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Optimization of Reaction Parameter in an OBR

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Abstract
In this paper, it is planned to study Transesterification reaction on castor oil in a oscillatory baffled reactor using potassium hydroxide as a catalyst. The variables chosen for the study were Residence time, Oil to methanol ratio, Catalyst concentration; and Reaction Temperature. The effects of these variables on the viscosity of biodiesel were studied, since this is one of the important specifications in ASTM standard. Apart from viscosity other properties like sp.gr, acid value, and sap value were also determined for the biodiesel product. As supply of fossil fuel is limited whilst energy demand continues to rise, hence alternative renewable fuels have received increasing attention for future utilization.

Key Words: Biodiesel, transesterification, castor oil

Introduction
Oscillatory baffled reactors (OBRs) are a form of reactor, in which tubes fitted with orifice plate baffles have an oscillatory motion superimposed upon the net flow of the process fluid. Through the interaction of the baffles with the oscillatory motion of the fluid uniform mixing and enhanced transport rates are achieved, whilst maintaining conditions approximating plug flow.[1]

Oscillatory baffled reactors (OBRs) are a form of plug flow reactor that can be used for long reactions, as the plug flow is not dependent on achievement of a certain velocity in the reactor leading to much more compact designs. One class of long reactions is that of bioreactions/fermentations. Since one major advantage of the OBR is its highly uniform, controllable mixing, which can be used to enhance gas-to-liquid mass transfer, it should be suitable for biological reactions.

It was observed[1] that oscillatory flows through geometries in which a surface was presented transversely to the flow could produce uniform and efficient mixing patterns. The optimal geometry for uniform mixing of fluid within a tube was found to be equally spaced orifice plates 1.5 tube diameters apart of 0.25 fractional open cross-sectional area. In the resultant ‘oscillatory flow reactor’, each baffle essentially behaves as a stirred tank, hence, if there are enough baffles in series, a good approximation to plug flow can be achieved.

Biodiesel as an alternative fuel, has many merits. It is derived from a renewable, domestic resource, thereby relieving reliance on petroleum fuel imports. It is biodegradable and non-toxic. Compared to petroleum-based diesel, biodiesel has a more favorable combustion emission profile, such as low emissions of carbon monoxide, particulate matter and unburned hydrocarbons. Carbon dioxide produced by combustion of biodiesel can be recycled by photosynthesis, thereby minimizing the impact of biodiesel combustion on the greenhouse effect. Biodiesel has a relatively high flash point (150 C), which makes it less volatile and safer to transport or handle than petroleum diesel. It provides lubricating properties that can reduce engine wear and extend engine life. In brief, these merits of biodiesel make it a good alternative to petroleum based fuel and have led to its use in many countries, especially in environmentally sensitive areas.

The application of external energy in the pulsing form (oscillatory flow mixing - OFM) has, for a long time, been a common practice to improve reaction performance, namely mass transfer rates in chemical engineering units. The general principles associated with the pulsing column were established by Van Dijck (1935), at the Royal Dutch/Shell Laboratory in Amsterdam, in the 1930’s .Since then, a number of techniques, based on several principles, have been developed and adapted for their applications to very different fields .

Types of oscillating devices
In general, oscillating equipment may be classified in two main types:

a) Alternating motion of some intrinsic elements of the column. It is worth mentioning the reciprocating plate columns , in which the pulsation is generated by means of an upwards-downwards motion of plates and the columns with oscillating piston , where a plug is coupled to the bottom of the column .

b) Oscillation is generated by the hydraulic transmission of a perturbation to the liquid contained in the column. This perturbation is typically generated by e.g. systems using positive displacement pumps (plug or membrane) to introduce the feed into the column and the pneumatic oscillating systems. In the latter example, the oscillation is generated by means of a pressurized gas which propels the liquid contained in a parallel branch to the column. The self-propelled oscillators are based on a different concept. In this case, fluid oscillating is the result of the liquid entering the columns, through a pulsation chamber. Once the pressure in the chamber is high enough, the membrane covering the feeding tube injects the liquid into the column; this membrane then closes the inlet tube again, which creates a cyclical feed system. In contrast with the previous pulsators, in this system, the motion of the liquid in the column is always generated in the upward direction.

