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The Effects of Trans-Esterification of Castor Seed Oil Using Ethanol, Methanol and their Blends on the Properties and Yields of Biodiesel

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ABSTRACT

The effects of ethanol, methanol and their blends at different percentage mixtures on the properties and yields of biodiesel at varied trans-esterification times and temperatures using sodium hydroxide as a base catalyst have been investigated. At 70^{o}C, the optimum yields were: for ethanol 88.4%, 94.2%, 94.8%, and 95.2% and for methanol, 90.6%, 95.6%, 96.0%, and 96.4% at 1 hour, 2 hours, 3 hours and 4 hours respectively. The biodiesel yields increased as time of reaction progressed for both solvents but the yields obtained from methanol were generally higher than those from ethanol. A mixture of both solvents at 50% each produced the overall highest of biodiesel yield of 98.6% at 70^{o}C and in 4 hours compared to either solvent used alone at the same time and temperature. The properties such as densities, viscosities, flash points and pour points of the biodiesels tested were found to conform to ASTM standards. The average values were as follows: densities at 15^{o}C, were 0.8951, 0.8876 and 0.8832g/cm^3; viscosities (at 40^{o}C) were 4.7160cSt, 4.7380cSt and 4.5055cSt; flash points were 140.9^{o}C, 147.4^{o}C and 161.6^{o}C while for pour points they were -2.4375^{o}C, -1.6875^{o}C and -6^{o}C for ethyl, methyl and ethyl/methyl biodiesel respectively.

Keywords: *Trans-esterification, Castor oil, Solvents blends, Biodiesel yields, Effect of temperature and time.*

1. INTRODUCTION

Most of the world's energy needs are supplied through fossil fuel sources (petroleum, coal and natural gas).[1] With the current environmental concerns posed by fossil fuels and their limited availability, the search for alternative energy such as hydroelectricity, biomass, wind, nuclear, geothermal, hydrogen and solar energy is on the rise. And biodiesel has attracted keen attention from numerous researchers all around the world as a possible source of alternative and renewable energy to replace or complement petro-diesel. Biodiesel is renewable and does not contribute to global warming due to its closed carbon cycle. [2] It is non-toxic, biodegradable and an excellent substitute for petroleum based diesel fuel. [3] Biodiesel can be produced by the reaction (trans-esterification) of a vegetable oil or animal fat with a low molecular weight alcohol in the presence of a strong acid or base as catalyst, this produces a mixture of fatty acid alkyl esters (biodiesel) and glycerol. [1] Although, Alkali catalyzed trans-esterification has been most frequently used industrially, mainly due to its fast reaction rate. [4, 5] The direct use of vegetable oils in fuel engines is problematic. Due to their high viscosity (about 11–17 times higher than diesel fuel) and low volatility, they do not burn completely and form deposits in the fuel injector of diesel engines.[6] There were problems of coking, carbon deposits due to incomplete combustion, oil ring sticking, thickening, lower volatilities, incorrect vapourization characteristics, lubricating problems and high viscosity.[1] This is why vegetable oils have to undergo trans-esterification process to overcome these problems and to make them useable and compatible with diesel engines. Most oil bearing seeds can serve as feedstock for biodiesel production. Some of which include castor, jatropha, soybean, canola, waste vegetable oil, etc. Trans-esterification involves the conversion of the oils to their alkyl esters by reaction with alcohol. And it has been the most commonly used method for the production of biodiesel. Ethanol and methanol have been the common alcohols used in the trans-esterification of these oils to biodiesel in the presence a base or an acid catalyst. Unfortunately, not much research has been conducted at investigating the combined effect of both alcohols in the trans-esterification of vegetable oils into biodiesel. Different vegetable oils especially those common to a particular locality have been used. Rapeseed oil for instance, is the predominant feedstock for biodiesel production in the European Union (EU) while soybean oil is predominant in the United States [7] and Canada. However, the use of oils from edible food crops for the production of biodiesel is discouraged as shortages may arise in the long run. Developing countries like Nigeria cannot afford to use edible oils to produce biodiesel as it would lead to acute food shortages. This is because larger population of her rural dwellers is mere subsistence farmers that still depend on hoe and cutlass rather than mechanized farming. Nigeria is blessed with a host of non-edible oils such as rubber seed, jatropha and castor oils which should be exploited. Hence castor oil was chosen as feedstock because it is non-edible and would not compete with food crops alleviating concerns about food shortages that may arise from the production of biodiesel from food crops. The oil content of castor seed has been found to be between 46-55% by weight [8] and is relatively high compared to soybean (20%) and rapeseed (40%).[9] Castor seed (Ricinus communi) is a species that belongs to the Euphorbiaceae family. The common names are castor oil plant, and Palma Christi. It has its origin in Africa but can be found in tropical and subtropical countries of the world. The castor plant is drought-resistant and can thrive in arid conditions. [10] It is non-edible as it contains ricin, a poisonous protein substance. Castor oil also known as or ricinus oil [11] contains approximately 90% of ricinoleic acid, [12] which makes the oil to be readily solubility in alcohol at room temperature. [13,14] Whereas, other vegetable oils have different compositions of fatty acids and are usually insoluble in alcohol at room temperatures, castor oil dissolves easily in alcohol, ether, glacial acetic acid, chloroform, carbon sulfide, and benzene. [8, 15] Castor oil is viscous, pale yellow, non-volatile and non-drying oil with mild odour and a bland taste. [9, 11] It has a boiling point of 313°C and has a density of 961 kg/m^3. [16] The castor biodiesel has very interesting properties (very low cloud and pour points) that show that this fuel is very suitable for use in extreme low temperatures (e.g. winter).

