

# Indu-Diesel and Bio-Diesel as Renewable Fuel Synthesis from Used Automotive and Frying Oils

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## Abstract

Generally, the types of used oil by human to meet its necessary requirements regardless of its source, whether industrial or vegetarian or animal are considered as one of environmental contaminants which ceaseless due to continue consumption and continuation put forward of it in huge amounts by a large proportion of population in cities and rural areas. Automotive engines need to replace their lubricating oil after passing a distance reach to (1000 – 1500) km to ensure the safety and not affected the car engine leading to poses not inconsiderable oil quantity of industrial origin to environment, if accounting the number of cars and the amount of replaced oil by each car engine after commuting the aforementioned distance. Human need to an almost daily basis for frying the food which is eating it during the three daily meals leaving great amounts of agricultural or animal origin oils due to the large number of population. Both types form contamination because it contains a harmful or undesirable materials for human and environment together, especially if throw these oils directly without treatment. This research tackles the ability of producing two types of diesel fuels, the first one is indu-diesel which prepared from used automotive oils (UAO) and the second type is bio-diesel that synthesis from used frying oil (UFO) using concentrated sulfuric acid and thermal treated eggshell for two types of produced diesel fuel (PDF), then complete the production process by applied clay treatment with faujasite type Y-zeolite for indu-diesel from UAO and ethanol for bio-diesel from UFO and after that all required test were performed for two types and compared the results with ordinary diesel of fossil fuel origin. The results show that convergence in values of tests for both types of PDFs with ordinary diesel with a relative vantage for indu-diesel on bio-diesel. Thus, obtained a petroleum product (diesel) which can be used directly as fuel or mixing with ordinary diesel from one side, and from another side it can get-rid of UAO and UFO by economic, benefit, useful and eco-friendly method.

**Keywords:** : indu-diesel, biodiesel, ordinary diesel, automotive oil, frying oil, renewable fuel, eggshell and PDF

## 1. INTRODUCTION

Fossil fuel (petroleum) is considered as the second most important fluid used by human after water where uses for a long time in the lighting and cooking purposes and today is used in most walks of life (Mohammed et. al., 2008). Ground, marine and air means of transportation of all kinds and electrical generation power plant depended mainly on fossil fuel (Mohammed and Abbas, 2014). It is also used as major energy source required for the operation of various large and small factories and plants (Mohammed et. al., 2008 and Mohammed and Abbas, 2014). Fossil fuel is considered by itself as a source for many main chemical substances like lubricating oils, petroleum derivatives of all kinds' liquids and gases, petrochemicals, fertilizers, pesticides, plastics, fabrics, nylon, artificial silk, synthetic leather, medicines and others (Abbas and Abbas, 2013e). The fossil fuel at the present time is the primary source of consumed energy in the world where its consumption exceeds coal, natural gas, hydropower, nuclear power, renewable energies (Abbas, 2013 and Abbas, 2014b). Increasing the need for fossil fuel on a daily and continuously basis with the increase in population and development of life not only to meet the basic and necessary requirements of heating oil, cooking gas and transportation fuels but also to operate the increase in labor force which imply the establishment of further factories, plants and workshops and construction of new electric power plants, therefore need to fossil fuel to operate (Abbas, 2014b). Today, most of required fossil fuel kinds are gasoline and diesel fuel. Fossil fuel is found nearly in most area of glob but not in commercial quantities or sufficient amounts to fill the increasing local need and continuing demand which expected increases to about 40% by 2025 (NETL, 2005). With increasing the demand for gasoline and diesel fuels will increase the production of fossil fuel, thus increase the environment pollution (Abbas et. al., 2012).

Generally, the environment is suffering from acute pollution in some regions on the world with many types of contaminants. In addition to the pollution with heavy metals like lead (Pb), zinc (Zn), cadmium (Cd), chromium (Cr), Arsenic (As), copper (Cu), mercury (Hg), tin (Sn), Iron (Fe), lithium (Li), etc. resulted from wastes of tanneries, battery factories, metals coating and other industrial activities (Abbas and Abbas, 2013a; Abbas and Abbas, 2013b; Abbas and Abbas, 2013c; Abbas and Abbas, 2013d and Abbas and Abbas, 2014a), the pollution with dyes from textile mills (Abbas and Abbas, 2014b), thermal pollution resulted from electric power plants (Abbas and Abbas, 2014a), agricultural pollution with various types of agricultural wastes such as rice husk (Abbas and Abbas, 2014c), banana peels (Abbas et. al., 2014 and Abbas, 2014d), orange peels (Abbas, 2014e), pomegranate peels (Abbas, 2014c and Abbas and Ali, 2014), eggplant peels (Gharib et. al., 2014), potato

peels (Joodi and Abbas, 2014b and Abbas, 2014f), tea leaves (Joodi and Abbas, 2014a and Abbas, 2014g) etc., electronic pollution with electrical and electronic devices and old computers and others (Abbas and Abbas, 2014a and Abbas and Abbas, 2014b) and nuclear pollution of radiation leakage (Abbas, 2013), in addition to all previous types of pollution the environment experiencing in each moment and no less important degree of pollution with fossil fuel firstly when extracted from subsoil, secondly at refine it in refining plants and thirdly when combustion of it in means transportations or motors or factories or other different activities (Mohammed et. al., 2008 and Mohammed and Abbas, 2014). Due to concerns about the depletion, fluctuation of production, marketing of fossil fuel in addition to rise its price to record levels occasionally from one hand and to ensure the nation and economic security, providing sources energy can depended on them and been economic, clean and little contaminant to environment so, a lot of countries (which don't have this natural wealth in its territories or its territorial waters) have resorted to search and elicitation for many alternatives to this necessary material which indispensable in peacetime and wartime alike (Mohammed et. al., 2008 and Mohammed and Abbas, 2014).

From these alternatives are the renewable energies such as solar, wind, nuclear, hydropower, geothermal and wave's energies. However, these energies, in spite of it depend on cheap and available sources but it cannot be relied upon these energies on an ongoing basis either because it require either high-tech technology or volatility their sources or shortage the efficiency of energy produced like the intensity of sunlight and wind speed or the lack of availability in many countries such as geothermal and ocean energies (Abbas and Abbas, 2014a and Abbas and Abbas, 2014b) or because they represent a significant threat in the event of control loss on it like nuclear energy, and a best example of this, the last catastrophe of Fukushima in Japan at 2011 (Abbas, 2013). One of renewable energy types that represent an appropriate balance among cost of conversion, efficiency of produced substance, availability and cheapen of its raw material and low contaminating wastes to environment compared to various types of fossil fuels is Biomass energy which is the energy that dependent on produce biofuel from agricultural substances or agricultural residues or wastes of agricultural origin (Abbas, 2014b). Besides using agricultural residue in water treatment processes via adsorption or of various types of contaminants such as heavy metals (Abbas and Abbas, 2014a), dyestuffs (Abbas and Abbas, 2014b), pesticides (Abbas, 2014a), organic acids (Abbas et. al., 2013a; Abbas et. al., 2013b and Abbas and Abbas, 2014c), inorganic matters (Abbas, 2014a) and radioactive elements (Abbas, 2013); can employing them in the preparation of both kinds of biofuels (bioethanol and biodiesel) (Abbas, 2014e), an example of that, bioethanol which can be produced from various types of agricultural wastes like rice husks, orange peels, banana peels or others and biodiesel which can be synthesis from vegetable oils and wastes of used cooking oil (Uddin et. al., 2013).

