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THE EFFECT OF CRITICAL PARAMETERS ON THE OIL SEPARATOR VESSEL (102D1) TO REDUCE THE VALUE OF FATTY ACID ESTER (FAE) IN THE GLYCERIN WATER PRETREATMENT UNIT AT PT. DOMAS AGROINTI PRIMA

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ABSTRACT

Glycerin water produced from the hydrolysis process in the splitting column still contains impurities such as oil or fatty acids that are carried along with glycerin water or other impurities found in the raw oil itself such as solid particles and gum. Oil or fatty acids that are still present in glycerin water must be removed or lowered to the maximum permissible limit before further processing in glycerin evaporation units and glycerin distillation units. The quality standard set for glycerin water is the value of fatty acid ester (FAE) in glycerin water. Glycerin water will be treated first in the glycerin water pretreatment unit to remove the oil and fatty acid content. Oil separator vessel (102D1) is one of the tools in the glycerin water pretreatment unit, is one of the important ones to control in the glycerin water pretreatment unit. Where in this oil separator vessel there will be an initial separation between oil and fatty acid with glycerin water. Optimization of separation in oil separator will determine the decrease in FAE value in (102D1) treated glycerin water end product. Observations were made on the oil separator vessel, namely temperature and pH to see both parameters of this process against the decrease in the value of fatty acid ester, data collection for temperature and pH was carried out 3 times a day for 6 days for each still paremeter to determine the value of fatty acid ester. From the observed data on the oil separator device, there are two parameters that determine the separation of oil and fatty acid from glycerin water, namely temperature and pH. The results obtained that temperature and pH greatly affect the value of fatty acids, where the higher the temperature, the lower the FAE value, from the observation data the optimum temperature is 90 – 95 oC. pH also greatly affects the value of fatty acid ester after oil separator, where the clover's pH is low in its fatty acid ester value. From the observed data, the optimum pH is pH 2 – 3.5

Keywords: *Fatty Acid Ester, Oil Separator, Pretreatment Glycerin Water, pH, Temperatur*

1. INTRODUCTION

The development of the oleochemical industry in Indonesia continues to grow rapidly in line with the increasing market demand for oleochemical products both in Indonesia and in the international market. The large market demand for oleochemical products is based on increasing production capacity. Companies or industries continue to strive so that the process can run optimally beyond capacity and avoid unplaned downtime or breakdown. Splitter is the main tool or main unit that functions as a breaker of oil or triglycerides into fatty acids and glycerin by the hydrolysis process method. Fatty acid or so-called crude fatty acid that comes out of the top of the splitter column will be flowed into a storage tank for further processing through the process of fractionation and distillation to become various kinds of fatty acid products. While

glycerin water that comes out of the bottom of the splitter column will also flow into the storage tank for further processing through several stages of the process (pretreatment, evaporation and distillation) until it becomes the final product refined glycerin (Operating Manual Glycerin Plant, JJ Lurgi). Glycerin water is glycerin obtained from the process of hydrolysis of oil or triglycerides in a splitting column where oil or triglycerides will separate into fatty acids and glycerin. Fatty acid will come out of the top of the spliter column while glycerin dissolved with water will come out of the bottom of the spliter column called glycerin water. Oil or fatty acid found in glycerin water is a impurities. In addition, impurities in glycerin water also come from raw oil itself such as; non-glycerin organic material, sap or glue or other solid particles. Before glycerin water is further processed in the glycerin evaporation and glycerin distillation units until it becomes a finish product, these oils and fatty acids must be removed or lowered to the specified standard limit. To remove or reduce the oil content is carried out in the glycerin water pretreatment unit. This pretreatment unit is very important and critical. If glycerin water processing in the pretreatment unit fails, it will cause disruption to the next process and product quality will also be disrupted.

The glycerin water pretreatment unit is very important, the process stages in this unit must be absolutely ensured to run well. There have been several times that glycerin evaporation units and glycerin distillation units have experienced quality problems and decreased production capacity due to the performance of pretreatment units that are not optimal. For this reason, it is very important to keep the pretreatment unit run properly and know the critical points that can affect quality, these critical points must be maintained so that the quality of the products produced from this unit can be maintained according to standards.

