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The Effect of Column Height, Packing Materials and Azeotrope Separation on the Purity of Ethanol Sourced from Ogogoro (Local Gin)

[1]V.E. Efeovbokhan, [2]J.D. Udonne, [1]D.E. Babatunde and [1]S.E. Sanni
[1]Department of Chemical Engineering, Covenant University, Ota, Nigeria
[2]Department of Chemical Engineering, Lagos State University, Epe, Nigeria

Abstract: Locally brewed Ogogoro, a mixture of ethanol and water in the ratio of 28:72 by volume was separated into its constituents by means of fractional distillation. The mixture of 95.5% ethanol and 4.5% water (percentage by volume) formed an azeotrope with a boiling point of 78.2°C and thus could not be further purified by distillation. The ethanol/water mixture after distilling to the azeotrope was shaken with calcium oxide to form the non-volatile compound, calcium hydroxide which altered the stability of the azeotrope and hence further separation of the constituents of the mixture. Nearly all of the calcium hydroxide was removed by filtration and the filtrate was redistilled to obtain higher puzrity ethanol relative to the azeotrope. The percentage purities of the ethanol were 96.5, 97% with the highest purity being 97.5%. The mass of calcium oxide used was varied at a constant time of 1 h and the effect on the percentage of ethanol achieved were recorded as 95.5, 97.7, 97, 96.5 and 96.5% from the initial percentages 95, 95, 95.5, 95.5 and 95.5% ethanol, respectively. By comparing masses, 10 g of calcium oxide gave the highest percentage purity of ethanol after distillation which was 97.5%. Also, in order to determine the effect of time on product purity, the duration for drying the ethanol/water mixture with constant mass of 10 g calcium oxide was varied from 1-5 h. It was observed that increase in drying time for the ethanol/water mixture after 1 h did not lead to increase in the purity of ethanol distilled. The effects of increase in height of column as well as number of random packing was investigated and found to increase the efficiency of the distillation process. Using the double fractionating column of height 77 cm with 145 random glass packing improved the purity of the ethanol from 40-2% as compared with 86% which was the percentage purity obtained when a single fractionating column of height 46 cm with 92 random glass packing was used.

Key words: Ethanol, local gin, purity, fractional distillation, azeotrope

INTRODUCTION

Nigeria imports 90 million litres of industrial alcohol annually while 80 million litres of these is used by the liquor industry (Odejayi, 2005). Hence, there is need to encourage and redirect our local people who have been brewing alcohol for centuries. Though, their major aim has been for consumption purposes but government and other relevant agencies can embark on enlightenment campaigns amongst local manufacturers, so as to make the brewed alcohol available for bio-fuel (ethanol and biodiesel) production rather than being consumed (Efeovbokhan, 2013).

Interest in deriving energy and other value-added products from renewable bio-resources has been on the increase due to major factors such as their compatibility with the environment, relative ease of accessibility, non-renewability of fossil fuels and their diversified use for many value-added products.

Over the years, bioethanol has continuously gained attention as an excellent source of energy and other bio products making it possess an unlimited potential for economic growth. This is partly due to the versatility of ethanol and its variety of uses such as fuel, manufacture of beverages and useful chemicals, solvent for paints, varnishes, drugs, fluid in certain thermometers, preservation of biological specimens, etc. (Denise, 2010).

Palm wine may be distilled to produce a strong drink "ogogoro" or local gin (Kolawole *et al*., 2007; Idonije *et al*., 2012). Local gin (Ogogoro), a West African alcoholic beverage has been found to contain ethanol as its active component. Ogogoro is brewed by the distillation of the fermented sap of any of raffia palm (Raphia hookeri), coconut palm (Cocus nucifera) and oil palm (Elaeis guinesis). The saps are of low alcohol content when freshly tapped but with fermentation the alcohol content increases significantly (Isidore, 2001). These palms are widely distributed in West Africa and significantly, in the southern part of Nigeria with high density of raphia hookeri in the swampy coastal areas. The alcohol concentration of Ogogoro ranges between 30-60% depending on levels and degrees of fermentation, (Elijah *et al*., 2010).