Moreover, the fuels can synthesis from industrial wastes also; for instance, motors and machines that used fossil fuels in different types used also lubricating oils which must be replaced after passing certain distances, thus constitute a new source of environmental pollution in addition to the pollution produced from fossil fuel combustion in inner combustion engines (Uddin et. al., 2013 and Abbas, 2014e). One of applicable surrogates for fossil fuel in means of transportations is the fuel that synthesis from used lubricating oil wastes, which can produce from it the diesel named "indu-diesel". Currently, these materials (i.e., bio-diesel and indu-diesel) doesn't considered as fuel by themselves only, but it can be mixed with fossil fuel and used in motors and engines, to say the least. For example, instead of consumed 100% diesel by the engine, it can consume between 80 – 85% of fossil fuel and the rest is the alternative fuel (i.e., bio-diesel or indu-diesel). In this paper, the using of two types of wastes first one: wastes of agricultural or biological origin which is used frying oil (UFO), and the second: wastes of industrial origin which is used automotive oil (UAO) to synthesis of two kinds of diesel was conducted.

The types of diesel produced were bio-diesel from UFO and indu-diesel from UAO and all possible tests were achieved for these two types and compared with the natural diesel produced from fossil fuel. Thus, two problems are solved in same time; the first problem is provided new materials from available and cheapen sources can be used them to decrease the amount of ordinary fuel employed to operate the engines or motors and so, lowering the increased and continued demand on fossil fuel albeit with few percent and the second problem is minimize the pollution resulted from combustion of fossil fuel and lubricating oil replacement by economic, benefit, useful and eco-friendly method.

## 2. EXPERIMENTAL WORK

Not only during the use of the oil in the engine of motor vehicles or frying, but also during collection, storage or mixing with oil waste from other sources, the oil is degraded and contaminated with a number of impurities. These impurities are on one side the additives of the oil itself or some food residue that cannot be removed with ordinary filtration process. On the other side they result from the degradation process of the oil during its use, from the mechanical wear of the engine parts and from the combustion of the fuel or during the cooking process. During storage and mixing, water, dirt and other objects might enter the used oil as well. The most commonly

used process for many years and in particular for small to medium scale applications is the Acid-Clay process.

### 2.1 Preparation of Calcium hydroxide

100 g of Eggshell Waste (ESW) were collected freely from the local restaurants in Baghdad and washed triplicate with running tap water and once with distilled water to ensure remove all dirties and impurities from ESW. ESW were crushed perfectly with mortar and calcined in the furnace at 500°C for one hour then reacted with distilled water to produce calcium hydroxide.

### 2.2 Preparation of Indu-diesel from Used Automotive Oil

Used Automotive Oil (UAO) was collected freely from some local garages for washing and maintenance of cars in Baghdad. 100 g of UAO were firstly filtered from any impurities and dirt, fed to 250 ml three necks round bottom flask vertical, equipped with a water-cooled reflux condenser (one neck for thermometer, other neck for feeding materials and the third neck for condenser) placed on a laboratory heating mantle with magnetic stirrers (Electrothermal EMA series) and treated gradually with different weight ratios of concentrated sulfuric acid (98% H<sub>2</sub>SO<sub>4</sub>) varied from (1 to 10% based on the weight of UAO) and leave the solution for one hour. After that (1 – 10% of calcium hydroxide (Ca(OH)<sub>2</sub>) strong base (prepared in section (2.1) above) based on the weight of UAO) were adding to the mixture and leave the mixture for 30 minutes. Then adding Y-zeolite which prepared according to (Abbas and Abbas, 2013e and Mohammed and Abbas, 2014) with a ratio between (1 – 10% based on the weight of UAO) added to previous mixture. All previous steps were performed at different temperatures ranged between (55 – 100 °C) for various time periods ranged from (10 – 150 minutes) and different agitation speed ranged between (100 – 600 rpm). Finally all flasks' content was transferring to filtering process to filter produced indu-diesel from the other layer which is black asphalt sludge.

### 2.3 Preparation of bio-diesel from Used Frying Oil

Used Frying Oil (UFO) was collected freely from some local restaurants in Baghdad. 100 ml of UFO were firstly filtered from any impurities and dirt, fed to 250 ml three necks round bottom flask angled, equipped with a water-cooled reflux condenser placed on a laboratory heating mantle with magnetic stirrers (Electrothermal EMA series) and treated gradually with different weight ratios of concentrated sulfuric acid (98% H<sub>2</sub>SO<sub>4</sub>) varied from (1 to 10% based on the weight of UFO) and leave the solution for one hour. After that (1 – 10% of calcium hydroxide (Ca(OH)<sub>2</sub>) strong base (prepared in section 2.1 above) based on the weight of UAO) were adding to the mixture and leave the mixture for 30 minutes. Then ethanol which was prepared according to (Abbas, 2014e) with a ratio between (1 – 10% based on the weight of UFO) added to previous mixture. All previous steps were conducted at different temperatures ranged between (55 – 100 °C) for various time periods ranged from (10 – 150 minutes) and different agitation speed ranged between (100 – 600 rpm). Finally all flasks' content was transferring to separating funnel allowing the solution mixture to settle for 20 – 30 minutes using a ring stand to separate produced the layer of bio-diesel from the other layer which is glycerine.

## 3. PROPERTIES OF PRODUCED DIESEL FUEL (PDF)

### 3.1 Density

Density is defined as the mass of substance per the volume occupied by it. Density of PDF ( $\rho_{PDF}$ ) can be measured by two methods, the first one is commonly method using graduated cylinder after washing it with warm distilled water and then wiped carefully with clean cloth piece. Weighting the mass of above washing and clean graduated cylinder empty ( $m_e$ ) and the mass of it after filled with determined volume of PDF ( $m_f$ ). The density of PDF is obtained by dividing the difference between two masses over the volume used ( $V_{PDF}$ ). The calculation is repeated three times for accuracy and taking the mean value.

$$\rho_{PDF} = \frac{m_{PDF}}{V_{PDF}} = \frac{(m_e - m_f)_{PDF}}{V_{PDF}}$$

Where:  $m_e$ : is the weight of washed, clean and empty graduated cylinder,  $m_f$ : is the weight of graduated cylinder filled with PDF and  $V_{PDF}$ : is the volume of PDF used = 50 ml. The second method was conducted using Automatic Density Meter (Koehler K86210 Germany) conforms to (ASTM D1250, D4052 and D5002). The specific gravity of PDF is the ratio between density of PDF and the density of pure water at the same temperature.

$$SG_{PDF} = \frac{\rho_{PDF}}{\rho_w}$$

Where:  $SG_{PDF}$ : is the specific gravity of PDF and  $\rho_w$ : is the density of pure water.

