

The Arroyo Fermentation Process For Alcohol and Light Rum From Blackstrap Molasses¹

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This process is primarily an improved procedure for the most economical and efficient fermentation of blackstrap molasses for conversion of its sugars into alcohol or light rums. The process was developed during the period of the last World War, and was designed in the first place to provide a ready and profitable means of expansion to the Puerto Rican alcohol and rum industry at a time when expansion was greatly desirable, but when new equipment, or even enlargement of existing factories was precluded due to conditions created by the war itself.

After the pilot-plant tests conducted in semi-commercial scale at the "Destileria de los Asociados de Borinquen" of Hato Rey, Puerto Rico, proved the commercial value and efficiency of the process, it was soon adopted by this distillery and three others from among the largest distilleries on the island. In the course of time over 100,000 liters of Proof Spirits were being produced daily by this process on the island. The Arroyo Process thus provided means to greatly increase the daily molasses mashing capacity of these distilleries without the enlargement of their existing fermentation equipment. In some cases the increase in daily processing of molasses amounted to from 50% to 100% over former performances. Besides providing for this remarkable increase in productive capacity, the process also increased fermentation yields, and efficiencies and made great economies in production costs.

Scientific Foundations of the Process

From a scientific and technical standpoint the process rests fundamentally upon two bases:

(1) A pre-treatment process for the raw material BEFORE it enters the actual "Thin Mashing" stage for fermentation, through which chemical, physical, and biological modifications of this material are effected; transforming it into a much fitter and improved material for the practice of alcoholic fermentation.

During this pre-treatment of the molasses in

¹Due to lack of space, tables have been omitted.



Rafael Arroyo

the form of a "Thick Mash," purification and conditioning take place; with the following results:

(1) Elimination of a large proportion of molasses impurities such as ash, gums, other organic non-sugars, and mechanically dispersed solid impurities. Besides, occluded bad odorous gases and other volatile substances undesirable in the molasses are also eliminated.

(2) Elimination of all the vegetative flora of microorganisms that usually infect this raw material, causing many and varied troubles during, or after, the fermentation.

(3) As inversion of sucrose into the corresponding reducing sugars is effected during the pretreatment and after storage of the pretreated material, the distillery yeast used as fermentation agent will later find a medium rich in directly fermentable hexoses.

(4) The nutrient requirements for the yeast are readily and economically corrected during this operation, by adding the necessary nutriment when deficiencies have been found to exist after analysis of the raw molasses. The molasses in

the "Thick Mash" is thus converted into a perfectly conditioned raw material as regards nutrients for the nourishing of the pitched yeasts.

(5) Physical improvements are also effected by which the unhandy, viscous, sticky molasses, is converted into a readily fluid, easy to handle and to sample mass. In this way subsequent operations in the confection of the "Thin Mash" for actual fermentation, are greatly simplified.

(6) Through the withdrawal of a large portion of the non-sugars, the concentration of valuable fermentable sugars is increased. In like manner a better ratio of total sugars to ash is obtained. This fact is of the greatest consequence in securing later on quick fermentation, high yields, and high alcoholic concentrations in the resulting fermented mashes or "beers."

(7) Through the withdrawal of mineral constituents (except the very soluble salts of potassium that remain in solution) the purity and concentration of these potash salts are greatly increased when potash recovery from the slops is practiced at the distillery.

(II) A novel method of using this pretreated material, or "Thick Mash" during fermentation, based upon the incremental addition of the "Thick Mash" at selected and controlled periods, to the fermenters.

Through the application of this novel method of fermentation the following features are attained:

(1) Very high and unusual degrees of alcoholic concentrations are developed during fermentation. By this we mean alcoholic concentrations of between 10 - 12.5 percent by volume in the fermented mash.

(2) Due to these high alcoholic concentrations that are developed in the fermented mash, every fermenter is guaranteed against loss due to infection from detrimental bacteria, as the high alcoholic concentration serves as a preservative against such bacterial infections.

(3) Very high amounts of sugars are readily fermented without fear of reaching inhibitory concentrations of either sugars or non-sugars in the fermenting medium. Usually from 18.0 to 23.0 grams of total sugars are fermented per 100 milliliters of mash.