2. METHODOLOGY

2.1 Instrument used: Anton paar density meter DMA 38, Thermostatic viscosity bath, Automated Pensky-Martens closed cup flash point tester, Pour point bath,

2.2 Apparatus used: Hot plate magnetic stirrer, retort stands, burette, pipette, 500mL separating funnel, condensation unit, thermometer, 50mL measuring cylinder, 500mL three-neck flat bottom flask, 250mL and 500mL beakers, 250ml conical flask and water hose, were provided by the Department of Chemical Engineering, Covenant University, Ota Nigeria.

2.3 Reagents used: Castor oil, ethanol, methanol, hydrochloric acid, sodium hydroxide pellets, were purchased from Angos Nigeria Limited, Lagos state, Nigeria. The distilled water was provided by Chemical Engineering Department, Covenant University, Ota Nigeria. All experiments were carried out in the Chemical Engineering laboratory, Covenant University, Ota, Nigeria.

2.4 Procedure

2.4.1 Experimental Set-up

The 500mL three-neck flat bottom flask (the reactor) was set-up with the middle opening (or neck) carrying a reflux condensation unit, one of the two side arms carried the thermometer and the second side arm carried a ground stopper which served as feed point to the reactor. The set-up was mounted on the hot plate magnetic stirrer. The condenser was in-turn connected to a tap for in- and out-flow of cold water to prevent loss of alcohol during the trans-esterification reactions.

2.4.2 Trans-Esterification of the Castor Oil

This was divided in to three parts namely; Trans-esterification of the oil using ethanol, methanol and then a combination of the two alcohols at predetermined ratios. 46.5g of ethanol (absolute) and 50.0g of the oil were first measured in two separate beakers that have been previously washed and dried. 0.5g (1% by weight) of the catalyst (pure potassium hydroxide) was weighed and dissolved in the ethanol and the resultant solution was then charged into the reactor and pre-warmed. The stirrer speed was set to 4rps (revolution per second) and the oil which was also pre-warmed in a separate beaker was carefully added to the sodium ethoxide in the reactor and was then subjected to heating at different temperatures and varied times of trans-esterification reactions. At the end of the reaction, the reaction mixture was poured into a separating funnel and left to stand for approximately 24hours, this was to allow for distinct phase separation. The phases were then separated to recover the biodiesel for further purification and drying.

The above procedure was repeated but instead of ethanol, methanol and a combination of ethanol-methanol mixtures were used.

3. RESULTS AND DISCUSSION OF RESULTS

3.1 Effect of Temperature and Time on Biodiesel Yield

Trans-esterification reactions were carried out at varying temperatures of 55^{o}C, 60^{o}C, 65^{o}C and 70^{o}C using methanol and ethanol and a combination of the two alcohols. Figures 3.1, 3.2 and 3.3 illustrate the effect of temperature on the yield of biodiesel. For trans-esterification with ethanol the yield of biodiesel progressively increased with temperature as the time was held constant. At constant time of 1 hour, the biodiesel yield was 64% at 55^{o}C, 80% at 60^{o}C, 86% at 65^{o}C, and 88.4% at 70^{o}C. The biodiesel yield also increased as the time of reaction increased at the same temperatures investigated. At constant time of 2 hours, the biodiesel yield was 75% at 55^{o}C, 85.6% at 60^{o}C, 89.6% at 65^{o}C, and 94.2% at 70^{o}C. Furthermore, at constant time of 3 hours, the biodiesel yield was 88.4% at 55^{o}C, 89.6% at 60^{o}C, 92% at 65^{o}C, and 94.8% at 70^{o}C whereas, at constant time of 4 hours, the biodiesel yield was 85.2% at 55^{o}C, 91.8% at 60^{o}C, 94% at 65^{o}C, and 95.2% at 70^{o}C. Exactly the same trend was also observed for trans-esterification of castor oil with methanol except that the bio-diesel yields generally were higher for methanol than with ethanol at the same reaction times and temperatures considered. The highest percentage yield of biodiesel was 96.2% with methanol trans-esterification compared to 95.2% obtained from ethanol trans-esterification in 4 hours and at 70 ^{o}C. The converse is true when trans-esterification times were varied and temperatures kept constant.

