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Increased Concentration of Bioethanol by Rectification Distillation Sieve Tray Type

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***Abstract*—** Bioethanol is one of the alternative energy which can be used to replace fossil fuels. Bioethanol used are typically high alcohol concentration. In the process of making bioethanol is usually produced with low alcohol content, hence the need for a purification process so that the consentrations are increased. One of bioethanol purification process is rectification distillation. This study aimed to test the rectification distillation apparatus sieve tray type and verify whether the alcohol content measurement using a pycnometer according to alcoholmeter. The results showed that the apparatus can be used to increase the ethanol concentration of 38% to 53%. This research can be demonstrated that the measurement of the concentration of ethanol by pycnometer method not much different from using alcoholmeter.

Keywords— bioethanol, concentration, distillation.

INTRODUCTION

Bioethanol should be developed as an alternative energy to replace fossil fuels. Ethanol is a fuel that is converted from sugar , starch and lignocellulose. The use of bioethanol as a gasoline blend to be used as a motor vehicle fuel will reduce emissions. In this case of bioethanol as a substitute for MTBE (methyl tertiary butyl ether), that is both toxic and contaminate the groundwater [1]. Bioethanol is good for the environment because it is derived from plants, it has improved Lifecycle CO_2 performance because the plants use as feedstock take CO_2 from the atmosphere as they grown. This mean that almost all the CO_2 produced by burning the fuel is balanced by CO_2 taken from the air in the first place so it can reduced CO_2 emissions [2].

The process of making bioethanol generally through three stages namely saccharification, fermentation and purification fermented. Bioethanol fermentation usually results in low levels of ethanol as has been done by [3] ie bioethanol fermentation by Saccharomyces cerevisiae on cassava hydrolyzed acid yield 7.18% (v/v) and cassava were hydrolyzed enzymatically by 9.59 % (v/v). Theoretically ethanol fermentation by Saccharomyces cerevisiae produce ethanol 12-18 % (v/v) as in [4].

At this stage of purification of bioethanol is usually carried out by distillation. Distillation is a way to separate the components of the solution or mixture is a liquid or gas by basing on the difference in boiling point components contained in it [5]. The main requirement in the operation of the separation of components by distillation is the composition of the vapor must be different from the liquid composition to be a balance solutions, with enough components can evaporate. Liquid boiling temperature is the boiling point of the liquid at atmospheric pressure are used [6].

Modern distillation systems are multi-stage, continuous, countercurrent, vapor-liquid contacting systems that operate within the physical laws that state that different materials boil at different temperatures. Trays are the most common contactor in use. The primary functions desired of tray contactor in a distillation tower are mixing rising vapor with a falling fluid, allow for separation after mixing, provide path for liquid to proceed down the tower and provide path for vapor to proceed up the tower [7].

The measurement of alcohol content is an important quality control aspect in the final steps of bioethanol production process. The practice currently in use by industry involves measurements taken from a high-quality hydrometer, and correcting for the temperature of the sample. A hydrometer is a measurement tool for determining the specific gravity of a liquid known as alcoholmeter. More rigorous and high-cost methods exist, including the use of a digital density meter, and gas chromatography. A gas chromatograph is very accurate in determining the concentration of hydrocarbons and was therefore one of the methods that was studied for determining the amount of ethanol in a sample of beer [8]. According Pardosi [9] measurement of the ethanol content using gas chromatrography and density methods provide almost the same results were not significantly different meaning. In this study conducted testing tools sieve tray type distillation rectification and comparison of results from measurements of bioethanol using a hydrometer and pycnometer.

Distillation is the purification of gases or liquids by taking advantage of their boiling point differences. Ethanol and water have a fairly large difference in boiling point, but only up to a certain concentration. At 1 atmosphere and about 95 volume % ethanol, the boiling point of this mixture has a boiling point less than either of the pure components and is known to be a minimum boiling azeotrope [13].

METHOD

Distillation equipment tested in this study is a distillation apparatus rectification sieve tray type with a maximum capacity of 3 liters, made of stainless stell. Made distillation apparatus consists of the main parts of the steam boiler, under the column (bottom), the top column / distillation column, and a condenser. The distillation column 1 meter long with a disc containing stainless steel without holes are sliced one of its edges. The discs are arranged alternately alternate slices intended to serve as a vapor way up or liquids down. Figure 1 shows this distillation system.

