

# Preparation and Characterization of Biodiesels Produced From Waste and Pure Hydrogenated Cooking Oils of Various Brands Manufactured In Pakistan

Suryyia-Manzoor<sup>1</sup>, Mazhar-Abbas<sup>2</sup>, Tariq-Mahmood-Ansari<sup>3</sup>,  
Muhammad-Qasim<sup>4</sup> and Zaib-Ullah-Khan<sup>5</sup>

<sup>1,2,3</sup> Institute of Chemical Sciences, Bahauddin Zakaraiya University,  
Multan, 60000, Pakistan

<sup>4</sup> HDIP, Petroleum Testing Centre,  
Multan, 60000, Pakistan

<sup>5</sup> Faculty of Engineering Science and Technology, Hamdard University,  
Karachi, Pakistan

## Abstract

Biodiesel was produced from the pure animal fat, hydrogenated oils and their waste frying oils by a base catalyzed transesterification with methyl alcohol in the presence of NaOH. The various fuel parameters like specific gravity, flash point, cetane number, pour point, cloud point, sulfur contents, water contents, copper corrosion, Conradson carbon residue, kinematic viscosity, total acid number, phosphorous contents, total acid number, sulphated ash and sediments were determined and found analogous to ASTM standards. The fuel properties of biodiesel produced from pure hydrogenated vegetable oils was markedly different from the biodiesel produced from their waste fried oils. Pure hydrogenated vegetable oils, their waste fried oils and the biodiesels were analyzed by FT-IR. The recent research suggests that the biodiesels produced from the waste fried hydrogenated vegetable oils are comparable to those obtained from pure hydrogenated

oils in terms of fuel properties and are highly economical.

**Keywords:** Waste hydrogenated oils, transesterification, biodiesel

## 1. Introduction

The significant environmental consequences caused by the use of fossil fuels and the waste generated by them are the serious concerns of the present time. Improper management of waste not only contributes to the numerous adverse effects on public health but also has a major role in increasing global warming effects. The latter problem has been identified as the most critical environmental issue currently. Among the detrimental effects of global warming are loss of lives, extinction of species and submerging of islands due to a rise in

sea levels. Recently, there has been a lot of clamors regarding the environment and its sustainability [1].

Growing concern about atmospheric impact, the elevated cost of petroleum derivatives and the shrinking of fossil fuels have provoked the investigators to discover the substitute of petroleum sources. Biodiesel has gained a huge interest over the globe due to its biodegradability, domestic origin renewability and less toxic gas emissions [2, 3].

The disposal of waste veggie oils has remained a constant environmental issue. Mainly the waste veggie oil is dumped in to the water bodies which cause to form an oily layer on the surface of water to reduce the oxygen exchange and disturb the ecosystem. The disposal of oil waste furthermore cause problems in sewerage system jamming the flowing pipes and producing offensive odour. In addition the depreciation results of sewer pipelines could be reason the overflow of system which broken out different fatal diseases. Due to these reasons the recovering and reusing of it is very important [4]. The production of biodiesel from waste veggie oils can be a possible route to manage this issue, because the main constituents of veggie oils are triesters of glycerols [5]. The transportation and handling of biodiesel is safe due to its low flash point (150 °C), while on other hand, it is more stable as compared to ordinary fuels [6]. The biodiesel acquired significance importance in last few years as an alternative fuel. The production of biodiesel form dissipation of veggie oil is relatively simple and possesses many ecological benefits [7].

A grand financial dare for the production and commercialization of the biodiesel is the high fee of its feedstock. Furthermore, the use of edible oils as feeding stock for the production of bio-fuels can cause a harmful impact to the atmosphere, since it needs large area for their cultivation. Therefore it is very essential to find out the new sources for the feed stock of biodiesel [8].

It is claimed by the International Energy Agency (IEA) that the world's consumption of basic energy demand will increase by 55% between the 2005-2030 with the average rate of 1.8% every passing year. If this fashion continues, the whole world will face a massive energy disaster. To solve these problems thoroughly, carbon neutral and renewable biodiesel is indispensable for the atmosphere and economic point of view [9].

