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Multi alcohols continuous unit for biodiesel production: Design and automation

Unidad continua de múltiples alcoholes para la producción de biodiésel: Diseño y automatización

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Abstract

In this paper, the evaluation and implementation of a control grid for a continuous biodiesel production plant was carried out using a layout that allows the use of ethanol (C_2H_5OH) and methanol (CH_3OH), and a capacity of $100 \, L/h$. First, the production system was divided into 8 subsystems, in order to identify and detail all the sub-processes that happen in the plant during the production of biodiesel. Then, it was developed careful studies of the systems and identification of the processing steps that demand a greater need for automation, using applied control strategies according to the specific need of each one, such as: feedback, on-off, ratio and proportion, split rang, and cascade. Thus, utilizing PFD (Process Flow Diagram) and P&ID (Piping and Instrumentation Diagram), it was possible to obtain a control loop capable of integrating the entire plant, aiming at greater stability of all process variables, which in turn result in a better functioning of the plant, as well as a final product capable of meeting the most varied quality standards, from different raw materials.

Keywords: Continuous unit, biodiesel, industrial automation, control strategies.

Resumen

En este artículo se realizó la evaluación e implementación de una cuadrícula de control para una planta de producción continua de biodiesel mediante una configuración que permite el uso de alcohol etílico (C_2H_5OH) y alcohol metílico (CH_3OH), con capacidad de 100 L/h. Primero, se dividió el sistema de producción en 8 subsistemas, con el fin de identificar y detallar todos los subprocesos que ocurren en la planta durante la producción de biodiesel. Luego, se desarrollaron estudios minuciosos de los sistemas e identificación de los pasos de procesamiento que demandan mayor necesidad de automatización, utilizando estrategias de control empleadas de acuerdo a la necesidad específica de cada uno, tales como: feedback, on-off, razón y proporción, split rang y cascada. Por lo tanto, con el uso de DFP (Diagrama de flujo de proceso) y DTI (Diagrama de tuberías e instrumentación) fue posible obtener un lazo de control capaz de integrar toda la planta, buscando una mayor estabilidad de todas las variables del proceso, lo que a su vez resulta en un mejor funcionamiento de la planta, así como con un producto final capaz de cumplir con los más variados estándares de calidad, a partir de diferentes materias primas.

Palabras clave: Unidad continua, biodiesel, automatización industrial, estrategias de control.

1 Introduction

Despite rising costs, predictable future scarcity, and increased pollutant emissions from burning fossil fuels (Sensoz *et al.*, 2000), many countries around the world are still heavily dependent on oil as the main source of electricity and transport fuel (Yusuf *et al.*, 2011). Thus,

it is necessary to search for new renewable, reliable and economically viable energy sources. Among the numerous sources researched and developed, such as solar, wind, and geothermal, the use of biomass for the production of biofuels stands out, which in the most optimistic scenario, will contribute with about half of the total energy demand in developing countries until 2050 (IPCC, 1997).

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Biofuels are liquid or gaseous fuels - bioethanol, biomethanol, biodiesel, biohydrogen, and it can be produced from a variety of bio-raw materials, such as vegetable oil and animal fat, and represent especially for the transport sector, a promising option for the future. Among all biofuels, biodiesel has received the most attention, due to the similarity between biodiesel and conventional diesel, in terms of chemical structure and amount of energy (Yusuf et al., 2011). Once it is perfectly miscible, and physically and chemically similar to mineral diesel oil, biodiesel can be used pure or mixed with the former in any proportion, in diesel cycle engines, without the need for significant or costly adaptations (Ramadhas et al., 2004; Hassan et al., 2013). Additionally, biodiesel has other economic and environmental advantages over petrodiesel, such as its non-toxicity, biodegradability, increased octane number, sulfur-free, better combustion profile and emission characteristics (Hasheminejad et al., 2011).

Biodiesel is a transesterification reaction product of any raw material that contains free fatty acids and/or triglycerides and alcohol, with or without a catalyst, as occur in the supercritical fluid method (SCM) (Wang, et al., 2009). This process is widely used in biodiesel plants due to its high performance under standard working conditions and for resulting in a better quality product (Shahid et al., 2011). However, efficient conversion to biodiesel depends, in addition to the nature of the raw materials, on the operating temperature and on the reaction time control (Ramadhas et al., 2004).

In this context, to ensure that the process is able to achieve not only a high quality end product, but also to maintain continuous, efficient and high quality production (Oğuz et al., 2015), it is necessary to formulate an automation strategy through the development of a control grid, which is robust to disturbances and changes at the set point, in order to provide an adequate performance to the biodiesel production process (Regalado-Méndez et al., 2016). Automation in a productive plant brings several benefits, such as the reduction of human labor during production, the ability to interfere in the production process at any level, and the minimization of maintenance costs (Özcan et al., 2005), etc., using basic components like sensors, controllers and actuators (De-Jesus, 2013). The automated system aims to optimize the process, increasing the production rate, precision and quality, including cost reduction (Prudente, 2007). In general, it involves the selection of controlled and manipulated variables (CVs and MVs), the definition of the control structure, adjustment, etc. (Rivas-Perez et al. (2016); De-Jesus, 2013).

There are some papers, in the literature, involving automation of biodiesel plants, such as those proposed by Birchal and Birchal (2013) and Oğuz et al. (2015), however, they are based on small units of production by batches of biodiesel, which involves reduced stages of the process and with the use of few tanks. The objectives are varied, but basically include the study of a preliminary programming logic, defining the process requirements for each equipment that constitutes the plant under study (Oğuz et al., 2015), and implementation of programmable logic controls (PLC) for automation (Worm, 2012), control loop proposals using and comparing techniques such as feedback, split range, loss technique, Tyreus-Luyben, Nichols-Ziegler, Skogestad, and plant-wide (De-Jesus, 2013; Munir et al., 2013; Silva, 2015; Regalado-Méndez et al., 2016), and implementation of new types of control, sensors and forms of programming and automation (González-González et al. (2020); Dhar and Mazumdar, 2012; Wali et al., 2013).

