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TIME, ENERGY EFFICIENCY IN THE PRODUCTION OF BIODIESEL, AND PRODUCTS DESTINATION IN A BIOREFINERY

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ABSTRACT

This manuscript deals with the production of biodiesel from triglycerides (TG); soybean oil was the primary raw material. The transesterification reaction was used to convert triglycerides into monoalkyl esters. The catalytic mixture of methanol and potassium hydroxide (KOH) was used in the transesterification reaction. The anhydrous refining of biodiesel was used to reduce side reactions during the purification process. The treatment of glycerol by acidification with H3PO4 and its subsequent purification was a convenience process aiming to form the desired byproducts. The conversion of residual fatty acids to soaps through the saponification reaction or into esters through the esterification reaction with H2SO4 can be done observing the market of those products. The monitoring of the transesterification reaction in real time with laser spectroscopy allows the variation of the temperature during the reaction process without losing the reaction endpoint. The use of only one reaction vessel for the accomplishment of several unit operations aiming the reducing of the reaction time in the production process was well succeeded. The main resulting products are biodiesel, glycerol, fertilizer, and soaps. As expected the biodiesel can be used as a source of energy, the glycerol can be further refined but this process was not explored at this manuscript, the resulting salts can be used as fertilizers, and the soups can be sold and used as a soup.

Keywords: *transesterification, real-time LASER spectroscopy, byproducts, energy efficiency.*

1. INTRODUCTION

Let us assume that you have some previous knowledge about the biodiesel industry and let's explore some possibilities in how to make a production facility more productive and lucrative. To get acquainted with this type of industry lets do a quick review of some of the process involved in the biofuel industry.

It is suggested the reading of the references about transesterification [1-5], about esterification [6-10], about saponification [11-15], about acidification and neutralization reactions [16-20], monitoring of chemical reactions in real time [21-25].

The transesterification reaction is the process used to convert an ester into another ester by the exchanging the organic group R″ of an ester with the organic group R′ of alcohol. This reaction is catalyzed by the addition of a base catalyst. Figure 1 summarizes the transesterification reaction [1-5]. The transesterification reaction was chosen as the primary reaction in the production process of this chapter.

The esterification reaction is used to produce esters when the starting products are not esters. When carboxylic acids are heated in the presence of alcohols and an acid catalyst, usually concentrated sulphuric acid (H_2SO_4 95.0-98.0%), the reaction products are esters, water, and the residue of the catalyst. Figure 2 summarizes the esterification reaction [6-10].

The saponification reaction is used to produce soap, and it is possibly an interesting alternative to treat the residues of biodiesel production. Figure 3 summarizes the saponification reaction [11-15].

The acidification and neutralization reactions [16-20] may be used to convert residual soaps into free fatty acids (FFA) and to neutralize the catalyst respectively.

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The monitoring of chemical reactions in real time with LASER spectroscopy [21-25] can be done with a setup equivalent to Figure 5. This technique is used to determine the endpoint of the transesterification reaction and follows its kinetics trough time.

2. MATERIALS AND METHODS

Suppose that you have at your disposal a reactor, similar to the one in Figure 6. It is not relevant that the components of the reactor are inside or outside the reactor, in any of the cases, there shall be pros and cons to consider, and you should evaluate what will suit you better.

2.1 DEVELOPMENT

The present experiments were conducted in the laboratory, and the lab results were scaled up to the proposed reactor. Commercial soybean oil (triglyceride), potassium hydroxide (catalyst) and methanol (alcohol) were used. The chemical reaction that occurred in the reactor was transesterification, followed by acidification, esterification or saponification. Suppose that you will operate the reactor of Figure 6 according to the procedures below.

Considering Figure 7, the generalized aspects of the system and the following operations are carried out to produce biodiesel in this reactor:

1. Whereas, the catalyst is prepared in the mixer vessel (6) of Figure 7. Add the volume of methanol contained in the vessel 5 of Figure 2 to the reactor. For this experiment it was used 130ml;
2. By titration, determine the mass of the catalyst, stored in the vessel (4) of Figure 7, to be used;
3. Add the catalyst (KOH) to the methanol, in this case, and it was used approximately 10g;
4. Dissolve the catalyst in methanol for *in situ* production of the catalytic mixture;
5. Add the oil to the reactor;
6. Activate the reactor agitation system;
7. Turn on the reactor heating system. The initial oil temperature was 20,64 °C (69.15 F);
8. Activate the reaction monitoring system(real-time LASER spectroscopy);
9. Under constant stirring add the previously prepared catalytic mixture to the reactor. (Date of acquisition of the data 9/27/2011 Time that the first transesterification reaction started: 10:55:57 HH:MM: SS. The end of the monitoring time: 12:58:37 HH:MM: SS. The total amount of samples was 7082 points. Total obtained data: 63738 points. Total monitoring time: 2:02:40 HH:MM:SS)
10. Allow the reaction to proceed with increasing temperature until the chemical equilibrium is established by the monitoring system (graphic example in Figure 8). The temperature at the chemical equilibrium was 57.31 °C (135.16 F);
11. After the reaction reaches chemical equilibrium, turn off the stirring system and heating system;
12. Activate the electrostatic separation system;
    a) Simultaneously initiate the preparation of the catalytic mixture for the second transesterification reaction.
    b) Repeat steps 1-4 by adjusting the volume of the reagents to 26 ml of methanol and 2 g of KOH.
13. Allow the separation of glycerin and biodiesel;
14. Separation of the glycerin is readily determined by the optical system, a sharp variation in absorbance indicates that the glycerin has been separated (example in Figure 3);
15. After the glycerin separation from the biodiesel phase, turn off the electrostatic separation system;
16. Move the glycerin into the vessel 15 of Figure 7;
17. Maintain the product of the first transesterification reaction in the reactor. Believe that it's purity is not less than 80% conversion of raw material into fuel or make a *H^1NMR* test to confirm (De Boni *et al.*, 2013)
18. Activate the reactor agitation system;
19. Start the heating system of the reactor. The initial temperature in the second transesterification reaction was 39.01 °C (102.21 F);
20. Activate the reaction monitoring system;
21. Under constant stirring add the previously prepared catalytic mixture to the reactor. (Date of acquisition of the data 27/9/2011 Time that the second transesterification reaction started: 13:09:35 HH:MM:SS. The end of the monitoring time: 14:32:38 HH:MM:SS. The total amount of samples was 4798 points colected. Total obtained

data: 43182 points. Total monitoring time: 1:23:03 HH:MM:SS);

22. Allow the reaction to proceed, with the elevation of temperature, until the chemical equilibrium is established by the monitoring system (graphic example in Figure 9). The temperature at the chemical equilibrium was 56.31 °C (133.36 F);
23. After the reaction reaches the second chemical equilibrium, turn off the stirring and heating system;
24. Activate the electrostatic separation system
    a) Simultaneously initiate the preparation of the catalytic mixture for the third transesterification reaction.
    b) Repeat steps 1-4 by adjusting the volume of the reagents to 5 ml of methanol and 0.4 g of KOH.
25. Allow the separation of glycerin and biodiesel;
26. Separation of the glycerin is readily determined by the optical system, a sharp variation in absorbance indicates that the glycerin has been separated (example in Figure 5);
27. After the glycerin separation from the biodiesel phase, turn off the electrostatic separation system;
28. Conducting the glycerin into the vessel 15 of Figure 7;
29. Maintain the product of the second transesterification reaction in the reactor. Believe that it's purity is not less than 96% of conversion of raw material into fuel.
30. Activate the reactor agitation system;
31. Activate the reactor heating system. The initial temperature in the third transesterification reaction was 32,02 °C (89.63 F) (Figure 10);
32. Activate the reaction monitoring system
33. Under constant stirring add the previously prepared catalytic mixture to the reactor (Date of acquisition of data 27/9/2011. The time that the third transesterification reaction started: 14:40:05 HH:MM:SS. The end of the monitoring time was 16:48:13 HH:MM:SS. The total amount of samples was 7397 points. Total obtained data: 66573. Total monitoring time: 02:08:08 HH:MM:SS);
34. Allow the reaction to continue, with the elevation of temperature, until the chemical equilibrium is established by the monitoring system (there is a graphical example in Figure 6). The temperature at the chemical equilibrium was 56,05 °C (132.8 F);
35. After the reaction reaches the second chemical equilibrium, turn off the stirring and heating system;
36. Activate the electrostatic separation system;
37. After the glycerin separation from the biodiesel phase, turn off the electrostatic separation system;
38. Conducting the glycerin into the vessel 15 of Figure 7;
39. Maintain the product of the third transesterification reaction in the reactor. Believe that its purity is not less than 99% conversion of raw material into fuel.
40. Switch on the stirring system
41. Turn on the low-pressure system;
42. Use the flash distillation system;
43. Remove the residual alcohol present in the biofuel, reserving the same for later reuse;
44. After completion of alcohol withdrawal, switch off the low-pressure system and stop using flash distillation;
45. Add under constant stirring, the cleaning agent to initiate refining process (for this example, use the sorbent of your choice or availability on the local market as *Magnesol®*, *Eco2Pure®*, *Amberlite®*, *Purolite®,* or another type of clay);
46. Allow interaction of the biofuel with the refining agent;
47. After the refining/cleaning agent has completed its purpose, activate the recirculation filtration system;
48. After the filter has wholly removed the cleaning agent, transport the fuel fluid to a suitable container (Figure 7, vessel 14);
49. Clean the filter as necessary.
50. To treat the crude glycerol of vessel 10, at the proper moment, when the reactor (11) is empty move the content of vessel 10 to vessel 11. Add phosphoric acid from vessel 17. This will acidify the medium producing a salt (K_3PO_4) that can be placed in vessel 12. Free fatty acids that can be placed in vessel 9, and yellow glycerol that is moved to vessel 15. The yellow glycerol can pass further treatment to be sold as distilled glycerol, but it will not be explored in this chapter.
51. The FFA from vessel 9 can be converted into biodiesel or soap, depending on what is the best economic opportunity. To convert the FFA into biodiesel, the content of vessel 9 is moved to vessel 11. An esterification reaction will take place,

