

# Feedstock Processing Experiments Preliminary Report

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## **1 Executive Summary.**

The authors undertook a study to determine a time and cost effective process for converting stale bakery waste into fuel ethanol. The resulting process meets or exceeds all of our goals and objectives, and has proven to work reliably over a wide range of waste material (stale bread, pastries, bagels, and muffins). The presence of mold on the feedstock does not appear to adversely impact the results, even though the process uses relatively low temperatures.

The most significant finding is that we do not need alpha-amylase enzyme for successful processing of this type of feedstock. Our process is successful using only gluco-amylase. Not only does this reduce the cost of consumables, it makes the overall process simple and time effective. We require only one pH adjustment (to approximately pH = 4.5) at the beginning of processing and this pH adjustment is sufficient for the complete starch to sugar conversion process as well as for subsequent fermentation. Our process only requires cooking the mash at 115 degrees F for a total of 4 hours. This low temperature could be provided using flat panel solar thermal heating, eliminating the recurring cost of energy for processing purposes. We also found that continuous agitation of the mash is essential for obtaining good, reliable results.

Bakery waste is ubiquitous in the urban environment and our experimental results show that it is an excellent (and free) source of feedstock for urban dwellers who do not have the ability to grow their own energy crops. The cost of small scale ethanol production is always a challenge and we believe that our results represent a pathway to cost-effective small scale fuel ethanol production.

The results presented in this report are preliminary. Our experimental protocol calls for laboratory distillation of our processed samples in order to validate our estimates of ethanol yield. To date, however, we have been unable to secure the necessary government permits for laboratory distillation. We have our “beer” in storage and we intend to update this report with final results after obtaining our permit and distilling our many samples.

This report contains a comprehensive compilation of our learnings during this year-long experimental process. It contains detailed learning about processing stale bakery waste and some learning about fermentation (although a detailed study of fermentation is outside of the scope of this effort). We also document what we have used in our experiments: the consumables, measurement equipment, and our experimental processing equipment. We hope that the knowledge that we have gained will help others to research additional aspects of small scale ethanol production, including fermentation, distillation, storage and distribution, and quality assurance.

## 2 **Terminology.**

The following terminology is used consistently throughout this document:

- **Feedstock**: For the purposes of this report, the term “feedstock” refers to stale bakery waste, including various types of bread loaves (often moldy), pastries, bagels, and muffins.
- **Mash**: Starchy feedstock is ground up and mixed with water, forming a “mash”. The mash is then pH adjusted and cooked in enzymes to reduce it to a “wort”.
- **Wort**: A solution of fermentable sugars in water resulting from the feedstock processing of a mash. The wort may contain other water soluble and insoluble material resulting from processing the mash. Yeast is pitched into the wort, which then ferments in fermentation vessels to convert the fermentable sugars into ethanol.
- **Beer**: In this context, a “beer” is a solution of ethanol in water (along with non-fermentable material) that is the result of yeast fermentation of the wort. After fermentation is complete, the beer is then distilled to produce high proof alcohol fuel.

### 3 Background and Results.

#### 3.1 Summary Results and Findings.

We (the authors) undertook a study to determine a “least cost recipe” for processing stale bakery waste into ethanol for fuel. The parameters for this study were driven by the ultimate objective of ethanol fuel production by urban dwellers. The implication of this objective is discussed in detail in section 3.2, below. In summary, this objective requires that the feedstock be processed into a fermentable “wort” and placed into proper fermentation vessels within one working day’s time.

We conducted many experiments on batches of 2.6 lbs of “feedstock” mixed into approximately 4 quarts of water. The feedstock was “grated” using a kitchen food processor and added to tap water. After processing, we achieved a wort with potential alcohol (as measured by a hydrometer) of between 7% and 13% alcohol by volume (%abv). Every batch that we created subsequently fermented completely. There were no failed batches. As of this writing, we are awaiting a still permit in order verify the actual alcohol content of our beer batches via laboratory distillation. We performed a few experiments utilizing one quart less and one quart more water, as the density of starchy material in the feedstock varied greatly between batches. This topic is elaborated upon in section 3.3, below.

After adding the grated feedstock to water, we adjusted pH and cooking temperature to process the starch down into fermentable sugars using enzymes. The most significant finding of these experiments is that alpha-amylase (AA) enzyme is not required in order to achieve our primary objective. We found that we are able to do just as well using only gluco-amylase (GA) to reduce the mash to a wort. We observed that the thick, lumpy mash thinned out into a smooth and relatively uniform solution within a minute after adding GA enzyme. However, full reduction of starch, as tested using iodine (Lugol’s solution) required approximately 4 hours of cooking at approximately 115 degrees F.

The overall recipe that we developed for cooking this feedstock down into a fermentable wort is as follows:

- 1) Prepare a “yeast starter” solution. We prepared ½ cup of water and mixed in approximately 2 tablespoons of table sugar. The solution was then heated to between 93 and 97 degrees F and ½ to 1 tsp of distillers yeast was added. The solution was left to stand at room temperature, loosely covered, and stirred thoroughly about once every hour, until it was needed to be “pitched” into the wort. The yeast starter solution reconstitutes the yeast from its dehydrated storage condition and allows the yeast to reproduce before it is needed for fermentation of the wort.



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- 2) Grind the feedstock. We used a kitchen food processor with a “grating” blade to grind our feedstock before mixing with water to form our mash.
- 3) Add the feedstock to water. Approximately 2.6 lbs of feedstock was generally added to 4 quarts of water. This ratio produced between 7% and 13% estimated alcohol by volume (%abv); the variability being in the feedstock. We found that we could generally estimate if more or less water should be used, based upon how many serving bowls it took to hold 2.6 lbs of ground feedstock. This is discussed further in section 3.3, below. We believe that properly estimating the necessary water would give more consistent results and avoid the “thick mash” condition, discussed further in section 3.3. However, we almost always used 4 quarts of water to maintain controlled conditions from batch to batch. We only varied the water content on a very few occasions, just to see if this hypothesis was correct (it is).
- 4) Adjust pH. Because we finalized on using GA enzyme alone, we targeted pH at 4.5, and accepted pH in the range of 4.3 to 4.8. When the pH of the initial mash was higher than this range, we added dry citric acid to reduce the pH down into this indicated range.
- 5) Raise temperature to 115 degrees F. We performed a lot of experiments under carefully controlled conditions reducing cooking temperature to find a minimum that met our criteria. We found that a nominal 115 degree F was this minimum. Lowering cooking temperature to 105 degrees F resulted in incomplete starch conversion even after 5 hours of cooking.
- 6) Add GA enzyme. We experimented with a dry enzyme and a liquid enzyme. For the experimental conditions, we found that we needed 4.8 grams of the dry GA enzyme or 0.8 ml of the liquid GA enzyme, with one gallon batches of mash, in order to achieve results that met our overall objectives. These values would be proportionally scaled up for production sized batches, as too little enzyme results in longer processing times.
- 7) Cook for 4 hours. We found that cooking for 4 hours at 115 degrees F after adjusting pH and adding GA enzyme resulted in complete or near complete conversion of starch to fermentable sugars, as indicated by an iodine test. Cooking temperature was thermostatically controlled using an electric burner and homemade controller. We found that continuous agitation is important and we always cooked using a motorized agitator to keep the mash under continuous mixing. If the mash was not mixed continuously, a fair amount of starchy material would stick to the sides and bottom of the cooking vessel and not get reduced to sugar. Continuous agitation, described further in section 5 of this document, left virtually no residue stuck to the cooking vessel.



- 8) Rapidly cool to under 97 degrees F. Rapid cooling down to the temperature that the yeast can survive is important in order to limit exposure of the wort to microbial infection. We transferred the one gallon batches to a clean and sterilized 2 gallon stainless steel cooking pot sitting in a kitchen sink partially filled with ice water. We measured wort temperature with a laboratory thermometer and pitched the yeast when the temperature got below 97 degrees F.
- 9) Add yeast starter and yeast nutrient. For one gallon batches, we added 1 tsp of DAP yeast nutrient and our ½ cup of yeast starter solution. We stirred well and then transferred our wort to glass carboys with “S” airlocks to ferment. Fermentation time was generally under 6 days, but fermentation could be as long as 2 weeks if the ambient temperature was low or if starch/dextrin conversion was incomplete. The latter is discussed further in section 3.3, below.

This process has been repeated with various different “stale bakery” feedstock and has proven to be reliable and repeatable. The elimination of AA enzyme is a significant cost saver in two ways: (a) the cost of the AA enzyme itself, and (b) AA requires significantly higher cooking temperatures and therefore added energy costs. Using only GA enzyme at a cooking temperature of 115 degrees F meets our time objective and should be easy to heat with flat panel solar collectors, eliminating cooking energy cost entirely. However, our experiments were carried out at ambient room temperature using a 1 KW thermostatically controlled electric heater with the resulting heater power on-time of between 1400 and 1600 seconds (thus, using approximately 0.4 Kwh of electricity for cooking heat for a one gallon mash).

### 3.2 Experimental Goals and Criteria.

We had two major goals when we began this series of experiments: (1) learn how to convert starch into ethanol, and (2) develop a recipe to utilize “urban waste material” to make ethanol in a cost effective manner. Our overall objective was to address small scale fuel ethanol production for the urban dweller.

