Performance Study of Oxidation Stability Test Rig By Using Pongamia Pinata And Coconut Biodiesel With The TBHQ Antioxidant

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Abstract- *The scope of the project is to produce a new oxidation stability test device for testing the biodiesel and make a good marketable product in low price when compare to other existing test devices. For fulfilling our objective, need to conduct experiment on test rig and comparing the results with standard PetroOXY test device. The vegetable oils, animal fats and their biodiesel suffer with the drawback of deterioration of its quality when it is in contact with oxygen. The present available methods of finding the oxidation stability of biodiesel are Rancimat test [EN 14214] method and Petroxy [ASTM 6751 – 07b] method. In this research project a new oxidation stability test rig is designed and fabricated by the reference of standard methods. The test procedure and initial conditions of our test rig is made similar to the Petroxy test by maintaining the constant temperature at* 140^0C *and replace compressed air by oxygen. The Pongamia pinnata and coconut biodiesel is used for experiment; the same samples are tested in both our new test rig and standard Petroxy test device and compare the results. The TBHQ antioxidant is used here for enhancing the oxidation stability of the biodiesel.*

Keywords- Oxidation Stability, Pongamia pinnata, Coconut, Biodiesel, TBHQ, New Test rig

I. INTRODUCTION

Biodiesel represents an alternative to petroleum-based diesel fuels and it is consist of mono-alkyl esters of fatty acids, extracted from plant oils and animal fats. The raw materials of biodiesel are oils from soy, palm, corn, rape seed and canola etc. New seed oils are under consideration include mustard seed, Pongamia pinnata, peanut, sunflower, jatropha and cottonseed. The commonly used animal fats include those derived from poultry, beef and pork. Biodiesel provides engine performance similar to that obtained with diesel fuel. Because biodiesels are energetically same with diesel fuel, interest is shift on vegetable oils as alternate fuels. Performance and efficiency at low engine ratings are same expected for operation on diesel fuel. The work related with the use of organic oils as fuel in diesel engines, harmful exhaust emissions, particularly sulpher, hydrocarbon (HC), smoke and carbon monoxide (CO) are considerably reduced as compared to diesel. However piston rings and cylinder liner wear are greater than expected for operation on diesel fuel.

India can produce about 4 – 5 million tons per year of biodiesel, which is about 10% of our current diesel demand of about 46 million ton. In Karnataka bio-fuels production from non-edible oil initiated by the Karnataka government and Karnataka state bio-fuel information and demonstration center is taking much interest in bio-fuel and initiated bio-fuel production and R&D center in every district of Karnataka. KSBF I&D center is funds NIE-CREST for generation of biodiesel, conducting the awareness programs to educating the formers. But apart from all these advantages bio-fuel or Biodiesel has some serious drawbacks, short comes and problems compare to conventional fuel.

The oxidation stability of the biodiesel is defined as the ability of the biodiesel to resist the oxidative degradation when contact with oxygen is called oxidation stability of biodiesel [4] it directly impact on the storage capacity of the biodiesel. The thermal stability also affects the storage stability of the biodiesel. The thermal degradation due to formation of sediments and deposits during the storage by the effect of temperature leads to degrading the biodiesel is called thermal degradation. The thermal stability of the biodiesel is important factor to know the quality and performance along with the oxidation stability. The oxidative degradation of biodiesel caused due to reaction with oxygen and thermal degradation occur by storing biodiesel at high temperature. The new standard test methods is required to find the oxidation stability and thermal stability of biodiesel, this is the main objective of the project.

Oxidation Stability of Biodiesel

The long chain free fatty acid ester is present in biodiesel due to presence of unsaturated fatty acids in vegetable oils and animal fat, FFA leads to accelerate the oxidation by expose to air and high temperature during storage, may yield the polymerized compounds. [1] Auto oxidation of biodiesel cause degradation of fuel quality by affecting the stability parameters.

