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THE USE OF HIGH-TEST MOLASSES DISTILLERY WASTES

Prepared For

THE UPR CENTER FOR ENERGY AND ENVIRONMENT RESEARCH
BIOMASS ENERGY PROGRAM

CENTER FOR ENERGY AND ENVIRONMENT RESEARCH
UNIVERSITY OF PUERTO RICO — U.S. DEPARTMENT OF ENERGY

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ABSTRACT

The Puerto Rican rum industry earns revenues of more than $200 million yearly. A by-product of the rum production is distillery stillage, a muddy brown liquid, a waste effluent from the making of alcohol. It has a high biological oxygen demand (BOD), equivalent to that of raw sewage. Normally, Puerto Rican rum distillers dump this material into land and coastal waters. Federal regulations starting in 1983 will prevent this. The present rum industry and its future expansion for rum or gasohol production is treatened by proper disposal of the stillage. This paper presents possible solutions to this problem.

The stillage is an organic liquid (8-10% total solids) with a high content of inorganic salts, especially potassium (8% K_2O, dry-weight basis). The three rum distillers in Puerto Rico produce about 710,000 gallons of stillage daily. The pollutant value of this material is equivalent to the daily sewage of a city of over one million inhabitants. There exists a potential to produce 730 million gallons of stillage yearly if the 70,000 acres of sugarcane lands are put into high-test molasses production for alcohol.

There are available methods for the utilization of stillage to minimize its organic content, and therefore, its ability to pollute. Possible products and uses from stillage are fertilizer, animal feed supplement, fuel, glycerol, ammonium sulfate, activated carbon, cement additive, corrosion inhibitor and agglutinin agent for animal feeds. They all present various degrees of anti-economic factors. The choice favored by the Puerto Rican rum distillers for stillage utilization will vary, according to distillery site, from irrigation as a fertilizer to cane fields, to a bio-gas fuel source by anerobic digestion, followed by aerobic digestion to lower the BOD level sufficiently to be dumped into the ocean. Yet, uses of stillage as a fertilizer and animal ration can supply needed materials and reduce imports. Economic considerations are discussed.

1/ Research Consultant

INTRODUCTION

Distillery waste (or stillage [1/]) is an amber to muddy brown liquid with a characteristic odor. It is the effluent from the first distillation column in the making of alcohol. This waste product is an aqueous suspension of organic solids and minerals which has a high biological oxygen demand BOD) content equivalent to that of raw sewage. For every gallon of anhydrous alcohol made, approximately 12-17 gallons of distillery waste are produced.

The rum industry of Puerto Rico earns revenues of more than $200 million yearly. It is presently threatened by the industry's inability to dispose properly of its stillage. A total of 200 million gallons of stillage, with a BOD of 30,000-40,000 ppm, are dumped into land and coastal waters. The Environment Protection Agency (EPA) has ruled that this coastal water dumping must stop unless the stillage is properly treated before 1983 (1).

A sugar mill energy project submitted to the Office of the Governor of Puerto Rico by CEER-UPR (2) proposes the production of high-test molasses and boiler fuel. The high-test molasses will be used by the rum industry which now imports over 80% of its supply (3). If put into full-scale action by the Puerto Rican sugar industry on its 70,000 acres, it could produce 122 million gallons of high-test molasses capable of making 56 million gallons of alcohol for rum and gasohol with a residual 734 million gallons of stillage.

The present rum industry and its future expansion for rum or gasohol production is treatened by proper disposal of the stillage. The object of this paper is to present possible solutions to this problem.

1/ The waste effluent which will be referred to as stillage in this paper is also known as distillery slops, distillery spent wash, distillery wastes and vinasse (<u>mosto</u> in Spanish and <u>vinhoto</u> or <u>vinhaca</u> in Portuguese).

STILLAGE

1. Source

Stillage is derived as a residual product in the production of alcohol by fermentation. Its source is the effluent from the bottom of the first distillation column in the making of alcohol.

Alcohol is made by fermenting a dilute solution of cane molasses with yeast for one or two days and then distilling the fermentation "beer" to recover the ethyl alcohol. The fermentation process begins as the yeast first converts the sucrose into equal parts of glucose and fructose. These in turn are converted into alcohol and carbon dioxide. The resulting fermentation product, known as "beer or wine" or mash (8 to 12% alcohol) is then fed continuously through a multi-column distillation process to separate the ethyl alcohol from the spent molasses and fermentation by-products (stillage).