Most of the literature on small scale ethanol production is oriented toward farmers who would utilize part of their farmland to grow energy crops for personal or small scale cooperative use. However, some proponents (e.g. Blume<sup>1</sup>) have postulated that urban food waste could be a source of feedstock as well. Urban waste, in this context, includes stale bakery products (bread, doughnuts, pastries, bagels, muffins, etc.) whose edible shelf-life has expired, fruit waste from canneries, sugar waste from milling and candy manufacturing, and even alcohol waste from commercial beer brewing, winemaking, and spirit distilling. We chose to work with stale bakery waste because it is ubiquitous in the urban environment; available from every supermarket, boutique bakery outlet, coffee and doughnut shop, bagel shop, and local food banks.

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<sup>1</sup> “Alcohol Can Be A Gas”, by David Blume; ISBN 9780979043772, 9780979043789.

We found that most bakery product retailers donate material that has passed its quality shelf-life to non-profit organizations. Only a small portion of this donated material actually makes it into the human food chain before it become inedible. At that point, the material is discarded. We obtained our feedstock from one such source: a residential outpatient mental health facility that routinely receives food donations and ends up discarding approximately 90% of it uneaten because it has become too stale or even moldy. This small facility received food donations from two Safeway supermarkets and one Starbucks coffee shop. They routinely discarded about 15 – 20 pounds of bakery material per week. However, when the people who gathered donations learned about our project, they collected a lot more material; in excess of 50 pounds per week, from these same sources. Our conclusion is that there is a substantial amount of stale bakery product material that is human inedible to be found in any local neighborhood of an urban area.

The implications of urban dwellers making their own ethanol fuel go far beyond the feedstock, however. Urban dwellers are presumed not to have sufficient personally-owned land, properly zoned, to process substantial quantities of feedstock into a beer, not to mention meeting the government requirements to site a still. Urban dwellers have a choice of leasing/renting commercial/small-scale industrial space or traveling to a production site in the rural outskirts. Our initial assessment was that only the latter would be cost effective, meaning that a small producer co-op would need to own or lease land that is at least one hour drive from their homes. The fact that urban ethanol fuel producers would have to travel substantial distances to their production site has a great many implications. The fact that urban ethanol fuel producers would not live on the production site has further implications. The major implications that drove our experimental goals and objectives were:

- “Feedstock processing” must be accomplished within one day, including two way travel time. This means that the feedstock must be transported to the processing site, the processing equipment prepared (cleaned and sterilized), the feedstock ground and mixed with water, the processing to a wort accomplished, the resulting wort transferred into fermentation tanks, and the site and equipment cleaned up, all within a single day (daylight hours). This led us to set a limit on the feedstock processing time itself of 5 hours maximum.
- We presumed that urban dwelling producers would have a “day job” and only be available for ethanol production work on selected weekend days. Fermentation cannot be accomplished in one day, so we set a goal to have fermentation complete within one week (7 days), which is reasonable for most distillers yeasts and summer temperatures (we did not, however, set out to explore fermentation in detail, and only fermented the wort to ensure that it was indeed fermentable). Therefore, the feedstock would be processed on a weekend day and left to ferment for a week. The next weekend, the completed beer would be distilled.

- These limitations would appear to have significant repercussions for distillation. Unless a still can be engineered and permitted to run unattended, a small continuous still design would seem to violate our assumptions. A large capacity batch still would seem to be required. Distillation of a previously fermented batch would, presumably, take place while a new batch of feedstock was being processed. However, our goals and objectives did not include distillation, except for laboratory distillation as part of the feedstock processing analysis (supporting ethanol yield calculations).
- Fuel distribution is also an issue for urban dwellers. It would either be necessary for co-op members to travel long distances to the processing site to obtain their fuel (uneconomical), or the distilled fuel would be trucked to the urban area and distributed either at a central location or directly delivered to the co-op members. A suitable fuel tanker truck would be required, but could serve both to store and to transport the resulting fuel. Further consideration of fuel storage and delivery was outside of the scope of this study.

Our other primary concern was the cost of producing the fuel. Even given that the feedstock would be obtained for free, the land, facilities, equipment, consumables, and labor are not usually free. Urban dwellers may be presumed to have access to commercial ethanol fuel in the form of E85. An urban co-op could, perhaps, simply contract for wholesale denatured ethanol for member use. Consequently, fuel production cost, even given “in-kind” labor by co-op members, is a significant issue.

Consideration of all of the cost factors involved in turning bakery waste into fuel is beyond the scope of this study. However, we assumed that even if only the cost of consumables was significant (everything else donated), it would be necessary to produce alcohol fuel for under \$2 per gallon in order to become cost effective in people’s minds. Leaving \$1 per gallon cost for distillation (an assumption), we set a limit on the cost of feedstock processing consumables at \$1 per gallon of azeotropic ethanol. This includes: (a) cost of cleaning/sterilizing chemicals, (b) cost of chemicals to adjust pH, (c) cost of energy for cooking the feedstock, and (d) cost of enzymes. The cost of yeast and yeast nutrients should also be considered, but cultivation and propagation of yeast for production purposes was beyond the scope of our experimental work.

### **3.3 Detailed Discussion of the Findings.**

This section presents some of the detailed findings of our feedstock processing experiments. The most significant findings were summarized in section 3.1. Additional findings and observations are reported here.

#### **3.3.1 Enzymes:**

The most significant finding of our series of experiments is that alpha-amylase (AA) enzyme is not needed for reducing stale bakery products to a sugar-water wort. We found that gluco-

amylase (GA) alone works well. This finding is very significant because of its many implications:

- a) The cost of AA enzyme is eliminated, reducing the overall cost of consumables.
- b) The energy requirements for cooking are reduced, and the temperatures necessary are compatible with flat panel solar heaters, potentially eliminating the recurring cost of cooking energy (a consumable). Starch hydrolysis with AA enzyme requires cooking at above 170 degrees F; too high for flat panel solar. Cooking with GA alone allows us to use temperatures of around 115 degrees F, which are easily achieved with flat panel solar. Even if solar energy is not used, the overall cooking energy requirements are reduced by a factor of 4/5 or higher.
- c) Hydrolysis and liquefaction using AA enzyme requires a pH of between 5.0 and 7.0. Most of our feedstock samples naturally fell into this range, but some sourdough stock was naturally lower in pH. Use of AA would require raising the pH with a base, and then lowering it for saccharification using GA enzyme. By using GA only, we found that the natural pH was either already in the range where GA works best (generally pH of 4.0 – 5.0) or that the natural pH was always higher and required us to use acid to reduce it. We had base available (calcium carbonate) but we never had to use it to raise pH for our processing work.

We have several theories as to why we could skip directly to saccharification for our bakery waste feedstock. Our primary theory is that that baking process itself breaks down sufficient bonds for the GA enzyme to work. An alternative theory is that the bakery products already have other enzymes in them, e.g. from sprouted grains (a natural source of amylases) or enzymes being added as part of the baking process. We noted that the ingredients listed on all packaged feedstock that we obtained always included either sprouts or enzymes, or both.

The GA dosage that we determined meets our requirements is (per gallon of mash):

- 0.8 ml per gallon of mash (liquid GA)
- 4.8 grams per gallon of mash (solid GA)

Reducing the GA dosage below these levels increases the processing time. Increasing the dosage may reduce processing time, but we did not investigate this further because this dosage meets our time requirements at minimal cost of consumables.

Raising the temperature can reduce processing time, but there are limits beyond which the GA becomes deactivated. Reducing temperature slows the conversion process markedly; however, conversion appears to continue even at room temperature (see the subsection on Fermentation, below, for further discussion of this). Cooking at approximately 115 degrees F was found to be the lowest temperature that met our processing time requirements.

### 3.3.2 Visual Indications:

When the ground up feedstock is mixed with water and agitated, it creates a relatively granular mixture. Some, but not all of the feedstock, dissolves in the water. Most of the solid matter does not dissolve and produces a thick and lumpy (and sometimes sticky) mixture. Very shortly (often less than one minute) after adding GA enzyme at 115 degrees F, the mixture changes markedly. It becomes much smoother and consistent in appearance and somewhat thinner (much easier to stir). This is a clear indication that the GA enzyme is working and is aiding in starch hydrolysis and liquefaction. Iodine testing indicates that starch is present at this point in the process. It takes 3 - 4 hours of cooking for the starch to be fully converted to sugar (saccharification).

### 3.3.3 Yeast Starter:

We always prepared a yeast starter solution and allowed it to cultivate while we were processing the feedstock. Our yeast source was a package of commercial dehydrated distillers yeast. The packaging indicated that the yeast needed to be rehydrated at between 93 and 97 degrees F. We chose to rehydrate the yeast in a solution of 2 tablespoons of table sugar mixed into ½ cup of tap water. The sugar-water solution was heated to above 97 degrees F in a microwave oven and then allowed to cool to below 97 degrees, at which time we added approximately ½ to 1 teaspoon of yeast. We mixed the yeast and sugar-water thoroughly, remixing each hour. It sat exposed to air, loosely covered, until we needed to pitch the yeast into the final wort.