Oil separator which is the first stage for separation between oil and glycerin water where oil in emulsion form mixes with glycerin water. At this stage there will be gravitational separation where oil that has a lower specific gravity will separate and rise to the surface. The performance of the oil separator will affect the results of the next stage. Therefore, the performance of oil separators is very important to reduce the value of FAE and other impurities.

In the study, we want to see the influence of critical process parameters on the glycerin wate pretreatment unit oil separator tool which is important to control, know the critical points that are optimu on the glycerin water pretreatment oil separator tool and know good prevention and maintenance efforts to prevent problems related to the quality of the refine glycerin operator product in running the glycerin water pretreatment unit properly and correctly.

2. RESEARCH METHODS

2.1 Tools and Equipment Performance Indicators

2.1.1 Tools used

The equipment that is the focus as an object of observation is the oil separator vessel (102D1) Pretreatment Glycerin Water.

2.2 Equipment Performance Indicators

The performance indicators of the Glycerin Water Pretreatment unit are as follows:

1. Glycerin water temperature in vessel oil separator
2. pH conditions in the oil separator vessel

2.3 Equipment Performance Evaluation Plan

In order to optimize the glycerin water pretreatment unit, observation and data collection will be carried out directly in the field and data from the equipment design for the 102D1 device

2.3.1 Temperature

The temperature condition of glycerin water will be observed to determine the separation between oil and glycerin water that takes place in the vessel oil separation (102D1) at a predetermined temperature standard. To

find out how various conditions of glycerin water temperature affect the separation of oil and glycerin water.

2.3.1 pH

The pH condition of the oil separation vessel (102D1) will be observed and see the separation of oil and glycerin water at the pH standard that has been set.

3. RESULT AND DISCUSSION

The observation data for various temperatures at pH 2 – 3 can be seen in table 1

Tabel 1. Data from temperature observation at pH 2-3

Date	Hour	Temperature	FAE (ml NaOH 0,5 N)
7 oct 2023	09.00	70	7.33
	17.00	71	7.2
	23.00	70.5	7.2
8 oct 2023	09.00	75	6.8
	17.00	75	6.9
	23.00	75.2	6.7
9 oct 2023	09.00	80	5.5
	17.00	80.5	5.4
	23.00	79.8	5.4
10 oct 2023	09.00	85	4.3
	17.00	85	4.4
	23.00	85.2	4.2
11 oct 2023	09.00	90	3.4
	17.00	90.5	3.3
	23.00	90.2	3.5
12 oct 2023	09.00	95	3.2
	17.00	95.1	3.3
	23.00	95	3.1

Observation data for various pH at a temperature of 90 – 95 ^{o}C can be seen in Table 2

Tabel 2. Data From the observation pH againts temperature 90-95^{o}C

Date	Hour	Temperature	FAE (ml NaOH 0,5 N)
16 oct 2023	09.00	1.2	3.0
	17.00	1	3.1
	23.00	1.1	3.2
17 oct 2023	09.00	2	3.2
	17.00	2.1	3.3
	23.00	1.9	3.4
18 oct 2023	09.00	3	3.3
	17.00	3.1	3.4
	23.00	3	3.3
19Oct 2023	09.00	4	4.8
	17.00	4.1	4.9
	23.00	3.9	4.6
20Oct 2023	09.00	4.9	5.1
	17.00	5	5.3
	23.00	5	5.3

3.1 Discussion

Glycerin water produced from the hydrolysis process in the splitter column still contains impurities in the form of oil and fatty acids. At the bottom of the splitter column, it is expected that there will be a perfect separation between glycerin water as a heavy phase with oil and fatty acid as a light phase. But in reality, there are still fatty acids and oils carried along with glycerin water. The oil and fatty acids contained in glycerin water are in very small sizes or emulsified in glycerin water.

Glycerin water before further processing is carried out in the evaporation and distillation unit to produce refined glycerin must be cleaned of impurities. The unit that functions to eliminate impuritis is the pretreatment unit. The pretreatment process of glycerin water consists of several stages, namely oil separator, treatment vessel and filtration. One of the quality standards that must be considered in the final product of pretreatment is the value of fatty acid ester (FAE). Special temperature and pH observations have been made on the 102D1 oil separator.