Industrial applications of oscillating reactors

The oscillating reactors were firstly used in separation processes in order to enhance the contact between the phases and, consequently, to improve mass transfer rates. Since then, they have been applied to a number of systems, either chemical or biochemical, under several configurations. In the last three decades, the number of publications resulting from the study of OFM has increased several times, which demonstrates that this is a technology creating an increasing interest in the scientific community. Several patents are currently protecting novel oscillating devices' designs and/or their commercial applications.

Materials and Method

In this Paper Castor oil was used to manufacture biodiesel in OBR using transestrification reaction. The most common way to produce biodiesel is by transesterification, which refers to a catalyzed chemical reaction involving vegetable oil and an alcohol to yield fatty acid alkyl esters (i.e., biodiesel) and glycerol. Triacylglycerols (triglycerides), as the main component of vegetable oil, consist of three long chain fatty acids esterifies to a glycerol backbone. When triacylglycerols react with an alcohol (e.g., methanol), the three fatty acid chains are released from the glycerol skeleton and combine with the alcohol to yield fatty acid alkyl esters (e.g., fatty acid methyl esters or FAME). Glycerol is produced as a by-product. Methanol is the most commonly used alcohol because of its low cost and is the alcohol of choice in the processes developed in this study. In general, a large excess of methanol is used to shift the equilibrium far to the right.

Reaction:

Triglyceride + Methanol → Diglyceride + Methyl Ester

Diglyceride + Methanol → Monoglyceride + Methyl Ester

Monoglyceride + Methanol → Glycerol + Methyl Ester

Overall:

Triglyceride + 3 Methanol → Glycerol + 3 Methyl Ester

Experimental Set Up

Initially the range for operating condition was taken according to physical properties and stichiometric ratio. Methanol having boiling point 62^{0}C, therefore higher level for reaction temperature was taken 60^{0}C. As per stichiometry reaction carried out at 3:1 methanol to oil molar ratio. The reaction is a reversible one, so, an excess of methanol is necessary to drive the equilibrium towards methyl ester formation. Hence actual experimental tests had taken at higher molar ratio.

For each experiment, Castor oil of measured 100ml was added in an OBR and heated to specific temperature. Next, make dissolved solution of measured quantity of methanol and sodium hydroxide. By keeping required temperature constant add methanol, Potassium hydroxide solution in OBR where Oscillation having speed of 100 RPM . The reaction time is varied in the range of 30 to 60 min.

Results and Discussion

The product obtained during various runs was analyzed for specific gravity, viscosity, acid value and sap. Value. Specific gravity of biodiesel was determined at room temperature (37^{0}C). Kinematic viscosity was determined at 40^{0}C. The quality of biodiesel was measured mainly in terms of viscosity, since this one of the important specification in ASTM standards. Hence the quality of biodiesel is discussed in terms of change in viscosity with the variables like residence time, temperature, oil to alcohol ratio. The stirring speed selected for the all runs 460$\pm$10 rpm as higher stirring speeds produces lot of vibrations.

Effect of various parameters on biodiesel properties

Effect of residence time

Data are presented for changes of viscosity of biodiesel product for different reaction time are shown in table-1, and figure-1. Experiments work were carried out varying the residence time in the OBR from 30 minute to 90 minutes keeping other parameter like reaction temp 30^{0}C,oil to alcohol molar ratio 1:9 ,stirring speed 460$\pm$10. The data sows viscosity decreases with increasing time from 30min to 45 min. further value of viscosity increases with increasing time. Hence optimum reaction time may be taken as 45 min. with lowest viscosity 14.10cSt.

The variation of specific gravity with residence time appears as follow similar trend as that of viscosity. That is specific gravity decrease from 0.9096 to 0.882 as residence time is increased from 30 to 45 minutes, further increase in residence time from 60 to 90 minutes the specific gravity increases 0.882 to 0.9026

While acid value of biodiesel product decreases with increasing reaction time and saponification value decreases with increasing reaction time and further increases.

