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Biodiesel Production of *Capparis Spinosa* Oil via Trans-Esterification Reaction by Using NaOH Catalyst and Its Pilot Synthesis Design

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ABSTRACT

Energy obtained from renewable sources has increased its participation in the energy matrix worldwide, and it is expected to maintain this tendency. Both in large and small scales, there have been numerous developments and research with the aim of generating fuels and energy using different raw materials such as alternative crops, algae and waste cooking oil. *Capparis spinosa* seed (containing 32% triglycerides) is used as an excellent source for biodiesel production. In this work, biodiesel (Fatty acid methyl ester) is produced by trans-esterification of *Capparis spinosa* oil, in the presence of methanol and sodium hydroxide as an alkaline catalyst. According to data collection, pilot of biodiesel production is simulated by using HYSYS v3.2 software. The yield of biodiesel in the presence of sodium hydroxide was considered 94%. The results of FTIR and GC-MS spectra showed trans-esterification reaction has been complete. According to the results, the produced biodiesel is in the range of ASTM Standard.

1. INTRODUCTION

From Industrial Revolution began in the late 18th century and early 19th century, energy is considered essential for economic growth and promotion of human life. By countries industrialization, the needs for fuel and energy is increasing day by day, so to provide energy and getting rid of the environmental impact of fossil fuels, these fuels should be replaced by other renewable fuels [1, 2]. So far gasoline and diesel are the most common fuel in transport section, in many countries, including Iran [3]. Cars that use gasoline or diesel fuel, release hazardous substances with complex chemical compounds [4]. Although several methods to reduce pollution, including vehicle inspection programs or installation of automotive exhaust emission control systems used in developed countries, these programs in large cities, has not reduced pollutants emissions sufficiently. Since the first generation of fuel and chemicals obtained from biomass (e.g., bioethanol from feed grain) has many shortcomings, much attention around the world is being given to developing subsequent generations of biofuel and chemicals from lignocellulose wood and plant biomass, which has no nutritional value for humans [5, 6]. Among the various renewable fuels, biofuels have the ideal conditions to be consumed and produced in both gas and liquid forms. Biodiesel is considered the most common and the most widely used biofuels. Biodiesel is renewable, sustainable, biodegradable, and emits low greenhouse gases [7, 8]. The primary sources of biofuels can be wood waste, agricultural waste, sugar cane, cereals, and vegetables oil. Every year, nearly 40 billion tons of biomass is produced but only 200 million tons are used [9]. To produce biodiesel, the feedstock can be classified into three main groups: vegetable oils (edible and non-edible), animal fats and waste oils. In addition, algae have been introduced in recent years as Group IV [4, 10]. High production costs of biodiesel are caused mostly by the cost of raw materials, whose share in the cost exceeds 80%, which implies that the production cost can be significantly lowered if the raw material or part of it would be replaced by a suitable alternative raw material [11, 12]. There are more than 4,000 plants on earth from which oil can be obtained [13]. Depending on weather conditions, various types of plants are grown for biodiesel production. Plant oils such as canola in Canada, sunflower in Europe, soybeans in America,

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palm kernel in Southeast Asia, Coconut oil in the Philippines are used to produce biodiesel [14, 16].

Fatty acid methyl esters (Biodiesel) have been found favorable to be used as a blend component of petro-diesel fuel due to lack of aromatics, negligible sulfur content, higher lubricity and very high cetane values [17- 19]. In developing countries the importance of fuels and oils from plant sources is growing. Thus *Capparis spinosa* plant is native plant in Iran and the seed of this plant comparing to others, contains significant amounts of oil. The seed oil of *Capparis spinosa* plant is non-edible, hence it is valuable in the industrial position. Noticing the agricultural industry and oilseeds cultivation plants compatible with the climate of our country, is another benefit of this plant for the production of biodiesel [20, 21]. *Capparis spinosa* is the scientific name of this plant, while its local name is "Caper". It is vernacular of the tropical West and Central Asia, these seeds contain 30-35% oil, which can be a notable source for biodiesel production. *Capparis spinosa* seed contains 19-22% protein, 26% fiber and 30- 35% oil. Major saturated fatty acids of *Capparis spinosa oil* are Linoleic acid (42-51%), Oleic acid (40-46%), Palmitic acid (3%), Palmitic acid (12%), and stearic acid (2%) [22].

