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Chapter 8

PROCESSING STEPS SPECIFIC TO CELLULOSE MATERIALS

CELLULOSE CONVERSION

Cellulose feedstocks, which include a wide variety of material from corn stalks, wood, straw, and cotton, to old newspapers (paper) and trash, are potentially good sources of alcohol. If fully converted, for example, a ton of old newspapers would yield up to 70 gallons of alcohol. Cellulose materials are also extremely cheap and, often, free.

Cellulose is converted by either enzymes or acid hydrolysis. Nova Laboratories produces special enzymes called "Cellulast" and "Cellobiase 250L" for conversion of cellulose to fermentable glucose. Other manufacturers make similar products. The acid process involves either strong acid and relatively low temperatures, or weak acid and high temperatures. The strong acid process has the problem that the glucose is destroyed almost as fast as it is formed unless the contact time with the acid is very brief. The weak process requires acid proof pressure cooking equipment as described earlier. Again, for the obvious reasons, these methods are not recommended on a small scale.

The main problem with cellulose as an ethanol feedstock is getting at the cellulose itself. In a plant, cellulose is encased in a substance called "lignin" in much the same way that a steel reinforcing rod is encased in concrete. Lignin is the substance that gives wood its strength. To get at the cellulose, the lignin must be dissolved away. The paper industry uses substances like sulfur dioxide, calcium bisulfite, sodium sulfate, sodium sulfide, and sodium hydroxide (lye) to dissolve lignin. Concentrated mineral acid, mentioned earlier, also dissolves lignin. Unfortunately, as it is dissolving the lignin, the strong acid also converts and then destroys the glucose.

Commercial processes are being developed to process cellulose into alcohol with the use of strong acid without destroying the cellulose. However, the process is complicated and economically feasible only on a very large scale.

The only alternative to dissolving the lignin is to reduce the cellulose material to as fine a state as possible so that at least some of the cellulose may be recovered. This is done by powdering,

grinding or pressing. The yield of cellulose is directly proportional to how finely the starting material is reduced.

Other cellulose materials are somewhat easier to process than those with high lignin content. Some forms of paper, like newspaper, are almost pure cellulose and are easily converted by either the enzyme or acid process.

Also, in order for a plant to produce cellulose, it must first produce glucose, which is the sugar we are trying to obtain. Therefore, plants that are processed while they are still wet and green have the advantage of having fermentable sugar already available. These materials can be simply fermented without conversion and considered as low-yield saccharine feedstocks.

Chapter 9

YEAST AND FERMENTATION

YEAST

Yeast is an organism belonging to the vegetable family. The yeast itself does not take a direct part in the fermentation process, but it secretes a complex of enzymes that act upon the sugar and convert it to alcohol and carbon dioxide gas.

The yeast used in alcoholic fermentation is a special strain bred to be tolerant to variations in pH and resistant to alcohol. In the past, distilleries bred and propagated their own yeast strains. The yeast was kept alive in cultures and grown in batches of ever-increasing size to be used in the fermenters. Keeping yeast alive and growing cultures is a tricky business that requires precise control of temperature, nutrients, and the like. However, a simplified method is described later. Fortunately, special active dry yeast is available. To use it, you merely add warm water to reactivate it and then add it to the mash in the fermenter. Two pounds is sufficient for 1000 gallons of mash. It is available from Universal Foods Corporation as listed in the appendix. This yeast should be rehydrated for 15 minutes prior to use at a temperature of 100-105 deg Fahrenheit, or it can be added dry to the fermentation tank prior to filling.

In a pinch, it is possible to use ordinary baker's yeast from your grocer's shelf. However, this yeast is not bred for alcohol tolerance, and you will probably not get the yields associated with the distiller's yeast.

YEAST PROPAGATION

It is possible to grow and propagate your own yeast cultures if you observe certain precautions. Above all, the conditions must be absolutely sterile. Ordinary boiling water does not kill all of the bacteria present. It is necessary to use a pressure cooker. Make a solution of (proportionately) one cup sugar, one cup flour and two quarts water. Place the solution in a pressure cooker and boil at elevated pressure for at least 45 minutes. Without opening the pressure cooker, cool the solution to about room temperature. Then open the container and add a cake of baker's or distiller's yeast. Close the container and keep it in the refrigerator. The yeast will slowly grow. Some carbon dioxide will be given off, so be sure to leave the vent open. If desired, the yeast slurry can be transferred to jars. Just be sure they are sterile and remember to poke a small hole in the lid to let the carbon dioxide escape.