The objective of the present work is to propose an automation grid, using feedback control, split range, cascade and other techniques, for a continuous production biodiesel plant with multipurpose characteristics, being able to process different vegetable oils, as long as they fit the appropriate specifications for adequate fuel production. The plant's versatility extends to transesterification agents: alcohol and alkali used in the conversion of vegetable oils into biodiesel. In this way, the process was designed to employ ethanol or methanol, and as a catalyst aid, the sodium hydroxide and/or potassium hydroxide.

2 Materials and methods

The development of this project was performed from the utilization of technical standard destined to the automation of industrial processes: Instrument Society of America, ISA 5.1 - Instrumentation Symbols and Identification (2009). To this end, it was developed the block and process flowcharts of the continuous production unit of ethanol or methanol biodiesel. Then, through the analysis of the necessary steps for the production of biofuel, control strategies were implemented according to the specifications of each subsystem.

Table 1. Concentration of solutions and reagents for the production of 100 l/h of biodiesel.

| NaOH | 0.5% |
|---------------------------|--------|
| KOH | 0.5% |
| Methanol | 22.0% |
| Catalyst solution | 12.0% |
| Ethanol | 35.0% |
| Reactional temperature | 60°C |
| Reactional residence time | 40 min |
| Decanting time | 40 min |

In this context, after defining its tags and production flow, the plant was modeled using the Solidworks® software and arranged in order to respect the previously defined flows of reagents and products required for the process. For the elaboration of control strategies, it was essential to develop the plant's PFD (Process Flow Diagram) and P&ID (Piping and Instrumentation Diagram), since these flowcharts are used to monitor the production process data specified in the preliminary design of the unit. The monitoring at the installation site was carried out at the plant installed at the Center for Technology and Industrial Quality of Ceará (NUTEC), located at the Brazilian state of Ceará. The monitored process parameters are shown in Table 1.

In this way, in order to obtain a final product with the desired quality and characteristics, (i.e., non-toxicity, biodegradability, sulfur-free), the continuous unit must meet the requirements specified in the Table 1, whose set points were established based on volumetric flow rates and mass concentrations that follow the lines of reagents, products and by-products. Thus, the control strategies were established in on-off, fee-back, cascade, split range and ratio-proportion. The choice of strategy implementation was chosen according to the operational system of each stage of the process provided in the PFD and P&ID.

3 Analysis and discussion of results

3.1 Presentation of the plant and its subsystems

The biodiesel production plant presented in this article can be divided into eight subsystems for better understanding, as shown in Figures 1 and 2. The first subsystem aims to prepare the raw materials that will participate in the biodiesel processing, which are

vegetable oil, catalyst and alcohol. To obtain biodiesel, there is no novelty regarding the sequence of steps that must be followed for its production, however, great care must be taken in handling the components, for example, when adding a certain amount of alcohol to dissolve the catalyst. The transesterification for continuous production, highlighted in Figure 1 by subsystems 02 and 04, can be performed in CSTR (Continuous Stirred-Tank Reactor) and PFR (Plug Flow Reactor) reactors, depending on the fixed parameters required by the final product, as well as the possibility of being carried out in one or two transesterification processes. Before using more than one type of reactor, like is the case of this plant, an evaluative study is necessary, regarding the types of reactors that could be applied in biodiesel plants. The CSTR reactors have motors and agitators that increase their cost, and with an efficiency gain proven when using more than one reactor, there can be a reduction costs in possible expansions of production plants that use only CSTR reactors. Thus, according to Rodríguez-Mariano et al. (2015), CSTRs have been intensively studied by the control community for the past two decades.

Then, there is a phase separation stage for each of the transesterification processes, aiming to separate the lighter phase, which consists predominantly of linear esters (biodiesel). The densest phase is formed mainly by glycerol. The separation subsystems 03 and 05 differ mainly because the latter has an alcohol depletion column. This subsystem is activated only when the alcohol used in the processing is ethanol, in order to break the emulsion of the mixture and facilitate the separation of the glycerin phase. Subsystems 06 and 07 aim to purify biodiesel. This purification occurs by washing, using liquidliquid extraction columns, in order to extract traces of glycerin and reduce the soap content obtained from the saponification reaction between catalyst and vegetable oil; acid treatment, in the last column of the system, to hydrolyze the remaining traces of soaps; dehumidification; and filtration.

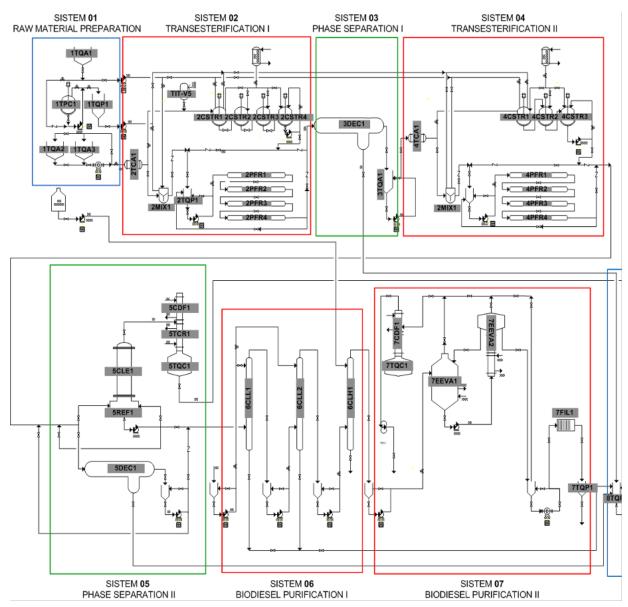


Fig. 1. Basic engineering diagram of the subsystems 01 to 07.

