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# **Distillery**

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# CAPSTONE PROJECTS FINAL REPORT

SENIOR DESIGN MECE 471 HONORS PROJECT MECE 497

By

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# **Abstract**

The purpose of the senior design project for this group is to create a cost-effective distillation column (still) that produces a quality liquor. Traditionally, a copper still is used in the whiskey-making industry. Copper reacts with volatile sulfur compounds to prevent them from being in the final product. The downside of using copper as the material of construction for the distillation column is that copper is very expensive. An economical alternative would be to use a stainless steel still. This project tests the effectiveness of the steel still with copper mesh packing in reducing the concentration of sulfur volatile compounds in the final product.

This project demonstrates that there is not much difference between a copper still and a steel still with copper packing regarding sulfur volatiles and ABV of the distillate. Because of that, it is much more economical to use a steel still when distilling ethanol. This can significantly save costs for distilleries that are looking to buy new distillation columns since the price to build a steel still is a small fraction of the cost of an equally sized copper still.

If one were to continue off this project, and had enough money to spare, it would be beneficial to make a like-geometry copper still to compare with. Additionally, if future researchers have access to a higher quality testing method, there may an improvement in the accuracy of the results.

# Contents



# <span id="page-4-0"></span>**1. Outline of Subject Matter**

### <span id="page-4-1"></span>**1.1 Introduction**

The purpose of the senior design project for this group is to create a cost-effective distillation column (still) that produces a quality liquor. To make whiskey, a mixture of grain and water is heated to make "wort", then fermented to produce ethanol. To boost the alcohol concentration, or alcohol by volume (ABV), this fermented wort is distilled in a still. Traditionally, a copper still is used in the whiskey-making industry for two main reasons: 1. Copper is highly thermally conductive to promote effective heat transfer and 2. Copper reacts with volatile sulfur compounds to prevent them from being in the final product, thus making it taste better. The downside of using copper as the material of construction for the distillation column is that copper is very expensive. An economical alternative would be to use a stainless steel still that has copper mesh packing in the column. This project tests the effectiveness of the steel still with copper mesh packing in reducing the concentration of sulfur volatile compounds in the final product. A number of liquor stills already exist in the market today, giving a benchmark of how much stills cost. The end goal will be to make modifications to the stainless steel still giving it copper components so that the sulfur compounds can still be removed. The cost of the final still will be compared to the copper still to show that cost of the modified on will be less expensive to manufacture. Additionally, the group will be comparing the sulfur compound concentrations of the modified still to the steel and copper still giving an upper and lower bound of the target we are trying to hit.

In order to create a product that is accepted by the customer and by society, the group had to keep in mind societal considerations and professional responsibilities. When creating this project idea, the interest of the customer was what was kept in the forefront of our minds. Multiple things were considered in the importance of this project. This product acknowledges the attitudes towards product quality for the people buying the product coming from the stills as well the buying habits of those needing to buy the still to create thus product. This still is being aimed at those who want to start learning and investing in their own liquor company, while also being able to save some money on this product so that they can invest it elsewhere in the company. The hope of this is to be able aid, even if it is just slightly, in the creation of new small business owners, like the people doing this project. In helping the potential new business owners, it will therefore help the economy of state and country.

When creating this still, public health and safety are kept in high regard in the decisions being made. As this is a device making a consumable product, we are taking precautions to use materials that are safe for the people. Follwing FDA Code 4-202.11, the still is smooth, free of imperfections, free of sharp internal angles, corners, and crevices, and finished to have smooth welds and joints [1]. Multiple cleaning agents are used constantly on the equipment being used for brewing and distillation. This is to ensure the following of FDA Code 4-602.11 [1]. This is also in accordance to Code 4-701.10 "equipment food-contact surfaces and utensils shall be sanitized" and Code 4-701.11 "UTENSILS and FOOD-CONTACT SURFACES of EQUIPMENT shall be SANITIZED before use after cleaning" [1]. In the creation of the still, it is also noted that it is structurally sound and heat safe as the still will be heated when in use.