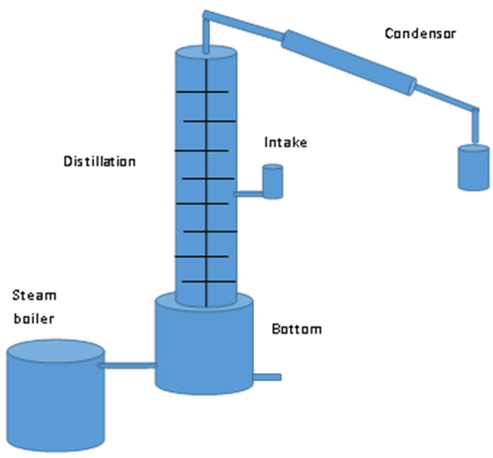

Fig 1. Distillation equipment system

Distillation system would contain the following elements; a feed composed of the two component to be separated, a source of energy to drive the process (steam), purified product consisting primarily of the feed component with lower boiling point, a bottoms product containing the component of the feed posessing the higher boiling point and an overhead heat exchanger (condenser), normally water-cooled [7].

The following testing procedures distillation apparatus that is connected to the electric current so that the temperature on the boiler to rise and reach the boiling point of water so the steam flows to the distillation column (bottom and top). Bioethanol with low concentration inserted into the intake so that the ethanol flows down to the bottom of the column. In the distillation

process takes place on record the temperature of boiler, bottom, the distillation column, water enters the condenser and condenser water out. After the distillation process, measured concentration of ethanol in purified product and bottom product by hydrometer that call alcoholmeter and picnometer.

Ethanol Concentration Measurement

The amount of alcohol present in a solution can be determined by measuring the specific gravity of the liquid and relating the gravity to standard tables of conversion of specific gravity to alcohol by weight in water-alcohol mixtures. One simple but very accurate way to do this is through the use of a specific volumetric container called the pycnometer and the use of a multi-place analytical balance. The pycnometer is a small flask with a very narrow opening at the top, which is drilled to accept a snugly fitting ground glass stopper. After the flask is filled and the top fitted on (excess liquid being displaced by the stopper), the flask is wiped dry and is ready to be weighed. Because the top opening is so narrow, the volume contained in the flask is always exactly the same within a very small error limit. Specific gravity is the density of an unknown liquid compared to (divided by) the density of water. Density is weight/volume. Because the volume in the pycnometer is always almost exactly the same, the specific gravity of a liquid is equal to the weight of the liquid, which fits in the pycnometer divided, by the weight of water, which fits in the pycnometer. As density is related to temperature, the pycnometer and contents need to be equilibrated to a set temperature (usually exactly 20 ^{0}C), and must be dried quickly and thoroughly before weighing [10]

$$SG = \frac{Weigh\ \ of\ bioethanol\ at\ 20\ oC\ in\ a\ certain\ volume}{Weigh\ \ of\ pure\ water\ at\ 20\ oC\ in\ the\ same\ volume}$$

The amount of alcohol present determined by Table apparent relative density of aqueous ethanol [11]

Specific gravity is also commonly measured by the use of a hydrometer. A hydrometer is a measurement tool for determining the specific gravity of a liquid. Typically, a hydrometer is made of glass and has a weighted bulb on the bottom filed with lead so that the tool will float in a liquid. The hydrometer has measurement increments on the upper half for taking readings when the hydrometer reaches its equilibrium point in the liquid. When the hydrometer is floating properly, the bottom of the meniscus will read the correct specific gravity value of the liquid trying to be measured [8].