Finally, in subsystem 08 (Figure 2), alcohol, used in excess, is recovered by evaporation followed by condensation, and subsequent dehydration, so that it can be reused in the process. The list of components existing in the subsystem used in the processing of biodiesel are shown in Table 2, together with their respective tags, as well as the main parameters used to control each subsystem. A prototype of the production unit was developed to enable the processing of biodiesel from different oilseeds, using ethyl or methyl alcohol. The construction and disposition of

equipment at the plant for the continuous production presented in the PFD, in Figures 1 and 2, and then, in Figures 3 and 4. Figure 3 represents the 3D of the plant, which allows observing the spatial layout of the unit and the allocation of equipment, while in Figure 4, the arrangement of some equipment in the plant is presented in an organized way. Thus, there are the CSTR and PFR reactors shown in Figures 4(a) and (b). The ethanol decantation and depletion systems, in the case of the ethyl route, are shown in Figures 4(c) and (d).

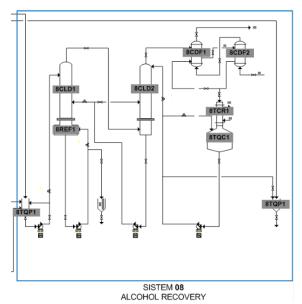


Fig. 2. Basic engineering diagram of the subsystem 08.

The columns for washing, evaporating moisture and recovering alcohol, used in excess at the beginning of the process, are demotivated in Figure 4(e), (f) and (g). In this Figure, the tagging described in Table 02 is presented in order to facilitate the monitoring of the process described in the PFD.

Once the steps have been described with all subsystems, the evaluation of the implementation of the control strategies should include all the parameters that are present in this plant. The system presented in this work aims to make the production of ethyl and methyl flexible, therefore, during the process of adjusting the control strategies to be used, one must ensure that there will be a reduction in the response time in relation to the disturbances that may occur in the parameters being controlled. It should also be noted that if using sensors and transducers, as well as making an appropriate installation of the final control elements, there will be a considerable influence in qualitative terms on the strategies proposed here, as well as on the quality of the final product obtained.

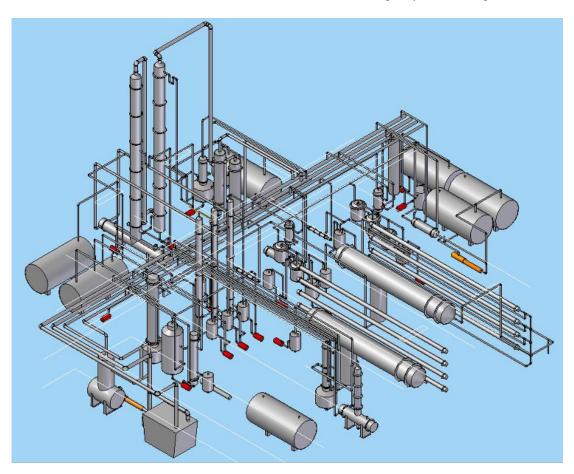


Fig. 3. Three-dimensional rendering of the production plant.

Table 2. List of components existing in the system.

| Subsystem | TAG | Equipment | Controlled/Manipulated parameters |
|---------------------------------|---------------------------------------|----------------------------------------------------------------------------|------------------------------------------------------------------------------------|
| 1 | 1TQA1 1TPC1 1TQP1 1TQA2 - A3 | Anhydrous alcohol tank Catalyst tank Lung catalyst tank Vegetable oil tank | -Alcohol flow and liquid level in 1TPC1; -Liquid level in 1TQP1; |
| | 2TCA1 | Vegetable oil heater | -Flow of alcohol and catalyst solution in 2CSTR1 and 2MIX1; |
| 2 | TIT-V5 | Tracer tank | -Temperature, pressure and level of tanks CSTR1 to CSTR4; |
| | 2MIX1 | Static mixer | -Liquid level and temperature in 2TQP1; |
| | 2TQP1 | Lung tank | -Flow and temperature from PFR1 to PFR4; |
| | 2CSTR1-4 | Transesterification reactor | -Fractions of the reaction products using an in-line chromatograph; |
| | 2PFR1-4 | Transesterification reactor | |
| 3 | 3DEC1 | Horizontal decanter | -Flow of the reacted medium at the inlet of 3DEC1; |
| | 3TQA1 | Collector tank of light phase | -Volume of glycerin in the lower region of 3DEC1; |
| | 4TCA1 | Vegetable oil heater | Flow of alcohol and catalyst solution in 2CSTR1 and 2MIX1; |
| 4 TIT-V5 4 4MIX1 4TQP1 4CSTR1-3 | TIT-V5 | Tracer tank | -Temperature, pressure and level of tanks CSTR1 to CSTR4; |
| | 4MIX1 | Static mixer | -Liquid level and temperature in 4TQP1; |
| | - | Lung tank | -Flow and temperature from PFR1 to PFR4; |
| | 4CSTR1-3 | Transesterification reactor | -Fractions of the reaction products using an in-line chromatograph; |
| | 4PFR1-4 | Transesterification reactor | |
| 5 | 5CLE1 | Depletion of alcohol | -Flow of the ester phase at the inlet of the horizontal decanter; |
| | 5REF1 | Reboiler | -Volume of glycerin in the lower region of the decanter; |
| | 5CDF1 | Alcohol condenser | -Liquid level in 5CLE1; |
| | 5TCR1 | Alcohol cooler | -Flow of thermal oil in 5REF1; |
| | 5TQC1 | Condensate collector | |
| | 5DEC1 | Horizontal decanter | |
| | 6CLL1-2 | Wash column | -Flow of the process water in 6CLL1 and 2; |
| 6 | 6CLH1 | Acid treatment column | -Flow of diluted acid at 6CLH1; -Ph control in 6CLH1; |
| 7 | 7CDF1 | Water condenser | -Flow of thermal oil in the evaporator liners and distillation tower; |
| | 7TQC1 | Condensate collector | -Humidity within 7EEVA1 and 2; |
| | 7EEVA1 | Evaporator | |
| | 7EEVA2 | Evaporator | |
| | 7FIL1 | Filter press | |
| | 7TQP1 | Dry biodiesel collector | |
| | 8CLD1-2 | Alcohol distillation column | -Flow of thermal oil for 8REF1; |
| | 8REF1 | Reboiler | -Liquid level in 8TQC1; |
| | 8CDF1-2 | Alcohol condenser | -Flow of alcohol vapor leaving 8CLD2. |
| 8 | QTCD1 | Alcohol cooler | |
| 8 | 8TCR1 8TQC1 | Alcohol cooler Condensate collector | |