mixing the alcohol from vessel 5 with the sulfuric acid of vessel 16 in the vessel for the preparation of catalyst (6). This catalytic mixture is added over the FFA, and it is allowed to the esterification reaction to take place. Not all the details will be presented here.

52. If the saponification reaction is to take place, just move the FFA from vessel 9 to vessel 11 and add a strong mixture of KOH from vessel 4 and water from vessel 8. Allow the reaction to the procedure for a few hours and the final product will be liquid soap.

3. RESULTS AND DISCUSSION:

To execute the transesterification reaction 3 times ensures that the final product will achieve more than 99% of conversion from triglycerides to biodiesel. The removal of the crude glycerol in each transesterification step ensures the forward of the reaction with the use of smaller amounts of the catalytic mixture.

The reaction time was optimized with the use of an appropriate monitoring system. Monitoring of the transesterification reaction with real-time LASER spectroscopy allowed initiating the reaction at room temperature and increase the supply of energy (heat) throughout the reaction, reducing the energy consumption during the fuel production and the endpoint of chemical reaction of transesterification was detected by the monitoring system in the same way as it would do if the reaction was carried out under constant temperature. The endpoint is assumed when the reaction curve assumes a linear behavior, as in Figure 11.

The H_3PO_4 was chosen to acidify the crude glycerol due to the conversion of the catalyzer (KOH) into fertilizer (K_3PO_4). Other acids may be used as needed or to form other salts.

4. CONCLUSIONS:

The final main product, biodiesel, was obtained with the same characteristics as the one produced by traditional methods. The production costs were lower since the input of energy was smaller and the production time was shorter than traditional methods.

Performing the transesterification reactions near to the stoichiometric amounts reduced the refining time of the final product, as there were less unreacted raw, i.e., methanol, materials to be removed from the main product by flash destilation.

The ability to convert part of the byproducts (FFA) into biofuels or soaps allows the company to quickly change its production to serve the market sectors that offer greater profitability without compromising their productive capacity.

5. ACKNOWLEDGMENTS:

CAPES case: 9109/11-0

PETROBRAS/CNPq case: 142120/2009-0

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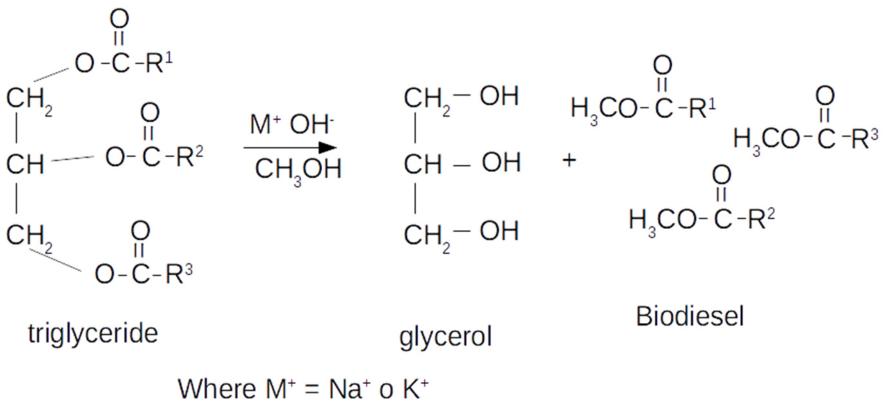


Figure 1: Transesterification: ester + alcohol → different alcohol + different ester