To use the yeast culture, merely remove a teaspoon or so, place it in another (sterile) container, feed it some sugar and warm it to room temperature. When it becomes active, it is ready for the fermenter. If at any time your refrigerated culture goes bad (due to bacterial contamination) it must be thrown out and the procedure started again. Also, yeast cultures should not be frozen.

FERMENTATION

All that is necessary to begin fermentation is to mix the activated yeast and the cooled, pHadjusted mash in the fermentation tank. Aside from the considerations of pH as discussed earlier, the most important thing during the fermentation is temperature control. When the fermentation begins, carbon dioxide gas will be given off. At the height of fermentation, the mash will literally "boil" from the carbon dioxide produced. The reaction also produces some heat. The optimum temperature for the fermentation process is between 70-85 deg F., and it is desirable not to let the temperature go much above 90-95 deg F. Cooling is readily done with the use of ice bags, as discussed earlier, or by the use of a cooling coil. A less desirable method of controlling temperature is to dilute the mash.

The actual time required to ferment a mash varies with the material being fermented, the pH, temperature, and several other factors. It can take from one to four days. You will know that the fermentation is complete when the mash ceases bubbling and the yeast cake, which forms on top, sinks to the bottom. At this point, the fermented liquor is known as "beer" and it is ready to be distilled.

It is advantageous to distill the beer as soon as possible. Occasionally, if it is allowed to sit, it will turn to vinegar. Vinegar is alcohol that has been oxidized to acetic acid. Certain enzymes present after fermentation act as catalysts and allow any air present in the mash solution to react with the alcohol to form acetic acid. In fact, if you want to produce vinegar, all you have to do to start the reaction is to bubble air through the fermented mash. Once the vinegar reaction has set in, the mash is lost. There is no cure. The only prevention is to separate the beer from the mash sediment and distill it as soon after fermentation is complete as possible.

It is also advantageous to use a fermentation lock as described in Chapter 13, to prevent alcohol vapors from escaping the fermenter. Otherwise, the CO2 gas can carry with it a considerable amount of alcohol. Note that the small, glass fermentation locks available from wine-making supply houses are suitable, at most) for a 5-gallon container. Larger containers must have proportionately larger fermentation locks or a dangerous amount of pressure will build and the vessel could explode.

It is permissible to open the fermenter to check progress and take samples for pH analysis, etc. as long as care is taken not to introduce bacteria that could contaminate the mash.

FERMENTATION BY-PRODUCTS

The principle products of fermentation are alcohol, carbon dioxide, and fermentation residue. The alcohol is distilled from the beer and used as fuel . The carbon dioxide gas in large distilleries is usually compressed or made into dry ice. Another use for the gas would be to pipe it into a greenhouse. The plants will then use it in the photosynthesis cycle, removing the carbon and giving off oxygen. Lacking a use for the carbon dioxide, it can be simply vented into the air as it is totally non-polluting and non-toxic.

What will be left is a lot of water and solids. A portion of the water can be used for backslopping. The remaining solids contain proteins, vitamins, minerals, fats, and yeast cells. All of the nutrition value of the original feedstock, except the starch or sugar that has been turned into alcohol, survives intact. It may be fed to cattle, or if suitably processed it can be used for human consumption. However, in the wet state, it will keep for a maximum of 3-5 days depending on conditions. After this it will begin to rot. Therefore, for long term storage these residues (stillage) must be dried. This can be done by straining out the solids and spreading them in a thin layer to dry in the sun, by use of rotary grain dryers, or similar equipment.

NOTE OF CAUTION

Alcohol produced for human consumption is made under special conditions and purified to a high degree. Ethanol that is produced according to the procedures in this book will contain fusel oils (high boiling alcohols), aldehydes, and ketones. None of these chemicals affect fuel performance but, if ingested, could cause fatal poisoning at worst or a horrible hangover at best. In addition, if the distillation equipment used later on is not tinned copper or stainless steel, many toxic metal oxides can be introduced to the alcohol. Solder, for example, contains a lot of lead and can react to form poisonous lead oxides. So besides being illegal, drinking your fuel could be hazardous to your health!

Chapter 10

INDIVIDUAL RAW MATERIALS

This chapter contains specific processing information and recipes for individual raw materials. If a material you are interested in using is not listed, you can usually approximate an appropriate process by using the information about a similar material. Note that feedstock materials are not consistent in the amount of fermentable materials, moisture content, and many other factors. The figures given here are averages. More specific information about particular materials can be obtained from your state agricultural service, or the material in question can be tested by an agricultural laboratory for a modest fee.

Remember, then, the following information is intended only as a guide.

