

DESIGN AND CONSTRUCTION OF A REFLUX COLUMN DISTILLATION UNIT FOR BIO-ETHANOL PRODUCTION FROM SUGARCANE SUBSTRATE

J. O. OLAOYE

Agricultural & Biosystems Engineering Department, University of Ilorin, P. M. B.
1515, Ilorin, 240003, Nigeria; E-mail: jolanoye@unilorin.edu.ng

ABSTRACT

A bio-ethanol distilling tank was designed and constructed to distil ethanol from sugarcane substrate. The machine has a capacity to process 200 litres of substrate at full load of the boiler. The distiller has an *Internal Reflux Still Condenser (IRSC)* that controls the internal re-distillation process and the separation of the final output. The column diameter was 40 mm. An anaerobic fermentation of substrate was adopted before distillation could be carried out. The fermented substrate was adjusted to an optimum pH level value of 4-5 by addition of 0.1 M H₂SO₄ and the optimum temperature was within the temperature range of 29-38°C. A charcoal pot was used as heat source. The results of the machine evaluation showed that optimum yield occurred at 0.0325 ratio of substrate to ethanol yield. An average distilled product of 2.1 litres was obtained at highest ratio of substrate to ethanol yield of 0.033 when the distillation time was 45 minutes. The total distilled products after 1½ hours of distillation was estimated at 4.25 litres. It was observed that the fermentation and distillation processes were done *in situ*, and could definitely affect clear separation of the fermentable portion of the fermented sugar solution.

Key words: Biomass, Biofuel, Ethanol, Distillation, Energy Crop, Alternative Energy

1.0 INTRODUCTION

Biofuels are renewable energy sources that are derived from living plant materials. They are extracted from vegetable or animal matter (biomass) and may be in liquid or gaseous form. Biofuel has potentials to be used as a substitute for fossil fuels like petrol or diesel. Biofuels are produced through simple scientific process of production without serious need for sophisticated machines and equipment as required in the case of oil and gas production. In bioethanol production microorganisms are responsible for the natural digestion of complex plant materials to produce ethanol in the presence of oxygen.

Ethanol is made by the fermentation of sugars. Sugarcane or starch from potatoes, corn, or other cereals can be the raw material. The yeast enzyme changes the simple sugars into ethanol and carbon dioxide. The fermentation reaction represented by the simple equation shown below is actually very complex because of other substances, including fusil oil glycerin, and various organic acids that are produced in unwanted side reaction.



The fermented liquid, containing from 7 to 12 percent ethanol, is concentrated to 95 percent by a series of distillations. When ethanol is mixed with gasoline for use as automobile fuel, it is generally referred to as gasohol or biofuel (Rosentrater, 2006). According to Henniges and Zeddies (2006), most of the results of research output on bioethanol development are directed towards the pretreatment of the energy crop. The process is to improve the rate of conversion, yield and efficiency for more economical ethanol production from the crop energy. These involve various technologies to advance pretreatment activities prior the process of fermentation.

1.1 Biofuels as Sustainable Source of Energy for the Future

Production of biofuels has the beneficial effect in increasing a sustainable fuel supply for the future. Biofuel does not cause as much air pollution and environmental hazards as petroleum fuels. It does not contribute to carbon dioxide greenhouse effect problem such as the global warming (Keney) and DeLuca, 1992). Ethanol is an octane booster and anti-knocking agent. The activities through the production chains of biofuels provide jobs and socio-economic developments in rural areas. The use of ethanol as fuel is capable of reducing the adverse foreign trade balance. The cost benefit ratio of production of biofuels may be higher compare to fossil fuel but biofuel does not contribute to greenhouse effect problem which is a major problem with other known energy source and this projects acceptance of biofuels compared to other known energy source (COLMAC, 2009; Van Gerpen et al, 2007).. The traditional method in feedstock distillation involves application of condenser that consists of coil of pipes or insertion of pipes in a fin jacket. The result is usually characterized by the mixture of products distillation and water.

The main objective of this study is to design and construct a bioethanol distillation unit with a reflux column for production of ethanol from fermented substrate using sugarcane products as the biomass feedstock.