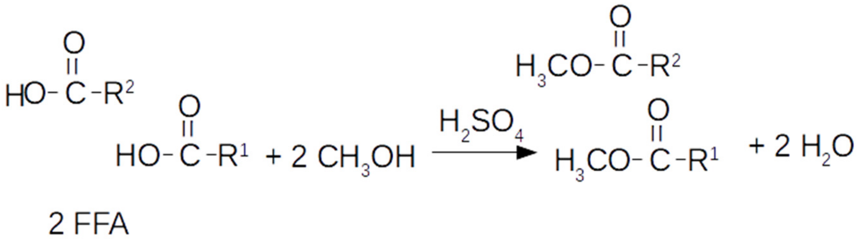

$$HO\text{-}\overset{\overset{O}{\|}}{C}\text{-}R^2 \quad HO\text{-}\overset{\overset{O}{\|}}{C}\text{-}R^1 + 2\,CH_3OH \xrightarrow{H_2SO_4} H_3CO\text{-}\overset{\overset{O}{\|}}{C}\text{-}R^2 \quad H_3CO\text{-}\overset{\overset{O}{\|}}{C}\text{-}R^1 + 2\,H_2O$$

Figure 2: Esterification, free fatty acidy (FFA) + alcohol → ester + water

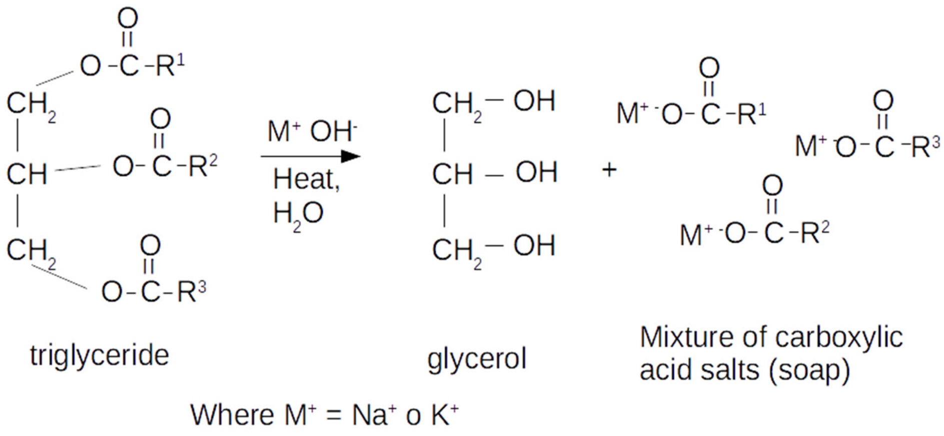


Figure 3: Saponification: Triglyceride + Alkaline metal → glycerine + soap

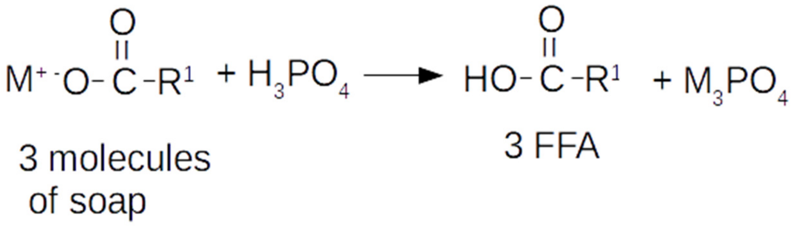

$$M^{+-}O\text{-}\overset{\overset{O}{\|}}{C}\text{-}R^1 + H_3PO_4 \longrightarrow HO\text{-}\overset{\overset{O}{\|}}{C}\text{-}R^1 + M_3PO_4$$

Figure 4: Acidification, soap + phosphoric acid → FFA + phosphoric acid salt.

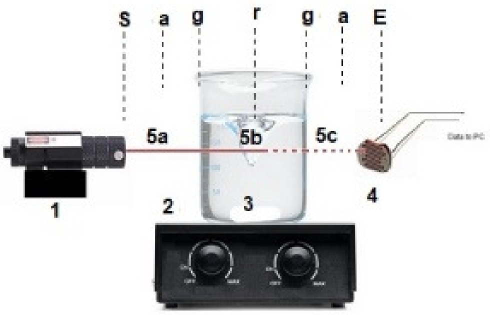


Figure 5: Schematic view of the setup of the reaction system monitored with real-time with LASER spectroscopy. Legend: 1- LASER source; 2- Heating plate; 3- Reactor; 4- Transducer; 5a- Incident LASER bean; 5b-Interaction between the LASER and the medium; 5c- output LASER radiation; S – propagation plane start point; E – propagation plane ending point; a – air environment; g – glass environment; r – reaction environment