### 3.2 Viscosity

viscosity is the ratio of the shear stress magnitude applied on the fluid to the velocity gradient that it produces. It

is described the internal friction present between two layers of a moving fluid which resists the flow of fluid. So viscosity is another type of characteristic bulk property of fluid. The viscosity can be obtained by the Ostwald viscometer. Viscosity ( $\mu$ ) of PDF can be calculated from the following equation:

$$\mu_{PDF} = A \cdot \rho_{PDF} \cdot t$$

Where:  $t$  = falling time of a PDF for a particular distance through the tube of Ostwald viscometer,  $\rho_{PDF}$  = density of PDF and  $A$  = calibration constant of Ostwald viscometer, which is numerically equal for every liquid flowing past a particular distance through the tube of Ostwald viscometer. The value of  $A$  can be easily determined by putting the chart value of corresponding to room temperature. Using the above equation the viscosity of sample can be determined. Another method can be achieved to determine the dynamic viscosity of sample which is used to detect the viscosity of PDF. This method was performed using Dynamic Viscosity by Master Series Rotational Viscosity apparatus (Koehler K447-ML Germany).

### 3.3 API gravity

API gravity (American Petroleum Institute gravity) is an inverted measure scale of the petroleum liquid relative density (specific gravity) to indicate the heaviness or lightness of crude oils or petroleum liquids or other liquid hydrocarbons compared to water density at 60°F or 15.5°C. Universally API gravity calibration is expressed in API degrees (or degrees API) on a hydrometer instrument because it has no units mathematically. The API measure scale was designed so that most values of petroleum liquid products would lie between 10 and 70 degrees. For any oil if API gravity is greater than 10 degree, it is lighter and floats on water; if less than 10 degree, it is heavier and go under water. In general, petroleum liquid with API greater than 30 degree is termed light; between 22 and 30 degrees, medium; below 22 degree, heavy; and below 10 degree, extra heavy. Therefore, the oil that has the same density as pure water at 15.5°C (i.e., with a specific gravity of 1.0) has an API gravity equal to 10 degree. For example, on average, API gravity of asphalt is 8 degree; API gravity of Brent Crude is 35.5 degree, API gravity of gasoline is 50 degree and API gravity of diesel is 32.5 degree. The equation that describes the mathematical relationship of API gravity of PDF is:

$$\text{API gravity}_{PDF} = \frac{141.5}{SG_{PDF}} - 131.5$$

### 3.4 Calorific value

(also known as heating value or energy value or the heat of combustion of a substance) is the quantity of energy released by a material (commonly food or fuel) as heat produced during combustion reaction when a specified amount of substance undergoes complete combustion with oxygen under standard conditions (i.e. temperature of 273.15K or 0°C or 32°F) and an absolute pressure of 100 kPa or 14.504 psi or 0.987 atm or 1 bar)). For produced diesel fuel (PDF) the completely combustion reaction is typically a hydrocarbons reacting with oxygen to produce carbon dioxide and water vapour. Calorific value may be expressed as [unit of energy (J or cal or Btu)/unit of substance (mol or kg or lb)] and it can measure conventionally using bomb calorimeter. The calculation of calorific value divided into two types, the first one is **Gross Calorific Value (GCV)** (also known as higher calorific value or higher heating value or gross energy) of a fuel which is meaning the quantity of heat released by a specified amount (initially at 25°C) immediately when combusted and the products have returned to a temperature of 25°C, which takes into account the latent heat of vaporization of water in the combustion products. This type can be calculated from the following equation:

$$\Delta H_{GCV} = \frac{t \cdot w - e}{m}$$

Where:  $t$  : Temperature difference between final temperature and initial temperature,  $w$  : Energy equivalent of the calorimeter = 2426 cal/°C,  $e$  : Correction in calories for heat of combustion of fuse wire =  $2.3 \times c$  when using nickel-chromium fuse wire,  $c$  : Fuse wire consumed in firing, cm,  $c = Lw_{bf} - Lw_{af}$ ,  $Lw_{bf}$  : Length of wire before firing = 10 cm,  $Lw_{af}$  : Length of wire after firing, cm,  $m$  : Weight of sample in g. The GCV can be calculated using Automatic Calorimeter (Koehler K88890 Germany) conforms to (ASTM D240, D4809, D5865, D1989, D5468 and E711). The second type is **Net Calorific Value (NCV)** (also known as lower heating value) of a fuel which is meaning the quantity of heat released when specified amount is combustion (initially at 25°C) and returning the temperature of the combustion products to 150°C, which assumes the latent heat of vaporization of water in the reaction products is not recovered. The mathematical formula that describes this type of calorific value is represented by the following equation:

$$\Delta H_{NCV} = \Delta H_{GCV} - 50.683 H$$

Where:  $H$  : Weight percentage of hydrogen in fuel.

### 3.5 Acid number

(also known as neutralization number or acid value or acidity) is the mass of potassium hydroxide (KOH) in



milligrams that is required to neutralize one gram of chemical substance i.e. it shows the amount of alkali required to neutralize unit mass of the oil. So, the acidity is one of the important chemical properties. The acid number is a measure of the amount of carboxylic acid groups in a chemical compound, such as a fatty acid, or in a mixture of compounds. In a typical procedure, a known amount of sample dissolved in organic solvent (often isopropanol), is titrated with a solution of potassium hydroxide with known concentration and with phenolphthalein as a color indicator. The acid number is used to quantify the amount of acid present, for example in a sample of biodiesel. It is the quantity of base, expressed in milligrams of potassium hydroxide that is required to neutralize the acidic constituents in 1 g of sample.

$$AN = (V_{eq} - b_{eq})N \frac{M_{KOH}}{w_{diesel}}$$

Where:  $V_{eq}$ : is the volume of titrant (ml) consumed by the diesel sample and 1 ml of spiking solution at the equivalent point,  $b_{eq}$  is the volume of titrant (ml) consumed by 1 ml of spiking solution at the equivalent point,  $M_{KOH}$  is the molecular weight of KOH which is equal to 56.1 and  $w_{diesel}$  is the mass of the sample in g.  $N$  is the molar concentration of titrant (N) and it can be calculated by the following equation:

$$N = \frac{1000}{V_{eq}} \cdot \frac{w_{KHP}}{M_{KHP}}$$

Where:  $w_{KHP}$ : is the mass (g) of Potassium Hydrogen Phthalate (KHP) in 50 ml of KHP standard solution,  $V_{eq}$  is the volume of titrant (ml) consumed by 50 ml KHP standard solution at the equivalent point, and  $M_{KHP}$  is the molecular weight of KHP which is equal to 204.23. There are standard methods for determining the acid number, such as ASTM D 974 and DIN 51558 (for mineral oils, biodiesel), or specifically for Biodiesel using the (European Standard EN 14104 and ASTM D664) are both widely utilized worldwide.