(4) With the same capacity of fermenter this process will produce from 50.0 to 100.0 percent more gallons or liters of spirits in the fermented mash than when the standard fermentation methods are followed. For instance, other methods of fermentation usually yield only from 5 to 7 percent alcohol by volume in the resulting "beers," while this method yields from 10 to 12.5 percent. And this is accomplished within the same or less time taken by other fermentation methods to ob-

tain from 5 to 7 percent alcohol in fermented mash.

(5) The fact that this method of fermentation is able to produce such high percentages of alcohol in a short period of fermentation, accompanied by high yields on sugars used, accounts for high economy of production and low expenses for power and steam.

Blackstrap Molasses As Raw Material

Blackstrap molasses as a raw material for alcohol and rum fermentation is a substance of variable composition. Molasses from different countries varies a great deal in physical and chemical characteristics, and even that produced within a single country or locality will exhibit variations due to the cane variety from which the blackstrap was produced, the cultivation methods followed at the plantations, physical and chemical characteristics of the soils, fertilizing practices followed, the methods of sugar manufacture employed at the factories, and the quality of the marketable sugars produced. Some of these differences in the constitution of molasses bear a direct influence on the suitability of the blackstraps for alcohol and rum fermentation. In extreme cases (as for instance Hawaiian blackstrap) the unsatisfactory composition of the raw material has prevented the development of a profitable alcohol and rum industry in that country.

Among the most important shortcomings of blackstrap molasses from the standpoint of their fitness for rum and alcohol fermentation, may be mentioned: (1) low total sugars, (2) high total non-sugars, (3) high ash, (4) high gums, (5) deficiencies in yeast nutriment, especially as to nitrogen and phosphorus; (6) variable degrees of microbiological contamination. The presence of the high non-sugar constituents has made it necessary to employ relatively high dilutions at mashing, thereby further augmenting the deleterious effects of low total sugars and nutriment deficiencies.

One of the objects of the process herein described further on, is to take account of the natural deficiencies of blackstrap molasses, and in a large measure to correct them; and therewith in a practical and inexpensive way to increase the alcoholic concentration of the fermented mash, to raise the alcoholic yields based upon the total sugars content of the substrate, and greatly to improve the molasses mashing capacity of the distillery without sacrificing high fermentation efficiencies or the length of the fermentation cycle.

It has long been recognized by alcohol and rum producers, as well as by fermentation experts and research workers, that the desired improvements in rum and alcoholic fermentation comprise higher economy in production, less capital outlay for initial installations, higher purity and increased

value of by-products, higher alcoholic concentrations in fermented mashes, maximum distillery output per dollar invested, and in general, higher yields and fermentation efficiencies. A fermentation process that will fulfill the above desiderata will then represent a true step of progress in rum and alcoholic fermentation. With these aims in view was the process under discussion developed.

Accomplishments of Objectives

The first desideratum, that is, that comprising the favorable changes in the chemical composition of the raw material, is accomplished by the preparation of a purified and conditioned "Thick Mash," which is conditioned and purified through: (1) a great reduction in the ash content, (2) a similar reduction in the total non-sugars, (3) an increase in total sugars concentration in the molasses due to the removal of non sugars; (4) an increase in the ratio of invert sugars to sucrose, and of total sugars to ash; (5) correction of pH value and of yeast nutriment deficiencies; and (6) elimination of all forms of vegetative microorganisms that may contaminate the substrate.

The second desideratum, that is, that pertaining to increases of alcoholic concentrations in the fermented mashes, increased yields and fermentation efficiencies, and cost savings, is obtained through the use of the conditioned and purified "Thick Mash" in a special fermentation procedure whose main feature depends on the incremental feeding of the "Thick Mash" to the fermenters.

A feature of the process therefore, is the production of a "Thick Mash" which is conditioned and purified as above stated, and to effect the fermentation by the employment of successive portions of this "Thick Mash" under controlled conditions.

