^{-3}.

3.6 Viscosity of Oil and Its Parent Biodiesel

The viscosity of a fluid is a measure of its resistance to flow [39]. It is also called dynamic viscosity. In brief there is another expression, it is kinematic viscosity. The kinematic viscosity "v" related to dynamic viscosity "η" through the density "d" $v = \eta/d$ [40]**.** Thus it is affected by density.Safflower oil has a viscosity of 27.4mm^2/s forFWcompared to 33.1mm^2/s for PTSW. The viscosity of fat and oil greatly depends on the structure of the compound, and it is affected by factors such as chain length, position, nature and number of double links [41]. Also,kinematic viscosity is the primary reason that prevents direct use of crude vegetable oil [33]. It wasreported that direct use of vegetable oils as fuel for engines is problematic[42]. Because of their high viscosity 20 times higher than diesel fuel and it caused low volatility and don't burn completely and this is in agreement with [43]. Generally, viscosity tends to increase with increasing degree of saturation and chain length. The trans double bonds impart higher viscosity than cis double bond [44, 45].Viscosity of crude oil was very high but it was reduced after esterification. The viscosity of safflower biodiesel was 3.21 and 3.9 mm/s for FW and PTSW treatments respectively. Results were in agreement with [46] who reported that due to triglyceride molecular weight before esterification and that was harmony with [47] and [48].Reducing viscosity is the major reason why vegetable oil transesterified to biodiesel [45].If fuel viscosity is high, the injection pump will be unable to supply sufficient fuel to fill the pumping chamber [45]. The viscosity of biodiesel FAME is also affected by contents of mono unsaturated fatty acid MUFA and poly unsaturated fatty. Lower contents of poly unsaturated fatty acid(PUFA) in FAMEcauses lower viscosity of biodiesel [33, 49]. Reduction of viscosity may be due to inclusion of methoxy moiety in the biodiesel molecule in addition to fatty acid component with the removal of high viscous glycerol moiety.It wasreported that the kinematic viscosity must be between 3.5 and 5 mm^2/S. They also reported correlation between viscosity and purity of methyl ester[50]. Because of that the produced biodiesel from the two treatments comply with the standards (table 3).

3.6 Iodine Value

The iodine value is an important measure that allows determination of the unsaturation degree of the oil[50] within a mixture of fatty materials, regardless of the relative shares of mono, di, tri and polyunsaturated compounds [51]. This property greatly influences fuel oxidation [50]. However the higher degree of unsaturated FAME is not suitable for biodiesel [52]. The unsaturated molecules react with atmospheric oxygen and are converted to peroxide, crosslinking with other unsaturated molecules and may become polymerized [53, 54]. It wasreported these results and added to their The IV of fatty acid compounds depend on M W [55].

The oil iodine value (IV) before esterification was very high as compared after esterification. IV of safflower oil is 120.64 and 135 g I_2/100 g oil for FW and PTSW respectively. This is in agreement with the results obtained from GC results and also it reflects the increase of unsaturated fatty acid percent (table 2) in the oil extracted from PTSW. Although IV differs from that reported by [56] for two types of safflower, high linoleic (67.8-83.2%) and high oleic acids (73.6%), and the reported IV for both is 135-150 and 90-100 g I_2/100 g oil respectively, while the oil under investigation recorded IV of 120.6 (FW) and 135 (PTSW). This may be explained as the obtained oil (FW) is neither high in linoleic acid (27.1%) nor high in oleic acid (32.7%), while oil (PTSW) has increased unsaturation; it recorded 135 g I_2/100 g oil.

IV for the esterified oil for both of the treatments was 89.1 and 97.6 g I_2/100 g for FW and PTSW respectively. Such reduction in IV compared to values recorded for raw oil may be attributed to the increase in molecular weight resulted from methoxy moiety addition.Therefore, biodiesel manufactures put broad lines for the value of the iodine number known standard specifications. The obtained IV values (for esterified oil) correspond to the standard specifications for biodiesel production worldwide for example in Germany standard "DIN E 51606" was <115 and also in France. In Sweden standard "SS 15 54 36" was <125, finally in Austria "ON C1191" was <120 [56].