SUGAR/STARCH CONTENT vs ALCOHOL

On the average, the amount of alcohol that can be produced from a given feedstock will be about half (on a weight/weight basis) of the convertible starch or sugar content. Ethanol weighs about 6.6 pounds per gallon. A ton of grapes, for example, with a 15% sugar content is capable (assuming 100% extraction) of producing about 150 pounds or 22.7 gallons of alcohol. Corn, with 66% convertible starch should produce 660 pounds or 100 gallons. Remember, this is only an approximation and actual yield depends on many interrelated factors.

SACCHARINE MATERIALS

The process of fermenting saccharine materials is relatively simple and straightforward. The steps involved are usually: (1) extracting or crushing, (2) pH adjustment through acid or backslopping, and (3) fermentation. Dilution is usually not necessary because the extracted juices often contain less than the 20% maximum of fermentable material. Exceptions to the above are the various types of molasses that do not require extraction, but usually require dilution.

FRUITS

The following are some fruits and their average sugar content: grapes, 15.0%; bananas, 13.8%; apples, 12.2%; pineapples, 11.7%; pears, 10.0%; peaches, 7.6%; oranges, 5.4%; prickly pear, 4.2%; watermelon, 2.5%; and tomatoes, 2.0%.

Allowing 75% extraction with apples, for example, the total fermentable material would be about 9% of the original weight. On this basis, a ton of apples would yield about 13 gallons of alcohol. Assuming an 80% extraction with grapes, a ton should yield about 17 gallons. With watermelons and a 90% extraction, a ton would yield only about 3 or 3-1/2 gallons. Clearly, some materials are better than others.

In all the above cases, the percentage of fermentable material in the extracted juice is low enough so that dilution is unnecessary and undesirable. To ferment these materials, the juice need only be adjusted to the proper pH (between 4.8-5.0) and the yeast added at the usual rate of 2 pounds per 1000 gallons of mash. To provide proper nutrients to the yeast, backslopping of about 20-25% by volume is desirable.

Also, all of the above materials may be simply crushed or pulped instead of extracted in a press. This way the total sugar content is available for fermentation. If you are using simple batch distillation equipment that does not require the beer to be strained, this method is recommended.

MOLASSES

Beet or cane molasses is the residue from the manufacture of sugar. These materials, if available, are excellent sources of alcohol. They contain 50-55% fermentable sugar, and a ton should yield between 70-80 gallons of alcohol.

Molasses with a sugar content above 15-20% will need to be diluted. Since most molasses is

low in the nutrients necessary for proper yeast growth, backslopping is of particular advantage. Up to 50% stillage (by volume) may be used. Also, most molasses is naturally alkaline, and acid will be needed in addition to the stillage to obtain the proper pH value.

CANE SORGHUM

Cane sorghum is a good alcohol source because it is easily grown and averages about 14% fermentable sugar content. The main drawback to using this material is that the extraction requires heavy-duty shredding and pressing equipment. An alternate process is to shred the stalks as much as possible and dissolve the sugar by heating (not quite to a boil) with a minimum amount of water. The process must be repeated several times to retrieve most of the sugar. Note that in this type of process, two extractions of one gallon each are better than one extraction of two gallons.

A conservative 65% extraction should yield about 13-14 gallons of alcohol per ton. Acidification to proper pH is necessary and backslopping to about 25% can be tolerated.

SUGAR BEETS

Sugar beets are an excellent material for ethanol production. They contain about 15% sugar, 82% water, and the rest in various solids. The juice can be extracted in a press, or the beets can be crushed and fermented as described in the section on fruits. Because the beets contain a certain amount of starch, the addition of small quantities of malt (1-2% by weight) or enzyme will greatly improve the alcohol yield. Adjustment of pH is, of course, necessary, and backslopping in the 20-25% range is desirable. A ton of beets should produce 20-25 gallons or more of alcohol.

SUGAR CORN WASTES

Stalks from sugar corn contain 7-15% sugar and should be considered as an alcohol source if they are available. The stalks need to be shredded and extracted in a manner similar to sugar cane or sorghum stalks. A relatively efficient operation should yield 8-18 gallons of alcohol per ton of material. Again, backslopping to 20-25% and acidification are necessary.

STARCHY MATERIALS

Starchy materials generally require milling, cooking, and conversion prior to fermentation. Exceptions are materials, such as potatoes and sweet potatoes, that do not require milling, and materials, such as artichokes, that do not require conversion. Relatively high alcohol yields often offset the necessary additional manufacturing steps, and most starchy materials are good alcohol sources.

