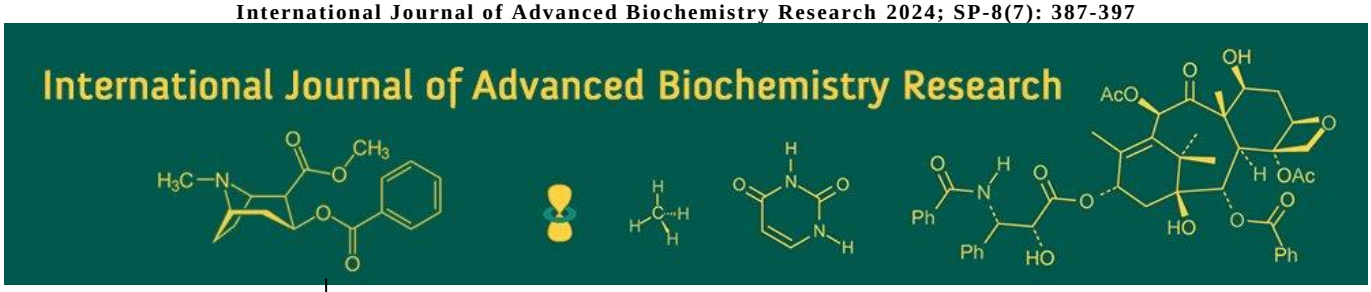

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Investigations into methanol recovery from biodiesel in a batch-type production facility

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Abstract
The amount of unused methanol extracted, the volume of the reaction tank, and the power required to agitate the products of the transesterification reaction were extrapolated for 500ℓ/day (125ℓ/batch) and 1000ℓ/day (250ℓ/batch) biodiesel plants. Three dependent variables, namely the amount of methanol recovered, the time required, and the energy consumed, and one independent variable, the volume of biodiesel to be produced, were selected for the extrapolation. Graphs were plotted and an equation with the highest goodness of fit (R^2) value was adopted. The predicted amounts of unused methanol to be recovered for 125ℓ and 250ℓ batch capacities of the batch-type biodiesel plant were found to be 10,684.19 g and 21,299.06 g, respectively. The time required and energy consumed during methanol recovery for the 125ℓ batch capacity was found to be 1,590.84 minutes and 36.02 kWh, respectively. In contrast, for the 250ℓ capacity, their values were found to be 3,251.22 minutes and 73.17 kWh, respectively. The volumes of the reaction chamber, having a cylindrical shape, for producing 125ℓ and 250ℓ of biodiesel per batch were calculated and found to be 0.29 m^3 and 0.65 m^3, respectively. The power required for agitating the mixture of end products of the transesterification reaction during the production of 125ℓ and 250ℓ of biodiesel per batch was found to be 1.94 kW and 6.18 kW, respectively.

Keywords: Unused methanol, biodiesel, transesterification

1. Introduction
Energy is the backbone of all research and inventions, ensuring technologies remain in motion and contributing to a sedentary and ease-loving lifestyle for humans. This energy cannot be created but can be transformed into other forms. The existing energy is found in our surroundings in various forms like petroleum products, wind, solar, etc.
Petroleum products are the most consumable sources for generating energy. The global petroleum oil and natural gas liquid reserves were reported as 1,573 billion barrels [1]. The global demand for crude petroleum oil in 2024 is forecasted to average around 102.9 million barrels per day (mb/d) and represents a modest increase from the previous year, [2]. However, this consumption results in the release of harmful gases and pollutants, causing the following changes in the ecosystem:
1. Disruptions to the global climate
2. Depletion of stratospheric ozone
3. Irreversible losses of biological diversity
4. Changes in the structure and functioning of ecosystems around the world [3]

To protect the environment from impending danger and to preserve what our forefathers left for us, there is an urgent need to adopt environmentally friendly techniques to produce power. One such alternative is biodiesel. As the name suggests, biodiesel is derived from biologically produced derivatives and has physio-chemical properties similar to diesel. Biodiesel can be produced from vegetable oil by reacting it with a super-stoichiometric amount of alcohol in the presence of a catalyst. The end products of this transesterification reaction are biodiesel (alkyl ester of vegetable oil), glycerol, and unused alcohol.
If this unused methanol remains in biodiesel, it has detrimental effects on the environment and various components of internal combustion engines. Excess alcohol can deteriorate natural rubber seals and gaskets of the engine, and corrode materials made of aluminium and

zinc. It lowers the flash point of biodiesel, making it difficult to handle. It also reduces the viscosity, density, and cetane number of biodiesels. Therefore, there is an urgent need to remove alcohol from biodiesel and the glycerol-rich phase. This forms the basis of the research conducted.

2. Materials and Methods

2.1 Selection and Determination of Composition of Vegetable Oil

Palm oil was used for producing biodiesel. The Gas Chromatography technique was utilized to determine the composition of palm oil. The results of the Gas Chromatography analysis are shown in Table 1.

Table 1: Composition of Palm Oil Used as a Reactant

S. No.	Fatty Acid	Percentage (by mass)
1	Palmitic Acid	33.73
2	Stearic Acid	4.0
3	Oleic Acid	50.0
4	Linoleic Acid	2.76
5	Myristic Acid	2.5
6	Others	7.0

2.2 Batch Type Biodiesel Plant

A batch-type biodiesel plant unit consists of a mixing tank with a mode of heat generation such as an electric heater or fluid jacket to carry out the reaction. An impeller is used for proper churning and mixing of the reactants and products obtained. Settling tanks are used to store the products of the reaction for a certain period to separate them from each other through the gravimetric method, and a washing tank is used to wash one of the products obtained after the completion of the reaction.

A batch-type biodiesel plant with a maximum capacity of 100ℓ/day (25ℓ/batch) was selected for carrying out the production of biodiesel. An electric heater with a power rating of 1.0 kW was used for heating the fluid inside the reaction chamber.