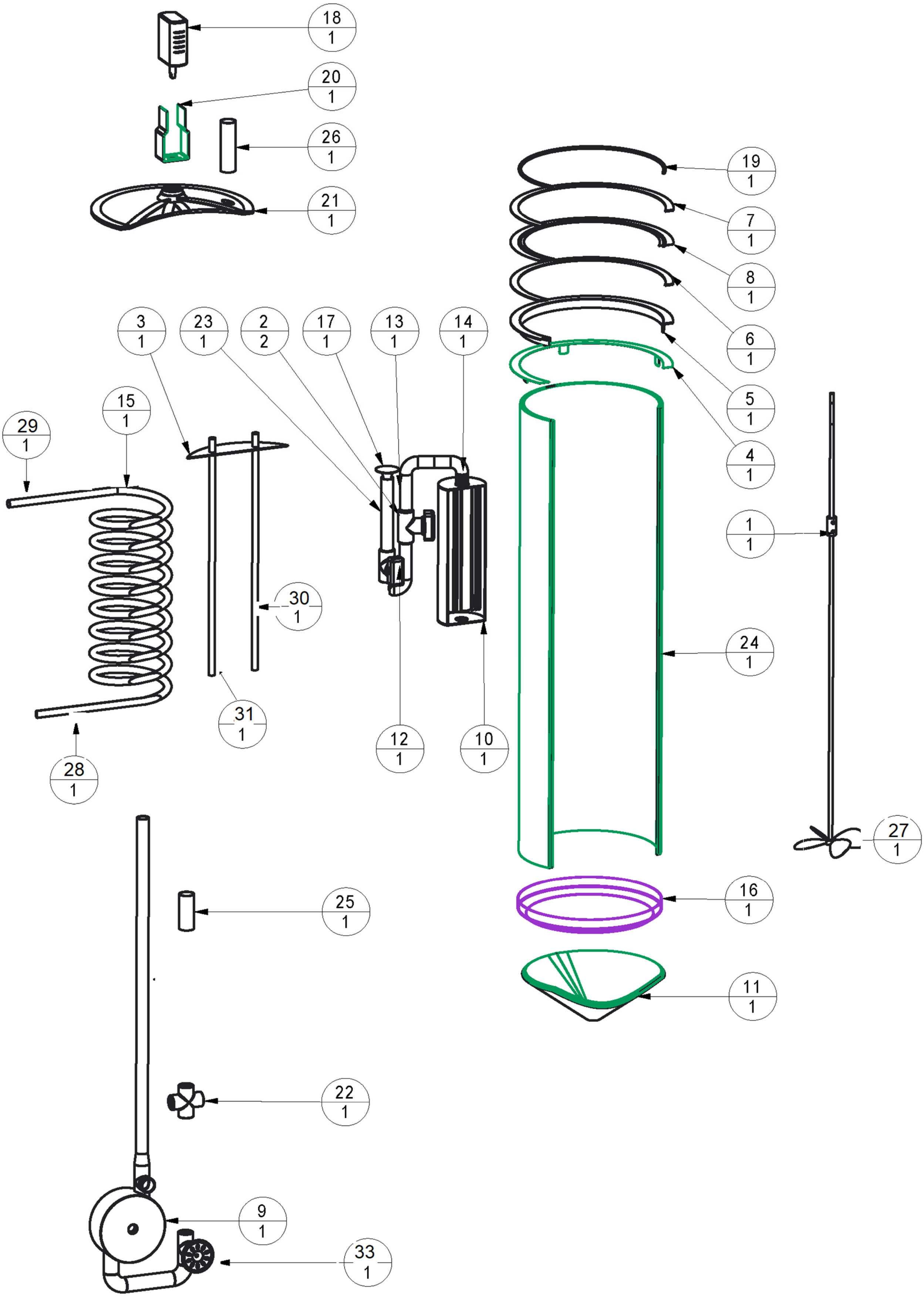


Figure 6: Reactor overview

Table 1: Reactor's components of Figure 6.

Number	Component	Amount
1	Shaft coupling	1
2	Actuator filter	2
3	Electrodes Bars	1
4	Upper flange base	1
5	External upper flange base	1
6	Inner upper flange base	1
7	External upper flange base	1
8	Upper flange base	1
9	Water pump	1
10	Housing filter	1
11	Bottom cone	1
12	90-degree bend fitting	1
13	Curve 90-degree filter input	1
14	90 Degree Curve Filter	1
15	Heating spiral	1
16	Tube seal - cone	1
17	Pressure Housing	1
18	Shaker motor	1
19	O-ring 304mm 5mm	1
20	Engine mount	1
21	Top cover structure	1
22	Exit cover	1
23	Wetting Tube	1
24	Main tube	1
25	Exit Tube 2	1
26	Vacuum tube	1
27	System shake propellers	1
28	Heating fluid outlet	1
29	Heating fluid inlet	1
30	Positive electrode	1
31	Negative electrode	1
32	Pipe	1
33	valve	

