Kobe Maristela
CHEM 345
9/29/2019

Biosynthesis of Ethanol from Molasses

Reference: "EthanolExpt" Handout; Chemistry lessons: simple and fractional distillation; Green lessons: renewable feedstocks, catalysts, design for degradation; "Distillation" movie

Purpose: To distill molasses and make ethanol by using simple and fractional distillation

Table of Reagents:

Reagents	Amount	MW	BP (°C)	MP (°C)	Density
Molasses	50 mL	201.22 g/mol	106.6 °C		1.4 g/cm^3
$Ca(OH)_2$		74.09 g/mol	2850 °C	580 °C	2.21 g/cm^3
Yeast	0.5 g	274.3 g/mol	105 °C		
Water		18.02 g/mol	100 °C	0 °C	0.997 g/cm^3

Balanced Chemical Equation:

$$C_{12}H_{22}O_{11}\ (sucrose) \rightarrow \mathbf{4}\ CH_3CH_2OH + \mathbf{4}\ CO_2$$

Safety:

- Glass may crack due to high temperature
- Do not distill until dry
- Contains explosive residue (Alkenes/ethers)

Experimental Procedures	Data & Observations
Part 1: Set up and fermentation **1.** Mix 50 mL molasses with 50 mL DI water in 250 mL filter flask. Add 0.5 g yeast and stir!	
2. Add rubber stopper to filter flask. Attach rubber hose to side of filter flask and insert short straight section of glass tube to other end of rubber tube. Dip straight glass tube into test tube ~2/3 full of $Ca(OH)_2$	*Observations:* • Color of Molasses: Black
3. Store in drawer until next lab	

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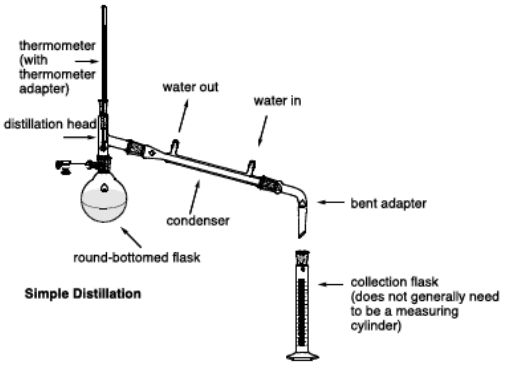
Part 2: Simple Distillation **4.** Decant ~50 mL of ethanol solution into 100 mL round bottom flask. Add boiling stone to 100 mL round-bottom flask.	
5. Assemble distillation apparatus as shown below. Add water hose and turn water on (gentle trickle). Add thermometer to apparatus and lower the flask into the mantle.  	
6. Have TA check before plugging in. Wait until thermal equilibrium (boiling point). Collect alcohol fraction until boiling is below of water (100 °C). Record initial boiling point and boiling point after each 2 mL distillation collection. *** Distillation → ~2 mL/min ***	*Observations:* • The molasses boiled over contaminating our collection • The molasses continues to boil and go up the tube
7. After collection, stop the distillation. Add stopper to flask with ethanol and put back in your drawer until next lab. Clean up and throw away the molasses to the appropriate container.	

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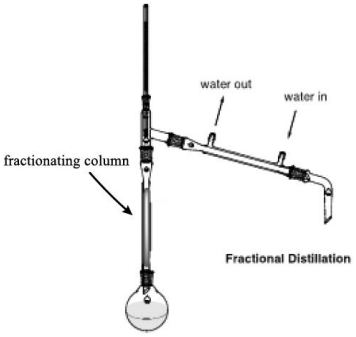
Part 3: Fractional Distillation

8. Assemble fractional distillation apparatus as shown below (simply add in the fractionating column). Have TA check before plugging in.



Observations:

- Molasses rising up the fractional tube
- Molasses slowly reaching the top of the fractional tube; turned temperature down

9. Distill mixture slowly and collect several fractions, which are identified by rapid change in temperature. Record temperature range for each fraction. Stop collection below 97 °C.

10. Stop distillation before flask is dry. Temperature can rise rapidly that will result of the flask cracking if no heat absorption is present.