Corresponding Author: V.E. Efeovbokhan, Department of Chemical Engineering, Covenant University, Ota, Nigeria

Leveraging on the abundance of these palms in Nigeria and West Africa as a whole, local large scale production of bioethanol from Ogogoro can be of valuable economic importance which serves as a means of boosting our local industries, making available an indigenous substitute for imported ethanol and enhancing foreign exchange via the exportation of the ethanol. Despite, the versatility of the use of bioethanol and its economic prospects, a key issue in its production is the manufacture of products with high level purity. Commercial ethanol contains 95% by volume of ethanol and 5% water (Davis, 2008). Research has shown that phase stability is a major problem in gasoline mixture when water is present; the presence of very small amounts of water can cause alcohol-gasoline mixtures to separate into gasoline and water-alcohol phases and these separate phases are vastly different in their combustion properties. Some of the aromatics from the base gasoline also separate with the alcohol-water, leaving the gasoline phase with very low octane number. The maximum purity of ethanol obtainable by distilling alcohol is around 96%. This is as a result of the azeotrope formed by the ethanol-water mixture. Fractional distillation can concentrate ethanol to 95.6% by weight (89.5 mole %). The mixture of 95.6% ethanol and 4.4% water (percentage by weight) is an azeotrope with a boiling point of 78.2°C (Rousseau and James, 1987) and cannot be further purified by distillation (Moore, 1962; Hilmen, 2000).

However, the separation of the ethanol-water azeotrope can be achieved by introducing a drying agent (such as CaO, $CaSO_4$, BaO, K_2CO_3, etc.) or an entrainer which influences the volatility of one of the constituents of the mixture or modifies the azeotropic composition and exhibits immiscibility with one of the components; extractive distillation may also be used (Perry and Green, 1984).

The purpose of this study is to demonstrate that purer samples with higher percentages of ethanol can be obtained through fractional distillation (Arthur and Brian, 1989; Kister and Henry, 1992) of local gin (Ogogoro). Ethanol as a vital industrial solvent has a high demand for its purer form for the production of many value added products, biofuels, inclusive cannot be over emphasized.

MATERIALS AND METHODS

Materials: Ogogoro, the local gin (28.08% purity) used for this research work was purchased from a local gin brewer in Ota, Ogun state and the calcium oxide of 99% purity is sigma aldrich analysed.

Apparatus: The apparatus used were liebig condensers (Pyrex), 500 mL flat bottom flask (Pyrex) which served as the distilling flask, 500 mL conical flask (Pyrex) as the distillate receiver, distilling head (Pyrex), 500 mL measuring cylinder (Pyrex), beakers (Pyrex), thermometer (i.e., calibrated 0-360°C), hot plate with magnetic stirrer, random glass chips, random copper chips, plastic beads, pycnometer (Specific gravity bottle), weighing balance (Pioneer, Ohaus), retort stands, rubber tubings and corks

Procedures

Variation of column height and type of packing: The 500 mL of the local gin (Ogogoro) was measured into a flat bottom flask which was connected to the distillation column, thermometer and magnetic stirrer via a cork. The flat bottom flask was placed on a hot plate which served as heat source for the distillation process. Cold water at a steady flow rate was turned on slowly and channeled to the inlet of the condenser of the distillation set up. The heat source was adjusted until the distillate came out at a rate of about one drop per second. The due point when the first drop of distillate was received and the temperature at which the distillation process stopped were noted.

Variation of number of packings using constant height: Distillation column of height 70.2 cm and random copper chips were used in investigating the effect of number of packings on the purity level of the Ogogoro distillates. The height of distillation column was kept constant, whereas the number of packings were varied for the different runs; 20, 40, 60, 80, 100, 120 and 140 pieces of random copper chips were loaded into the distillation column before the fractional distillation setup was erected and distillation performed. The experiment was repeated using packings such as glass and plastic chips.

