Development of an improvised convertible distillation apparatus for teaching and learning chemistry

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ABSTRACT

Distillation is an important concept in chemistry as it involves separation techniques which are widely used in various industries. However, despite its significance, it is hardly understood and appreciated because it is rarely performed in laboratory experiments. Distillation requires expensive apparatus to conduct which most schools cannot afford. Hence, an improvised convertible distillation apparatus for simple, fractional and steam distillation was developed using common household and recyclable materials. Improvised measuring devices were also fabricated to determine the purity of the distillates. The performance of improvised apparatus was assessed and compared with the standard apparatus using real samples. The improvised apparatus, using fractional distillation set-up, produced 91.3% alcohol from alcoholic beverage while the simple/steam distillation setup, yielded distilled water from the salt-water sample and essential oil from pomelo (*Citrus maxima*) peel with percentage yield of 0.56%, respectively. The improvised apparatus for fractional distillation yielded a higher alcohol content ($\bar{x}$=91.3%; 95%CI=91.0, 91.6) than the standard apparatus ($\bar{x}$=85.7%; 95%CI=85.3, 86.1) while the efficiency of separation of the improvised apparatus for simple/steam distillation was comparable with standard apparatus. Thus, the improvised apparatus offers a cheaper alternative for conducting distillation process in chemistry experiments. The cost of performing distillation process is further reduced by using improvised measuring devices to measure the purity of the distillates in lieu of chemicals and reagents. Aside from being cost-effective, the improvised apparatus is easy to construct, durable, user-friendly and safe to use.

***Keywords**:* chemistry experiments, distillation, improvisation, low-cost

INTRODUCTION

Laboratory plays an important role in effective teaching and learning chemistry. Concepts in chemistry are better understood and appreciated if practical works are conducted in a well-equipped laboratory. It focuses more on student-centered teaching approach, which is more effective in the mastery of skills and deep understanding of concepts in science than the teacher-centered approach (Granger et al. 2012). In the Philippines, the lack of science equipment and facilities (Marinas undated) has been a serious concern for public school teachers, particularly in rural areas. This occurs as a result of an inadequate budget to meet the demand for acquiring expensive laboratory materials and equipment (Padolina and Magno undated). Thus, the concept of improvisation became popular in conducting laboratory experiments as it helps address the problem of lack of equipment due to financial constraints and brings out creativity among students and teachers. Improvisation could be defined as the making of alternative instructional materials from the use of locally available resources (Ndirangu 2003) and it is often advocated as a low-cost solution in terms of equipment and chemicals (Kimel 1998). It brings out the same learning results as standard materials.

Several low-cost apparatus for chemistry experiments have been developed from simple laboratory glassware (Yitbarek 2012) to sophisticated laboratory instruments to address the problem of insufficient budget. Muhammad and Lawal (2015) made use of common household materials as an alternative to standard laboratory equipment and reagents. An ordinary syringe was used as vacuum source in vacuum filtration (Zhilin and Kjonaas 2013) and as electronic buret for acid-base titration (Cao et al. 2015). To introduce the concept of spectroscopy to undergraduate chemistry laboratory, Wigton et al. (2011) developed a low-cost portable fluorimeter using a 360 nm light emitting diode (LED) for excitation and a silicon photodiode for detection. LEGO blocks were utilized to make an inexpensive visible light absorption spectrophotometer (Albert et al. 2012) and were also used to construct a simple, inexpensive, and robust colorimeter (Asheim et al. 2014). And recently, a spectrophotometer using a smartphone's light sensor as a detector and an application to calculate and display absorbance values was constructed and tested by Hosker (2018).

While there are growing interests in the fabrication of inexpensive laboratory apparatus for teaching chemistry in the past few years, only a few studies have been dedicated to the development of low-cost distillation apparatus (Babu et al. 2002). Distillation is considered one of the most important separation techniques in chemistry. It is widely used in the production of essential oils (Stratakos and Koidis 2016) and alcohol (Cho et al. 2013) and the extraction of medically important organic compounds in plants (Singh 2016). However, distillation apparatus that are available in the market are very expensive and most parts are usually made of glass which is susceptible to cracks and breakages. Additionally, conventional distillation set up employs open flame as the heat source which constitutes a fire hazard. Literature for improvised distillation apparatus is limited and no studies have been found as to the development of an improvised distillation apparatus that can be used interchangeably for simple, fractional and steam distillation processes. It is, therefore, the aim of the study to develop an improvised distillation apparatus using household and recyclable materials for simple, fractional and steam distillation that is safe to use, cost-effective, durable and easy to assemble. The study also aims to fabricate improvised measuring devices to test the purity of the distillates instead of using chemicals and reagents.

METHODS

The study was conducted in three stages. The initial stage includes the modification and characterization of an electrothermal cup as heat source and distilling pot. This was followed by the development and optimization of interchangeable distilling lid which allows the use of improvised apparatus for simple, fractional and steam distillation. This stage also includes the fabrication of improvised devices for measuring conductivity and density to determine the purity of the distillates collected from simple and fractional distillation, respectively. The last stage was performance and cost evaluation of the fabricated distillation apparatus. Real samples were used to compare the performance of improvised distillation apparatus with a commercially available standard distillation apparatus. Correlation analysis and paired t-test at 95% confidence interval were used as a statistical tool to compare the efficiency of improvised apparatus with standard apparatus (McDonald 2014; Kim 2015). Statistical values were computed using Analysis Toolpak in Excel.

