https://doi.org/10.30799/jacs.196.18040401

J. Adv. Chem. Sci. – Volume 4 Issue 4 (2018) 593–597

ISSN: 2394-5311

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Methanolysis of Low FFA Waste Vegetable Oil using Homogeneous Base Catalyst for Biodiesel Production: New Process Design

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ARTICLE DETAILS

Article history:
Received 13 October 2018
Accepted 30 October 2018
Available online 21 November 2018

Keywords:
Waste Vegetable Oil
Biodiesel
Process Design
Simulation

ABSTRACT

Biodiesel is an alternative liquid biofuel consists of a mixture of methyl or ethyl esters. It can be produced through transesterification where low molecular weight alcohol (e.g. methanol or ethanol) reacts with lipid or fat (triglyceride) to produce biodiesel (fatty acid methyl ester FAME) and glycerol as a valuable byproduct. The most common method of biodiesel production is base catalyzed transesterification where alkaline material such as potassium hydroxide is used as a catalyst. This paper presents a suggested new process design of a biodiesel plant that treats about 24 tons/ day of low free fatty acids, FFA, waste vegetable oil and WVO.

1. Introduction

Recently, different schemes were suggested for biodiesel production through various processes, e.g. homogeneous base catalyzed transesterification, two-step process, heterogeneous catalyzed transesterification, etc., [1-4]. Sajid et al. [5] implemented a probabilistic economic risk analysis of biodiesel production through alkali catalyzed transesterification. The process description showed that after reaction between jatropha oil and methanol in presence of NaOH, the products are transferred to a separation tank where ester and glycerol layers can be separated by means of gravity. Methanol associated with the ester layer is removed using a falling film evaporator then the partially purified biodiesel is directed to a liquid-liquid extraction unit to be washed by water in order to remove any residues of methanol, glycerol and NaOH. Finally, the moisture content is eliminated from crude biodiesel in a flash evaporator to produce pure biodiesel. After decantation, crude glycerol is neutralized by HCl to remove the remaining catalyst and convert it to salt. In addition, soaps are converted to free fatty acids that can be easily separated in a splitter. The final step is to evaporate the methanol to obtain pure glycerol. Aside from this study, Sajid et al. [6] developed another continuous two-step process to produce biodiesel from waste vegetable oil at a rate of 45000 ton per year. In this process, the oil is first treated through acid catalyzed esterification in order of decreasing the free fatty acids content to below 2% and then transesterified with methanol underutilization of an alkali as a homogeneous catalyst. Process flow diagram showed that after reaction and two layers separation, the ester layer is first flash vaporized for methanol removal, which is then recycled, then the product is washed by warm water which is heated by the hot cooling water goes out from the methanol cooler after flash vaporizer as a figure of heat integration. Water droplets associated with washed biodiesel are eliminated using a flash vaporizer and the purified product is then directed to a storage tank. Crude glycerol passes by two purification steps, i.e. neutralization for catalyst removal and vaporization for methanol removal. Pretreatment process consists of six steps. The first one is to filter the oil to remove any solid particles then the oil is reacted with methanol in presence of sulfuric acid. Stream from esterification step is washed with glycerol in order to remove sulfuric acid. To recover methanol used in this process, the bottom glycerol stream from the glycerol washer is distilled then methanol can be recycled. Glycerol-sulfuric acid stream is neutralized by calcium oxide and the produced calcium sulfate separated whereas the other methanol-glycerol stream is directed to an evaporator to remove methanol, while glycerol is then recycled.