2.0 MACHINE DESCRIPTION

A bioethanol distillation tank was designed and constructed (Figure 1). The machine has a capability to process 200 litres of substrate. The machine was designed to distil materials fermented products such as sucrose from sugarcane juice and sugarcane products or by - products. The *Internal Reflux Still Condenser (IRSC)* is a device that controls the internal re-distillation process and the separation of the final output. The column of the IRSC was packed with reflux of a uniform surface area and the column was sparsely packed or jammed. The condenser uses a surface sump pump. The capacity of the pump is 1.5 hp and it has potential to circulate water within the flow range of 750-1200 l/hr.

2.1 Design Assumptions and Calculations

A cylindrical tank was considered as the boiler or distilling tank and the capacity of the distilling tank of about 200 litres to process 0.2 m³ of substrate. The gravimetric volume and volumetric volume of the boiler were determined by the expression presented in Equations 1 and 2. The maximum height of the tank was chosen as 0.9 m.

$$G_v = V_b \rho_b \tag{1}$$

where,

G_v = Gravimetric Capacity
 V_b = Volumetric Capacity
 ρ_b = Nominal density of the product

$$V_b = \pi r^2 h \tag{2}$$

where,

V_b = volume of a cylinder (m^3)
 r = radius of the boiler (m)
 h = height of the boiler (0.9 m)

The thermal and engineering properties of the substrate, boiling medium, heat source and substrates were established from various sources as follows: the specific heat capacity of ethanol is 2.44 J/g/K at density of 790 kg/m³, the latent heat of vaporization is 38.56 kJ/mol and the boiling temperature is 78.5 °C (BIODIESEL, 2009)., the system will be powered by charcoal, of density, 208 kg/m³ (SIMETRIC & Co, 2009)., the calorific value of charcoal is 30,970 kJ/kg (WORLDENERGY, (2008).

The rate of flow of water into the cooling jacket is preferred to be slower than the flow rate of flow of ethanol vapour into the reflux column for effective distillation. On the principles of continuity of fluid flow, ethanol flows at volumetric flow rate of 0.003 m³/s, as such a much slower speed of 0.001 m³/s is selected for the pump for effective cooling. The quantity or volume of water needed to cool ethanol vapour in the condenser determines the volume of the water tank. The volume of water was calculated by equating the quantity of heat energy lost by ethanol vapour to the heat energy gained by water, (Equation 3). Volume of water needed at the maximum operating capacity was 273.9 litres and a water reservoir of 300 litres was selected (BIODISEL, 2009).

$$M_w C_w (\dot{E}_a - \dot{E}_{25}) = M_{eth} \times L_{eth} \tag{3}$$

Where

M_w = mass of water (kg)
 C_w = specific heat capacity of water (4.186 J/g °C)
 \dot{E}_a = the highest desired temperature water should get to (40°C)
 \dot{E}_{25} = room temperature of water (25°C)
 M_{eth} = mass of ethanol (kg)
 L_{eth} = latent heat of fusion of ethanol (108.99 J/g)

2.2 Heat Source and Heat Energy Requirement for Distillation Process

The required heat energy to raise the temperature of the still from 38.5 °C to 78.5 °C, the boiling temperature of ethanol was estimated from Equation 4 and the quantity of heat energy, Q to change ethanol to vapour at boiling phase was determined using Equation 5.

$$Q = MC\Delta\Theta \quad (4)$$

$$Q = ML \quad (5)$$

Where

- M = Mass of fermented feedstock (kg)
- C = Specific heat capacity of fermented stock (J/g°C)
- $\Delta\Theta$ = Change in temperature (°C)
- L = the latent heat of vaporization (J/g).

The required heat energy to raise the temperature to a desired temperature, 78 °C is 14824 kJ, 132k J is the quantity of heat energy to change ethanol to vapour and the total quantity of energy required to vaporize ethanol is 14956.0 kJ/S. The quantity of charcoal (mass) needed to supply the quantity of heat energy needed to vaporize ethanol was calculated as 30.970 kJ / kg. Therefore, the required quantity of charcoal was given as 0.4829 kg / s

2.3 Rate of Discharge and Pump Power

The ethanol vapour rises by 0.003 m³ in the reflux column every second. The Heat supplied, Q excites the particles at corresponding kinetic energy level. The velocity at which ethanol vapour rises in the reflux column was presented by Equation 6 and the diameter of the reflux column was determined using Equation 7.

$$v = \sqrt{\frac{2 C \Delta\theta A^2 l^2}{L}} \quad (6)$$

$$d = \sqrt{\frac{4 v^2}{A^2 \pi}} \quad (7)$$

where:

- v = Volumetric flow rate of the Vapour (m³ s⁻²)
- A² = rate of flow per cross sectional area (m² s⁻²)
- C = Specific heat capacity of ethanol (J/g °C)
- l = Column length (m)
- d = diameter of the column (m)
- L = the latent heat of vaporization / fusion (J/g)

From Equations 6 and 7 the volumetric flow rate at which ethanol vapour rises up the reflux column and the column diameter were calculated as 0.0038 m³ s⁻² and a 40 mm (1.5"), respectively. Copper pipe was selected because of its inert property which can limit reaction with vaporised ethanol.

The reflux column has a water jacket surrounding it to influence the cooling action of the vaporized ethanol. An alternating current pump was installed to circulate water through the condensing unit. The pump capacity, p was determined based on the volume of water to be circulated from the retention reservoir to the condenser, total head of circulation and the demand flow rate. Eqn. 8 was applied according to Khurmi (2005) and 1 hP pump was recommended.

$$p = \bar{n} g q H \quad (8)$$

where

- \bar{n} = fluid density, (for water, $\bar{n} = 1000 \text{ kg m}^{-3}$)
- q = flow rate (m^3/s)
- H = $h + h_f$
= height of the Column above pump level + frictional loss

The flow analysis through the reflux column and the condenser unit was estimated. The volumetric flow rate of discharge was estimated as $2.75 \times 10^{-3} \text{ m}^3 \text{ s}^{-1}$ when a condenser of 0.019 m diameter was used. This corresponds to the half of the reflux column diameter. Therefore, the biofuel distiller has capacity to distil $5.65 \times 10^{-2} \text{ m}^3$ of ethanol in 1 hr at a maximum loading capacity of the biostill tank for substrate to bioethanol yield of 1:1 or at a higher ratio.

The orthographic projection of the distiller is presented in Fig. 1 showing the front and end views of the machine. The boiler tank was first constructed before the pipe setting and assemblage. Appropriate selection of the best materials was ensured to enable the best quality of the reflux distillation tank and ethanol quality, and to meet design standards. The detailed drawing of the machine is presented in Figure 1.

2.4 Manufacturing Process

The construction of the machine was carried out at the fabrication workshop of the Agricultural and Biosystems Engineering Department, University of Ilorin, Ilorin, Nigeria. The fabrication techniques involve cutting of galvanised metal sheets into desired sizes, and welding of the same and other joints ensuring toughness and strength putting prospective usage in mind. The parts were welded perfectly with firm joints to avoid vapour loss. The boiler tank was first constructed before the pipe setting and assemblage. Appropriate selection of the best materials was ensured to enable the best quality of the reflux distillation tank and ethanol quality, and to meet design standards. The exploded view is presented in Figure 1 and the display of machine setup was shown in Figure 2.

3.0 MATERIALS AND METHODS

3.1 Required Materials for Bioethanol Distillation

The materials that were used for bioethanol distillation procedure include fermented feedstock (sugar solution), 0.1 M H_2SO_4 solution to adjust the pH level of substrate, pure tepid water to inoculate the yeast, yeast as a catalyst to enhance fermentation process, heat source and condensation device. The pH was measured using a Lab meter (Model PHM 92).



Figure 2: Machine Setup during Distillation Process

3.2 Method of Substrate preparation for Distilling Bioethanol

An anaerobic fermentation of substrate was carried out at the processing Laboratory, Agricultural and Biosystems Engineering Department, University of Ilorin, Ilorin where the fermentation and distillation conditions were monitored. The anaerobic fermentation is slower than aerobic fermentation but the anaerobic fermentation guarantees more alcohol and CO₂ yield. The object of distillation is to remove ethanol from the fermented sugar solution. The substrate was loaded into the fermenting tank for yeast growth. The distilling tank was designed to handle 200 litres of substrate, the tank was charged to about ¼ of its full capacity and the distillation process was monitored after fermentation.

The temperature of the fermentation tank and its content was adjusted and maintained at 28-30 °C and the acidity level of the content was monitored to reach pH of about 4-5 for optimum salinity for yeast growth. The yeast was introduced as catalyst to enhance the conversion of sugar into alcohol and CO₂. The substrate was allowed to ferment for about 48 and 72 hours. The fermentation and distillation processes were conducted in situ. A heat source of charcoal was introduced as shown in Figure 2 for the distillation.