GRAINS

Grains must be milled, diluted, cooked, and converted prior to fermentation. However, they contain large amounts of potentially fermentable material. The average content of convertible starch and sugar in some typical grains are: barley, 50%; maize (indian corn), 66%; oats, 50%;

rye, 59%; sorghum seed, 67%; and wheat, 65%. Alcohol yield per ton is dependent on how completely the starches are converted to fermentable sugar, but should be between 70-100 gallons.

After milling, the grain must be diluted prior to cooking and fermentation. The average dilution is between 56-64 gallons per 100 pounds of grain, depending on moisture and starch content. The method of cooking with minimal water and adding the balance prior to conversion, as described previously, has the dual advantage of reducing the energy needed for cooking and shortening the cooling time. Premalting with malt or enzymes is generally desirable.

Cooking is accomplished by heating the diluted and premalted mash to a slow boil and holding at this temperature for 30-60 minutes. Generally, the mash is sufficiently cooked when it is soft and mushy. The mash is then cooled to 145-150 deg F and the malt slurry is added. The malt slurry consists of about 2-1/2 pounds of dried or green malt per gallon of water as described in the section on malting.

On a weight/weight basis, corn or wheat will require about 8-10 pounds of malt per 100 pounds of grain. Rye will require about 10-12 pounds of malt for the same 100 pounds of grain. Other grains will fall somewhere in between. The malt slurry is stirred constantly during conversion. For wheat, the conversion will be complete in 5-15 minutes. Corn will require about 30 minutes, and rye between 30-60 minutes. The actual time, as well as the minimum amount of malt necessary, can be determined through trial mashes and the starch test as described in the section on malting.

When the conversion is complete, the mash is cooled to 70-75 deg F. and yeast slurry is added. Note that most grain mashes have an acceptably low pH and often do not need much adjustment. Backslopping should be limited to 20-25%.

The following is the general procedure for converting corn with Miles Laboratories enzymes. The procedure for other materials and other enzymes will differ slightly, and the manufacturer's recommendation should be followed.

After milling, the grain is partially diluted (slurried) at a ratio of 35 gallons of water per 100 pounds of grain. The pH is adjusted above 5.5 with an optimum range of 6.0 to 6.5. "Premalting" or liquefaction, is accomplished by the addition of 0.3 ounces of Taka-Therm enzyme.

The mash is then slowly heated. Gelatinization will begin at about 150 deg F. and the mash will rapidly thicken. Constant stirring is necessary at this point. At about 160 deg the liquefying action of the enzyme will begin. Heating may be more rapid after the liquefying action of the enzyme begins to take effect. After the mash reaches 200-212 deg an additional 1.3 ounces of Taka-Therm enzyme is added.

After the mash has been held at a slow boil for 20-30 minutes, an additional 33 gallons of water is added to complete dilution and cool the mash.

When the mash has cooled to 135-140 deg the pH is adjusted to 4.2 with acid and Diazyme L-100 enzyme is added at a ratio of 4 ounces per 100 pounds of grain. This enzyme completes the conversion in about 30 minutes and, after cooling to 70-80 deg, the mash is fermented in the usual manner.

JERUSALEM ARTICHOKES

Jerusalem artichokes deserve special mention as a source of alcohol because they contain between 16-18% fermentable material. In addition, the starches present can be converted without the use of malt or enzymes if cooked for a sufficient length of time. A ton should yield about 25 gallons of alcohol. To prepare artichokes for fermentation, they should be crushed to a pulp and cooked for 2-3 hours. If the starch test (described in Chapter 7) indicates that some unconverted starch is still present, conversion with small amounts of either malt or enzyme might be needed. Shorter cooking times are possible if a greater amount of malt or enzyme is used. For example, a 30 minute cooking time should be sufficient with a conversion using 3-6% malt or the equivalent amount of enzyme. Dilution is not necessary because the root usually contains 79-80% water. After cooking, the pH is adjusted and fermentation commenced in the usual manner.

POTATOES

Potatoes contain between 15-18% fermentable material and are a traditional source of alcohol. On the average, a ton of potatoes will yield about 22-25 gallons of alcohol. Damaged or sprouted potatoes are not objectionable, and the use of sprouted potatoes will reduce the amount of malt or enzyme required for conversion.

Commercially, potatoes are usually cooked with steam, under pressure. An acceptable alternate method is as follows: The potatoes should be shredded or cut up and placed in the cooker with as little water as possible; cover the cooker and steam until the potatoes are reduced to a soft mass. Premalting to reduce viscousness is a definite advantage. After cooking, the mash is cooled to the conversion temperature. Usually only 3-4 pounds of malt per 100 pounds of potatoes are all that is required. The mash must be constantly stirred during conversion, which will take about 15-20 minutes.