Initiation

$RH + I \longrightarrow R^* + IH$

Propagation

$R + O2 \longrightarrow ROO^*$

$ROO^* + RH \longrightarrow ROOH + R^*$

Cycle repeats

Termination

$R^* + R^* \longrightarrow R - R$

$ROO^* + ROO^* \longrightarrow$ Stable product

Figure 1.1: Oxidation reaction in biodiesel

Oxidation reaction Figure 1.2, leads to formation of end by-products like alcohols, aldehydes, shorter chain of carboxylic acids and gum like sediments in the biodiesel it results in blocking in the fuel filter and fuel injection nozzle, deposits formation in the combustion chamber of the engine and other components of fuel supply system. Because of these problems oxidation stability is an important parameter for addressing the quality and purity of biodiesel. The oxidative degradation of biodiesel can be controlled by the addition of antioxidants.

Existing Standard Test Methods

Rancimat Test [EN 14214]

The Rancimat test has two methods one is long term storage stability test and other one is rapid acceleration oxidation stability test. Oxygen is passing continuous to the sample at the rate of 20 liters per hour and maintaining constant temperature at 1100C. The free fatty acids are oxidized during the reaction with oxygen. The volatile and secondary reaction productions are formed at the end of the experiment and transferred into the measuring vessel by the air stream and absorbed from measuring solution, it is increase by the absorption of by-products of oxidation reaction. The time until the conductivity of biodiesel suddenly rise at a point is called induction time; Rancimat test characterizes the oxidation stability of oils and fats. The Standardized oxidation stability period of biodiesel is 6hr.

Petroxy Test [ASTM 6751 – 07b]

The Petroxy test method is capable of testing the biodiesel, lubricant oil and conventional diesel oxidation stability. This equipment has 20ml capacity reactor in that 5ml sample is taken for experiment. The oxygen supplied to reactor at 7bar pressure and then starts the experiment by heating upto constant temperature at 1400C. The expansion of oxygen molecule due to heat input leads to increase the pressure in reactor. The termination of the experiment when pressure drops 10% of the maximum pressure, then automatically end the experiment and display the experimental results.

II. OXIDATION STABILITY TEST RIG AND METHODOLOGY

The new oxidation stability unit having test rig set-up and automated control system these two is important parts of this experiment. In reactor set-up reactor shell is heart of the test rig in which the biodiesel sample is poured to find oxidation stability. This reactor design is done based on the DGMK 714 standards. The solid works 3D model as shown in the Figure 3.1 and also determine the maximum working pressure of the reactor by using longitudinal stress formula. The biodiesel is heating is done by induction heating band. The heating band is controlled continuously to maintain the temperature at 1100C till the completion of the experiment as per the previously followed procedure

Figure 2.1: Designed 3D view of the reactor unit

The pressure relief valve is used for safety purpose it maintains the compressed fluid pressure within predetermined

working pressure of the reactor if pressure more than predetermined pressure than relief valve is opened and excess pressure went out by axillary path from the reactor. The 10bar capacity pressure relief valve is used and 7bar is the working pressure of test rig.

Control System and Monitoring system

The development of control system including temperature monitoring system and pressure monitoring system, for this project thermocouple and relay circuit is used for temperature monitoring where thermocouple sense the change in temperature and sends analogue signal to signal conditioner to covert from an analogue input to digital output which is fed to MODBUS. The control system ensures the operating temperature in reactor should be kept constant. In pressure monitoring system the pressure transducer sense pressure inside the reactor and sends analogue signals to signal conditioner for getting the output digital values.

The both monitoring systems are programmed in the microcontroller 8028 which is flexible configuration option for 8 channels accepting universal input and 4 relay to serve these applications. This unit has separate Numeric display for group and process value. The Figure 3.3 shows the automatic controlling and monitoring system.

