



Dehydration of an ethanol-water mixture by sorption using barley as the sorbent
by James Patrick Law

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in
Chemical Engineering
Montana State University
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Abstract:

Experiments were conducted to determine the feasibility of utilizing barley as a dehydration agent for the separation of an ethanol-water mixture. The variables tested were mass flux, column geometry, grain particle size distribution, and the number of regenerations of the barley.

The mass flux was varied between 0.035 and 0.35 gm/min-sqcm. A vapor phase, fixed-bed adsorption apparatus was used with three different adsorption columns. The adsorption columns used were 1.5" (diameter) X 14" (length), 1.5" X 24", and 2.5" X 23". Three particle size distributions of ground barley were tested. The particle size distributions were obtained by grinding the barley in a hammer mill with different screen sizes. The screen sizes used were 1/8", 1/16", and 1/32". A total of five regeneration runs were made with the 1/32" hammer-milled barley. The initial feed concentration was 75 weight percent ethanol in all runs.

The best results were obtained with the smallest particle size distribution and the lowest mass flux. The yield varied between 0.029 and 0.191 grams 99-wt% ethanol produced per gram dried barley. The column geometry had an effect at the low mass fluxes, but had no effect at the high mass fluxes. Effects of column diameter were larger than the effects of column length on the yield. Grain regeneration is necessary if the barley is to be used to produce fermentation ethanol. Ethanol along with water was adsorbed into the barley.

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of a thesis submitted by

James Patrick Law

This thesis has been read by each member of the thesis committee and has been found to be satisfactory regarding content, English usage, format, citations, bibliographic style, and consistency, and is ready for admission to the College of Graduate Studies.

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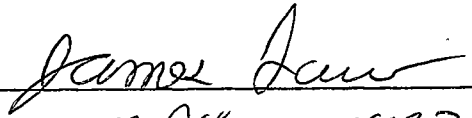
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ABSTRACT

Experiments were conducted to determine the feasibility of utilizing barley as a dehydration agent for the separation of an ethanol-water mixture. The variables tested were mass flux, column geometry, grain particle size distribution, and the number of regenerations of the barley.

The mass flux was varied between 0.035 and 0.35 gm/min-sqcm. A vapor phase, fixed-bed adsorption apparatus was used with three different adsorption columns. The adsorption columns used were 1.5" (diameter) \times 14" (length), 1.5" \times 24", and 2.5" \times 23". Three particle size distributions of ground barley were tested. The particle size distributions were obtained by grinding the barley in a hammer mill with different screen sizes. The screen sizes used were 1/8", 1/16", and 1/32". A total of five regeneration runs were made with the 1/32" hammer-milled barley. The initial feed concentration was 75 weight percent ethanol in all runs.

The best results were obtained with the smallest particle size distribution and the lowest mass flux. The yield varied between 0.029 and 0.191 grams 99-wt% ethanol produced per gram dried barley. The column geometry had an effect at the low mass fluxes, but had no effect at the high mass fluxes. Effects of column diameter were larger than the effects of column length on the yield. Grain regeneration is necessary if the barley is to be used to produce fermentation ethanol. Ethanol along with water was adsorbed into the barley.

INTRODUCTION

Since the 1974 Arab oil embargo and the ever increasing tensions in the Middle East, the United States' need for alternate energy sources has grown tremendously. Ethanol has long been identified as a possible candidate for the extension of petroleum-based transportation fuels. Ethanol fuels could play an important role in the transition period between today's petroleum fuels and the synthetic fuels of the future.

The idea of using ethanol to supplement petroleum reserves is by no means new. Brazil has been testing different blends of gasoline and ethanol, which is called gasohol, since the 1920s [1]. In 1975 Brazil launched a program called Proa'lcool, whose purpose was to decrease Brazil's dependence on foreign oil. Under this program the average blending level of gasoline is 20-vol% (volume percent) ethanol. In addition to the increased use of gasohol, there has been an increase in the number of pure ethanol driven vehicles [2].

Gasohol with up to 20-vol% ethanol can be used without any engine modification. Researchers say the optimum blend is 10-15-vol% ethanol [3].

A major advantage of mixing ethanol with gasoline is that ethanol is an octane booster. Pure ethanol has a research octane number (RON) of 106-108 [4]. In general, ethanol will increase the octane number of gasoline by 0.5 units for every 1-vol% addition of ethanol [5]. Several oil companies are currently marketing super unleaded gasoline, which is a mixture containing 90-vol% unleaded gasoline and 10-vol% ethanol.

Ethanol can be produced by the fermentation of grains. The starch in the grain is converted into glucose by the addition of an enzyme. A specific yeast is then added to convert the glucose into ethanol and carbon dioxide. This is carried out until the ethanol concen-

tration reaches approximately 10-vol%. At that point, the ethanol is in sufficient concentration to kill the yeast [6].

Ethanol which is to be used for mixing with gasoline must be nearly anhydrous. A 20-vol% ethanol-gasoline mixture will tolerate only 1-vol% water at 70 F [7]. Simple distillation cannot be used to produce anhydrous ethanol because there is a minimum boiling point azeotrope at 94.4-wt% (weight percent) ethanol. There are, however, several methods available to produce anhydrous ethanol; the most common being azeotropic distillation. This involves using a third compound, such as benzene, to break up the minimum boiling azeotrope. There are, however, new methods being investigated that require less energy. Several of these methods are shown in Table 1, along with the approximate energy required to produce 1 liter of anhydrous ethanol [8].