All kinds of fatty acids origin e.g. plants or animals' lipids can be used for the production of biodiesel. The use of these kinds of sources can be a serious threat to the food chain resulting in food crisis. That's why, the use of non eatable oils or waste cooking oils as raw materials is in focus currently which can be converted to biodiesel through the transesterification method [10-13].

## 2. Experimental

### 2.1 Chemicals

Isopropyl alcohol, sodium hydroxide, methanol and n-hexane were obtained from Merck. Acetic acid, hydrochloric acid and sulphuric acid were bought from Riedel-de-Haen, Germany. Oxalic acid was obtained from Fluka, Germany and toluene from Aldrich, Germany. All the chemicals used were of analytical grade.

### 2.2 Synthesis of biodiesel

Four different hydrogenated edible oils and animal fat was purchased from the market.

These hydrogenated oils and fat were converted into the biodiesel by using base catalyzed transesterification process. The procedure was accomplished by performing the following steps:

#### 2.2.1 Synthesis of the sodium methoxide

Methanol (200mL) was taken with the help of graduated cylinder and poured into the 500 mL of conical flask. NaOH was weighed accurately (3.70g) and added in methanol containing flask. The mixture was shaken for about one hour on the hot plate by means of magnetic stirrer till sodium hydroxide was dissolved and sodium methoxide was obtained.

#### 2.2.2 Transesterification process

Pure hydrogenated vegetable oil firstly was heated at elevated temperature (120°C). This was done to remove the water contents. Then this hydrogenated oil was cooled to 50 °C. 1 kg of hydrogenated oil was weighed and transferred to the sodium methoxide solution. The mixture was placed on hot magnetic plate and stirred at 250 revolutions per minute. During this time period, the temperature was maintained between 55 °C for about 80 minutes.

### 2.2.3 Separation of biodiesel

Upon the completion of transesterification reaction, the covering of the flask was removed to evaporate the extra methyl alcohol for about half an hour. Then the mixture was transferred to the separating funnel and the separating funnel was left for a night in vertical position. Two distinct liquid phases appeared inside the separating funnel. The upper layer was of crude biodiesel and the lower layer was of crude glycerin. The crude biodiesel layer was removed from the glycerin layer by transferring it in to the conical flask.

### 2.2.4 Purification of biodiesel

The unreacted reagents were eliminated by washing the crude biodiesel with de-ionized water (30 % by volume of biodiesel) in the separating funnel. The washing procedure was repeated three times. After that, the biodiesel was dried with Na<sub>2</sub>SO<sub>4</sub> and followed by filtration to obtain pure biodiesel.

### 2.3 Production of biodiesel from waste hydrogenated oils (WHOs)

The waste hydrogenated oils (WHOs) were obtained after the frying process in four different brands of pure hydrogenated oils and animal fat.

These waste hydrogenated oils and waste animal fat were converted in to biodiesel by base catalyzed transesterification method as mentioned in section 2.2.2.

### 2.4 FT-IR analysis

The pure hydrogenated cooking oils, waste frying oils and their respective biodiesels were analyzed by FT-IR. The spectra were obtained in the range of 4000 cm<sup>-1</sup> to 650 cm<sup>-1</sup> using FT-IR spectrophotometer (Cary 630 FTIR, Agilent USA).

### 2.5 Analytical methods

The different parameters of biodiesels were investigated in the accreditation of the standards of American Society of Testing Materials (ASTM).

ASTM D-1298 standard method was used for the determination of the specific gravity. The flash point of biodiesels was determined by adopting the ASTM D-

93 standard test method while the cetane number of biodiesel was determined by ASTM D-976 standard.

The ASTM D-97 standard test was employed for the determination of the pour point and ASTM D-2500 standard test for the cloud point. Sulfur contents were calculated by following the IP-63 standard test method.

ASTM D-473 standard test was used for the calculation of sediment by extraction while ASTM D-95 for the water contents. Copper corrosion, Conradson carbon residue, kinematic viscosity, total acid number, phosphorous contents and sulphated ash were evaluated by following the ASTM D-130, ASTM D-189, ASTM D-445, ASTM D-664, ASTM D-4951 and ASTM D-874 standard tests respectively.