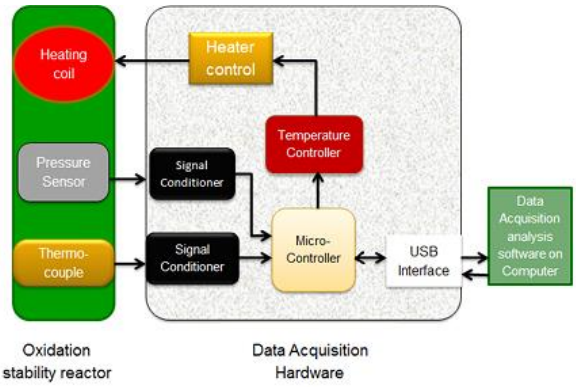

Figure 2.2: Automation of Control and Monitoring system

III. METHODOLOGY AND TEST PROCEDURE

The design and development of the new oxidation stability test rig have follow the working principle of existing standards like Petroxy and Rancimat oxidation stability test methods, in particularly it works on the basis of Petroxy [ASTM 6751 07b] test method. The test procedure, initial conditions and co-relations are followed by the Petroxy test apart from difference is the quantity of sample used for the test, in Petroxy test 5ml of biodiesel sample is used for the test but in our new test rig 215ml of biodiesel is used for every sample. The reactor designed for 1:3 ratios volume fraction of bio-fuel and the oxygen to be filled in the reactor, it is by the reference of the rotating bomb oxidation test method and the Petroxy test method. [9], [7].

The test rig consisting of monitoring system which is fully automated by MSIBUS 8028 microcontroller and online data acquisition system. The induction heating coil is placed on the circumference of the reactor which is connected to relay for cut-off and on the electric connection when biodiesel reach at predefined temperature range. The test rig consisting pressure transducer it is connected to microcontroller through signal conditioner which converts analogue input data to digital output. Similarly thermocouple connected to microcontroller through the signal conditioning device.

The previously we considered some preliminary test conditions which are not matching with the standard Petroxy test method which are initial pressure considered as 5bar, 1100C biodiesel temperature to be maintain constant throughout the experiment and compressed air is supplied for the experiment which are make more fluctuation in the result.

The standardization of the test rig is main objective of this project, for standardization it need to compare the results with standard test method for that it need to change some of the parameter as in the standard test method. The initial pressure is increased to 7bar; constant biodiesel temperature is maintained at 1400C and 99% pure oxygen is supplied to reactor shell which clears the path for standardization.

Experiment Procedure

The below steps are followed to conduct the experiment in new oxidation stability test rig and standardization is done by comparing results with the Petroxy test results, which needs to similar test procedure and preconditions in both device. The following steps are used to conduct the experiment.

- Clean the reactor shell and end cap with ethanol and dry it with clean tissue paper.
- The test sample B100 Pongamia pinnata biodiesel of 215ml i.e. 1:3 ratio of biodiesel and compressed air for the experiment
- The reactor and end cap tightened by nut and bolt joint and silicon ‘O’ ring placed in between to prevent the pressure leakage.
- Oxygen is supplied from the cylinder at 7bar pressure to reactor shell.

- The thermocouple and pressure transducer are connected to the MASIBUS 8028 microcontroller system.
- The reactor is fixed to the stand and enclosed with the safety hood.
- Then switch on the automated control system and the heating coil to increase the temperature of the reactor upto 1400C and keep it constant throughout the experiment.
- By automation heating element gets on and off to maintain the constant temperature.
- The maximum test duration is 16hrs.
- The oxidation stability of biodiesel calculated by reducing the pressure by 10% from the maximum pressure
- The test is carried out to the different biodiesel and blends of the Biodiesel.

IV. RESULT AND DISCUSSION

The oxidation stability of both Pongamia pinnata and coconut biodiesel with and without TBHQ antioxidant samples are prepared for experiment. The experiment on Pongamia pinnata biodiesel is conducted with previous test procedure and supplied compressed air to the test rig, with this condition done the experiment and got the results. The same sample tested in Petroxy test device and got oxidation stability result; both the results compared and noticed that deviation in the results due to change in test procedure and preconditions. The changes made in the test rig and control system to set constant temperature at 140^0C and provide oxygen supply to reactor for remaining all experiments. The below Table 5.6 shows the oxidation stability of biodiesel without adding TBHQ antioxidant and the results of both Pongamia pinnata and coconut biodiesel are not meet the standard duration of oxidation stability i.e. 3 hours in Petroxy test ASTM standard.