Detailed Mechanism of the Process

The process starts with the weighing of the molasses and the water to be employed in the preparation of the "Thick Mash." The proportion of the diluent varies with the Brix density of the molasses to be used; but it may be stated that enough water is added so that the resulting "Thick Mash" will have a Brix density within the range 55 - 60 degrees. Corrections for nitrogen and phosphorus deficiencies are made, if necessary, by the incorporation of the calculated amounts of ammonium sulphate and calcium superphosphate respectively. Under Puerto Rican conditions it has been found that the addition of the ammonium salt up to about 0.5% on the weight of the molasses, and of the superphosphate up to about 0.1% also on the weight of the molasses, is almost always satisfactory. The final ratio of phosphoric acid (as P₂O₅) to nitrogen should, however, be not less than 1:5, nor greater than 1:2; for best results during fermentation. Similarly the ratio of total sugars to ash should never fall below the value

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of 6.5:1 if a rapid and vigorous fermentation is to be expected. The next step is the adjustment of the pH value. This is done by the incorporation of concentrated sulphuric acid to obtain a value within the range of 4.5 - 5.2 pH in the "Thick Mash." Commercial grades of all chemicals involved are used. The ammonium sulphate and the calcium superphosphate may be the same quality used as fertilizing material.

The accompanying drawing conventionally illustrates the successive steps in the practicing of the process.

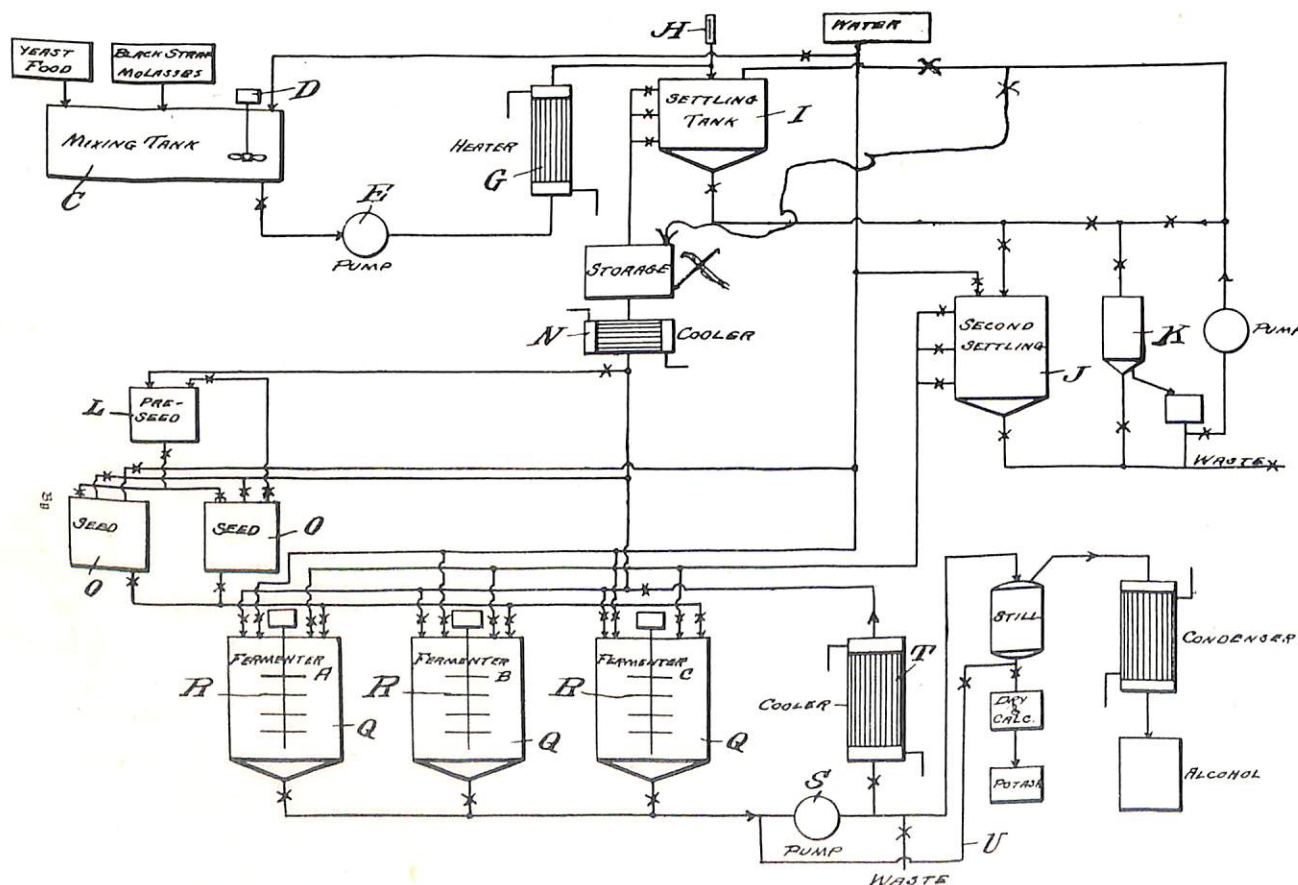
After thorough mixing of the above mentioned ingredients in the mixer tank (C), a "Thick Mash" of from 55 - 60 degrees Brix, and with a pH value between 4.5 - 5.2, results. This "Thick Mash" is then pumped through a multitubular steam heater (G), where its temperature is raised to exactly 80.0 degrees Centigrade. To facilitate the temperature control, a variable speed electric pump (E) is used to force the liquid through the heater (G), and the heater is provided with necessary regulating standard Centigrade thermometers. While the "Thick Mash" is being pumped through the heater (G) it must be kept well stirred up, so as to prevent the leaving of precipitated impurities at the bottom of tank (C). For this purpose the "Thick Mashing" tank (C) is provided with an agitator such as the motor driven mechanical stirrer (D).