There are several methods to produce and apply biodiesel: direct use of vegetable oils, micro-emulsions [23], thermal cracking (pyrolysis) [24] and trans-esterification [25]. Direct use of vegetable oils is not applicable to most of actual diesel engines, as the high viscosity would damage the engine. Biodiesel obtained from micro-emulsion and thermal cracking methods would lead to incomplete combustion due to a low cetane number. Trans-esterification is the most common method for biodiesel production due to its simplicity and it has been widely studied and industrially used to convert vegetable oil into biodiesel [26]. The most widely used industrial method for the commercial production of biodiesel from vegetable oils/fats is a base catalyzed trans-esterification process using KOH or NaOH as the homogeneous catalyst and MeOH as the lower alcohol (Figure 1.) [27, 28].

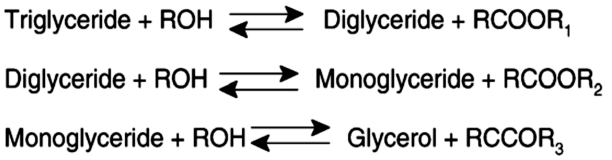

Figure 1. Trans-esterification reaction steps [29].

In this study, oil was extracted from *Capparis spinosa* seed and trans-esterification reaction was carried out in the presence of methanol and sodium hydroxide. Different molar ratios of oil in methanol (1:4, 1:6, and 1:8) were tested, resulting in 1: 6 was chosen as the optimum molar ratio. In addition, the catalyst 1% by weight of oil was used in the reaction. FTIR and GC-MS spectra of biodiesel were taken; also some fuel standards for biodiesel were measured. Finally, biodiesel production in pilot scale was simulated with HYSYS v3.2.

2. MATRIALS AND METHODS

2.1. Materials and equipments Sodium sulfate (Na_2SO_4) (99%), Phenolphthalein ($C_{20}H_{14}O_4$), Isopropyl alcohol, Sodium hydroxide 99% (NaOH), N-Hexane, Methanol (99%) all were supplied by German company Merck. *Capparis spinosa* supplied from Kazeroon city in Fars province of Iran.

GC-MS analysis was performed on 2200/3800 varian, Australia, column: VF-5Ms, flow rate: 1ml/min, injection temp: 28^{O}C, Mntel 1000ml, Centrifuge Kokusan H-108N made by Tokio, RLABINCO Stirrer M-81, Rotary meter Stero Glass by 202/201, made by S.R.L italian. Rotary evaporator instruments by Kentron, Strike 202.

2.2. Extraction oil from *Capparis spinosa* For this purpose, Soxhlet extractor, which is an oil extraction system, and normal hexane solvent were used. In this way 200 grams of *Capparis spinosa* seed was put into a 1000 ml flask, 500 ml of Normal Hexane was added and the solution was left for 5 hours so that the chemical reactions complete. Finally solvent was extracted using a distiller to achieve the pure oil (64 grams).

2.3. Biodiesel production by trans-esterification of *capparis spinosa* oil 50 grams of *Capparis spinosa* is put into a flask and 1 gram of sodium sulfate (Na_2SO_4) is added to water absorption of oil and the flask is placed on a heater stirrer for 20 minutes to be stirred at 60º C. Then sodium sulfate is separated from oil. Afterwards, 10 grams of methanol and 0.5 grams of catalyst (NaOH) are added. After that we'll put the packed flask on a heater stirrer at 60 ºC and 600 rpm of stirrer for a period of 3 hours. Then the product is poured into a decanter to separate Glycerol from biodiesel (Estric phase). Glycerol was in the lower part and biodiesel in the higher part due to their density.

Obtained biodiesel is purified from catalyst, methanol and glycerol three times using 10% volumic hot water (60-70 ºC) leeching. 47 grams of biodiesel is coughed which is 94% of the oil weight.

According to the calculations and measurements of fatty acids of *Capparis spinosa* oil, because acid number is less than 1, so there is no need to carry out esterification and thus, directly trans-esterification reaction was done. The type of catalyst is determined, according to the free fatty acid content. In this study, the homogeneous catalyst of sodium hydroxide base is used which is a phase in this reaction, in addition side reaction (Saponification) does not occur.

3. RESULTS AND DISCUSSION

Table 1. shows that in order to achieve efficiency the molar ratios of 1:8 and 1:6 should be used more than 90%. The molar ratio of 1:8 consumed high content of methanol and no significant increase efficiency, so the molar ratio of 1:6 is more affordable and was selected to the pilot design. The ratios of the compounds in the pilot simulation as same as laboratory optimum ratios were considered.

