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Oil industry waste: a potential feedstock for biodiesel production

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ABSTRACT

The worldwide rising energy demands and the concerns about the sustainability of fossil fuels have led to the search for some low-cost renewable fuels. In this scenario, the production of biodiesel from various vegetable and animal sources has attracted worldwide attention. The present study was conducted to evaluate the production of biodiesel from the oil industry waste following base-catalysed transesterification. The transesterification reaction gave a yield of 83.7% by 6:1 methanol/oil molar ratio, at 60°C over 80 min of reaction time in the presence of NaOH. The gas chromatographic analysis of the product showed the presence of 16 fatty acid methyl esters with linoleic and oleic acid as principal components representing about 31% and 20.7% of the total methyl esters, respectively. The fourier transform infrared spectroscopy spectrum of oil industry waste and transesterified product further confirmed the formation of methyl esters. Furthermore, the fuel properties of oil industry waste methyl esters, such as kinematic viscosity, cetane number, cloud point, pour point, flash point, acid value, sulphur content, cold filter plugging point, copper strip corrosion, density, oxidative stability, higher heating values, ash content, water content, methanol content and total glycerol content, were determined and discussed in the light of ASTM D6751 and EN 14214 biodiesel standards. Overall, this study presents the production of biodiesel from the oil industry waste as an approach of recycling this waste into value-added products.

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1. Introduction

The fast-growing human population and the urge of high life standards have necessitated large-scale exploitation of fossil fuel-derived energy resources. This large-scale exploitation of fossil fuels results not only in the depletion of such non-renewable energy resources, but also in global warming due to the release of greenhouse gases (especially CO₂) during their burning.[1–3] In order to fulfil the increasing demand of fuels and to combat with the uncertainty in availability of the fossil fuels, there is a need to develop renewable fuels as a stable energy alternative that will meet world demand while mitigating climate change to a considerable extent. In this regard, the development of biofuels from biomass resources has attracted worldwide attention because of being renewable and environment friendly.[1–7]

Biodiesel derived from plants, animals or organic oils by transesterification is an environment-friendly, renewable and biodegradable energy resource which is considered as an alternative to the petroleum-based fuel.

[2, 4–8] Biodiesel is composed of mono-alkyl esters of long-chain fatty acids and have been reported to be primarily obtained from a number of conventional as well as non-conventional vegetable fats, including soybean oil,[4, 5, 9] palm oil,[10] olive oil,[11] safflower oil,[12] jojoba oil,[13] hazelnut oil,[14] cotton seed oil,[15] rapeseed oil,[4, 16] mahua oil,[17] tobacco seed oil [18] and jatropha seed oil.[5, 19] These oils are converted into first-generation biodiesel by esterification or the transesterification process. [20, 21] Although the ‘first generation’ biofuel has environmental benefits and helps to improve the supply of domestic energy, however, there are some concerns associated with the production of such biofuel which set the limits for their increased production. One of the major disadvantages of first-generation biofuel is its increasing competition with the food supplies and high costs as an emission abatement technique. [1] The high cost of first-generation biodiesel feedstock and bioethanol led to the emergence and the need of second-generation biofuel. [1, 22] The second-generation biofuel predominantly makes use of the non-food

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biomass like cellulose or lignocellulose. [1, 23] These include such materials as stover from sugar, bagasse, municipal solid waste, crop residues, short forest crops and vegetable grasses. [23]

Keeping the context in view, the present study was conducted to produce biodiesel from the waste of oil industry using the optimized conditions for esterification reaction. The quality of the biodiesel was evaluated by fourier transform infrared spectroscopy (FTIR) and GC-FID (gas chromatography-flame ionization detector) analysis as well as by estimating its basic fuel properties.