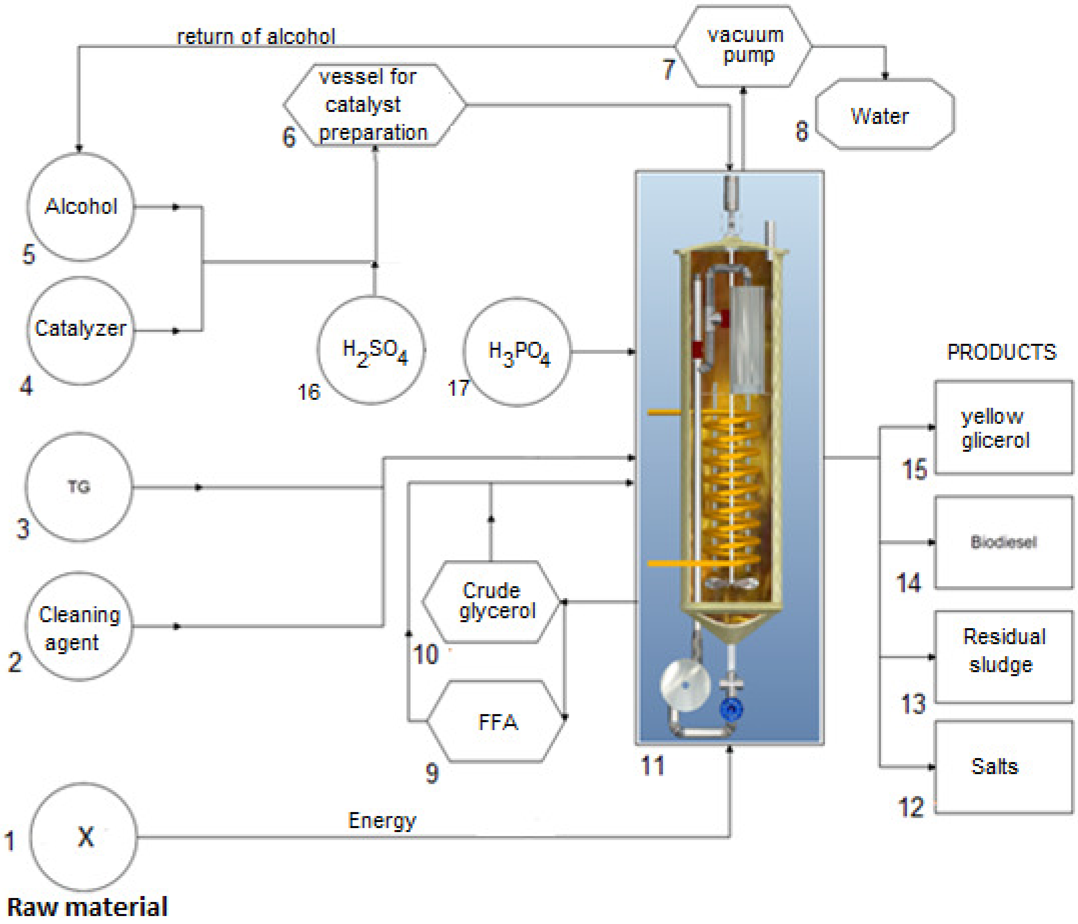


Figure 7: Generalized aspects of the system. Where: 1- Stock of wood or burning oil; 2- Stock of cleaning agent; 3- Stock of triglycerides (fat or oil); 4- Stock of catalyze ; 5- Stock of methanol; 6- tank for the preparation of the catalytic mixture ; 7- vacuum pump; 8- water storage tank; 9- FFA vessel; 10-Crude glycerol vessel ; 11- reactor; 12 – salt container; 13- residues; 14- biodiesel container; 15- yellow glycerin container.