From the steam heater the "Thick Mash" is

discharged into the "Thick Mash" receiving and settling tank (I), of which one or several may be in use. This tank is a cylindrical iron tank preferably insulated for the conservation of heat, and having a conical bottom. The conical section should have a capacity equal to about 20% of the total volume of "Thick Mash" to be stored in the tank. In this tank the "Thick Mash" is stored hot, and is allowed to settle for at least eight hours. The impurities of the "Thick Mash" will settle down into the conical bottom by this time, leaving a clear liquid on top. The temperature sustained in the "Thick Mash" results in the pasteurization of the liquid, so that essentially a sterile "Thick Mash" is provided, and at the same time the sucrose of the total sugars is permitted to reach a substantial degree of inversion through the action of temperature and the acidity of the medium. In this way the total sugars become high in directly fermentable hexoses.

The precipitated organic and inorganic impurities settle down into the conical bottom of the tank, mixed, of course, with some of the liquid "Thick Mash." This sedimentation is disposed of at this stage of the process in either or both of two ways:

(1) The sediment in the form of magma may be dropped into tank (J) situated underneath the thick mash receiving tank (I), where it may be diluted with water and well stirred up. This further dilution permits a quick second sedimentation, the solid impurities settling at the bottom, leaving



Diagrammatic sketch of the Arroyo Process of fermentation for alcohol and rum.

clean sweet water above. This sweet water is used in the preparation of "Thin Mash" at the fermenters. The solid impurities are either washed off into the sewer, or centrifuged in the special centrifuge (K) for further recovery of sweet water, and then dumped out.

(2) The sediment may be fed directly to the centrifuge (K) that will separate the solid impurities from the material, returning the clean run-off to either the "Thick Mash" receiving tank (I) or to the storage tank (X). Procedure No. 2 is to be preferred in most cases.

The conditioned and purified "Thick Mash" in tank (I) is now ready for the preparation of "Thin Mash" for fermentation purposes. Immediately before using, the "Thick Mash" is passed through a heat exchanger, or cooler (N) in order to bring its temperature down to about 40 degrees Centigrade, or less. This "Thick Mash" cooler is connected directly to the yeasting section (L), the seed tanks (O), and the main fermenters (Q), for supplying all of them with "Thick Mash" for the preparation of "Thin Mash" used in fermentation. At the yeasting section (L) provision is made for the preparation of absolutely sterile "Thin Mash" that is used for the further propagation of the laboratory pure yeast starter. All other "Thin Mash" are prepared directly from the "Thick Mash" through merely diluting it to the desired density.

