

FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA
SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY



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BIENNIAL ENGINEERING CONFERENCE

Theme:

**DECAY IN INFRASTRUCTURE - A CHALLENGE
TO SCIENCE AND ENGINEERING RESEARCH
IN REALISING VISION 20-2020**

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Technology, Minna Main Campus*

on

26th – 28th June 2008

**THEME: DECAY IN INFRASTRUCTURE – A CHALLENGE TO SCIENCE AND ENGINEERING
RESEARCH IN REALISING VISION 20 – 2020**

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PRODUCTION OF BIODIESEL FROM USED FRYING OIL

by

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ABSTRACT

Used frying oil (fried palm oil) was treated with silica gel so as to reduce some of the poor quality parameters of the oil especially the Peroxide and Free Fatty Acid Values. The free fatty acid and peroxide values of the used treated oil subjected to adsorbent treatment was reduced by 31.14% and 47.8% respectively. The pretreated used fried palm oil was converted into methyl esters as biodiesel via transesterification reaction using Sodium hydroxide. The methyl esters produced had comparable fuel properties as petroleum diesel; hence it can be used as a petrol-diesel substitute.

KEYWORDS: *Methyl Esther, Tranesterification, Free Fatty Acid, Peroxide value, Total Polar -Material*

INTRODUCTION

Most of the energy needs for domestic and industrial consumption come from early few precious natural resources. Energy from petroleum source is non-renewable and oil reserves have been shrinking in recent years. This will automatically lead to exhaustion of this non-renewable energy source in the near future. (Phillip, 1992). However, the biggest challenge for developing countries in relation to energy consumption is to develop and implement technology, i.e. that can help reduce the emissions of gasses and particulate matter which have both local and environment impact. In order to be more environmentally conscious, there

is a need to use energy resources preferentially, with reference to the ones that create fewer pollutants as by-products. Palm oil meets this requirement as it has no unpleasant room odour, high resistance to oxidation and high content of nutrients from its fatty acid composition and does not polymerize easily. The indicators of poor oil quality includes: elevated free fatty acids (FFA), high total polar material (TPM), change of colour, high foaming property, low iodine value and increased viscosity. Literature reveals that colour and stability of used frying oil can be improved using adsorbents such as zeolite, activated carbon, activated clay, aluminum hydroxide gel etc. (Loh et-al, 2006) For improving the quality parameters such as total polar materials, oxidation products polymerization, FFA, Colour and oil stability, silica gel was found to be the most suitable adsorbent in reducing FFA, TPM, oxidation products, viscosity and oil decolourization without altering the chemistry and stability of the recovered oil. (Loh et-al, 2006)

The scarcity of diesel fuel in Nigeria is on increase geometrically. (Alhassan and Isah, 2006) This scarce resource is causing low productivity in our industrial sector and other businesses thereby having negative effect on the economy of the nation. Fossil fuel causes air pollution to our environment with emission of CO₂ and other green house gases. Based on this fact, there is need for the production of an alternative diesel fuel from used frying palm oil in order to counter the difficulties and improve the economy of this nation.

Alhassan, and Isah, (2005) worked on production and characterization of bio-diesel from cotton seed oil, while Loh et-al (2006) looked at improving the colour and stability of used frying oil using adsorbents such as zeolite, activated carbon, activated clay, and silica gel. This research on the production of biodiesel from pre-treated used frying oil will provide an alternative source of energy to the conventional crude oil. Also, it will help to effectively and efficiently harness and exploit petroleum alternative.

EXPERIMENTAL AND RESEARCH METHODOLOGY

Materials

Used frying oil was obtained from local bean cake seller. The cooking oil in the villages is mainly palm olein.

Pretreatment of the used frying oil.

300ml of the oil was first dissolved in 100ml of n hexane. The mixture was mixed with equal weight of silica gel. The mixture of oil, hexane and Silica adsorbent was stirred continuously for 30 minutes using Gallen kamp orbital shaker. After the reaction time, whatman filter paper of size A2 was used to filter the mixture and transferred into a round bottom flask and kept in an incubator for vacuum filtration. Thereafter, the filtrate was vacuum dried using Gallen kamp oven dryer to remove solvent and water. The pretreated oil remaining was then analyzed for free fatty acid and peroxide value (PV) before conversion into methyl ester. (Cormicks, 2006, Paul and Mittal, 1997)

Determination of Free Fatty Acid in Palm Oil (Acid Value)

Weighing dish containing about 5g of the oil was weighed and poured into the conical flask and then 50ml of hot neutral alcohol together with few drops of phenolphthalein added and shake vigorously. The mixture was titrated against 0.5M Sodium hydroxide solution with constant shaking until the pink color appeared. From the volume of 0.5M alkali used, the percentage acid present in term of oleic acid was then calculated. (Hoover, 1966)

Determination of Peroxide Value

20ml of glacial acetic acid and 10ml chloroform were added to 1g of oil sample and then boiled for 1 minute. The mixture was then transferred into a flask containing 20ml of 5% potassium iodide solution; then few drops of starch solution were added and titrated with 0.025M $\text{Na}_2\text{S}_2\text{O}_3$ to a faint yellow color. Also, 1ml of starch indicator was added and continued with titration until the blue color disappears. (Hoover, 1966)

Preparation of Methyl Ester.