The first distillation or alcohol stripping column separates the alcohol from the fermentation mash. This stripping process yields the major waste component of the alcohol production process, the stillage.

In the production of rum, the alcohol from the first column is fed to a second distillation or aldehyde column (fig. 1). Here the ketones, aldehydes, low-boiling esters (the more volatile or lower boiling fractions in the alcohol, called "heads") are collected and removed. This fraction is removed to improve rum quality. The heads may be redistilled to recover whatever alcohol remains. The recovered heads is often used as a boiler fuel since it is combustible. This is presently the case with the Puerto Rico Distillery Co. at Arecibo.

The product is drawn off the bottom of the aldehyde column and sent on to the rectifying column, where the fusel oils and amyl alcohols are removed. These waste products may become part of the waste stream, or may be recovered and used as solvents or fuels.

If the alcohol is destined for anhydrous alcohol for gasohol, rather than rum production, the aldehyde column is eliminated and dehydration and benzene recovery columns are added (Fig. 2). The dehydration column is used to eliminate the water (about 5%) from the alcohol. Anhydrous alcohol (100% alcohol) containing no water can be mixed with gasoline to make a gasohol mixture or used as is for a motor fuel.

The amount of stillage per volume of alcohol produced depends on the source of the fermentable material. Table 1 shows the amount of stillage produced from various fermentable-alcohol sources. For sugarcane, be it from molasses or cane juice, the volumetric relationship between alcohol and stillage remains the same. Where high-test molasses (normally 25% more total sugars) is used instead of regular black-strap molasses (50-65% total sugars), the stillage remains similar, as all molasses sources used are diluted in the fermentation process to similar concentrations.

The relationship of about 13 gallons of stillage for every gallon of alcohol is based on anhydrous alcohol. The rum industry uses a proof gallon of rum which is defined as one gallon of rum at 100 proof (50% alcohol). Thus one proof gallon of rum would produce about 6.5 gallons of stillage.

2. <u>Composition</u>

Stillage can be characterized physically as an aqueous suspension of organic solids and minerals whose particle size varies from a coarse dispersion to a molecular solution.

The chemical analyses of stillage from rum and anhydrous alcohol distilleries are given in Table 2. The stillage derived from molasses is higher in all components than that obtained from cane juice in anhydrous alcohol distilleries. Yet, stillage from a rum or anhydrous alcohol distillery is rather similar even though the rum distillery values presented are from Puerto Rico and the anhydrous alcohol distillery values from Brazil.

The highest component content in stillage is total solids with the majority being organic. Potassium is present in greatest quantity for the inorganic components followed by S and Ca. Calcium values are high, primarily due to the treatment in the sugarcane factory of the cane juices with lime to aid in clarification. The source of the high K and Ca values in the stillage in the molasses (Table 3) where these minerals, because of their solubility carries over into the stillage.

The distribution of solids in stillage is given in Fig. 3. The major organic dissolved solids are glycerol and sugars with K, S, and NaCl being the major dissolved inorganic solids.

3. Quantity

The problem of stillage disposal or utilization is magnified by the huge volume of this material. For every gallon of alcohol produced, there is about 13 gallons of stillage to be handled. The Puerto Rican rum industry produces the following volume of stillage daily (4):

Company and Location	Gallons Stillage/day
Bacardí, Cataño	300,000
Bacardí (formerly Schenley), Cataño	60,000
Puerto Rico Distillers, Arecibo	200,000
Destillería Serrallés, Ponce	150,000
Total........	710,000

For a 300 day operating year, the stillage amounts to 213 million gallons yearly.

If the 70,000 acres of land to be alloted for sugarcane under the modern agricultural plan of Puerto Rico (5) were to be used to produce high-test molasses and bagasse as advocated by CEER (2), the alcohol produced from this acreage would be 56 million gallons which would give 734 million gallons of stillage to be disposed of yearly. Such large quantities of stillage, more than three times the amount now produced, will place a challange of how to dispose of it properly from environmental as well as economic considerations.