Some other works compared between different processes of production from economical and technical prospects [7-10]. Gaurav et al. [11] introduced a catalytic distillation scheme for bio diesel production and compared it with the conventional alkali process. In the case of conventional process, they suggested a scheme for a continuous biodiesel production process where a plug flow reactor is utilized for the reaction step followed by a flash separator, decanter and a set of distillation columns to remove and recycle excess methanol without usage of any washing steps. On the other hand, the catalytic distillation process consists of a reactive distillation column as the main component of this continuous process followed by a decanter and set of distillation columns for biodiesel and glycerol purification. It was found that the new process, i.e. catalytic distillation, reduced the fixed and operating costs significantly which may make the process of biodiesel production more economical and feasible. Glisic et al. [12] presented techno-economic analysis of green diesel production from waste vegetable oil by three processes, conventional process, supercritical methanol process and waste vegetable oil hydrogenation process; similar process designs for both conventional alkali catalyzed transesterification and supercritical methanol process were suggested by Lee et al. [13]. The process mode was continuous for all the suggested schemes. In the conventional process the type of the reactor was CSTR while in the supercritical methanol method the reactor was a plug flow one. From the process flow diagrams for the conventional method of the two studies it can be observed that the resulted reaction mixture is introduced to a distillation column directly to recover excess methanol. Water washing is then applied to dissolve glycerol and alkali homogeneous catalyst in a liquid-liquid extraction column where the top stream, which consists of esters, oil and traces of methanol and water, is then distilled to obtain three streams of almost pure biodiesel, oil and a vent stream of water-methanol mixture. The bottom stream of the extractor is acidulated with phosphoric acid for catalyst removal to produce a good fertilizer. Water-methanol-glycerol mixture enters a distillation column to separate methanol and water in order to obtain pure glycerol. Supercritical alcohol method consists of a plug flow reactor in which non-catalytic transesterification takes place followed by a set of distillation column in addition to a decanter to separate different products, i.e. biodiesel and glycerol, besides unreacted reactants, i.e. unreacted oil

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https://doi.org/10.30799/jacs.196.18040401

Cite this Article as: E.G. Al-Sakkari, S.T. El-Sheltawy, A. Soliman, I. Ismail, Methanolysis of low FFA waste vegetable oil using homogeneous base catalyst for biodiesel production: New process design, J. Adv. Chem. Sci. 4(4) (2018) 593–597.

and excess methanol, as well. In another research study, Chilev and Simeonov 2014 [14], simulated a process for biodiesel production from vegetable oils through alkali catalyzed transesterification similar to the one described by Glisic et al. 2016 [12] and Lee et al. [13]. However, this later process contained some figures of task integration, mainly heat integration, such as heating biodiesel-unreacted oil stream and stream of reactants entering the reactor with the hot biodiesel stream leaving biodiesel-unreacted oil distillation column. Another example of heat integration is the heat exchange between washing water, reaction mixture leaving reactor and the bottom product of the methanol recovery distillation column which is positioned directly after the reactor. Excess methanol and unreacted oil are recycled back to the process in order of decreasing materials loss. This paper presents a suggested new process design for biodiesel production plant that treats about 24 tons/day of low free fatty acids FFA waste vegetable oil WVO. The novelty of this process is the adequate use of task integration concepts that can lead to obtain a feasible process for biodiesel production. Moreover, the comparison with different alternatives and previously stated processes gave the ability to achieve the optimum design from the point of view of both technicality (e.g. task integration, energy efficiency, etc.) and economy.

2. Process Design

The suggested process for biodiesel production has been discussed in this section. Fig. 1 illustrates the flow sheet of this process.