3.1. Biodiesel characteristics Properties of biodiesel are evaluated by fuel standard test, and the results are compared with ASTM D6751 standards. Freeze point (FP), Viscosity (VS), Flash point (FP), Density (DN), Pour point (PP), and Cloud point (CP) of fatty acid methyl esters of *Capparis Spinosa* seed oil were empirically determined. Biodiesel was compared to ASTM standards and the result indicated in Table 2. Biodiesel characteristics reveal that biodiesel is in permissible range.

TABLE 1. Conditions of experiments

No.	Amount Catalyst (wt. %)	Oil/ Methanol ratio (mol/mol)	Yield of biodiesel (%)	Number of repetitions
1	1	1:4	83	3
2	0.5	1:4	80	3
3	1	1:6	94	3
4	0.5	1:6	90	3
5	1	1:8	95	3
6	0.5	1:8	94	3

3.2. Biodiesel FTIR spectra We used FTIR spectra to ensure the reaction progress and triglyceride conversion to biodiesel (methyl ester), by using a NICOLET 8700 Thermo scientific, USA spectrophotometer.

By comparing oil FTIR with biodiesel FTIR of *Capparis spinosa* in Figures 2. and 3. one can assume that spectra are similar to each other and only in some cases (the fingerprint area) are different. Most of the differences are related to the area of 1500-1000 cm^{-1}. The only change that occurs is because of the reaction in the oil due to glycerin out and replaced methanol in the hydrocarbon chain.

In triglycerides, groups O-CH_2 and O-CH exist. Peak of 1163 cm^{-1} and 722cm^{-1} in the triglycerides spectrum belonging to the tensional vibration of CO stretching group attached to -CH_2 .This turns to 1171 cm^{-1} peak in methyl ester a drive from tensional vibration of CO group which is linked to -CH_3. Also, the 1197 cm^{-1} and 1436 cm^{-1} peak belongs to the bending vibration and swinging O-CH_3, which does not exist in the triglycerides spectra. Typical absorptions for acylglycerols were detected at 3006 cm^{-1} (C–H olefins), 2924-2854 cm^{-1} (aliphatic C–H stretching), 1464 cm^{-1} (aliphatic C–H bending).

TABLE 2. Characteristics of oil-derived biodiesel from *Capparis spinosa* in the presence of NaOH

PROPERTY	ASTM METHOD D6751	BIODIESEL
Density (g/cm^3)		0.88
Viscosity (mm^2/s)	1.9- 6	4.3
Flash point ^oC	<130	167
Freeze point ^oC		-10
Pour point ^oC		-8
Cloud point ^oC		-2

The biodiesel impurities include FFA, alcohol, water, mono glyceride and di glyceride. All of them have OH functional group which displayed a peak at 3200-3500 cm^{-1}, so the absence of this peak indicates the purity of the biodiesel. Peak at 1743 cm^{-1} is related to C=O functional group in methyl esters. Peaks at 1150- 1350 cm^{-1} are related to the torsional vibrations of CH_2 groups. Figure 3. shows the reaction progress in kinetics point of view.

After the trans-esterification reaction, separation of phases and purification of samples produced, FTIR spectra were taken, to ensure to perfect synthesis of methyl esters were compared with the FTIR spectrum of methyl stearate as standard. (Figure 4.)

FT-IR spectra of biodiesel are almost similar to the spectrum of methyl stearate (standard), and the only difference in the 1654 cm^{-1} and 3006 cm^{-1} region that methyl stearate does not have group C = C , so there is no peak in this area. Considering that perfectly matches the spectrum obtained from biodiesel with methyl stearate can be concluded that biodiesel synthesis is completely done.

3.3. GC chromatogram analyses GC-MS analysis was performed to characterize methyl esters in biodiesel. Figure 5. shows oil-derived biodiesel of *Capparis spinosa* GC spectrum. Fatty acid methyl esters are recognized in GC Spectrum which is shown in Table 3.

According to GC spectrum of biodiesel, sharp peaks are in 21 and 22.6 minutes the area taken by MS of these two kinds and compared with the standard of fatty acid, understanding that methyl linoleate and methyl oleate have the highest fatty acid in *Capparis spinosa* oil. And according to Table 3. methyl linoleate and methyl oleate have the highest fatty acids in *Capparis spinosa* oil [20, 21].