4. Pollutant Value

Stillage can be a pollutant if not disposed of properly. It does not contain viruses or pathogenic bacteria. However, its greatest pollutant problem is due to its organic matter content which gives it a high biological oxygen demand (BOD) equivalent to raw sewage. In Brazil, human sewage production per day is calculated to have a BOD value of 54,000 ppm (6). Stillage from the Puerto Rican rum industry has a BOD of 30,000 to 40,000 ppm (4, p. 21). Thus one gallon of stillage has a BOD equivalent to the sewage produced daily by 1.5 persons. The dumping of 360,000 gallons of stillage daily into the coastal waters by the Bacardí distilleries in Cataño has a BOD equivalent to the sewage of a city of about 540,000 inhabitants.

Stillage also contains several compounds included in the list of toxic materials (priority pollutants) identified by Congress in the Clean Water Act (Table 4). Although, it is felt that the high BOD of the effluent is the cause of major damage to marine organisms in the coastal waters where the stillage is discharged (4).

The Environmental Protection Agency (EPA) has ruled after a study of the rum distillery effluent discharges into the coastal waters of Puerto Rico and the Virgin Islands that said effluents are harmful to the marine environment and this dumping must stop unless the stillage is properly treated before 1983 (1). Thus the present rum industry and its future expansion for rum or gasohol production is threatened by proper disposal of the stillage.

The reduction of the pollutant effect of stillage can be accomplished by:

1. Degradation of the stillage to inert forms in relation to the environment. For the organic residues present, this can be accomplished by chemical or biological oxidation which eventually produces CO_2 and water as the non-pollutant residues.

2. Process the stillage transforming it into commercial products (recycling) such as fertilizer salts or animal feeds.

The amount of BOD reduction by treatment of stillage is as follows:

Process	% BOD Reduction
Anaerobic fermentation	80
Aerobic fermentation	90
Evaporation to 60% total solids	95
Incineration 3/	$>$ 95
Reverse osmosis (15% total solids)	94

Suggested processes of reducing the BOD of the stillage and/or recycling the stillage into commercial products will be presented in the follwing sections of this report.

UTILIZATION

Stillage should not be considered as a waste material, but rather as a raw material with possibilities for conversion into useful products with potential

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3/ This does not consider the effects of chimney emissions on air quality.

economic value. Even if the money generated by the product utilization just covers its conversion costs or usage, it should be considered of beneficial value inasmuch as it eliminates a pollutant from the environment.

The possible processes and products for the utilization of stillage is given in Fig. 4.

1. Fertilizer

Stillage can be classified as a dilute liquid organic fertilizer with a high content of K. It has about 90-93% water and 7-10% solids of which 75% is organic. The 25% inorganic solids has about 63% K_2O on a dry-weight basis. However, stillage should not be regarded merely as a K fertilizer. Its N is mostly organic in colloidal form, available over a longer range of time than most inorganic N sources. The P is about two-thirds in organic form, thus making it much more available than inorganic P sources. Also it contains large amounts of such important secondary elements as Ca, S and Mg as well as needed trace elements such as Mn, Cu, and Zn.

In its natural state, as it comes from the distillery (at about 70°C), stillage offers the most immediate and economic use as a fertilizer if it can be used directly for land application. It is being used as a fertilizer on a large commercial scale by the Braxilian sugarcane growers. Faced with huge quantities of this material, about 34 billion gallons per year by 1985, the Brazilian sugar-alcohol industry has made rapid strides in applying stillage as a fertilizer for their sugarcane decreasing the use of commercial fertilizers.

(a) Influence on Soils and Production: Stillage applied to the soil raises soil pH, mostly by virtue of its Ca content, for 30 to 40 days after application. The soil returns to its original pH value six months after application (7). The cation exchange capacity on light-textured soil is increased by large applications of stillage (8). This action is short-lived, if not repeated, but annual applications tend to keep this value elevated. Levels of K, Ca and Mg are increased in the cation-exchange complex by stillage application. The physical properties of the soil as regards to resistance to erosion (9) and water-infiltration capacity (10) have been improved by stillage application.

Numerous experiments in Brazil have shown increases in cane tonnage per acre with the use of stillage (11, 12). Increases up to 30% in cane production have been obtained. Also noted is the increase in number of ratoons per field when stillage is used on sandy soils.

Increases in cane tonnage of up to 38% using stillage on a partially reclaimed saline-sodic soil were obtained in Puerto Rico (13). In Trinidad, increases of up to 45% in cane tonnage were shown on a first ratoon application of stillage to a sandy loam (14).