Determination of concentrations (wt.%) of the ethanol: Specific gravity of un-distilled local gin (Ogogoro) and all the distillates obtained during the research work were determined at 30°C. An empty pycnometer was weighed and the mass was recorded as m_1. The pycnometer was then filled with the sample and weighed again, the mass obtained was recorded as m_2. Subsequently, the pycnometer was emptied, rinsed thoroughly and dried; the pycnometer was then filled with distilled water and reweighed, the mass was recorded as m_3. The specific gravity of the sample was then calculated using Eq. 1:

$$\text{Specific gravity} = \frac{M_2 - M_1}{M_3 - M_1} \quad (1)$$

The specific gravities obtained from the calculations carried out above were then used to determine the corresponding concentration by weight of ethanol present in the Ogogoro distillates thus: a plot of standard values of specific gravities against the corresponding concentrations (wt.%) of ethanol-water mixture at 30°C as obtained from "Perry's Chemical Engineers' Handbook" was made using Microsoft Excel. The equation obtained is:

$$y = -0.000007x^2 - 0.0014x + 0.9949$$

whose correlation coefficient R^2 value is 0.9998. This equation was then used to determine the concentrations (wt%) of samples for which their specific gravities were earlier determined.

RESULTS AND DISCUSSION

The different results obtained include the effects of packing types on the purity of the ethanol (distillate) at varied column heights-from 28-105.5 cm are clearly displayed on Table 1-5. The results of the specific gravities of ethanol-water mixtures (30°C) vs ethanol concentration (Fig. 1), the effects of packing type and column height vs purity of ethanol from local gin (ogogoro) Fig. 2, the number of copper chips packing on ethanol purity (Fig. 3) and varied weights of CaO on the purity of ethanol (Fig. 4) are also shown.

Relationship between standard specific gravity and purity of ethanol-water mixtures: The plot of the specific gravity against the purity of ethanol obtained gave a nearly linear graph (Fig. 1) with a negative slope (Table 6-8). This shows that as the level of purity of ethanol in the ethanol-water mixtures increases, the specific gravity of the mixture correspondingly reduces. It was observed that columns with no packing materials generally gave the highest specific gravity values or lower purity ethanol distillates (Table 1-6). Also, the height of the column affected the specific gravity of the ethanol-water mixtures. As the height is increased, the specific gravity of the distillates obtained reduced which implied higher purity for the distillates (Table 1-6).

The effect of column packing material on separation efficiency: Un-distilled local gin (Ogogoro) of 28.08% purity was subjected to fractional distillation process using different types o f column packing materials. This was done in order to provide more surface area for the ascending vapour and also increase vaporization-condensation cycles. Packing materials were loaded into the distillation columns while unpacked distillation columns of same heights were also investigated which served as controls for the experiments.

Results from the experiments showed that unpacked fractionation columns yielded ethanol distillates with lower purity than those of packed distillation columns. It

Table 1: Packing materials and varied column heights in local gin purification

	Height of column used (cm)					
Packing material used	1	2	3	4	5	6
Unpacked column	28	33.3	42.2	61.3	70.2	103.5
Glass	28	33.3	42.2	61.3	70.2	103.5
Copper	28	33.3	42.2	61.3	70.2	103.5
Plastic	28	33.3	42.2	61.3	70.2	103.5

Table 2: The effect of packing type on the concentration (wt%) of the distillate at column height of 28 cm

Height of distillation column = 28 cm					
Types of packing	No. of packing materials	Initial volume (mL)	Distillation temperature range (°C)	Specific gravity of distillate (30°C)	Concentration of distillate (wt.%)
None	0	500	78.5-79.0	0.8352	81.13
Glass	50	500	78.5-79.0	0.8144	89.19
Copper	70	500	78.0-79.0	0.8133	89.60
Plastic	40	500	78.5-79.5	0.8188	87.48