### 3.6 Pour point

it is the lowest temperature at which the oil remains pourable and shows flow a characteristic, meaning it still behaves as a fluid. Or it is the temperature of oil at which it becomes semi solid and ceases to flow i.e. loses its flow characteristics. Pour point is an important determining factor to select the type of oil for many purposes from among a group with otherwise identical characteristics due to its ability of appraising oil flow properties at low temperature. For instance, the pour point of aviation fuel and liquid additives is an important consideration for aircrafts in upper atmosphere and drilling operations in the Arctic and Antarctic respectively. There are two methods for measuring the pour point of oils which are Manual Method under (ASTM D5853 and ASTM D97) and Automatic Method according to (ASTM D5950). The automatic method was used to test pour point of PDF using Automatic Cloud and Pour Point Analyzer with Touch Screen (Two-stage), 220V 50/60Hz (Koehler KLA-3-TS/2 (220) Germany) conforms to (ASTM D97, D5853 and D5950).

### 3.7 Cloud point

it is the highest temperature point on the solid-liquid phase envelop at which at least a detectable amount of one component of dissolved solids in a visible solution of certain petroleum oil as a homogenous liquid mixture (e.g., paraffin wax or other solid substance) is no longer completely soluble in the solution (i.e., the mixture of liquids starts to become immiscible) and will begin to form, insulate, solidify, congeal and separate from an identical solid-liquid solution and precipitating as a featured, discrete and crystalline second phase when the clear petroleum oil solution subjected for cooling (under specific and standardized conditions of in the time frame of the measurement resulting to distinctly cloudiness, turbidness and haziness appearance is observed in the certain petroleum oil solution at this point or temperature due to bonding of paraffin wax. Cloud point is relevant to several applications with different consequences and indicates the tendency of the petroleum oil to plug filters or small orifices at cold operating temperatures because the presence of solidified wax in diesel or solidified biowax in biodiesels thickens the oil and clogs fuel filters and injectors in engines. The wax also accumulates on cold surfaces (e.g. pipeline or heat exchanger fouling) and forms an emulsion with water. Therefore certain petroleum oils like diesel must be maintained at temperatures above the cloud point. Two methods are used to determine cloud point, the first one is Manual Method approved with (ASTM D2500) while the second procedure is Automatic Method consistent with (ASTM D5773). The automatic method (which also known as Constant Cooling Rate Method) was used for testing cloud point of PDF by Automatic Cloud and Pour Point Analyzer with Touch Screen (Two-stage), 220V 50/60Hz (Koehler KLA-3-TS/2 (220) Germany) conforms to (ASTM D2500, D5771, D5772 and D5773).

### 3.8 Flashpoint

is the lowest temperature at which a combustible particular volatile organic solid compound can vaporize in a specified apparatus to give off sufficient vapour of a liquid near its surface and generate an ignitable mixture to initiate a combustion reaction in air, flash and burn momentarily when exposed to a brief flame if ignited by

flame or spark under certain conditions. The flash point is often used as a descriptive characteristic of liquid fuel, and it is also used to help characterize the fire hazards of liquids. “Flash point” refers to both flammable liquids and combustible liquids. There are various standards for defining each term. Liquids with a flash point less than 60.5 or 37.8 °C (140.9 or 100.0 °F) — depending upon the standard being applied — are considered flammable, while liquids with a flash point above those temperatures are considered combustible. A decrease in flash point indicates contamination by dilution of petroleum oils with unburned fuel. Increasing of flash point indicates evaporation of the light components from the petroleum oil. There are two basic types of flash point measurement: open cup and closed cup. The type was applied to test PDF is closed cup using Pensky-Martens Closed Cup Flash Tester with Electric Heating, 115V, 50/60Hz (Koehler K16200 Germany) conforms to (ASTM D93).

### 3.9 Fire point

is the lowest temperature at which frying oil is produced enough flammable vapour of a volatile combustible substance to form an air-vapour mixture above its surface, by that given frying fuel will sustain combustion and burns continuously in air for at least 5 seconds after ignited (as when heating is continued after the flash point has been determined) by a small open flame. At the flash point, a lower temperature, a substance will ignite briefly, but vapour might not be produced at a rate to sustain the fire. Most tables of material properties will only list material flash points, but in general the fire points can be assumed to be about 10 °C higher than the flash points. However, this is no substitute for testing if the fire point is safety critical. It is done by open cup apparatus. The fire point test of petroleum oils can be conducted by Cleveland Open-Cup Flash Tester with Electrically Heated, 220-240V 50/60Hz (Koehler K13990 Germany) Conforms to (ASTM D92).

### 3.10 Auto-ignition point

is the lowest temperature where a flammable substance or mixture will spontaneously ignite or auto-ignite and combust in normal atmospheric conditions without any influences of external ignition source (i.e., presence of a flame or spark). This temperature is required to supply the activation energy that is necessary for combustion. Auto-ignition point is usually applied to a combustible fuel mixture because the temperature at which a chemical will ignite decreases as the pressure or oxygen concentration increases. The auto-ignition temperature is used to specify safe operating, storage and handling procedures for process plant operating at elevated temperature. The Auto-ignition temperature test of PDF is typically measured using an auto-ignition apparatus, 220-240V, 50/60Hz, Single Phase, 7.7A (Koehler K47000 Germany) Conforms to (ASTM E659). The test is repeated 10 times to confirm the lowest auto ignition temperature has been found.

### 3.11 Aniline Point

is the minimum equilibrium solution temperature at which equal volume of pure, fresh aniline and petroleum oil sample under test (or any hydrocarbon fluid) will completely miscible (clear) (i.e., dissolve in one another) under standardized conditions. Aniline point serves as an indication of the type of hydrocarbons present in the solvent, used in some specifications to determine the aromatic content of oils and to calculate approximate heat of combustion. The aniline point of oil should be high to minimize damage to elastomer parts, but this may indicate oils of less solvency and lowered ability to disperse asphaltic materials in oil muds. The aniline point test of PDF is performed using an Automatic Aniline Point Apparatus, 220-240V 50/60Hz (Koehler K10290 Germany) conforms to (ASTM D611 Method E).

### 3.12 Smoke point

the smoke point is not temperature; it is the highest distance in millimeters reach by the flame of petroleum product when it is burned (not only combustion) without smoke forming under standard conditions tested. Smoke point is used as measure of the burning cleanliness of petroleum product. So, it is an indicator of the burning qualities of petroleum products. The smoke point of PDF can be determined using Smoke Point Lamp apparatus (Koehler K27000 Germany) conforms to (ASTM D1322).