Time of run (min)	30	45	60	90
Kinematic viscosity, (cSt)	18.12	14.10	14.51	`15.15
Specific gravity	0.9096	0.890	0.90	0.9026
Acid value	1.26	0.57	0.64	0.68
Sap value	177.6	173.4	174.3	174.9

Table No.1 Variation of Viscosity with Time

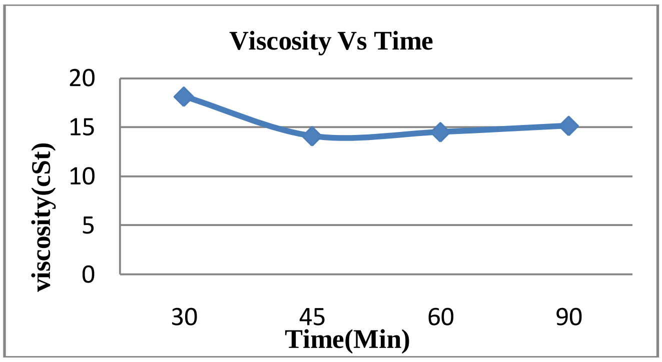


Figure-1: variation of Viscosity Vs Reaction Time

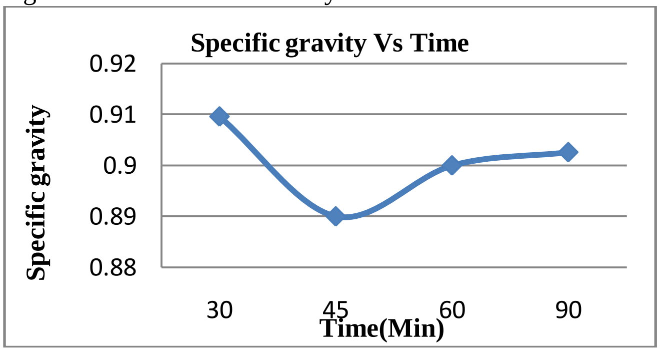


Figure-2: variation of SP.G Vs Reaction Time

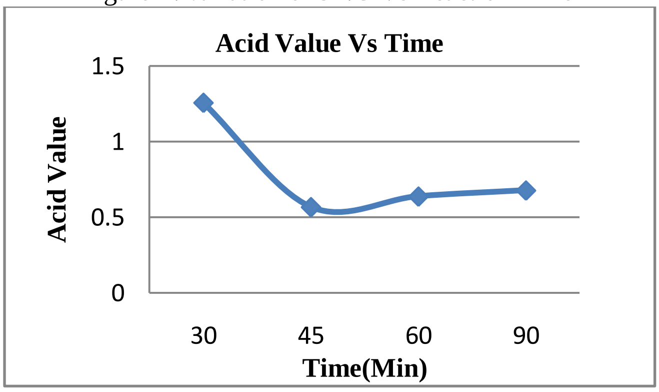


Figure-3: variation of Acid value Vs Reaction Time

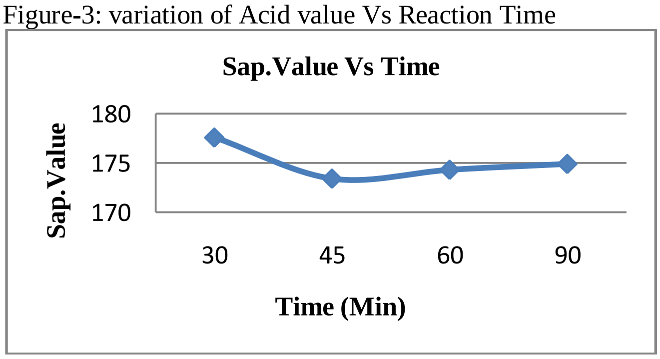


Figure-4: variation of Sap value Vs Reaction Time

Oil to methanol mole ratio

Variation of viscosity with oil to alcohol ratio as shown in table-2. Here oil to methanol ratio is change from 1:6 to 1:12; all the runs were carried at 30^{0}c with batch residence time a 45 minutes. This residence time chosen because optimum value as per earlier discussion. It observed that as oil to alcohol ratio is increases 1:6 to 1:9 mole ratio the product viscosity decreased from 18.18 to14.10 cSt. Further increase oil to alcohol ratio that is 1:12 two layers are not formed. Probably there may not be any reaction at this higher oil to alcohol ratio.