For the ethanol-methanol blends different percentage ratios, 0-100, 30-70, 40-60, 50-50, 60-40, 70-30 and 100-0 were used for the trans-esterification reactions. The optimum reaction conditions of 4 hours and temperature of 70^{o}C were used. Figure 3.4 and table 3.9 show the yield of biodiesel from their various blends used. The yield of biodiesel increased progressively from 96.4%, at 0:100 ethanol: methanol blend, reaching a maximum of 99.6%, at 50:50 ethanol: methanol blend before it began to decrease steadily to a value of 95.2% at 100:0 ethanol: methanol blend. This gives an indication that blending of alcohols results in higher biodiesel yield. From Tables 3.1 to 3.9, the densities of bio-diesel at 15^{o}C range from 0.8803- 0.8997g/cm^3 which conform to ASTM standard of 0.88-0.9g/cm^3. The densities of the biodiesel produced from ethanol were within the range of 0.8909-0.8997 with an average value of 0.8951, the biodiesel produced from methanol had density values within the range 0.8845-0.8898 with an average value of 0.8876 while the biodiesel produced from the ethanol-methanol blend had density values within the range 0.8823-0.8841. From the results, it is clear that the density produced from the ethanol-methanol blend had the lowest density.

Viscosity values (at 40^{o}C) of all the biodiesel produced were within the range 4.2909-5.2189cSt which conform to ASTM standard of 1.9-6.0cSt. The viscosity of the biodiesel produced form ethanol was within the range 4.2909-5.0094cSt with an average value of 4.7160cSt, while that of the biodiesel produced from methanol was within the range 4.2909-5.2189cSt with an average value of 4.7380cSt and the viscosity of the biodiesel produced from the ethanol-methanol blend was within the range 4.2909-4.7614cSt with an average value of 4.5055cSt. Flash point values of the biodiesel was within the range 131-168^{o}C which conform to ASTM standard (>130^{o}C). The flash point of the biodiesel produced form ethanol was within the range 131-148^{o}C with an average value of 140.9^{o}C, the flash point of the biodiesel produced from methanol was within the range 131-158^{o}C with an average value of 147.4^{o}C while the flash point of the biodiesel produced from the ethanol-methanol blend was within the range 155-168^{o}C with an average value of 161.6^{o}C. Pour point values of the biodiesel were within the range -6-0^{o}C which also conform to ASTM standard. The pour point of the biodiesel produced from ethanol was within the range -6-0^{o}C with an average value of -2.4375^{o}C, while the pour point of the biodiesel produced from methanol was within the range -3-0^{o}C with an average value of -1.6875^{o}C and the pour point of the biodiesel produced from the ethanol-methanol blend was -6^{o}C.

4. CONCLUSION

The rate of trans-esterification of castor oil increased with reaction time and temperature. Higher yields were obtained from the trans-esterification of castor oil with methanol compared to ethanol. However, its trans-esterification with blends of ethanol and methanol gave higher yields compared to the yields obtained from both alcohols used alone. Equal percentage blend of 50: 50, ethanol: methanol, gave the highest yield of 99.6%. The pour point, flash point, density at 15^{o}C and viscosity at 40^{o}C of the biodiesel produced were within the allowable limits set by the American Society for Testing and Materials (ASTM) making castor oil a suitable feedstock for high quality biodiesel. The use of biodiesel as fuel provides useful solutions to various environmental problems and will go a long way to ameliorating impending energy crises. It is renewable in nature, biodegradable, and will reduce emissions of green house gases. The use of castor oil as biodiesel feed stock should be greatly encourage because it is a non-edible crop and would not compete with acreage dedicated for food crops.

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Table 3.1: Results of Trans-esterification with Ethanol 55^{o}C

Sample Name	Raw materials			Conditions		Products		Biodiesel Properties			
	Castor Oil (g)	Ethanol (g)	NaOH (g)	T (^{0}C)	t (hrs)	Biodiesel (g)	Biodiesel Yield (%)	Density at 15^{o}C (g/cm^3)	Viscosity at 40^{o}C (Cst)	Flash Point (^{0}C)	Pour Point (^{0}C)
A1	50	46.5	0.5	55	1	32.0	64.0	0.8993	4.6103	140	-3
A2	50	46.5	0.5	55	2	37.5	75.0	0.8965	4.5289	147	-6
A3	50	46.5	0.5	55	3	40.2	80.4	0.8959	4.2909	139	-3
A4	50	46.5	0.5	55	4	42.6	85.2	0.8997	4.7214	142	0