Because cooking and conversion times will vary, depending on starch content and the like, test mashings and the use of the starch test is recommended. Once converted, the pH should be checked and the mash fermented in the usual manner.

For specific procedures for the use of enzymes to convert potatoes, consult the manufacturer. Otherwise, about half the amounts listed in the corn recipe should be sufficient.

SWEET POTATOES

Sweet potatoes average about 22% starch and 5-6% sugar for a total of 27-28% fermentable material. A ton should yield up to 40 gallons of alcohol. Sweet potatoes are cooked and converted in a manner similar to potatoes with the exception that they contain only about 66%

water and some dilution is necessary.

CELLULOSE MATERIALS

The following "recipe" for the conversion of cellulose is based on the use of two enzymes available from Novo Laboratories. "Celluclast" enzyme is produced from a variety of the Trichoderma viride fungus and is active in breaking cellulose into cellobiose and glucose. The former is not a fermentable sugar, therefore, a second enzyme, "Cellobiase" is used in conjunction with Celluclast to convert the cellobiose. Together the two enzymes have the ability to convert cellulose to sugar with near 100% efficiency.

However, in order for the enzymes to work, the cellulose must be accessible. Any cellulose material should be shredded, ground, or otherwise reduced to as fine a state as possible.

After shredding, or whatever, the material is mixed with as little water as possible to make a thick, soupy mass. The pH is adjusted to between 4.5-6.0, and the enzymes are added.

It will be impossible to determine the exact amount of "accessible" cellulose, and the amount of enzyme needed must be estimated. Generally, dry cellulose materials such as wood, straw, corn cobs, etc. will have the lowest yields. This is because the cellulose is encased in lignin, and the amount that is ultimately accessible to the enzymes is proportional to how finely the material is divided. Materials such as grass clippings and all moist, green cellulose containing materials will have the next highest yields. This is partly because the lignin content is lower and partly because some fermentable glucose is already present. The highest yields will come from materials such as paper and cotton that are almost pure cellulose.

A trial amount of enzyme for all of the above Materials would be about 2% Celluclast and 0.2% Cellobiase on a weight/weight ratio to available cellulose. Thus, if wood chips were estimated to have 5% by weight available cellulose, about 0.1% of the first enzyme is needed and 0.01% of the second. This would work out to 32 ounces of Celluclast and 3.2 ounces of Cellobiase per ton of wood. Newspapers, on the other hand might have 50-80% available cellulose and the amount of enzyme needed would be greater.

Optimum temperature for the enzyme reaction is 140 deg F. The mash should be held at this temperature for about 16 hours. The temperature should then be reduced to 80-90 deg and fermentation commenced in the usual manner. Prior to adding the yeast, the pH should be checked and adjusted to the optimal range for the yeast strain.

It is suggested that trial conversions and fermentations be made to determine the minimum amount of enzyme needed to produce maximum yield.

A simplified "recipe" for green cellulose material of almost any kind is shredding followed by fermentation. As noted earlier, plants first produce glucose (a fermentable sugar) and then convert the glucose to cellulose. Yields will be based entirely on the amount of glucose present as cellulose is not converted by this method.

MULTIPLE ENZYME TREATMENT

All materials used in the production of ethanol will contain some cellulose. Therefore, it might be worthwhile to experiment with small amounts of cellulose enzymes in conjunction with the other processes. Saccharine materials might benefit from a separate cellulose conversion step. Starchy materials could have the cellulose enzymes added during conversion in addition to the starch enzymes. Depending on the amount of available cellulose, this procedure could dramatically increase yields.

Chapter Index Chapter 1 AN OVERVIEW Chapter 2 BASIC FUEL THEORY **Chapter 3 UTILIZATION OF ALCOHOL FUELS Chapter 4 ETHANOL PRODUCTION - GENERAL DISCUSSION** Chapter 5 PROCESSING STEPS COMMON TO ALL MATERIALS **Chapter 6 PROCESSING STEPS SPECIFIC TO SACCHARINE MATERIALS** Chapter 7 PROCESSING STEPS SPECIFIC TO STARCHY MATERIALS **Chapter 8 PROCESSING STEPS SPECIFIC TO CELLULOSE MATERIALS Chapter 9 YEAST AND FERMENTATION Chapter 10 INDIVIDUAL RAW MATERIALS** Chapter 11 DISTILLATION Chapter 12 DRYING THE ALCOHOL Chapter 13 MASHING AND FERMENTATION EQUIPMENT **Chapter 14 DISTILLATION EQUIPMENT Chapter 15 SOLAR STILLS** Chapter 17 PUTTING IT ALL TOGETHER Chapter 18 THE FUTURE

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