PFD of biodiesel production was drawn in Figure 6. In the input of fluid package as the general NRTL in this pilot design, it's been tried to use all substances in the environmental condition to cut down on expenses. (In 4 to 6 Tables are shown)

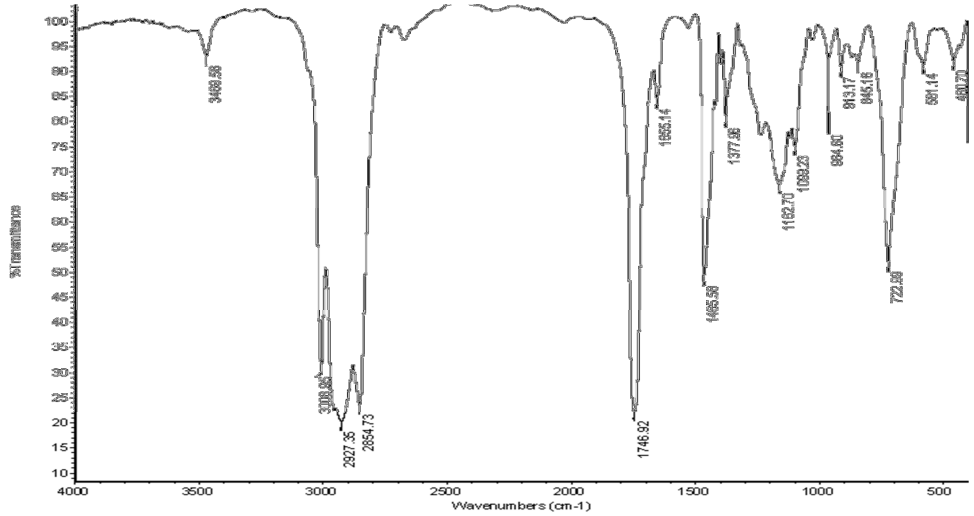

Figure 2. FTIR spectrum of *Capparis spinosa* oil

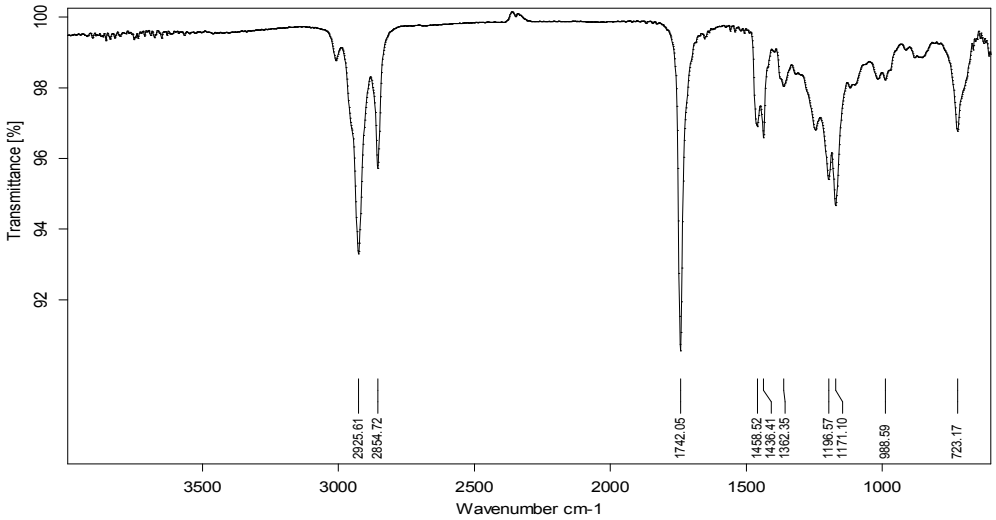

Figure 3. FTIR spectrum of biodiesel

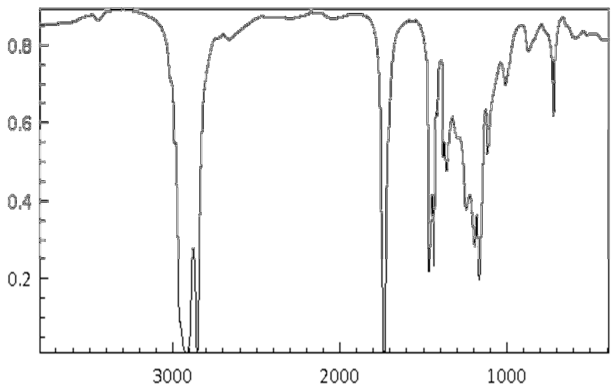

Figure 4. FTIR spectrum of Metyl Stearate

Trigly flow which its Characteristics are mentioned, enter heater E-100. This is for preheating of Ingredients for better reactions. E-100 output flow is TG flow which is heated up to 60º C. This flow directly enters reactor. Methanol flow consists of two parts: f-Me flow which is fresh methanol and cool "returning methanol from distilling column which is cooled down to the temperature of fresh methanol".