## 3. Results and Discussion

### 3.1 FT-IR analysis

Infrared spectroscopy is considered as one of the chief analytical techniques for the characterization of various compounds due to its quickness, screening, quality and cost effectiveness for the analysis. IR spectroscopy is generally used to detect the conversion of triglycerides in to biodiesel [14-15].

The C–O stretching bands in ester are highly informative. All esters show strong C–O stretching absorption in the region of 1300 – 1030 cm<sup>-1</sup>. The normal C=O absorption in ester appears in the range of 1770 – 1725 cm<sup>-1</sup>.

The comparison shows that in the pure hydrogenated oil, waste hydrogenated oil and their biodiesel, the C=O group has the same band at 1752 cm<sup>-1</sup> and the C-O stretching vibrations give strong peak at 1171 cm<sup>-1</sup> due to C-O-C and medium peak at 1246 cm<sup>-1</sup>, whereas in their biodiesel these values changed to 1253 cm<sup>-1</sup>, 1179 cm<sup>-1</sup>, and 1127 cm<sup>-1</sup> respectively (fig. 1- 4).

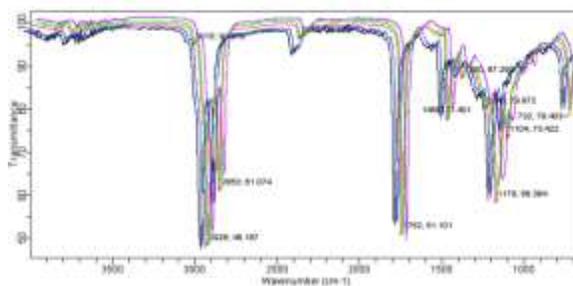


Fig. 1 FT-IR spectra of pure hydrogenated oils and animal fat.

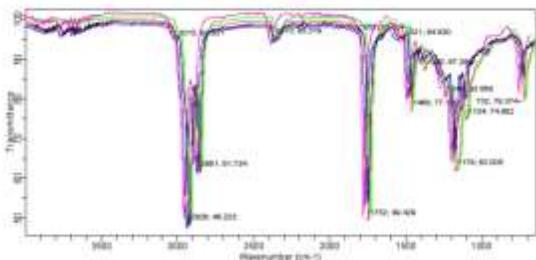


Fig. 2 FT-IR spectra of waste frying oils and waste animal Fat.

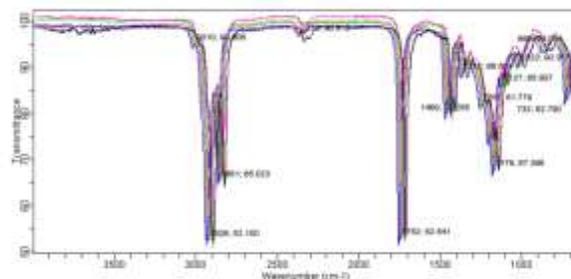


Fig. 4 FT-IR spectra of biodiesel from waste hydrogenated oils and waste animal fat

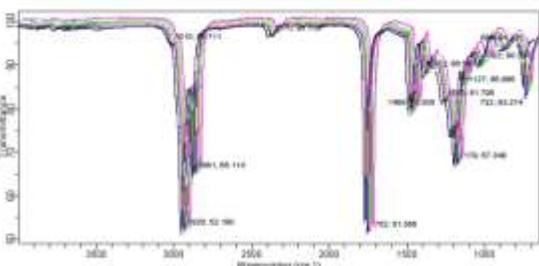


Fig. 3 FT-IR spectra of biodiesel from pure hydrogenated oils and animal fat.