The actual fermentation is carried on in the following manner:

The fermenters (Q) are of the closed type, and preferably constructed of polished iron. They are equipped with mechanical stirrers (R) and with recording thermometers and pH indicators. They are also connected to a circulating pump (S) that draws the fermenting liquid from a point near the bottom of the fermenter and circulates it through cooler (T), delivering the cooled mash at the top of the fermenters. The end of the delivery pipe is actually submerged into the upper layers of the fermenting liquid. The cooling device is situated outside the fermenters, and one such cooler and pump can take care of three fermenters. This device is used for maintaining the control of the temperature of the fermentation. The obtaining of high alcohol in the fermenter depends in a great measure upon the proper and efficient working of this cooling arrangement. Therefore, fermentation temperature is rigorously controlled between 28 and 33 degrees Centigrade by circulating the fermenting mash through the cooler as soon as the temperature of the liquid approaches 33 degrees Centigrade.

The first step in the preparation of "Thin Mash" at the fermenter is to introduce into the fermenter all of the water of dilution that is to be used as

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such for the fermentation operation. Then 0.5 of the total amount of "Thick Mash" to be used is added gradually, with continuous stirring to insure thorough mixing. In this way an initial "Thin Mash" is prepared within the fermenter, having a Brix density that may vary between 20 and 23 degrees. The pH is readjusted, if necessary, and the mash is then inoculated with a footing or seed equivalent to from 10 to 15 per cent the volume of total "Thin Mash" to be made. Gentle stirring of the liquid in the fermenter is maintained while the yeast footing is being incorporated. Fermentation usually starts within one hour from the time of seeding. When the Brix density of the fermenting liquid has dropped to one half or less of its original value (usually after 12 to 14 hours), another increment of "Thick Mash" is passed into the fermenter, taking care to keep the liquid well in motion with the mechanical agitator while this new increment is being added. In order to avoid heavy foaming at this time, a small amount of Turkey Red Oil (sulphonated Castor Oil) is previously added to the liquid in the fermenter. The amount of increment of "Thick Mash" added this time may amount to 0.3 of the total "Thick Mash" to be added to the fermenter. Any adjustment of pH necessary may be accomplished at this time. Fermentation is allowed to continue during about 6 more hours, and then the last increment of "Thick Mash" is added to the fermenter, observing the same precautions as before. This last increment will amount to 0.2 of the total "Thick Mash" to be added. Thus the total "Thick Mash" has been introduced by adding 0.5, then 0.3, and lastly 0.2 of the total. That is, during the first 18 or 20 hours of fermentation all of the "Thick Mash" has been passed into the fermenter; and from 10 to 16 hours after the addition of the last increment, fermentation will have finished in most cases, thus making a total fermentation time of from 30 to 36 hours. "Beers" of from 10 to 12.5 percent alcohol by volume are obtained. The yields of alcohol are based upon the total sugars (as invert) vary between 44.0 and 48.0 percent by weight. Fermentation efficiencies (based upon a maximum possible industrial yield of 48.55 percent alcohol on the weight of total sugars) may vary in the range 90.00 to 98.0 percent. The concentration of alcohol in the fermented mashes will be influenced to a large extent by the original quality of the raw material. Blackstrap molasses of very low total sugars content and high specific gravity will yield maximum alcoholic concentrations of around 10.0 to 11.0 percent by volume of fermented mash; while molasses of initial low non-sugars and high total sugars will produce alcoholic concentrations of between the values 11.0 and 12.50 percent by volume. In the case of blackstraps of exceptionally high quality, alcoholic con-

centrations of 13.6 percent by volume have been obtained in industrial practice of the process.

The above described procedure of fermentation may be modified in order to: (1) Increase the simplicity of operations; (2) Reduce initial cost of installation; (3) Reduce the operating expense, especially as to the use of steam; (4) When fuel is very expensive or difficult to obtain.