Table 3: Effect of packing type on the concentration (wt%) of the distillate at column height of 33.3 cm

Height of distillation column = 33.3 cm					
Types of packing	No. of packing materials	Initial volume (mL)	Distillation temperature range (°C)	Specific gravity of distillate (30°C)	Concentration of distillate (wt.%)
None	0	500	78.0-79.0	0.8396	79.42
Glass	75	500	78.5-79.5	0.8167	88.30
Copper	80	500	78.5-79.0	0.8138	89.41
Plastic	57	500	78.5-79.0	0.8182	87.73

Table 4: Effect of type of packing on the concentration (wt.%) of the distillate using the 42.2 cm column

Height of distillation column = 42.2 cm

Types of packing	No. of packing materials	Initial volume (mL)	Distillation temperature range (°C)	Specific gravity of distillate (30°C)	Concentration of distillate (wt.%)
None	0	500	78.5-79.0	0.8313	82.68
Glass	110	500	78.0-79.0	0.8134	89.55
Copper	120	500	78.0-79.5	0.8122	89.99
Plastic	70	500	78.5-79.5	0.8181	87.78

Table 5: Effect of type of packing on the concentration (wt.%) of the distillate using the 61.3 cm column

Height of distillation column = 61.3 cm

Types of packing	No. of packing materials	Initial volume (mL)	Distillation temperature range (°C)	Specific gravity of distillate (30°C)	Concentration of distillate (wt.%)
None	0	500	78.5-79.0	0.8274	84.18
Glass	125	500	78.5-79.0	0.8120	90.06
Copper	150	500	78.5-79.5	0.8089	91.25
Plastic	97	500	78.5-79.0	0.8164	88.40

Table 6: Effect of type of packing on the concentration (wt.%) of the distillate using the 70.2 cm column

Height of distillation column = 70.2 cm

Types of packing	No. of packing materials	Initial volume (mL)	Distillation temperature range (°C)	Specific gravity of distillate (30°C)	Concentration of distillate (wt.%)
None	0	500	78.0-79.0	0.8237	85.64
Glass	160	500	78.5-79.0	0.8064	92.16
Copper	190	500	78.5-79.5	0.8083	91.46
Plastic	110	500	78.0-79.0	0.8118	90.16

Table 7: Effect of type of packing on the concentration (wt.%) of the distillate using the 103.5 cm column

Height of distillation column = 103.5 cm

Types of packing	No. of packing materials	Initial volume (mL)	Distillation temperature range (°C)	Specific gravity of distillate (30°C)	Concentration of distillate (wt.%)
None	0	500	78.5-79.5	0.8208	86.74
Glass	235	500	78.5-79.5	0.8072	91.87
Copper	270	500	78.5-79.0	0.8073	91.85
Plastic	167	500	78.5-79.5	0.8105	90.63

Table 8: Summary of the effect of variation of column height and type of packing on the concentration (wt.%) of the distillate

Height of distillation column (cm)	Concentrations (wt.%) of the ethanol samples obtained from the different types of packing materials			
	No.packing (empty column)	Glass	Plastic	Copper
28.00	81.13	89.19	87.48	89.60
33.30	79.42	88.30	87.73	89.40
42.20	82.68	89.55	87.78	89.99
61.30	84.18	90.06	88.40	91.25
70.20	85.64	92.16	90.16	91.46
103.5	86.74	91.87	90.63	91.85