### <span id="page-4-3"></span><span id="page-4-2"></span>**1.2 Design**

### **1.2.1 Design procedure**

At the beginning of the design process, we started with the designing of the steel still. We knew that we wanted to keep the size on the smaller end, so we chose to make a 10 liter still. This measurement is in reference to the side of the pot at the bottom. The length of the column was chosen to be 2 feet and the diameter 2 inches. These measurements came from research. According to [2], columns are

generally 2 inch or 3 inch in diameter. This particular site would do 2 inches for 30 liters, meaning 2 is plenty for 10. The length of the column is 24 inches plus an approximate 5 inches for the 90-degree bend. The optimal height ratio is 15:1 on the low end. At 14.5:1 for ours, this fits [3]. Other components used are two thermocouples for temperature monitoring. One is on the pot while the other is at the top of the column. A copper pipe was used in this steel still in the condenser part. This was decided because the product would be in a liquid state by that point, no longer having a reaction of the vapor with the copper. This was coiled into a circle inside of an 8-liter pot with room to put ice in.

Taking from what was taught in Concepts of Design, we started with an initial function diagram, figure 1, to get our thoughts down. After this, a basic preliminary design, figure 2, was drawn



#### **Figure 2: Preliminary Design**

The next part of the design process was determining how to incorporate copper into the column. We knew that it was going to be some sort of component inside of the column itself as that is where the vapor must pass through. To help gather our thoughts we made an objective tree, figure 3, and weighted decision matrix, figure 4.



**Figure 3: Objective Tree With Weighting Factors**



#### **Figure 4: Weighted Decision Matrix**

From this information, it was decided to not go with a permanent fixture inside of the column such as copper fins. After each time copper is used in a distilling process, it needs to be thoroughly cleaned. This was a big reason in not choosing fins as it would be very hard to clean them inside the column. Plus, if the fins were too big, it may inhibit the vapor moving up the column. This left using copper chips or a thin wire mesh. In the weighted decision matrix, the two came very close, but we ended up choosing the copper wire mesh.

#### <span id="page-6-0"></span>**1.2.2 Design details**

Present the detailed design, with diagrams and component values. Show how the design equations were applied. Give equations and diagrams with specific design values and data. Place large data tables in an appendix. Circuit diagrams that are too large to be readable on a single page should be broken into pieces for presentation. The full diagram may be included in an appendix. Use photographs only as necessary and treat them, along with all other graphics except tables, as *figures*.

The first step after we had decided on the design of the still was making a basic model in SolidWorks to get all of our thoughts down. This is shown in figure 5.



#### **Figure 5: SolidWorks Model**

In order to connect the pieces together, some of which being different materials that could not be welded together, we decided to use clamps and gaskets. Some soldering was also used. With all these materials a simple Bill of Materials was formed, figure 6.



**Figure 6: Bill of Materials**

We then took this Bill of Materials and searched for existing parts from local hardware stores. To keep track of all the parts needed and what were sourced, a detailed drawing was made, figure 7.



#### **Figure 7: Detailed Sketch with Parts**

From this point all that was needed was to assemble the still. This was done with the help of a friend of ours. A secondary 10-liter copper still was purchased online. Early on in the design process we were planning on making both the copper and the steel still so as to ensure the size and shape was the same for it to be a control. Unfortunately, the cost to buy the parts ourselves was extremely high compared to buying a prebuilt one online. It would have cost us the same amount to buy a singular 10-liter copper cooking pot as it was the buy a completed still online. We acknowledge the difference in shape and column length, but this was beyond our control. The completed steel still is shown in figure 8, and the copper still is in figure 9.



**Figure 8: Completed Steel Still**



#### **Figure 9: Purchase Copper Still**

The last part that needed to be dealt with was the copper mesh insert. All this required was purchasing copper wire from a store and wrapping it upon itself in the shape of the column. We ensured that the diameter of the mesh column was approximately half an inch wider than the diameter of the column. This was so that when inserted into the column it would stay snug and not fall out.

### <span id="page-11-0"></span>**1.3 Verification**

The verification of the design includes two main parameters for the finished product: 1. The distillate is a high-proof alcohol that is at least 40% ABV and 2. The distillate has a low concentration of volatile sulfur compounds. Combined, these two parameters ensure that the still design creates a quality product.