2. Materials and methods

2.1. Materials and preliminary analysis

The yellow-coloured oil industry waste, originating from soybean and canola oils, used for this study was collected from Kashmir Banaspati, United Ghee Industries, Mansoorabad, Faisalabad. Before the start of this experiment, the waste was tested for free fatty acids (FFA), iodine value and peroxide value following the standard American Oil Chemists' Society methods F 9a-44, Cd 1-25 and Cd 8-53 and Cd 3-25, respectively. [12, 24] Pure standards of fatty acid methyl esters () were obtained from Sigma-Aldrich (UK). All other chemicals and reagents were of analytical grade and purchased from Merck chemical company.

2.2. Transesterification reaction

The transesterification reaction was carried out in a 250 mL three necked round bottom flask equipped with a thermostat, a sampling outlet, a mechanical outlet, a reflux condenser and a mechanical stirrer set at 750 rpm, as already reported by Rashid and Anwar. [12] The whole process of biodiesel production in the present study has been shown in a flow chart in supplementary data. Initially 10 g of waste oil sample was taken in the three necked round bottom flask and was pre-heated on a heating plate for a few minutes at 40° C. A mixture of 0.5 g anhydrous NaOH/60 mL of anhydrous methanol was added in this pre-heated oil sample. The reaction was preceded for 80 min at 60°C and 750 rpm. It was then allowed to cool at room temperature which resulted in the separation of the reaction mixture into two layers with the upper layer of methyl ester and the lower layer of glycerol (mixture of glycerin, remaining catalyst and the soap formed during the reaction). The two layers were separated by sedimentation and the fraction of methyl ester was then purified by distillation at 60°C. The methanol and water present in this fraction were removed by heating it on a hot plate for a

few minutes at 100°C. The yield of the methyl esters was calculated as:

$$\text{Yield of methyl esters (\%)} = \frac{\text{Grams of methyl esters produced}}{\text{Grams of oil industry waste used in reaction}} \times 100.$$

2.3. Analysis of the biodiesel from the waste of oil industry

2.3.1. Fatty acid profile by gas chromatography

The biodiesel samples were analysed by gas chromatography by running the samples on Shimadzu Gas Chromatograph (model 17 – A) fitted with a FID set at 260°C and a DB-Wax column (30 m × 0.25 mm). The temperature of the column was programmed from 120°C to 240°C at a linear flow rate of 2°C/min with hold up times of 2 min and 10 min at initial and final stages, respectively. Helium was used as a mobile phase with a flow rate of 30 mL/min. All the quantification was done by chromatography station for windows (CSW32) software (Data Apex Ltd. Prague, Czech Republic). The comparison of fatty acids was taken as relative percentages of total peak areas and compared with the FAME standards.

2.3.2. FT-IR spectral analysis

Thermo-Nicolet 6700P FTIR Spectrometer (equipped with diamond-attenuated total reflectance (ATR) sampling accessory, a deuterated triglyceride sulphate detector and KBr optics) was used to estimate the biodiesel produced from the waste of oil industry. It had a removable ZnSe crystal mounted in the spectrometer's sample compartment. The background spectra showed the existence at clear ATR accessory.

2.3.3. Fuel properties

Specific fuel properties of the biodiesel produced from the waste of oil industry were determined. The density of the biodiesel was determined by a digital density analyser (PAAR, DMA 38) following the American Society for Testing and Materials (ASTM) D 5002. By using a calibrated viscometer, the kinematic viscosity was measured following the ASTM D 445 and cetane number was measured using the ASTM D 613. The flash point was estimated by following the ASTM D 93, ash content was determined according to ASTM D 874 and the sulphur content was measured by using energy-dispersive X-ray fluorescence spectrometry (Model lab X -3000) following ASTM D 4294. Cloud and pour point determinations were made using ASTM D 2500 and ASTM D 97, cold filter plugging point (CFPP) was

determined using a CFPP apparatus (ISL, model CPP97-2) following the procedure of ASTM D 6371 and the acid value was measured following ASTM D 974. Analyses of the higher heating values (HHV) were performed by following ASTM D 4868. The oxidative stability of FAME was determined by the Rancimat method EN 14112, which utilizes 3 g of material per test. Detection of copper corrosion was made by the copper strip tarnish test following ASTM D 130. The water content (%), methanol content (%) and the total glycerol content (%) were determined by following the ASTM D95, En14110 and ASTM 6584 methods, respectively.