### 3.13 Octane number

(also called octane rating) is a numerical representation value used to indicate the ignition quality of petroleum motor fuel for knock resistance compared with a standard reference fuel comprises of a mixture of 2,2,4-trimethylpentane (isooctane) (which has an octane number of 100 (i.e., minimal knock)) and heptane (of zero octane number (i.e., bad knock)) which would have the same anti-knocking capacity as the fuel under test. This does not mean that the petroleum fuel contains just isooctane and heptane in these proportions, but that it has the same detonation resistance properties, because some petroleum fuels are more knock-resistant than isooctane, the definition has been extended to allow for octane numbers higher than 100. There are many types of octane number like Research Octane Number (RON), Motor Octane Number (MON), Anti-Knock Index (AKI) (also

called pump octane number), Observed Road Octane Number (RdON) and Aviation gasoline octane ratings. The most common type of octane rating worldwide is the Research Octane Number (RON). The RON can be applied for PDF using Automatic Fuel Blending System for Octane and Cetane Reference apparatus; 220-240V 50/60Hz with 10 channels (Koehler K87994 Germany) conforms to (ASTM D2699, ASTM D2700 and ASTM D613).

### 3.14 Cetane number (CN)

is a measurement of the combustion quality indicating the ignition properties of the readiness of diesel fuel to auto-ignite when injected into a diesel engine during compression ignition relative to cetane (which is alkane hydrocarbon named Hexadecane of chemical formula  $C_{16}H_{34}$ ) as a standard. It is an important factor in determining the quality of diesel fuel. Cetane number relates to the delay between when fuel is injected into the cylinder and when ignition occurs. Cetane number of PDF was conducted using the same apparatus used for determined of octane number in section 3.13 previously and also implemented using Cetane Ignition Delay – CID 510 Fully Automated Derived Cetane Number (DCN) Analyser. Cetane number can be estimated mathematically according to the following empirical equation:

$$CN = 0.72 \times DI + 10$$

Where: **DI** the Diesel Index.

### 3.15 Cetane Index (CI)

is value used as a substitute for the cetane number of diesel fuel. The cetane index is calculated based on the fuel's density and distillation range according to (ASTM D86). There are two methods used to estimate cetane index, the older one is "two-variable equation" method according to ASTM D976. By this method cetane index can be calculated from the following equations:

$$CI_{cal.} = -420.34 + 0.016 G^2 + 0.192 G(\log M) + 65.01(\log M)^2 - 0.0001809 M^2$$

or

$$CI_{cal.} = 454.74 - 1641.416 D + 774.74 D^2 - 0.554 B + 97.803 (\log B)^2$$

Where: **G**: API gravity, Degrees @ 60 °F, determined by Test Method D287 or D1298, **M**: mid-boiling temperature @ 50% volume, °F, determined by Test Method D86 and corrected to standard barometric pressure. **D**: density at 15 °C, g/ml, determined by Test Method D1298 and **B**: mid-boiling temperature, °C, determined by Test method D86 and Corrected to standard barometric pressure. (ASTM D4737) is the newest method for calculating cetane index and is sometimes referred to as "the four-variable equation" method. Cetane Index according to this method can be evaluated from the following equation:

$$CI_{cal.} = 45.2 + 0.0892 T_{10N} + (0.131 + 0.901 B) T_{50N} + (0.0523 - 0.42 B) T_{90N} + 0.00049 [(T_{10N})^2 - (T_{90N})^2] + 107 B + 60 (B)^2$$

Where:

$$B = \exp(-3.5 D_N)$$

$$D_N = SG - 0.85$$

$$T_{10N} = T_{10} - 215$$

$$T_{50N} = T_{50} - 260$$

$$T_{90N} = T_{90} - 310$$

$T_{10N}$ ,  $T_{50N}$  and  $T_{90N}$  Distillation expressed in D86 in °C

Cetane index in some crude oil assays is often referred to as Cetane calculi, while the cetane number is referred to as Cetane measure.

### 3.16 Diesel Index (DI)

is an empirical expression describe diesel fuel rating based on its ignition quality and can be calculated by two mathematical formulas, the first one involving its cetane number as aforementioned explain in section 3.15 while the second formula depending on its gravity and its aniline point as in the following equations:

From Cetane Number:  $DI = \frac{1}{0.72} (CN - 10)$  and, From gravity and aniline point:  $DI = \frac{1}{100} (G \times A)$

Where **G** is the API gravity and **A** is the aniline point, °F

### 3.17 Water Content

Diesel fuels also contain small amounts of water. Hydrocarbon type and bulk temperature control the amount of dissolved water that a fuel holds. As the temperature decreases, the amount of water dissolved in the fuel will also decrease, which may lead to a water layer forming on the bottom of the fuel storage tank. To prevent subsequent bacterial contamination, as well as the pumping of water into the fuel distribution system, draining the lowest level of all tanks regularly should minimize this layer. Many diesel fuel treatments are offered to control this water content. These additives typically contain glycol or various types of alcohol (most often Isopropanol) which absorb their own weight in water (they are hydroscopic), and emulsify the water back into

the fuel. These additives are very popular, especially in the trucking and marine industry. But they carry with them a very serious risk. The water content in PDF can be tested by Compact Volumetric Karl Fischer Titrator apparatus (Koehler K90375 Germany) conforms to (ASTM D789, D803, D890, D1123, D1152, D1193, D1348, D1364, D1568, D1631, D2072, D2575, D3401, D4017, D4377, D4672, D5460, D5530, E180, and E203). This piece of equipment measures the parts per million (ppm) of water content in PDF. Although there is no official maximum water content in petrol, with the introduction of ethanol blended petrol, 1000 ppm is considered the maximum level of water is suspension within the fuel. In diesel, under the European standard for road diesel, a maximum water content of 200 parts per million mandated.

### 3.18 Ash Content

The "ash content" is a measure of the total amount of minerals present within petroleum fuel, when it is completely burned, the remaining solid is called ash and it shows the fuel purity. The highest ash content of most diesel fuels limits to 100 ppm. (Inorganic particles and oil-soluble, metallo-organic compounds both contribute to the ash content; but, only inorganic particles will cause wear). The ash content of PDF can be achieved using Micro Conradson Carbon Residue and Ash Tester, 230V 50-60Hz (Koehler K41090 Germany) conforms to (ASTM D4530, D482 and D189).

### 3.19 Carbon Residue

is the amount of carbon that is made by a chemical process, such as heating up oil. It is mainly considered to be a by-product of fuel. When fuel is burned and used by a motor vehicle engine, it produces exhaust that contains carbon monoxide. Carbon residue is what the leftover particles of a fuel product are transformed into. There are tests that can determine the amount of residue that remains after certain fuels are heated or burned. High amounts of residue can be damaging to the environment. For example, high levels of carbon monoxide can lead to an increase in the concentration of greenhouse gases in the atmosphere. These gases are thought to contribute to global warming — an overall increase in the average earth temperature over decades. The test method that is used to calculate the amount of carbon residue is known as Ramsbottom Carbon Residue (RCR). This test is used to determine how much residue a fuel is likely to leave. It also helps calculate the fuel's tendency to combust or burn. The carbon Residue of PDF can be calculated also using using Micro Conradson Carbon Residue and Ash Tester, 230V 50-60Hz (Koehler K41090 Germany) conforms to (ASTM D4530, D482 and D189) and using Ramsbottom Carbon Residue Apparatus (Koehler K27100 Germany) conforms to (ASTM D524). Both of these apparatus are equivalent in that they return the same numerical value.