Modification and Characterization of Improvised Heat Source

Figure 1 is the schematic diagram depicting a complete set-up for improvised distillation apparatus which was basically composed of electrothermal cup, interchangeable cup lid, condenser, light dimmer, digital multimeter, and submersible aquarium pump. A one-liter stainless steel electrothermal cup was used as the heat source and as distilling pot. Bended glass tubing was inserted and fixed on the side of the cup to serve as a water level indicator.

The light dimmer and digital multimeter were integrated into the power plug of the electrothermal cup in a manner shown in Figure 2. The light dimmer was used as the power switch and voltage regulator while the digital multi-meter served as the readout device for voltage monitoring. The heating performance of the modified electrothermal cup was characterized by heating 500 ml of distilled water using different voltages set from 50 to 200 volts with 50-volt increment.

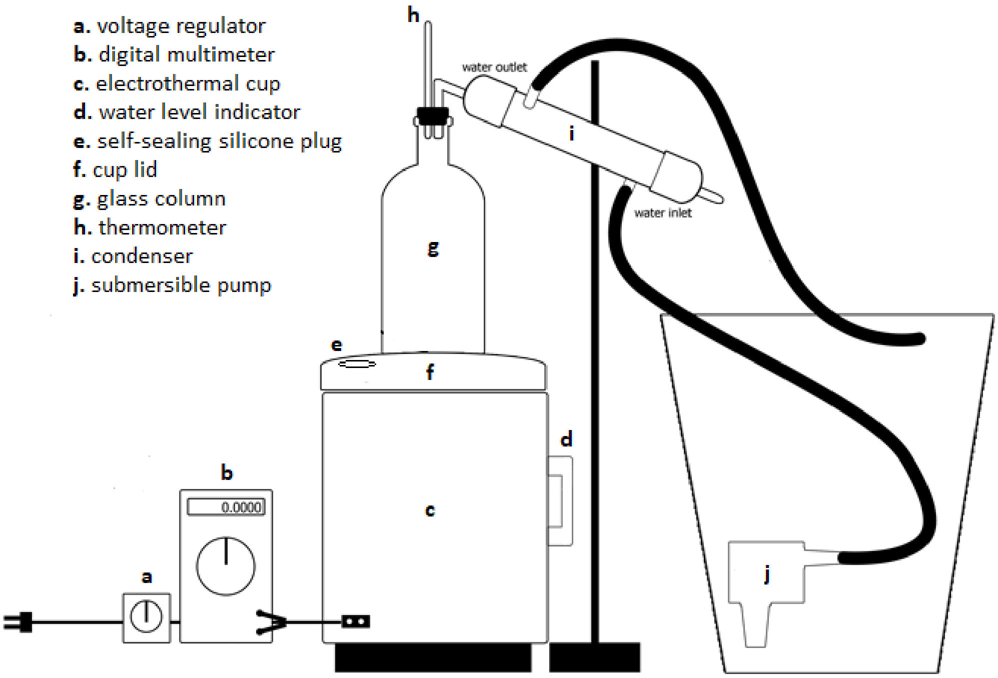

Figure 1. Schematic diagram of improvised distillation set-up

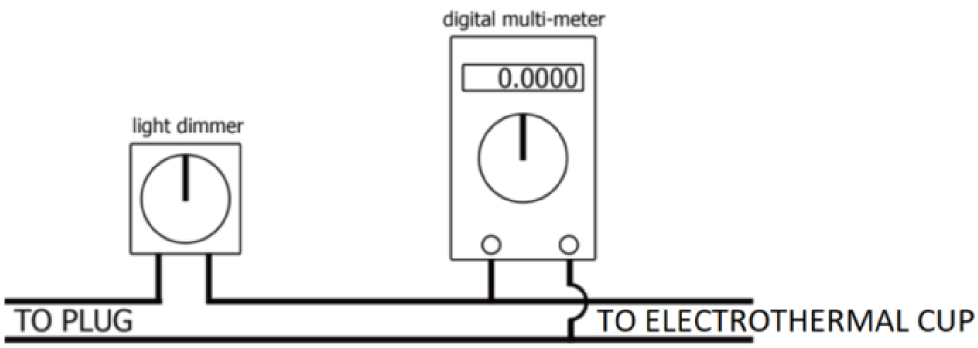

Figure 2. Modified power plug of electrothermal cup equipped with light dimmer and digital multimeter.

Fabrication and Optimization of Measuring Devices and Interchangeable Distilling Lid

Fabrication of improvised measuring device. The circuit for improvised conductivity tester was adapted from Katz and Willis (1994) with some modifications. Copper electrodes were replaced by TRS earphone jack and a 9-volt AC/DC adaptor was provided in addition to the 9-volt battery as the power source (Figure 3). Electrical components were housed in an empty permanent marker.