3. Results and discussion

3.1. Preliminary chemical properties of the oil industry waste

The preliminary analysis of the waste from oil industry was performed and it exhibited the FFA value of 2.1%, iodine number of 114.7 g of I₂/100 g of waste and peroxide value of 129.6 meq/kg of the waste. Since, the FFA value is less than 3% so the base (NaOH)-catalysed transesterification reaction was performed for the production of biodiesel from the waste of oil industry.

3.2. Quality of the biodiesel produced from the oil industry waste

Using the optimized conditions already reported by Rashid and Anwar, [12] 83.7% of biodiesel yield was

achieved at a temperature of 60°C with methanol/oil molar ratio of 6:1. The fatty acid composition (relative percentage) of the oil industry waste methyl esters (OIWMEs) determined by gas chromatography is specified in Figure 1 and Table 1. As indicated in Table 1, linoleic, oleic, elaidic, cis-11-eicosenoic, erucic, tridecanoic, linolenic, behenic, heptadecanoic, arachidic and palmitic acids were the main components of the OIWMEs with relative contributions of 31%, 20.7%, 6.3%, 6.3%, 6.0%, 5.9%, 5.8%, 5.2%, 4.3%, 3.2% and 2.5%, respectively. However, small amounts of pentadecanoic, myristoleic, stearic, palmitoleic and lauric acids with relative contributions of 1.1%, 0.6%, 0.5%, 0.4% and 0.2%, respectively, were also found to be present. The linoleic acid was found to be a dominant component (31%) of the OIWMEs which is in accordance with a number of previous studies indicating for a relatively higher percentage of linoleic acid in the biodiesel produced from a number of different sources, including jatropha, safflower and waste cooking oils. [12, 25, 26] Overall, OIWMEs had about 22.9%, 40.3% and 36.8% of the saturated fatty acids (SFA), monounsaturated fatty acids (MUFA) and polyunsaturated fatty acids (PUFA), respectively.

The presence of methyl esters was further confirmed by the FT-IR spectral analysis of the oil industry waste and OIWMEs. It is evident from Figure 2 that there were differences in the spectra of oil industry waste and OIWMEs, and three sharp peaks representing the presence of ester, carbonyl and methyl groups were observed in the OIWMEs.