Table 3.2: Results of Trans-esterification with Ethanol 60^{o}C

Sample Name	Raw Materials			Conditions		Products		Biodiesel Properties			
	Castor Oil (g)	Ethanol (g)	NaOH (g)	T (^{0}C)	t (hrs)	Biodiesel (g)	Biodiesel Yield (%)	Density at 15^{o}C (g/cm^3)	Viscosity at 40^{o}C (Cst)	Flash Point (^{0}C)	Pour Point (^{0}C)
A5	50	46.5	0.5	60	1	40.0	80.0	0.8963	4.9980	145	-3
A6	50	46.5	0.5	60	2	42.8	85.6	0.8924	4.6953	132	0
A7	50	46.5	0.5	60	3	44.8	89.6	0.8947	4.9904	148	-6
A8	50	46.5	0.5	60	4	45.9	91.8	0.8974	5.0094	140	-3

Table 3.3: Results of Transesterification with Ethanol 65^{o}C

Sample Name	Raw materials			Conditions		Products		Biodiesel properties			
	Castor Oil (g)	Ethanol (g)	NaOH (g)	T (^{0}C)	t (hrs)	Biodiesel (g)	Biodiesel Yield (%)	Density at 15^{o}C (g/cm^3)	Viscosity at 40^{o}C	Flash Point (^{0}C)	Pour Point (^{0}C)

									(Cst)		
A9	50	46.5	0.5	65	1	43.0	86.0	0.8917	4.7669	138	-6
A10	50	46.5	0.5	65	2	44.8	89.6	0.894	4.8495	142	-3
A11	50	46.5	0.5	65	3	46.0	92.0	0.8938	4.6500	147	0
A12	50	46.5	0.5	65	4	47.3	94.6	0.8909	4.5306	139	-3

Table 3.4: Results of Transesterification with Ethanol 70°C

Sample Name	Raw materials			Conditions		Products		Biodiesel properties			
	Castor Oil (g)	Ethanol (g)	NaOH (g)	T (^{0}C)	t (hrs)	Biodiesel (g)	Biodiesel Yield (%)	Density at 15°C (g/cm^3)	Viscosity at 40°C (Cst)	Flash Point (^{0}C)	Pour Point (^{0}C)
A13	50	46.5	0.5	70	1	44.2	88.4	0.8914	4.3211	131	0
A14	50	46.5	0.5	70	2	47.1	94.2	0.8965	4.6024	140	-3
A15	50	46.5	0.5	70	3	47.4	94.8	0.8991	4.8854	146	0
A16	50	46.5	0.5	70	4	47.6	95.2	0.8928	5.0049	138	0

Table 3.5: Results of Trans-esterification with Methanol 55°C

Sample Name	Raw materials			Conditions		Products		Biodiesel properties			
	Castor Oil (g)	Methanol (g)	NaOH (g)	T (^{0}C)	t (hrs)	Biodiesel (g)	Biodiesel Yield (%)	Density at 15°C (g/cm^3)	Viscosity at 40°C (Cst)	Flash Point (^{0}C)	Pour Point (^{0}C)
B1	50	32.4	0.5	55	1	35.0	70.0	0.8893	4.2909	139	0
B2	50	32.4	0.5	55	2	39.1	78.2	0.8865	4.3575	148	-3
B3	50	32.4	0.5	55	3	41.0	82.0	0.8859	4.5786	151	-3
B4	50	32.4	0.5	55	4	44.2	88.4	0.8897	4.5570	156	-3

Table 3.6: Results of Trans-esterification with Methanol 60°C

Sample Name	Raw materials			Conditions		Products		Biodiesel properties			
	Castor Oil (g)	Methanol (g)	NaOH (g)	T (^{0}C)	t (hrs)	Biodiesel (g)	Biodiesel Yield (%)	Density at 15°C (g/cm^3)	Viscosity at 40°C (Cst)	Flash Point (^{0}C)	Pour Point (^{0}C)
B5	50	32.4	0.5	60	1	41.4	82.8	0.8863	4.9140	131	-3
B6	50	32.4	0.5	60	2	43.6	87.2	0.8854	5.2189	142	0
B7	50	32.4	0.5	60	3	45.0	90.0	0.8887	4.4561	154	-3
B8	50	32.4	0.5	60	4	47.0	94.0	0.8874	4.5786	150	0

Table 3.7: Results of Trans-esterification with Methanol 65°C

Sample Name	Raw materials			Conditions		Products		Biodiesel properties			
	Castor	Methano	NaOH	T	t	Biodiesel	Biodiesel	Density	Viscosity at	Flash	Pour

	Oil (g)	l (g)	(g)	(^{0}C)	(hrs)	(g)	Yield (%)	at 15^{o}C (g/cm^3)	40^{o}C (Cst)	Point (^{0}C)	Point (^{0}C)
B9	50	32.4	0.5	65	1	44.3	88.6	0.8897	5.1022	138	0
B10	50	32.4	0.5	65	2	45.9	91.8	0.8869	4.9823	149	-3
B11	50	32.4	0.5	65	3	47.3	94.6	0.8898	4.4987	158	0
B12	50	32.4	0.5	65	4	47.5	95.0	0.8872	4.9613	146	0