### 3.20 Average Molecular Weight

Diesel fuel, like other petroleum products, is not one specific chemical compound but a complex mixture of organic compounds separated into ranges by their boiling point. For this reason, diesel doesn't have a specific molecular weight. However, you can talk about an "average molecular weight" of the mixture. EPA lists an estimated average molecular weight for diesel of 233 g/mole. The actual average molecular weight of diesel will vary from sample to sample depending on what grade of diesel it is, feedstock of the diesel, and the refining process of the diesel.

**3.21 Lead and Sulfur Content:** The Lead and Sulfur content in PDF were measured spectrophotometrically using Automatic Colorimeter 115-240V, 50/60 Hz (Koehler K13550 Germany) Conforms to (ASTM D156, D1209, D1544, D1925, D6045 and D6166).

## 4. RESULTS AND DISCUSSIONS

### 4.1 Effect of (Sulfuric Acid/ Raw Material) Ratio (Acidification Process)

During the process of sulfuric acid treatment, the UAO feedstock is mixed with concentrated sulfuric acid to remove most of the impurities, polymers, asphalts, degraded additives and products of oil degradation process. The effect of (sulfuric acid/raw material) weight ratio on the % Produced Diesel Fuel (%PDF) was explain in Figure 1. From this figure it is clear that the %PDF is increased with the (sulfuric acid/raw material) weight ratio increases too until the ratio reach to 7 in indu-diesel and 8 in bio-diesel. This may be due to that when the ratio of (sulfuric acid/raw material) increased the amount of impurities in the UAO is decreased because the sulfuric acid react with the impurities forming sulfates and insoluble sulphur containing rejected compounds (sludge asphalt) which will settle in the bottom of reactor. So indu-diesel (i.e., PDF from UAO) is increased because the hydrocarbons founded in the UAO will be alone without compete with other impurities. On the other hand for the bio-diesel (i.e., PDF from UFO) the amount of sulfuric acid has a significant role on conversion of Free Fatty Acid (FFA) to methyl ester. Increase the amount of sulfuric acid lead to increase the percentage of FFA conversion. When the ratio reach to 7 and 8 wt% the %PDF was kept constant without large variation. This means that these ratios were efficient and any increase in sulfuric acid amount not affected on the %PDF.



Therefore the optimum weight ratios of (sulfuric acid/raw material) for both indu-diesel and bio-diesel were 7 and 8 wt% respectively.

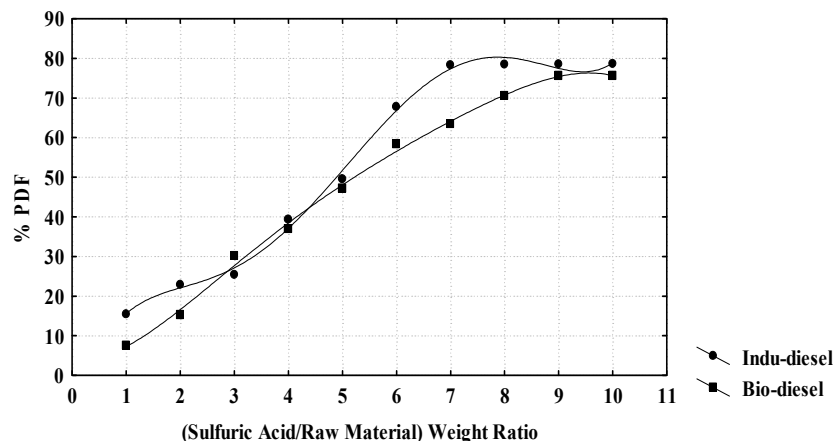


Figure 1 Effect of Effect of (Sulfuric Acid/ Raw Material) Ratio on the % PDF

#### 4.2 Effect of (Calcium Hydroxide/Raw Material) Ratio (Neutralization Process)

When the acidification process was accomplished, the oil is heated at 100 °C to let the water content evaporate and remove other volatile matter such as petrol or organic solvents which might have been mixed into the UAO. The mixture was then allowed to cool and kept undisturbed for 2 hours for deasphalting and settling of acid sludge from acid treated oil and then subject to a neutralization when calcium hydroxide is added. **Figure 2** illustrates the effect of (calcium hydroxide/raw material) weight ratio on the % Produced Diesel Fuel (%PDF). This figure show that the increasing in the amount of calcium hydroxide lead to increasing in %PDF. The calcium hydroxide is considered as a strong base and when added to the acidification mixture it can neutralize any excess of sulfuric acid by reacting with it to form calcium sulfate which settle with impurities and water. When the (calcium hydroxide/raw material) weight ratio reach to 8 wt% the %PDF is approximately remain constant. This may be meaning that the amount of calcium hydroxide is sufficient to neutralize any excess of sulfuric acid in the solution and this result is agreed with the results obtained in previous *section 4.1*. The optimum (calcium hydroxide/raw material) ratio for both indu-diesel and bio-diesel was 8 wt%.

#### 4.3 Effect of (Y-Zeolite/UAO) Ratio and (Ethanol/UFO)

For the indu-diesel, the next step after neutralization process is clay treatment using prepared Y-zeolite; an adsorption process would be taken place. Figure 3 elucidates the effect of (Y-zeolite/UAO) weight ratio on the % Produced Diesel Fuel of indu-diesel (%PDF). Figure 3 shows that when increasing in the amount of Y-zeolite, the %PDF was increasing too. This result may be due to the fact that any increasing in the amount of Y-zeolite (which behave as adsorbent material) means increasing in the surface area of the material so, it can adsorb excess quantity of impurities that don't settled in the acidification step like heavy metals and undesired hydrocarbons may be found in the UAO before or after treatment. Thus, the %PDF wasn't increased only but the Y-zeolite treatment process used to improve color and oxidation stability of the treated UAO especially for when the (Y-zeolite /UAO) ratio override 8 wt% because there is an excess amount of clay can behave as bleaching agent after the adsorption process is completed. Moreover, for the bio-diesel, the next step after neutralization process is treatment with ethanol using ethanolic alcohol; a transesterification process would carry out. It's obvious from Figure 3 that the continuous increasing in the % Produced Diesel Fuel of bio-diesel (%PDF) is related with the increasing of (ethanol/UFO) weight ratio because the excess amount of ethanol lead to increasing calcium ethoxide ions forming which will necessary to convert FFA to bio-diesel by replacing the glycerol part from triglyceride with three ethoxide groups for each molecule of triglyceride. In this reaction the ethanolic alcohol play another role, besides it is the main source of ethoxide it behaves as a catalyst for transesterification process which catalysis the cleavage of triglyceride to glycerine and FFA from one side, and from another side provides the required ethoxide ions to perform transesterification reaction. Therefore, when the (ethanol/UFA) increased above 8wt% the %PDF (bio-diesel) was increased because there is enough amount of alcohol to produce sufficient ethoxide ions and the excess behave as catalyst for triglyceride conversion reaction. This result is identical with the results obtained in previous *section 4.2*. The optimum (ethanol/UFA) ratio for %PDF (bio-diesel) was 10 wt%.