We found that rehydrating the yeast in a warm sugar-water solution allowed the yeast to propagate, as evidenced visually by the frothing and clouding of the solution. We stirred the yeast starter approximately once every hour in order to ensure that the solution was well oxygenated, such that the yeast would propagate aerobically rather than make ethanol anaerobically. In almost all cases, this process resulted in a yeast starter solution that was extremely cloudy (indicating that a lot of yeast cells were present), with a thick “head” of foam at the top. This was our indication of a successful yeast starter. In two instances, the yeast failed to rehydrate during the first hour or so after preparation (reasons unknown). In these instances, we simply re-made the yeast starter. Therefore, another benefit of preparing a yeast starter is to ensure that the yeast is healthy and robust before pitching it into the wort. In every experiment that we performed, this yeast starter always worked; we always got fermentation of our wort up and running very quickly.

### 3.3.4 Dry and Moldy Feedstock:

Our feedstock samples varied widely in the type of bakery products that we obtained as well as in its condition. Some of the feedstock was quite moist and often sticky; other samples were so dry that they crumbled, or were as hard as rock. Some samples were so tough that we feared burning out the motor on the food processor that we used to grate it with. Other samples almost grated themselves. Very dry and very moist samples created their own problems with too-thick and too-thin mashes; a topic that is discussed below.

Some of our feedstock was moldy; some extremely so. So much mold was present at times that we felt it necessary to take strict precautions on handling and to clean up using copious quantities of chlorine based cleaner. However, we found that the presence of mold did not appear to impact the results. Our final recipe does not involve boiling (or any high temperatures) and the final pH range is natural for some of the feedstock. On this basis, we do not believe that our processing conditions killed the mold. We originally feared that the mold might kill the yeast, but it did not. Some of our wort samples were very green in color owing to the mold. These worts fermented as well as those with little or no mold and the resulting liquid beer was a golden color in every case. The lees and non-soluble residue at the bottom of the fermenters after fermentation, however, did retain the greenish tinge of the mold, even after fermentation. We suspect that this is a residue of the original mold and that the ethanol in the beer killed the mold, but we did not try to establish this positively. In any event, we figure that distillation heat will kill any mold that remains. However, this is one more reason to clean thoroughly and *sterilize* the equipment between batches.

### 3.3.5 Thick and Thin Mash:

We found that some of our feedstock samples were very moist and other samples were bone dry. The very moist samples weighed a lot per unit volume, owing to the high water content. Conversely, the very dry samples weighed comparatively little per unit volume. We always used 2.6 pounds, by scale weight, of feedstock, regardless of the volume that this weight of feedstock took up. We grated the feedstock into kitchen serving bowls of approximately 2 quarts capacity each. We found that 2.6 pounds of a very moist feedstock would take up only one serving bowl, while the same weight of a very dry feedstock would take up a two full bowls or more.

As might be expected, the moist feedstock ended up being low in starch (per pound weight) and the dry feedstock was much higher in starch, per unit weight. Although the feedstock contained solid material other than starch and sugar, we found that the “number of serving bowls” was a good indicator of the thickness of the resulting mash (after adding water) as well as of the final estimated alcohol content. In fact, some of our batches were so dry that after adding 4 quarts of water and processing as usual, the resulting wort was so thick that the hydrometer wouldn’t float and we could not therefore obtain an estimate of the alcohol content. We also found that this “thick mash” situation apparently did not complete processing within our 5 hour time window, even though the starch test seemed to indicate that it had completed. We determined this by noting an excessively long fermentation time. Generally speaking, fermentation (under summer conditions) would complete in 5 – 6 days. However, these “thick mash” situations would continue to ferment for 2 weeks or more. We surmise that the “thick mash” made it difficult for the enzymes (or iodine of the starch test, for that matter) to get to all of the starch molecules during processing, but the enzymes continued to work (albeit slowly) during fermentation, prolonging the total fermentation time. Fermentation times are discussed further in section 3.3.10 below.



Very moist feedstock, conversely, gave us a very thin mash and resulted in low estimated %abv levels. Again, we attribute this to low starch and sugar content because so much of the weight (which was our measurement standard) was in moisture. Thin mashes processed and fermented well, in general, but low resulting alcohol content means higher distillation energy costs.

On one occasion, we repeated a thick mash experiment using the same feedstock but mixed in 5 quarts of water, vs. the normal 4 quarts. On another occasion, we repeated a thin mash experiment, but using 3 quarts of water vs. the normal 4 quarts. In both cases, we obtained a mash that processed and fermented closer to the norm than if we had used 4 quarts of water. This validated our assumptions regarding thick and thin mashes and we conclude that the “serving bowl” measurement was an excellent indication of the amount of water that should be used in order to more or less normalize a mash to produce a nominal 10% abv beer.

We believe that if a wort is obtained that is too thick to float a hydrometer, water should be added prior to fermentation, until the hydrometer floats. We did not, however, actually try to add water to a wort just prior to fermentation in our experiments. We always added water only prior to cooking or during cooking of the mash down into a wort.

### 3.3.6 Agitation:

Our initial experiments were conducted with more or less continuous hand stirring with a kitchen stirring paddle. We found this to be totally inadequate. Sticky matter tended to cling to the inside and bottom of the pot – anywhere where the stirring spoon missed. We rapidly concluded that continuous agitation is needed. We concluded that a successful agitator would need to:

- be motor driven, so as to operate continuously, at a constant speed;
- contain a sweeper arm to keep material moving near the sides and bottom of the cooking vessel;
- create vertical, as well as horizontal, motion of the mash in order to continuously mix at all depths; and
- not interfere with temperature measurement of the mash, necessary for cooking temperature control.

A highly successful motorized agitator was developed from these requirements. It is described in section 5 of this document.

Our agitator motor was a gearmotor operating the agitator mechanism at 80 rpm. Our initial guess, based upon observation during the early, manual stirring, was that somewhere between 30 and 60 rpm would be sufficient. We ended up at 80 rpm only because we were able to find an inexpensive gearmotor with the necessary torque that happened to run at this speed. We did not perform any experiments using different agitator speeds, but we believe that lower speeds (30 – 60 rpm) would work as well. We were not able to try any different or modified agitator designs, so we can only definitively say that the design we used works very well. We continue to believe



that a good agitator should create a vertical motion in the mixture as well as sweep the sides and bottom to prevent starchy material from sticking there prior to liquefaction and saccharification of the mash.

### 3.3.7 Cooking Temperature Control:

The literature and specifications for amylase-family enzymes indicate that they work well over a fairly broad temperature range. However, we set out to try to control the mash cooking temperature very closely in order to ensure repeatability from one experiment to another. We used an existing, homemade controller, reprogrammed specifically to support these experiments, to thermostatically control the temperature of the mash. The controller turned AC power on and off to a 1 KW single burner electric stove. The equipment details are presented in section 5 of this document.

The temperature controller was thermostatic in nature and programmed with a  $\pm 0.5$  degree F hysteresis. Nevertheless, upon initial application of AC power to the stove, the temperature overshot the set point significantly. Although the controller removed AC power to the stove at 115.5 degrees F, the mash temperature continued to climb to over 126 degrees F, before slowly beginning to cool. We attribute this to a significant amount of thermal storage (thus lag) in the electric stove. Clearly, a 1KW stove was too powerful for this application; a lower wattage stove would have taken longer to attain the necessary mash temperature, but we believe that it would have overshoot considerably less.

After the initial temperature overshoot, the mash cooled down to 114.5 degrees F, at which time the controller reapplied AC power to the stove. There was a little undershoot (to about 113.9 degrees) seen in the mash temperature, and similarly small overshoot on the high side once the mash had reached the point of the controller cycling the AC power to the stove around the set point of 115 degrees F.

Note that 115 degrees was the final temperature that we established, based upon a large number of experiments. We started off using a corn processing recipe that included premalting, boiling for 45 minutes to one hour, cooling to between 170 and 190 degrees F for AA enzyme processing and then cooling to between 120 and 140 degrees F for GA enzyme processing. This initial recipe took a lot of cooking energy at high temperatures and took longer to achieve a fully processed wort than the recipe that we ended up with. We gradually and methodically deleted steps and reduced cooking temperature within the steps until we achieved this final result. This partly explains why we did not choose to try and mitigate the initial overshoot<sup>2</sup>. In summary, we

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<sup>2</sup> Since we had the complete ability to write software for our controller, we could have employed a more sophisticated control algorithm. We chose not to do so because by the time that we got down to cooking initial temperatures where the overshoot was significant, we had already gathered a lot of data using the simple thermostatic control algorithm and chose to not change the baseline processing methodology but simply to record the facts. We do not believe that this large initial overshoot compromised the results in any way and we believe that the GA enzyme works well over a considerable temperature range and that the maximum temperatures seen in the overshoot are well below the temperature that would disable or compromise the enzymes.

do not believe that very precise temperature control is needed – only that it be kept between about 115 degrees F on the low side and under 130 degrees or so on the high side.