Table 1. Common Ethanol-Water Separation Processes and the Approximate Energy Required to Produce One Liter of 100% Ethanol from a 10-vol% Ethanol Mixture.

Separation process	Energy requirements (KJ/l)
Vacuum distillation	7,600
Azeotropic distillation	7,400
Distillation to 95-wt%, and molecular sieve	6,100-7,600
Distillation to 95-wt% and low temperature blending with gasoline	5,860*
Carbon dioxide extraction	2,500**
Distillation to 95-wt%, and dehydration with calcium oxide	2,170
Distillation to 75-wt%, and dehydration with starches	< 2,000
Solvent extraction	1,000**

*Results in the direct production of gasohol.

**Requires a high capital investment.

This project looks at using the starch present in barley to remove the water from an aqueous ethanol mixture. Barley was chosen as a desiccant for several reasons; first, past research at Montana State University has shown great promise for the use of barley in

ethanol water separation [9]. Barley can also be used as a feedstock to produce fermentation ethanol. Also, barley is an abundant cash crop in Montana and several other states.

Dehydration is not a new method for separating ethanol and water. Calcium oxide (CaO) has long been used as a desiccant in the dehydration of many gases and vapors, including ethanol and water mixtures [10]. The main disadvantage of using CaO as a desiccant is that it reacts with the water to form a hydroxide. This makes the regeneration of the solid very energy expensive. It has been known for many years that starches, such as those found in barley, could be used as a desiccant [11]. However, it was just recently found that starches would preferentially adsorb water vapor over ethanol vapor [12].

Sorption

There are three types of sorption:

1. Absorption
2. Physical Adsorption
3. Chemical Adsorption

First, adsorption should be distinguished from absorption. Absorption is the bulk penetration of a vapor or gas into the structure of the solid, thus forming a solid solution. Adsorption is the phenomenon where molecules of a gas or vapor will stick to the surface of the solid, but not form a solid solution. The term sorption is used when both processes may occur simultaneously.

Physical adsorption should be distinguished from chemical adsorption, sometimes called chemisorption. Physical adsorption is similar to condensation of a vapor. The gas or vapor is held to the solid surface by van der Waals forces. Chemical adsorption involves the formation of a chemical bond between the solid and the adsorbed liquid.

There are several important differences between physical and chemical adsorption listed below [13]:

1. Physical adsorption will occur on any gas-solid system, provided that the temperature and pressure are suitable. Chemisorption will occur only when the gas and solid are able to form a chemical bond.
2. The heat of physical adsorption is on the order of the heat of vaporization for a vapor and the heat of liquefaction for a gas. The heat of chemisorption is on the order of the heat of reaction.
3. Physical adsorption is easily reversible by simply reducing the pressure or increasing the temperature. Chemisorption is much less reversible and requires much more rigorous conditions.
4. Several adsorbed layers can be built up with physical adsorption, but only one layer will occur with chemisorption.
5. Chemisorption will occur at much lower pressures and much higher temperatures than physical adsorption.
6. Physical adsorption occurs almost instantaneously, whereas chemisorption may occur instantaneously or might require an activation energy.

The adsorption of water onto biological materials is almost always physical adsorption [14].

Theories of Physical Adsorption

For a given weight of adsorbent the amount of gas or vapor adsorbed at equilibrium is a function of temperature and pressure only. Adsorption data is generally given in terms of isotherms. An isotherm is a plot of the amount of gas or vapor adsorbed versus pressure at a constant temperature. There are five types of isotherms for the adsorption of gases or vapors on solids. These are shown in Figure 1. Type I isotherms are associated with systems