2.3 Preparation of Palm Oil Ester

Palm oil was reacted with methanol to produce its methyl ester using a methanol to palm oil molar ratio of 6:1. The catalyst (KOH) concentration was taken as 1% w/w of palm oil. The reaction temperature was maintained at 65 °C for 60 minutes. The reaction was carried out at 1.0 atm pressure. Different volumes of untreated biodiesel (without methanol recovery) viz. 10, 12.5, 15, 17.5, and 25ℓ were produced. The transesterification reaction can be represented as:

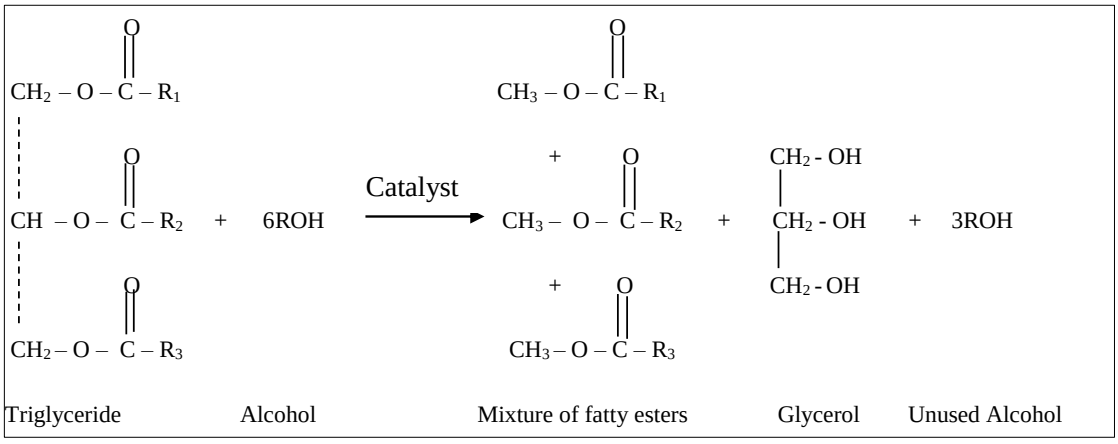

Fig 1: Transesterification of palm oil

2.4. Methanol recovery

During the methanol recovery process, the temperature of end products was increased from 65 °C to 90 °C. Unused methanol was recovered from the mixture of end products obtained during the transesterification reaction which is a mixture of biodiesel, glycerol and unused methanol while producing different volumes of treated biodiesel (methanol recovered) namely 10, 12.5, 15, 17.5 and 25ℓ by using a rotary vacuum flask evaporator.

Three graphs have been plotted viz. temperature rise vs. time, rate of volume of methanol recovered vs. time and cumulative methanol recovery vs. time to determine the effect of energy consumed, time interval and capacity of the condenser on methanol recovery.

2.5. Purity of methanol

The recovered methanol was tested to its level of purity by comparing it with the peak obtained for pure methanol by using the Gas Chromatography technique. The amount of methanol that remained in untreated and treated biodiesel was also analysed through a Gas Chromatograph.

2.6. Mathematical modelling

Mathematical models namely duration of methanol recovery, energy consumed thereof and amount of methanol recovered were prepared by using the results obtained during methanol recovery while producing different volumes of treated biodiesel viz. 10, 12.5, 15, 17.5 and 25ℓ. The equation was developed with the highest value of goodness of fit (R^2) for each model, which forms the basis for predicting the amount of methanol that could be recovered, the time required to obtain that amount and the energy consumed thereof for higher capacity biodiesel plant namely 500 ℓ /d(125ℓ/batch) and 1000ℓ/d(250ℓ/batch).

2.7. Scale up

The volume of the mixing chamber and the power required to agitate the fluids inside it were scaled up for 125ℓ/batch and 250ℓ/batch of biodiesel production in a batch-type biodiesel plant.

3. Results and Discussion

Table 2(a) and (b) show methanol recovery for 10, 12.5, 15, 17.5 and 25ℓ of biodiesel production. Graphs were plotted

between different dependent variables namely temperature rise, rate of quantity of methanol recovered cumulative methanol recovery and one independent variable, time.

3.1 Temperature Rise vs. Time

The temperature vs. time graph indicated in Fig 2 shows the rate of increase of temperature of products with respect to time. The temperature of products obtained after the transesterification reaction for different volumes of biodiesel to be produced was increased from 65 °C to 90 °C. Before reaching 90 °C, temperature was almost directly proportional to the increase in time irrespective of the volume of biodiesel to be produced.

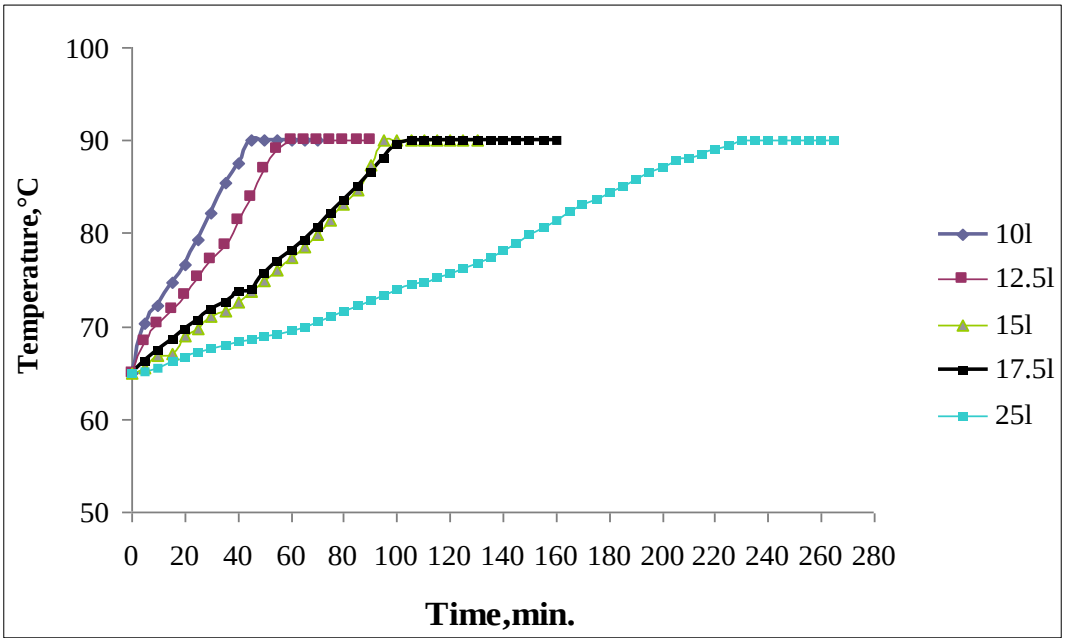

Fig 2: Rate of increase of temperature during methanol recovery

The time taken to reach 90 °C was found to be directly proportional to the different volumes considered. Thus, during methanol recovery from the end products of the transesterification reaction, the time taken to reach 90 °C from 65 °C was 45, 60, 95, 105 and 235 min. for 10, 12.5, 15, 17.5 and 25ℓ of biodiesel production respectively.

