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Design and development of bubble-cap tray column for continuous transesterification plant in coconut-oil-based biodiesel production

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ABSTRACT

A study was carried out on production of biodiesel from coconut oil using alkali catalyzed transesterification process by two different reactive distillation columns, viz. sieve tray and bubble-cap tray column. Coconut oil with free fatty acid content of 1.2% was used for transesterification. In laboratory scale batch system, among all the variations adopted, it was found that maximum methyl esters yield of 97.7% was obtained with 6:1 molar ratio of methanol-to-oil, 1% NaOH at 60ºC reaction temperature. A continuous flow transesterification using sieve tray reactive distillation column yielded 93.75% methyl ester with 5.55 min average reaction time at 65ºC under 4:1 molar ratio (methanol-to-oil). The excess alcohol was found to be 11.4 ml. Based on performance evaluation of sieve tray reactive distillation column, a new bubble-cap tray reactive distillation column was designed and developed for continuous biodiesel production. Maximum methyl ester yield was 96.2% with 5.55 min average reaction time at 65ºC under 4:1 molar ratio. Excess alcohol was found to be 17.1 ml. Daily average electricity consumption was found to be 13.6 kWh for continuous transesterification biodiesel production and processing cost was worked out as ₹ 26.03. Regression analysis was employed to evaluate the effects of molar ratio and temperature on the yield of methyl esters. The R^2 value was 0.87 for sieve tray reactive distillation column and 0.92 for bubble-cap reactive distillation column which confirmed the validity of the predicted model. Based on the findings from the study, it was found out that continuous transesterification was more feasible and efficient way for production of biodiesel. It was concluded that bubble-cap tray reactive distillation column was found to be an effective than sieve tray column due to its increased yield (1 time), excess alcohol recovery (1.5 times) and better fuel properties.

Key words: Biodiesel, Bubble-cap tray column, Distillation columns, Sieve-tray column, Transesterification

The production of biodiesel from vegetable oil consists of a simple chemical process called the transesterification. The transesterification process can be carried out chemically or enzymatically through batch or continuous system. Batch processes require larger reactor volumes, thus requiring higher capital investment and they are inherently less efficient than continuous processes which allow the users to accommodate variations in feedstock type, composition and quantity. The major drawbacks of the batch process include low productivity, larger variation in product quality, and more intensive labor and energy requirements. One of the keys to making biodiesel a viable and profitable energy source is the use of a continuous flow transesterification process to reduce time and cost, thereby increasing production and profit. The methods of continuous transesterification process that can be used to produce biodiesel are supercritical, reactive distillation, continuous stirred-tank reactor, ultrasonic, oscillatory flow reactor and static mixing. Of these processes, reactive distillation was found to be most adaptable due to its own process advantages because of simultaneous chemical reactions and distillation processes in a countercurrent column.

In transesterification methods commonly used for producing biodiesel are batch process and continuous process. The most common method for producing biodiesel is the one-step or two-step batch transesterification process. Batch processes suffer several disadvantages compared to continuous processes: Batch processes require larger reactor volumes, thus requiring higher capital investment; they are inherently less efficient than continuous processes owing to their start-up and shut down nature; there are batch-to-batch variations in the quality of the products and labour costs are higher with batch processes (Darnoko 2000). Increasing biodiesel consumption requires optimized production processes allowing high production capacities, simplified operations, high yields, and the absence of special chemical requirements and waste streams. So, continuous

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transesterification is preferred over batch processes in commercial production because of higher biodiesel recovery, lesser retention time, lesser alcohol to oil molar ratio, excess methanol recovery and these processes result in consistent product quality and low capital and operating costs per unit of product (Singh *et al*. 2004).

Keeping in view the above perspectives, the present investigation on development of continuous transesterification unit was taken with the aims of development of reactive distillation column for continuous transesterification of vegetable oils including optimization of the process parameters such as reaction time, reaction temperature, chemical inputs, etc.

MATERIALS AND METHODS

The aim of this work was to develop different types of reactive distillation (RD) column in continuous transesterification unit and study the properties of biodiesel. The study was carried out on coconut oil which was procured from local oil mills. Coconut oil was selected based on its availability and comparatively cheaper cost. The properties such as free fatty acid, kinematic viscosity, specific gravity and cloud point were analyzed.