Specific gravity also follows a similar change at oil to alcohol ratio 1:9 the specific gravity of biodiesel layer is found to be lowest 0.882. Acid value and sap value also give similar trend and have lowest value of acid value 0.37 and 170.9 at this oil to alcohol ratio that is 1:9.From the above discussion it appears oil to alcohol ratio 1:9 and residence time of 45 minutes is optimum the parameters for biodiesel production. Viscosity decreases up to 1:9 mole ratios. Specific gravity initially decreases up to 1:9 mole ratio, and further increases up to 1:10 mole ratio and. Acid value shows similar trend as that of viscosity.

Oil to alcohol Mole ratio	1:6	1:9	1:10	1:12
Kinematic viscosity, (cSt)	18.18	14.10	19.84	Nil
Specific Gravity	0.9036	0.8820	0.9112	Nil
Acid value	0.97	0.57	0.84	Nil
Sap value	173.4	176.1	176.8	Nil

Table No.2

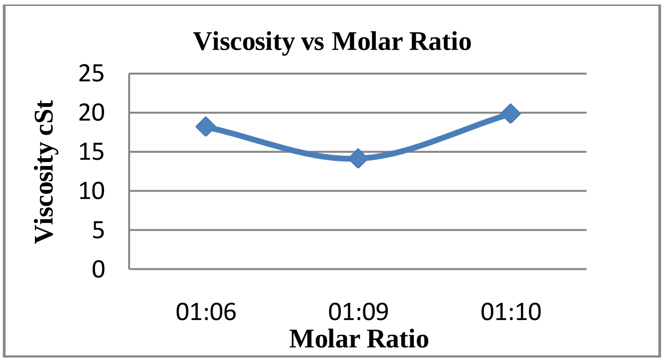


Figure-5 Variation of Viscosity Vs Molar Ratio

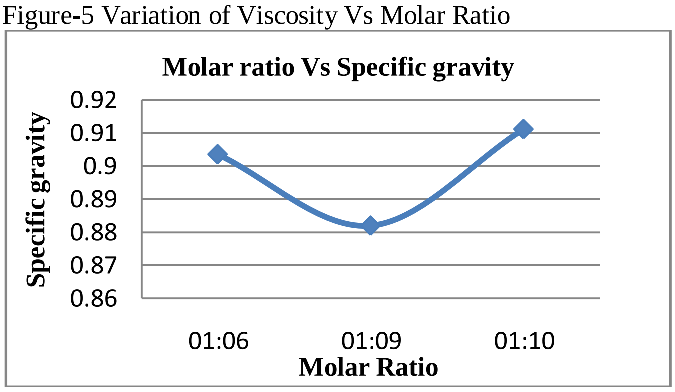


Figure-6 Variation of Molar ratio Vs Specific gravity

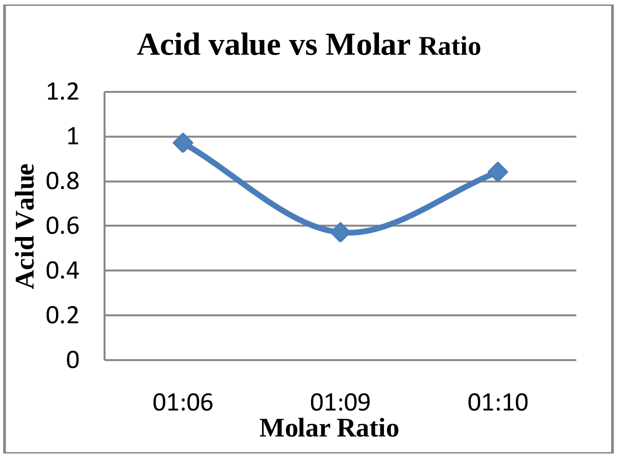


Figure-6 Variation of Acid value Vs Molar Ratio

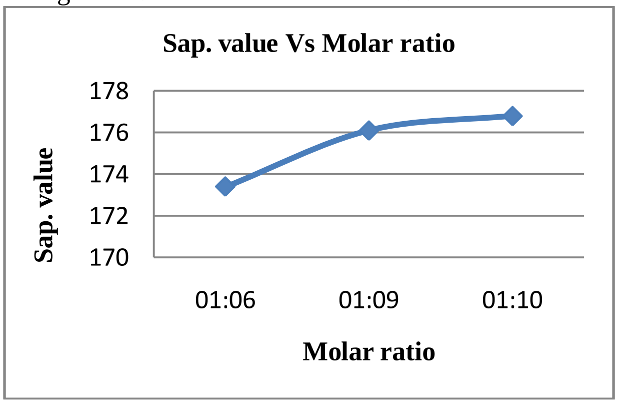


Figure-7 Variation of Sap.value Vs Molar Ratio

Effect of temperature

The effect of temperature on the properties of biodiesel like viscosity, specific gravity, acid value sap value and run time are shown in the table 4.3.3K. Runs were carried out at two temperatures 30 and 50^{0}c maintaining reaction time is 45 minutes. It was observed that at lower temperature that 30^{0}c the biodiesel product gave lower viscosity 14.10 cSt.Other properties like specific gravity, acid value and sap value are lower at 30^{0}c than 40^{0}c. The oil to alcohol ratio chosen for this runs is 1:9, since this is optimum. (Table-2)

Specific gravity increases with increasing temperature, the same changes are observed in acid value. It was observed that at lower temperature that 30^{0}c the biodiesel product gave lower Viscosity and further increases up to 50^{0}c. Acid value shows similar trend as that of viscosity. Sap value shows similar trend as that of specific gravity.

Reaction Temperature(^{0}c)	30	40	50
Kinematic viscosity, (cSt)	14.10	18.06	20.6
Specific gravity	0.890	0.9006	0.9010
Acid value	0.56	0.86	0.91
Sap value	173.4	176.1	176.8