Table 3.8: Results of Trans-esterification with Methanol 70^{o}C

Sample Name	Raw materials			Conditions		Products		Biodiesel properties			
	Castor Oil (g)	Methanol (g)	NaOH (g)	T (^{0}C)	t (hrs)	Biodiesel (g)	Biodiesel Yield (%)	Density at 15^{o}C (g/cm^3)	Viscosity at 40^{o}C (Cst)	Flash Point (^{0}C)	Pour Point (^{0}C)
B13	50	32.4	0.5	70	1	45.3	90.6	0.8868	5.1691	158	0
B14	50	32.4	0.5	70	2	47.8	95.6	0.8845	4.7733	140	-3
B15	50	32.4	0.5	70	3	48.0	96.0	0.8895	4.6070	147	-3
B16	50	32.4	0.5	70	4	48.2	96.4	0.8876	4.7624	152	-3

Table 3.9: Results of Trans-esterification with Methanol 70^{o}C

Sample Name	Raw materials				Conditions		Products		Biodiesel properties			
	Castor Oil (g)	Ethanol (g)	Methanol (g)	NaOH (g)	T (^{0}C)	t (hrs)	Biodiesel (g)	Biodiesel Yield (%)	Density at 15^{o}C (g/cm^3)	Viscosity at 40^{o}C (Cst)	Flash Point (^{0}C)	Pour Point (^{0}C)
C0	50	0	32.4	0.5	70	4	48.2	96.4	0.8876	4.7624	152	-3
C1	50	13.95	22.68	0.5	70	4	48.4	96.8	0.8803	4.4194	159	-6
C2	50	18.6	19.44	0.5	70	4	49.4	98.8	0.8824	4.4792	168	-6
C3	50	23.25	16.2	0.5	70	4	49.8	99.6	0.8818	4.7614	155	-6
C4	50	27.9	12.96	0.5	70	4	48.1	96.2	0.8841	4.3602	164	-6
C5	50	32.55	9.72	0.5	70	4	47.8	95.6	0.8831	4.5075	162	-6
C6	50	46.5	0	0.5	70	4	47.6	95.2	0.8928	5.0049	138	0