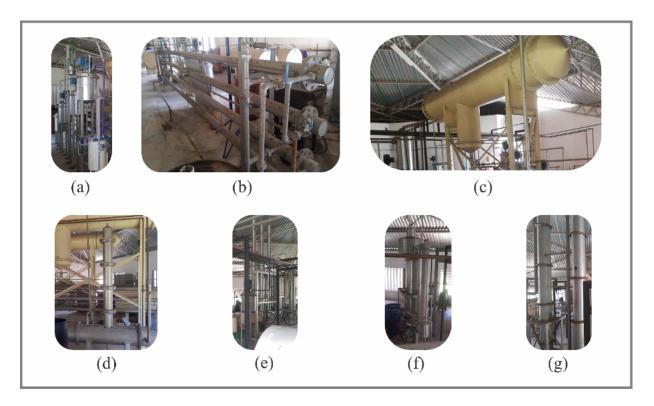


Fig. 4. Construction and presentation of equipments: (a) 2CSTR1-4, (b) 2PFR1-4, (c) 3DCE1, (d) 5CLE1/5REF1, (f) 6CLL1-2/6CLH1, (f) 7EEVA1-2, and (g) 8CLE1-2.

3.2 Subsystems and control techniques

3.2.1 Subsystems 01 and 02

For the implementation of control strategies, the plant was initially divided into subsystems, so that a more careful analysis of the processing steps that occur during the production of biodiesel could be made, and thus, identify the process variables that demand the greatest need for control. Figure 5 presents a processing flowchart of the production plant, showing briefly the steps that make up the plant's operation.

The raw material preparation stage, system 01, consists of supplying the reagents in the initial stage of the process. Thus, the beginning of the process is characterized when there is a simultaneous supply of these products from vegetable oil tanks and catalyst dilution tanks, in addition to alcohol. The alcohol, methyl or ethyl, is initially used to dilute the catalyst (NaOH or KOH), so that it can be pumped through the pipes of the plant easily and efficiently, then the vegetable oil is pumped through a heat exchanger for the transesterification subsystem I. The alcohol and the catalyst diluted in alcohol

(catalyst solution) are pumped simultaneously, both for the transesterification subsystem I and for the transesterification subsystem II.

Figure 6 shows both the plant feeding system and the transesterification subsystem I, represented by subsystems 01 and 02. For the control of the feeding system, it was initially created a node 01-S01, which encompasses two control strategies. The first one consists of a time controller, inserted in the alcohol line that enters in the dilution tank of catalyst 1TPC1 and in the impeller motor of the tank. After receiving from controller the command to start the process, there will be an activation of the valve that allows alcohol to enter the tank 1TPC01. The alcohol will continue to enter the tank for the pre-set amount of time. Then, after the time is computed, the controller will start the tank impeller motor. This impeller will operate for the pre-set time, shutting down after the time has elapsed. This whole process guarantees that the catalyst solution obtained will have been well diluted.

The second strategy for node 01-S01 consists of one control by ratio and proportion applied to tanks 1TPC1 and 1TQP1.

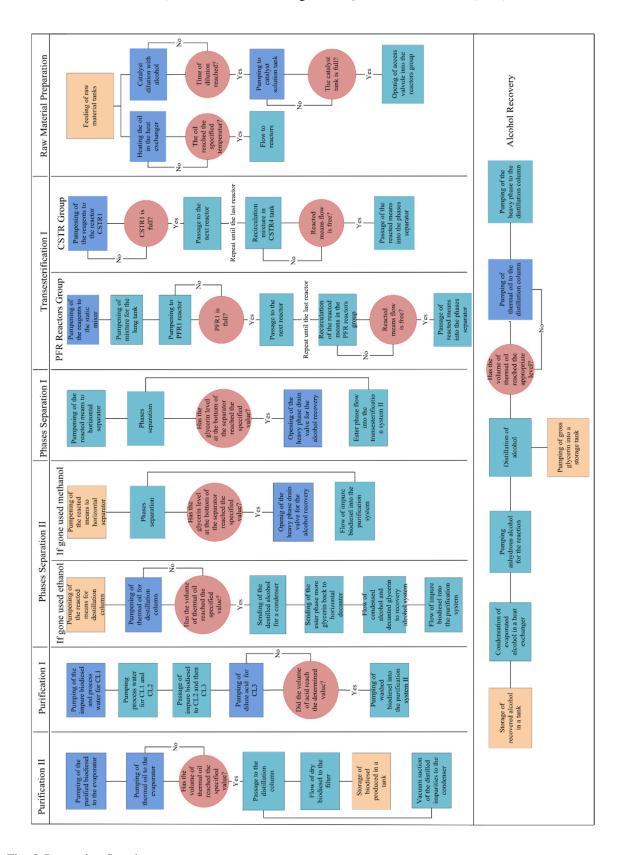


Fig. 5. Processing flowchart.

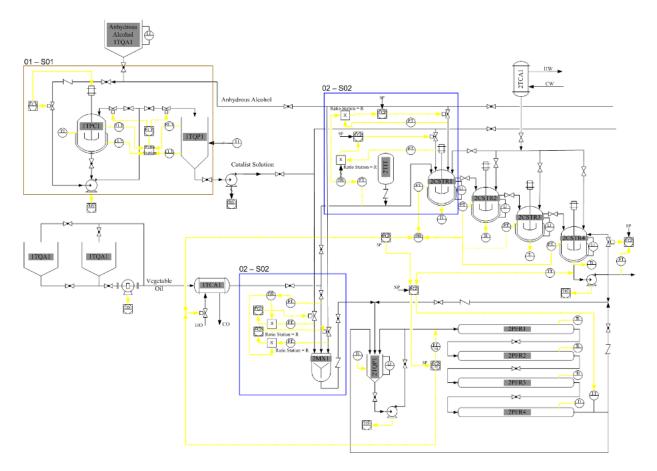


Fig. 6. Control diagram for subsystems 01 and 02.