The rate of increase of temperature decreases as the volume of products to be heated increases till reaching 90 °C. For 15 and 17.5ℓ, the slope remains constant because the rate at which energy is transferred becomes constant. This meant that for volumes ranging from 15 to 17.5 ℓ, the rate of increase of temperature is independent of the volume considered.

There is an increase in duration at 90 °C with the increase in volume of products because a large amount of methanol was still to be recovered. This duration was found to be 25, 30, 40 and 60 min. during production of 10, 12.5, 15 and 17.5ℓ biodiesel, respectively. The longest duration occurred for 17.5 l because a large amount of methanol was still to be recovered. However, for 25ℓ, the duration was decreased because till reaching 90 °C a large proportion of unused methanol was recovered.

3.2 Quantity obtained per unit time interval vs. Time

Fig 3 shows the rate at which a particular volume of liquid methanol is recovered after condensation of methanol vapours. During the recovery of methanol from the mixture of products of transesterification reaction obtained during 10 and 12.5ℓ of the biodiesel production in a batch-type biodiesel plant, there is an abrupt increase in the volume of methanol obtained at a particular time interval due to complete conversion of methanol vapour into the liquid state. After reaching a peak value, which is almost the same for both, 147 ml for 10ℓ and 145 ml for 12.5ℓ, but achieved at different time intervals, 10^{th} min. for 10ℓ and 20^{th} min. for 12.5ℓ, which could also be called as maximum efficiency of the condenser which is to condense vapour into liquid form under a particular time interval, there is a decrease in the volume of liquid methanol obtained as the evaporation rate decreases with the increase of time. The graph for methanol extraction from the mixture of end products obtained during the production of 15 and 17.5ℓ treated biodiesel shows a decrease in the peak value, 120 ml for both, which was obtained at 30 and 35 min., respectively. While considering 25ℓ of biodiesel production, there was a decrease in the maximum capacity of the condenser and the condensation rate remains almost constant (50^{th} to 130^{th} min and 160^{th} to 220^{th} min.). The maximum conversion rate of methanol vapours into liquid was decreased because of the formation of a liquid film of methanol on the outer layers of the double-helical tubes of the condenser, which in turn delays the total amount of methanol to be recovered. Thus, increasing the energy consumed during the methanol recovery process.

The maximum amount of methanol recovered at an interval of 5 min. from the mixture of end products obtained after transesterification reaction during the production of 10, 12.5, 15, 17.5 and 25ℓ biodiesel was found at 72.3, 73.3, 71, 72.7 and 69.2 °C, respectively, having values of 147, 145, 120, 120 and 87.5 ml, respectively. Till reaching these temperatures the capacity of methanol recovered was 274, 475, 594.38, 640 ml and 749.99 ml, which is 27.42, 36.67, 37.3 34.55 and 28.41 % of the total methanol recovered respectively for different volumes of 10, 12.5, 15, 17.5 and 25ℓ considered, respectively.

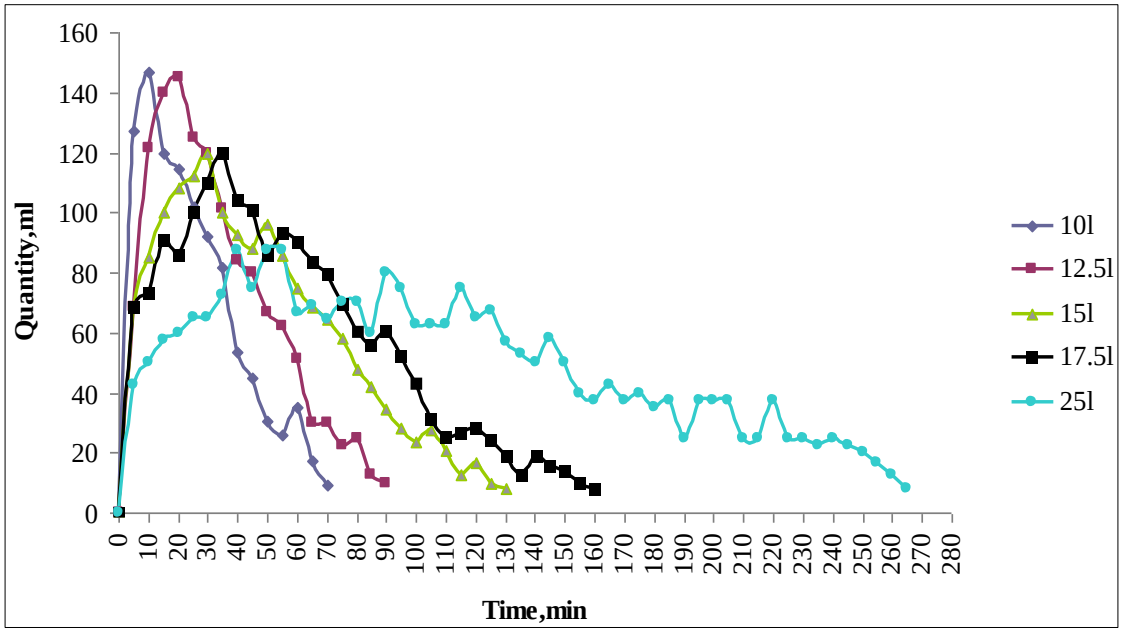

Fig 3: Condensation rate of methanol in a rotary vacuum flask evaporator

Table 2(a): Amount of methanol recovered during 10l, 12.5l, 15l, 17.5 and 25ℓ of treated biodiesel production