The heavier the liquid, in which the hydrometer is placed, the higher it will float. A scale placed on the narrow top of the hydrometer (the stem) can be calibrated in terms of specific gravity or concentration directly. The weight of liquid in the meniscus formed by the liquid around the stem adds to the weight of the hydrometer itself in forcing the hydrometer into the liquid. For best accuracy, a hydrometer should be used in the manner in which it was designed to be used. Because of the surface effects on the meniscus the hydrometer must be very clean. The hydrometer should be used on a "fresh surface". The surface is provided by filling the hydrometer cylinder completely to the point where it overflows the top. A hydrometer placed into the liquid to be tested slowly and spin it slowly to keep it from touching the sides of the container. As the hydrometer settles to a steady level, refresh the surface with a little more of the test liquid. Wait about 30 seconds and read the level at the very top of the meniscus. A dirty hydrometer placed in a liquid with an improperly formed meniscus can easily be off by 0.2.or more [10] This specific gravity can also be known as relative density, because the density of the measured liquid is determined by its density compared to the density of water [8].

		Ethanol concentration (%)	
Ethanol position	Volume (ml)	hydrometer	pycnometer
Intake	1000	37	36,49
Distillate	186	30	29,29
Bottom	886	10	10,54

RESULT AND DISCUSSION

a. Measurements of Bioethanol with Alcoholmeter and Pycnometer

When samples of bioethanol obtained from the distillation process a bit and does not allow it to be measured with alcoholmeter or hydrometer, often alcohol content measurement was conducted by using a pycnometer. Research at this stage to determine whether alcohol content measurement method using pycnometer measurements can represent the level of alcohol with alcoholmeter. Bioethanol grading 90% diluted so that the levels are lower and divided into several samples. The samples were then measured alcohol content with alcoholmeter and pycnometer and the results as listed in Table 1 below.

Table 1. Results of measurements of bioethanol with alcoholmeter and pycnometer

Sample	Alcoholmeter (% alcohol)	Pycnometer (% alcohol)
1	99	100
2	84	85
3	87	89
4	89	90
5	91	92
6	94	95
7	83	84
8	81	82
9	78	80
10	76	77
11	73	75
12	70	71
13	67	69
14	64	66
15	62	64

The measurement results were analyzed by statistical analysis of comparative two independent samples for ordinal data using the median test and found that the combination of the data median of 81.5. At the 5% significance level and degrees of freedom 1 obtained $\chi^2_{0,05} = 3{,}481$ and the value $\chi^2_0 = 0$. Based on these values it can be concluded that there is no significant difference between the measured concentrations of alcohol with Alcoholmeter and with pycnometer. It shows that the use of pycnometer in measured concentrations of bioethanol can be used to determine how much of alcohol content if the sample being measured slightly. These results are in accordance with that done by Tekaligne, *et all* [12] that the difference of alcoholic content measured using Hydrometer and Pycnometer are not much significant, ie the lowest concentration of bioethanol production (4.67 and 4.84 %) through Hydrometer and Pycnometer measurements were obtain at fermentation time.

b. Bioethanol Distillation Process

The purification process is done using a tool bioethanol distillation sieve tray-type rectification twice experiments ie by adding heating elements in the column above (distillation column) and without additional elements.

Table 2. Concentration of ethanol before and after distillation with the heating element

Temperature distillation process observed in steam (T steam), bottom, (T bottom), the top column/distillation column (T tower), the condenser water sign (T Con Inp)

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and water condenser exit (T Con Out) in order to obtain the data as follows.

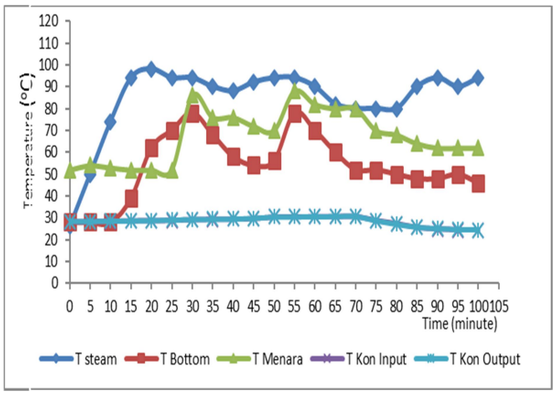

Fig 2. Graph of the temperature distribution in the distillation process with heating element

Table 3. Consentration of ethanol before and after the distillation process without heating element

		Ethanol concentration (%)	
Ethanol position	Volume (ml)	hydrometer	Pycnometer
Intake	916	38	37,66
Distillate	26		52,95
Bottom	932	22	22,92

As in the first test, the temperature distribution in the distillation process was also observed in steam, bottom, towers and condensers. Below is the distribution of distillation temperature without the use of the heating element.