The control starts with the reading of the liquid level in the tanks, followed by sending the values read to a ratio station that determines the set point for the flow controller installed in the catalyst solution line. In this way, if the 1TQP1 is at a low level (LL) and 1TCP1 is at a high level (HL), the controller will allow the catalyst solution prepared in 1TPC1 to reach 1TPQ1. If 1TQP1 is empty, the controller will send a signal to the control panel informing that it is necessary to refill it with catalyst and activate the timer. Thus, it is possible to control the liquid level in each tank so that they always remain between their respective minimum (LL) and maximum (HL) levels.

In subsystem 02, it occurs the transesterification stage, which, in general, consists of a reaction where an ester is obtained from the replacement of the glycerin molecule with another alcohol (Geris *et al.*, 2007). In this proposed project, the transesterification process can be performed by any of the reactor groups (CSTR or PFR). If the reactors are of the CSTR type, the process begins with the supply of reagents. After the entry of these reagents, the impeller

of the rotors will be responsible for mixing these components. However, if PFR type reactors are used, the mixture will be made by the static mixer 2MX1. The temperature condition of the reactional system, throughout all the reaction, must be maintained at 60°C, and the amount of reagents that must be supplied for the reactor groups is according to a specific preestablished proportion, as shown in Table 01.

The control of subsystem 2 starts in the preheating stage of vegetable oil in the heat exchanger 1TCA1, using thermal oil. For this situation, it was implemented a cascade control that acts on the thermal oil line that feeds the exchanger. The control consists, initially, of a temperature controller that receives information from the line of the reactional medium when leaving the reactor groups. The preheated oil can be directed to the CSTR or PFR reactors. Due to this possibility of sending oil to any of the reactors, the cascade system will use two secondary controllers to send information about which reactors will receive this oil. In the case where vegetable oil will be sent to CSTR type reactors, the primary controller (A)

will be responsible for informing the set point for the secondary controller (B). This second controller is responsible for the information about the pressures in the tanks, and after receiving the information about the set point, it will send a signal to the flow controller, which is in the oil line of the heat exchanger. Already, if the vegetable oil is directed to the PFR type reactors, the primary controller (A) will be responsible for sending the information about the set point to the secondary controller (B). In the same way as in the previous case, the secondary controller will be responsible for the information about the pressures in the tanks, and after receiving the information about the set point, it will send a signal to the flow controller, which is in the 1QCA1 oil line.

The implementation of the cascade control described above aims to obtain a more accurate response about the mixture. As explained earlier, only vegetable oil is preheated. Thus, the process will be inaccurate and with a considerable error, if the temperature control of the mixture is made only from information about the amount of heat that is supplied to the vegetable oil. In this way, a dynamic set point correlating some state variables of the mixture provides the system with a more stable and faster response. In addition, with a more precise control over the mixture, it is possible to obtain, in the absence of other disturbances, a constant alcohol evaporation rate in the CSTR reactors, which eliminates the need for control temperature of the cooling water of the 2TCA2 heat exchanger. The remaining part of subsystem 2 consists of two nodes, 01-S02 and 02-S02, which apply to the CSTR and PFR reactor groups, respectively. In each of these two nodes, it was implemented a ratio and proportion control system, where the first one is in the alcohol line and the second one is in the catalyst solution line. The procedure is as follows: the controller receives from the vegetable oil line the information about what is the flow that passes through it. This information is then, transmitted to the ratio stations, which are responsible for calculating the set points. Once the set points are established, this information is provided to the flow controllers, which are on the oil lines. Thus, the supply of new reagents for the process will be maintained according to a proportion ratio with the flow of vegetable oil. This system is implemented both at the entrance of the 2CSRT1, giving access to the CSRT reactor group, and at the entrance of the 2MX1 static mixer, giving access to the PFR reactor group.

3.3 Subsystems 03 and 04

Subsystem 03 was developed to perform the separation of the ester and glycerin phases, which consists of the separation of the light and heavy phases of the reactional medium. This step takes place in a horizontal separator (3DEC1), where the layers, heavy and light, are divided due to the difference in density. As the reactional medium enters the horizontal decanter, the heavy phase accumulates, by the action of gravity, at the bottom of the unit, while the light phase flows through the main top outlet. The heavy phase accumulated at the bottom, flows through a valve that accesses the alcohol recovery system, while the light phase goes to the lung tank 3TPQ1, from where it is sent to the subsystem 04. For the control of subsystem 03, it was used only a single node, where a simple feedback control is applied, which consists of a control level of glycerin accumulated in the bottom of the decanter. This system is composed of a glycerin biosensor, responsible for monitoring in real time what is the amount of glycerin that is present in the medium (Šefčovičová et al., 2008). The biosensor is implemented at the bottom of the decanter, where it will be responsible for sending a signal to the level transducer, informing that the amount of glycerin is increasing in the reservoir. This level transducer will be responsible for transmitting to the flow controller, a command to open and close the valve that gives access to the heavy phase into the alcohol recovery system.

The subsystem 04 represents the second stage of transesterification. During this second stage, the objective is to ensure that reagents, which were supplied in the first stage, will be converted into a new reagent with a higher quality when compared to its quality in the initial stage. Thus, the system was subdivided into a stage of heating of the reactional medium, followed by two nodes 01-S04 and 02-S04, which are based on the same principles as the nodes of subsystem 02. The heating stage of the reactional medium is developed according to the same logic presented in the preheating stage of the vegetable oil, in system 01. As this subsystem works similarly to the subsystem used for preheating vegetable oil, mentioned earlier, it was decided to use a cascade control system here. Nodes 01-S04 and 02-S04 were implemented following the same purpose as nodes 01-S02 and 02-S02 of subsystem 02, respectively. Therefore, they also present control strategies by ratio and proportion in the alcohol and catalyst solution lines, with the difference that the flow of these products now follows the flow

of the reactional medium. The control strategies implemented in subsystems 03 and 04, as well as the automation flowchart are shown in Figure 7.