10ℓ of biodiesel				12.5ℓ of biodiesel			15ℓ of biodiesel			17.5ℓ of biodiesel		
Time (min,)	Temperature (°C)	Quantity of methanol recovered (ml)	Cumulative methanol recovery (ml)	Temperature (°C)	Quantity of methanol recovered (ml)	Cumulative methanol recovery (ml)	Temperature (°C)	Quantity of methanol recovered (ml)	Cumulative methanol recovery (ml)	Temperature (°C)	Quantity of methanol recovered (ml)	Cumulative methanol recovery (ml)
0	65	0	0	65	0	0	65	0	0	65	0	0
5	70.4	127	127	68.3	68.75	68.75	65.5	68.75	68.75	66.2	68.75	68.75
10	72.3	147	274	70.4	121.25	190	66.8	85.4	154.15	67.4	72.92	141.67
15	74.7	119.5	393.5	71.8	140	330	67	100	254.15	68.6	90.83	232.5
20	76.7	114.5	508	73.3	145	475	68.9	108.23	362.38	69.7	86	318.5
25	79.3	102	610	75.3	125	600	69.8	112.5	474.88	70.7	100	418.5
30	82.2	92	702	77.2	120	720	71	120	594.88	71.8	110	528.5
35	85.4	82	784	78.8	101.4	821.4	71.6	100	694.88	72.7	120	648.5
40	87.5	53.4	837.4	81.4	84.12	905.52	72.7	92.5	787.38	73.8	104.14	752.64
45	90	44.84	882.24	84	80.11	985.63	73.7	87.83	875.21	74	100.84	853.48
50	90	30.54	912.78	87	66.67	1052.3	74.9	96.33	971.54	75.7	85.67	939.15
55	90	25.81	938.59	89	61.9	1114.2	76	85.83	1057.37	77	93.33	1032.48
60	90	35.3	973.89	90	51.42	1165.62	77.3	74.71	1132.08	78.1	90.15	1122.63
65	90	17.28	991.17	90	30	1195.62	78.5	68.67	1200.75	79.4	83.5	1206.13
70	90	8.14	999.31	90	30	1225.62	79.9	64.5	1265.25	80.7	79.5	1285.63
75				90	22.5	1248.12	81.5	58	1323.25	82.1	69.5	1355.13
80				90	25	1273.12	83.1	47.5	1370.75	83.5	60.5	1415.63
85				90	12.5	1285.62	84.7	42	1412.75	85.1	56	1471.63
90				90	10	1295.62	87.3	34.75	1447.5	86.6	60.5	1532.13
95							90	28.25	1475.75	88.2	52.5	1584.63
100							90	23.33	1499.08	89.6	43.32	1627.95
105							90	27.83	1526.91	90	31	1658.95
110							90	20.5	1547.41	90	25.56	1684.51
115							90	12.5	1559.91	90	26.36	1710.87
120							90	16.66	1576.57	90	28	1738.87
125							90	10	1586.57	90	24	1762.87
130							90	8.33	1594.9	90	18.75	1781.62
135										90	12.5	1794.12
140										90	18.8	1812.92
145										90	15.66	1828.58
150										90	13.66	1842.24
155										90	10	1852.24
160										90	8.33	1860.57

Table 2(b): Amount of methanol recovered from 25 ℓ of biodiesel

25ℓ of biodiesel			
Time (min.)	**Temperature (°C)**	**Quantity of methanol recovered (ml)**	**Cumulative methanol recovery (ml)**
0	65	0	0
5	65.2	42.66	42.66
10	65.6	50	92.66
15	66.2	57.33	149.99
20	66.7	60	209.99
25	67.3	65	274.99
30	67.7	65	339.99
35	68	72.5	412.49
40	68.3	87.5	499.99
45	68.6	75	574.99
50	68.9	87.5	662.49
55	69.2	87.5	749.99
60	69.5	66.66	816.65
65	69.9	68.83	885.48
70	70.5	64.2	949.68
75	71.1	70.5	1020.18
80	71.7	70	1090.18
85	72.3	60	1150.18
90	72.8	80	1230.18
95	73.4	75	1305.18
100	73.9	62.5	1367.68
105	74.5	62.5	1430.18
110	74.8	62.5	1492.68
115	75.2	75	1567.68
120	75.6	65	1632.68
125	76.2	67.14	1699.82
130	76.8	57.14	1756.96
135	77.3	52.86	1809.82
140	78.2	50	1859.82
145	79	58.33	1918.15
150	79.8	49.98	1968.13
155	80.6	40	2008.13
160	81.5	37.5	2045.63
165	82.3	42.5	2088.13
170	83.2	37.5	2125.63
175	83.8	40	2165.63
180	84.5	35	2200.63
185	85.1	37.5	2238.13
190	85.8	25	2263.13
195	86.5	37.5	2300.63
200	87.2	37.5	2338.13
205	87.9	37.5	2375.63
210	88.2	25	2400.63
215	88.5	25	2425.63
220	89	37.5	2463.13
225	89.5	25	2488.13
230	90	25	2513.13
235	90	22.5	2535.63
240	90	25	2560.63
245	90	22.5	2583.13
250	90	20	2603.13
255	90	16.66	2619.79
260	90	12.5	2632.29
265	90	8.33	2640.62

The fluctuations during the downward motion of the curve are more abrupt and clearly can be seen methanol recovery process. There is an increase in the volume of methanol recovered at 60th (35.3 ml) and 80th min. (25 ml) from the mixture of end products obtained for 10 and 12.5ℓ of biodiesel production respectively. During methanol extraction from the mixture of end products obtained during 15ℓ biodiesel production, this fluctuation was observed at 50th min. (96.33 ml), and 105th min. (27.83 ml), and for 17.5 ℓ observed durations were 55th min. (93.33 ml), 90th min. (60.5 ml), 115th min. (26.36 ml), 120th min. (28 ml) and 140th min. (18.8 ml). For 25 ℓ of biodiesel production, there appeared a continuous fluctuation in the amount of methanol to be recovered. This condition appears due to two reasons: water inlet temperature, and mass and gravity factor. During a higher rate of methanol recovery, the dominant factor is water inlet temperature. With the decrease in temperature, there is a spurt increase in the rate at which methanol is

recovered. This justifies that the lower the temperature of inlet water, the higher the rate at which methanol would be recovered and the lesser will be the total duration for methanol recovery. Conditions during low evaporation rates are entirely different. Here, during the conversion of vapour into liquid state, liquid drops formed are not too heavy to fall. Also, in a vertical condenser, the drops at the upper level have to fall through several restrictions in the form of helical tubes. When another layer of vapour comes, it condenses and hence the pre-existing droplets become heavier and fall, thus contributing to increasing the volume of methanol recovered.

The volume of methanol obtained during the downward trend of the curve increased with the increase in the volume of methanol to be obtained under equal time zones. The curve obtained during methanol extraction from the mixture of end products obtained during the production of 10 and 12.5ℓ, and 15 and 17.5ℓ and 25ℓ treated biodiesel are closer to each other while the curve of 12.5 and 15ℓ and 17.5 and 25ℓ are apart or in other words, former has steep slope while latter has gradual slope because a large amount of methanol is still to be recovered, thus a large amount of evaporated methanol was coming out, which requires a longer duration of time to condense.