Temperature, pH, pressure built up within the distillation boiler and volume of ethanol yield were monitored over time. The procedure was replicated twice.

3.3 Determination of Ratio of Total Substrate to Volume of Ethanol Yield

The level of sugar formation for conversion of starch to ethanol was determined at fermentation stage by the rate at which the yeast enzyme converts the sugar to sucrose or fructose and releases CO₂. This process establishes the fermented state of the substrate and represents the total distillate or ethanol yield. The fermented substrate was adjusted to an optimum pH level value of 4-5 by addition of 0.1 M H₂SO₄, and the optimum temperature was confirmed at 29-38 °C when the mash ceases bubbling and the yeast cake, which forms on top, sinks to the bottom. Substrate concentration plays dominant role in the yield of ethanol during distillation procedure. The volume of substrate was measured at 30 minutes interval starting from the time the temperature of the distiller attains 78.5 °C and the ratio of the volume of ethanol yield to the total substrate in the boiler was evaluated and recorded. A graduated measuring cylinder was used to collect drops of ethanol yield and the volume of ethanol collected at 30 minutes interval was determined and recorded. The total volume of distillate was read on a graduated scale on the biofuel distillation tank.

4.0 RESULT AND DISCUSSION

The temperature variation of the prepared substrate was monitored during fermentation process. Table 1 shows the changes in temperature in the fermentation tank against time taken during fermentation process. It takes about 3 days before the fermentation process was completed. The temperature of the fermentation tank attained a steady state of average temperature of 38.5 °C. The initial steady rise in substrate temperature was due to the action of Yeast enzyme on the substrate to release CO₂ under anaerobic condition. The steady state of the substrate temperature at 38.5 °C indicated that the product is ready for distillation especially when it was observed that the pH values were maintained consistently at the optimum and starting values during the fermentation procedure.

4.1 Temperature Variation during Distillation before Ethanol Yield

Table 2 shows changes in temperature against time during the distillation process. It was observed that it takes about 3 hours to boil the substrates to the boiling point of ethanol when charcoal pot was used as heat source. Mathewson (1980) noted that when the mixture of water and alcohol is boiled, vapours with a greater concentration of alcohol will be formed and liquid with a lesser concentration of alcohol will remain behind. The separation of ethanol and water is initiated at this stage. Water and alcohol do not form an "ideal" mixture, the separation cannot be done in one clean step. A reflux column created the distillation and re-distillation of the mixture. The observed boiling point composition for liquid and vapour phases could be compared to Matherson (1980). The upper limit of temperature variation indicates the condition at which the reflux ratio can be determined. The boiling of the substrate takes about 3 hours to attain boiling point of ethanol

4.2 Effects of the Ratio of Substrate to the Volume of Ethanol Yield and Distillation Rate

Rate of distillation of ethanol is influenced by the ratio of substrate to the ethanol content of the substrate. Distillation process is predicated on the principle of conservation of matter. Distillation of ethanol evolves through liquid / vapour phase and since matter can neither be created nor destroyed the volume of ethanol yield per unit time must be evaluated from the overall substrate through evaluation of the ratio of substrate to distillate or ethanol yield. Table 3 presents the relationship between the volume of fermented substrate and the total ethanol yield per unit time. Table 3 indicated that optimum yield is observed at higher ratio of substrate to ethanol yield. An average distilled product of 1.6 l was obtained at highest ratio of substrate to ethanol yield of 0.033 and when the distillation time was 45 minutes (Figure 3). The total distilled products after 1½ hours of distillation was estimated at 4.25 litres. It was observed that the fermentation and distillation processes were done *in situ*, this would definitely affect clear separation of the fermentable portion of the fermented sugar solution from slurry and invariably affect the final yield (Rosentrater, 2006). The test conducted was limited to ¼ capacity of the biostill and this was lower than the loading capacity recommended by Bolling and Suarez (2001). Bolling and Suarez (2001) reported that in operation, the boiler or still pot should be filled to about ¾ level capacity. It was observed that if the tank is not filled to the optimum capacity, vapour rising through the column will loose energy and cool down before it reaches the end of the column through to the condenser, as such the yield will be low. The highest distillation rate was attained at the time corresponding to highest ratio of substrate to ethanol yield as clearly indicated by Figure 4. The trend of the distillation rate curve follows the pattern presented by the substrate to ethanol yield curve as shown in Figures 3 and 4. This observation showed clearly that constant and sufficient heat source was used for the distillation process and this indicated that regression in the distillation process was avoided.