For all distillation testing throughout the project, the same procedure was followed for creating every batch of fermented wort. The procedure is as follows:

- 1. Obtain 8.5# of dehydrated rye, 6# of dehydrated corn, and 2# of dehydrated 6-row barley.
- 2. Put the grain into a closed 5-gallon bucket and shake the bucket vigorously to evenly mix the grain.
- 3. Heat 3 gallons of water in a large pot to 160 degrees Fahrenheit and turn off the heat source.
- 4. Immediately transfer the grain from the bucket to a brew bag and place in the heated water.
- 5. Using a spoon, mix up the grain to get rid of any dry clumps of grain (dough balls) to ensure even extraction.
- 6. Stir the mixture every 10 minutes and continuously monitor the temperature to maintain 155 degrees Fahrenheit, turning the heat source on and off as necessary to maintain the set temperature.
- 7. After 45 minutes of brewing, turn off the heat source and remove the brew bag from the wort. Holding the brew bag above the pot, let the bag drain for 3 minutes.
- 8. Transfer the hot wort from the pot to a sanitized fermentation bucket and let cool to 90 degrees Fahrenheit.
- 9. Using a sanitized cup, scoop out approximately 2 cups of wort and pour into a sanitized hydrometer. Use the hydrometer to measure the starting gravity (S.G.) of the wort.
- 10. Add one packet of SafAle BE-256 yeast to the bucket and shake the closed bucket for 1 minute to disperse the yeast and incorporate oxygen into the wort.
- 11. Place the airlock in the hole on the top of the fermentation bucket and let the wort ferment for 1 week.
- 12. After 1 week of fermentation, measure the final gravity (F.G.) of the wort using the same method as in step 9. If the F.G. is not 1.000, let the fermentation continue for another 4 days to allow further fermentation.

Using the method outlined above, we were able to consistently achieve a fermented wort that has a starting ABV of 4.5%.

The first stage of testing for the project was to see if we could achieve the 40% ABV target for the finished product. This testing was done on a benchtop laboratory setup as seen in figure 10.



**Figure 10: Setup for Benchtop Distillation in the Lab**

To do this, the fermented wart was placed in a round-bottom flask that was submerged in an oil bath on a hot plate, a simple distillation was carried out. During the distillation, the temperature of the round-bottom flask was monitored, and the distillation ended when the temperature reached 99 degrees Fahrenheit. At this point, the amount of ethanol in the distillate is considerably lower than at the start. The distillate was collected into four different "cuts", with the first cut being the first 10% of the overall mixture, and the remaining four cuts being 22.5% of the mixture each. The first cut was discarded because it contains a higher concentration of methanol (up to 10% of the first cut is methanol) which is toxic.

After the cuts were cooled down to room temperature, the density of each of the cuts was measured using a pycnometer. The measurements were plotted against the calibration curve to calculate the ABV of the solution.

Several methods can be used to test the ABV of a solution, including pycnometer density

measurements, surface tension testing, viscosity, etc. For this project, we used pycnometer density measurements because the equipment was accessible, and the procedure was already known by our team. Since the density of an ethanol + water solution changes based on the concentration of ethanol, if the density of the sample of an unknown ABV is plotted against a calibration curve, the ABV can be easily determined. To create the calibration curve, the densities samples of known concentrations of ethanol were measured using a pycnometer and plotted on a curve to create a calibration curve. The calibration curve can be seen below in figure 11.



**Figure 11: Calibration Curve for Determining Ethanol Concentration**

As seen in figure 11, as the concentration of ethanol in solution increases, the density decreases. This is due to the density of ethanol being lower than water. To test the ABV of our unknown samples, we first determined the mass of solution within a pycnometer of constant volume and then plotted it on the calibration curve. The results from that testing can be seen below in table 12.



**Table 12: Ethanol Concentration Results of Unknown Samples from the Copper Still, Steel Still, and Steel Still with Copper Packing.**

The distillation performed is a simple batch distillation, which is where the vapors are taken directly from the still to the condenser in a batch process. Our batch process is a batch rectifier, which consists of a reboiler, column, and condenser. The reboiler is filled with the wort and heated to boiling temperature to vaporize it. We chose a simple batch process for our distillation because the column has a basic design (which cuts down on manufacturing costs), and ethanol and water have a wide enough gap in boiling points to allow easy separation for our 40% ABV target.