3.3 Cumulative Methanol Recovery vs. Time

The cumulative methanol recovery vs. time graph represented in Fig. 4 shows the total volume of methanol obtained after a time interval. Graphs of different volumes considered show similar trends with respect to time. The total volume of methanol recovered from the mixture of end products of transesterification reaction during the production of 10, 12.5, 15, 17.5 and 25ℓ treated biodiesel was found to be 999.04, 1295.62, 1594.9 and 1860.57 and 2640.62ml respectively. Total time required to extract the total methanol from the mixture obtained during production of different volumes of biodiesel viz. 10l, 12.5l, 15l, 17.5 and 25ℓ after transesterification reaction was found to be 70 min., 90min., 130min. 160 and 265min. respectively. It was found that the methanol recovered for different volumes of treated biodiesel viz. 10, 12.5, 15, 17.5 and 25ℓ to be produced was 81.4, 84.5, 86.7, 86.7 and 86.13 % of the unused methanol which remained as a part of end products obtained during the transesterification reaction respectively. While 40.7, 42.25, 43.35, 43.35 and 43.06% of methanol used as a reactant in the transesterification reaction was recovered from the mixture of end products obtained during the production of 10, 12.5, 15, 17 and 25ℓ treated biodiesel respectively. This shows the maximum value of methanol recovered during a recovery process in a batch-type biodiesel plant.

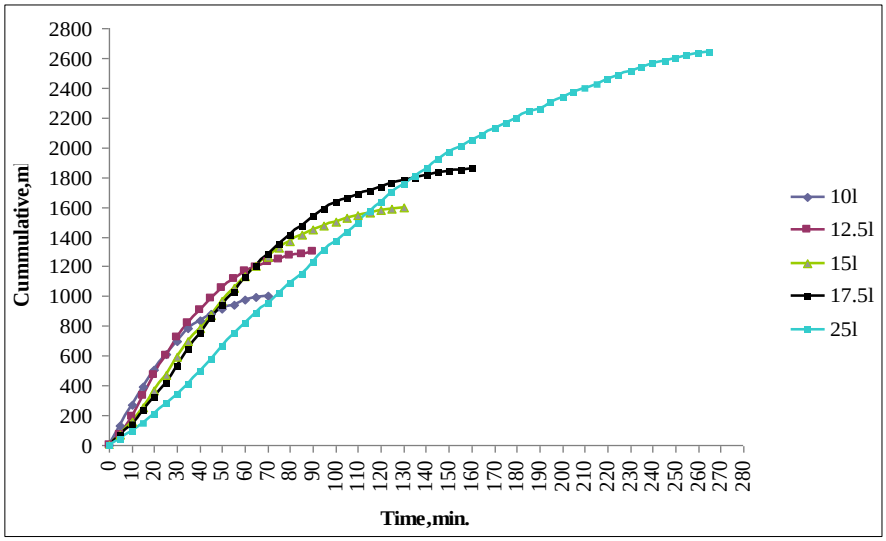

Fig 4: Amount of methanol recovered at different intervals of time

During the first half of the total duration required for methanol recovery, the percentage of methanol recovered for 10, 12.5, 15, 17.5 and 25ℓ was found to be 78.47, 76.07, 75.28 and 76.07 and 69%, respectively. It is clear that from the Fig. 4 that 35, 45, 65, 80 and 135min. are required to recover about 75% of the excess methanol that remained unused during the transesterification reaction from the mixture of end products obtained during the production of 10, 12.5, 15, 17.5 and 25ℓ biodiesel respectively. However, the remaining period of 35, 45, 65, 80 and 130min. was utilized for the recovery of only 25% of the methanol from the mixture of end products of transesterification reaction for different volumes viz. 10, 12.5, 15, 17.5 and 25ℓ of treated biodiesel production, respectively.

3.4 Amount of products obtained experimentally

The statistics of different products namely biodiesel, glycerine and methanol obtained after the completion of the process is presented in the form of mass balance equations. The results show there is a deviation from the calculated mass of products.

Table 3 shows the difference between theoretical and experimental values of biodiesel produced for 10, 12.5, 15, 17.5 and 25ℓ biodiesel production and was found to be 371.09, 668.37, 903, 1056.88 and 1556 g respectively. Thus, shows that there was a decrease in the mass of biodiesel produced because the conversion of palm oil into biodiesel was not achieved up to 100%. The total conversion of palm oil into biodiesel for different volumes viz. 10, 12.5, 15, 17.5 and 25ℓ was found to be 96.15, 94.25, 93.44, 93.44 and 93.22% respectively. This could be due to the formation of soap [4] while another factor was that the pressure was maintained at 101.3kPa. It has been shown that with the increase in pressure applied on the reaction chamber of 0.10, 0.50, 0.85 and 1.2 MPa there is an increase in product yield to 94.4, 100, 102.8 and 103.2% respectively [5]. But for

glycerol, there is an increase in the mass obtained experimentally. For 10, 12.5, 15, 17.5 and 25ℓ the difference was 548.97, 856.67, 1095.15, 1288 and 1889.67 g, respectively. Two reasons explain this fluctuation; first conversion percentage was lesser than the theoretical one and second methanol is still present at the interstices of glycerol. There is a decrease in the amount of methanol obtained because a greater amount is bonded with the molecules of biodiesel and glycerol. Also, vacuum pressure and the condenser can never be 100% efficient.

Table 3: Variation in the amount of products obtained after the transesterification reaction

Volume of biodiesel to be produced(l)	Biodiesel g (E)	Biodiesel g (T)	Difference (E-T), g	Glycerol g (E)	Glycerol g (T)	Difference (E-T) g	Methanol recovered, g	Unused Methanol, g	Difference (E-T), g
10	8264.41	8635.5	-371.09	1470	921.03	587.97	783.24	961.33	-178.09
12.5	10126.23	10794.37	-668.37	2005.74	1151.67	856.67	1015.76	1201.67	-185.91
15	12050.25	12953.25	-903	2476.07	1381	7095.15	1250.48	1442	-191.52
17.5	14055.24	15112.12	-1056.88	2897	1608.81	1288	1458.86	1682.33	-223.47
25	20032.97	21589	-1556	4192	2302.33	1889.67	2070	2403.33	-333.09

E – Experimental T - Theoretical

3.5 Purity of recovered methanol

The results obtained from gas chromatography have been presented in Table 4. The purity of recovered methanol was found by calculating the areas under the peaks obtained from chromatograms using the Gas Chromatography technique. Both, pure methanol and experimentally recovered methanol chromatogram have been shown in Fig. 5 and Fig. 6, respectively. The corrected area of pure methanol used as a reactant during the transesterification reaction was found to be 3,124,051,536 units and its corresponding value for methanol which was recovered after completion of the methanol recovery process was found as 3,120,350,423 units. There is a decrease in
the area of recovered methanol as compared to pure methanol which was found to be 3,701,113 units. The purity of recovered methanol as compared to the pure methanol was found to be 99.87%.