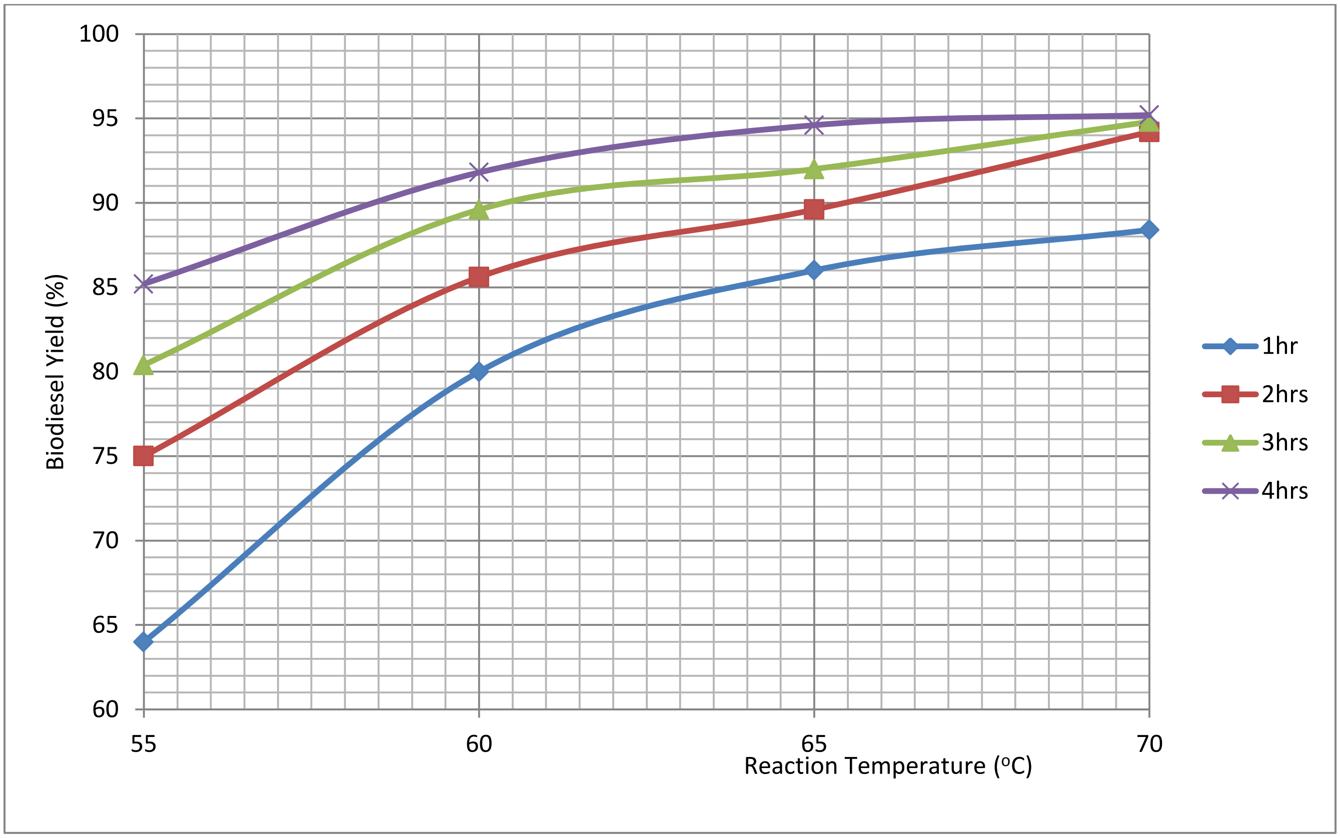

Fig.3.1: Ethyl bio-diesel yield at varied reaction temperatures and fixed times

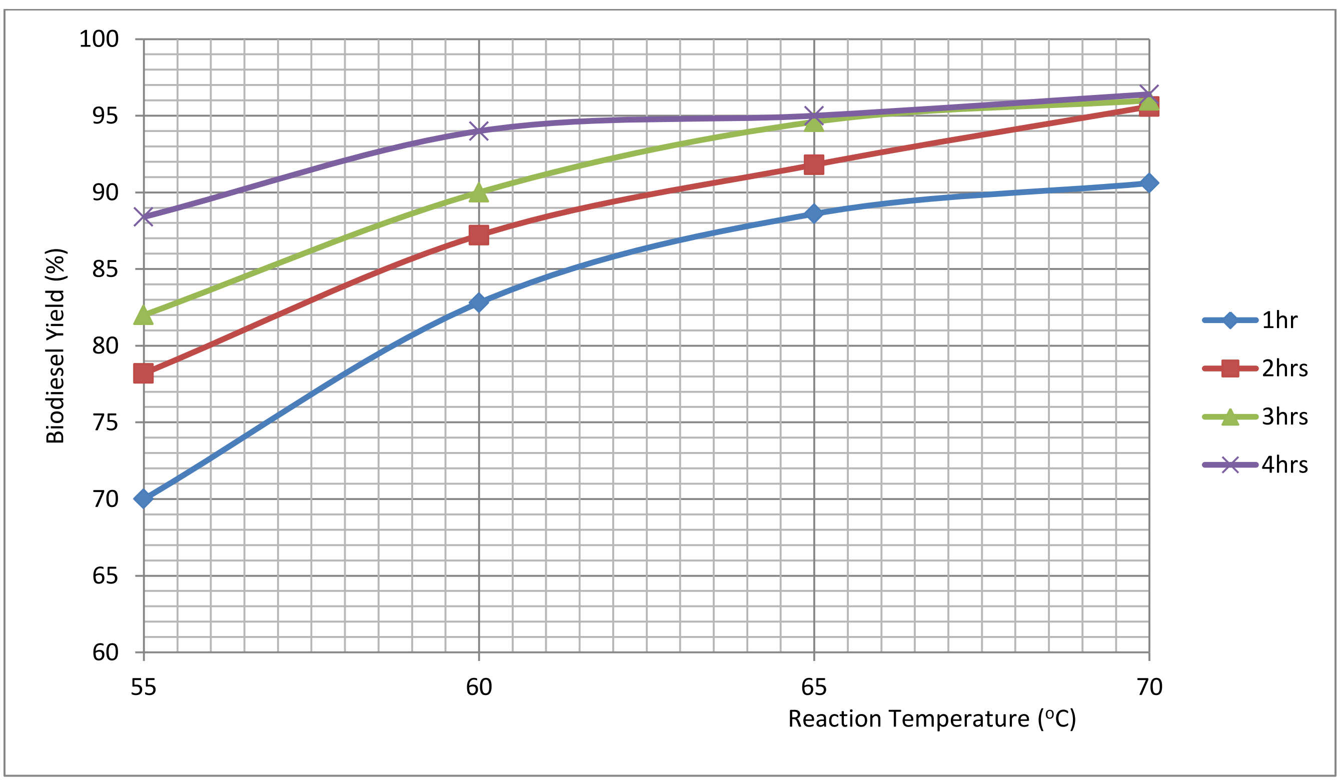

Fig.3.2: Methyl bio-diesel yield at varied reaction temperatures and fixed times