In such cases the heat treatment during the "Thick Mashing" operations is eliminated, and the pH adjustment takes place within the more acid range of 4.0 to 4.5 pH. The "Thick Mash" prepared in mixing tank (C) is in this case directly centrifuged in special centrifuge (K), for elimination of precipitated solid impurities. The clear run-off is pumped to storage tank (X), from where it is distributed for use in the formation of "Thin Mash." The separated solid impurities are introduced in tank (J) made into the form of a magma again by dilution with water, and recentrifuged for the recovery of the sugars left in them during the first separation of solids. The second clean run-off is sent either to storage tank (X) or directly to the fermenters for the making of "Thin Mash." We have, then, that in this modification some of the most expensive pieces of equipment are eliminated, and also floor and head space. Among the eliminated equipment we have: heater (G); receiving and settling tank (I), and cooler (N). Moreover, steam and cooling water are also saved.

While these remarks are made upon the saving of steam in this modification of the process, it is well to bear in mind that even when the heat treatment is given to the "Thick Mash" the process also saves steam to the distillery. The small amount of steam used at this stage of the process is more than repaid many times by the great economy that results during distillation of the high alcohol "beers" produced. Also in the great reduction in volume of mash to be prepared, fermented, and distilled for the obtainment of a given amount of alcohol or rum.

Also there exist ways to economize steam when the heat treatment is used. One of these ways is the use of hot water from the condensers of the still in the confection of the "Thick Mash." In a large installation of the process the saving of heat units by the use of this expedient amounted to 40.0 percent of the heat units formerly used when using cold water in the mashing tank (C).

In cases when the distillery works as an adjunct of the sugar factory and is located at convenient distance from it, another opportunity presents itself for the economy of heat during "Thick Mashing." This is effected by using exhaust steam from the sugar factory for heating the "Thick Mash." Still another optional manner to prepare the hot "Thick Mash" with the saving of heat

units, is to use hot clarified cane juice from the sugar factory instead of cold water in the mixing of molasses for this purpose. This method is highly beneficial when dealing with very poor grades of blackstrap molasses, since a solution of high purity and rich in sugars is added. This method is particularly advantageous when the distillery is engaged in the production of high class rums. The weight of clarified, dilute juice to be added in such practice will represent about 50.0 percent of the weight of the blackstrap molasses used in the preparation of the "Thick Mash."

The disadvantages of using the modification involving "Thick Mashing" by the cold process are: (1) The sterilizing effect of the added heat is, of course, lost. (2) The "Thick Mash" must be used daily as prepared; that is, it will not stand storage for a long time after its preparation; while the hot "Thick Mash" may be kept several days in storage before using it in cases of necessity. (3) The inverting action upon the sucrose of the molasses is lost in a large degree from lack of heat, although a higher acidity is used.

RECOVERY OF BY-PRODUCTS

When the importance and capacity of the distillery justifies the entering into the phase of valuable by-products recovery; or when according to the laws of the country, slops disposal (other than mere dumping into adjacent rivers, lakes or ocean) becomes necessary, this process affords an opportunity for recovery of valuable by-products with least expense, and in a highly concentrated and pure condition.

(1) *Reduction of Slops to Solid Condition for Disposal as Soil Amendment and Fertilizing Material.*

The greatest expense connected with slops recovery rests upon the high cost of evaporation of the water therein contained. In this relation this process becomes the least expensive, as the slops deprived of alcohol that pass away from the base of the "beer column," are of much higher dissolved solids content than those obtained by any other known commercial process for alcohol or rum. Our slops leave the stripping column at an average Brix concentration between 13 and 16 degrees. This means that they almost double the amount of solids in slops obtained from ordinary mashing and fermenting procedures. Hence, for the obtainment of a given amount of solids through evaporation the saving of steam is almost 50 per cent over that required for the obtainment of the same amount of solids from ordinary slops. This is a decided advantage in case slops recovery in solid form becomes a necessity.