### **3.3.8 pH Adjustment and Range:**

We found that the natural pH of our feedstock ranged from about 6.5 to 4.5. The lower pH was associated with sourdough, although not all sourdough had a natural pH this low. After we established that AA enzyme was not required for processing this feedstock to a fermentable wort, we found that the natural pH of our feedstock was either higher or “just right” for the GA enzyme. Different manufacturers of GA enzymes list different pH ranges, but they generally fall into pH 4.0 – 5.0. Our target, therefore, was pH = 4.5, and we accepted pH in the range of 4.3 – 4.8, after adjustment with citric acid.

We used dry citric acid to adjust pH. We selected citric acid simply for safety reasons. We were experimenting in a kitchen with kitchen gloves, apron and hardware-store eye protection. We did not want to provision ourselves with safety equipment and supplies that would be necessary if stronger acids were used.

We generally added 0.5 – 1.0 grams of citric acid at a time to our mash, let it agitate for a few seconds, and then measured pH, and adjusted again as necessary. On one occasion, we needed to add as much as 8.0 grams (total) of citric acid to adjust pH, but on all other occasions, the necessary dosage was under 5.0 grams (total). This was for one gallon size mashes. In summary, it took surprisingly little of this relatively weak acid to correct pH to the necessary value.

We did not experiment with the effects of changing pH on our final recipe, as we felt that some pH adjustment would be required in any event and the cost difference would be negligible. Theory, as well as published data tends to show that a higher pH results in increased processing time, whereas too low a pH may deactivate the enzyme.

It should be noted that the pH range that we targeted is a good range for yeast fermentation as well. We did not adjust pH further and we fermented at whatever pH we achieved for the GA enzyme. Final pH readings were taken on the wort just prior to pitching in yeast nutrient and yeast starter. These were generally the same as the initial, citric acid adjusted, pH readings and in no case did they vary by more than pH 0.2 from the initial, citric acid adjusted, pH reading. In these instances, the final pH was higher than the pH we measured when we added the GA enzyme. We attribute the variation, if any, to measurement error in our meter, as we do not believe that any chemical reactions take place that would change the pH.

### **3.3.9 Starch Testing Using Iodine Measurement:**

We tested for the presence of starch using Lugol's (2% iodine) solution. We tested prior to adding any enzymes, and in all cases we obtained a strong positive indication for starch, as discussed below. Cooking at 115 degrees F with GA enzyme definitely reduced the starch

content with time. However, we had substantial difficulty in determining when no starch was present.

The main source of difficulty with the iodine test was the variation in color of the feedstock mash. Some was fairly light and whitish in color, and some was very dark brown, with every hue and shade in-between. Needless to say, a dark brown mash color made it extremely difficult to discern iodine color.

We found that color (hue) observation is quite subjective in any event. We ultimately took two samples in identical white sample cups and put a drop of iodine in one, stirred and compared with the other sample. This at least gave us a control sample to compare with.

Since the actual color (hue) was often very hard to discern, we settled on a metric of the time that it took for the darker iodine-added sample to lighten until we could not tell the difference from the control (no iodine added) sample. We found that the initial, pre-cooking samples would take 30 seconds to several minutes to lighten to the control sample (if it ever did). We established this metric as the baseline for strong starch reaction. We performed many experiments where we took samples for starch testing every ½ hour during the cooking process and noted that the time for the iodine-added sample to fade to the same darkness as the control sample decreased until the “final” test where the time for the iodine added sample to fade completely was under 5 seconds. Longer cooking times did not change this result. Therefore, we adopted this “5 second” rule as our metric for “starch conversion complete”.

We did not have any test for dextrin, which is an intermediate product of the conversion of starch into fermentable sugars. Our 4 hour cooking time result is based upon a negative starch test (as described above) within about 2.5 hours of cooking with GA, leaving an extra 1.5 hours for dextrin conversion. We do not know if this time is too long or too short, but we can say that fermentation would complete within 5 – 6 days if we followed this starch reduction process (see below for a discussion of fermentation times).

### **3.3.10 Fermentation Times.**

Our objective was to obtain fermentation time of less than 7 days. We found that under “summer” conditions, the process we ended up with would complete fermentation in 5-6 days. Completion of fermentation was judged at the point where there was no pressure at all seen in our S-type airlocks. Generally speaking, noticeable fermentation (positive pressure in the airlocks) was observed within one hour of pitching the yeast starter and transferring the wort into the fermentation vessels (1 gallon un-insulated glass carboys with S-type airlocks). By the next day, there was substantial bubbling seen in the airlocks – up to one or two bubbles per second. We often observed an impressive head of foam – one to three inches in height – on top of the fermenting wort, during this very active fermentation period. After the second day, fermentation slowed noticeably, often down to longer than 5 seconds per bubble. However, it took at least 3

more days for fermentation to slow to the point where no pressure at all was observed in the airlocks.

As stated above, we fermented every batch that we produced, even those where we had a clear indication of residual starch after 5 hours of processing. In these latter cases, we noticed that fermentation would continue well beyond 7 days. The full fermentation time for these “residual starch” batches varied between 10 days and 18 days in our experiments. We attribute this to the fact that the GA enzyme remains unchanged in the wort and continues to break down starch and dextrin, even under the room temperature conditions of fermentation. The conversion process is very slow at this point, but it is present and we found complete fermentation time, under controlled room temperature conditions, to be a good indicator of whether residual starch or dextrin were present in our wort.

On two occasions, our wort was very thick as a result of very dry feedstock and insufficient water. The hydrometer would not float in this thick wort and we could not, therefore, obtain an estimated %abv. In these two instances, we also found that fermentation times were much longer than normal. We are forced, therefore, to conclude that these “thick mash” cases contained residual starch or dextrin, even though the iodine test was negative. We theorize that the thickness of the mash impeded both the GA enzyme from getting to some starch molecules and also impeded this starch from reacting with the iodine in the normal manner. Our conclusion is that such a thick mash (really thick wort) should be thinned out by adding extra water, until a hydrometer will float in it. We did not, however, validate this extra thinning process by actual experiment.

We always fermented at ambient room temperature in a heated and air conditioned house. Our location (northern California) is characterized by 2 seasons: a summer season, which is hot and sunny and a winter season, which is generally cold and rainy. Most of our experiments were conducted in the summer season. The indoor temperature in the summer season was held to a maximum daytime temperature of 76 degrees F by air conditioning. Nighttime temperatures would generally remain above 70 degrees F but could be as cool as 68 degrees F. The very last set of experiments occurred when the season suddenly changed to winter, and indoor temperatures were generally held to 68 degrees F during the day and 64 degrees F during the night via central heating.

We found that fermentation times lengthened significantly when the sudden seasonal change occurred. This 7 degree F or so mean temperature difference caused fermentation times to almost double! We therefore conclude that outdoor fermentation apparatus should be provisioned with some means to keep fermentation vessel temperatures above 75 degrees F, or accept significantly longer fermentation times (in excess of our 7 day requirement).

In no instance did ambient room temperature get higher than 76 degrees F. The manufacturer's stated temperature for rehydrating our distillers yeast was between 93 and 97 degrees F. There

was no danger of killing our yeast due to excessive ambient temperature during fermentation. However, outdoor equipment in hot climates may need some means of keeping fermentation temperatures below that which would kill the yeast. Yeast fermentation is somewhat exothermic and will tend to self heat, albeit our small samples did not exhibit any noticeable self-heating.

We used 2 glass carboys to ferment our 1 gallon of wort. The carboys were each 1 gallon in capacity, but we needed two of them because (a) the insoluble mass and dead yeast (lees) added to the volume of the water, and (b) substantial foaming action needed “headroom” in the fermentation vessels or else it would foam out through the airlock.

### **3.3.11 Distillation.**

As of this writing, we have not obtained a permit to allow us to perform laboratory distillation of the beer that we created in our various experiments. We are storing the beer in corked (cleaned and sterilized) wine bottles until our permit application is approved.

The distillation process itself was never part of our experimental objectives. However, we wish to perfect our skills at distillation and to then use these skills to distill the beer from our experiments in order to determine the actual alcohol content that we achieved. As of this report writing, we have only been able to estimate the %abv of our beer via triple scale (sugar) hydrometer testing of the wort. In some instances, the wort was too thick to even float the hydrometer and therefore we do not have %abv estimates for these experimental runs. We had originally thought that we could read the alcohol content of the beer directly via an alcohol hydrometer. However, residual soluble and non-soluble material in our beer consistently gives us a specific gravity reading above 1.000, even though we are certain that fermentation has completed, as noted in the subsection on Fermentation, above. We currently believe that if we distill the beer to near azeotrope, we will leave this other material behind and consequently be able to accurately and definitively determine ethanol content via direct volume measurement.

We have a laboratory glassware reflux still available to us and permission to perform our distillation in a certified bio-chemical laboratory. We originally contacted the TTB (US Treasury Bureau of Alcohol and Tobacco Tax and Trade; <http://www.ttb.gov/>) more than 8 months ago. They informed us that there are no minimums below which we could distill without a permit, and we therefore submitted a permit application. The application has bounced back and forth a number of times since then due to two factors: (1) change of TTB personnel assigned to review and approve our permit (with differing opinions about what is needed coming from the different people involved), and (2) complexities in our relationship with the laboratory. Specifically, the laboratory that has granted us permission to distill in their facilities leases the physical space from a commercial owner and TTB has been confusing, unclear, and changing in their opinion about what documentation they need to approve our permit (e.g. the latest iteration is TTB wanting a copy of the lease agreement between the laboratory and the property owner, in addition to our agreement with the laboratory and a written certification from the property owner that on-site distillation is acceptable to them).