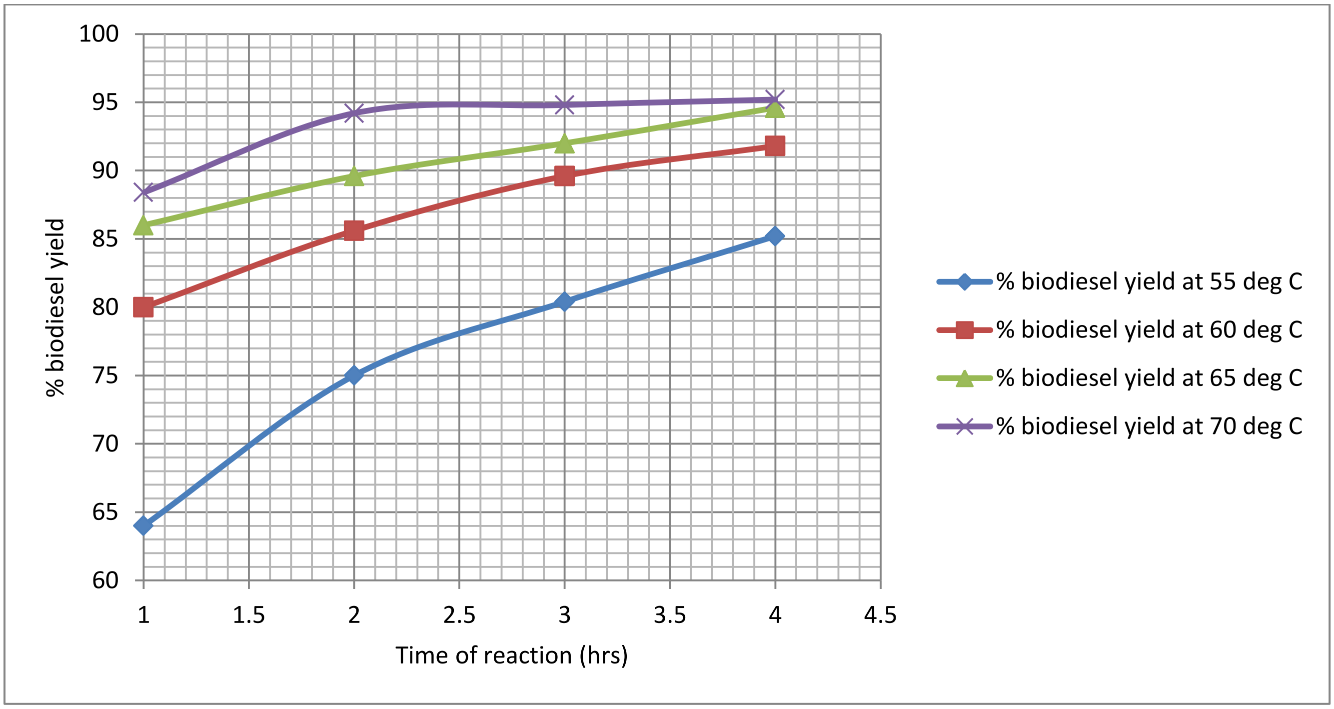

Fig.3.3: Ethyl bio-diesel yield at varied reaction times and fixed temperatures

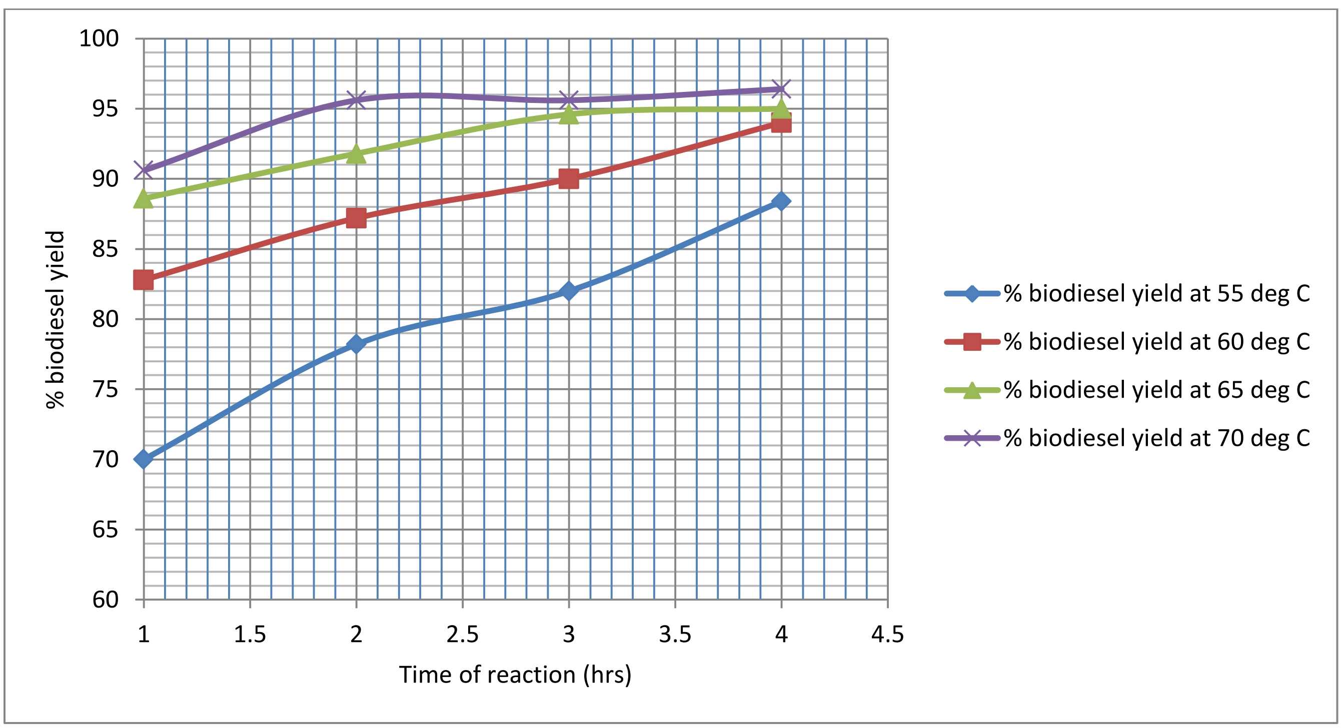