In phase II separation, biodiesel is separated from glycerin and alcohol, and then, it is sent to the purification system. The separation can occur in two ways, depending on the alcohol used in the process. If methanol is used, the separation occurs just like in the phase I separation system (at this stage, the ester can be sent back to the beginning of the process, or be taken to the sewage system, or it can be sent to the decanter). On the other hand, if the alcohol used in the transesterification reaction is ethanol, the reacted medium, from the transesterification system I, is sent to a reboiler and an alcohol depletion column, so that the emulsion formed in the mixture is broken. From this equipment, two streams are then separated, impure biodiesel and alcohol vapor. The first one can proceed either to the next subsystem (purification I) or return to the phase II separation entrance (being taken either to the decanter or to the alcohol depletion column again), while the second one is directed, in order, to an alcohol condenser, an alcohol cooler and a condensate collector. The alcohol recovered, as a result of these steps, goes to subsystem 08 (alcohol recovery).

3.3.1 Subsystems 05 and 06

The control of the separation system II was divided into three nodes. The first one, 01-S05, corresponds to a ratio-proportion control applied to the hot oil line of the 5REF1 reboiler from the measurement of the flow rate of the reactional medium (MR). This information is sent to a flow controller of the MR line and a ratio station, which, in turn, determines the necessary set point for another flow controller installed in the thermal oil line. Once the plant has continuous operation, a constant proportion of the flow rate of MR and thermal oil will be decisive for maintaining the alcohol evaporation rate. This avoids, for example, having a control of the flow of cold water that stops through the condenser and alcohol cooler. The second one, node 02-S05, involves only a level control applied to the condensate collector tank, so that the alcohol volume is always between the extreme levels, opening the flow valve at the high level (LH) and closing at the low level (LL).

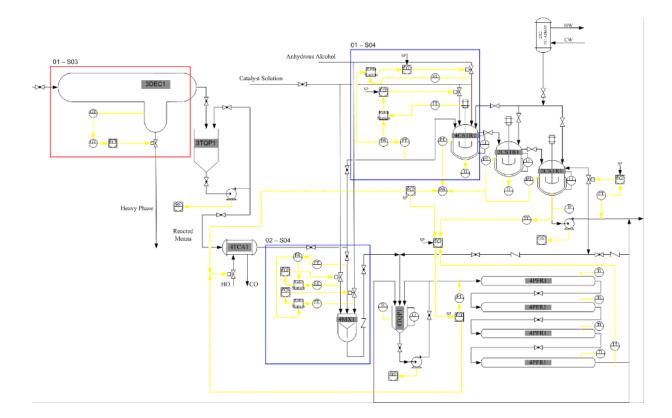


Fig. 7. Control diagram for subsystems 03 and 04.

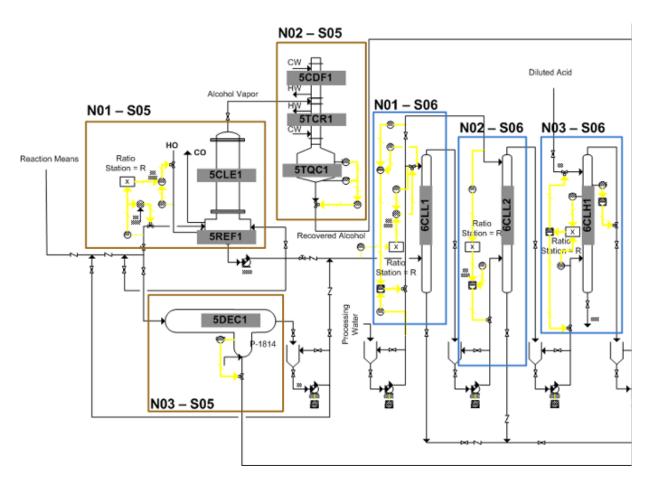


Fig. 8. Control diagram for subsystems 05 and 06.

Already the node 03-S05 has the same control logic explained in subsystem 03 through the utilization of a glycerin sensor. Subsystem 05, together with subsystem 06, is shown in Figure 8.

In subsystem 06, the biodiesel purification occurs through washing and acid treatment processes. The washing takes place in two stages, in columns 6CLL1 and 6CLL2, by adding water to biodiesel in order to extract traces of glycerin and reduce the soap content obtained from the saponification reaction between catalyst and vegetable oil. After this stage, the biodiesel is subjected to an acid treatment in 6CLH1 in order to hydrolyze the traces of still existing soaps, from which fatty acids and inorganic salt are removed. For this purpose, on node 01-S06, a split range control was used, in which the flow controller receives information on the process water flow from the supply lines of the first two columns and acts to allow the passage of sufficient water volume for both. At the same time, a ratio and proportion control system is employed on the part surrounding the 6CLL1

column feed valve. The use of this system is to prevent that the amount of water, which has to be sent to the process of removing impurities from the biodiesel present in this column, to be insufficient.

In node 02-S06, a ratio and proportion control system was used for the same purpose mentioned above. In the 6CLH1 acid treatment, two sensors, one of Ph and the other one of flow, send signals to the ratio station that establishes a control set point in the flow controller, which acts on the valves that regulate the passage of diluted acid and biodiesel moist. This ratio and proportion control was used in order to obtain a faster response to possible disturbances in the diluted acid and washed biodiesel feed lines, which could compromise the product characteristics, since in the continuous process part of the biodiesel could go to the other steps with acidic or basic nature. In order to have greater security, in this project, it was decided to add another Ph sensor, where it will be associated with a flow controller that acts directly on the outlet valve of column 03, in order to interrupt the passage

of biodiesel if the desired Ph is not reached in the acid treatment process.