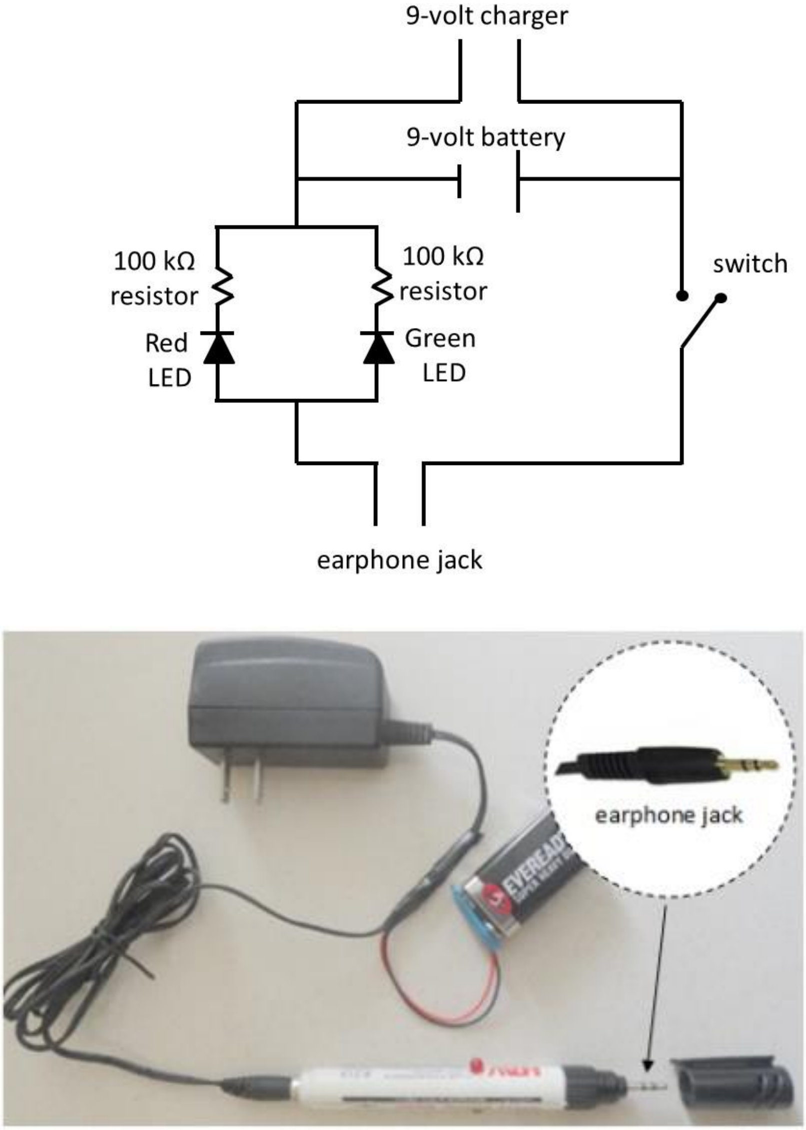

Figure 3. Circuit diagram for modified conductivity meter (top) and fabricated conductivity tester (bottom).

Improvised pycnometer was built from an empty bottle of nail polish and capillary tube (Figure 4). The capillary tube was inserted and fixed to a rubber plug which tightly covers the bottle. The performance of improvised pycnometer was compared against a standard pycnometer using different concentrations of ethanol.

Figure 4. Improvised pycnometer (left) and standard pycnometer (right).

Interchangeable distilling lid. The glass column with an external diameter of 55 mm was made from clear soda bottle which bottom was cut off. An open-end glass column was placed over the pre-cut hole at the center of the cup lid. A 19-cm long glass column was used for simple/steam distillation while a longer glass column filled with column packing was utilized for fractional distillation (Figure 5). A hole with self-sealing silicone plug was provided on the cup lid for simple/steam distillation to facilitates replenishment of vaporized water during steam distillation. Glass column for fractional distillation was constructed from several pre-cut glass bottle joined together by silicone sealant. A stainless steel mesh was placed inside the lower part of the glass column to support column packing. The length of the glass column, as well as the type and size of column packing, for fractional distillation were investigated. Glass marbles and stainless steel sponge were used as column packing.

Figure 5. Electrothermal cup (a) and distilling lid for simple/steam distillation (b) and fractional distillation (c).

The condenser was constructed using one-foot (30.28 cm) long polyvinyl chloride (PVC) pipe (size 1”), PVC caps and 7 mm glass tubing. PVC caps were used to seal both ends of the pipe. A 22-inch (55.88 cm) glass tubing was bent on one end and inserted through the length of the pipe passing both caps. Two pieces of one-inch (2.54 cm) glass tubing were inserted on the side of the pipe opposing each other and proximate to both ends of the pipe to serve as water inlet and outlet. All joints were sealed with two-part epoxy resin to prevent leakages.

Performance Evaluation

Three hundred milliliters of samples were used for simple and fractional distillation while 250 grams of plant sample were utilized for steam distillation. Distilled water and absolute ethanol were used as reference standards throughout the study. Boiling stones were added to the samples to prevent bumping. A submersible aquarium pump was utilized to re-circulate water coming out of the condenser. Ice was added to the water reservoir to provide a continuous supply of cold water to the condenser. All measuring instruments were calibrated prior to use. The improvised apparatus applied for all types of distillation process were run against standard distillation apparatus. The Vigreux column was used as the standard fractionating column. Three trials were conducted for each type of distillation process.

Simple distillation. The performance of the simple distillation set-up was evaluated by separating water from the salt-water mixture. Three hundred milliliters of water samples were placed inside the cup. The voltage was set at 150 volts. The temperature was monitored and recorded at the first drop of the distillate. The purity of distillates was determined by measuring its conductivity using the fabricated conductivity tester and results were compared with the conductivity scale given in Table 1.

Table 1. Degree of electrical conductivity of sample solution based on lighted light emitting diode (LED).