We intend to continue to pursue our permit and to distill the beer that we now have in storage. We intend to update this *preliminary* report to a *final version* when we have actual, finalized data from distillation.

### **3.4 Further Discussion of the Findings.**

We achieved the objectives set forth in section 3.2, above, with results summarized in section 3.1 and expanded upon in section 3.3. After completing this activity and documenting our experimental results, we believe that there are additional experiments that might be useful to perform, albeit outside of our original scope of work. We currently have no specific plans to perform these additional experiments, but we wish to document our thoughts and ideas here.

#### **3.4.1 Yeast Starter and Nutrient.**

We reported on the yeast starter that we used in section 3.3.3, above. This yeast starter approach worked well for us and gave us confidence that if fermentation ever did not start up, it would be because of something in the wort and not a problem with getting the yeast rehydrated and growing strongly. It is because of this that we believe that a yeast starter is necessary for the “urban producer” model, as this model needs a guarantee that fermentation will start up and complete without anyone present on-site to monitor it. We noted in section 3.3.3 that on two occasions our initial yeast starter failed and we had to redo it. However, fermentation never failed with this yeast starter approach.

The problem now is that we do not currently know exactly how to scale this for pilot and production scale use. Our yeast starter batch size was ½ cup. This worked well for our 1 gallon wort batches. It makes sense that linearly scaling the starter solution size to the wort size would produce similar results. Therefore, we could say with some confidence that a 500 cup yeast starter would work well for a 1000 gallon wort. However, since yeast is a living, growing substance, we wonder if linear scaling is necessary. A smaller size yeast starter might take a little bit longer to get vigorous fermentation going, but we do not know how much longer. One of our key objectives is reduction in consumable costs, and reduction in the yeast used as well as reduction in sugar would have some impact on the overall cost per gallon of the resulting fuel.

A second issue is that our yeast starter has a pH of around 8.8, whereas the wort has a target pH of 4.0 – 5.0. This significant difference in pH does not appear to have deterred the yeast starter from adapting to the wort. Nevertheless, we wonder if a yeast starter medium that was closer in pH to the wort would produce even better results (i.e. faster fermentation startup). For example, would rehydrating the yeast in warm orange juice (more acidic than sugar-water) produce a superior starter solution for this application? We believe that this question is worth exploring.

The cost of yeast for fermentation is hard to estimate, as yeast is a living organism that can be cultured at home. The cost of our yeast nutrient, on a per gallon of fuel ethanol basis is substantial, but we used small packages and we did not experiment with reduction or elimination



of the nutrient because fermentation costs were beyond the scope of our study. We also believe that the cost of the DAP yeast nutrient can be substantially reduced when purchased in quantity, and that the nutrient can be synthesized at “home scale” for even lower cost. However, the topic of yeast and nutrient cost should be explored in further detail.

### 3.4.2 Cleaning and Sterilization Procedures.

The literature is clear that proper cleaning and sterilization of equipment is essential to successful ethanol production. Consequently, we paid careful attention to this in our experiments. Prior to each and every experiment, we thoroughly hand washed and rinsed each piece of equipment using dishwasher soap and tap water in a clean, stainless steel, kitchen sink. We carefully hand dried each piece with clean towels and then sterilized each piece using a hand held steamer. We made sure to place each cleaned piece of equipment on a clean surface prior to use. The cleaning and sterilization process took place within one hour (often less) of the beginning an experiment. After each experiment was concluded, we again hand washed and rinsed each price of equipment until it was completely clean of all feedstock derived material. Where possible, we ran the equipment through a kitchen dishwasher with sterilization cycle after hand cleaning it. In instances where significant mold was present in the feedstock, we performed post-experiment washing of the equipment with a chlorine based cleaning solution.

We believe that the objectives of our cleaning and sterilization procedures must be carried through as feedstock processing is scaled up to pilot and production sized equipment. However, these exact procedures are not scalable to large vessels that cannot be hand washed in a kitchen sink or dishwasher. We believe that different procedures must be used at a larger scale that results in the same (or better) level of infection control as in our experiments. Perhaps pressure washing with a commercial, food grade sanitizing agent would be a suitable approach. We believe that procedures need to be developed that are suitable for production scale equipment cleaning and sanitizing.

### 3.4.3 Shortest Time Processing.

Our goals and objectives led us to try and minimize cooking temperature and enzyme quantities while staying within our 5 hour time limit. As such, we did not gather sufficient experimental information to report out what conditions of cooking temperature and pH produce the *shortest possible* processing time. We did observe that, to some degree, cooking at higher temperatures seemed to shorten the minimum necessary cooking time. However, the result is not linear and we did not run enough samples through at specific higher temperatures (higher than our final 115 degrees F result) to quantify the gain in cooking time. Likewise, we did not intentionally vary the pH of our mashes to see what the tradeoff would be between increasing acid (to lower the pH) and possible decrease in cooking time. We are satisfied that our final results meet all of our experimental goals and objectives, but it would be interesting to explore such potential optimizations further.



## **4 Measurement Equipment, Materials and Consumables.**

This section describes the measurement equipment, materials and consumables that we found necessary and useful to process our bakery waste feedstock into ethanol for fuel. The items described in this chapter, or their functional equivalent, are needed for pilot and production scale implementation of our experimentally-determined process, described in section 3.1 above.

### **4.1 Laboratory Measurement Equipment and Materials.**

#### **4.1.1 pH measurement.**

We elected to use a low cost pH meter: Milwaukee Instruments PH600AQ



**Milwaukee Instruments PH600AQ**

The meter can be purchased from:

[http://www.amazon.com/Milwaukee-Instruments-PH600AQ-Tester-Calibration/dp/B005H78ZI0/ref=sr\\_1\\_1?s=industrial&ie=UTF8&qid=1327379811&sr=1-1](http://www.amazon.com/Milwaukee-Instruments-PH600AQ-Tester-Calibration/dp/B005H78ZI0/ref=sr_1_1?s=industrial&ie=UTF8&qid=1327379811&sr=1-1)

We found this meter to be generally reliable and to hold its calibration reasonably well – most of the time. Occasionally, it required recalibration on almost every measurement. Calibration is simple, but touchy. The meter is not as accurate as laboratory quality equipment using 3 point or more calibration, but it seems to be fine for the purpose of adjusting pH in a mash.

It is mandatory to have buffered calibration solution on-hand and highly recommended to store the meter with the probe immersed in the calibration solution. We used General Hydroponics standard reference solution pH 7.0, available at:

[http://www.amazon.com/General-Hydroponics-7-0-Calibration-Solution/dp/B001D0CKYK/ref=pd\\_sbs\\_indust\\_2](http://www.amazon.com/General-Hydroponics-7-0-Calibration-Solution/dp/B001D0CKYK/ref=pd_sbs_indust_2)



#### **pH Calibration Solution.**

It is also necessary to have distilled water available. The pH meter is calibrated using the calibration solution, then cleaned in distilled water, the measurement taken, cleaned again in distilled water, and replaced in the calibration solution for storage. Ordinary gallon jugs of distilled water can be purchased inexpensively in any grocery or hardware store.

#### **4.1.2 Starch Test.**

We used the standard Lugol's solution (2% iodine) to test for the presence of starch.



#### **Lugol's solution.**

It can be purchased from:

[http://www.amazon.com/J-Crows-Lugols-Iodine-Solution-2/dp/B001AEFM9Y/ref=sr\\_1\\_1?ie=UTF8&s=hpc&qid=1304715200&sr=1-1](http://www.amazon.com/J-Crows-Lugols-Iodine-Solution-2/dp/B001AEFM9Y/ref=sr_1_1?ie=UTF8&s=hpc&qid=1304715200&sr=1-1)

The solution comes with a dropper cap. We took small samples in white measuring cups from the measurement set, below.

#### 4.1.3 Sugar Hydrometer.

A “triple scale” sugar hydrometer and test jar is a required piece of equipment. A typical hydrometer is:



**Hydrometer and Test Jar.**

The hydrometer can be purchased at:

[http://www.amazon.com/RiteBrew-Hydrometer-Triple-Scale/dp/B000E60U6Y/ref=pd\\_bxgy\\_misc\\_img\\_c](http://www.amazon.com/RiteBrew-Hydrometer-Triple-Scale/dp/B000E60U6Y/ref=pd_bxgy_misc_img_c)

The corresponding test jar is:

[http://www.amazon.com/Monster-Brew-Home-Brewing-Supplies/dp/B0067MZP64/ref=pd\\_sbs\\_misc\\_23](http://www.amazon.com/Monster-Brew-Home-Brewing-Supplies/dp/B0067MZP64/ref=pd_sbs_misc_23)

#### 4.1.4 Laboratory Thermometer.

We used a temperature probe and homemade controller to monitor and control cooking temperature. Nevertheless, a decent digital laboratory thermometer is needed for measuring temperature for yeast rehydration, yeast pitching in the wort, and temperature control for pH and hydrometer measurements. We used the following thermometer:



**Laboratory Thermometer.**

This thermometer can be purchased at:

[http://www.amazon.com/Fisher-Scientific-Digital-Thermometers/dp/B0015SGXAM/ref=sr\\_1\\_1?s=industrial&ie=UTF8&qid=1327380414&sr=1-1](http://www.amazon.com/Fisher-Scientific-Digital-Thermometers/dp/B0015SGXAM/ref=sr_1_1?s=industrial&ie=UTF8&qid=1327380414&sr=1-1)

#### **4.1.5 Laboratory Digital Scale.**

Weight measurement for dry acid and dry enzymes requires an accurate digital scale. For our one gallon mash sets, we required measurement accuracy to 0.1 gram and thus we selected this handy, battery operated pocket scale:



**Pocket Scale.**

This scale is available at:

[http://www.amazon.com/American-Weigh-Signature-Digital-Pocket/dp/B002SC3LLS/ref=pd\\_sbs\\_indust\\_1](http://www.amazon.com/American-Weigh-Signature-Digital-Pocket/dp/B002SC3LLS/ref=pd_sbs_indust_1)

This scale can weigh up to 1000 grams. It can therefore support dry acid and dry enzymes for batches as high as 100 gallons of mash. A larger scale would be required for larger batches, for which 1.0 gram increments would be sufficient.