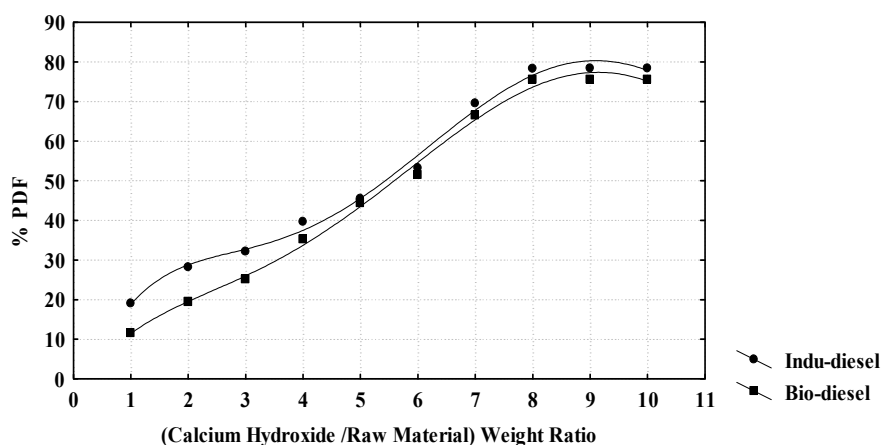


Figure 2 Effect of (Calcium Hydroxide/Raw Material) Ratio on the % PDF

#### 4.4 Effect of Agitation Speed

Effect of agitation speed was studied in the range from 100 to 600 rpm keeping all other conditions constants and Figure 4 shows the effect of agitation speed on the %PDF (both indu-diesel and bio-diesel). It's clear from Figure 4 the %PDF was increased with the agitation speed increased too until reach to 400 rpm where %PDF was stopped increase and remain constant. These results may be due to the facts that the agitation speed plays a very significant role in the yield production of PDF (both indu-diesel and bio-diesel) using acidification, neutralization and transesterification processes, because raw materials (UAO (of industrial source) and UFO (of biomass source)) are immiscible (each one alone) with acid, alkaline and alcohol solutions forms a separate phases and lower agitation speed shows lower product formation. So, there are multi-phase in the reaction system. Under such condition, agitation facilitates and enhances the mixing of the different component of reactants mixture, influencing the rate of the chemical reaction and thus increases the %PDF as a result to overcome the mass transfer limitations and form a homogeneous phase of hydrocarbons or ethyl esters and ethanol. Thus, the raw materials dissolve in the mixture. On the other hand (for bio-diesel) higher agitation speed favours formation of soap. This is due to the reverse behaviour of transesterification reaction. This explains why the %PDF increased with increase the agitation speed. The optimum agitation speed for better both indu-diesel and bio-diesel was taken as 500 rpm. However, at the optimum speed (i.e., 450 rpm) the single phase is established in the reaction mixture, therefore above this speed the agitation becomes inconsequential and the %PDF (both indu-diesel and bio-diesel) no longer exceeds.

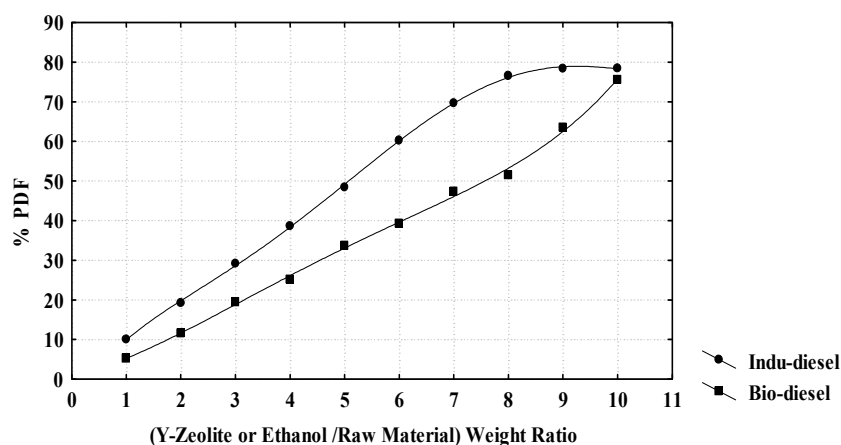


Figure 3 (Y-Zeolite/UAO) Weight Ratio and (Ethanol/UFO) Weight Ratio on the % PDF

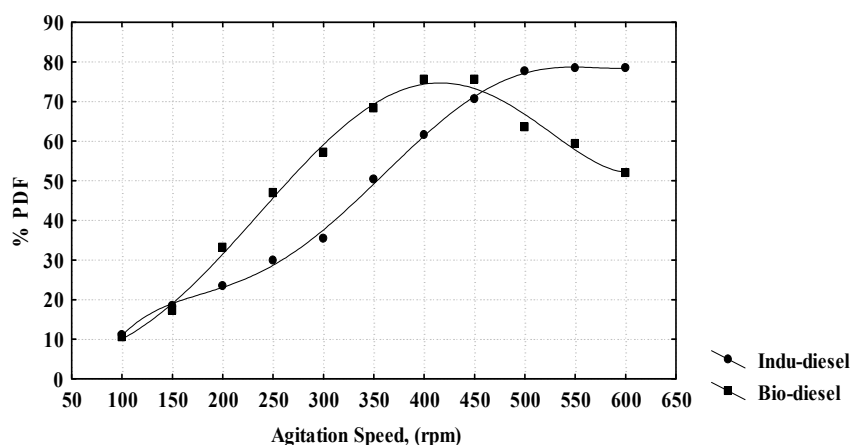


Figure 4 Effect of Agitation Speed on the % PDF

#### 4.5 Effect of Reaction Temperature

Figure 5 shows the results acquired by varying the reaction temperature from 55°C to 100°C. From those results it's appeared that the %PDF was increased with increasing reaction temperature until reach 80 °C for bio-diesel and the decreased but continues increasing for indu-diesel. Reaction temperature is an important factor that will affect the % PDF (both indu-diesel and bio-diesel) due to the type of heat reaction. The obtained results indicate that the reaction was typically endothermic, (i.e., increasing rate of reaction related to increase in reaction temperature). When the reaction temperature was increased, the viscosity of the raw material oils (UAO and UFO) was decreased, so the solubility of oils was increased in the reaction mixture. This lead to increase the rate of oils conversion, thus increase % PDF. The yield of PDF was continued for indu-diesel from the raw material UAO in the range of temperature but for bio-diesel the % PDF is stopped at 80 °C and decline after that. This may attributed to the excess of evaporation of ethanol when rise the temperature beyond the boiling point of ethanol which is 78.73°C. When the ethanol is evaporated the ethoxide produced is decreased and its amount became insufficient to convert triglyceride in FFA to bio-diesel, besides that the role of behave ethanol as a catalyst was lowered or absence too. Therefore, the % PDF (bio-diesel) was decreased while this doesn't happen in the case of % PDF (indu-diesel) from UAO since the boiling point of UAO is higher than 100°C. The maximum % PDF was obtained at optimum reaction temperature which was 80°C for bio-diesel and 100°C for indu-diesel.