Scale	Red LED	Green LED	Conductivity
0	Off	Off	None
1	On	Off	Medium
2	On	On	High

Fractional distillation. The efficiency of the fractional distillation set-up was determined using 80-proof liquor. The glass column was wrapped with aluminum foil leaving an air space between the column and the foil to serve as an insulator (Lancaster 2017). Three hundred milliliters of the sample were transferred into the cup. The voltage was initially set at 150 volts and adjusted to 100 volts after the temperature reached ten degrees below the boiling point of ethanol (78°C). The voltage was further adjusted upon the first drop of distillate to maintain a drop rate of 20 drops per minute (Yoder et al. undated) while maintaining the temperature close to 78°C. Fractions were collected every 10 ml of distillates in a graduated cylinder placed in an ice bath. The temperature was monitored and recorded for each fraction collected. Distillation process was continued until a sharp increase in temperature was observed and distillation was stopped when the temperature reached the boiling point of water. The percentage composition of ethanol and water in each fraction was determined indirectly by comparing the density of each fraction with the Table for Concentration of Ethanol-Water Mixture Versus Specific Gravity at Various Temperature (Perry et al. 1997). The density was measured using an improvised pycnometer. The performance of improvised apparatus for fractional distillation was compared with standard apparatus for fractional distillation with Vigreaux column.

Steam distillation. The efficiency of the improvised steam distillation was evaluated based on the physical properties and percentage yield of oil obtained from the plant sample. Two hundred fifty grams of grated pomelo peel and 400 ml of distilled water were transferred into the electrothermal cup. The water level was marked and monitored through the water level indicator. Glass vessel with distilled water was inserted into the self-sealing silicone plug to replenish the water lost during steam distillation.

Steam distillation was carried out at 100-volt setting and continued until no visible droplet of oil was observed in the distillate.

RESULTS

Performance of Modified Electrothermal Cup as Heat Source

Simple, steam and fractional distillation share basic apparatus consisting of heat source, distillation pot, distilling head, thermometer, condenser, and receiving flask. A fractionating column placed between the distillation pot and condenser differentiates fractional distillation from simple and steam distillation set-up.

A modified electrothemal cup was used as the heat source and distillation pot. The effect of a voltage input to heating rate was examined by heating distilled water to boiling using different voltages. The temperature was monitored and plotted against time and Figure 6 shows that the heating rate is proportional to the voltage input. The higher the voltage input, the faster the water boils. The sharp increase in temperature was observed for higher voltages while a gradual increase was observed at 50 volts. The boiling point of water could be reached in less than 10 minutes at 200-volt setting.

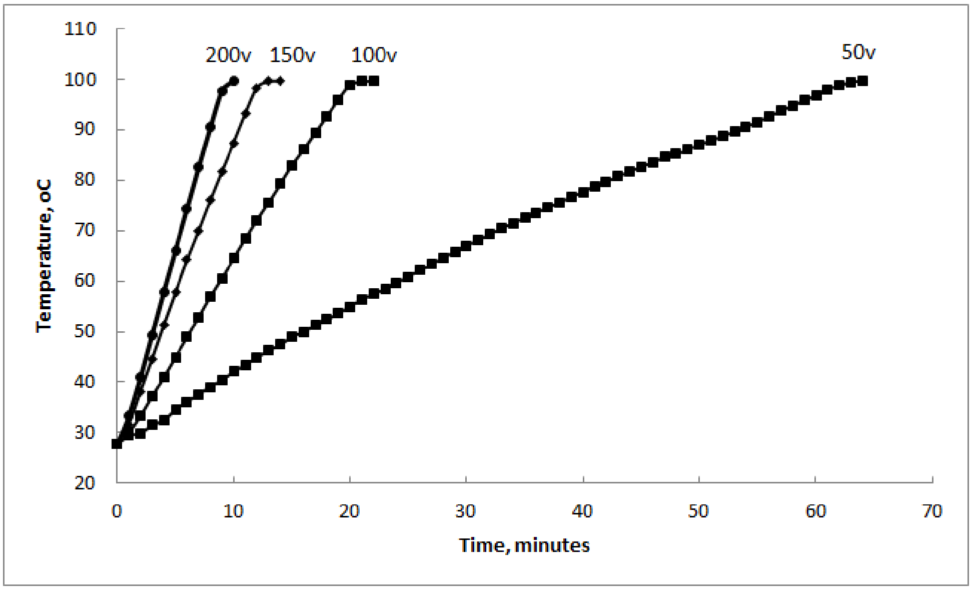

Figure 6. The heating rate of the modified electrothermal cup at different voltages.

Performance of Improvised Pycnometer and Fractional Distillation Lid

The density of an aqueous solution of ethanol varies depending on the amount of alcohol present in the solution. The density of prepared solutions having different concentrations of ethanol was measured using the improvised pycnometer and Figure 7 shows that the density of sample solution decreases as the percentage of alcohol in solution increases. The performance of the improvised pycnometer was found to have a strong correlation (R = 0.9996) with standard pycnometer as shown in Figure 8.