(2) *Potash Recovery from Slops.*

The same arguments as used above will apply to the subject of potash recovery from concentrated slops in respect to fuel requirements for evapora-

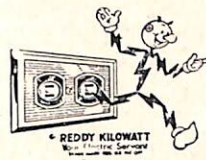
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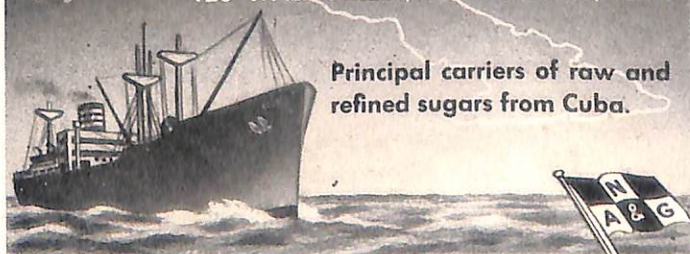
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tion purposes. But there exists another great advantage when this process is in use at the distillery desiring to recover the potash as such: During "Thick Mashing" mineral constituents are withdrawn from the molasses; but not the very soluble salts of potassium, which remain in solution in the purified "Thick Mash" in a very pure and enriched condition of concentration. Therefore, the ash resulting from the incineration of the concentrated slops is mainly composed of potash salts. Up to 43 to 45 percent of potash (as K₂O) has been obtained from the ash resulting from the slops produced in the operation of this process. Ordinary slops will produce an ash with from 32 to 35 percent of K₂O. Another decided advantage of this process in the field of by-products recovery.

(3) *Yeast Recovery from Fermentation Tanks.*

It is well known that under ordinary conditions of mashing, the yeast that may be recovered from the fermenters is often badly contaminated with high amount of ash. This fact naturally lowers the protein content of the yeast recovered and inhibits its use and reduces its price. It is different with the yeast recovered from distilleries using the present fermentation procedure. Due to the great cleaning and demineralizing effect exerted upon the raw material during "Thick Mashing," the yeast that is recoverable after the alcoholic fermentation will be found of high purity, high protein content; and of low ash contamination. A third advantage of the process in the field of by-products recovery.

(4) *Recovery of CO₂*

In the case of CO₂ recovery from the fermenters, either for liquid or solid CO₂ (Dry Ice) manufacture, this process produces a purer and more concentrated supply of the CO₂ gas per unit volume of mash under fermentation. This is due to the absence of contaminating microorganisms in the mash which by the initiation of "side fermentations" may generate other gaseous products of fermentation that will interfere with the purity of the CO₂ generated by the main alcoholic fermentation. Also, due to the fact that per unit volume of fermenting liquid a greater amount of sugars are converted to alcohol and CO₂ gas in a given period of time than is possible in other procedures of fermentation. Hence, per unit time of fermentation very large, pure, and concentrated amounts of the CO₂ gas are available.

MAIN ADVANTAGES OF PROCESS

The more outstanding advantages of this fermentation procedure may be summarized as follows:

(1) Initial cost for a new distillery will be more economical than otherwise when the process is used, because the site and buildings will be smaller; all the process equipment will likewise be

of smaller size than under old methods of fermentation; including fermentation, distillation and power and steam generating and transmitting equipment.

(2) Operating costs are also more economical for this process, due to compactness of equipment, small number of process units, great economy in time, steam, and labor; and greater daily output of finished products per dollar spent in operating expenses. Another important item making for economical operation is the lack of "fermenter failures" due to contaminations of the material before, during, or after fermentation.

(3) Old distilleries adopting the process have been able to increase their molasses mashing capacity by from 50 to 100 percent over previous performance.

(4) Old establishments with no desire for immediate expansion of production, have adopted the process for more economical production of their daily output. This is possible because the amount of equipment used will be less, sometimes one-half of the already existing. For instance one-half the number of fermenters may accomplish the work of fermentation, thus reducing the volume of mash to half of former value. This means, of course, less labor, less attendance, less cleaning and preparation of fermenting vats, and what is much more important *less steam used* for the same production of liters or gallons of spirits. Similarly, one still may be made to produce the same amount of rum or alcohol that formerly took the operation of two stills. All of these features bring economy of production and greater efficiency in a general way.

(5) Both for old and new installations, the process offers assurance of efficient performance, minimum risks of fermentation losses through infections, compactness and ease of operation, minimum labor requirements, and greatest possible daily output of finished products for size of distillery and of distillery equipment.

(6) When the question of by-products recovery arises, this process offers unusual opportunities for the most economical recovery of all by-products, as already explained before.

NOTE: This article was a contribution of the writer to the VIIth International Congress of Agricultural Industries held in Paris during July, 1948.

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