Glycerin water before entering the oil separator is heated first using a heat exchanger. From the results of observation and data collection for different temperatures in oil separator 102D1, it can be seen that temperature greatly affects the value of fatty acid ester (FAE). Temperature data collection starts from 70 ^{o}C and every 5^{o}C increase to 95^{o}C. From the data, it can be seen that the temperature of glycerin water in the oil separator is inversely proportional to the value of the fatty acid ester produced, the higher the temperature, the lower the value of the fatty acid ester. This happens because at high temperatures, the density of oil and fatty

acids will be low so that the oil and fatty acids contained in glycerin water in small sizes as a light phase of glycerin water will be easier to separate and rise to the surface. In the data table, the observation results show that at a temperature of 70^{o}C the value of fatty acid ester is 7.2, at a temperature of 75^{o}C the value of fatty acid ester is 6.8, at a temperature of 80^{o}C the value of fatty acid ester is 5.43, at a temperature of 85^{o}C the value of fatty acid ester is 4.3, at a temperature of 90^{o}C the value of fatty acid ester is 3.4 and at a temperature of 95^{o}C the value of fatty acid ester is 3.2

The value of fatty acid ester continues to decrease along with the increase in temperature, it can be seen that the lowest value of fatty acid ester is obtained at a temperature of 90 – 95 oC. So the optimum condition for the temperature in the oil separation vessel 102D1 is 90 – 95^{o}C. In its operation, the temperature in the oil separator 102D1 has been controlled automatically using a control valve so that the temperature stability of glycerin water entering the oil separator can be maintained continuously. Observations for temperature are limited to a temperature of 95^{o}C to avoid evaporation due to boiling water if the temperature reaches 100^{o}C.

From the observation data of FAE values on temperature changes, there is a large influence. This occurs because the density of oil or fatty acids in glycerin water gets heat into the system which is a form of energy. When this heat energy is absorbed by the oil or fatty acid contained in glycerin water, it turns into kinetic energy which causes the oil or fatty acid molecules to begin to vibrate at a higher speed. This increase in speed causes more collisions than usual thus pushing each other. The movement of oil molecules or fatty acids away from each other causes the expansion of the substance thereby increasing its volume. Because the volume is inversely proportional to the density, the increase in temperature causes a decrease in density, therefore the fatty acids or oils in glycerin water will be faster and easier to separate to the surface.

Discussed based on the theory from the reference: *Savitri Devi (2023) " Does density change with temperature " www://techniescientist.com*

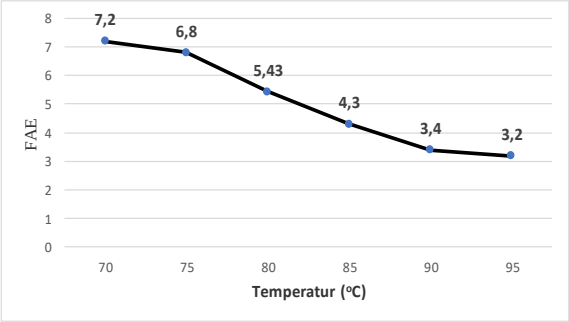

Figure 1. FAE vs Temperature value chart

Glycerin water before entering the oil separator, in addition to being heated, is also injected with a 32% HCl solution to acidify glycerin water until the pH value is determined. In addition to observing the temperature of the decrease in the value of fatty acid esters in glycerin water, changes in the value of fatty acid esters in glycerin water were also observed under different pH conditions. From the results of observation and data collection for different pH at a temperature of 90 -95^{o}C. Data collection for pH was carried out at different pHs starting at pH 1 and adding 1 to pH 5.