The same samples of Pongamia pinnata and coconut biodiesel is tested in standard PetroOXY test method here the oxidation stability results are lower compare to our test device, but this is the standard test method and should follow this results as a correct results. The variation of the result due to high rate of heat transfer between steel reactor shell and biodiesel it results problem in maintaining the constant temperature and also need inaccuracy in the automatic monitoring system.

Table: Oxidation stability of Biodiesel without antioxidant

Experiment on Oxidation stability of *Biodiesel without anti-oxidant*				
Particulars/Properties	Pongamia pinnata		Coconut biodiesel	
Test rig used	On New test rig at NIE-CREST	On Petroxy test at HPCL	On New test rig at NIE-CREST	On Petroxy test rig at HPCL
Temperature maintained	140^0C	140^0C	140^0C	140^0C
Pressure Provided	7 bar	7 bar	7 bar	7 bar
P_{max} Obtained during experimentation	9.58 bar	9.99 bar	9.38 bar	11.24 bar
Supply	Oxygen	Oxygen	Oxygen	Oxygen
Oxidation stability obtained	52 min.	35.05 min	38 min.	29.58 min.

The oxidation stability of biodiesel is improved by adding the additives like antioxidants, here also used TBHQ antioxidant to prevent the oxidation reaction in the biodiesel. The biodiesel and antioxidant sample is prepared by considering the optimum amount of antioxidant per liter of biodiesel and prepared using magnetic stirrer. The Pongamia pinnata and coconut biodiesel with antioxidant samples are tested with both our test rig and PetroOXY test method and the results are shown in below Table 5.7. The results of Pongamia pinnata and coconut biodiesel in our test rig are 169 min. and 125 min respectively and the results of same biodiesel samples are 138.21 min and 88.36 min. respectively. Here, also variation between our test results and PetroOXY test result for the same reason as above mentioned but positive thing is addition of TBHQ antioxidant helps in improving the oxidation stability of the biodiesel.

Table: Oxidation stability of Biodiesel without antioxidant

Experiment on Oxidation stability of Biodiesel with antioxidant				
Particulars / Properties	Pongamia pinnata		Coconut biodiesel	
Test rig used	On New test rig at NIE-CREST	On Petroxy test at HPCL	On New test rig at NIE-CREST	On Petroxy test rig at HPCL
Temperature maintained	140^0C	140^0C	140^0C	140^0C
Pressure Provided	7 bar	7 bar	7 bar	7 bar
P_{max} Obtained during experimentation	9.59 bar	10.70 bar	9.48 bar	9.86 bar
Supply	Oxygen	Oxygen	Oxygen	Oxygen
Oxidation stability obtained	169 min	138.21 min	125 min	88.36 min

V. CONCLUSION

The standardization of our new oxidation stability test rig need to similar in the test procedure and preconditions, for that changed the constant temperature of experiment from 1100C to 1400C by referring the Petroxy test method and oxygen supply is provided instead of compressed air for the experiment. The test procedure and preconditions is

maintained as same in the Petroxy test method for next samples of Pongamia pinnata and coconut biodiesel. Then conduct the experiment in both our test device and PetroOXY test device, then compare both results this time also small variation in the results, due to inconsistent in maintaining the constant temperature due to convective heat transfer from reactor shell to biodiesel sample even cut-off the heating coil power and lack of accuracy in controlling & monitoring system.

The additives also added for both Pongamia pinnata and coconut biodiesel the result of oxidation stability varies when compare to Petroxy test results because of above said reason, but creditable thing is the oxidation stability of both biodiesel samples are increased drastically, it is shown in the above comparison result. The properties which are influencing the oxidation stability of the biodiesel is found out for both fresh biodiesel (before the experiment) and oxidized biodiesel (after the experiment), the results of biodiesel with TBHQ anti-oxidant is good compare to the without anti-oxidant in both oxidation stability and properties of the sample. The standardization of our test rig is not possible due to variation of oxidation stability result in our test rig, because of above disadvantages in the test rig.