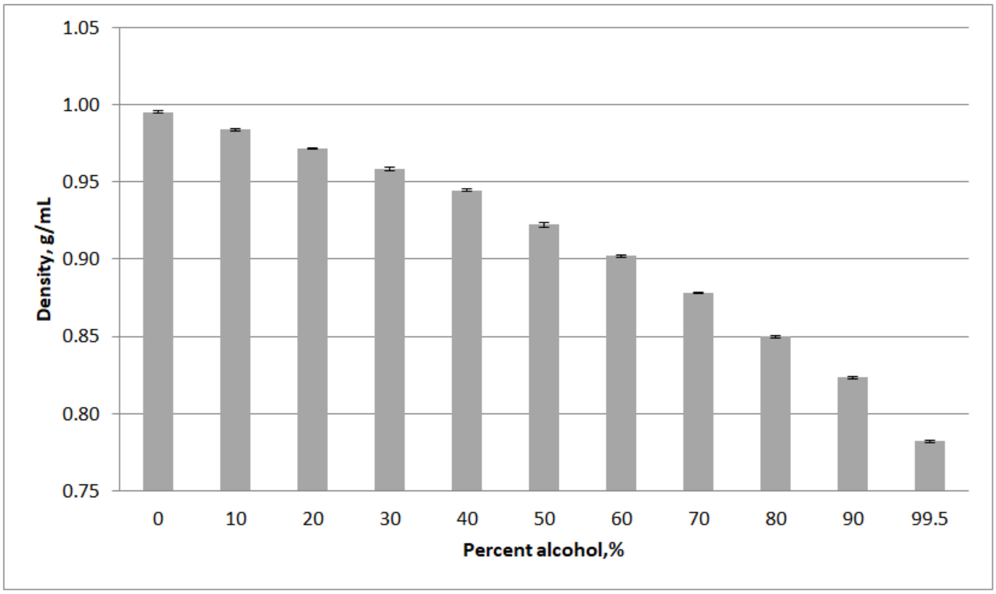

Figure 7. The density of ethanol at different concentrations using improvised pycnometer (number of replicates = 3).

The developed distillation apparatus can be converted easily into any other type of distillation set-up by simply changing the lid. Miscible liquids with a boiling point difference of less than 40°C are efficiently separated by fractional distillation than by simple distillation. The fractional distillation has the effect of several simple distillation processes in a single distillation apparatus and this is achieved with the use of a fractionating column.

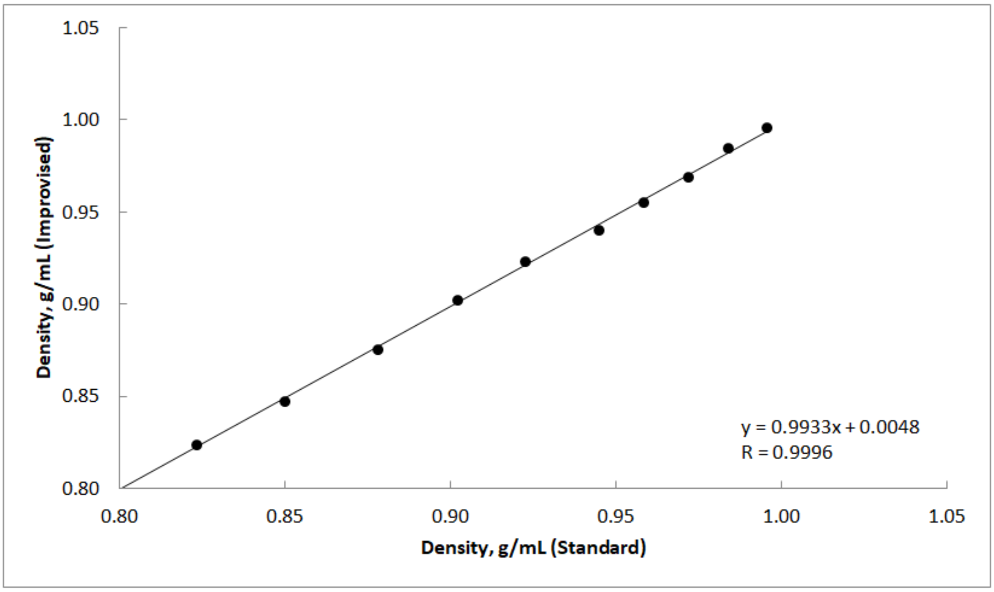

Figure 8. Correlation of density determined using improvised and standard pycnometer for different alcohol concentrations.

Figure 9 shows the distillation plot for different types of column packing placed in a 19-cm long glass column. A column packing made from 15.9 mm glass marble yielded a distillation plot with temperature that continuously increases with the volume of distillate while for column packing made from 11.1 mm glass marble and stainless steel sponge, several fractions were collected first at almost the same temperature before a sharp increase in temperature was observed.

Glass columns having different lengths and filled with 11.1-mm glass marbles were also investigated. Figure 10 shows that temperature increases continuously as the volume of distillate increases for the shortest column while the longer columns demonstrated steady temperature for several volumes of distillates before a sharp increase in temperature was observed.

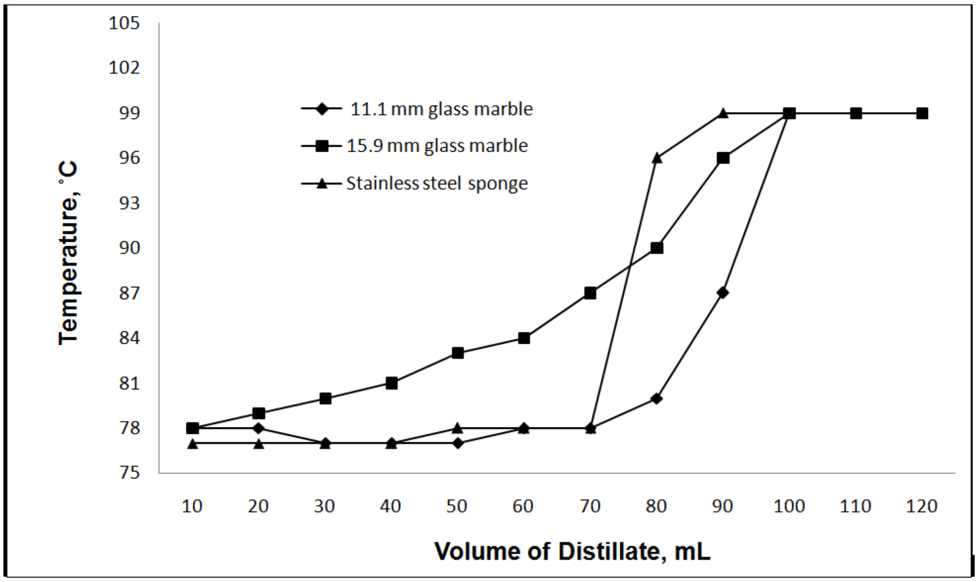

Figure 9. Effect of different types and sizes of column packing on the efficiency of separation of alcohol from sample mixture using 19 cm column (number of replicates = 3).