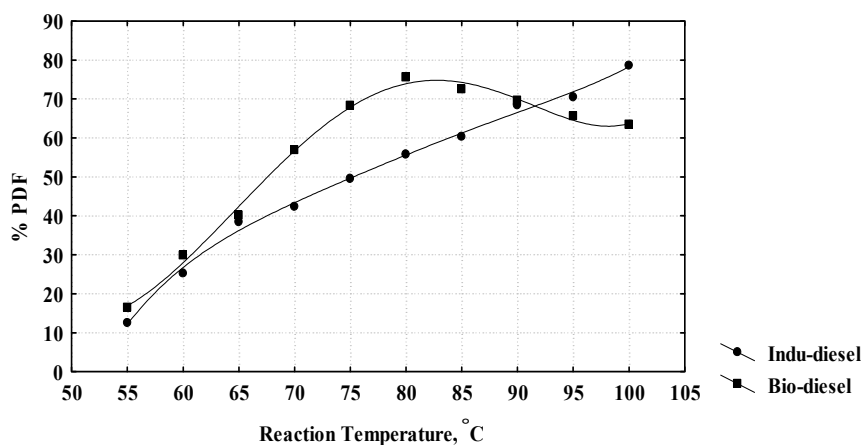


Figure 5 Effect of Reaction Temperature on the % PDF

#### 4.6 Effect of Processing Time

To study the effect of processing time on the % PDF, the processing time was changing in the range between 15 and 150 minutes. Figure 6 shows that the % PDF was increased with increasing processing time for indu-diesel but reach to 120 minutes and decline after that for bio-diesel. Processing time is a factor influences the yield of PDF, when the reaction starts the rate of reaction was slow due to agitation speed and mixing conditions and also dispersion of raw material oils and other reactants. Then the reaction rate is increased rapidly and the maximum % PDF was been at 120 minutes after that the conversion decreased lead to lower the rate of PDF for bio-diesel but the % PDF was continued for indu-diesel. This may be due to the fact that further increasing of processing time

results in reduction of bio-diesel due to the reverse reaction of transesterification reaction and thus, leads to loss of esters as well as soap formation. The optimum processing time was 120 minutes for % PDF (bio-diesel) while it was 150 minutes for % PDF (indu-diesel).

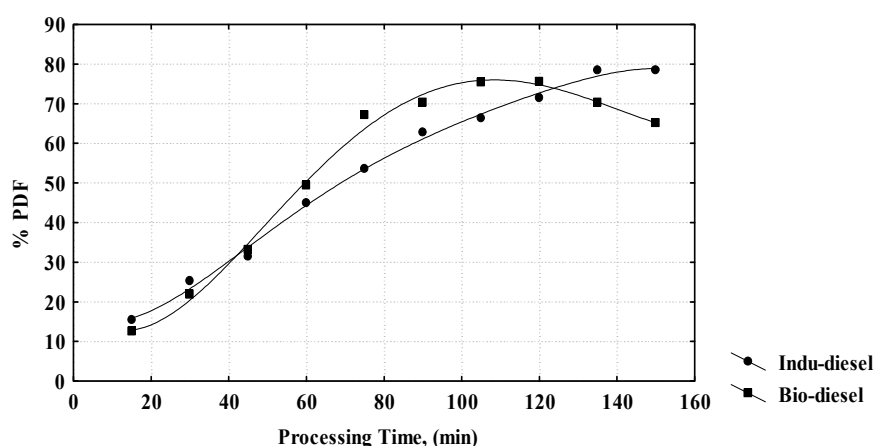


Figure 6 Effect of Processing Time on the % PDF

#### 4.7 Properties of PDF

The tests conducted for the PDF were tabulated in **Table 1**

**Table 1** Properties of indu-diesel, Bio-diesel and Ordinary diesel

Property	Indu-diesel	Bio-diesel	Ordinary diesel
Density ( $\text{kg.m}^{-3}$ )	859	852	832
Viscosity ( $\text{kg.m}^{-1}.\text{s}^{-1}$ )	$3.22 \times 10^{-3}$	$3.76 \times 10^{-3}$	$2.85 \times 10^{-3}$
API gravity (degree)	34.58	33.23	38.57
Gross Calorific Value ( $\text{MJ.kg}^{-1}$ )	40.186	42.582	45.490
Net Calorific Value ( $\text{MJ.kg}^{-1}$ )	37.351	40.055	42.117
Acid number (mg KOH/g diesel)	0.416	0.827	0.5
Pour point ( $^{\circ}\text{C}$ )	-11	-8	-17
Cloud point ( $^{\circ}\text{C}$ )	-13	-12.5	-14
Flashpoint ( $^{\circ}\text{C}$ )	73	65	60
Fire point ( $^{\circ}\text{C}$ )	70	77	68
Auto-ignition point ( $^{\circ}\text{C}$ )	215	225	210
Aniline Point ( $^{\circ}\text{C}$ )	50	49	51.6
Smoke point (mm)	7	5	8
Octane number (RON)	16	13	20
Cetane number	39.18	39.92	44.7
Cetane index	37	35	40
Diesel Index	40.54	41.56	48.2
Water Content ( $\text{mg.kg}^{-1}$ ) or (ppm)	0.14	0.21	0.05
Ash Content % (m/m)	0.01	0.008	0.003
Carbon Residue % (m/m)	0.223	0.192	0.125
Average Molecular Weight	156	104	230
Lead Content ( $\text{mg.kg}^{-1}$ ) or (ppm)	0.109	0.197	0.205
Sulfur Content ( $\text{mg.kg}^{-1}$ ) or (ppm)	11.95	20.44	13.51

## 5. CONCLUSIONS

The following conclusions can be deduced:

- There is ability to convert UAO and UFO to indu-diesel and bio-diesel respectively using acidification, neutralization and clay treatment for UAO or alcohol esterification for UFO method, thus it can removed two types of wasted used oils by eco-friendly method.
- It can be used other wastes to perform the conversion process for both UAO and UFO such as eggshell which can be used as a source of calcium hydroxide after thermal treated and reacted with pure water, rice husk which can be used to prepare faujasite type Y-zeolite used as clay in clay treatment and orange peel which can be used as a source of ethanol used to achieve esterification process.
- The % PDF is increased with increasing (acid/raw material) weight ratio, (base/raw material) weight ratio,



(clay or ethanol/raw material) weight ratio, agitation speed, reaction temperature and processing time.

d. The maximum percentage of Produced Diesel Fuel (%PDF) by this way reaches to approximately 78% for indu-diesel from UAO at (acid/raw material) ratio, (base/raw material) ratio, (clay/raw material) ratio, agitation speed, reaction temperature and processing time of 7%, 8%, 8%, 500 rpm, 100°C and 150 minutes respectively and 75% for bio-diesel from UFO at (acid/raw material) ratio, (base/raw material) ratio, (clay/raw material) ratio, agitation speed, reaction temperature and processing time of 8%, 8%, 10%, 450, 80°C and 120 minutes respectively.

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