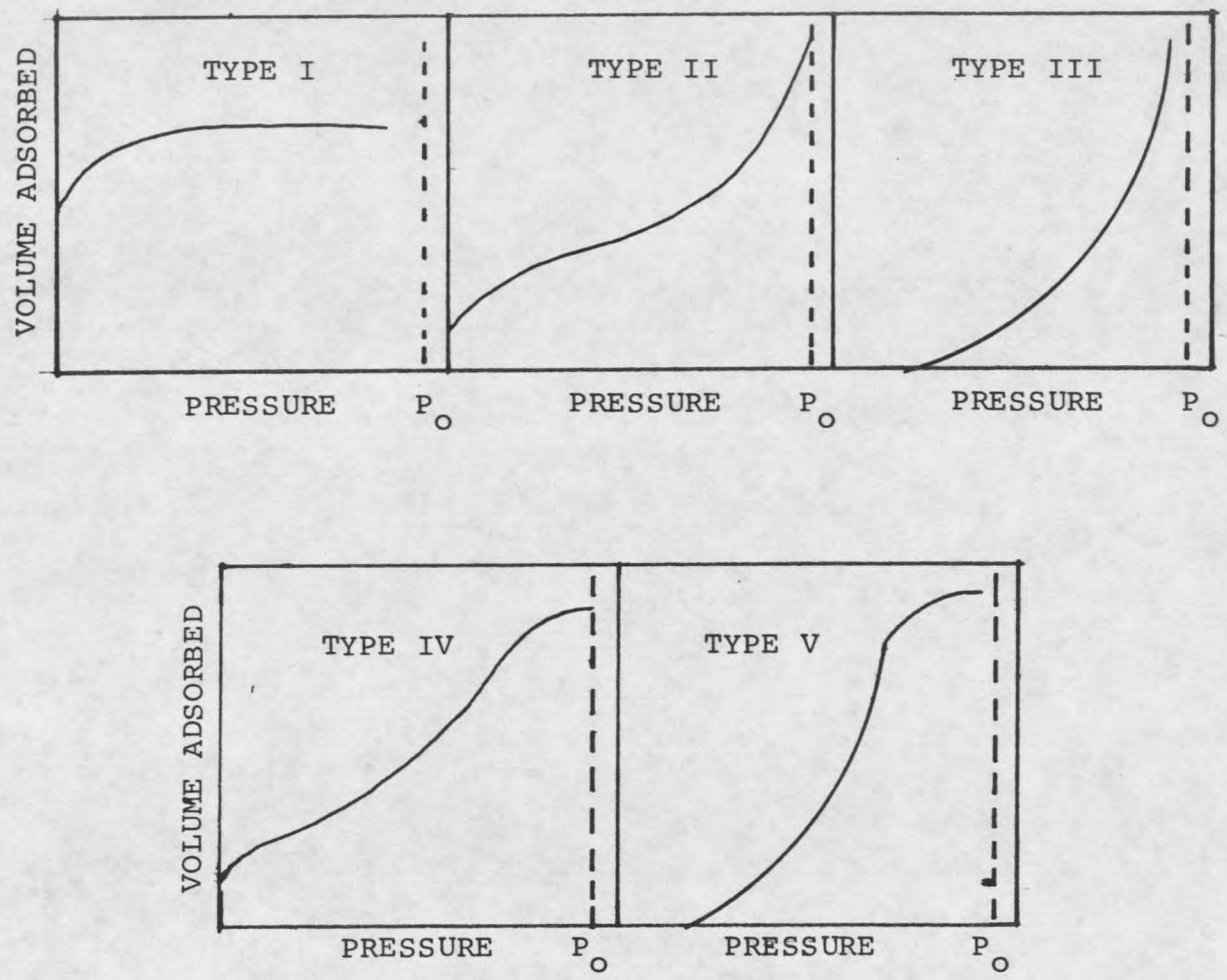


Figure 1. The five general types of adsorption isotherms. P_0 is the saturation pressure.

where adsorption is monomolecular. Types II and III isotherms are associated with systems where multilayer adsorption occurs. Types IV and V isotherms are associated with systems where a highly porous adsorbent is used [15]. The isotherms for biological materials are most similar to the type II isotherm [16].

Nygoddy and Bakker-Arkema [17] developed an isotherm equation for the sorption of water by starches. They obtained their equation by combining the three following adsorption theories:

1. Brunauer, Emmett, and Teller's (BET) theory of multilayer adsorption.
2. Zsigmond's capillary condensation theory.
3. Polyany's adsorption potential theory.

The most used theory of adsorption is the BET theory of multilayer adsorption. The BET equation appears to represent the shape of actual isotherms fairly well. It is also used to find the surface area of a substance because it yields a good value for the volume of gas required to form a monomolecular layer on the surface of the substance. This theory says that adsorption will occur on one layer until it is completely full, then adsorption will continue by building more and more layers [18].

Zsigmond's capillary condensation theory says that the adsorbate exists as a condensed liquid in the capillaries of the sorbent. The adsorbate's properties are the same as the bulk liquid phase [19].

Polyany's adsorption potential theory says the adsorbent exerts strong attractive forces. These forces attract gas molecules in the vicinity and as a result many layers can build up [20].

Physical adsorption of a gas or vapor on a solid is a spontaneous process, which causes a decrease in the Gibb's free energy of the system. The process also involves a decrease in entropy when the gas or vapor goes from the free gas to the adsorbed film. It follows from the equation,

$$\Delta G = \Delta H - T\Delta S$$

that physical adsorption must always be an exothermic process.

Desorption is the process by which the adsorbate is removed from the solid. Biological materials show a hysteresis effect present between the adsorption and desorption isotherms. During desorption more water is contained in the solid at a given pressure than during adsorption. Young and Nelson [21] tried to explain this phenomenon by assuming there are three types of water sorption in biological materials. First, there is a layer of water adsorbed directly on the outer surface of the cell. Then, there are several layers of condensed molecules that adhere to the adsorbed layer. There are also molecules that pass through the cell wall and become adsorbed into the interior of the cell. It was proposed that two or more adsorbed layers are required to push the adsorbed molecules into the interior of the cell. During desorption the water bound to the surface would have to be removed before the water in the cell. This would mean that at a given pressure more water would be present in the cell during desorption than during adsorption.

Physical Adsorption in Packed Beds

The unsteady-state adsorption of gases in packed beds is very common in industry. The main reason for this is the ease of operation and low cost of the adsorption systems. Adsorption is used in many recovery and purification operations such as: decolorizing mineral and vegetable oils, purifying air, dehydration of gases and vapors, and concentrating valuable solutes from liquid solutions [22].