We used 4 cup paper coffee filters to hold the dry material for weighing. The coffee filters worked for this application, but weight measurement was difficult because the filters were a little too flexible and the center of mass shifted on the scale. This solution is usable and we did not need to find another solution, but we recommend using light, rigid plastic measuring cups designed for laboratory use. The plastic measuring cup set that we purchased, below, was good for volume measurement but these cups are too massive for weight measurement to within 0.1 grams.

### 4.1.6 Measurement and transfer.

It is handy to have a set of measuring cups and spoons, such as:



**Measuring cups and spoons.**

This set can be purchased from:

[http://www.amazon.com/Progressive-GT-3520-International-19-Piece-Measuring/dp/B0014Y4X3G/ref=pd\\_sbs\\_indust\\_1](http://www.amazon.com/Progressive-GT-3520-International-19-Piece-Measuring/dp/B0014Y4X3G/ref=pd_sbs_indust_1)

We also found it handy to have a turkey baster for extracting samples of mash and wort, such as:

[http://www.amazon.com/Heat-Resistant-Nylon-Baster-Rubber/dp/B002U9JSIE/ref=pd\\_sim\\_sbs\\_k\\_3](http://www.amazon.com/Heat-Resistant-Nylon-Baster-Rubber/dp/B002U9JSIE/ref=pd_sim_sbs_k_3)



**Handy Turkey Baster.**

## **4.2 Consumables and Consumable Costs.**

### **4.2.1 Feedstock.**

We tested a wide variety of “bakery waste” feedstock including many types of bread (white, wheat, sourdough, pumpernickel, french, herb, etc.) in the form of no-preservative paper wrapped loaves, preservative added plastic wrapped loaves, rolls, and cocktail slices. We also test various types of bagels, muffins, pastries, and doughnuts. We basically found that all of this material works. We only used feedstock that was no longer edible and is available at no cost (headed for the dumpster). 26 lbs of feedstock makes 10 gallons of mash and approximately 1 gallon of azeotropic ethanol.

Mr. Jack Cutter provided us with the feedstock material. The material was donated to the agency he works for by two Safeway supermarkets and one Starbucks coffee shop. We only accepted material that became inedible and was headed for the dumpster.

### **4.2.2 Water.**

Ten gallons of fresh water are needed for every 26 lbs of feedstock to make a mash with approximate fuel grade ethanol content after processing and distillation of 1 gallon. We did not assess the cost of water in our analysis, but the stripped beer after distillation should definitely be recycled and put to useful purpose, either for another batch of mash or some other use.

### **4.2.3 Acid.**

Sourdough feedstock may not require pH adjustment, but other feedstock has a higher natural pH than needed for processing with GA enzyme and for fermentation. We used dry citric acid to lower the pH, when needed, for our experimental 1 gallon mash batches. We purchased 2 oz (56.7 grams) packets from Mile High Distilling for this purpose (cost = \$1.25 per package):

<http://www.milehidistilling.com/citric-acid-2oz/>

Since we generally required 2 – 5 grams of citric acid to pH adjust our test meshes (if needed at all), one packet is sufficient for 10 - 25 experiments. The amount of citric acid would need to scale linearly with the batch size, so a 100 gallon mash batch would require 200 – 500 grams of

citric acid. The cost, therefore, is somewhere in the neighborhood of \$0.50 - \$1.25 per gallon of fuel grade ethanol. This is somewhat outside of our cost range, but we believe that larger and much more cost effective size packages of citric acid can be purchased, or a stronger liquid acid could be used with proper safety precautions.

#### 4.2.4 GA Enzyme.

We originally used a dry powdered GA enzyme at \$14.99 for a 1 lb bag:

<http://www.milehidistilling.com/products/gluco-amylase-enzyme-1-pound.html>

We found that we needed 4.8 grams of this enzyme per 1 gallon mash batch. This equates to 0.01 lb of enzyme per gallon of mash, or 1 lb per 100 gallon mash batch, thus \$1.50 per gallon of fuel grade ethanol (at 10% abv for the mash). This cost exceeds our goals and objectives, but the packages are small. We also investigated and experimented with a liquid GA enzyme available in larger quantities from an industrial distributor. We obtained samples of the SEBamyl-GA enzyme for testing from Specialty Enzymes of Chino, CA ([www.specialtyenzymes.com](http://www.specialtyenzymes.com)). We obtained a price quote for a 25 KG (5 gallon jerry can) of this enzyme for \$12.95. 5 gallons = 18,927 ml. At 0.8 ml of GA enzyme per gallon of mash, one jerry can will process over 23,000 gallons of mash, or approximately 2,300 gallons of fuel ethanol (at 10% abv). That's less than a penny per gallon of ethanol! We also found that the liquid enzyme was easier to measure than the dry powdered form.

#### 4.2.5 Yeast and Yeast Nutrient.

We purchased standard distillers yeast for our experiments, at \$7.99 for a 1 lb bag:

[http://www.amazon.com/Distillers-Yeast-DADY-bulk-pack/dp/B0064O7T2I/ref=pd\\_sim\\_misc\\_1](http://www.amazon.com/Distillers-Yeast-DADY-bulk-pack/dp/B0064O7T2I/ref=pd_sim_misc_1)

There is the possibility to use faster (higher temperature) turbo yeast and also lower temperature one-step yeast, but we did not experiment with these other types. The cost of yeast per gallon of ethanol fuel is hard to predict because we use a yeast starter and culture the yeast prior to use. In theory, one small sample of yeast could be cultured and used indefinitely at a site, requiring sugar-water or sugary juice in order for the yeast to grow.

We assumed that our wort would be lacking in nutrients and purchased DAP yeast nutrient to add to our wort (at \$11.97 per 1 lb bag):

[http://www.amazon.com/Yeast-Nutrient-1-lb/dp/B0064H0MWY/ref=pd\\_bxgy\\_misc\\_img\\_b](http://www.amazon.com/Yeast-Nutrient-1-lb/dp/B0064H0MWY/ref=pd_bxgy_misc_img_b)

We used the recommended dosage of 1 tsp of nutrient (weight = 4.1 grams) per gallon of wort. We did not experiment with quantities of yeast nutrient to see if we needed as much (or needed it at all). A 1 lb bag should be good for over a hundred gallons of mash, or about \$1 per gallon of fuel grade ethanol. However, the nutrient can be purchased at a fraction of this cost in higher quantity and can be synthesized at home for even lower cost.



## **5 Detailed Description of Experimental Equipment.**

This section describes the equipment that we used to conduct our experiments with. This equipment is judged to be suitable only for experiments with small batches of feedstock and the particular equipment items described herein would not be expected to be suitable for pilot and production scale facilities.

### **5.1 Feedstock Preparation.**

The feedstock was sliced on a kitchen cutting board to fit in a standard kitchen food processor:



**Food Processor and Grating Blade.**

The grating blade, as shown in the figure above, was used to grate the feedstock into something resembling a course powder. The grated feedstock was placed into serving bowls and weighed until we obtained exactly 2.6 lbs. We used a standard digital kitchen scale to weigh out the feedstock, subtracting the weight of the empty serving bowls:

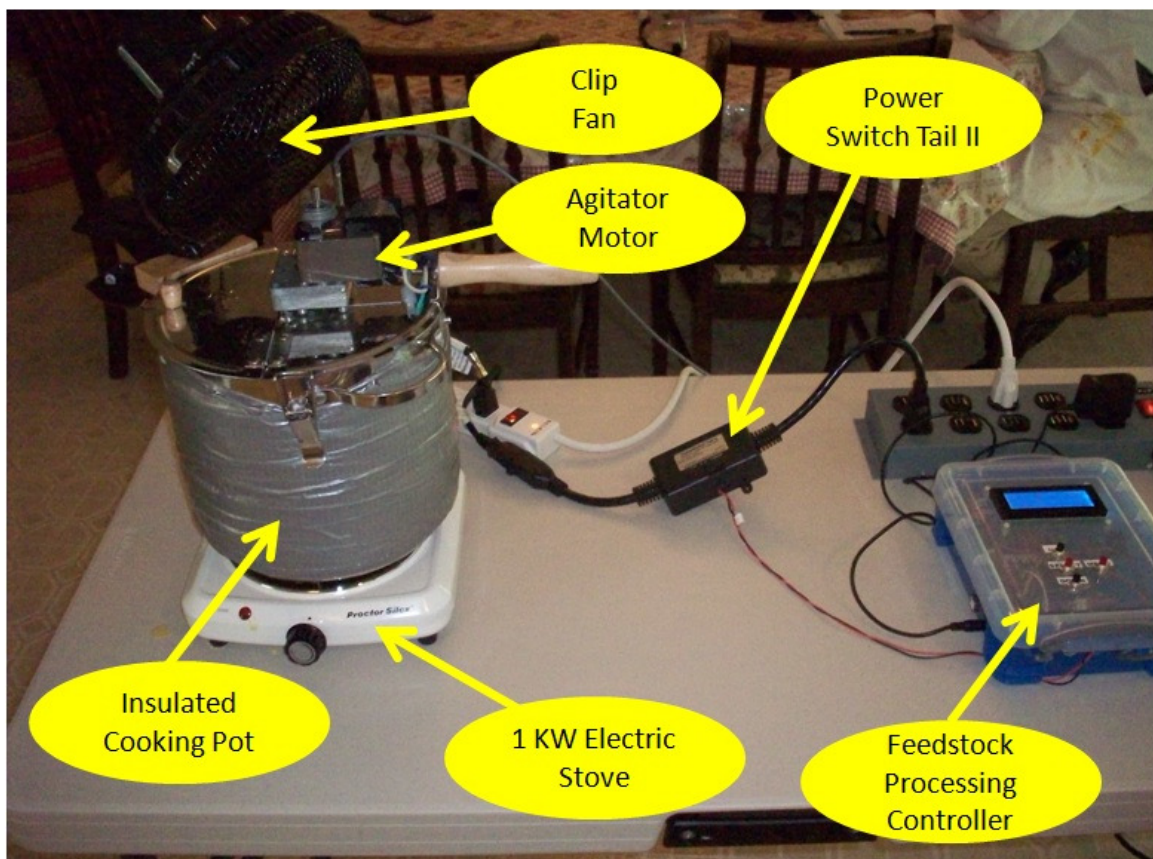


**Kitchen Scale for Weighing Feedstock.**

## **5.2 Cooking and Mash Processing Equipment.**

### **5.2.1 Overview.**

The figure below shows an overview of the equipment setup that we used for cooking the mash:



**Cooking Equipment Overview.**

A two gallon stainless steel cooking pot was insulated with fiberglass pipe wrapping and secured with duct tape. The pot cover was fabricated using a hinged cover from a stainless steel corn popper with a gearmotor mounted to the top turning an agitator mechanism within the pot. The pot cover was also fitted with a compression fitting for a temperature probe.

We found that the agitator motor that we selected required some air blowing over it or else the motor would overheat and stop running. We purchased a clip fan and clipped it to one of the pot handles, secured by a small block of wood. The airflow from the fan was more than sufficient to keep the motor cool.

Cooking heat was provided by a 1 KW electric stove. The stove power was supplied through a “power switch tail II” device, which is an opto-isolated relay with AC connectors built in. The AC power to the stove was controlled by a homemade “Feedstock Processing Controller”, which provided a number of useful functions within one device. A temperature probe fitted through the top of the cooking pot provided real-time mash temperature readings to the Feedstock Processing Controller, which in turn controlled the power to the stove through the Power Switch Tail II device. The agitation was constant; only the temperature was controlled.

The individual pieces of equipment are described further below.

### 5.2.2 Feedstock Processing Controller.

The Feedstock Processing Controller is an Arduino-based microcontroller with software specially designed to support this project. It is based upon the Open Source Controller design by Bob Glicksman, one of the authors of this paper. Complete hardware, software and documentation for this Controller can be found at:

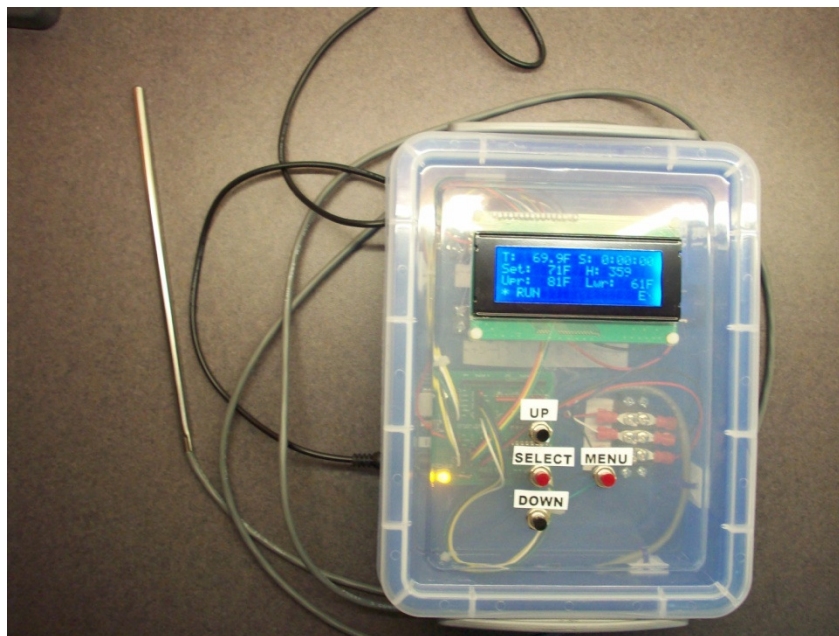
[http://www.liquidsunenergy.com/projects/projects\\_home.html](http://www.liquidsunenergy.com/projects/projects_home.html)

We had the Controller hardware handy and decided to use it to help instrument our experiments. However, there is nothing that the Controller does that could not be implemented with several, readily purchasable, off the shelf instruments.

The Controller performs the following functions:

- 1) Thermostatic temperature control of electric stove to regulate cooking temperature of the mash.
- 2) Mash temperature limit monitoring and alarming, in the event of a temperature control problem.
- 3) Countdown timer for the various steps in our experiments.
- 4) Stove AC power on time accumulator, to provide an estimate of cooking power consumption.





**Feedstock Processing Controller, with Temperature Probe.**

### **5.2.3 Power Switch Tail II.**

We used the Power Tail Switch II to allow the Controller to turn AC power on and off. This device contains an electromechanical relay with optically isolated digital control. It comes in a molded package with AC plug and socket connections, so that the AC power is completely insulated. The device is available from: <http://www.powerswitchtail.com/Pages/default.aspx>



**Power Switch Tail II with control wiring from the Controller.**

#### 5.2.4 Temperature Probe.

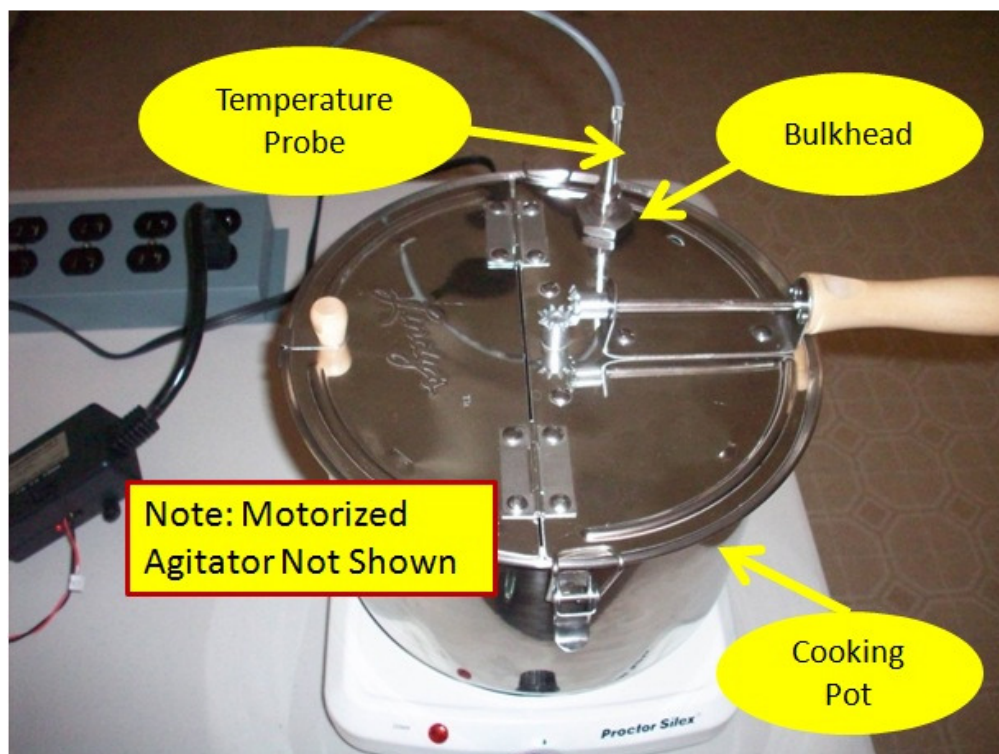
The Open Source Controller is designed to use DS18B20 temperature measurement chips. We elected to purchase a pre-made stainless steel temperature probe that uses this chip internally. The probe is available from:

<http://www.brewershardware.com/8-Temperature-Sensor-DS18B20.html>

The same vendor also sells stainless steel bulkheads with high temperature compression fittings to provide a pass-through for the probe through a vessel top or wall. The bulkhead is:

<http://www.brewershardware.com/WLFM14C14.html>

We used this bulkhead to anchor and seal the temperature probe through the top of our cooking vessel:



**Mounting of Temperature Probe in Cooking Vessel.**

#### 5.2.5 Electric Stove.

We purchased a 1 KW electric single burner stove from:

[http://www.amazon.com/Proctor-Silex-34101-Proctor-Silex-Burner/dp/B000690WNU/ref=sr\\_1\\_2?ie=UTF8&qid=1329871503&sr=8-2](http://www.amazon.com/Proctor-Silex-34101-Proctor-Silex-Burner/dp/B000690WNU/ref=sr_1_2?ie=UTF8&qid=1329871503&sr=8-2)

This stove has a temperature control knob to adjust an internal temperature thermostat. The internal thermostat is not calibrated, it is not accurate, and, in any event, it would not control mash temperature. We had to make sure to turn the control knob to the maximum (Hi) to disable this internal stove function. With the knob set to Hi, the Controller could regulate the stove by cycling the AC power on and off through the Power Switch Tail II device.

### 5.2.6 Cooking Pot Top and Agitator.

This one component of our experimental equipment required a great deal of fabrication. After several manual experiments on a stovetop, we realized that we needed a covered pot with easy access for sample taking and chemical introduction and, most importantly, continuous agitation. After some degree of research, we purchased a stainless steel 6 quart corn popper with a hinged top and manual crank driven agitator:

<http://www.amazon.com/gp/product/B0034D5BIQ>

The 6 quart pot was a little too small and the manual agitator was neither convenient nor capable of stirring the mash, which could be very thick before enzyme treatment. Our manual experiments led us to estimate that a gentle agitation at 30 – 60 rpm should do the job nicely, as long as the agitation was continuous. After some research, we found an inexpensive gearmotor with what we estimated would be sufficient torque to continuously stir the mash. The motor we found ran at 80 rpm, which was a little higher than we specified but performed the task nicely. The motor that we used is:

<http://www.surpluscenter.com/item.asp?item=5-1572&catname=electric>

The figure below shows the motor affixed to the stainless steel corn popper top:



**Motor Affixed to Corn Popper Top with Hinged Lid for Easy Access to the Mash.**



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We used the top and the cover securing hinges from the corn popper, but a larger, 2 gallon stainless steel pot. Our initial manual experiments led us to believe that a suitable agitator should provide a good vertical motion of the mash, as well as some sweeping action to keep the starch from sticking to the sides and bottom of the cooking vessel. Based upon these requirements, Mr. Gene Halverson (aircraft mechanic and expert machinist) produced the agitator design shown below, as well as mounted the motor to the corn popper lid and welded the cover securing hinges to this larger stainless steel pot.



**Agitator Detail.**

One final problem that we had was that the gearmotor was designed to have a fan blade moving air over it. Without cooling air running constantly over the motor, the motor would overheat and its thermal cutout would shut it off. We did not have a fan blade to fit the high speed shaft on the motor and even if we did, a high speed blade spinning on top of our cooking pot would be unsafe. We therefore purchased a “clip fan” and clamped it to one of the pot handles using a wooden block for support:

Clip Fan: <http://www.officedepot.com/a/products/769395/Holmes-Oscillating-Clip-Fan/>

The spring loaded clamp that holds the clip fan to the pot handle could easily be released (no tools required) to move the fan out of the way briefly whenever we needed to open the pot lid to take samples for pH testing, iodine testing, adding acid and adding enzymes.





**Clip Fan Providing Motor Cooling.**

We also decided to insulate the sides of the pot in order to minimize heat loss through the stainless steel pot walls, particularly as we had a fan blowing over the top. We wrapped the sides of the pot with fiberglass pipe insulation purchased from a local hardware store, and then secured it in place by covering it with duct tape (see picture above).

### **5.3 Fermentation And Bottling Equipment.**

#### **5.3.1 Fermentation Equipment.**

We used two, 1 gallon glass carboys as fermentation vessels.

[http://www.amazon.com/1-Gallon-glass-Jug/dp/B0064O8Z76/ref=sr\\_1\\_4?s=miscellaneous&ie=UTF8&qid=1327516457&sr=1-4](http://www.amazon.com/1-Gallon-glass-Jug/dp/B0064O8Z76/ref=sr_1_4?s=miscellaneous&ie=UTF8&qid=1327516457&sr=1-4)

It was necessary to use two vessels for a 1 gallon mash because fermentation was often vigorous and would foam out through the airlocks unless we provided a lot of headroom in the carboy.

We used S-Type airlocks to keep air out of the fermenters while allowing CO<sub>2</sub> gas to escape.

[http://www.amazon.com/RiteBrew-S-Shape-Airlock/dp/B0057JBABM/ref=pd\\_sim\\_misc\\_93](http://www.amazon.com/RiteBrew-S-Shape-Airlock/dp/B0057JBABM/ref=pd_sim_misc_93)

We prefer S-Type airlocks because it is very easy to see the pressure in the fermentation vessel and thereby assess fermentation progress. However, care must be taken to prevent foaming of material into the airlocks, as S-Type airlocks are extremely hard to clean once they are so fouled.

The figure below is a photo of one of our batches fermenting in the carboys and airlocks:



**Beer Fermenting in the Carboys.**

Heavy, but quite typical foaming can clearly be seen in the photo above. The clear ethanol/water beer is the layer below the foam and the lees and other non-soluble matter is at the bottom.

### **5.3.2 Bottling the Beer.**

We intend to distill the beer in order to positively measure the alcohol levels that we achieved. However, we have not received the government still permit as of the date of this report. Therefore, we decided to bottle up the beer after fermentation was complete and store until we are able to distill.

We used empty wine bottles and standard wine corks to store the beer. Before bottling, however, we found that we needed to strain the liquid beer out of our fermentation bottles, as the bottom part had a lot of non-soluble material in it but also contained a lot of liquid beer. Separation of this solid matter was quite difficult. We poured the material from the carboys through a fine kitchen strainer into a clean and sterilized 2 gallon stainless steel kitchen pot. We then passed this material through a very fine metal coffee filter into a second clean and sterilized 2 gallon stainless steel kitchen pot. The straining process was slow and tedious, requiring constant stirring to get the material through the filters. Even so, we found a lot of very fine matter mixed into the beer. We attempted a third stage filtering with paper coffee filters, but these clogged easily and we could not filter further. We observe that as the bottled beer sits upright in storage, some of this matter settles to the bottom of the bottles. However, if we are to distill all of the beer (which we need to do to obtain a true alcohol production measurement) we will have to rely on boiling in the still to leave this material behind.

We added a crushed Campden tablet (sodium bisulfate) to each beer batch prior to bottling to act as a sterilizing agent during storage. We hope that this will keep invasive bacteria (such as turns ethanol to vinegar) at bay until we can distill our product.

## **Appendix A. Author Biography.**

- **Bob Glicksman:** Bob holds a Masters degree in electrical engineering and has over 40 years industrial experience in the design, development and application of signal and image processing for military and medical applications. Since retiring in late 2009, Bob has been active in researching and supporting small scale fuel ethanol production, with the following accomplishments:
  - Co-developer and co-administrator (both with Lucy Geever) of the Liquid Sun Energy website, to promote and foster small scale community supported energy.
  - Designed and developed a microcontroller-based the Open Source Controller to support an open source distillation project.
  - Wrote software to adapt the Open Source Controller to support the needs of these feedstock processing experiments.
  - Authored and published a paper about using ethanol in internal combustion engines.
  - Co-author and co-researcher (with Mark Kent and Lucy Geever) of the feedstock processing experiments and of this report.
- **Lucy Geever:** Lucy is a flight instructor and Air Transport Pilot. She holds multiple flight instructor certificates. She has a BA in history. She is interested in sustainable manufacturing, sustainable agriculture, community supported agriculture and community supported energy. Her activities in the area of community supported energy include:
  - Co-developer and co-administrator (both with Bob Glicksman) of the Liquid Sun Energy website, to promote and foster small scale community supported energy.
  - Developer and Administrator of the “Alcohol Fuel – San Francisco Bay Area -- Community Supported Energy (CSE)” Facebook site.
  - Experimentation with small scale cultivation of energy crops, worms and composting.
  - Co-author and co-researcher (with Mark Kent and Bob Glicksman) of the feedstock processing experiments and of this report.
- **Mark Kent:** Mark is an organic chemist with over 20 years experience in industrial and commercial process chemistry. He holds a Masters degree in Chemistry and is active in science education and tutoring young people in scientific techniques, principles, applications and laboratory procedures. His activities in the area of community supported energy include:
  - Fuel production quality analysis techniques.
  - Chemical process analysis for ethanol production.

## **Feedstock Processing Experiments Preliminary Report**

- Co-author and co-researcher (with Lucy Geever and Bob Glicksman) of the feedstock processing experiments and of this report.