100ml of the pretreated use frying oil was measured and poured into a conical flask, then converted to methyl ester using 1g of Sodium hydroxide pellet which was first dissolved in 45ml of methanol. The solution was poured into a conical flask containing the oil. The mixture was heated to 60 °C and stirred continuously until the fatty acid is converted to biodiesel. The products were decanted for each run of transesterification reaction. (Choo, et-al, 1993)

Analysis of Methyl Esters Produced.

Determination of Density

The mass of the oil was measured using a density bottle.

Determination of Specific Gravity

A known volume of methyl ester was weighed. The ratio of the weight of methyl ester to that of the same volume of water gives the specific gravity.

Refractive Index Test

The refractive index was determined by placing a drop of oil sample on the glass slab of the refractometer and the adjustable hands of the meter was adjusted to give the reading at 25°C. (Mayagi, A. et-al, 2003)

Sulphur Test

The sulphur content of the methyl ester produced was determined using ASTM D2622 method. The sample was

placed in an X-ray beam and the intensity of the Sulphur X-ray fluorescence of LOVI BOND-57 sulphur analyzer was measured. (Mayagi, A. et-al, 2003)

Pour Point Test

The methyl ester was poured into the test jar in the appropriate level. The test jar was tightly closed by cork into which the thermometer was inserted. The position of the cork was adjusted and the thermometer fits the cork tightly. The thermometer and the cork were co-axially and the thermometer bulb was immersed such that one end of the capillary is 3mm below the surface of the oil.

The oil was heated with stirring to 46°C in water bath maintained at 48°C. The fuel was cooled to 35°C in the bath. The disk was placed in the bottom of the jacket. The ring was placed around a test jar 25mm from the bottom. The test jar was inserted into the ice jacket. The jacket was supported containing the test jar, firmly in a vertical position in a cooling bath. After preliminary heating, the methyl was cooled to a lowest temperature at which the movement of the methyl ester was observed and recorded as pour point. (Phillip, 1992, Mayagi, et-al, 2003)

Flash Point Test

Procedure for determination of flash point by Pensky-martens (ASTMD₉₃), the cup was filled with specific level with a sample of methyl-ester. The temperature of the sample was increased rapidly at first and then slowly as the flash point is approached at 200°C a small test flame was passed across the cup. At a point, the vapour above the surface of the testing sample was ignited with the aid of a testing flame across and the temperature at this point was noted and recorded, where the flame causes the oil sample to ignite and burned consciously for some seconds. The temperature at which this occurs is at the fire point. (Akoh and Reynolds, 1998)

Distillation Characteristics

Heat was supplied to the distillation flask in a gradual process and the initial boiling point was observed and recorded, with the tip of the condenser away from the walls of the graduated cylinder. The cylinder was moved immediately so that the tip of the condenser touches its inner wall. The heating was

recorded so that the time from initial boiling point to 10% volume was recorded.

However, the heating was continuously regulated so that the uniform average rate of condensation for 10% recovery interval could be obtained. In the interval between the initial and the final boiling point, all volumes in the graduated cylinder and the thermometer reading corresponding to this were recorded. While the condenser tube continues to drain to the graduated cylinder, the volume was measured and recorded. After cooling the flask, its content was poured into the cylinder. The flask was allowed to drain until no appreciable increase in the volume of the liquid in the cylinder was observed. The value obtained for the percentage methyl-ester recovery was added to the percentage residue and the total recovery was then obtained. The percentage total recovery was deducted from 100 to obtain the percentage loss. (Akoh and Reynolds, 1998)

RESULTS

The data obtained for the percentage Improvement for peroxide and free fatty acid was tabulated in Table 1, while the characteristics of the used frying oil after pre-treatment by silica gel are shown in Table 2.