Table No. 3 Variation of Viscosity with Reaction Temperature

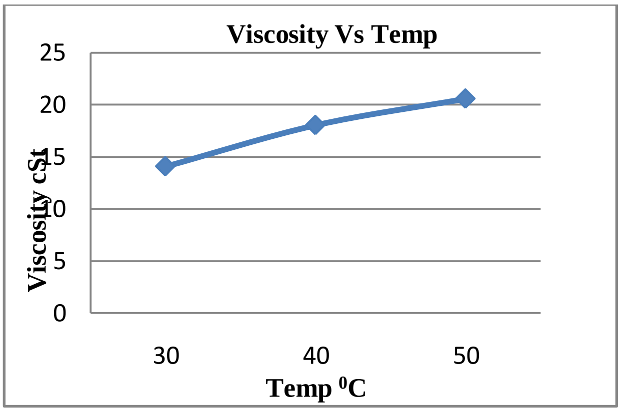


Figure-8Variation of Viscosity with Temperature

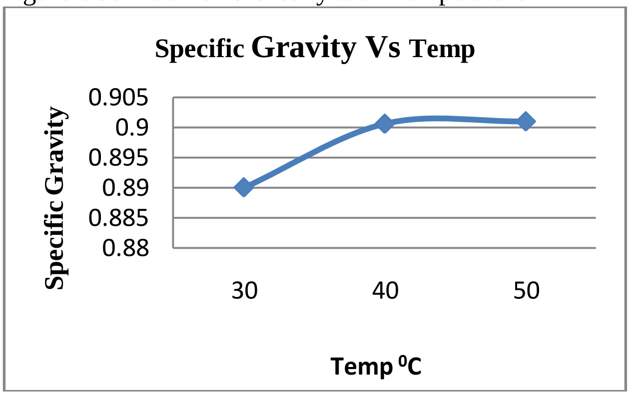


Figure-9 Variation of Specific Gravity with Temperature

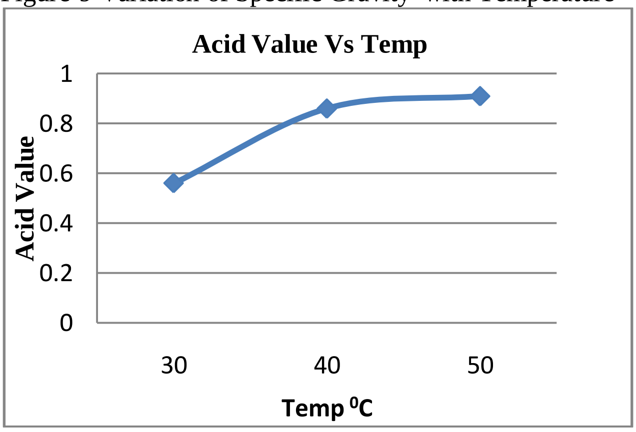


Figure-10 Variation of Acid Value with Temperature

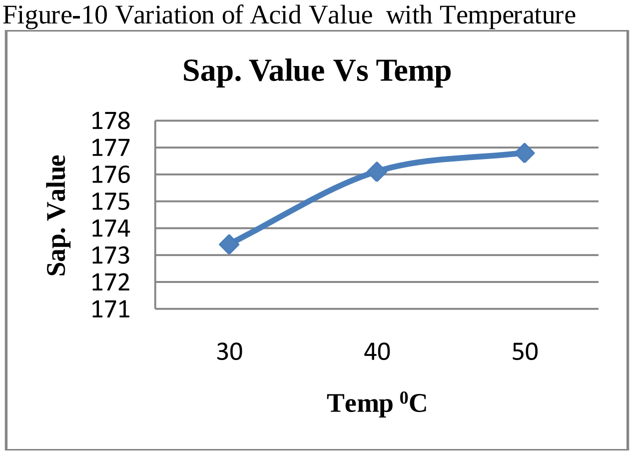


Figure-11Variation of Sap. Value with Temperature

Effect of catalyst concentration

The effect of catalyst concentration on various properties biodiesel are reported in the table 4.3.4K . In these runs catalyst concentration varied from 0.5 to 2 weight % based on amount of oil employed in the reaction. Other experimental conditions like oil to methanol mole ratio 1:9,reaction temperature 30^0c, stirrer speed 460$\pm$10 rpm and run time 45 minutes are kept constant, and catalyst concentration is varied. These variables are selected because they were at optimum, giving biodiesel product of viscosity 14.10 cSt ,where 1 weight % catalyst concentration gave lowest value of viscosity 14.10 cSt.

The specific gravity of the product is lowest at 1 wt % catalyst concentration and increases with increasing catalyst concentration up to 2wt% . The specific gravity of product was increased and also at lowest concentration at 0. 5 wt%, specific gravity was higher. Probably lower catalyst concentration 0. 5 wt % conversions are very low. Therefore from above data, from table 4.4K, 1wt% catalyst concentration may be optimum in the range operating condition studied.Viscosity decreases with initially up to 1 wt. % catalyst concentration and then increases up to 2 wt. catalyst concentration. Specific gravity initially decreases up to 1 wt. % catalyst concentration and further increases. Acid value shows similar trend as that of viscosity. Sap value is practically constant after 1 wt. % catalyst concentration.

Catalyst concentration Weight %	0.5	1	1.5	2.0
Kinematic viscosity, (cSt)	16.48	14.10	16.91	18.36
Specific gravity	0.9028	0.882	0.9017	0.9136
Acid value	0.98	0.57	0.59	0.59
Sap value	175.2	174.6	173.4	173.5

