Physical Characteristics of Palm Fatty Acid Distillate

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ABSTRACT

Palm fatty acid distillate (PFAD) is a widely used product due to its low cost being a by-product of (edible) palm oil manufacturing process. In this paper, the physical properties of palm fatty acid distilled (PFAD) produced in Malaysia will be discussed. The major physical properties obtained during this study are; the specific gravity at 28°C, moisture content, viscosity at 40°C, colour at 28°C, Pour point, Flash point and Oxidative stability values were 0.87±0.1g/ml, 0.63±0.1%, 96.35±0.1 cSt, 2R-20Y, 35±1°C, 135±1°C and 178±1°C, respectively. A rather significant outcome is the great benefit of using PFAD for bio lubricant, which offers many positive characteristics, such as; it increases the viscosity, viscosity index, flash point and oxidative stability. Therefore PFAD has a good potential for biodiesel and bio lubricant industries.

KEY WORDS: Palm Oil, Fatty Acid Distillate, Physical Properties.

1. INTRODUCTION

PFAD is considered as a core raw material for many oleo chemical industries, including; plastics, medium-grade cleaners, animal foods, and other intermediate products. (Dumont and Narine, 2007). Several studies also investigated the possibility of producing biodiesel using Free Fatty Acid (FFA) that is extracted from PFAD (Chong, 2007). PFAD has a promising potential to be used in low cost palm biofuel second generation feedstock. PFAD can be a source of fatty acid for non-food applications in several industries PFAD is retrieved from the process of deodorization with a high FFA content. The refinery process of CPO involves three steps; degumming, bleaching and deodorization. The Free fatty acid (FFA) content in the palm oil is about 3-5% originally, this value will be reduced to less than 0.01% via neutralization and deodorization processes. During deodorization process, the volatile odorous compound is extracted and re-collected as by-product with low value, PFAD (Majd, 2018). During neutralization process, alkali solution is added to be reacted with FFA, this alkali then removed in a soap form by centrifugal force. Deodorization process includes steam distillation under vacuum where steam is sparked into de-aerated oil. The volatile components are separated away and separately condensed as the oil passes through different sections. At this stage, FFA is recovered as PFAD mixed with other volatile components. PFAD is a light brown semi solid at room temperature, it melts into brown liquid when higher temperature is applied (Figure.1). The main objective of this study is to investigate physical properties of PFAD collected from Sime Darby Plantation oil refinery for physical.

Figure.1. Palm fatty acid distillate at room temperature

2. MATERIAL AND METHOD

Sample: Palm fatty acid distilled (PFAD) was collected from Sime Darby Plantation oil refinery located in Selangor, Malaysia.

Physical Analysis: Methods used to perform all physical properties analyses were AOCS official methods and MPOB testing method.

Viscosity and Viscosity Index: Moderate viscosity index improves the characteristics of a good lubricant. Viscosity index is a scale of numbers that indicate the changes in kinematic viscosity with changes in temperature. It is indicative of the quality of lubricating oil that is associated to any changes in kinematic viscosity related to temperature change. Viscosity index is used to describe the engine lubricating oil in the automotive industry. Rheometer used for kinematic viscosity measurement is physica MCR 301 model made by Anton Paar Instruments in Germany. The viscosity and viscosity index were made according to ASTM methods; ASTM D 2270-04 (ASTM 2005). A total of 1 ml of sample was added on a hot plate heater set at 40 °C and 100 °C. Viscosity index (VI) of
PFAD was measured on the basis of the L and H values, and the kinematic viscosity (U) at 40 °C, using equation (1).

\[
VI = \frac{(L-U)}{(L-H)} \times 100 \ \text{Equation} \ ... \ (1)
\]

Triplicate measurements were made and results were reported as a mean ± Standard deviation.

**Color:** The palm fatty acid distilled color was determined by means of lovibond Tintometer F/10508, in accordance with MPOB test method (Ainie, 2005). The PFAD sample were put in an oven and melted at 60 °C. Afterwards, the liquefied samples were placed in an inch cell, level was up to three-quarters full. The color was then determined at 28 °C to obtain the best match with the standard color slides of red and yellow indices.

**Density and Specific gravity:** The density of PFAD was measured using a delicate balance. The weight of one milliliter of PFAD measured by a balance was recorded at room temperature. The specific gravity was determined according to the Lund relationship (Gunstone, 2011) equation (2).

\[
\text{Specific gravity (28 °C)} = \text{oil density} + 0.00030 \ \text{SV} + 0.00014 \ \text{IV} \ ... \ (2)
\]

Where SV is an indicate to the saponification value of the oil and IV is the iodine value of oil.

**Moisture Content:** The moisture content of the PFAD was determined using an oven method. An estimated 5 g of PFAD was poured into weighted aluminum pan and dried in the oven adjusted at 100°C. Afterwards removed from oven, then cooled and stored in desiccator until weighed. The water content was calculated with the following equation (3).

\[
\text{Water content (\%)} = \left( \frac{M_A - M_R}{M_A} \right) \times 100 \ \text{(3)}
\]

Where; \( M_A \) = wet weight (g) = (wet weight + pan weight) – (pan weight) \( M_R \) = dry weight (g) = (dry weight + pan weight) – (pan weight).