### 3.2 Characterization of biodiesel

The different properties of biodiesel produced from virgin hydrogenated edible oils and their waste fried hydrogenated oils were ascertained and compared. Each biodiesel was analyzed for the determination of the specific gravity, flash point, cetane number, pour point, cloud point, sulfur contents, sediments, water contents, copper corrosion, Conradson carbon residue, kinematic viscosity, total acid number, phosphorous contents and sulphated ash. The chemical and physical properties of the biodiesels are mentioned in the tables 3.1, 3.2, 3.3, 3.4 and 3.5.

**Table 1.** Comparison between BDV and BDW from hydrogenated oil of sample 1.

Sr. No.	Parameter	Standard method	BDV sample 1 Hydrogenated oil	BDW sample 1 Hydrogenated oil	Specification
1	Specific Gravity @ 60/60°F	ASTM D-1298	0.8734	0.8779	Report
2	Flash Point (PMCC) (°C)	ASTM D-93	99	105	130 min.
3	Cetane (Calculated)	ASTM D-976	53.5	54.5	47 min.
4	Pour Point (°C)	ASTM D-97	3	3	-
5	Cloud Point (°C)	ASTM D-2500	6	9	-
6	Sulfur Contents (Wt %)	IP-63	0.04	0.02	0.05 max
7	Sediment by Extraction (Wt %)	ASTM D-473	L 0.01	L 0.01	0.05 max
8	Water contents (Vol. %)	ASTM D-95	L 0.05	L 0.05	0.05 max
9	Copper Corrosion; 3hrs @ 100°C	ASTM D-130	1a	1a	1a
10	Conradson Carbon Residue (Wt %)	ASTM D-189	0.05	0.04	0.05 max
11	Kinematic Viscosity at 40°C (cSt)	ASTM D-445	3.39	3.18	1.9-6.0
12	Total Acid Number (mg KOH/gm.)	ASTM D-664	0.14	0.15	0.05 max
13	Phosphorous contents (Wt %)	ASTM D-4951	0.0007	0.0007	0.001 max
14	Sulphated Ash (Wt %)	ASTM D-874	0.019	0.020	0.02 max

**Table 2.** Comparison between BDV and BDW from sample 2 Hydrogenated oil.

Sr. No.	Parameter	Standard method	BDV sample 2 Hydrogenated oil	BDW sample 2 Hydrogenated oil	Specification.
1	Specific Gravity @ 60/60°F	ASTM D-1298	0.8819	0.8819	Report
2	Flash Point (PMCC) (°C)	ASTM D-93	102	102	130 min.
3	Cetane (Calculated)	ASTM D-976	55.5	55.5	47 min.
4	Pour Point (°C)	ASTM D-97	3	3	-
5	Cloud Point (°C)	ASTM D-2500	9	9	-
6	Sulfur Contents (Wt %)	IP-63	0.05	0.05	0.05 max
7	Sediment by Extraction (Wt %)	ASTM D-473	L 0.01	L 0.01	0.05 max
8	Water contents (Vol. %)	ASTM D-95	L 0.05	L 0.05	0.05 max
9	Copper Corrosion; 3hrs @ 100 °C	ASTM D-130	1a	1a	1a
10	Conradson Carbon Residue (Wt %)	ASTM D-189	0.05	0.05	0.05 max
11	Kinematic Viscosity at 40°C (cSt)	ASTM D-445	4.18	4.18	1.9-6.0
12	Total Acid Number (mg KOH/gm.)	ASTM D-664	0.12	0.12	0.05 max
13	Phosphorous contents (Wt %)	ASTM D-4951	0.0006	0.007	0.001 max
14	Sulphated Ash (Wt %)	ASTM D-874	0.016	0.016	0.02 max