3.7 Cetane Number

CN is a significant expression of diesel fuel quality. It is actually a measure of a fuel's ignition delay (ID) and start of combustion of the fuel. Higher speed diesels operate more effectively with higher CN [54]. High CN correlated with reduced nitrogen oxides NO_x exhaust emission. The structure of fatty acid can also influence emission. Emission of NO_x being reduced by increasing saturation due to shorter ID times [57]. [42]also reported that longer fatty acid carbon chains and the more saturated the molecules, the higher CN will be.Safflower biodiesel have almost the same CN for the two treatments (FW and PTSW). It was 32.3 and 32.4 for FW and PTSW respectively.[58]studied the parameters affecting the cetane number of biodiesel and said that generally, CN increased with chain length. It was also affected by number of double bonds and their position in the chain [46, 57, 59]. [60]observed that the CN of rapeseed which had 94% unsaturated is lower than that other biodiesel fuel. [61]studied the effect of single double bond in the fatty acid profile of biodiesel on its properties and the relationship with CN. They recorded that CN of saturated fatty acid higher than mono unsaturated fatty acid and poly unsaturated was very low. [62]recorded that CN influence the combustion process and engine performance and it is affected by physical properties that was able to predict CN.This value only is not commensurate with the standard specification for the production of biodiesel. It may be improved by blending or utilizing another alcohol such as ethanol and iso-propanol.

IV. Conclusion

It can be concluded that safflower oil extracted from plants irrigated with primary treated sewage water (PTSW) possesses some differences in chemical compositions compared to that extracted from plants irrigated with freshwater (FW). The differences are increase in polyunsaturated fatty acids and variations in some saturated fatty acids. Also, results proved that PTWS safflower oil is a potential candidate for biodiesel production. The PTWS safflower biodiesel complies with the international standards except for its cetane number (CN), which can be enhanced by blending with or utilizing iso-propanol in transesterification.

Table 2: Safflower chemical composition in various countries carried out by different authors (%)

Location	Variables studied	Linoleic C18:2	Oleic C18:1	Stearic C18:0	Palmitic C16:0	C14:0	C20:0	C22:0	Remarks	author
Uzbekistan	--	78.00	9.30	2.50	8.90	0.2	0.3	0.3		[23]
Iran	Genetic profiles	55.82-75.23	15.40-35.26	1.70-3.81	7.05-9.74					[18]
Iran	Irrigation regimes	67.28-72.65	19.05-21.35	3.19-7.95	1.22-3.31					[17]
Turkey	17 varieties	17.00-83.40	8.20-73.90	0.80-3.60	6.60-9.50					[13]
Turkey	Genotype and sowing dates	38.70-74.10	15.50-48.20	2.10-3.60	6.00-9.80					[14]
Egypt	FW	27.94	32.67	2.37	22.58	1.39	2.63	1.83	4.66 (Linolenic, C18:3)	This work
Egypt	PTSW	20.09	57.39	1.26	10.48	1.39	1.41	0.96	4.97 (Linolenic, C18:3)	This work
Standard safflower oil		71.00-75.00	16.00-20.00	2.00-3.00	6.00-8.00					[35]

Table 3: International standards for the production of biodiesel compared to that obtained in this study

Parameter	Unit	Austria C1191	France	Germany DIN E51606	Italy UNI 10635	Sweden SS 154536	USA D 6751-02	EU EN 1421	This study
Density @ 15C^o	g cm^{-3}	0.85-0.89	0.87-0.90	0.875-0.900	0.86-0.90	0.87-0.90	-	0.890-0.900	0.890
Viscosity@ 40C^o	mm^{2}s^{-1}	3.5-5.0	3.5-5.0	3.5-5.0	3.5-5.0	3.5-5.0	1.9-6.0	3.5-5.0	3.9
IV	g I_2/100 g oil	<120	<115	<115	-	<125	-	<120	97.6
CN	-	> 49	> 49	> 49	-	> 48	> 47	> 51	32.3

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