The progressive level of ethanol yield with time to the observed maximum yield prior the eventual gradual decrease to no yield as presented in Table 3 and the trend of distillation rate as shown in Figure 4 affirmed that ethanol yield was well regulated by the reflux column and the reflux material packed in the column.

The properties of the distilled product were not examined. It was however noted that the distilled ethanol may not be pure as it may still contains some water. Anhydrous ethanol is required and water has to be removed from the distilled product. This is part of the treatment to be investigated in future.

Table1 Temperature variation of the Substrate against Time of Fermentation

Time of Fermentation (hrs)	Temperature (°C)		pH Value	
	Replicate 1	Replicate 2	Replicate 1	Replicate 2
00.00	27.5	28.0	4.2	4.5
12.00	30.5	29.5	4.2	4.5
24.00	34.5	33.0	4.2	4.5
36.00	36.0	35.5	4.2	4.5
48.00	37.5	37.0	4.2	4.5
60.00	38.5	38.5	4.2	4.5
72.00	38.5	38.5	4.2	4.5

Table 2. Observation of the changes in temperature against time during the distillation process

Time of Distillation (minutes)	Temperature (°C)	
	Replicate 1	Replicate 2
00.0	38.5	38.5
20.0	42.5	41.5
40.0	45.5	43.0
60.0	50.0	55.5
80.0	55.0	59.0
100.0	60.5	61.5
120.0	65.0	64.5
140.0	76.0	75.5
160.0	80.0	80.0
180.0	81.5	82.0

Table 3. Ratio of Substrate with Time and Total Ethanol Yield

Time of Distillation (minutes)	Ratio of Substrate Distillate to Ethanol Yield		Volume of Ethanol Yield (L)	
	Replicate 1	Replicate 2	Replicate 1	Replicate 2
00.0	0.000	0.000	0	0
15.0	0.007	0.006	0.35	0.3
30.0	0.017	0.018	0.85	0.9
45.0	0.033	0.035	1.55	1.65
60.0	0.022	0.024	0.95	1
75.0	0.015	0.012	0.55	0.45
90.0	0.000	0.000	0	0
Total Volume of Ethanol Yield (L)			4.25	4.30