**Table 3.** Comparison between BDV and BDW from sample 3 Hydrogenated oil.

Sr. No.	Parameter	Standard method	BDV sample 3 Hydrogenated oil	BDW sample 3 Hydrogenated oil	Specification
1	Specific Gravity @ 60/60°F	ASTM D-1298	0.8730	0.8839	Report
2	Flash Point (PMCC) (°C)	ASTM D-93	95	98	130 min.
3	Cetane (Calculated)	ASTM D-976	53.5	54.5	47 min.
4	Pour Point (°C)	ASTM D-97	Zero	Zero	-
5	Cloud Point (°C)	ASTM D-2500	6	6	-
6	Sulfur Contents (Wt %)	IP-63	0.02	0.03	0.05 max
7	Sediment by Extraction (Wt %)	ASTM D-473	L 0.01	L 0.01	0.05 max
8	Water contents (Vol. %)	ASTM D-95	L 0.05	L 0.05	0.05 max
9	Copper Corrosion; 3hrs @ 100°C	ASTM D-130	1a	1a	1a
10	Conradson Carbon Residue (Wt %)	ASTM D-189	0.02	0.03	0.05 max
11	Kinematic Viscosity at 40°C (cSt)	ASTM D-445	3.29	3.11	1.9-6.0
12	Total Acid Number (mg KOH/gm.)	ASTM D-664	0.18	0.16	0.05 max
13	Phosphorous contents (Wt %)	ASTM D-4951	0.0008	0.0008	0.001 max
14	Sulphated Ash (Wt %)	ASTM D-874	0.018	0.020	0.02 ax

**Table 4.** Comparison between BDV and BDW from sample 4 Hydrogenated oil.

Sr. No.	Parameter	Standard method	BDV sample 4 Hydrogenated oil	BDW sample 4 Hydrogenated oil	Specification.
1	Specific Gravity @ 60/60°F	ASTM D-1298	0.8740	0.8779	Report
2	Flash Point (PMCC) (°C)	ASTM D-93	>110	>110	130 min.
3	Cetane (Calculated)	ASTM D-976	53.5	53.5	47 min.
4	Pour Point (°C)	ASTM D-97	3	Zero	-
5	Cloud Point (°C)	ASTM D-2500	9	6	-
6	Sulfur Contents (Wt%)	IP-63	0.02	0.04	0.05 max
7	Sediment by Extraction (Wt %)	ASTM D-473	L 0.01	L 0.01	0.05 max
8	Water contents (Vol. %)	ASTM D-95	L 0.05	L 0.05	0.05 max
9	Copper Corrosion; 3hrs @ 100°C	ASTM D-130	1a	1a	1a
10	Conradson Carbon Residue (Wt %)	ASTM D-189	0.04	0.03	0.05 max
11	Kinematic Viscosity at 40°C (cSt)	ASTM D-445	3.18	3.28	1.9-6.0
12	Total Acid Number (mg KOH/gm.)	ASTM D-664	0.15	0.19	0.05 max
13	Phosphorous contents (Wt %)	ASTM D-4951	0.0008	0.0008	0.001 max
14	Sulphated Ash (Wt %)	ASTM D-874	0.022	0.024	0.02 max

**Table 5.** Comparison between BDV and BDW from sample 5

Sr. No.	Parameter	Standard method	BDV sample 5	BDW sample 5	Specification.
1	Specific Gravity @ 60/60°F	ASTM D-1298	0.8720	0.8732	Report
2	Flash Point (PMCC) (°C)	ASTM D-93	108	>110	130 min.
3	Cetane (Calculated)	ASTM D-976	55.5	54.5	47 min.
4	Pour Point (°C)	ASTM D-97	3	Zero	-
5	Cloud Point (°C)	ASTM D-2500	9	3	-
6	Sulfur Contents (Wt%)	IP-63	0.04	0.03	0.05 max
7	Sediment by Extraction (Wt %)	ASTM D-473	L 0.01	L 0.01	0.05 max
8	Water contents (Vol. %)	ASTM D-95	L 0.05	L 0.05	0.05 max
9	Copper Corrosion; 3hrs @ 100°C	ASTM D-130	1a	1a	1a
10	Conradson Carbon Residue (Wt %)	ASTM D-189	0.04	0.02	0.05 max
11	Kinematic Viscosity at 40°C (cSt)	ASTM D-445	4.22	3.48	1.9-6.0
12	Total Acid Number (mg KOH/gm.)	ASTM D-664	0.14	0.25	0.05 max
13	Phosphorous contents (Wt %)	ASTM D-4951	0.0007	0.0005	0.001 max
14	Sulphated Ash (Wt %)	ASTM D-874	0.018	0.024	0.02 max

### 3.2.1 Specific Gravity

The biodiesels with low specific gravity values are preferred for use as fuel [16]. The specific gravity values of the biodiesel produced from pure hydrogenated oils were lower than those produced from waste hydrogenated oils. Hence, the biodiesel produced from the pure hydrogenated oils would be better than biodiesel produced from the waste hydrogenated oils according to present research work.