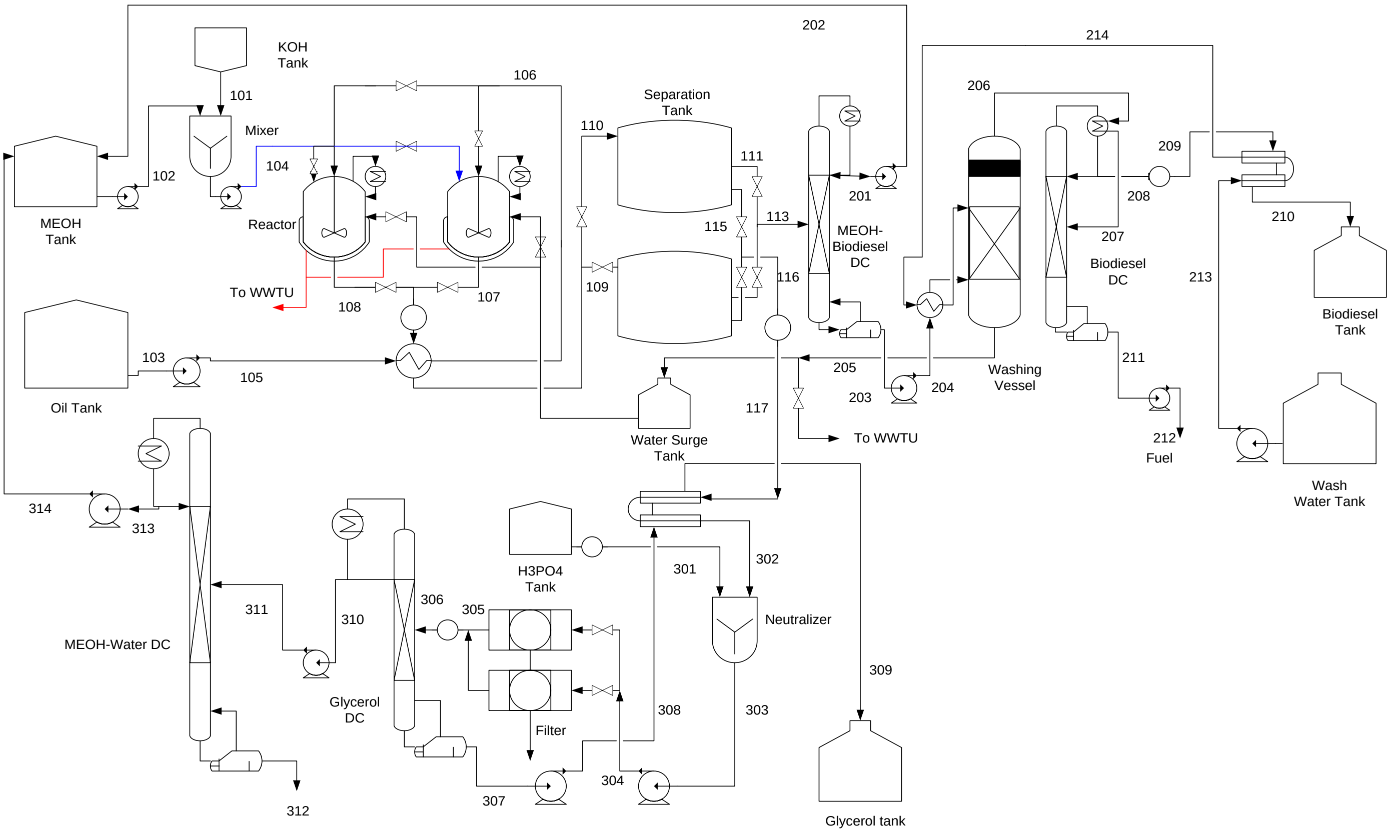

Fig. 1 Process Flow Diagram

2.1 Process Description

The process introduced in the present work consists of three major units. The first one is the production unit where waste vegetable oil is converted to methyl ester (biodiesel). The final element in the first unit is the gravity separator (decanter) where biodiesel (light layer) and glycerol (heavy layer) are separated due to density difference. A stream of each layer is then directed to the corresponding purification unit. The function of the second unit is to purify crude biodiesel to match ASTM D6751 standards. Heavy layer or crude glycerol is purified in the last unit as well as recovering the catalyst as a fertilizer using phosphoric acid.

2.1.1 Biodiesel Production Unit

Biodiesel is produced in this unit according to the optimum conditions previously investigated in an experimental study. The process is conducted in an isothermal batch reactor operating at 65 °C with methanol to oil molar ratio of 6:1 and catalyst, KOH, loading of 1 wt.% of WVO. The experimental optimum agitation speed is about 400 rpm, but on scaling up the reactor this value should be adjusted according to scaling up ratio that keeps the rate of mass transfer to be constant. At these conditions, an oil conversion of 95% is obtained. After completing the reaction, the produced mixture is used to heat up a stream of oil to be converted to biodiesel in another similar reactor as a way of heat integration. At the same time, cooling the produced mixture improves separation of glycerol and biodiesel layers in the decanter which has a residence time of 12 h to ensure efficient separation.