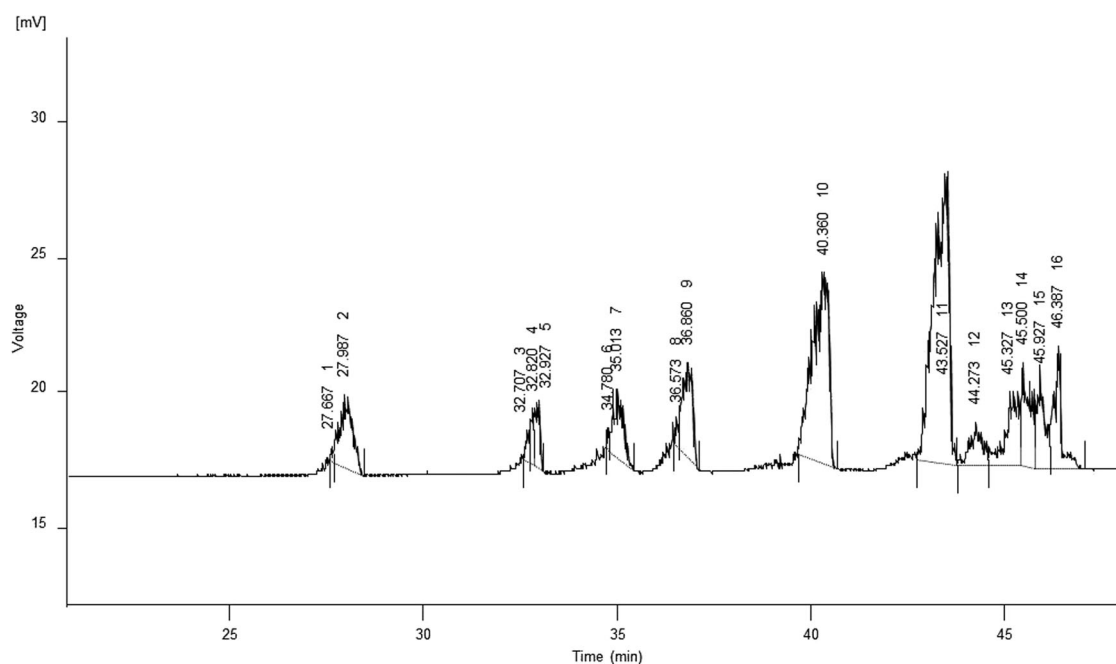


Figure 1. Fatty acid profile of OIWMEs.

Table 1. Fatty acid composition (g/100 g of fatty acids) of OIWMEs.

Fatty acid	C:D	Retention time	OIWMEs
Lauric acid	12:0	27.667	0.2
Tridecanoic acid	13:0	27.987	5.9
Myristoleic acid	14:1	32.707	0.6
Pentadecanoic acid	15:0	32.820	1.1
Palmitic acid	16:0	32.927	2.5
Palmitoleic acid	16:1	34.780	0.4
Heptadecanoic acid	17:0	35.013	4.3
Stearic acid	18:0	36.573	0.5
Elaidic acid	18:1	36.860	6.3
Oleic acid	18:1	40.360	20.7
Linoleic acid	18:2	43.527	31.0
Arachidic acid	20:0	44.273	3.2
Cis-11-Eicosenoic acid	20:1	45.327	6.3
Linolenic acid	18:3	45.500	5.8
Behenic acid	22:0	46.927	5.2
Erucic acid	22:1	46.387	6.0
SFA			22.9
MUFA			40.3
PUFA			36.8

Notes: SFA = saturated fatty acids; MUFA = mono unsaturated fatty acids; PUFA = poly unsaturated fatty acids; C:D = lipid numbers, where C is the number of carbons in the fatty acids and D is the number of double bonds in the fatty acid

3.3. Fuel properties of OIWMEs

The fuel properties of the biodiesel obtained by the transesterification of oil industry waste as compared to standards are summarized in Table 2.

Kinematic viscosity is the key fuel property for persuading the atomization of a fuel upon injection into the diesel engine ignition chamber and the formation of engine deposits. [27] The kinematic viscosity of the biodiesel produced from the oil industry waste was found to be 4.91 mm²/s. The ASTM D6751 and EN

14214 gave the kinematic viscosity limits ranging from 1.9 to 6.0 and 3.5 to 5.0 mm²/s, respectively. Thus, in the present study, the biodiesel produced from the oil industry waste has the kinematic viscosity within the range of the American as well as EU biodiesel standards. Moreover, this value is in accordance with the values reported in some previous studies.[12, 25]

The suitability of a fuel as an alternative to already existing fuels in the market is determined by its HHV which is a measure of the energy produced when fuel is completely burnt up and the utmost yield of the esters. The greater the yield of esters, higher will be its HHV. The HHV of biodiesel from the oil industry waste was about 44.7 MJ kg⁻¹. Although the specifications for HHV do not exist in ASTM D6751 and EN 14214 standards, however, we found that the HHV is in agreement with the values (45.2 and 39.8 MJ kg⁻¹) reported in the previous studies for biodiesel produced from safflower oil [12] and tobacco seed oil, [18] respectively.