### 3.2.2 Flash point

The smaller values of flash point of the given biodiesel indicate the presence of volatile components while higher its value reduces the chance to catch fire [17]. The flash point of biodiesel produced from pure hydrogenated oils was less than the biodiesel produced from waste hydrogenated oils yet both were according to ASTM standards.

### 3.2.3 Kinematic viscosity

Viscosity of biodiesels is an important parameter because it greatly affects the injection system of engine. Biodiesels have usually higher values of viscosity. Biodiesel of high viscosity burns leisurely, because they have very small amount of lighter hydrocarbons to produce smoke [8]. The BDV sample 1, 3 hydrogenated oils and sample 5 have high kinematic viscosity than BDW sample 1, 3 & 5. While BDV sample 2 & 4 hydrogenated oils have lower kinematic viscosity than that of their respective BDW hydrogenated oils.

### 3.2.4 Copper corrosion

The corrosiveness of a fuel to the metal is measured by Copper strip corrosion test. This test is conducted to provide the protection to the fuel tanks and other automobile engine components [18]. Copper strip corrosion values of the BDV and BDW hydrogenated oils and animal fat are in accordance with ASTM D-130 (1a max.), hence it can be deduced that the biodiesels are neutral.

### 3.2.5 Sulphated ash and sulfur contents

Smaller the sulfur contents and sulphated ash, the better will be the biodiesel's quality and atmospheric friendly [8]. The sulphated ash of BDV hydrogenated

oils and BDW of animal fat are lower than their BDW hydrogenated oils and animal fat. Hence on the basis of this result, the BDV are environmental friendly. While the sulfur contents of BDV sample 1 and sample 5 are higher than their BDW, BDV sample 2, 3 & 4 hydrogenated oils have low value of sulfur contents than their BDW.

### 3.2.6 Conradson carbon residue

Smaller value of conradson carbon residue results in the low amount of aromatic hydrocarbons and improves the quality of biodiesel [8]. The conradson carbon residue values of BDV sample 1, 4 & 5 have higher values than their BDW. While the BDV sample 2 hydrogenated oil and BDW of sample 2 have the same values. But the conradson carbon residue of BDW sample 3 is higher than the BDV sample 3.

### 3.2.7 Water contents and sediments

The biodiesel is of good quality if it has no sediments and less water contents [8]. All the biodiesel samples have the values of water contents and sediment in the range of ASTM standards.

## 4. Conclusion

In the current research study, the biodiesel was produced from the pure and waste hydrogenated oils of different brands available in Pakistan by base catalyzed transesterification method. The yield of biodiesel was improved by methanolysis of hydrogenated oils at 1:6 molar ratio of oil and methanol at 50 °C. The biodiesel properties (specific gravity, flash point, cetane number, pour point, cloud point, sulfur contents, water contents, copper corrosion, conradson carbon residue, kinematic viscosity, total acid number, phosphorous contents and sulphated ash) were characterized and were found according to ASTM standards. The FT-IR analysis was performed for the pure, waste hydrogenated oils and their biodiesels, which confirmed the production of biodiesel. Though the biodiesel synthesized from pure hydrogenated oils is of good quality than biodiesel synthesized from waste fried hydrogenated oil yet the values obtained were within the range specified by ASTM. However, the biodiesel produced from waste hydrogenated oil is economically superior to the pure hydrogenated oils. The results show that the biodiesel produced from waste hydrogenated oils could be the alternative of the common biodiesel and petro-fuels.

The controlled plantation & regular accumulation of waste hydrogenated veggie oils could be prospective biodiesel backups which can trim down the importation burden of crude oil.

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