The content of methanol present in both untreated and treated biodiesel has been analysed by using the Gas Chromatography technique, which is shown in Fig 7 and Fig.8, respectively. The area under the peak shows the percentage of methanol present in each untreated and treated biodiesel. The area under peak obtained for methanol was found to be 202324306 units and 5766551 units in the case of untreated and treated biodiesel respectively. There is a decrease of 196,557,755 units of the area in the case of treated biodiesel as compared to the untreated biodiesel, which is 97.14% of the area obtained under the methanol peak in the untreated biodiesel sample. The area under the methanol peak in both untreated and treated shows a large difference in value when compared with each other, which proves that there was a reduction in the per cent content of methanol in treated biodiesel.

The maximum corrected area of methanol for both untreated and treated biodiesel was found to be 3.63% and 0.07% respectively. This also confirms that there is a decrease in the amount of methanol content in the sample of treated biodiesel as the percentage of methanol content in the treated biodiesel sample is lesser than the untreated biodiesel.

Table 4: Results obtained for different samples analysed by using gas chromatography

Sample Name	Retention time min.	Start in time min.	End time min.	PK TY	Peak Height	Corrected Area	Corrected % max.
Methanol Pure	2.157	2.05	2.3	BB	78226856	3124051536	100
Methanol Recovered	2.153	2.05	2.27	BB	77267691	3120350423	100
Untreated Biodiesel	2.105	2.07	2.26	BB	17208382	202324306	3.63
Treated Biodiesel	2.105	2.08	2.18	BB	462252	5766551	0.07