TABLE 1. PERCENTAGE IMPROVEMENT (PI) FOR PEROXIDE VALUE AND FREE FATTY ACID (FFA) OF THE USED FRYING OIL WITH SILICA GEL AS AN ADSORBENT.

| Sample | Peroxide Value (PV) % | Percentage Improvement (P _i) % | % Free Fatty Acid (FFA) | Percentage Improvement (P _i) (FFA) % |
|---|-----------------------|--|-------------------------|--|
| Used frying oil before pretreatment (P ₀) | 6.70 | | 8.96 | |
| Used frying oil after adsorbent treatment (P ₁) | 3.50 | 47.8% | 6.17 | 31.14% |

TABLE 2 COMPARISM OF THE PROPERTIES OF PETROLEUM DIESEL AND BIODIESEL STANDARD VALUES WITH THAT OF METHYL ESTERS PRODUCED FROM PRE TREATED OIL.

| Properties Determined | Standard Values | | | Experimental Values |
|--------------------------------------|-----------------|-----------|--------------------|---------------------|
| | Diesel Fuel | Biodiesel | Low Sulphur Diesel | |
| Specific Gravity (at 25oC) | 0.82-0.87 | | 0.854 | |
| Density at 25oC (g/cm ³) | 0.82-0.88 | 0.87-0.89 | 0.83-0.86 | 0.85 |
| Flash point (oC) | >52 | >100 | >60 | 175 |
| Pour point (oC) | -10 to 0.01 | <-10 | | -6.5 |
| Refractive index (25oC) | | | | 1.35 |
| Sulphur content% | 0.05max | 0.01max | 0.03 | 0.02 |
| Distillation Characteristics | | | | |
| IBP (oC) | | | | 141 |
| FBP(oC) | | | | 347 |
| % total recovery | 90 | >98 | >98 | 97.74 |
| percentage residue | | | | 1.23 |
| percentage loss | | | | 1.03 |

DISCUSSION OF RESULT

From Table1, the calculated percentage improvement of the peroxide value is 47.8%, and that of the free fatty acid (FFA)

is 31.14%. This shows the effectiveness of silica gel used as adsorbent in pre-treatment of the used frying oil. A similar result was found previously by Miyagi and Nakajima (2003) and Loh, et-al (2006). The analysis of the biodiesel produced

from used frying oil as presented in Table 2 shows that the density and specific gravity at 25 °C are 0.85 and 0.854 respectively. The density of the standard biodiesel fuel is (0.87 – 0.89) g/cm³, comparing with the standard diesel fuel which is (0.82 – 0.88)g/cm³. From the values obtained, it was found that the methyl ester produced when characterized has value closer to that of petrol diesel. Thus, methyl ester is within the specific range for diesel fuel. The pour point of the methyl ester is -6.5°C and this is a temperature at which the movement of the oil will be observed when cooled. The pour point of petroleum diesel fuel is between -10 to 0.01. Comparing this with the values obtained for the methyl ester, there is a little difference which means that the methyl ester is pour faster than the petroleum diesel. (Choo, et-al, 1993)

The flash point of the methyl ester is 175°C, while that of petroleum diesel is within the range of 59 to 66°C.² The flash point of the methyl ester is not within the range of petroleum diesel, indicating that the methyl ester has a higher flash point than the diesel fuel. This implies that bio-diesel obtained from used oil will ignite easily than diesel thereby making it a safer fuel. The distillation recovery of the methyl ester is 97.74% and this is the maximum percentage recovered. The percentage residue is 1.23 and the percentage loss is 1.03 of the quantity distilled. The range of loss for petrol diesel is between 0.1 to 1.0 and the value obtained for methyl ester falls within this range which means that the pure product would in turn give a greater efficiency.

The various test carried out gives results that are relatively close to the values obtained for petroleum diesel. From this result, it can be deduced that the methyl ester from pretreated use fried palm oil can be used in an unmodified diesel engine. The methyl ester yield reasonable energy and significant reduction in carbon IV oxide emissions hence it is less toxic and more environmental friendly.

CONCLUSION

The fried palm oil (mainly palm olein), after pretreatment with the silica gel is a suitable feed stock for conversion to methyl ester by catalytic reaction called trans esterification using sodium hydroxide (NaOH). The characterization of the methyl ester produced give results that are relatively close to

the values obtained for petroleum diesel. From this result, it can be deduced that the methyl esters produced have fuel properties comparable to those of petroleum diesel thus it can be a diesel substitute for unmodified diesel engines. Apart from providing an alternative to fossil fuel, methyl ester is a renewable motor fuel which causes less environmental pollution, permit greater energy diversification and reduce fuel importation.

RECOMMENDATION

It is recommended that equipments such as sintered glass for filtration and vacuum dryer may be considered more effective than whatman filter paper and oven dryer that were used in the pretreatment of the fried palm oils as it saves time and give higher purity of products

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