2.1.2 Biodiesel Purification Unit

In this unit biodiesel is purified to match ASTM D6751 standards. First, the biodiesel layer in the separation tank is pumped to a distillation column to remove residual methanol associated with this layer and recycle it to methanol tank to minimize process losses. It was found that methanol content percentage in this layer is about 2 to 3 wt.%. Bottom products are oriented to a washing vessel to remove any traces of methanol, glycerol, soaps and catalyst residues using warm demineralized washing water. This vessel is equipped with a coalescer to produce a clear top product without water contaminations. It should be noticed that this operation can be improved by operating at laminar flow conditions as well as operating at relatively elevated temperature (e.g. 80 °C). After removing these traces, unreacted oil is removed using a vacuum distillation column. Vacuum conditions are used to avoid thermal cracking of biodiesel as well as unreacted oil. Finally, the produced purified biodiesel is pumped to a well-insulated storage tank where it is injected by some additives such as TBHQ in order to increase biodiesel stability and decrease or eliminate its oxidation during storage. These additives commonly added at a percentage of 1 wt.% of produced biodiesel as suggested by Chakraborty and Baruah [15] and Dwivedi et al. [16].

2.1.3 Glycerol Purification Unit

The purity of crude glycerol leaving the decanters is about 50% due to presence of excess methanol, catalyst (KOH) and free fatty acids. Neutralization of KOH using commercial phosphoric acid removes it from glycerol phase as a good fertilizer as well as it removes free fatty acids and

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make them float to the interface of glycerol phase which then can be skimmed and removed. On performing neutralization step, the produced glycerol is about 80% pure. This crude is then pumped to an atmospheric distillation column to remove excess methanol as well as water contaminated in the glycerol phase. The produced high grade glycerol is then cooled and stored. To further minimize process losses, excess methanol aqueous phase produced at the top of the previous atmospheric distillation column is directed to another distillation column to obtain, almost, pure methanol which is then recycled to methanol tank.

3. Material and Energy Balance

Appendix 1 summarizes the material and energy balances of the suggested process. It can be noticed that the flow rates of inlet and outlet streams of the batch reactor are much higher than those of the other streams; this is due to completing charging and discharging processes in short time. To avoid water accumulation in the system that is harmful for transesterification process, water traces in the recycled methanol from glycerol purification unit should be eliminated. This step can be done using a bed of silica gel or using an adsorption system in case of small flow rates instead of using distillation column. Thermodynamic model used was NRTL general.

4. Properties of WVO and Produced Biodiesel

4.1 Properties of WVO

In this study a waste vegetable oil mixture of (soybean and sunflower) having the properties shown in Table 1 was used.

Table1 Some properties of used waste vegetable oil (WVO)

Property	Value
Density kgm^{-3}	902
Acid Value mg KOH/g	2.5
Saponification Value mg KOH/g	200
Viscosity mm^2s^{-1} at 20 °C	50
Water content wt.%	<0.01%
Solid content wt.%	Nil

4.2 Properties of Produced Biodiesel

The properties of produced biodiesel and the according ASTM testing methods are shown in Table 2. The determined diesel index (DI) and cetane number (CN) take the values of 55.56 and 50 respectively.

Table 2 Properties of produced biodiesel

Property	Value	ASTM test
Viscosity, mm^2/s @ 40 °C	4.5	D445
Density, g/cm^3	0.875	D1298
Aniline Point, °C	84.5	D611
Flash Point, °C	140	D93
Cloud Point, °C	3	D2500
Pour Point, °C	-2	D5773
Acid Value, mg KOH/g	0.8	D974
Calorific Value, kJ/kg	39000	D240

Chemical composition of the produced biodiesel is illustrated in Table 3. Fractions of the produced methyl esters were characterized using Agilent HP 6890 GC system equipped with flame ionization detector FID. The column used was hp 5 column type.