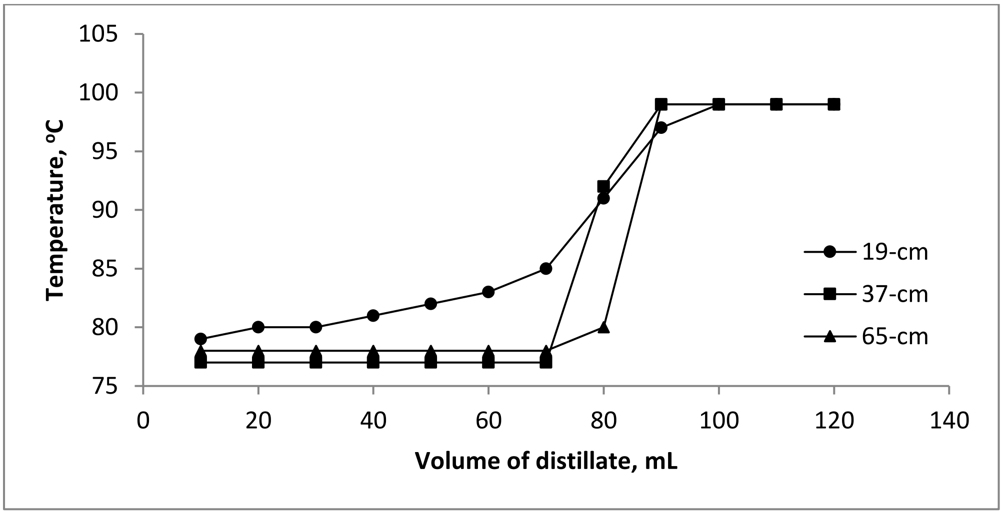

Figure 10. Effect of different lengths of the fractionating column on the efficiency of separation of alcohol from sample mixture (number of replicates =3).

The alcohol content of distillates collected from 65-cm long fractionating column yielded a higher percentage of alcohol than the distillates collected from 37-cm glass column (Figure 11).

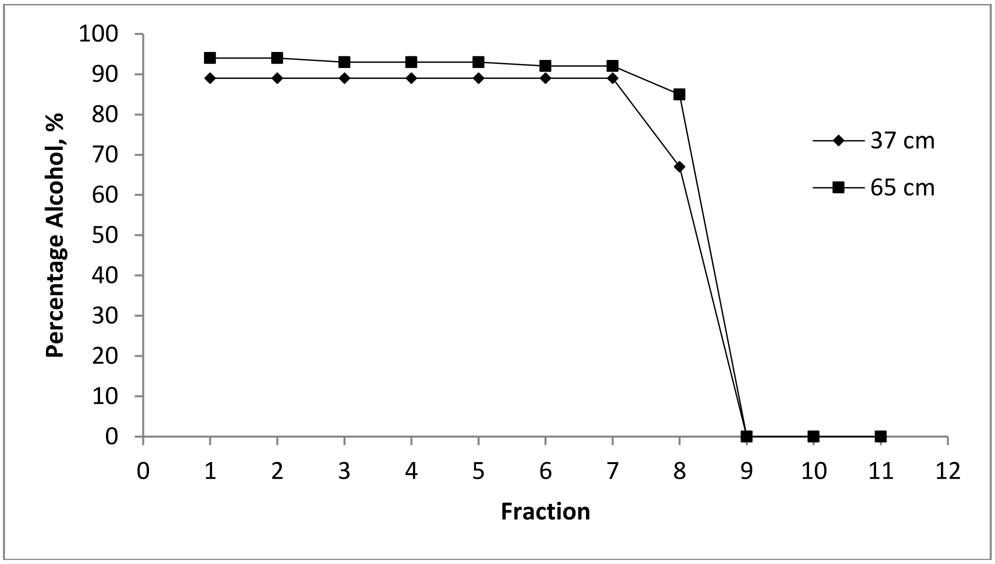

Figure 11. Percentage of alcohol at different fractions collected using different length of glass column (number of replicates = 3).

Performance and Cost of Improvised Distillation Apparatus

Simple distillation. The conductivity of distillates collected from the salt-water mixture by simple distillation set-up was tested using the improvised conductivity tester. Conductivity was observed when the light emitting diodes (LED) in the conductivity tester turned on which indicates the presence of salt in water. Salt-water mixture yielded high conductivity while standard distilled water and distillates collected from improvised and standard set-up for simple distillation produced zero conductivity (Table 2).

Table 2. The conductivity of distillates collected from improvised and standard apparatus for simple distillation.

Sample	Conductivity	
	Improvised	Standard
Salt-water mixture	2	2
Distillate	0	0
Standard distilled water	0	0

Fractional distillation. Figure 12 shows the performance of the improvised apparatus against standard apparatus for fractional distillation. The average density of distillates collected from improvised apparatus ($\bar{x}$=0.8056; SD=0.0015; 95%CI=0.8068, 0.8045) was lower than the average density of distillates collected from standard apparatus ($\bar{x}$=0.8176; SD =0.0024; 95%CI=0.8193, 0.8158).