For all three still setups (copper, steel, and steel with copper packing), the first cut was discarded because it contains a relatively high concentration of methanol compared to the rest of the cuts. Methanol comprises approximately 1% of the total distillate and is almost entirely removed in the first cut. The boiling points of methanol, ethanol, and water are 64.7 Celsius, 78 Celsius, and 100 Celsius respectively. The vapor pressure of methanol, ethanol, and water at STP are 13.02 kPa, 5.95 kPa, and 3.17 kPa, so the relative volatility of methanol/ethanol is 2.19, and ethanol/water is 1.87. Since the relative volatility of methanol is so high and the boiling point so low, this supports that it will be removed first during distillation.

For all the still setups, the second cut was above the threshold of 40% ABV, which is a success regarding the target alcohol concentration. It should be noted that after the second cut the ethanol concentration drops, so the remaining cuts will have to go through a repeat distillation in order to achieve the 40% ABV mark.

To test for the sulfur content of the distilled samples, a group of 7 people were given the samples to smell for sulfur odors in a blind smell test. The scale that was given to them is in figure 13, as well as the results from the smell test.





#### **Figure 13: Scale and Results from Sulfur Testing**

The results from the smell test show that the copper still had the least amount of sulfur odors, while the steel still had the most. The results from the testing also show that there isn't much difference between each of the stills; the range of the averages of all the stills is only 0.57. Repeat testing is recommended because we were limited in how many people we could get to participate in the smell test, and they were not trained beforehand

## <span id="page-17-0"></span>**1.4 Costs**



**Figure 14: Costs to Build the Steel Still and a Copper Equivalent Still**

Figure 14 is a complete list of all the required materials and labor that were used to build both the copper and steel stills. Looking at the total cost for the stills, the copper still costs approximately twice as much as the steel still. Most of the cost is in the materials that are used for the reboiler (cooking pot) section of the still. If the stills were going to be scaled up to a larger size, the difference in total cost between steel and copper would likely increase due to the large difference in cost between steel and copper.

## <span id="page-17-1"></span>**1.5 Conclusions**

Overall, it was determined that there is no significant difference between a steel still, steel still with copper packing, or a copper still regarding the sulfur content of the distillate. In our testing and surveying, the ABV of the distillate was very similar between all of the stills in addition to the sulfur content.

The design of the steel still was a success. The results from our testing of the ABV and sulfur content were aligned with the testing we did on the copper still and the benchtop lab testing. All three of the stills were able to achieve over a 40% ABV solution on the second cut. Additionally, there was not a significant difference of sulfur content detected by the people who were surveyed. While we are very confident in the results from the ABV testing due to low error and low deviation in repeated tests, there still lies some uncertainty in the results from our sulfur detection testing. Originally, we were going to use gas chromatography (GC) to test for the sulfur content of our solutions. This method of testing was not feasible for two main reasons: 1. The GC machine was not properly set-up like we had planned in earlier months and would have taken too long to prepare for testing, and 2. The concentration of sulfur volatiles was in the parts-per-billion (PPB) range, which is too small for a GC machine to detect. Because we couldn't use GC, we had looked into using a sulfur detector such as potassium permanganate, but this also wasn't feasible because it wouldn't be able to detect sulfur in the PPB range. This led us to using people as our detectors because the human nose is surprisingly capable of detecting the odors from hydrogen sulfide and other volatile sulfur compounds in the PPB range. However, the subjects that were used for this testing method were not trained on sulfur detection and we had a limited number of subjects (7). If repeat testing is going to be done in the **Example the still costs** of the still is the competent and the still is the costs of the still costs (taste testers) and the still costs of the still costs (taste testers) and the still Costs Costs (taste test is the cost

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### <span id="page-19-0"></span>**References**

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# <span id="page-20-0"></span>**Appendix A Possible Abbreviations**





\*Deprecated: use gigaelectronvolt (GeV).

\*\*Preferably called simply *kelvin.*





†The name *micrometer* (µm) is preferred.

‡The name *nanometer* is preferred.

§Although the use of the abbreviation psi is common, it is not recommended. See pound-force per square inch.