Continuous transesterification processes are preferred over batch processes in commercial production because continuous flow generally produces more fuel per unit of labour, high productivity and allows for larger scale projects, thus reducing the overall cost of production. In continuous transesterification process, the reactive distillation is found to be the most adaptable due to its own process advantages like alcohol to oil ratio, reaction temperature, etc. because of simultaneous chemical reactions and distillation processes in a countercurrent column, so a continuous flow reactive distillation process was developed for the production of biodiesel from vegetable oil. Earlier, the sieve tray type reactive distillation column was designed and developed by students of McGill University at Tamil Nadu Agricultural University. However, the performance of the reactor was not evaluated. Under this project work, the performance of existing sieve tray reactive distillation column was evaluated. Based on the performance evaluation, a new reactive distillation column tray, namely, Bubble-cap tray was designed and developed and its performance was evaluated and compared with sieve tray reactive distillation column.

Reactive distillation (RD) column: A glass vessel (3 l capacity) was used for oil tank and another glass vessel of 1 l capacity was used for methoxide solution. Gravity flow was maintained throughout the process. The RD column was used for transesterification, which consists of two parts, namely outer shell and a plate system. The column has 10 plates with an inner diameter of 82 mm, a weir height of 18 mm and distance between plates as 25 mm. The nominal hold-up on each plate was set as 2 ml, totaling 100 ml for the column, made of 2 mm thick stainless steel (316 L grade). The outer shell was made using a standard pipe (84 mm diameter), with a cone at the bottom (3/4" diameter) and a flange at the top. It has an inlet at the top (3/8" diameter) for the solution to enter into the plate system from pre-reactor made of 2 mm thick stainless steel (316 L grade). The removable lid was a cone with flange made of 5 mm thick, 116 mm diameter stainless steel (316 L grade) for fixing with outer shell. It has an outlet (3/8" diameter) for the methanol vapour to escape. The column has a jacket around it, as a means of thermal insulation (glass wool), to prevent heat-loss from the column. Heating coil (1 000 W) was used to maintain the desired temperature inside the reactor.

Capacity of reactor: The capacity of unit was designed (100 ml) based on the working volume of reactive distillation column.

Bubble-cap: A bubble-cap tray has riser or chimney fitted over each hole, and a cap that covers the riser. The cap is mounted so that there is a space between riser and cap to allow the passage of vapour. Vapour rises through the chimney and is directed downward by the cap, finally discharging through slots in the cap, and finally bubbling through the liquid on the tray.

Pre-reactor: Pre-reactor was used at the upstream of the reactive column to handle a substantial part of the reaction duty. The capacity of the reactor is 50 ml, made of glass. It consists of three openings; inclined pipe inserted at the desired liquid level (50 ml) and used as side openings on both the sides of reactor as inlet and outlet. Reactant is added through one of the side openings, a mechanical stirrer is placed at the central opening and other side opening acts as outlet.

Evaporator: An evaporator was used to drive methanol off the product mixture, made of glass (250 ml flask with 100 ml working volume), which was heated by 250 ml heating mantle (150 W). A vapour outlet was also added on the surface of the flask.

Decanter: The product from the evaporator was transferred to the gravity settler (5 l capacity) made of glass to separate glycerol phase from biodiesel.

Condenser: A water-cooled condenser (30 cm) made of glass was used to condense the methanol vapour from the RD column and evaporator. A 500 ml flask made of glass is used to collect condensed excess methanol vapors from the top of the column and evaporator.

The vegetable oil was checked for free fatty acid content. Up to 5% FFAs, the reaction can be catalyzed with an alkali catalyst. When the FFA level is above 5%, the acid treatment process can be used as a pre-treatment to convert the FFAs in high FFA feed stocks to methyl esters and thereby reduce the FFA level. Then the low FFA pretreated oil can be transesterified with an alkali catalyst to convert the triglycerides to methyl esters. The methanol and desired catalyst in desired amounts transferred to a container and stirred to completely dissolve the catalyst in methanol. The amount of methanol and catalyst was determined based upon the required methanol-to-oil ratio and catalyst concentration. A flow rate between the maximum and minimum was being adopted using flow controller. For molar ratio of 3:1, a flow rate of 6 ml/min and 0.6 ml/min was adopted for oil and methanol, respectively, during the