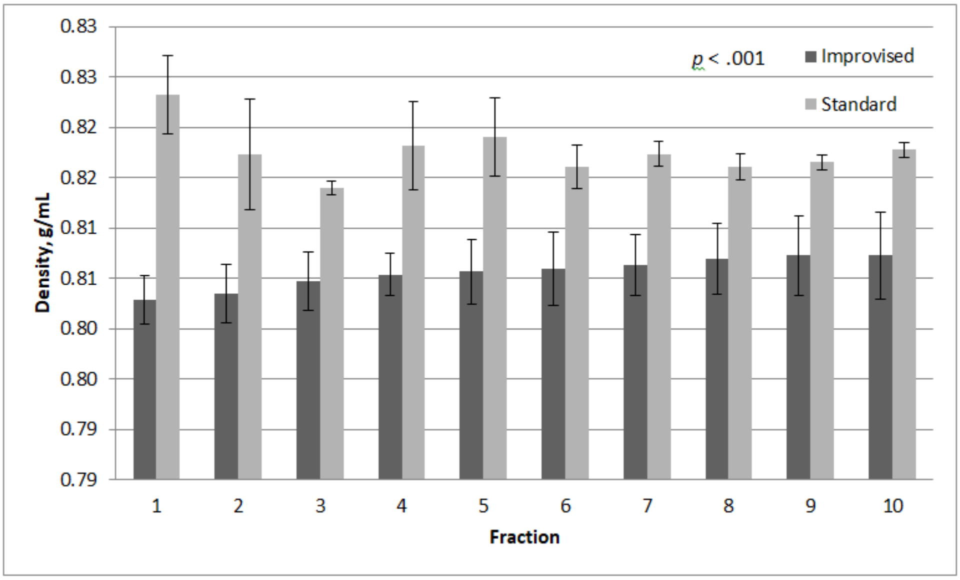

Figure 12. The density of the first ten fractions collected from improvised apparatus ($\bar{x}$=0.8056; SD=0.0015; 95%CI=0.8068, 0.8045) and standard apparatus ($\bar{x}$=0.8176; SD =0.0024; 95%CI=0.8193, 0.8158).

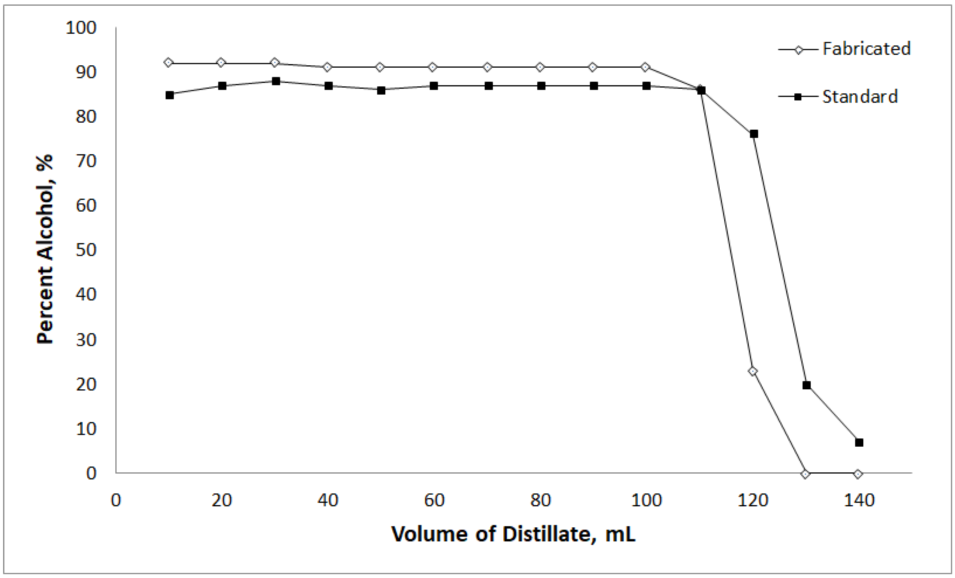

Figure 13. Percentage of alcohol for the first ten fractions collected using improvised and standard (Vigreaux) fractional distillation apparatus.

Steam distillation. Steam distillation is used to separate immiscible liquids. It is commonly used to extract essential oils from various plant materials. Since many organic compounds tend to decompose at their boiling point, water is introduced into the distillation apparatus. The water vapor carries small amounts of the vaporized compounds to the collection flask, where the immediate product is a two-phase system of water and the organic distillate, allowing for easy collection. As a proof of concept, the improvised distillation apparatus was used for steam distillation of pomelo peel to extract essential oil. Physical characteristics and percentage yield of essential oil extracted from pomelo peel using improvised and standard distillation apparatus are presented in Table 3.

Table 3. Comparison of essential oils produced from improvised and standard apparatus for steam distillation.

Apparatus	**Appearance**	**Odor**	**%Yield ($\bar{x}$)**	**95% Confidence interval, (n=3)**
Improvised	Clear	Citrusy	0.56	0.45 - 0.65
Standard	Clear	Citrusy	0.60	0.43 - 0.73

Cost evaluation. The cost for fabrication of one set of improvised distillation apparatus together with the measuring devices is 99% cheaper than purchasing its standard counterpart (Table 4).

Table 4. Cost of improvised distilling apparatus and standard distillation apparatus.