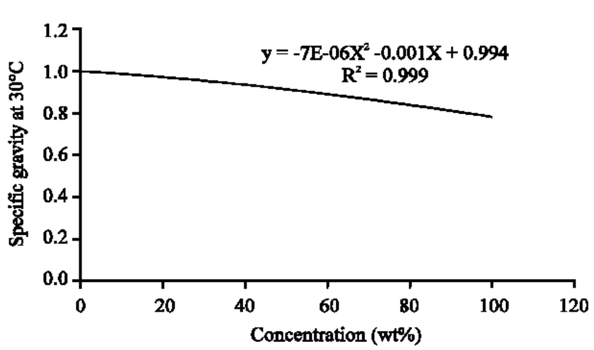

Fig. 1: Specific gravities of ethanol-water mixtures (30°C) vs. ethanol concentration

was observed that as the height of the distillation column (packed and unpacked) increased, the percentage of purity of the distillates also increased. The ascending vapours in the empty distillation columns encountered low surface area and reduced vaporization-condensation cycles in unpacked than those in the packed columns. The effect of the type of packing was also studied, using copper chips, glass chips and plastic beads as basis for comparison (Table 1-8). By visual inspection, it was observed that the random copper chips had relatively smaller piece sizes than the glass chips and the plastic beads and as such for the same column height, more number of copper chips was generally required to pack,

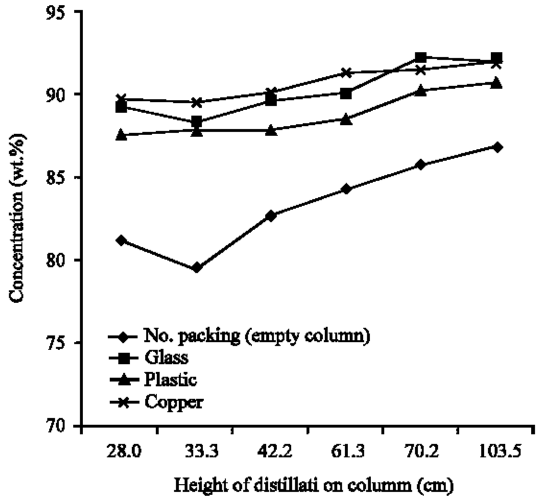

Fig. 2: Packing type and column height vs. purity of ethanol from Ogogoro

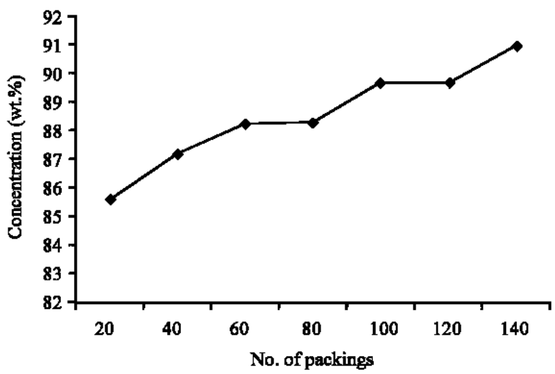

Fig. 3: Effect of number of copper chip packings on the etanol purity

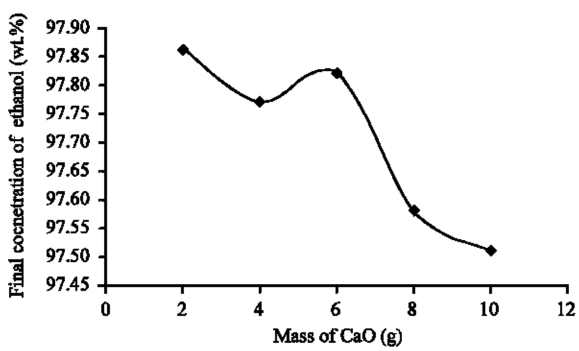

Fig. 4: Varied weights of CaO on the purity of ethanol

it than glass chips and plastic beads considering the space available for packing. This was closely followed by glass chips and plastic beads which gave the least number of packing materials. However, with packings, separation efficiency is enhanced due to the increased tendency of ethanol-vapour-escape relative to water through the packings. From the experiment carried out, it was observed that the columns packed with random copper chips yielded ethanol samples with higher purity than those packed with random glass chip and plastic beads while the distillation columns packed with the plastic beads yielded lowest purities of ethanol samples. Smaller piece sizes (copper chips) provided higher contact surface area per unit volume and higher separation efficiency while the glass chips provided less specific surface area than the random copper chips, hence lower separation efficiency than the random copper chips. Of all the three packing materials, the plastic beads provided the lowest surface area and hence the lowest separation efficiency relative to the copper and glass chips. Although, random copper chips gave the best results but for economic considerations, glass chips may be preferred as they are cheaper than copper; also, copper may degrade by corrosion in the long run.