In a packed bed where there is a vapor and strongly adsorbed solute, such as ethanol and water, the vapor enters the packed bed and at first the solid adsorbs the solute rapidly and effectively. The effluent from the bottom of the packed bed is practically solute free (see Fig. 2). The distribution of the adsorbate in the solid bed is indicated by the horizontal lines in Figure 2. The bulk of the adsorption takes place over a small adsorption zone,

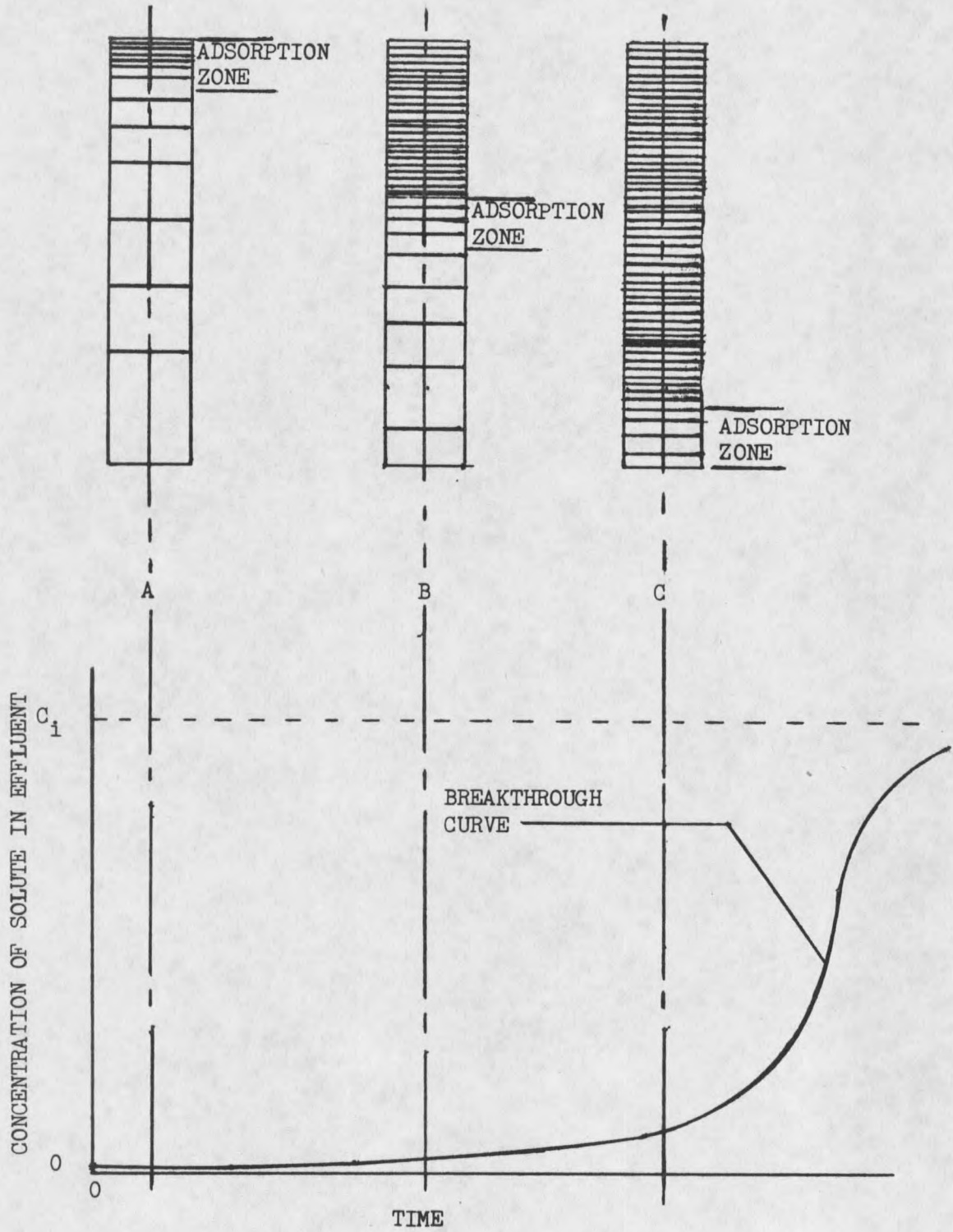


Figure 2. The adsorption wave and breakthrough curve.

which moves down the column like a wave. The speed of the wave is usually much slower than the linear velocity of the vapor in the column. As the adsorption zone gets closer to the bottom of the column, the concentration of the solute in the effluent starts to rise very rapidly. This is called the break point. Then the concentration of the solute in the effluent rises until it reaches the inlet concentrations. The portion of the curve after point C on Figure 2 is called the breakthrough curve [23].

If the process is highly exothermic a temperature wave will follow the adsorption wave. A rise in temperature of the effluent can be used as an indication of the start of the breakthrough curve [24].

Previous Research

There has been some previous work done with the separation of ethanol and water at Montana State University. Graham [25] did work with a 0.75" x 12" adsorption column and 1/16" hammer-milled Shabet barley. He obtained a correlation for yield as a function of initial temperature and mass flux. The correlation he obtained is shown below:

$$Y = 0.0229 / ((T-80)^{0.5209} F^{0.6436})$$

Y = Yield (milliliters 99-wt% ethanol produced per gram dried barley)

F = Mass flux (gm/min-sqcm)

T = Temperature (C)

He also determined that the initial feed concentration had little effect on the yield for feed concentrations between 75- and 85-wt% ethanol. Runs using specific barley particle sizes showed a decrease in yield for sizes larger than 0.83 millimeters.