Table 1 Pumping rate for oil and methanol

Molar ratio	Volume ratio (methanol-to-oil)	Flow rate (ml/min)			
		Oil		Methanol	
		Maximum	*Minimum*	*Maximum*	*Minimum*
3:1	0.170	42.74	8.55	7.27	1.45
3.5:1	0.198	41.74	8.35	8.26	1.65
4:1	0.227	40.75	8.15	9.25	1.85
4.5:1	0.255	39.84	7.97	10.16	2.03

experiment. For molar ratio of 3.5:1, a flow rate of 12 ml/min and 1.8 ml/min were adopted for oil and methanol, respectively, during the experiment. Similarly, for molar ratio of 4:1 and 4.5:1, a flow rate of 18 ml/min and 36 ml/min respectively, for oil; and 6 ml/min and 12 ml/min respectively, for methanol were adopted during the continuous transesterification experiment. A glass socket was attached with pre-determined pore size (0.6 ml/min, 1.8 ml/min, 6 ml/min, 12 ml/min, 18 ml/min and 36 ml/min) pipe. It can be accordingly placed and fitted for the required flow rate. The Pre-reactor, RD column and evaporator heaters were set to desired temperature and allowed to heat for the determined temperature. The oil and methoxide solution were filled in respective containers. The flow controller was used to adjust the required flow.

Physico-chemical properties of diesel and biodiesel blends such as kinematic viscosity, specific gravity, acid value, carbon residue, ash content and cloud point are found out as follows (IS 1460:2000 and Pramanik, 2002). The procedure followed to measure the different physico-chemical properties of fuel is detailed below.

Kinematic viscosity: The kinematic viscosity of sample was determined by Redwood viscometer (Fig. 3) using American Society for Testing Materials (ASTM 445-72) procedure. The kinematic viscosity of sample was calculated as follows.

Kinematic viscosity, cSt = $0.26t - 179/t$ (for $34<t<100$)
Kinematic viscosity, cSt = $0.24 \times t - 179/t$ (for $t>100$)

where, t - time taken for the flow of 50 ml of oil sample, s.

Specific gravity: The specific gravity of fuel blends was determined using specific gravity bottle method (IS: 1448-1972). The experiments were conducted at 40°C.

Cloud point: The ASTM method D-2500 was adopted for determining the cloud point of samples. A cooling bath was prepared using an empty Styrofoam packing box. Salt was added to the ice in the box to obtain temperatures in the range of -1 to -3°C. Samples of methyl esters (15 ml) were placed in a glass jar and the temperature was lowered until clouds of crystals appeared at the bottom of the jar.

Carbon residue and ash content: The carbon residue apparatus was used to determine the carbon residue of the samples (ASTM D524-IP14/65). Ash content of test fuels was analyzed by the following procedure explained in IS: 1448-1992. 100 g of test fuel was taken in 120 ml silica crucible.

Free fatty acid and acid value: The Association of Official Chemists Society (AOCS) Official Method Ca 5a-40 was used to determine the Free Fatty Acid (FFA) of fuels. The acid value is defined as the number of milligrams of KOH necessary to neutralize one gram of sample. The acid value of test sample was calculated by using AOCS Official Method Ca 5a-40.

The energy balance is the relation between the energy produced (output/kg biodiesel) and energy consumed (input/kg biodiesel). The energy input consists of several components (oil, methanol and catalyst). Total energy input was the addition of energy input for oil, energy input for methanol and energy input for catalyst. The energy output from the continuous transesterification plant is obtained by multiplying the calorific value of product (H_p) and output product yield (M_p). The energy output consists of several components (biodiesel, glycerol and excess alcohol). The energy output was the addition of energy output of biodiesel, energy output of glycerol and energy output of excess alcohol.

$$Q_i = M_f H_f$$
$$Q_o = M_p H_p$$

where, Q_i - energy input (MJ), M_f - feed consumption (kg), H_f - calorific value of feed (MJ/kg, Q_o - energy output (MJ), M_p - product yield (kg), and H_p - calorific value of product

Fig 1 Sieve-tray RD column

Fig 2 Bubble-cap tray RD column

Fig 3 Redwood viscometer

Fig 4 Conradson carbon residue (CCR) apparatus

(MJ/kg).

RESULTS AND DISCUSSION

Properties of coconut oil

The free fatty acid, specific gravity, kinematic viscosity and cloud point of coconut oil was found to be 1.2%, 0.92, 26.52 cSt, and 19 °C.

Laboratory scale batch system

Laboratory scale batch system reactor of one litre capacity was developed to produce biodiesel from vegetable oils. The preliminary trials were conducted with laboratory scale batch system. Laboratory scale batch system consisted of three necked round bottom flask, mechanical stirrer, water cooled condenser and water bath. The oil was added through one of the side openings of the flask. The mechanical stirrer was placed in the central opening of the round bottom flask. The condenser was connected with another opening of the flask. This set-up was placed in a water bath and heated electrically. 250 ml of oil was taken in three necked round bottom flask. Water cooled condenser connected with round bottom flask was used to condense methanol vaporized during the reaction. Mechanical stirrer was operated to maintain uniform temperature of the reaction mixture and the speed of the stirrer is adjustable. The required amount of catalyst was weighed, dissolved completely with methanol and methoxide solution was prepared. The oil was heated to about 60°C and methoxide solution was added with the oil. The reaction was carried out for a period of about 2 h and the mixture was transferred to a separating funnel and allowed to settle by gravity. The glycerol settled at the bottom and the biodiesel was collected from the top.