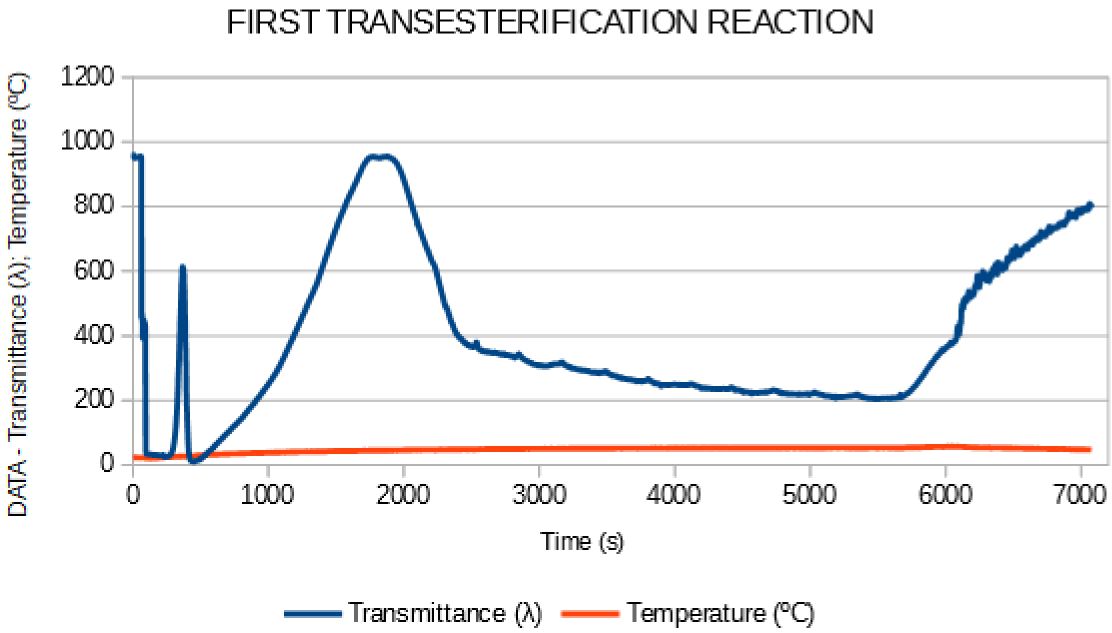


Figure 8 – First reaction of transesterification monitored with LASER spectroscopy and temperature monitoring.

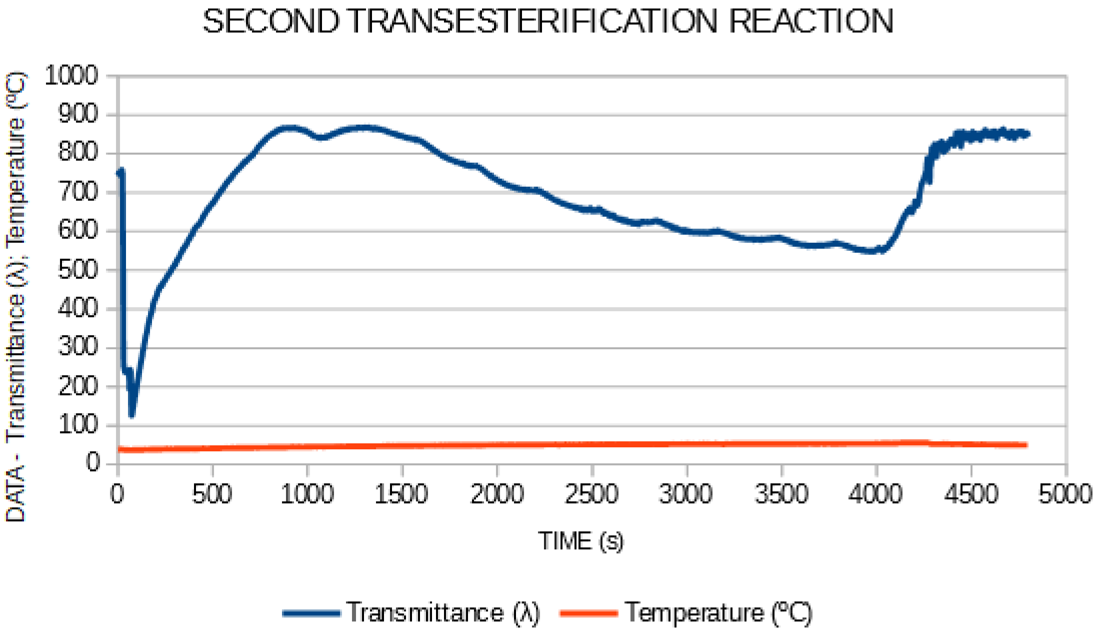


Figure 9 – Second reaction of transesterification monitored with LASER spectroscopy and temperature monitoring.