**Pour Point:** The pour point is the lowest temperature at which a liquid remains in a pourable state (meaning it maintains its fluid behavior). It is a routine method used to determine the low temperature fluid flow properties. Pour point and PPDs values were measured according to ASTM D97-05a method (ASTM 2005) with some modification. The pour point was determined by using a U-tube and thermometer ranging from -80°C to 0°C. 10 ml of the sample was placed in a U-tube with the thermometer attached. Then the sample was introduced in the freezer with a temperature of -80°C. The sample was made frozen completely by letting it freeze for 24 hours. The U-tube was held in a horizontal position (Yunus, 2003). After 24 hours the U-tube was taken out from the freezer and the first flowing of the sample in the U-tube was recorded. The measured temperature was archived as pour point temperature. The pour point tests were in triplicate and results were reported as a mean ± SD of triplicate measurements. Pour point measurements were carried out with a resolution of 1°C instead of the indicated 3°C to achieve more accurate results.

**Flash Point:** The flash point for any volatile liquid is defined to be the lowest temperature at which this liquid vaporizes to form an ignitable mixture in air. Flash point was determined according to the ASTM D 56-79 method using a Tag Closed Tester (ASTM, 2005). Thermometer from 0°C to 360°C temperature was used in this test. Approximately 10 ml of the test sample was poured into the testing cup. For safety reasons, the flash point test was carried out in a fume chamber as a precaution against the very high temperature. The temperature of the tested product was increased rapidly at first up to 100°C and then gradually at 5°C steps until the flash point was approached. Using a spark plug, a test flame was passed across the cup at specified intervals. The flash point is the lowest liquid temperature which ignites the vapor test sample when test flame was applied. The temperature readings were taken as the flash point, and performed in triplicate. The results were reported as a mean ± standard deviation of triplicate measurements.

**Oxidative Stability:** Oxidative stability is a chemical reaction that occurs with a combination of oil and oxygen. Theoretically, oxidation rate increased with time, reducing the lubricating quality with increasing temperature. Oxidation increases the viscosity of the oil. The OST of PFAD in the current study was determined using Pressure Differential Scanning Calorimetry (PDSC), of which is a highly-selected device to evaluate the OST of oleo chemicals. The Sample placed on a film less than 1 mm of thickness and inserted into an aluminium pan firmly using a pinhole lid. Oxidation was carried out with the presence of dry air at constant pressure of 200 psi. Heating rate of oxygen gas was 10 °C/min and it was an ideal flow rate throughout the course of the study. Temperature raised at a rate of 10 °C/min starting from 50 °C to 350 °C in order to analyze the oxidative stability of the PFAD. The OST of C was calculated from a plot of heat flow (W/g) versus temperature for each experiment. The OST is defined as the temperature at which a rapid increase in the oxidation rate under a constant pressure of 200 psi occurs (Salimon, 2010). Analysis under increasing pressure has brought better stability foundation for the limited sample evaporation and shortened the time of analysis. Evaluation of OST of lubricants can be performed under isothermal and endothermal conditions (Cerny & Zelinka, 2004). The OST test was performed in triplicate and results were reported as a mean ± standard deviation of triplicate measurements.
3. RESULT AND DISCUSSION

Physical properties: In terms of appearance, the PFAD is in a semi-solid state at room temperature. Table 1, represents the physical parameters of PFAD samples compared with the standard Malaysian palm oil distilled. These parameters include viscosity, specific gravity, moisture content, and color.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>PFAD</th>
<th>PFADMS</th>
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<tbody>
<tr>
<td>Color at 28°C</td>
<td>2 R – 20 Y</td>
<td>2 R – 20 Y</td>
</tr>
<tr>
<td>Specific gravity (g/ml)</td>
<td>0.87 ±0.2</td>
<td>0.86-0.88</td>
</tr>
<tr>
<td>Moisture content (%)</td>
<td>0.63 ±0.1</td>
<td>0.03-0.93</td>
</tr>
<tr>
<td>Viscosity (40°C) cSt</td>
<td>96.35 ±0.1</td>
<td>25-30</td>
</tr>
<tr>
<td>Viscosity (100°C) cSt</td>
<td>2.93</td>
<td>-</td>
</tr>
<tr>
<td>Viscosity index</td>
<td>-345.57</td>
<td>-</td>
</tr>
<tr>
<td>Pour point (°C)</td>
<td>35±1</td>
<td>-</td>
</tr>
<tr>
<td>Flash point (°C)</td>
<td>135±1</td>
<td>-</td>
</tr>
<tr>
<td>Oxidative stability (°C)</td>
<td>166.53-178.38</td>
<td>-</td>
</tr>
</tbody>
</table>

PFAD: palm fatty acid distillate from Sime Darby; PFADMS Malaysian palm fatty acid distillate (Hosseini, 2015). Color: Color is a very important feature that indicates product composition, purity and degree of deterioration, that’s why it can be useful to verify oil degradation, stability, in addition to its suitability for a specific usage (Rossi, 2001). The color of the melted oil sample was determined by comparative analysis using yellow and red Lovibond glasses of documented characteristics (Fengxia, 2001). The color of palm oil is generated by the existence of vastly colored materials and carotene that got extracted from the seed (O’Brien, 2009). Some of the colors can be removed by adsorption, bleaching and alkali-refining (Luthria, 2004). The yellow-red (2 R-20 Y) color of PFAD is due to the Vitamin E, Squalene and Others.