Adverse effects as to cane quality have been reported when using large quantities of stillage per acre, up to 10,000 gallons. The principal effects of high rates of stillage application has on cane quality is to retard maturation (probably due to excess N), lower the sucrose-percent-juice and increase the level of ash in the juice (15). High levels of ash in the cane juice interferes in the crystallization of the sugar. Research has shown that varieties differ greatly in their uptake of K from applied stillage and the resultant ash in juice and influence on maturity (16). Proper selection of varieties can diminish the adverse influence of high stillage applications on juice quality. Rates up to 750 gallons per acre of stillage from molasses does not appear to influence adversely cane quality.

(b) Methods of Application and Rates: In Brazil, stillage is applied by tank trucks and irrigation. The tank truck transports the stillage from the distillery to the field and applies it. Another method uses a tank truck to transport the stillage to the field where it is transferred to a tractor-powered tank for infield application. In general, the tanks are constructed of stainless steel or fiberglass, because of the corrosive nature of the stillage to ordinary steel or other metals.

Irrigation with stillage, called "ferti-irrigation", normally consists of pumping the stillage, in natural form or diluted with residual wash water from sugar mill or distillery, to reservoirs at high points in the field. By gravity, the stillage is distributed via canals and furrows to the field. Overhead portable irrigation equipment has also been used to apply diluted stillage.

The advantages and disadvantages of the various systems used for fertilization with stillage are given in Table 5. It should be remembered that whatever system is used, all of the daily stillage production of the distillery has to be applied in about 24 hours or it begins to ferment.

The economics of stillage application for fertilizing sugarcane depends on such factors as distance to area to be applied, rate of application, source of stillage and type of application equipment. An excellent study of the economics of stillage application by various systems has been made by Copersucar in Brazil (17). A comparative study of all systems showed the raltive costs of the various systems as follows:

System	Relative Application Costs
Tank truck, gravity flow	41
Tank truck, motor pump	44
Tank truck pump powered by take-off	43
Tank truck delivering to tractor-tank	53

Irrigation, overhead	46
Irrigation, furrow	96
Mineral fertilizer	100

Normally, stillage is applied to the ratoon crop in Brazil, after the cane is harvested, but before germination of the new ratoon takes place. This coincides with the availability of the stillage from the distillery and ratoon field operations. The plant crop rarely receives stillage, as new plantings occur when stillage is not available. However, some field experience has shown good results from applying the stillage to the field after the last harvest, before preparing the new field planting. Limited experiments in Brazil have shown that herbicides may be applied with the stillage in tank-truck application for weed control (18).

On a limited scale in Brazil, stillage has been composted with filter-press cake or mud and/or bagasse to make an organic fertilizer. Experiments are being evaluated as to the value of these composts for below the seed application in plant crops.

The rates of stillage application depends on its source, because its composition is somewhat variable. Inasmuch as stillage is used primarily as a potash fertilizer, rates are based on the amount of K needed by the crop for the particular soil. Bittencourt (19) has devised equations to determine application rates for various stillage sources using K analyses. Stillage is applied normally at rates of about 750 to 2000 gallons per acre when derived from molasses fermentation and up to 4,000 gallons per acre when derived from cane juice. Of course amounts of N, Ca, S and other fertilizer elements are also supplied by the stillage (Table 6).

(c) Concentrated Stillage Fertilizers: The concentration of stillage for fertilizer usage by removal of water decreases handling, storage, and transportation costs. The removal of water from stillage requires energy, and thus raises the cost of the concentrated product. At present there exists two commercial processes developed in Europe for the beet-stillage industry, but now being used in Brazil for sugarcane stillage. The "Borag" process concentrates the stillage to 60% total solids by use of multiple evaporators in the distillery (no outside energy source is needed) and then concentrates the material to a powder by use of chimney gases (20). "Conger" uses two processes to convert stillage to a concentrate of 50-60% total solids and then to a powder---one being a spray drier, the other a turbo-homogenizer (21). The powdered stillage is hygroscopic except with the turbo-homogenizer process which mixes the stillage with bagasse.

No experience exixts in Brazil for the use of concentrated stillage as a 60% total solid syrup or powder. In Europe, especially France, concentrated stillage, primarily from sugar beet-alcohol distilleries, is used with good results as a fertilizer for grapes, citrus, tobacco and asparagus. In Australia, a patent has been granted to mix the powdered, hybroscopic stillage with 15% superphosphate to make a granulated fertilizer (22).