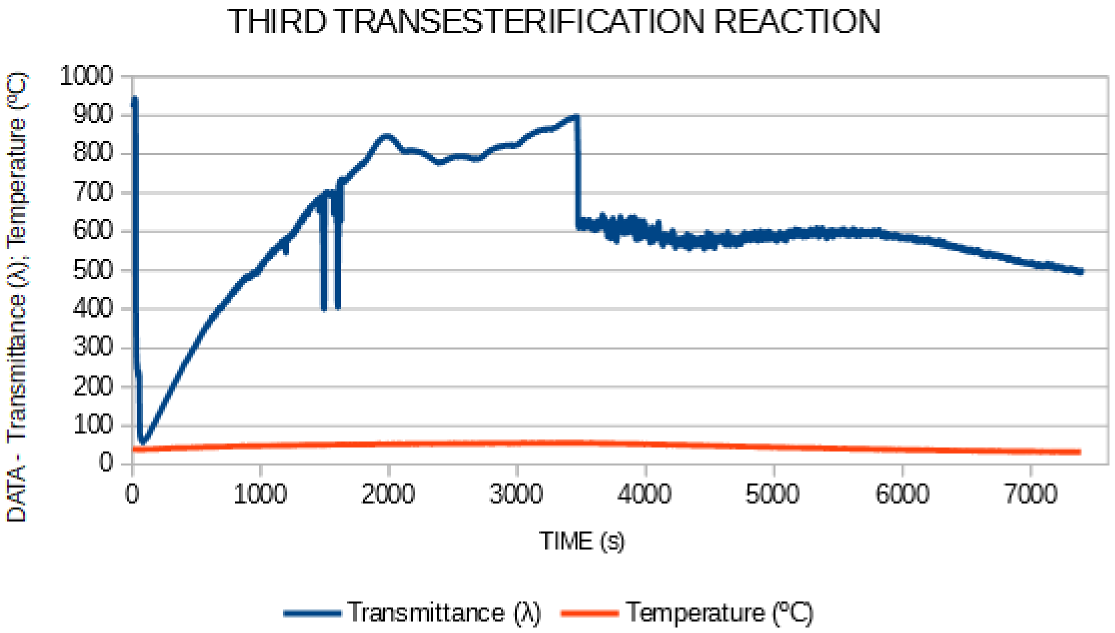
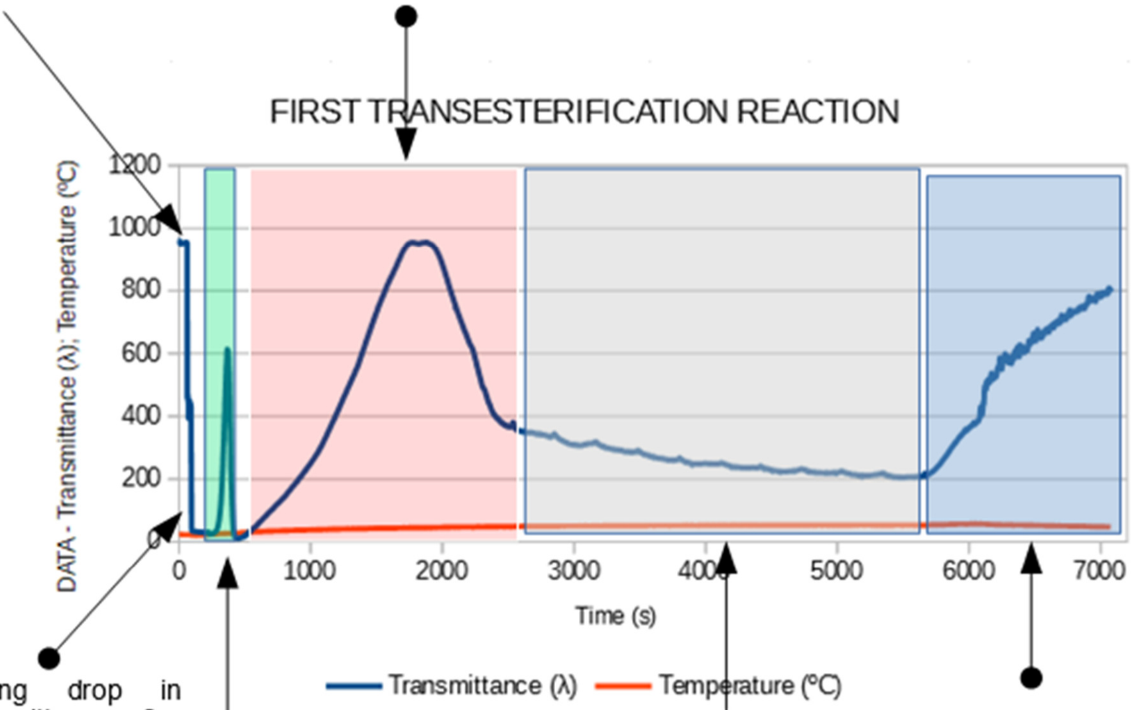


Figure 10: Third reaction of transesterification monitored with LASER spectroscopy and temperature monitoring. (In this graph the LASER was hit by the author's hand, and the distortion was caused by him fixing the sensor position. The glycerin separation was not monitored.)

Linear behavior before addition of the catalytic mixture.

Effective reaction time.



Strong drop in transmittance after addition of the catalytic mixture.

Moments of disturbance in the reaction, common just after the addition of the catalytic mixture. This peak may or may not appear depending on the position of the sensor within the reaction medium. This peak has no monitoring function or practical function and should be ignored when occurs, it is just an interference.

linear behavior of the instrumental reading, the reaction is in chemical balance. The reaction should have been interrupted much before 300s, but for demonstration purposes, it was allowed to proceed.

Glycerin separation phase

Figure 11: Comments on the monitoring process on the Graph.