RESEARCH OBJECTIVE

The purpose of this investigation was to determine the feasibility of using barley as a dehydration agent in the separation of an ethanol-water mixture. The following parameters were considered important in this investigation:

1. Feed space velocity
2. Barley particle size distribution
3. Adsorption column geometry
4. Number of regenerations
5. Initial barley temperature
6. Pressure
7. Initial feed concentration
8. Initial grain moisture content

Parameters 1-4 were varied while parameters 5-8 were held constant at the following values:

Initial grain moisture content	0%
Initial barley temperature	84-86 C
Feed concentration	75-wt% ethanol
Pressure	atmospheric (640 mmHg)

MATERIALS, APPARATUS, AND PROCEDURE

Materials

All the grain used in this project was Shabet barley. The barley was first separated into three parts, then each part was ground in a Bell Model 10 hammer mill with a different size screen. The three screen sizes used were 1/32", 1/16", and 1/8", thus giving three different particle size distributions. A comparison of the three particle size distributions obtained can be seen in Table 2. After grinding, all the grain was stored in sacks and kept in the refrigerator at 10 C.

The particle size distributions were found using screens, which varied in size from 16 mesh to 250 mesh. For a list of screen sizes used, see Table 2. The screens were stacked in order of decreasing mesh and then the sample, usually 115 grams, was placed on the top screen. A Ro-Tap was then used to shake and tap the screens. Two different samples were Ro-Tapped for each particle size distribution. The results that are shown in Table 2 are the average of the two runs. All grain was ground and sized when the moisture content was 7%.

All the ethanol used was anhydrous, and it was mixed with distilled water to form a 75-wt% mixture.

Apparatus

The equipment used consisted of five major parts:

1. Feed burette
2. Pump
3. Evaporator
4. Adsorber

Table 2. The Particle Size Distributions of the 1/8", 1/16", and 1/32" Hammer-Milled Barley.

Screen number	Screen size U.S. mesh	Screen opening millimeters	Percent of total sample		
			1/8" Hammer-milled barley	1/16" Hammer-milled barley	1/32" Hammer-milled barley
1	10	2.0	3.25	1.7	0.0
2	18	1.0	38.75	36.4	8.2
3	30	0.589	10.4	9.2	14.2
4	40	0.417	24.7	19.7	24.7
5	50	0.297	4.9	8.2	13.9
6	60	0.246	4.5	4.4	7.0
7	70	0.210	1.6	1.4	2.0
8	100	0.147	5.2	5.4	7.8
9	140	0.106	2.4	3.0	4.0
10	200	0.075	3.3	3.1	13.3
11	270	0.053	3.3	3.2	3.0
12	bottom	—	1.7	1.1	1.9

5. Condenser

A flow diagram of the complete apparatus is shown in Figure 3.

A 50 milliliter glass feed burette was used for runs 1-25; it was accurate to 0.1 ml. A 100 milliliter glass feed burette, accurate to 0.2 ml, was used starting with run 26. The reason for changing burettes was due to the volume of feed required.

A four channel, low pressure, peristaltic pump was used with 3/32" tygon tubing. The maximum output of the pump was approximately 4 ml/min for each channel. The higher flow rates were obtained by connecting two or more channels in parallel.

The aqueous ethanol was transported from the feed burette to the pump and from the pump to the evaporator via 3/32" I.D. plastic tubing.

The evaporator, which would vaporize the aqueous ethanol, consisted of one inch pipe filled with copper-clad BB's. The entire evaporator was wrapped with heating tape and was insulated. It was placed vertically so the aqueous ethanol would enter the top and drip down through the hot BB's. The evaporator was packed with BB's in order to supply a high surface area so complete vaporization could take place. A thermocouple was placed right below the output of the evaporator so the temperature of the exiting vapor could be monitored. The output of the heating tape for the evaporator and the other equipment was controlled by a Powerstat variable transformer.

After the vapor left the evaporator, it passed through a 12 inch section of 1/4" I.D. copper tubing. This tubing was wrapped with heating tape and insulated. The purpose of this section was to insure complete vaporization of the aqueous ethanol. When the higher flow rates were used (4.5 ml/min and above), the evaporator was not able to vaporize all the incoming aqueous ethanol. In those cases, the remainder of the aqueous ethanol was vaporized in this section of tubing. Complete vaporization was insured by monitoring the temperature at the end of this section of tubing.