Based on the fact that these two flows have the same properties, they can be mixed and enter the reaction.

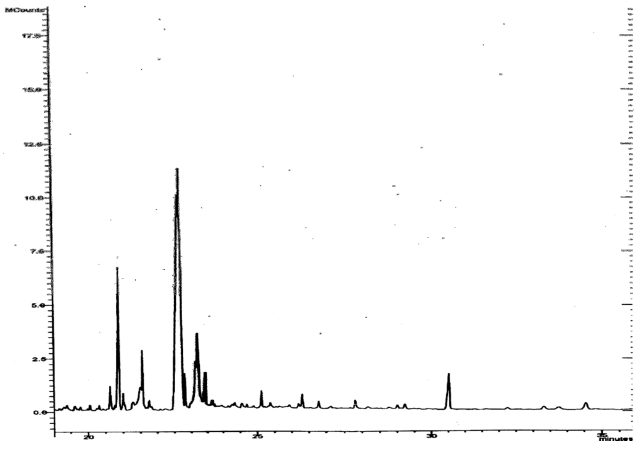

Figure 5. GC spectrum of biodiesel production

TABLE 3. Percentage of fatty acids in oil by GC-MS spectra

METHYL ESTER	PERCENT (%)
Methyl linoleate (C18:2)	42 – 45
Methyl oleat (C18:1)	44 – 48
Methyl palmitoleate (C16:1)	3
Methyl palmitate (C16:0)	10
Methyl stearate (C18:0)	2
Other	1>

TABLE 4. Input Oil Characteristics

STREAM NAME	TRIGLY
Temperature (°C)	30
Pressure (bar)	1
Molecular weight	282.5
Mass flow (Kg/h)	208.3
Density (Kg/m^3)	881.3
Kinematic viscosity (cSt)	31.67

TABEL 5. Input alcohol Characteristics

STREAM NAME	F-ME
Temperature (°C)	30
Pressure (bar)	1
Molecular weight	32.04
Mass flow (Kg/h)	45.83
Density (Kg/m^3)	780.8
Kinematic viscosity (cSt)	0.6508

TABLE 6. Input catalyst Characteristics

STREAM NAME	NaOH
Temperature (°C)	30
Pressure (bar)	1
Molecular weight	40
Mass flow (Kg/h)	1.563
Density (Kg/m^3)	1465
Kinematic viscosity (cSt)	0.09877

This flow which is called methanol flow is the output MIX-101 which is sent to MIX-101 strier. Catalyst flow, in this case sodium hydroxide (NaOH), is MIX-101's other input flow. The product of combination of methanol and sodium hydroxide exits MIX stirrer and enters the reactor to react with the preheated oil. Reactive ERV-100 is an equilibrium reactor which is selected due to the reaction which is an equilibrium. This reactor, with the volume of 3 m^3, performs trans–esterification reaction. Q reactor heat flow is used to keep the temperature on 60º C which will not cost a lot because the trans–esterification reaction is an exothermic reaction. In addition to the output flow of the reaction, a gas flow goes out from the top of the reactor, called gas1 flow. This flow is prepared to avoid accumulation of alcohol gases in the reactor to prevent from explosion. Mixture flow, which is reaction flow, exits reactor ERV-100 and its pressure is increased up to 1.5 bars with a pump. Feed tower flow which its pressure has been increased, is pumped to the distillation column to extract excess methanol and avoid its waste. Distillation column T-100 separates methanol from the reaction mixture. Distillation column condenses the methanol flow and gives it back in the liquid form. The pressure difference inside the column is considered to be 0.2 bar. Input feed of the column enters it from the middle.

Product flow, exiting the column from below, is 92 ºC so as not to have any methanol, but for extraction of glycerol it's better to reduce temperature. Accordingly, cooler E-102 is used to reduce the temperature to 30º C. In other words, cool-RB flow and product flow have the same composition, merely temperature has been decreased. This flow is transferred to Separator V-100 in order to separate glycerol and biodiesel phases from each other. Separator V-100 sends out glycerol from below and biodiesel from above. Gas 2 flow is for avoiding accumulation of vapors.