Fig.3.4: Methyl bio-diesel yield at varied reaction times and fixed temperatures

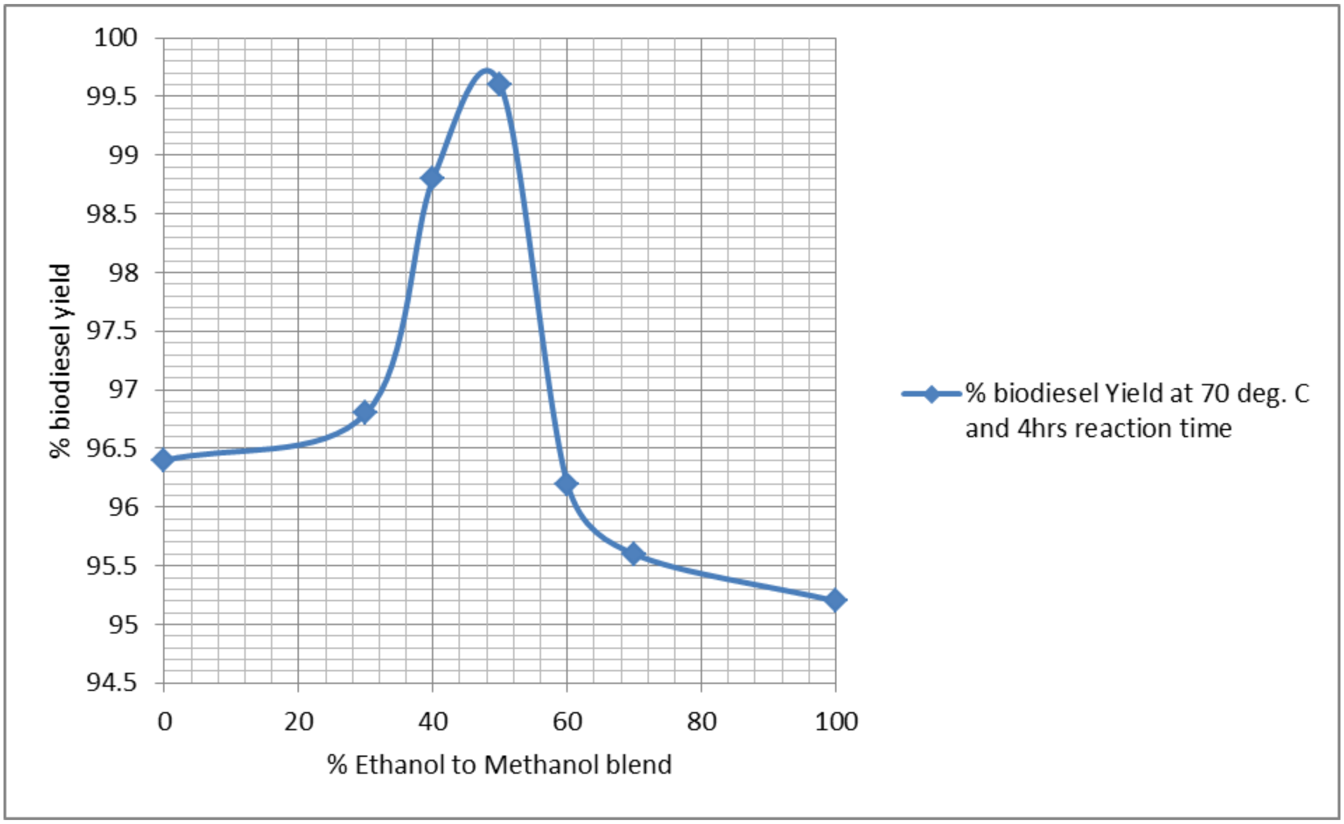

Fig.3.5: Effect of Ethanol: Methanol Blend on Percentage Bio-Diesel Yield at 70^{o}c and 4 Hours Reaction Time