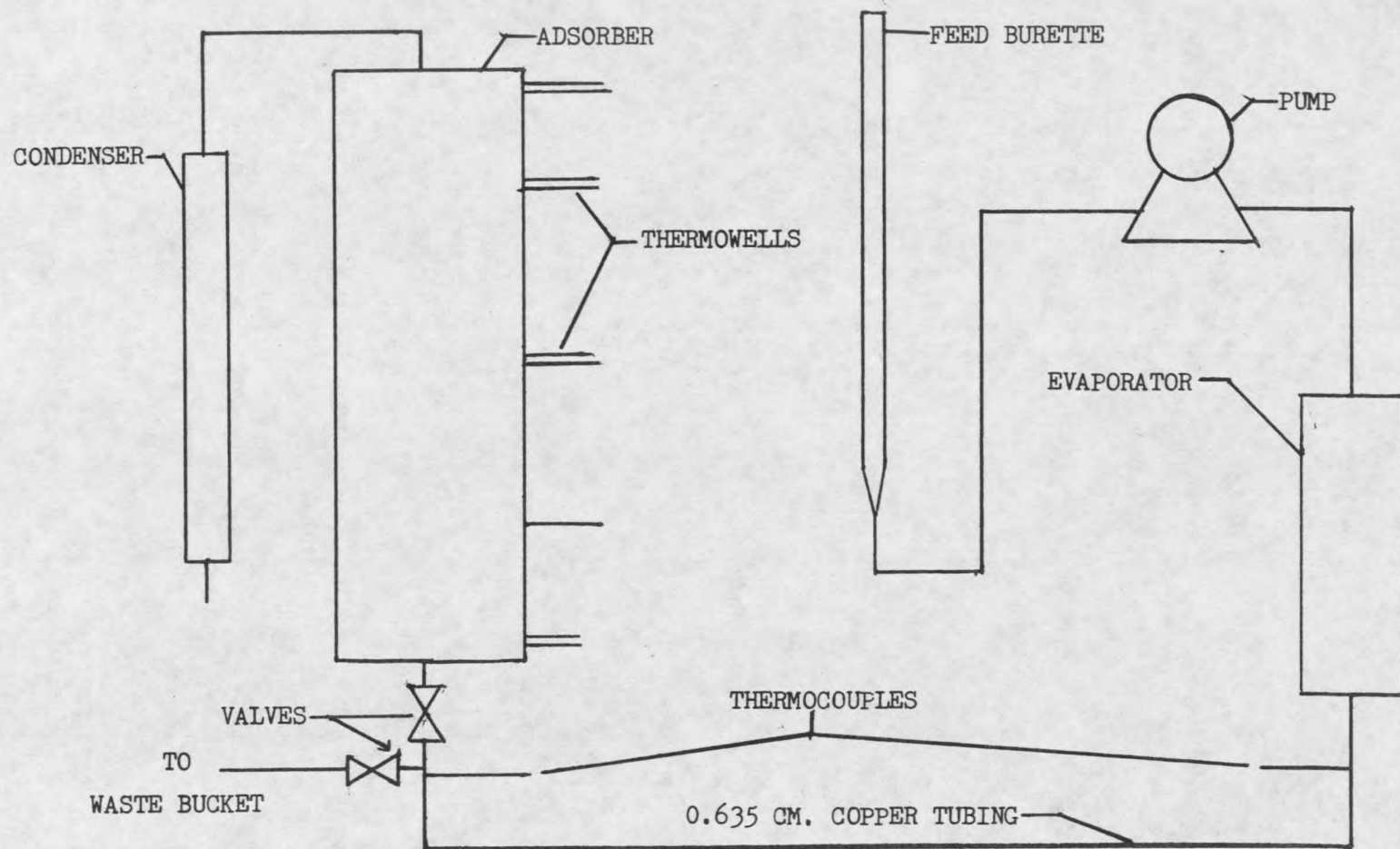


Figure 3. Flow diagram for the barley adsorption process.

There was a series of valves at the end of the 12 inch section of tubing. During pre-heat, the valve leading to the waste bucket was opened and the vapor was condensed and discarded. When the run was started, that valve was closed and the valve leading to the adsorption column was opened.

Four different adsorption columns were used. Runs 1-32 used a 1.5" (diameter) × 14" (length) copper column; runs 33-42 used a 1.5" × 24" copper column; runs 43-60 used a 1.5" × 24" black iron column; and runs 60-75 used a 2.5" × 23" black iron column. All regeneration runs, numbers 57, 62, 64, 65, and 75 used the 1.5" × 24" black iron column. The reason for switching from the copper column to the black iron column on run 43 was because leaks were developing at the entrance and exit of the copper column.

The purpose of the adsorber was to hold the grain in a fixed position. Heating tape was wrapped around the adsorber and the entire adsorber was insulated. One piece of heating tape was used with the 1.5" × 14" column. Two separately-controlled pieces of heating tape were used on the other columns. There were thermowells at the entrance, exit, and every 3-4 inches along the adsorption column. Thermowells were placed so the ends were at the center of the adsorption column. One thermowell was placed at the edge of the column about halfway up. The thermowells consisted of 3/16" copper tubing with the ends soldered shut. Thermocouples were placed inside the thermowells as far as they would go.

After the vapor left the adsorber, it entered a water-cooled condenser where the samples were collected in test tubes. The vapor was kept hot until it entered the condenser. This was done in order to prevent the vapor from condensing and dripping back into the adsorption column.

All the thermocouples used were chromel-alumel. A Cole-Palmer digital thermometer that was accurate to 1 C was used to record temperatures. The thermocouples used to measure the temperature of the vapor leaving the evaporator and the vapor entering the adsorber were held in place with a Swagelok fitting filled with epoxy.