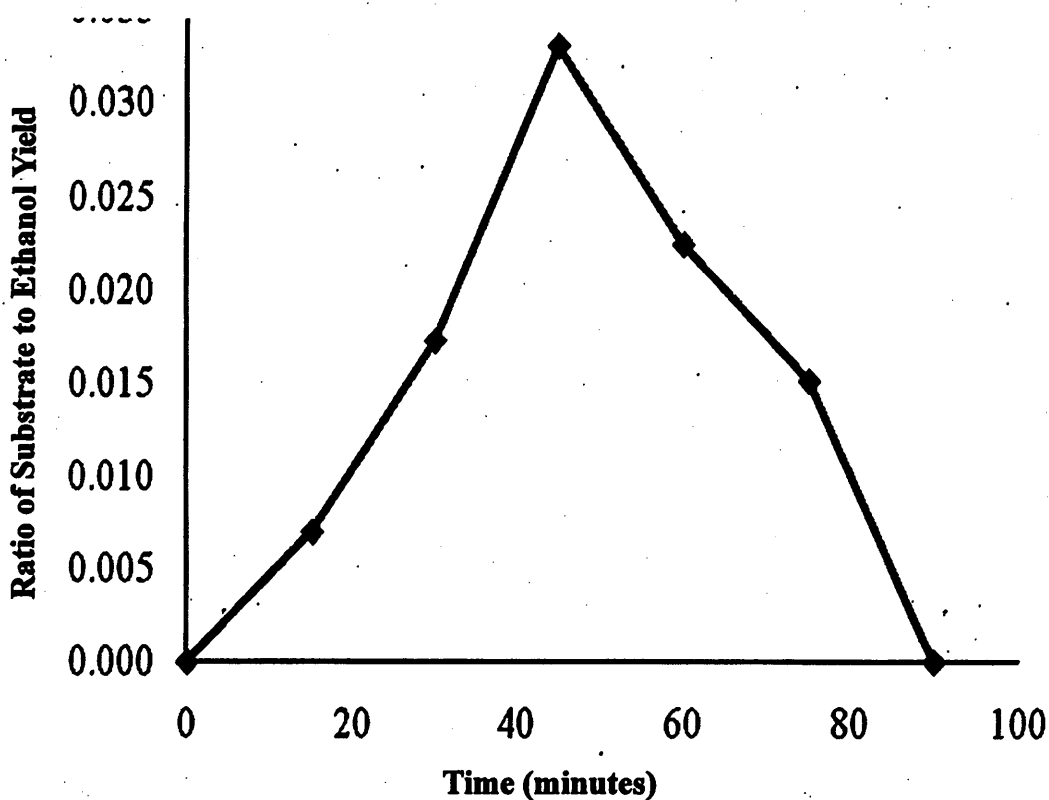


Figure 3: Ratio of Substrate to Etahanol Yield against Time of Distillation

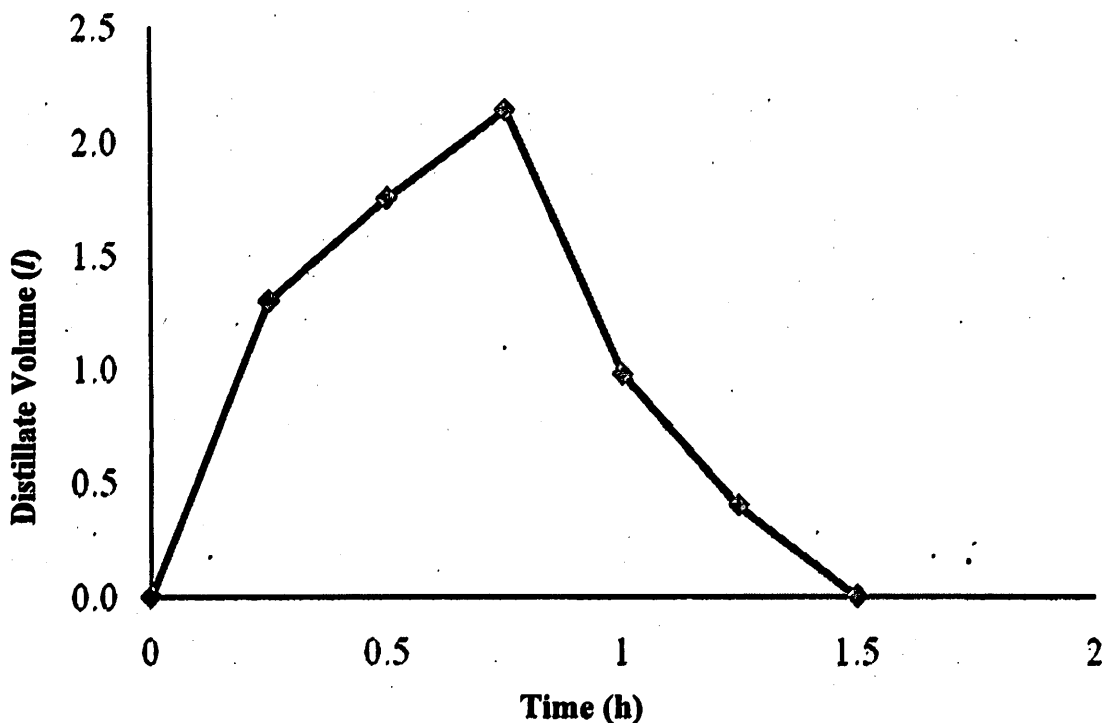


Figure 4: Rate of Ethanol Distillation at various Time Interval

5.0 CONCLUSIONS

A bioethanol distillation unit was developed to distil ethanol from sugarcane substrate. The machine has capacity at maximum loading state to distil 200 litres of substrate. The distiller has an *Internal Reflux Still Condenser (IRSC)* that controls the internal re-distillation process and the separation of the final output. The boiling of the substrate takes about 3 hours to boil to the boiling point of ethanol when charcoal pot was used as heat source. The rate of distillation of ethanol was influenced by the ratio of substrate to the ethanol content of the substrate. An average distilled product of 1.6 l was obtained at highest ratio of distillate to ethanol yield of 0.033 when the distillation time was 45 minutes. The total distilled products after 1½ hours of distillation was estimated at 4.25 litres.

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