Methyl esters yield and fuel properties were studied in laboratory scale batch process. The results indicated that the methyl esters yield was in the range of 73.29 to 97.72%. The ester yield increased with increasing catalyst concentration from 0.5% to 1%. At 0.5% catalyst concentration, methyl ester yield was 92.34, 91.84% with NaOH and KOH, respectively. But with 1.5% catalyst concentration, the methyl esters yield decreased. This negative impact was due to the formation of soap during transesterification (Sinha *et al*. 2008). Also it was reported that the esters yield reduced while increasing the amount of catalyst concentration from 0.5 to 1.5%. The most favorable condition for higher yield (97.72%) of methyl esters was 1% NaOH with 6:1 molar ratio at 60°C. This is in comparison with the biodiesel yield of 97.1% as reported by Rashid *et al.* (2008) at 60°C with 1% NaOH catalyst concentration using 6:1 methanol to oil molar ratio and Chitra (2005) resulted maximum methyl ester yield of 98% with 1% NaOH at 60°C reaction temperature. In the present study, it is also observed that 1% KOH as catalyst with 6:1 molar ratio at 60°C found the maximum ester yield (97.38%) was achieved. This is in comparison with the biodiesel yield of 98% with methanol-to-oil ratio (6:1 molar ratio) and 0.7% w/v KOH as an alkaline catalyst resulted by Shashikant (2006).

Singh *et al*. (2006) investigated that the soap was formed as the result of undesired side reactions between the catalyst and fatty acids, it was expected that the higher catalyst concentration would favour soap formation. Additionally, the use of potassium hydroxide as a catalyst led to higher level of soap formation during transesterification which led to the hydrolysis of esters to free fatty acids. Kinematic viscosity decreased with increased methyl esters conversion. It was found that 4.69 and 4.95 cSt at the time of higher methyl esters yield of 97.72 and 97.38%, respectively, with 1% catalyst at 60°C, also it was found to be within ASTM standards. There were no much variations in specific gravity throughout the process with catalyst and catalyst concentration. The results indicated that the specific gravity was 0.873, 0.876 and 0.878 using NaOH; 0.871, 0.874 and 0.879 using KOH with 0.5, 1 and 1.5% catalyst concentration, respectively.

Design of continuous transesterification plant

Capacity of reactor: Where, V_1 - working volume of reactor (RD column) (ml), RT_1 - retention time of RD column (min), Q - flow rate reactive solution (ml/min), h - height of liquid stagnation on plates (mm), and A_1 - total plate area in RD column (mm^2). A_1 = 5264 mm^2, where V_1 =100 ml; RT_1=2 to 10 min; Q = 50 ml/min; plate working area = 5000 mm^2. Then, column diameter, D = 82 mm. V_1, = 100 ml and RT_1 = 2 to 10 min.

Q, ml/min = V_1/RT_1
Plate working area, mm^2= V_1/h
A_1, mm^2 = (working area × % total area)/% working area

Table 2 Test results of laboratory scale batch process at 60°C with 6:1 molar ratio

Trial	Catalyst concentration (%)	Catalyst	Biodiesel yield (%)	Kinematic viscosity (Cst)	Specific gravity	Acid value	Carbon residue (%)	Ash content (%)	Cloud point (°C)
T_1	0.5	NaOH	92.34	5.711	0.873	0.493	0.05	0.011	6.3
T_2	1.0	NaOH	97.72	4.691	0.876	0.496	0.02	0.005	5.8
T_3	1.5	NaOH	75.07	5.664	0.878	0.793	0.06	0.020	6.5
T_4	0.5	KOH	91.84	5.906	0.871	0.507	0.07	0.015	6.6
T_5	1.0	KOH	97.38	4.950	0.874	0.499	0.04	0.007	5.8
T_6	1.5	KOH	73.29	5.776	0.879	0.809	0.06	0.024	7.1

Bubble-cap: Where, RT_2 - retention time of pre-reactor (min) (RT_2= 1 to 5 min) and V_3 - volume of pre-reactor (ml). Then, V3 = 50 mm, RT_2(max) = 5 min, and RT_2(min) = 1min; υl, = 1.3 l/kg; υv = 936.45 l/kg and assuming 50% methanol recovery, V_2 = 0.01779 m, h = 2 mm, r = 53.21 mm and assuming 5 caps in a plate, then r = 10.0 mm.