Procedure

Before the run could begin, the barley had to be dried to 0% moisture. This was accomplished by placing the grain in large aluminum pans with the grain 0.5-0.7 centimeters deep. The pans were placed in one of two ovens that were kept at 110 C. The samples were allowed to dry for 72-96 hours. It was found that this was sufficient to completely dry the barley.

The pump was then set up depending on the flow rate required. The maximum output of the pump was a 4 ml/min for each channel. To keep a constant flow rate during the run, 3/32" tygon tubing was used and changed often.

The adsorption column was then blown out with compressed air in order to insure that all the old grain was removed. Teflon tape was wrapped around the threads at both ends of the column. The bottom screen was then placed in the column and the cap was put on. A preweighed sample of barley (230 grams for the 1.5" X 14" column, 400 grams for the 1.5 X 24" column, and 900 grams for the 2.5" X 23" column) was added. The barley came to within 0.5-1.5 inches of the top of the column. The top screen was fitted into place and the top cap screwed on. Both the top and bottom caps were then tightened in a pipe vise to prevent leaks. The adsorption column was wrapped with heating tape and an insulation jacket was placed around the entire adsorber. The adsorber was fitted into place and the thermocouples were placed in the thermowells.

The Powerstats were turned on and the system was allowed to heat up. The valve to the adsorber was closed, and the valve to the waste bucket was opened.

When the temperature of the evaporator reached 80-83 C the pump was turned on and adjusted to the correct flow rate. The Powerstats controlling the evaporator were set so the output of the evaporator was 84-92 C. The temperature of the vapor entering the adsorber was kept at 100-110 C. When the high flow rates were used (above 4.5 ml/min),

the evaporator could not vaporize all the incoming feed. In those cases the power to the heating tape on the 12 inch section of tubing was turned up. This would allow complete vaporization to occur before the feed entered the adsorber.

The barley inside the adsorber was heated until a uniform 84-86 C was obtained. Then the vapor was routed up the adsorber and the run was started. At the start the level of the burette was recorded along with all the temperatures.

When the 1.5" x 14" column was being used, the power to the heating tape was turned off when the vapor was routed up the adsorption column. Due to the heat of adsorption, the temperature of the adsorber would rise to 90-95 C. The exception to this was the slowest space velocity (0.035 gm/min sqcm). In this case a small amount of heat had to be added to maintain the temperature above 84 C. When the other two columns were being used, the power to the bottom section of heating tape would be shut off at the beginning of the run. The upper section of heating tape was kept on so the outlet temperature would not drop below 84 C. Again, the only exception to this was the slowest space velocity where some heat addition was required from the lower section of heating tape.

Temperatures were recorded at various time intervals depending on the space velocity. Generally, temperatures were recorded after every 20-30 milliliters of feed additions. Sample tubes were changed at various times depending on run conditions. At the beginning of the runs, samples were changed every 10-15 grams. As the ethanol concentration started to drop, 3-4 gram samples were taken. A good indication of when the ethanol concentration would start dropping was when the exit temperature started to rise.

The run was stopped when the barley was saturated. The Powerstats were turned off, and the last sample was allowed to set for 10-15 minutes to collect the last bit of output.

The sample tubes were then weighed, and the samples were transferred to smaller sample bottles for analysis. A Beckman gas chromatograph with a Poropack-R packed column operated at 190 C was used to find the ethanol concentration. A calibration curve

of weight percent versus area percent was obtained by making up samples of known weight percents.

The column was allowed to cool for several hours. Then three samples of barley were taken, one from the top, one from the middle, and one from the bottom. These samples were dried in order to find the moisture content. The overall grain moisture content was taken as the average of the three individual moisture contents.

RESULTS AND DISCUSSION

A total of 75 runs were made and of that number, 54 were used in my analysis. The results for all the useful runs are shown in Appendix A along with a list of all rejected runs and the reason for their rejection. The purpose of the runs was to determine the effects of:

- Particle size distribution
- Space velocity
- Column geometry
- Multiple grain regenerations

Before the above points are discussed, a few general points should be mentioned.

As explained in the procedure section, the moisture content of the barley was taken as the average of the three small samples taken from the adsorption column. This method worked well for the 1.5" × 14" and the 1.5" × 24" adsorption columns. All the grain from two columns was dried and the actual moisture contents were 3-5% higher than the estimated moisture contents. The method did not work as well for the 2.5" × 24" adsorption column. All the grain from one column was dried and the actual moisture content was 13% lower than the estimated moisture content. This is the reason for the higher percentages (above 100%) of the amount of feed accounted for when the 2.5" × 23" adsorption column was being used.

The results in Appendix A show that some ethanol was adsorbed by the barley. The amount ranged from 12-35% of the ethanol in the feed. These figures were only estimates due to the method of calculating barley moisture content. That is the probable cause for the large range of values obtained. No trend could be found for the differences as a function of space velocity, particle size distribution, or adsorber geometry.

A typical breakthrough curve is shown in Figure 4. The concentration of water in the effluent was small for 5 minutes. Then the concentration rose very rapidly and leveled out at about 5-6-wt% below the inlet concentration. This phenomenon was most likely due to the fact that as the grain became saturated many adsorbed layers were built up. As stated in the Introduction, Young and Nelson [26] proposed that once two layers were built up, absorption of the water into the interior of the barley cells occurs. This would suggest that as the barley became saturated it would start to absorb water molecules making more sites available for adsorption. This process would occur at a much slower rate than the initial adsorption rate, thus removing only 5-6-wt% of the water in the feed. This would make sense, since initially barley cells contain some water that was removed when the barley was dried. Graham [27] obtained the same results with his experiments.