Glycerol flow mainly consists of glycerol and impurities like sodium hydroxide, biodiesel and etc. This flow, as the trans-esterification byproduct, can be purified and sold. Raw BIO flow, exiting for output V-100 is impure biodiesel, in fact; although it's main part is methyl ester (biodiesel), it's impurities which makes it useless.

To achieve pure biodiesel, raw BIO flow must be purified. To do so, water leaching is the best solution. Based on the fact that all impurities are water-soluble, they can be extracted by water leaching and obtain pure biodiesel. By pumping impure biodiesel to washing column T-101, on the one hand, and water flow entering the column, on the other hand purification process is done. Biodiesel flow which is pure is now available. Waste water flow is the effluent in which impurities are solved so it should be transferred to purification section to be recycled.

MIX-102 machine combines all gas flows and sends them to purge section to be burnt and removed from the system. Purge system prevents form gas accumulations and explosions.

Biodiesel production with mass flow rate of 195.8 Kg/h of leaving the process compared to the entrance mass flow rate of oil represents 94% efficiency.

4. CONCLUSION

Biodiesel was produced form oil then oil extracted form *Capparis spinosa* via trans-esterification using sodium hydroxide (NaOH) as the catalyst. Biodiesel production was carried out with different percentage of catalyst (0.5 and 1 wt% of oil) and different molar ratios of oil to methanol (1:4, 1:6, and 1:8). Optimal efficiency was 94% and studies showed that by increasing the reaction time no significant progress was achieved. The ratios of

the compounds in the pilot simulation as same as laboratory optimum ratios were considered. Biodiesel production pilot design was performed by HYSYS v3.2. This pilot can produce 5 tons of biodiesel daily. Oil-derived biodiesel produced from *Capparis spinosa* oil in the presence of biodiesel was compared to ASTM standards and the result indicated that this method is in permissible range.

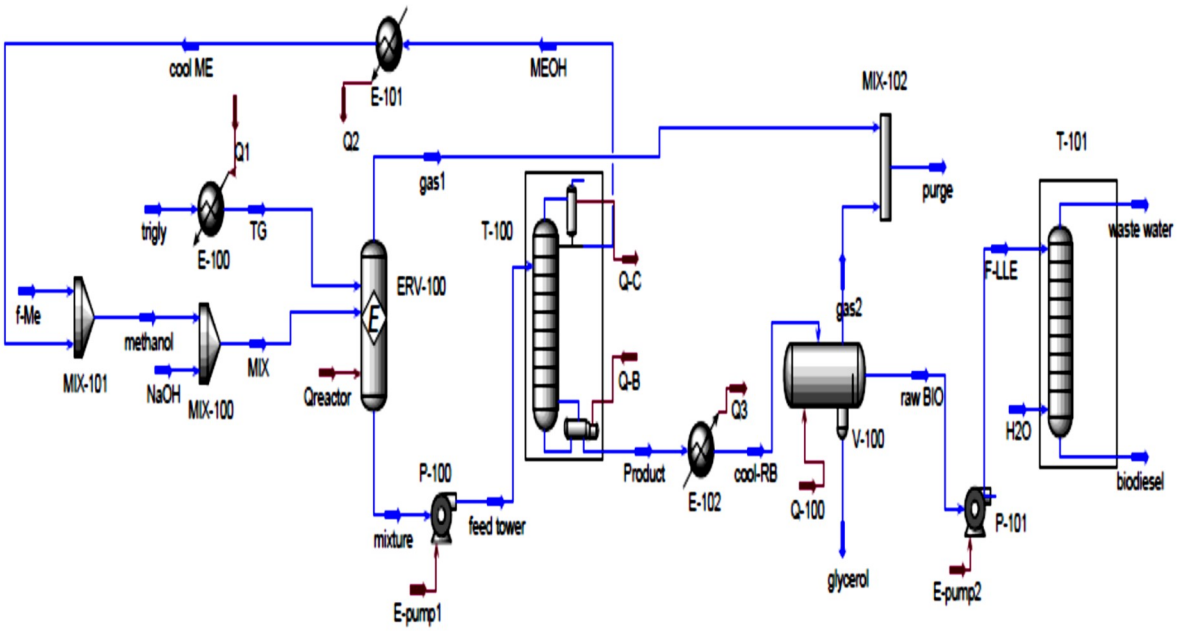

Figure 6. PFD of oil-derived biodiesel from *Capparis spinosa*

5. ACKNOWLEDGEMENT

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