The cetane number is the parameter which measures the tendency of self-ignition, facilitates the cold start and helps in less exhaust gases during combustion. A higher value of the cetane number is an indicator of more ignitability of the fuel. The cetane number of the biodiesel produced from the waste of oil industry was found to be 57.24 which is greater than ASTM D6751 and EN 14214 standards (Table 2). This value is also in accordance with the previous studies reporting for cetane values of 52.3, 53.7 and 51 for the biodiesel produced from safflower oil, [12] soybean oil [28] and tobacco seed oil, [18] respectively.

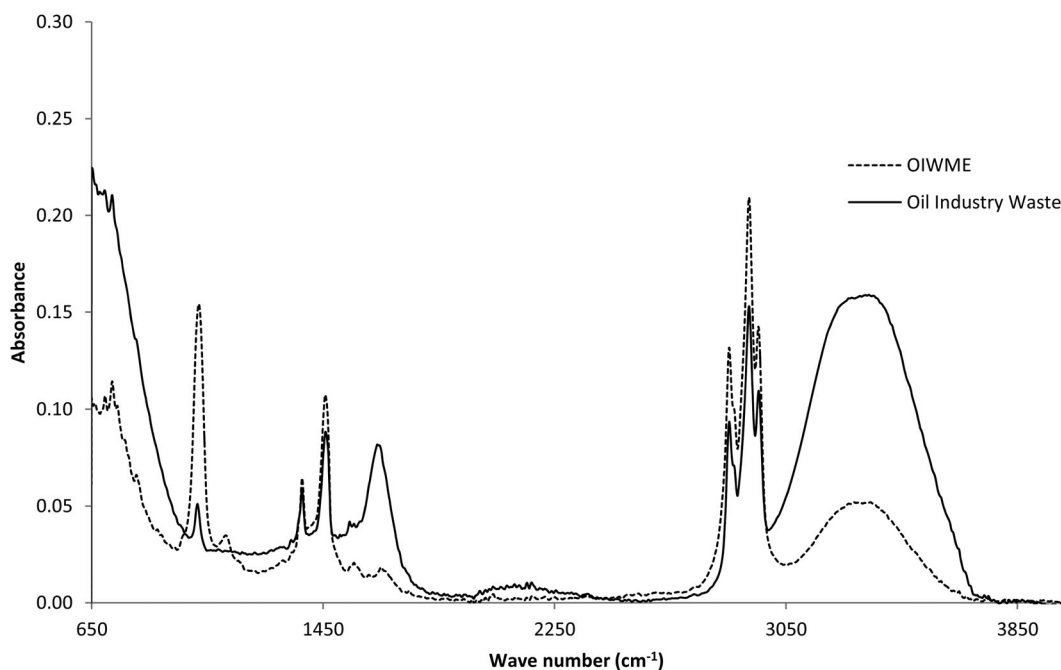
**Figure 2.** Comparison of FT-IR spectra of the oil industry waste and OIWMEs.

Table 2 . Fuel properties of OIWME.

Fuel property	Method	OIWME	ASTM	EN
	(ASTM)		D6751	14214
Kinematic viscosity (mm ² /s;40°C)	D445	4.91 ± 0.001	1.9–6.0	3.5–5.0
Cetane number	D 613	57.24 ± 0.02	47 min	51 min
Cloud point (°C)	D 2500	−5.1 ± 0.1	Report	− ^a
Pour point (°C)	D 97	−8.91 ± 0.001	− ^b	− ^a
Flash point (°C)	D 93	171 ± 0.02	93 min	101 min
Ash content (%)	D 874	0.01 ± 0.001	0.02 max	0.02 max
Sulphur content (%)	D 4294	0.021 ± 0.01	0.05 max	–
CFPP (°C)	D 6371	−2 ± 0.1	–	–
Copper strip corrosion	D 130	1a	No. 3 max	No. 1 min
Density (15°C), kg m ^{−3}	D 5002	894 ± 0.02	–	860–900
Oxidative stability (h)	EN14112	3.7	3 min	6 min
HHVs	D 4868	44.7 ± 0.002	–	–
Acid value (mg of KOH/g)	D 974	0.32	0.50 max	0.50 max
Water content (%)	D 95	<0.01	–	–
Methanol content (%)	EN14110	0.187 ± 0.015	–	–
Total glycerol (%)	D 6584	0.2525 ± 0.003	–	–

Notes: Values are mean ± SD of triplicates.