Table-4 Variation of Viscosity with Catalyst Concentration

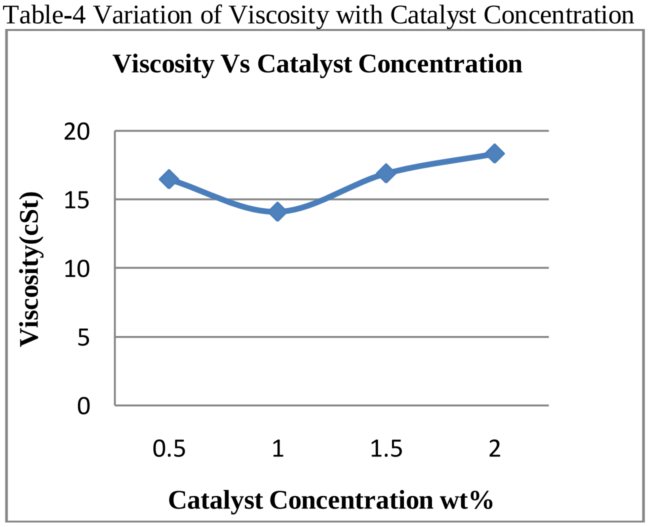
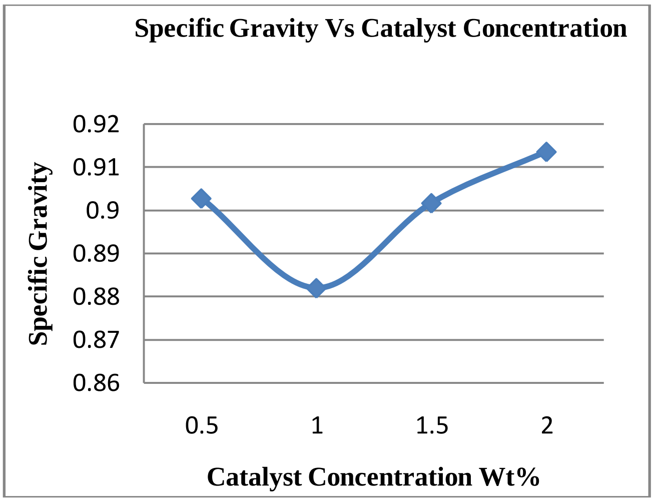


Figure-12Variation of Viscosity with Cat.Conc



. Figure-13 Variation of Sp. Gravity with Cat Conc

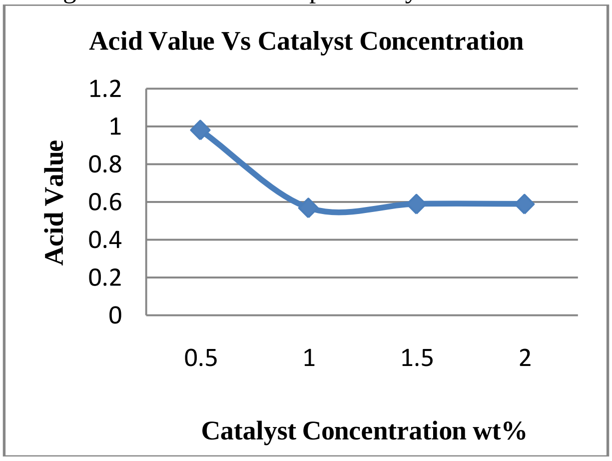


Figure-14 Variation of Acid value with Cat Conc

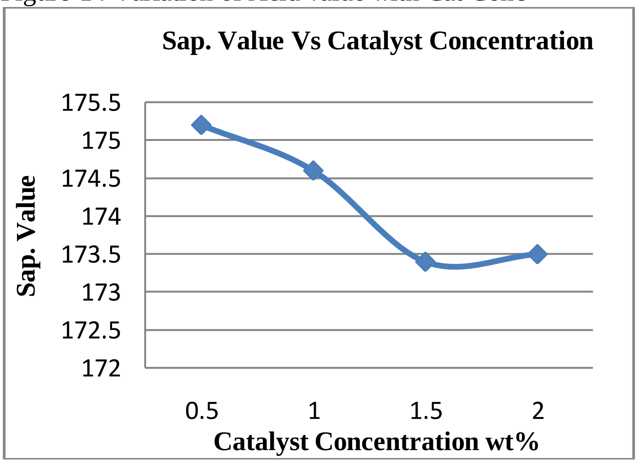


Figure-15Variation of Acid value with Cat Conc

Conclusion

Production of biodiesel by transeterification of castor oil has been studied in an OBR using potassium hydroxide as a catalyst. The variables chosen for the study were Residence time, Oil to methanol ratio, Catalyst concentration; and Reaction Temperature. The effects of these variables on the viscosity of biodiesel were studied, since this is one of the important specifications in ASTM standard. Apart from viscosity other properties like sp.gr, acid value, and sap value were also determined for the biodiesel product. From the above discussion it may be concluded that the optimum operating condition studied in the present work, oil to methanol mole ratio 1:9, temperature =30^{o}c,catalyst concentration 1 wt % and run time 45 min.

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