As stated before, the adsorption process releases heat causing the barley temperature to rise. The faster the adsorption takes place the higher the barley temperature rises. Figure 5 shows the maximum temperature of the barley versus mass flux for the 1/32" hammer-milled barley. This is typical of the other particle size distributions. The temperatures shown on Figure 5 are the average maximum temperature the barley attained at the center of the adsorption column. The temperature of the barley increased as the size of the adsorber increased, due to the fact that less heat could escape in the larger columns. The temperature of the barley would rise as the adsorption wave, as discussed in the Introduction, would move up the column. The fastest temperature rise observed was with the 1/32" hammer-milled barley, a mass flux of 0.212 gm/min-sqcm, and the 2.5" x 23" column. A temperature rise of 11 C was observed in 45 seconds. It should be noted that the thermocouples were placed in thermowells, which would tend to slow down their response. In most cases the temperature would increase 3-7 C in 3-4 minutes.

There were some radial temperature gradients in all the columns. These gradients would occur just after the adsorption wave would pass a point. The 1.5" adsorption col-

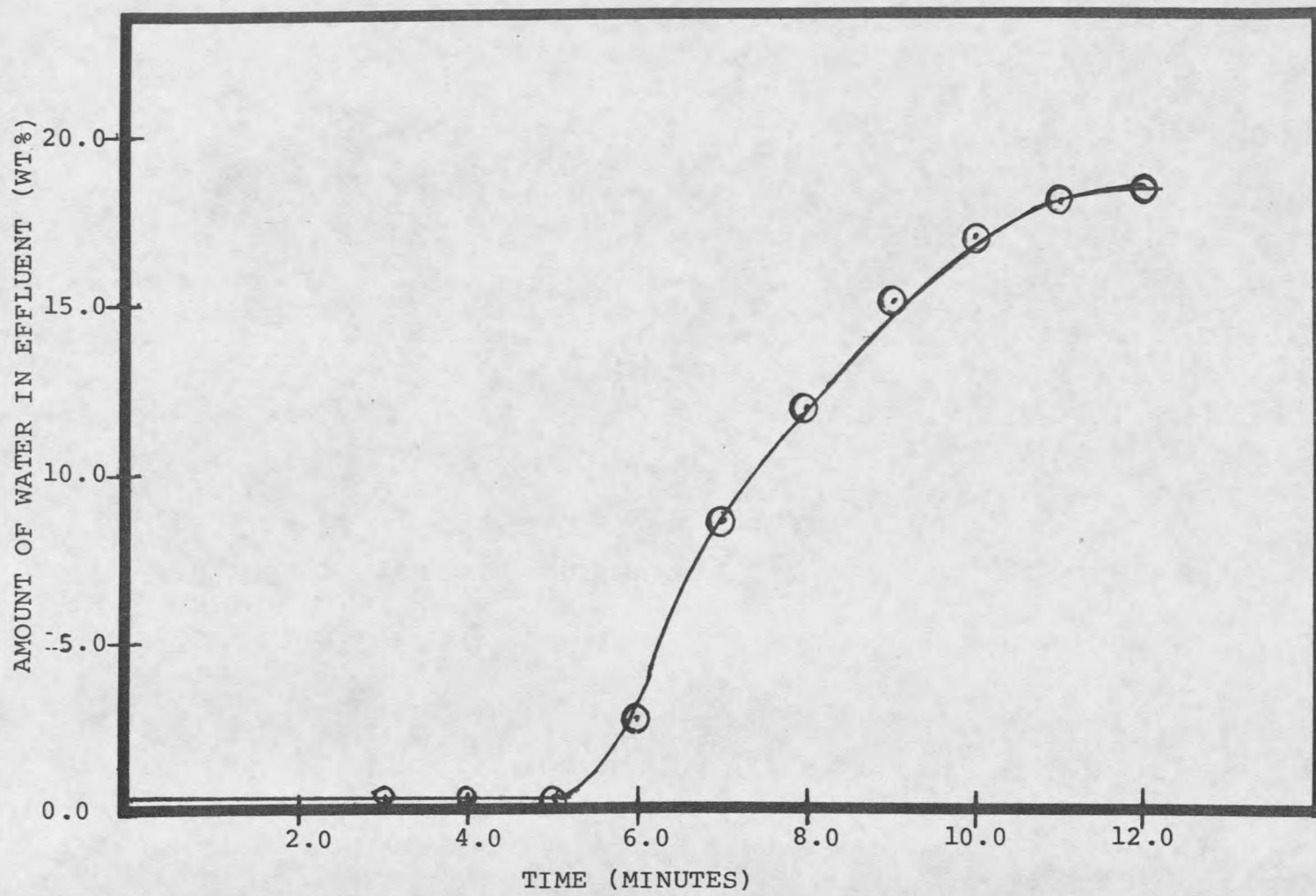


Figure 4. Breakthrough curve for Run 20.