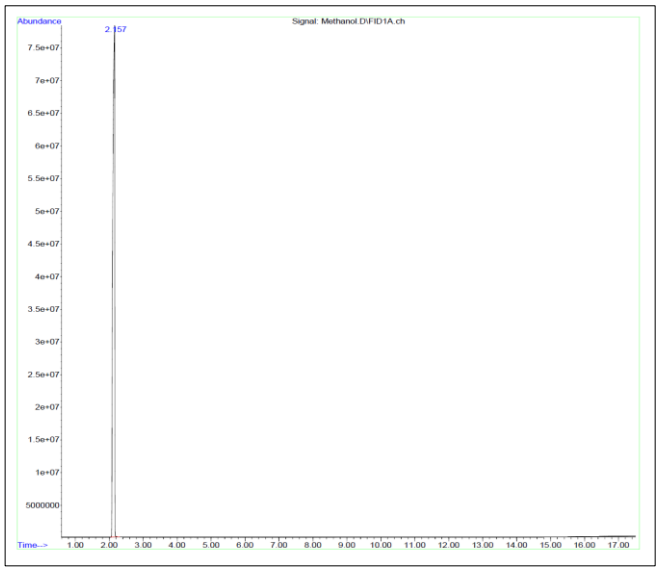

Fig 5: Chromatogram of pure methanol

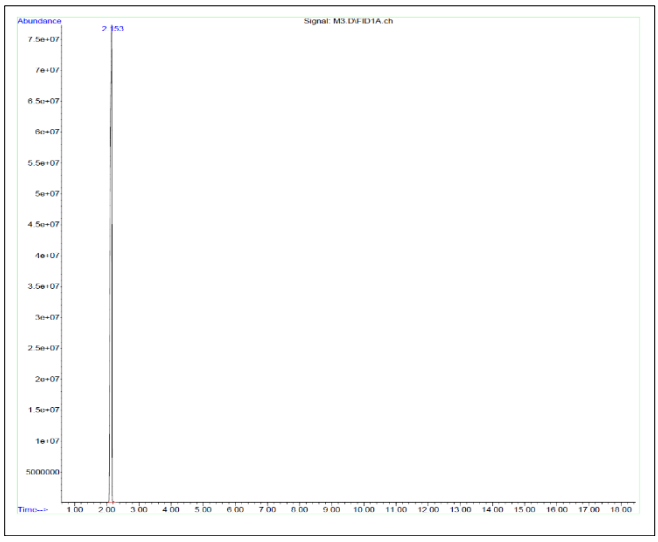

Fig 6: Chromatogram of recovered methanol

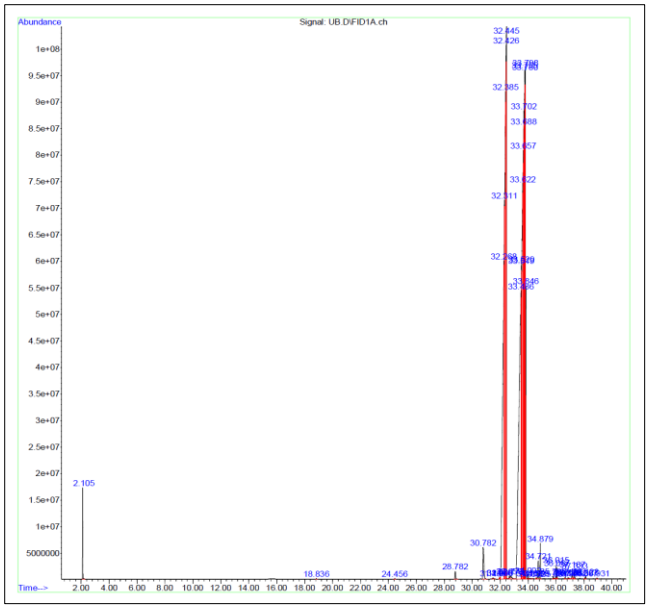

Fig 7: Chromatogram of untreated biodiesel

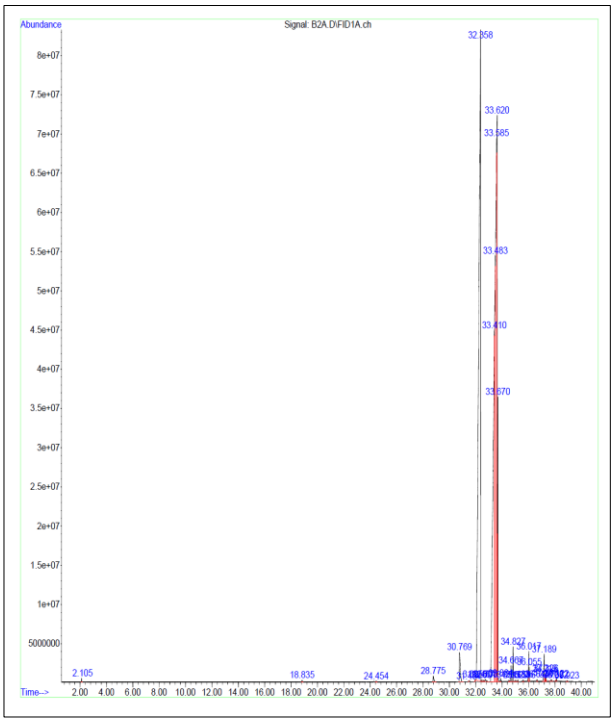

Fig 8: Chromatogram of treated biodiesel

3.6 Scale Up

3.6.1 Volume of Mixing Chamber

The number of revolutions (N) for the laboratory biodiesel plant, when 25ℓ of biodiesel is produced comes out to be 180.66 rpm, which was considered constant for the scale-up. The ratio of the diameter of the mixing tank to the height up to which the end products obtained after transesterification for the highest capacity of laboratory biodiesel plant i.e. 25ℓ /batch was found as 0.78 and considered constant for scale-up of cylindrical reactor volume meant for transesterification reaction. The total volume of mixture after the transesterification reaction as shown in Table 5 for the production of 125 and 250ℓ biodiesel inside the cylindrical tank was found to be 149.05 and 299.01ℓ. The height up to which the products of the transesterification reaction stand without agitation was found to be 0.6788 and 0.8553m while the diameter of

Table 5: Volume of products obtained during the production of 125 and 250ℓ of biodiesel

Volume of biodiesel to be produced (ℓ)	Theoretical volume of products of transesterification reaction (ℓ)			
	Biodiesel	Glycerol	Methanol	Total Volume
125	125	9.81	15.325	149.05
250	250	18.36	30.65	299.01

the cylindrical tank was found to be 0.5295 and 0.667m during the production of 125 and 250ℓ of biodiesel production. The height up to which the fluid would rise upwards during agitation was calculated and found to be 0.6386 and 1.045m, thus the total height for the cylinder during agitation came out to be 1.317 and 1.87m for 125 and 250ℓ of biodiesel production. The minimum volume of the cylinder restricting the overflow of liquid during agitating for the production of 125 and 250ℓ biodiesel was found to be 0.290 and 0.6536m^3 respectively.

3.6.2 Agitation

The impeller used for the laboratory is a turbine having twin straight blades and the working tank is devoid of baffles. The impeller is attached to the motor through a belt and pulley system, with a larger pulley attached to the impeller. The range of Power number for a turbine agitator with a vertical blade is between 0.93 to 1.08 [6]. Taking the Power number value to be 1, the power required for agitating the volume of the mixture obtained during the production of 125 and 250ℓ biodiesel was found to be 1947.77 W and 6186.31 W respectively.

3.7 Prediction of methanol recovery for higher capacity batch type biodiesel plant

Table 6 shows the amount of methanol recovered, energy consumed and the time taken to recover the known quantity of methanol from different experimental values obtained during the methanol recovery from the mixture of end products of transesterification reaction for 10, 12.5, 15 and 17.5ℓ of biodiesel production. These values, which are obtained experimentally, were considered for extrapolation through simple graphs, along with generating equations as per the highest goodness of fit, R^2.

For each dependent variable, viz. methanol recovered, energy consumed and time required during extraction of methanol graphs were plotted by considering the volume of biodiesel to be produced as an independent variable.

Table 6: Experimental values obtained during methanol recovery

Biodiesel, ℓ	Methanol, g	Energy, kWh	Duration, min
10	783.247	1.3	70
12.5	1015.77	2.25	90
15	1250.4	2.9	130
17.5	1458.87	3.85	160
25	2070.24	5.85	265

The Equation and goodness of fit (R^2) values for different graphs are as follows:

i) Methanol Recovered vs. Biodiesel Produced

$Y = 85.399X - 50.684$ …..1

$R^2 = 0.9987$

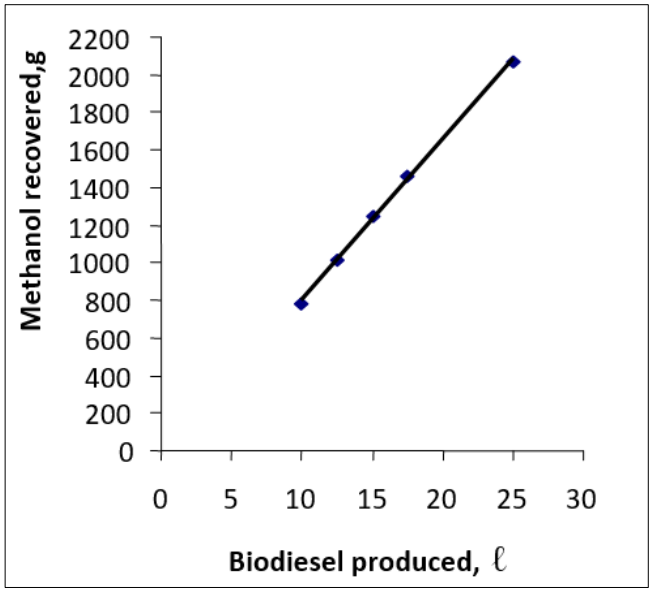

Fig 9: Methanol recovered vs. Biodiesel produced

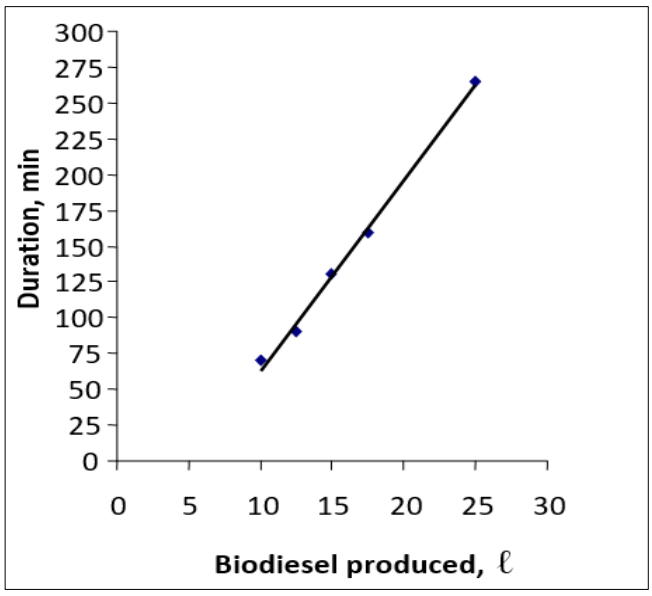

Fig 10: Energy consumed vs. Biodiesel produced

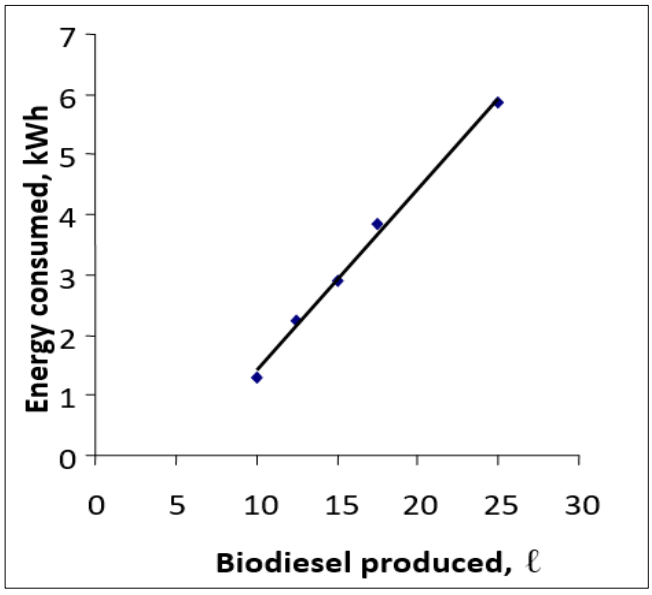

Fig 11: Duration vs. Biodiesel produced

ii) Energy Consumed vs. Biodiesel Produced
$Y = 0.3008X - 1.5821$ ….2
$R^2 = 0.9952$

iii) Duration vs. Biodiesel produced
$Y = 13.283X - 69.528$ ……3
$R^2 = 0.9957$

3.7.1 Biodiesel plant capacity: 500ℓ/d (125ℓ/batch)

The amount of methanol recovered per batch, energy consumed during methanol recovery per batch and duration required for recovery of methanol per batch, as predicted from Eq. 1, 2 and 3 were found to be 10684.19 g, 36.018 kWh and 1590.84 min. respectively. The percentage of methanol recovered was found to be 88.41% of the excess methanol that remained unused during the transesterification reaction (Table 7).

3.7.2 Biodiesel plant capacity: 1000ℓ/d (250ℓ/batch)

Equations 1, 2 and 3 were applied to predict the values of methanol recovered per batch, energy consumed during methanol recovery per batch and duration of methanol recovery process per batch. The methanol recovered was found to be 21299.06 g, which is 88.62% of the unused methanol (Table 7). The energy consumed and duration for the unused methanol to be recovered from the mixture of the end products of transesterification was found to be 73.168 kWh and 3251.22 min. respectively.

Table 7: The theoretical amount of products obtained while producing 125 and 250ℓ of biodiesel

Volume of biodiesel to be produced (ℓ)	Theoretical amount of products obtained after transesterification (g)		
	Biodiesel	Glycerol	Methanol
125	107943.75	11512.85	12016.67
250	215887.5	23025.7	24033.34

4. Conclusions

Three fourth of the total volume of methanol recovered experimentally was found to be 749.48, 971.71, 1196.25, 1395.42 and 1980.46 ml at 35th min., 45th min., 65 min., 80min., and 150th min., respectively for different volumes of treated biodiesel viz. 10, 12.5, 15, 17.5 and 25ℓ production. The total height and diameter of the biodiesel plant having a capacity of 500ℓ/d (125ℓ/batch) were found to be 1.317 m and 0.5295 m, respectively. The volume of the reaction chamber was found to be 0.29 m^3. For producing 250ℓ/batch (1000ℓ/d) biodiesel the height and diameter of the cylindrical tank meant for the transesterification reaction was found to be 1.870 and 0.6671 m, respectively. The volume of the reaction chamber was found as 0.6536 m^3. The power required to agitate the volume of the mixture obtained as an end product of transesterification reaction for 125ℓ/batch (500ℓ/d) and 250ℓ/batch (1000ℓ/d) having number of revolutions as 180.66 rpm was found to be 1947.77 and 6186.31 W, respectively. The total amount of unused methanol as predicted from the theoretical mass balance equation for 125ℓ/batch (500ℓ/d) and 250ℓ/batch (1000ℓ/d) biodiesel productions was found to be 12016.67 and 24033.34 g, respectively. The predictive amount of methanol recovered, the time required to obtain that amount and the energy consumed thereof for 125ℓ/batch (500ℓ/d) were found to be 10684.19 g, 1590.84 min. and 36.018

kWh, respectively. The predicted amount of methanol to be recovered is 88.41% of the unused methanol remaining in the mixture of end products obtained after the transesterification reaction. Prediction for the extraction of methanol from the mixture of end products obtained during the production of 250ℓ of biodiesel per batch (1000ℓ/d) was done through the equation obtained for the total amount of methanol recovered, the time required to extract the predicted amount of methanol and the energy consumed thereof, and was found to be 21299.06 g, 3251.22 min., and 73.168 kWh, respectively. The amount of methanol recovered was found to be 88.62% of the unused methanol remained in the mixture of end products obtained after the transesterification reaction.

5. Abbreviations

C : Carbon
G : gram
H : Hydrogen
ℓ : litre
ℓ/d : litre per day
kWh : kilowatt-hour
m : meter
min. : minutes
ml : millilitre
mb/d : million barrels per day
O : oxygen
R_1, R_2, R_3 : Alkyl groups

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