V_3, ml=RT_2Q

Evaporator: The temperature of evaporator is maintained typically above the boiling point of methanol. The residence time was maintained as 90-100 s (McGill University report), i.e. RT_3 =90 to 100 s

V_4, ml=RT_3Q

where, RT_3 - retention time (min), V_4 - working volume (ml), RT_3 =90 to 100 s, and V_4, ml=200

Decanter: Where, MV - molar volume (ml/mol), t - working time of plant (l/d), and V_5 - volume of decanter (ml), υl - specific volume of methanol in liquid state @ 60ºC (l/kg), υv - specific volume of methanol in vapour state @ 60ºC (l/kg), V2 - working volume of bubble-cap (m^3), r - radius of bubble-cap (mm) and $V_2 = \pi r^2 h$

Total mole, mol = Qt/MV_{oil}
MV, ml mol^{-1} = molar mass/density
V_5, ml = $Vol_{biodiesel} + Vol_{glycerol}$
$1\ mol_{oil} + 3\ mol_{methanol} \rightleftarrows 1\ mol_{glycerol} + 3\ mol_{biodiesel}$
MV = 714.08 ml mol^{-1}; Total mole =20.16 mol; $Vol_{biodiesel}$=20 478.6, $Vol_{glycerol}$ =1 474.1, V_5= 22 l

Evaluation of continuous transesterification plant

Methyl esters yield: The methyl esters yields in continuous process were studied and the results are presented in Table 3. In sieve tray reactive distillation column, the methyl ester yield varied from 66.75% to 93.75%, which depends on the reaction temperature, molar ratio and flow rate of methanol. The maximum was attained at a flow rate of 24 ml/min, 65ºC with molar ratio of 4:1. Similarly, the yield in bubble-cap tray ranged from 68.2% to 96.2%, and maximum was attained at the same parameters as mentioned above. Methyl esters yield increased with methanol supplied, reaction temperature, reaction time. The higher yield of biodiesel at increased methanol proportion may be attributed to the fact that excess quantity of methanol shifts the reaction to ester formation, as transesterification and esterification are reversible reactions (Yun *et al.* 2008). The methanol flow rate was higher (12 ml/min) with 4.5:1 molar ratio but reaction time (2.78 min) in reactive distillation column was lesser, it reduced methyl esters conversion rate. The optimum condition for methyl esters yield were 4:1 molar ratio, average reaction time of 5.55 min and methanol flow rate of 6 ml/min. Maximum conversions were obtained for large methanol amounts (Shashikant 2006). Each tray in a distillation column is designed to promote the contact between the vapour and liquid for better mass and heat transfer. Mass and heat transfer effected by varying flow rate. Ideally, the vapour and liquid leaving the stage are in equilibrium. Equilibrium is a function of the rate of mass and heat transfer between liquid and vapour in distillation and it has an effect on the efficiency of tray. In sieve tray, vapour flow through the tray deck to contact the liquid is controlled by the number and size of the perforations. This is in comparison with Singh *et al.* (2004) which reported that operating in reactive distillation system at 65ºC, with 4:1 molar produced maximum biodiesel. Higher molar ratios of methanol to oil led to greater conversion for a given reaction time (Freedman *et al.* 1984, Holser and Harry 2006, Leung and Guo 2006). In this present study, the maximum biodiesel yield found to be at 65°C with 4:1 molar ratio in 5.55 min average reaction time. Temperature influenced the reaction rate and higher yield was obtained at a higher temperature between 35 and 65°C. The conversion increased with reaction time. The first 15 min is the fastest period of the reaction, in which a conversion of 90% is possible and the optimal conditions were: 64°C with 1.3% KOH as catalyst (Zhou *et al.* 2006).

Effect of kinematic viscosity, specific gravity and acid value: The fuel properties such as kinematic viscosity, specific gravity and acid value were studied and results are presented in Table 4. The biodiesel obtained through sieve tray reactive distillation column had the viscosity in the range of 14.459 to 4.288 cSt, while that in bubble-cap tray ranged from 14.371 to 3.759 cSt, which indicated that the viscosity gradually decreased with the reaction temperature and also the conversion efficiency of the reactive distillation column. The maximum and minimum viscosity of the biodiesel was obtained at 57.5°C and 65°C respectively, with molar ratio of 4:1. The methyl esters of soybean oil had a viscosity between 4.2 and 4.6 cSt (Clements 1996). Specific gravity of a methyl ester depends on its molar mass, free fatty acid content, temperature and the mixing effect. Sieve tray reactive distillation column could be affected by the vapour to liquid density ratio due to which back mixing effect could occur, where the liquid carries the vapor downward through perforations. It led to reduction in reaction efficiency and product quality. But in the case of bubble-cap tray, there was no immediate direct contact between liquid and vapour phases, so there was no possibility of mixing effect. Owing to the decrease in free fatty acid content with