^aNot specified. EN 14214 uses time and location-dependent values for the CFPP instead.

^bNot specified.

Among the major problems associated with the commercial application of biodiesels are their cold flow properties. Cloud point (CP), pour point (PP) and CFPP are the main low-temperature flow properties which were studied for biodiesel produced from the waste of oil industry. These points must be sufficiently low as the engine will not be able to start if the biodiesel is frozen. The CP, PP and CFPP for the biodiesel from the oil industry waste were −5.1°C, −8.91°C and −2°C, respectively. ASTM D6751 and EN 14214 standards do not specify the values for these low-temperature flow properties because of large seasonal and geographical temperature variabilities, the CP, PP and CFPP for the biodiesel from the oil industry waste are relatively lower as compared to a number of the previous studies.[12, 25, 29]

Ash content (parameter related to the storage and safety of fuel and flammable materials) represents the amount of inorganic contaminants and the concentration of soluble metal soaps in a fuel. The ash content came out to be 0.01% (± 0.001) which is within the specified limits (max 0.02%) of ASTM D6751 and EN 14214 standards.

The sulphur content came out to be 0.021% (± 0.01) which is within the acceptable limit (max 0.05%) of the specifications provided by ASTM D6751 standards. As the low sulphur content helps to protect both the environment and engine life so the presence of relatively low sulphur content in the biodiesel plays an important role in improving its acceptability. Flash point was found to be 171°C (± 0.02) which is greater than the flash point values of the standards ASTM D6751 (93°C) and EN 14214 (101°C). A relatively higher value of the

flash point is also a point of advantage for the biodiesel, as it reduces the risk of the undesirable fire.

The density of the fuel is an important parameter for affecting the performance of the engine, as it determines the mass of the fuel and air–fuel ratio within the combustion chamber. [2] The density of biodiesel was found to be 894 kg m^{−3} (± 0.02) which is within the range of the specifications (860–900 kg m^{−3}) provided by EN 14214 standards. This value of density is also in accordance with the previous studies reporting the densities of 874, 880 and 890 kg m^{−3} for the biodiesel produced from safflower, rapeseed and soybean oils, respectively. [12] The oxidative stability of the biodiesel produced from the waste of oil industry was found to be 3.7 h which meets with the oxidative stability requirements of the ASTM D6751 standards reporting for minimum value of 3 h. The acid number for the biodiesel produced in the present study was found to be 0.32 mg of KOH/g which is within the specified limits of the ASTM D6751 and EN 14214 standards reporting for the maximum acid values of 0.5 mg of KOH/g. Similarly, the copper strip corrosion value of the biodiesel from the oil industry waste in the present study was also within the specified limits reported by ASTM D6751 and EN 14214 standards.

4. Conclusion

From the results of this study, it might be concluded that the oil industry waste may serve as an acceptable feedstock for biodiesel production by base-catalysed transesterification with methanol. The estimated fuel properties showed that the biodiesel meets the quality requirements of international standards including ASTM D6751 and EN 14214. The research might also serve as a basis for 'NO WASTE' strategy for protecting the environment by utilizing the pollution causing waste for the production of biofuel which is the need of hour in solving the energy crises in a developing country like Pakistan. This work also confirms the selective advantage of the second-generation biofuel over the first-generation biofuel for utilizing the wastes rather than crops or similar feedstock.

Disclosure statement

No potential conflict of interest was reported by the authors.

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