Improvised Apparatus	**Cost (PhP)**	**Standard Apparatus**	**Cost (PhP)**
Modified electrothermal cup, 1 L-capacity with voltage regulator and read-out device	740.00	Heating mantle, 500 ml-cap 500 ml boiling flask (2-neck)	45,900.00 6,900.00
Distilling lid	20.00	Distilling head	5,900.00
Fractionating column filled with column packing	132.00	Fractionating column (Vigreaux)	12,500.00
Condenser	52.00	Liebeg condenser	4,900.00
Conductivity tester	50.00	Conductivity meter	21,750.00
Pycnometer	10.00	Pycnometer with thermometer, 50 ml-cap	13,330.00
TOTAL	**1,004.00**	**TOTAL**	**111,180.00**

DISCUSSION

Modified Electrothermal Cup

The modified electrothermal cup served both as the heat source and distillation pot. The light dimmer incorporated into the power plug regulates the voltage input and permits variation on the heating rate. This allows the sample to be heated to its boiling point at a faster rate and regulate the temperature with ease during the distillation process. Unlike the Bunsen burner, the electrothermal cup is safe to use because it does not involve the use of open flame for heating and considering that the temperature controller is not within or near the heat source, the distillation process can be operated at a safer distance. Unlike the conventional distilling flask which is commonly made of glass, the electrothermal cup was made from stainless steel which is not prone to cracks and breakages. It has a wide mouth to facilitate easy cleaning as well as easy charging and discharging of plant materials during steam distillation. The cup was also provided with a plastic handle for easy handling. Further, the modified electrothermal cup could be used for other experiments which would require heating such as melting point and boiling point determination.

Improvised Measuring Devices

It is important to test the sample solution before and after the distillation process to determine its efficiency of separating individual components in a solution. Improvised measuring devices were fabricated to determine the purity of the distillate collected from the distillation process. It further lowers the cost of conducting distillation process in school experiments because it eliminates the use of expensive apparatus and chemicals for testing the purity of the distillates.

Improvised conductivity tester was used to determine the purity of distillates collected from the salt-water mixture using simple distillation set-up while improvised pycnometer was used to determine the purity of ethanol produced from alcoholic beverage using fractional distillation set-up. The conductivity tester presented by Katz and Willis (1994) was modified to have a dual power source. It can be powered either by electricity or by a 9-volt battery. The TRS earphone jack was used instead of copper electrodes to make the distance between the two electrodes constant throughout the experiments.

An improvised pycnometer was used to determine the density of different concentration of ethanol in aqueous solution and density varies as the concentration of ethanol changes. The density of the solutions could be used to determine the concentration of alcohol in the solution by converting it into a concentration unit using a suitable concentration table. The fabricated pycnometer was validated against standard pycnometer and aside from exhibiting high precision (RSD < 0.04%), it shows a strong correlation (R = 0.9996) with standard pycnometer over a range of ethanol concentrations.

Fractionating Column

Efficient separation of an ethanol-water mixture is represented by distillation plot characterized by a relatively stable temperature at which several fractions of distillates were collected and a sharp increase in temperature that reaches a plateau. The lower temperature plateau represents the lower boiling point component of the mixture while the higher temperature plateau represents the higher boiling point component. Distillates collected at the same temperature indicate a relatively pure substance while distillates collected at different temperatures constitute varying amounts of alcohol content as solutions with different concentrations of alcohol have a unique boiling point.

The efficiency of separation for fractional distillation is influenced by the size of the column packing and the length of the fractionating column Madson 2003; Pavia et al. 2005). Better separation of alcohol was achieved through the use of stainless steel sponge and smaller glass marbles with a diameter of 11.1 mm than the larger glass marbles with a diameter of 15.9 mm. This behavior could be due to the higher surface area available in stainless steel sponge and smaller glass marbles compared to the larger glass marbles. Higher surface area means greater contact between the rising steam and the condensed liquid descending down the column. However, smaller glass marbles were used favorably over stainless steel sponge due to the ease of charging and discharging it from the glass column.

The length of the fractionating column was also found to affect the efficiency of the separation of ethanol from water. Fractional distillation set-up utilizing 65-cm column produced a higher concentration of alcohol than set-up employing 37-cm column and subsequently, the longer column was used throughout the study. The increase in the efficiency of separation could also be attributed to the increase in the evaporation-condensation steps occurring in longer columns (Pavia et al. 2005).

Comparison with Standard Distillation Apparatus

The performance of the improvised apparatus for simple, fractional and steam distillation was compared with standard distillation apparatus. The efficiency of improvised apparatus for simple and steam distillation was found to be comparable with standard apparatus while improvised apparatus for fractional distillation was shown to be more efficient in purifying ethanol from alcoholic beverage than standard apparatus. Figure 12 shows that the improvised apparatus yielded distillates with a lower density (higher concentration of alcohol) and exhibited more reproducible results than the distillates collected from standard apparatus for the first ten fractions. A paired sample t-test was conducted to compare the performance of improvised and standard apparatus for fractional distillation. The statistical test shows that there was a significant difference between improvised and standard apparatus; $t(9) = -11.00$, $p < .001$. The results suggest that the improvised apparatus is more efficient in separating ethanol from alcoholic beverage and produces more reproducible results than the standard apparatus for fractional distillation that utilizes Vigreaux as the fractionating column.

The price of one set of improvised apparatus for simple, fractional and steam distillation was significantly cheaper than the standard apparatus (Table 4). The use of fabricated measuring devices instead of chemicals and reagents to test the purity of the distillates further reduces the cost of experiments involving distillation process. All materials used in the construction of the improvised apparatus are locally available and are easy to replace.

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