Table 3 Methyl esters yield in continuous transesterification process

Type of RD column	Flow rate (ml/min)	Molar ratio	Temperature (°C) 57.50	60.0	62.50	65.00
Sieve tray	6.6	3:1	66.75	76.5	83.50	88.00
	13.8	3.5:1	67.50	81.2	87.25	91.25
	24.0	4:1	68.50	85.0	90.25	93.75
	48.0	4.5:1	66.00	83.6	88.75	92.00
Bubble-cap tray	6.6	3:1	68.20	79.0	84.35	92.70
	13.8	3.5:1	70.25	82.5	89.96	94.32
	24.0	4:1	72.40	87.3	91.30	96.20
	48.0	4.5:1	71.60	84.6	90.76	95.65

Table 4 Effect of kinematic viscosity, specific gravity and acid value in continuous process

RD column	Temp (°C)	Molar ratio											
		3:1			3.5:1			4:1			4.5:1		
		Kinematic viscosity, Cst	Specific gravity	Acid value	Kinematic viscosity, Cst	Specific gravity	Acid value	Kinematic viscosity, Cst	Specific gravity	Acid value	Kinematic viscosity, Cst	Specific gravity	Acid value
Sieve tray	57.5	17.202	0.901	1.759	15.995	0.892	1.593	14.459	0.887	1.423	15.907	0.893	1.547
	60.0	11.175	0.884	1.156	10.186	0.882	1.024	9.754	0.880	1.010	12.745	0.881	1.288
	62.5	9.438	0.890	0.951	9.059	0.885	0.907	8.618	0.882	0.862	8.905	0.884	0.884
	65.0	6.242	0.885	0.637	5.277	0.878	0.524	4.288	0.873	0.412	4.664	0.871	0.422
Bubble- cap tray	57.5	15.900	0.893	1.580	14.831	0.889	1.478	14.371	0.886	1.415	14.984	0.888	1.506
	600	9.867	0.873	0.899	9.565	0.871	0.802	8.997	0.869	0.757	9.211	0.871	0.835
	62.5	8.791	0.869	0.869	7.274	0.867	0.733	6.939	0.865	0.697	7.052	0.867	0.714
	65.0	5.643	0.871	0.576	4.745	0.868	0.464	3.759	0.866	0.384	3.831	0.866	0.387

increase in reaction temperature, the acid value varies between 1.413 to 0.384 in bubble-cap tray and 1.423 to 0.412 in sieve tray reactive distillation column. This goes in line with the observation of Muthu *et al*. (2010) and Cvengros and Cvergrosova (2004) where, acid value reduced at higher temperatures. Muthu *et al*. (2010) reported that the optimal acid value conversion was achieved using 1% (wt.) catalyst with a methanol-to-oil molar ratio of 9:1, temperature of 65°C and reaction time of 2 h. In sieve tray reactive distillation column, the acid value at 57.5°C was 1.759 with 3:1 molar ratio, 1.593 with 3.5:1 molar ratio, 1.423 with 4:1 molar ratio and 1.547 with 4.5:1 molar ratio. At 60°C, the kinematic viscosity was 1.156, 1.024, 1.010 and 1.288 with 3:1, 3.5:1, 4:1 and 4.5:1, respectively. In the same manner, bubble-cap tray reactive distillation column recorded the acid value at 57.5°C was 1.580 with 3:1 molar ratio, 1.478 with 3.5:1 molar ratio, 1.415 with 4:1 molar ratio and 1.506 with 4.5:1 molar ratio. At 60°C, the acid value was 0.899, 0.802, 0.757 and 0.835 with 3:1, 3.5:1, 4:1 and 4.5:1, respectively. The acid number of biodiesel can be used to indicate the content of free fatty acids of the fuel and found that the acid number of a biodiesel increases 3 mg KOH/g/ 1% (wt.) water content in its raw oil (Cvengros and Cvengrosova 2004).