From the observation results, the following data were obtained: for pH 1 a fatty acid ester value of 3.10 was obtained, for pH 2 a fatty acid ester value of 3.30 was obtained, for pH 4 a fatty acid ester value was obtained a value of 4.59 and for pH 5 a fatty acid ester value was obtained a value of 5.26. From the observation data, it can be seen that the pH is directly proportional to the value of fatty acid ester, meaning that the glycerin acid water in the oil separator is a catalyst for a lot of oil and fatty acid ester as a light phase that is separate from glycerin water as a heavy phase. Glycerin water is treated in glycerin water pretreatment derived from splitting coloms *(autoclave sweetwaters)* by acidifying glycerin water by adding HCl solution.

Glycerin water acidification by adding HCl solution is as a coagulant *(Carl S miner & N.N Dalton 1953 " Glycerol " The Waferly Press Inc, Baltimore, MD, U.S.A.)*

Where small particles of oil and fatty acid contained in glycerin water in acidic environmental conditions will combine into a larger particle size so that it will be easier and faster to separate and rise to the surface as a light phase.

In the condition of pH 1, pH 2 and pH 3 it can be seen that the values of fatty acid esters are almost the same, meaning that at pH 1, pH 2 and pH 3 are optimal conditions but it needs to be noted that at pH 1 of course more doses of HCl will be consumed, if the difference in the fatty acid value produced is not too significant, then it is best to use pH 2 – 3. It is also very important to keep the pH not exceeding 3.5 because in the treatment vessel will be added $Ca(OH)_2$ which functions as a neutralizing pH as well as for the salinization process, where the oil and fatty acids that are still present in the glycerin water from the oil separator will become insoluble soap so that it can be separated by filtration.

In the oil separator for dosing, HCl has used a dosing pump but not automatically, to find out the pH only by using the pH indicator which can know the real-time pH through the monitor layer but for the pH setting is still done manually.

It should also be noted that oil and fatty acid material that has been separated to the surface in the oil separator must be periodically and routinely taken by skimming. Oil and fatty acids that have been separated on the surface if not taken over time will accumulate and will be carried back with the output from the oil separator to the treatment vessel so that it will have an impact on the further process.

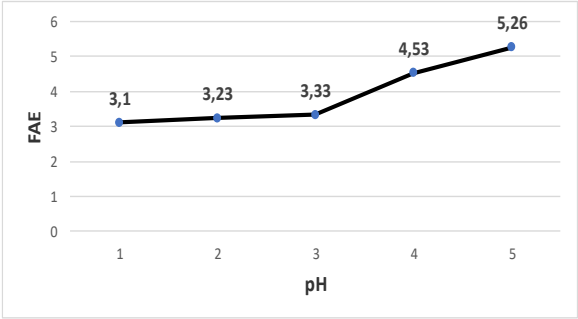

Figure 2 FAE vs pH value graph

From the observation results for temperature and pH, it can be seen that the value of fatty acid esters in the oil separator is greatly influenced by temperature and pH, so these two parameters are critical *parameters (critical control points)* that must be considered and controlled in the oil separator equipment.

4. CONCLUSION

4.1 Conclusion

Based on the results of the study, it can be concluded that:

1. Temperature and pH are process parameters that greatly affect the oil separator vessel 102D1, the heat catalyst is low in its FAE value, the low pH catalyst is in a low FAE value.
2. The optimum temperature in the oil separator vessel 102D1 is 90 – 95^{o}C and the optimum pH is 2 – 3.5
3. To maintain optimal oil separator performance, it is necessary to clean the vessel periodically every year, especially in the packing structure and routinely skimming the oil that is separated on the surface as many as 2 times per shift

5.1 SUGGESTION

To further optimize the performance of the oil separator vessel 102D1, it is recommended that HCl dosing can be done automatically so that pH stability can continue to be consistently maintained. In addition, it is recommended to add one more unit of oil separator of the same size that is

installed in series so that the residence time in the oil separator can be longer so that more oil or fatty acids are separated. It is recommended that the pH position of the transmitter be moved from the one previously installed at the end of the oil separator vessel to the beginning when the feed begins to enter the separator vessel, this is so that if the pH does not meet the standard, then adjustments can be made immediately so that the glycerin water that enters the oil separator can be immediately repaired pH. With the current pH position of the transmitter, when the pH reading is not standard, it is too late to anticipate, the oil separator has been filled with a non-standard pH.

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