The use of the ash from incinerating stillage for fertilizer had been practiced in the 1930's in Brazil (23). There was listed on the U.S. fertilizer market in the 1940's a vegetable potash made from distillery waste and containing 33% K_2O. Several processes exist today to produce potash ash fertilizer by incineration of concentrated stillage (24, 25). The combustion of concentrated stillage provides sufficient heat to carry out the evaporation of the natural stillage to 50-60% total solids as well as give a potash rich ash for fertilizer usage. Products with up to 42% K_2O content have been obtained with a production of 184 pounds K_2O per 1,000 gallons of stillage.

2. Fuel

Stillage can be fermented anaerobically to produce biogas: a mixture of gas containing 50-80% methane giving 5.3 cu.ft/gallon of stillage with a heating value of 1020 BTU's/cu.ft of methane. The mechanism of the anaerobic fermentation occurs in two stages. In the first stage the simple carbohydrates serve as a substrate for a group of acetogenic bacterias which metabolize the compost producing organic acids principally acetic acid. In the second stage, which is strictly anaerobic, the acids are transformed into CO_2 and methane (CH_4) principally by metanogenic bacteria. The anerobic fermentation can occur at two temperature levels each favoring specific bacteria: mesofillic phase (35-40°C) and thermofilic phase (50-60°C). There is evidence that the thermofilic phase can occur twice as rapidly as the mesofilic.

Although the process is rather simple, there are complicating factors of pH, temperature and interfering ions in the substrate. The two ions causing problems when in excess are K and S with strong inhibition occurring at 12,000 ppm for K and 200 ppm for S (26). The S as SO_4 favors formation of H_2S gas which inhibits the metanogenic bacteria. Thus for stillage produced from molasses fermentation, it may be necessary for pretreatment of the molasses to reduce the S content. Dilution to lower S levels causes added load levels making for a more expensive anaerobic fermentation operation.

The efficiency of methane production in an anaerobic fermentation can be increased by using two digesters instead of one. One digester give approximately 54% volume of methane gas in the biogas mixture coming from the digestor gives 77.4% methane volume in the biogas mixture. The caloric value of the biogas from one digestor is 550 BTU's/cu. ft of gas and 790 BTU's/cu ft from the second digestor giving 645 BTU's/cu ft for a mixture of the two digestors (27).

A schematic presentation of how stillage can be converted into steam for energy use by the distillery is shown in Fig. 5.

The wastes from the anaerobic-methane fermentation plant include stillage II which has a BOD reduction of about 80% from the original stillage. However, further treatment to reduce BOD to acceptable EPA levels may be required. The sludge from the methane fermentation also must be disposed of partly by re-cycling to the digestors then thickened and dried and incinerated. This material has a fertilizer value.

The anaerobic conversion of stillage to methane gas provides energy (1000 gallons of stillage provides gas energy equivalent to 0.52 barrels of oil) as well as reduce the BOD to less than 80% of former values.

As mentioned in the previous section on fertilizers, fuel energy is also obtained in the incineration of concentrated stillage to ash. However, the amount of energy obtained normally covers that needed for the concentration of the stillage.

3. Animal Feeds

Stillage can be used as part of the rations for animal feed by:

(a) Direct incorporation of concentrated stillage (60-80% total solids) into balance rations as a substitute for molasses.

(b) Drying the concentrated stillage to a powder using this powder as an agglutinant and feed ingredient.

(c) Producing unicellular protein from aerobic fermentation of the natural stillage and using this material for the protein fraction in conventional animal feed rations.

The K content of the concentrated or powdered stillage can cause a desequilibrium of the Na/Cl balance and cause intestinal disturbances in the animal if large amounts are consumed. The laxative effect of the K contnent of the concentrated or powdered stillage can be overcome by mixing bagasse into the feed ration thus allowing for higher levels of the stillage concentrate. In the feed ration, stillage concentrate is usually limited to 2% for poultry and swine and 10% for ruminants. In Europe, concentrated and dried slops from beet and barley fermentation has been used for over 10 years for animal rations. South Africa and Australia are using molasses stillage as concentrate or powder in animal rations. Known as MDS (molasses distillers solubles), the concentrated stillage (60-80% total solids) is sold for rations for ruminants. Research in Puerto Rico (28, 29) has shown that local concentrated stillage can be used as part of the feed ration for poultry, swine and ruminants.