Effect of carbon residue and ash content: The fuel properties such as carbon residue and ash content were studied and results are presented in Table 5. The carbon residue of the methyl ester from sieve tray reactive distillation column had values of 0.13, 0.09, 0.06 and 0.03 at 57.5°C, 60°C, 62.5°C and 65°C respectively, with molar ratio of 4:1. Meanwhile, the methyl ester from bubble-cap tray had carbon residues of 0.11, 0.06, 0.05 and 0.02 respectively at 57.5°C, 60°C, 62.5°C and 65°C with the same molar ratio. The carbon content of methyl esters obtained from sieve tray reactive distillation column was high when compared to bubble-cap tray because, the carbon content was directly proportional to the reaction conversion efficiency. Hence, when the conversion efficiency is low, it led to formation of triglycerides which had the effect on the carbon residue in the methyl ester (Harvey, 2003). The results indicated that the ash content was in the range of 0.139 to 0.008%. It varied with carbon residues. Fernando *et al*. 2007 showed that the carbon residue increased when concentrations of residual triglycerides reached up to approximately 10% (wt.). 0.104, 0.057, 0.048 and 0.013 were the ash contents of the methyl esters obtained from sieve tray reactive distillation column with molar ration of 4:1 at temperatures 57.5°C, 60°C, 62.5°C and 65°C respectively. Still, the ash content of the methyl ester with same molar ratio from bubble-cap tray reactive distillation column has differences and the range from 0.092 to 0.008 between temperatures of 57.5°C and 65°C.

Effect of cloud point on continuous transesterification process: The fuel property cloud point was studied. The result indicated that the cloud point was in the range of 4.3 to 16°C. The cloud point was in the acceptable limit (4.6°C and 4.3°C) at high conversion rate (65°C and 4:1 molar

Table 5 Effect of carbon residue and ash content in continuous process

Type of RD column	Temperature (°C)	Molar ratio							
		3:1		3.5:1		4:1		4.5:1	
		Carbon residue, %	Ash content, %	Carbon residue, %	Ash content, %	Carbon residue, %	Ash content, %	Carbon residue, %	Ash content, %
Sieve tray	57.5	0.16	0.139	0.14	0.122	0.13	0.104	0.14	0.119
	60.0	0.09	0.072	0.08	0.061	0.09	0.057	0.12	0.087
	62.5	0.08	0.055	0.07	0.051	0.06	0.048	0.08	0.049
	65.0	0.06	0.027	0.04	0.017	0.03	0.013	0.04	0.014
Bubble-cap tray	57.5	0.15	0.117	0.13	0.105	0.11	0.092	0.12	0.110
	60.0	0.08	0.053	0.07	0.040	0.06	0.031	0.07	0.052
	62.5	0.07	0.046	0.06	0.033	0.05	0.028	0.06	0.031
	65.0	0.05	0.026	0.03	0.014	0.02	0.008	0.03	0.010

ratio). A thermodynamic study was made by Imahara *et al.* (2006) and found that the cloud point of biodiesel could be determined only by the amount of saturated fatty acid methyl esters regardless of composition of unsaturated esters.

Excess alcohol: The excess alcohol amount were measured and presented in Fig 5. The excess alcohol in the sieve tray reactive distillation column was between 0.3ml to 15ml at 57.5°C and 65°C, respectively, but with different molar ratio of 3:1 and 4.5:1, respectively. On observing the bubble-cap tray reactive distillation column, the excess alcohol was in-between 0.7 ml to 18.2 ml with the same parameters as mentioned above. The result indicated that the excess alcohol varies in the range of 0.3 to 18.2 ml. Bubble-cap had a positive liquid seal, therefore could operate efficiently at very low vapor rates. Sieve tray relied on the flow of vapor through the holes to hold the liquid on the tray and cannot operate at very low vapor rates. It reduces the mass transfer rate, and hence the conversion efficiency was low. With increase in methanol flow rate, i.e. molar ratio, and at temperature of 65°C, the excess alcohol was high as the boiling point of methanol is 64.5ºC.

Statistical analysis

The experimental data obtained from continuous transesterification process using two types of reactive distillation column, namely, sieve tray and bubble-cap tray were analyzed using regression analysis by Statistical Package for the Social Sciences (SPSS). Models were developed to describe the relationships between the predicted response variable (methyl esters yield) and the independent variables (methanol-to-oil ratio and reactive distillation column temperature). The statistical analysis indicated the predicted R^2 value was 0.865 and the molar ratio of methanol-to-oil and temperature were the significant factors,10% and 1%, respectively, on the yield of methyl esters in sieve tray reactive distillation column. The empirical relationship obtained for methyl ester yield (y) by simple