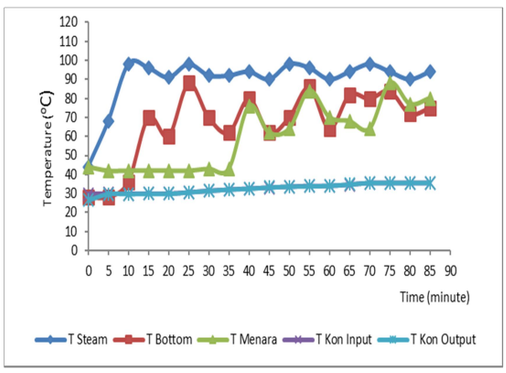

Fig 3. Graph of the temperature distribution in the distillation process without heating element

The distillation process begins with filling the water in the steam / boiler 3000 ml, as well as replenishing water in the tub as much as 11000 ml condenser, then connect the electric current appliance and boiler temperature set on the number of 100°C. The temperature regulation aims to boil water in the boiler so that the steam can enter the space under the column (bottom). Ethanol with low levels (37-38%) is inserted into the tube insertion material (intake) but the faucet is still closed. After the bottom temperature reached about 80 °C in the fill faucet is opened.

In accordance with Table 2 and Table 3 on the condition of ethanol in the distillation process can be seen that the measured levels of ethanol by the method pycnometer and use Alcoholmeter not much different, so if distillate obtained many do not enough for the measurement with Alcoholmeter, pycnometer methods can be used or can represent the data required.

The concentrations of ethanol after distillation process by heating obtained is lower than the initial ethanol content ie the levels of 37% to 30%, with total distillate volume of 186 ml and 886 ml volume bottom. The total volume of distillate and bottom as much as 1072 ml resulting in a volume increase as much as 72 ml of the input material. In the distillation process without using the heating element, can increase ethanol content material that is from 38% to 53% by volume of distillate by 26 ml and 932 ml volume bottom. The total volume of distillate and bottom in this process as much as 958 ml resulting in decrease by 42 ml from volume input. The ethanol yield in the process is strongly influenced by temperature distribution distillation apparatus.

Based on Figure 2 and Figure 3 it can be seen that there is a fairly sharp fluctuations in steam temperature, distillation column temperature and bottom temperature. These fluctuations are caused by temperature settings manually on steam heating element, resulting in a temperature difference large enough. Therefore replacement of heater temperature control from manual to digital necessary in order to set the temperature difference is not too much so that the temperature can survive at a certain point.

An overhead heat exchanger (condenser), normally water-cooled, to condense the vapor resulting from the boiling created by energy input. The overhead vapor, after condensation, is split into two streams. One stream is the overhead product, the other is the reflux which is returned to the top of the tower to supply the liquid downflow required in the upper portion of the tower [7].Difference in incoming water temperature and water out on the condenser is not far adrift from only about 0.1-0.3 even at a certain time did not differ between the temperature of incoming water and condenser water out. It shows that the exchange of heat transfer occurs is sufficient to change the vapor into a liquid.

In accordance with the data obtained can be explained that the addition of heating elements on the top of the column/tower does not need to be done. When the temperature reached 78°C tower means the ethanol has evaporated, and the expected water vapor will fall back toward the column down/bottom, so that in the container distillate contain high levels of ethanol. Refer to [7] analysis of the ethanol-water distillation system is mathematically straightforward when using molar quantities rather than the more common measurements of volume or weight. This is because of an energy balance principle called 'constant molal overflow'. Essentially, this principle states that the heat (energy) required to vaporize or condense a mole of ethanol is approximately equal to the heat (energy) required to vaporize or condense a mole water, and is approximately equal to the heat (energy) required to vaporize or condense any mixture of the two.

CONCLUSSION

The research showed that the alcohol content measurement method using pycnometer density results do not differ significantly from the measurement of the alcohol content using hydrometer or alcoholmeter The concentrationl of alcohol in ethanol may increase after purified by distillation rectification equipment type sieve tray without the heating element from 38% to 53% but these tools need to be studied further.

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