3.3.2 Subsystems 07 and 08

Subsystem 07 consists in the second stage of the biodiesel purification, by a dehumidification process through drying in an evaporator with vacuum recirculation. The second stage of the biodiesel purification (07) and the recovery of alcohol (08) are shown in Figure 9.

The automation proposed in these last subsystems is, in general, simpler. Moisture meters are associated with nodes 02-S07 and 03-S07, and are responsible for reading this variable and sending a signal to a flow controller. This component, in turn, acts on the thermal oil inlet valve in the evaporator jacket. In 7EEVA1 and 7EEVA2, there is a loss of alcohol vapor; therefore, it is necessary to place a control system in the supply line of the condensation tower, in order to have a greater control of the steam flow. This solution, controlling a supply line, is similar to the one used in 01-S05. Besides, it can be avoided control over the flow of cold water in 7CDF1. On the other hand, nodes 04-S07 and 05-S07 have only feedback controls for the flow of wet and dry biodiesel, respectively, in the pump discharge piping.

As is already known, alcohol, methanol or ethanol, is used in excess in relation to the stoichiometric

of the reaction and, therefore, in the later phases, there may be a large amount of this component. Such excesses are recovered by evaporation, followed by condensation and result in alcohol with a significant water content (hydrated). However, it is necessary that it goes through a dehydration process so that it can be reused in the process.

For the recovery of alcohol, subsystem 08, a distillation tower is used. This process occurs by heating the recovered heavy phase, causing the alcohol to evaporate, separating it from the glycerin. This, in turn, is recovered in a tank based on 8CLD2 and the alcohol vapor is sent to a set of heat exchangers, 8CDF1 and 2, where it is condensed and then sent to the final storage tank, 8TQP1. After storage in this tank, the condensed alcohol vapor can be reused in the process. Control 01-S08 is used with the function of manipulating the flow of thermal oil, aiming to control, also, the amount of heat in the process, according to the level of hydrated alcohol found in the reboiler. This mesh consists of two level sensors (high level and low level), associated with a flow controller. In 02-S08, a flow controller was used to maintain a constant flow of steam through the inlet line of the exchangers, so that the cold water line does not need to be automated. Then, as in 02-S05, in 03-S08, it was used a level control (high level and low level) that acts on the exhaust valve of 8TQC1.

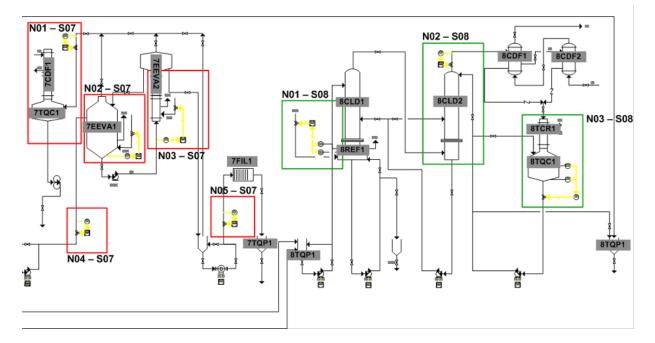


Fig. 9. Control diagram for subsystems 07 and 08.

Conclusions

In this present work, relevant points to the production process were highlighted using ethanol (C₂H₅OH) and methanol (CH₃OH) alcohols. In this perspective, this work stands out for presenting, in an integral way, an entire continuous process of biodiesel production with different production routes, enabling the evaluation of the implementation of control strategies. Additionally, it can be highlighted that the monitoring of flows in the PFD (Plug Flow Reactor) contributes to the appropriate choices of level, flow and temperature controls that must be followed for phase separation in the unit decantation system (3DEC1 and 5DEC1). Thus, the integration of the strategies, presented in the P&ID, allowed that all the stages present in the biodiesel production process, could follow their pre-established dynamics in the project. Finally, the strategies related to this unit make it possible for the description of the stages and further developments in the process to collaborate with the creation of technologies that will be developed and implemented on large-scale production.

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References

- Birchal, M. A. S., Birchal, V. S. (2013). Automação de uma planta de produção de biodiesel. *Exacta 6*, 139-145.
- De-Jesus, D. S. S. (2013). Design automation of the manufacturing process of a minibiodiesel plant. IFAC Proceedings Volumes 46, 32-39. https://doi.org/10.3182/20130911-3-BR-3021.00006
- Dhar, P., Mazumdar, A. (2012). Automation of biodiesel plant with bio-sensing technologies.
 In: 2012 1st International Symposium on Physics and Technology of Sensors (ISPTS-1).
 IEEE. March 7-10. India, Pune, 319-325.

- Geris, R., Santos, N. A. C., Amaral, B. A., Maia, I. S., Castro, V. D., Carvalho, J. R. M. (2007). Biodiesel from soybean oil: experimental procedure of transesterification for organic chemistry laboratories. *Química Nova 30*, 1369-1373. http://dx.doi.org/10.1590/S0100-40422007000500053
- González-González, R., Flores-Márquez, J. A., López-Sánchez, E., Rodríguez-Jimenes, G. C., Carrillo-Ahumada, J., García-Alvarado, M. A. (2020). Non-competitive \mathcal{L}_2/D control applied to continuous concentric tubes heat exchangers. Revista Mexicana de Ingeniería Química 19, 569-583. https://doi.org/10.24275/rmiq/Sim669
- Hasheminejad, M., Tabatabaei, M., Mansourpanah, Y., Khatami-far, M., Javani, A. (2011). Upstream and downstream strategies to economize biodiesel production. *Bioresource Technology 102*, 461-468. https://doi.org/10.1016/j.biortech.2010.09.094
- Hassan, M. H., Kalam, M. A. (2013). An overview of biofuel as a renewable energy source: development and challenges. *Procedia Engineering* 56, 39-53. https://doi.org/10.1016/j.proeng.2013.03.087
- IPCC (Intergovernmental Panel on Climate Change). (1997). Greenhouse gas inventory reference manual: revised 1996 IPCC guidelines for national greenhouse gas inventories. Report 3. Paris, France. Available at: https://www.ipcc-nggip.iges.or.jp/public/gl/invs6.html. Accessed: September 14, 2020.
- ISA (International Society of Automation). (2009). ISA5.1, Instrumentation Symbols and Identification. Retrieved from https://webstore.ansi.org/preview-pages/ISA/preview_ANSI+ISA+5.1-2009.pdf
- Munir, M. T., Yu, W., Young, B. R. (2013). Plantwide control: Eco-efficiency and control loop configuration. *ISA Transactions* 52, 162-169. https://doi.org/10.1016/j.isatra.2012.09.006
- Oğuz, H., Özcan, M., Yağci, M., Özkan, A. O. (2015). Automation of the two stage biodiesel production process/Iki Aşamali Biyodizel Üretim Süreci Otomasyonu. *International*