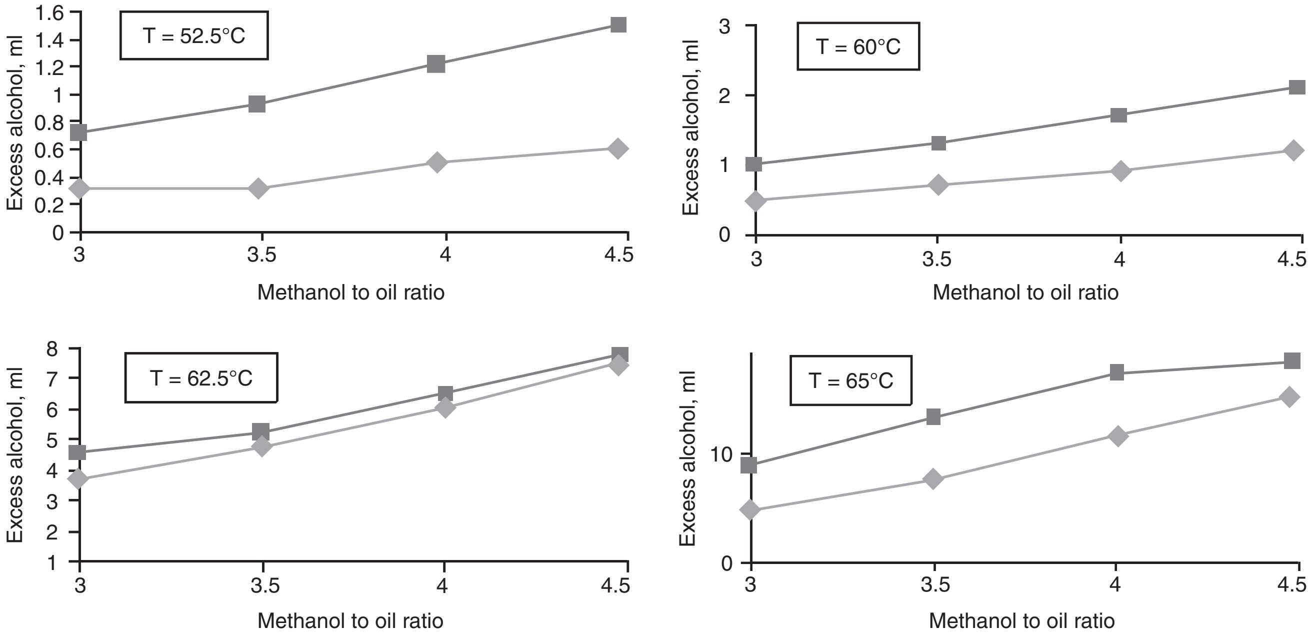

Fig 5 Excess alcohol in continuous transesterification process

Table 6 Electrical energy consumption in continuous transesterification plant

Component	Capacity (W)	Run time (h)	Electrical energy (Wh)
Mechanical stirrer	50	8	400
Hot plate heater	500	8	4000
RD column heating coil	1000	8	8000
Heating mantle	150	8	1200

Table 7 Energy analysis of continuous transesterification unit

	Particular	Energy content (MJ)	Total energy (MJ)	Loss (MJ)
Energy input	Coconut oil	325.555	392.08	45.995
	Methanol	65.376		
	NaOH	1.149		
Energy output	Biodiesel	291.2	346.085	
	Glycerol	53.325		
	Excess methanol	1.56		

linear regression analysis is,

$$y = 3.12a + 2.86b - 120.07$$

where, a – temperature (°C), b – molar ratio (methanol-to-oil).

The equation indicated that there is an increase of 1% of methyl ester yield with every increase of 3.12°C temperature and 2.86 units of molar ratio (methanol-to-oil). In case of bubble-cap tray reactive distillation column, the predicted R^2 value was 0.922. The molar ratio of methanol-to-oil (5%) and temperature (1%) were the significant factors on the yield of methyl esters which confirmed the validity of the predicted model. The empirical relationship indicated that there is an increase of 1% of methyl ester yield with every increase of 3.12°C temperature and 3.26 units of molar ratio (methanol-to-oil). From this analysis, the bubble-cap tray reactive distillation column is more reliable than sieve tray reactive distillation column.

$$y = 3.12a + 3.26b - 119.03$$

Energy analysis

The electrical energy consumed by the continuous transesterification plant was analyzed (Table 6 and Table 7). The electrical energy consumed in the continuous transesterification plant per day of 20 l biodiesel production was worked out as 13.6 kWh.

Economic evaluation

Capacity of the continuous transesterification plant was 20 l/day. The plant will be operated, for 8 h in a day. The processing cost of biodiesel production in continuous transesterification plant was worked out as ₹ 26.03/litre as on April 2015.

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