Table 3 Chemical properties of produced biodiesel

Fatty Acid	Palmitic	Stearic	Oleic	Linoleic	Linolenic
Carbon Number	16:0	18:0	18:1	18:2	18:3
Percentage	15	8	26.1	43.25	7.65

5. Heat Integration

To obtain a feasible process, material losses should be minimized as well as heat losses. These losses could be minimized through applying heat integration between hot streams to be cooled and cold streams to be heated. Examples of applying this concept in the suggested process are: using cold biodiesel-oil stream at the top of washing vessel to condensate hot vapors in the top stream of biodiesel distillation column, heating washing water by cooling biodiesel condensate to be stored. Other examples of heat integration are the integration between hot purified glycerol and cold crude glycerol streams as well as preheating of oil by hot products discharged from the reactor. Using hot washing water after washing process to be the source of heat for biodiesel batch reactor can be considered as a way of heat integration as well.

6. Modifications Present in the New Scheme Compared to Previous Schemes

The proposed process design introduces a modified one to previously proposed schemes. This section is going to justify the flow of the developed production process scheme compared to other previous schemes [4-7, 10-14, 17 and 18].

6.1 Separation Tank

Separation tank is put after reactor with a relatively long residence time to insure complete separation between phases and this enables the separation tank to be a storage tank with a reasonable stock to make the purification units work steadily. The reason of putting this equipment before distillation is to avoid emulsification and formation of gel like products due to presence of alkaline catalyst.

Centrifuges were not suggested in this scheme due to high energy consumption which affects process feasibility negatively.

6.2 Position of Distillation Column

Distillation column position is after the separation tank to ensure separation of catalyst from ester phase towards glycerol phase. Presence of this material can cause excessive fouling inside different parts of the column specially trays and reboiler which can lead to decrease its thermal and separation efficiency dramatically.

6.3 Washing Vessel

This vessel is equipped with a coalescer and operates at laminar flow conditions to insure elimination of water from ester phase before entering distillation column. This can also decrease the capital cost of the column as presence of water at these conditions needs special materials to avoid severe corrosion.

If the mixture of ester and glycerol layers is washed directly after reaction or even after separation of methanol, the chance of emulsification is high due to presence of potassium hydroxide which decreases separation efficiency and make it more difficult.

6.4 Neutralization Tank

Neutralization process is carried out after separation immediately to eliminate the catalyst from glycerol phase before entering methanol distillation column to avoid excessive fouling. Presence of water in this reactor can cause K_3PO_4 dissolution which prevents its separation, so supplying any water to this equipment was avoided. In addition, water, glycerol and fatty acids residues can make an undesired emulsion in presence of K_3PO_4.

6.5 Continuous or Batch Reaction Step

Continuous transesterification was also suggested but the volume of the reactor was about 20 times the batch one so it is not recommended to operate the reaction step continuously, and instead it is highly recommended to carry out the reaction in a batch reactor followed by a separation tank with a relatively high residence time in order to continuously supply the other equipment with their feed streams. Even if the reaction is carried out in CSTRs in series, the operating and fixed costs are higher than those in the case of using single batch reactor.

7. Conclusion

This study was about the production of biodiesel from waste vegetable oil through homogeneous base-catalyzed transesterification. A process design of a full scale plant that converts about 24 tons/day of oil into biodiesel was suggested. The process was designed according to the optimum reaction conditions (1 h, 1 wt.% catalyst loading, 65 °C and 400 rpm agitation speed) obtained from an experimental work taking into consideration some recommendations suggested after experimental study of product stability to produce a stable biodiesel such as removal of oil contaminations, good insulation of storage tanks and the addition of some additives like (TBHQ) to decrease biodiesel oxidation. Heat integration concept was applied to make the process feasible. Finally, a comparison was held between the new proposed process and the previously suggested ones in literature which showed the importance of the modifications and considerations taken in the new process.