The production of unicellular protein from stillage is accomplished by aerobic fermentation. The process is shown diagramatically in Fig. 6. The cooled stillage (35°C) is fermented with agitation to insure good industrial production and produce a homogeneous mass of tortula yeast. The cellular mass formed is washed and centrifuged to 90% moisture content, and then hot-air dried to 5% moisture content. Yields of 64 pounds of crude protein per 1000 gallons of stillage have been obtained. Reductions of up to 90% in BOD levels is also obtained in the residual stillage II effluent from the process. Also available from the alcohol-production process is saccharomyce yeast produced during the molasses fermentation process.

The advantages of producing unicellular protein from stillage is that it is a product with good market value and acceptance. There is an additional benefit of reducing the BOD of the stillage by about 90%. The disadvantages for the process are the high cost of investment and operation, narrow operational limits which require care and control including instrumentation and aseptic conditions to avoid unwanted biological infections reducing or stopping production.

4. Others

Although major attention has been given to use of stillage for fertilizer, animal feed or fuel, there are other possible use for this waste effluent. At present the other uses are minor compared with the aforementioned. Yet, proper development may give these other products economic status. The suggested other uses of stillage are:

(a) Agglutinant for animal feeds can be provided by adding either concentrated stillage (60% total solids) or powder. The stillage aids in the forming of pelletized animal feeds an can be used up to 10% in weight as the concentrate and 30% in weight as the powder for grass and legume mixtures (30).

(b) Retardation of the setting of cement and prevention of crusting on cement forms by addition of stillage in concentrate or powder. At present it is used principally in the Soviet Union (31). Some builders in Sao Paulo, Brazil are using it to prevent crusting on cement forms.

(c) Inhibition of corrosion of metals by acids can be accomplished by stillage derived from molasses. Although stillage in the natural state is corrosive to cast iron and mild steel, in dilute form (up to 20% natural, concentrated at 55% total solids) it gives inhibition against acid solutions prolonging the equipment life 8-20 times (32). The anticorrosive action is believed due to polymers, coloring matter, and reducing sugars present in the stillage.

(d) Glycerol can be obtained from concentrated stillage by extraction with a solvent and then distillation (33). Preliminary trials in Brazil revealed that the content of glycerol in their stillage was too low to make such a recovery process economically viable.

(e) Activated carbon and ammonium sulfate can be obtained by pyrolysis of the residue from the distillation of glycerol.

(f) An aggregating agent for soil stability can be obtained by use of stillage in treating heavy clay soils. This was done with salinized soil in Puerto Rico (34).

5. Intermediate Technology for Reduction of Stillage Volume

The possibility exists to reduce the volume of stillage obtained in the production of each gallon of alcohol. From the present 12 to 17 gallons of stillage for each gallon of alcohol, reductions up to 50% are possible. Such a concentration would prove of economic benefit, regardless of the resultant use of the stillage. Some of the methods to reduce stillage volume are as follows (17):

(a) Raising the alcohol concentration in the fermentation "beer" or "wine". Normally, the fermented molasses mixture is distilled off when it reaches 7.5° GL and gives 13.3 gallons of stillage per gallon of alcohol. By raising the alcohol level in the fermentation mixture before distilling, the volume of stillage could be reduced up to 25%. To do this, strains of yeast more resistant to higher alcohol levels will be needed, better control of temperature by refrigerated cooling towers and continuous fermentation will be used. Tate and Lyle (35) have developed a continuous fermentation process allowing alcohol concentrations up to 12% with subsequent reduction in size of distillery and stillage volume.

(b) Elimination of stillage dilution by waste steam. Normally distillation of the alcohol in the distillation column is done by using steam as the heat source directly into the column. By use of external heat sources or steam in serpentine heating tubes, the dilution of the stillage volume could be reduced by about 17%.

(c) Recirculation of the stillage by using it for dilution of the molasses rather than using water can achieve reductions in stillage volume from 20 to 30%. To permit a greater recirculation of the stillage, the Almotherm process (24) of molasses purification can be utilized to remove non-fermentable materials.

(d) Concentration of the stillage by external heat sources using multiple-effect evaporators or by direct contact with chimney-flue gases. One problem that exists in use of multiple-effect evaporators with stillage is the problem of incrustation primarily due to the high amounts of calcium sulfate present in the molasses. The purification of the molasses by Almotherm or other processes reduces greatly the incrustation problems as well as increase alcohol yield in the fermentation by 10%.