Effect of distillation column height on ethanol distillate purity: Starting with local gin (Ogogoro) of 28.08% purity, the effect of height of distillation column on purity of distilled gin was studied. The height of the distillation column was varied from 28-103.5 cm. Height of distillation column was found to impact on the level of purity of ethanol obtained (Table 1-8). Generally, as the height of column increased, the level of purity of the distillate (ethanol) also increased for each column type, i.e., unpacked and packed. However, to sustain this positive trend, caution must be put in place to prevent the temperature from rising above 80°C, so as not to allow water vapour to condense alongside the ethanol vapour thereby diluting the ethanol distillate thus reducing the ethanol purity.

Liquid holdup, a phenomenon which occurs when the loading on a packed bed is increased to the extent that the liquid begins to accumulate in the packed bed was observed in the distillation columns of height 33.3 cm that were packed with random glass and copper chips. This accounted for reduction in the separation efficiency recorded for a column height of 33.3 cm (Table 2). As loading continues to increase, the packing in the columns will reach a point where the efficiency begins to decrease.

The effects of variation of number of packings in a distillation column on ethanol distillate purity: Since, the random copper chips yielded the best results in the first experiment they were used to investigate the effects of the variation of number of packings on the purity of the ethanol samples. The distillation column of height 70.2 cm was used for the distillation process. It was observed that as the number of packing materials per unit volume increased there was a steady rise in the purity of the ethanol samples obtained from Ogogoro distillation (Fig. 3). Increase in number of packing materials per unit volume gives room for higher contact surface areas with the distilling gin which resulted in more vaporization-condensation cycles leading to higher separation efficiency (Fig. 3). The number of vaporization-condensation cycles that can occur within a fractionation column determines the level of purity that can be attained. A deviation from the observed increment was observed when 80 random copper chips yielded the same ethanol purity as 60 random copper chips and may be due to occurrence of liquid holdup when the column was packed with 80 random copper chips.

The effects of variation of the weight of calcium oxide (lime) on the percentage purity of ethanol: Dehydration with CaO was employed to help break or alter the ethanol-water azeotrope, so as to obtain ethanol of higher purity. Lime reacts with water to form calcium hydroxide ($Ca(OH)_2$). The calcium hydroxide formed is insoluble in ethanol, so separation from the ethanol is easy:

$$CaO(s) + H_2O(1) \rightarrow Ca(OH)_2(s)$$

In order to effectively increase the purity of the distilled gin, the possible effect of variation of mass of CaO used in the drying process of the distillate was investigated. Distilled gin with initial purity of 90.46% were dried with 2, 4, 6, 8 and 10 g of CaO. The result is as shown. It was observed from Fig. 4 that lower weights of CaO used as drying agents favoured higher purities of ethanol. For effectiveness of the drying process, CaO must be sufficiently enough as excessive amounts could cause a large proportion of calcium hydroxide to be formed and towards the end of the distillation; water is released which dilutes the alcohol again.

CONCLUSION

From the research findings, it is evident that the efficiency of fractional distillation can be increased by increasing the height of the fractionation column and the number of packings. Smaller piece sizes of random packings increase the efficiency of fractional distillation. Surface area is a very important factor that can affect the efficiency of fractional distillation. Azeotrope stability of ethanol-water mixture can be altered using calcium oxide which gives better performance and improved ethanol purity with smaller masses of CaO per unit volume.

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