- Journal of Automotive Engineering and Technologies 4, 254-260. https://doi.org/ 10.18245/ijaet.27257
- Özcan, M., Oğuz, H., Öğüt, H. (2005). Application of automation system in biodiesel production 4. In: *International Advanced Technologies Symposium*. September 28-30. Turkey, Selçuk Üniversity, 487-491.
- Prudente, F. (2007). Automação industrial. *PLC Teoria e aplicações* (in Portuguese). LTC, São Paulo, Brasil.
- Ramadhas, A. S., Jayaraj, S., Muraleedharan, C. (2004). Use of vegetable oils as IC engine fuels a review. *Renewable energy* 29, 727-742. https://doi.org/10.1016/j.renene.2003.09.008
- Regalado-Méndez, A., Romero, R., Natividad, R., Skogestad, S. (2016). Plant-Wide Control of a Reactive Distillation Column on Biodiesel Production. In: Silhavy, R., Senkerik, R., Oplatkova, Z., Silhavy, P., Prokopova, Z. (eds). Automation Control Theory Perspectives in Intelligent Systems. CSOC 2016. Advances in Intelligent Systems and Computing 466, https://doi.org/10.1007/978-3-319-33389-2_11
- Rivas-Perez, R., Sotomayor-Moriano, J., Perez-Zuñiga, C. G., Calderon-Mendoza, E. M. (2016). Design of a multivariable GPC based on an industrial PC for control of a reverse osmosis unit of a pharmaceutical industry. *Revista Mexicana de Ingeniería Química 15*, 259-273.
- Rodríguez-Mariano, A., Reynoso-Meza, G., Páramo-Calderón, D. E., Chávez-Conde, E., García-Alvarado, M. A., Carrillo-Ahumada, J. (2015). Comparative performance analysis of different linear controllers tuned for several Cholette's bioreactor steady states using multi-criteria decision making techniques. *Revista Mexicana de Ingeniería Química 14*, 167-204.
- Sensoz, S., Angin, D., Yorgun, S. (2000). Influence of particle size on the pyrolysis of rapeseed (*Brassica napus* L.): fuel properties of biooil. *Biomass Bioenergy 19*, 271-279. https://doi.org/10.1016/S0961-9534(00)00041-6

- Šefčovičová, J., Katrlík, J., uStefuca, V., Mastihuba, V., Voštiar, I., Greif, G., Bučko, M., Tkac, J., Gemeiner, P. (2008). A filtration probe-free online monitoring of glycerol during fermentation by a biosensor device. *Enzyme and Microbial Technology* 42, 434-439. https://doi.org/10.1016/j.enzmictec.2008.01.006
- Shahid, E. M., Jamal, Y. (2011). Production of biodiesel: a technical review. Renewable and Sustainable Energy Review 15, 4732-4745. https://doi.org/10.1016/j.rser.2011.07.079
- Silva, B. F. (2015). Desenvolvimento e avaliação de sistemas de controle plantwide aplicados na produção de biodiesel. *Tesis de Maestría en Ingeniería Química*, Universidade Estadual de Campinas, Brasil.
- Wali, W. A., Cullen, J. D., Bennett, S., Al-Shamma'a, A. I. (2013). Intelligent PID controller for real time automation of microwave biodiesel reactor. *International Journal of Computer and Information Technology* 2, 809-814.
- Wang, Y., Wang, X., Liu, Y., Ou, S., Tan, Y., Tang, S. (2009). Refining of biodiesel by ceramic membrane separation. *Fuel Processing Technology* 90, 422-427. https://doi.org/10.1016/j.fuproc.2008.11.004
- Worm, H. (2012). Estudo da automação de uma planta piloto para a produção de biodiesel. *Tesis de Maestría en Sistemas y Procesos Industriales*, Universidade de Santa Cruz do Sul, Brasil.
- Yusuf, N. N. A. N., Kamarudin, S. K., Yaakub, Z. (2011). Overview on the current trends in biodiesel production. *Energy conversion and management* 52, 2741-2751. https://doi.org/10.1016/j.enconman.2010.12.004

Appendix

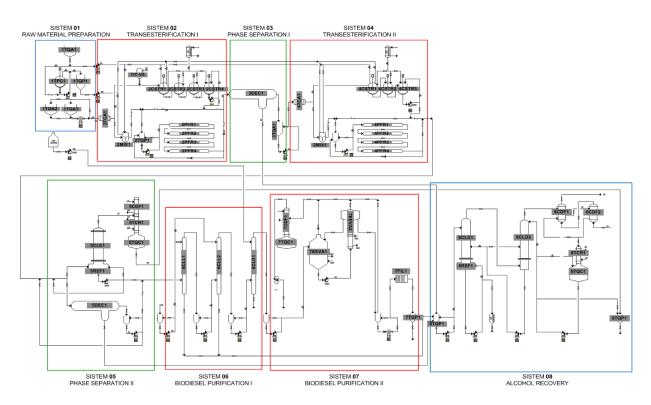


Fig. A1. PFD for the continous biodiesel plant.