11. Use volumetric flask → determine density of each fraction as well as mass and volume of each fraction.

$$density = \frac{mass}{volume}$$

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Post-lab Questions:

1.

Reaction: $C_{12}H_{22}O_{11}$ *(sucrose)* → ***4*** CH_3CH_2OH + ***4*** CO

Fractions	Volume Collected	Mass of Collection	Density	% Ethanol	% Yield of EtOH Molasses	% Yield of EtOH Sugar
1	*1 mL*	*0.856 g*	$\frac{0.8558\ g}{1\ mL}$ *0.856 g/mL*	**Work in chart** *75 %*	$\frac{0.8558\ g * 0.5}{8.02\ g} \times 100$ *5.34 %*	$\frac{0.8558\ g * 0.5}{8.42\ g} \times 100$ 5.08 %
2	*1 mL*	*0.818 g*	$\frac{0.8176\ g}{1\ mL}$ *0.818 g/mL*	**Work in chart** *90 %*	$\frac{0.8176\ g * 0.5}{8.02\ g} \times 100$ *5.1 %*	$\frac{0.8176\ g * 0.5}{8.42\ g} \times 100$ 4.86 %

- **Initial Boiling Point:** *22.1 °C*
- **Temp Ranges:**
  - *Fraction #1: 68 °C*
  - *Fraction #2: 76.5 °C*

Aqueous Alcohol (EtOH) Content							
Density g/mL	% EtOH by wt.	% EtOH by vol.	g EtOH per 100 mL	Density g/mL	% EtOH by wt.	% EtOH by vol.	g EtOH per 100 mL
0.989	5	6.27	4.95	0.856	75	81.30	64.17
0.982	10	12.44	9.82	0.843	80	85.49	67.48
0.975	15	18.54	14.63	0.831	85	89.48	70.63
0.969	20	24.54	19.37	0.828	86	90.25	71.23
0.962	25	30.46	24.04	0.826	87	91.02	71.84
0.954	30	36.25	28.61	0.823	88	91.77	72.43
0.945	35	41.90	33.07	0.821	89	92.53	73.03
0.935	40	47.40	37.41	0.818	90	93.27	73.62
0.925	45	52.72	41.61	0.815	91	93.99	74.19
0.914	50	57.89	45.69	0.813	92	94.72	74.76
0.903	55	62.89	49.64	0.810	93	95.44	75.32
0.891	60	67.74	53.47	0.807	94	96.11	75.86
0.880	65	72.43	57.17	0.804	95	96.79	76.40
0.868	70	76.95	60.74	0.789	100	100.00	78.9

Figure 1. Chart for Calculating % EtOH

2. The advantage of fractional distillation over simple distillation is that fractional distillation can purify liquids better, in a sense that it can separate substances to its respective fractions better. In addition, while the two distillations are similar to each other, the only difference between them is that the fractional distillation has a fractional column attached to it, which allows the liquid to cool down and only let vapor with a low boiling point to travel through.

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3. An azeotrope is two substances in a mixture that have a constant boiling point throughout the entire distillation process. In this experiment, 5% of the distillation should be water, which is a result of condensation with the ethanol. The other 95% should be the targeted ethanol we were supposed to make in the distillation. This ratio between water and ethanol is the result of the azeotrope mixture, which lowers the boiling point of the mixture and hinders us from getting the pure ethanol.

4. In first setting up our lab, it is critical to prevent air from entering the reaction vessel as this will result in the overoxidation of the ethanol which will turn it to acetic acid.

5. In correspondence to the "greenness" of this experiment, we did not produce any waste which is the first principle of Green Chemistry. All of our substances were able to be thrown down the sink as it was not hazardous to the environment. Furthermore, the substances that we used such as the molasses is renewable material, which corresponds to the 7^{th} Principle in Green Chemistry. Molasses, which we used to distill and gather the ethanol from is renewable in a sense that it is the byproduct of the processed sugar and not anything depletable such as fossil fuel. Finally, we did not incorporate any hazardous chemicals in our experiment that would be toxic to us or the environment, since our main ingredient was molasses.