Moisture Content: High-moisture content causes hydrolysis, where water reacts with oil or fat to break it down into DAG, MAG and FFA (O’Brien, 2008). Therefore, it is important to determine the moisture content of oil to prevent hydrolysis from occurring after distillation. The moisture content was determined to be 0.63±0.01 for PFAD, as shown in Table 1. In this regard, the high moisture content in oil can be attributable to the heavy raining season, poor storage conditions and lengthy period of storage. Moreover, in the sample with moisture content above the standard value 0.25% (Table 1), FFA can be formed by autocatalytic hydrolysis where the initially present FFA moieties catalyze and highly enhance subsequent formation of other FFA (Frank, 2013). This result is consistent with studies by Bonnie & Mohtat (2009), who reported moisture content of 0.03 – 0.24 for PFAD and Tan (2009), who reported a moisture content of 0.52±0.37 for crude palm oil.

Density: Density is significantly important characteristic of oils, due to the fact that weight is used as a basis for transaction when shipments of oil products are traded. Therefore it is essential to have the right and agreed values for density. This value varies and it relies on fatty acid composition and also on some minor components, as well as temperature (Gunstone, 2009). Equation (4) for determining density includes saponification value, iodine value, and temperature, as shown below.

\[
\text{Density (g/mL)} = 0.8543 + 0.000308 (\text{SV}) + 0.000157 (\text{IV}) - 0.00068t \ldots (4)
\]

Where, SV is the saponification value, IV is the iodine value and t is the temperature (°C). Table 1, shows the apparent densities at room temperature of PFAD to be 0.87±0.1. The Density of palm oil can be linked to the variance in the composition of fatty acid in the oil, where high saturated fatty acid content increases oil density.

Viscosity and Viscosity Index: Viscosity is defined as the resistance of a liquid to flow. Normally, viscosity decreases when temperature increases and in case of unsaturation, however it increases with molecular weight increase. The viscosity of PFAD found to be 96.35 and 2.93 at 40°C and 100°C, respectively, as shown in Table 1. A previous study found viscosities of high free fatty acid mixed crude palm oil to be 18.2 at 60°C (Somnuk, 2013). It can be noticed that the increase in temperature increases the movement of molecules and decreases the intermolecular forces. That movement leads the layers of the liquid to simply move over to create viscosity reduction (Mobin Siddique, 2010).

The clear effect of temperature on viscosity is illustrated by comparing viscosities of palm oil at different temperatures. The viscosities of palm oil measured to be 63.2, and 36.7 at 40°C and 100°C, respectively (Mobin Siddique, 2010).

Pour Point: Pour point is a vital factor to decide if the oil is well suited to be a potential lubricant or not. Being the lowest temperature point at which the liquid sample can be poured, this characteristic is essential for the lubricate to be used at low operating temperatures, especially in automotive and industrial fluids.

The pour point of PFAD was observed at 35°C, which is higher than the pour point value of palm oil (Table 1). The results show that oil from shorter saturated fatty acids give a favorable lower pour point. The pour point depends on the carbon chain length and the saturation degree. Oils with Longer saturated fatty acid have higher
Oxidative Stability: Oxidative stability is a chemical reaction that occurs with a combination of oil and oxygen. Oxidative stability is a measure of a substance (e.g. a lubricant or a fuel) resistance to oxidation. The oxidation reaction usually starts relatively slow then it suddenly speeds up. The reason for that the process occurs as a chain reaction. Measuring of the oxidative stability can be done with an accelerated method. Acceleration is done by influencing all experimental parameters to produce results in a rational period of time. The main parameters are higher temperature, pressure, and the flow rate of air (containing oxygen) to the sample. In the present study PFAD were tested for oxidative stability determination. PFAD showed high of oxidative stability values at the range of 166.53-178.38°C which would indicate their suitability to be used as a commercial lubricant without any antioxidant additives.

4. CONCLUSION

Several physical parameters of PFAD from Sime Darby were measured and compared with previous studies. These quality-determining parameters of PFAD showed comparable values to results from earlier evaluations, with small increases in values. A rather significant outcome is the great benefit of using PFAD for bio lubricant, which offers many positive characteristics; it increases the viscosity, viscosity index, flash point and oxidative stability. Therefore PFAD has a good potential for biodiesel and bio lubricant industries.

The results show that PFAD is a cheap raw material for fatty acid production with acceptable purity. PFAD is important raw materials in oleo chemical industries, such as soap, greases, cosmetic and pharmaceutical production.

5. ACKNOWLEDGMENTS

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