List of Abbreviations

FFA	Free fatty acids	K_3PO_4	Tri-potassium phosphate
FAME	Fatty acid methyl ester	TBHQ	tert-butyl hydroxyquinone
WVO	Waste vegetable oil	ASTM	American Society for Testing and Materials
CSTR	Continuously Stirred Tank Reactor	NRTL	Non-random two liquid (activity model)
KOH	Potassium hydroxide	D.I.	Diesel index
H_3PO_4	Phosphoric acid	C.N.	Cetane number
FID	Flame ionization detector		

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Appendix 1

Table 1 Summary of material and energy balance

Stream	**101**	**102**	**103**	**104**	**105**	**106**	**107**
Oil kg/h	0.00	0.00	12000.00	0.00	12000.00	12000.00	600.00
Methanol kg/h	0.00	2560.00	0.00	2560.00	0.00	0.00	1344.00
KOH kg/h	120.00	0.00	0.00	120.00	0.00	0.00	120.00
Biodiesel kg/h	0.00	0.00	0.00	0.00	0.00	0.00	11450.67
Glycerol kg/h	0.00	0.00	0.00	0.00	0.00	0.00	1165.33
H_3PO_4 kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Water kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00
K_3PO_4 kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00
TOTAL kg/h	120.00	2560.00	12000.00	2680.00	12000.00	12000.00	14680.00
Temperature °C	25.00	25.00	25.00	29.00	25.00	60.00	65.00
Pressure kPa	101.30	155.00	139.00	110.00	204.00	101.30	113.00
Enthalpy kJ/kmol	0.00	0.00	0.00	321.61	0.00	52668.00	431239.50
Heat flow kJ/h	0.00	0.00	0.00	26418.36	0.00	702240.00	45291203.31
Stream	**108**	**109**	**110**	**111**	**112**	**113**	**114**
Oil kg/h	600.00	600.00	600.00	50.00	50.00	50	50
Methanol kg/h	1344.00	1344.00	1344.00	31.09	31.09	31.09	31.09
KOH kg/h	120.00	120.00	120.00	0.10	0.10	0.10	0.10
Biodiesel kg/h	11450.67	11450.67	11450.67	954.22	954.22	954.22	954.22
Glycerol kg/h	1165.33	1165.33	1165.33	0.97	0.97	0.97	0.97
H_3PO_4 kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Water kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00
K_3PO_4 kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00
TOTAL kg/h	14680.00	14680.00	14680.00	1036.38	1036.38	1036.38	1036.38
Temperature °C	38.73	38.73	38.73	25.00	25.00	25.00	25.00
Pressure kPa	190.00	165.00	165.00	101.30	101.30	101.30	46.00
Enthalpy kJ/kmol	438870.85	438870.85	438870.85	0.00	0.00	0.00	0.00
Heat flow kJ/h	46092690.98	46092690.98	46092690.98	0.00	0.00	0.00	0.00
Stream	**115**	**116**	**117**	**201**	**202**	**203**	**204**
Oil kg/h	0.00	0.00	0.00	0.00	0.00	50.00	50.00
Methanol kg/h	80.91	80.91	80.91	28.11	28.11	2.98	2.98
KOH kg/h	9.90	9.90	9.90	0.00	0.00	0.10	0.10
Biodiesel kg/h	0.00	0.00	0.00	0.00	0.00	954.22	954.22
Glycerol kg/h	96.14	96.14	96.14	0.00	0.00	0.97	0.97
H_3PO_4 kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Water kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00
K_3PO_4 kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00

TOTAL kg/h	186.95	186.95	186.95	28.11	28.11	1008.27	1008.27
Temperature ^{o}C	25.00	25.00	25.00	42.57	42.57	160.00	160.00
Pressure kPa	127.20	127.20	150.00	40.00	140.00	50.00	140.00
Enthalpy kJ/kmol	0.00	0.00	0.00	1416.84	1416.84	82470.81	82470.81
Heat flow kJ/h	0.00	0.00	0.00	1244.61	1244.61	274730.37	274730.37
Stream	**205**	**206**	**207**	**208**	**209**	**210**	**211**
Oil kg/h	0.00	50.00	50.00	0.01	0.01	0.01	49.99
Methanol kg/h	2.98	0.00	0.00	0.00	0.00	0.00	0.00
KOH kg/h	0.10	0.00	0.00	0.00	0.00	0.00	0.00
Biodiesel kg/h	0.00	954.22	954.22	927.04	927.04	927.04	27.19
Glycerol kg/h	0.97	0.00	0.00	0.00	0.00	0.00	0.00
H3PO4 kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Water kg/h	1999.80	0.20	0.20	0.20	0.20	0.20	0.00
K3PO4 kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00
TOTAL kg/h	2003.85	1004.42	1004.42	927.25	927.25	927.25	77.18
Temperature oC	85.00	85.00	200.00	205.00	205.00	30.00	280.00
Pressure kPa	110.00	101.30	16.00	10.00	130.00	105.00	20.00
Enthalpy kJ/kmol	4519.26	37552.36	134235.45	131602.03	131602.03	3169.32	368977.81
Heat flow kJ/h	502566.33	121550.99	434498.70	406779.10	406779.10	9796.31	53819.54
Stream	**212**	**213**	**214**	**301**	**302**	**303**	**304**
Oil kg/h	49.99	0.00	0.00	0.00	0.00	0.00	0.00
Methanol kg/h	0.00	0.00	0.00	0.00	80.91	80.91	80.91
KOH kg/h	0.00	0.00	0.00	0.00	9.90	0.00	0.00
Biodiesel kg/h	27.19	0.00	0.00	0.00	0.00	0.00	0.00
Glycerol kg/h	0.00	0.00	0.00	0.00	96.14	96.14	96.14
H3PO4 kg/h	0.00	0.00	0.00	5.78	0.00	0.00	0.00
Water kg/h	0.00	2000.00	2000.00	1.02	0.00	4.20	4.20
K3PO4 kg/h	0.00	0.00	0.00	0.00	0.00	12.49	12.49
TOTAL kg/h	77.18	2000.00	2000.00	6.80	186.95	193.74	193.74
Temperature oC	280.00	25.00	66.23	25.00	58.38	57.00	57.00
Pressure kPa	101.30	140.54	130.00	101.30	101.30	110.00	140.00
Enthalpy kJ/kmol	368977.81	0.00	3102.15	0.00	4072.48	4009.48	4009.48
Heat flow kJ/h	53819.54	0.00	344682.80	0.00	15272.49	15272.49	15272.49
Stream	**305**	**306**	**307**	**308**	**309**	**310**	**311**
Oil kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Methanol kg/h	80.91	80.91	0.07	0.07	0.07	80.84	80.84
KOH kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Biodiesel kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Glycerol kg/h	96.14	96.14	96.14	96.14	96.14	0.00	0.00
H3PO4 kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Water kg/h	4.20	4.20	0.00	0.00	0.00	4.20	4.20
K3PO4 kg/h	0.00	0.00	0.00	0.00	0.00	0.00	0.00
TOTAL kg/h	181.25	181.25	96.21	96.21	96.21	85.04	85.04
Temperature oC	57.00	57.00	95.00	95.00	30.00	62.42	62.42
Pressure kPa	101.30	130.00	101.30	140.00	110.00	90.00	140.00
Enthalpy kJ/kmol	3839.84	3839.84	17006.92	17006.92	1113.83	239015.03	239015.03
Heat flow kJ/h	14400.04	14400.04	17809.67	17809.67	1166.40	658745.54	658745.54
Stream	**312**		**313**		**314**		
Oil kg/h	0.00		0.00		0.00		
Methanol kg/h	1.35		79.49		79.49		
KOH kg/h	0.00		0.00		0.00		
Biodiesel kg/h	0.00		0.00		0.00		
Glycerol kg/h	0.00		0.00		0.00		
H_3PO_4 kg/h	0.00		0.00		0.00		
Water kg/h	3.88		0.32		0.32		
K_3PO_4 kg/h	0.00		0.00		0.00		
TOTAL kg/h	5.23		79.81		79.81		
Temperature ^{o}C	85.60		61.60		61.60		
Pressure kPa	110.00		90		140.00		
Enthalpy kJ/kmol	272419.43		235466.23		235466.23		
Heat flow kJ/h	70204.50		588283.38		588283.38		

https://doi.org/10.30799/jacs.196.18040401

Cite this Article as: E.G. Al-Sakkari, S.T. El-Sheltawy, A. Soliman, I. Ismail, Methanolysis of low FFA waste vegetable oil using homogeneous base catalyst for biodiesel production: New process design, J. Adv. Chem. Sci. 4(4) (2018) 593–597.