There are other intermediate technologies which can be used to improve the quality of the stillage for by-product utilization. Some of these are:

(a) Magnetic separation consists of adding magnetite sands to the stillage as a precipitation flox and then removing the magnetite with the attached solids by means of high intensity magnetic fields. At present problems of dilution and destabilization exist (36).

(b) Reverse osmosis for treatment of cane juice or stillage to remove starches, dextrines, and other polysaccarides (37). The process has various applications in cleaning up industrial wastes such as cheese whey. It has been tried in pilot-plant stage for beet-industry wastes. The present problem consists of large surface area needed for the process which makes it uneconomical.

(c) Use of MgO instead of CaO for clarification of cane juice will give reduction in $CaSO_4$, which due to low solubility, causes incrustation of distillation columns and also interferes in stillage concentration. The problem here is that MgO costs more than CaO.

(d) Continuous fermentation in vacuum gives better alcohol production and reduces stillage volume by 40%. The problem here is the high energy costs needed to maintain the vacuum.

(e) Centrifuging stillage can reduce total solids 75% by weight and remove 90% of the suspended solids fraction, as well as reduce the BOD by 50%. This works well with stillage from wine making, but experience in Brazil shows only a 25% reduction in solids and 30% in BOD. Energy requirements are too high for the reduction achieved.

(f) Floculation and flotation of the stillage to remove solids can be used. Floculants such as aluminum sulfate or iron hydroxide or sulfate can cause possible problems of residual toxicity to the soil if the raw stillage is applied as a fertilizer. Inert organics such as non-convencional poli-electrolytes are being evaluated (38). At present the process is not economical.

(g) Yeast can be eliminated from the fermenter "beer or wine" before distilation by centrifugation. The centrifuged yeast may then be recycled for reuse in the fermenters, sold for animal feedstuff or land disposed. The Melle-Boinet technique can be used for yeast recovery (4, p. 23) shortening fermentation time 40 to 80% with up to 5% increase in alcohol yield.

(h) Removal of the unfermentable solids and dead yeast cells which settle to the bottom of the fermenters to form a sludge (fermenter bottoms) can reduce suspended inorganic and organic materials which could become part of the stillage. Instead of discharging to local sewar drains, the sludge can be removed by a pump to a holding tank and then to land disposal.

POTENTIAL USES OF STILLAGE IN PUERTO RICO

There are many potential uses of stillage in Puerto Rico rather than mere dumping of this material into coastal waters which will be prohibited by 1983. Technology exists for the development of most of the process and products. Research is needed to adopt the technology for Puerto Rican stillage under its own particular local conditions. Also economic investment is needed to put the processes and products into being.

1. Fertilizer

Puerto Rico is a heavy user of commercial fertilizers, especially for sugarcane, pineapples and vegetables. Yet, the use of stillage as a fertilizer is ignored except for the Serrallés rum distillery in Ponce which for years applies stillage to its own nearby sugarcane fields by mixing with irrigation water. The two largest rum distillers do not own agricultural land, and thus they have no ready fertilizer market for the stillage they produce. The Bacardí operation in Cataño has no agricultural lands nearby. However, the Puerto Rico Distillery operation, west of Arecibo, is located in a large agricultural area devoted to sugarcane, pineapples, and vegetable crops. Aside from sugarcane, the Serrallés operation in Ponce is in the center of a large agricultural area which extends east and west along the south coast and produces bananas, vegetables and orchard fruits.

Tank truck application of stillage as a fertilizer has proven economical in Brazil for distances up to 15 miles from the stillage source. However, application of stillage by tank truck to areas beyond economical irrigation application range has not been tried in the Island. Where distance becomes an economical limitation, the concentrated or powdered stillage may be used; but problems in the ease of application of these two forms remain to be solved.

The most practical form to apply stillage as a fertilizer material is the ash when savings in transportation are important. The incinerated ash would not have any N and P content, only K. Yet, it could compete with imported potash fertilizers. About 11,000 tons of potash are imported yearly for local fertilizer consumption. If all of the about 200 million gallons of stillage produced yearly by the Puerto Rican rum distillers were converted into potash ash, it would give about 18,000 tons of 42% K_2O potash fertilizer. More than enough to supply all local needs of this costly material.

The potential fertilizer production from stillage in Puerto Rico is given in Table 7.

An organic complex N-P-K fertilizer process has been developed by the Japanese using concentrated stillage as a raw ingredient. A dry granulated product is obtained through a series of chemical reaction involving concentrated stillage, sulfuric and phosphoric acids and ammonia. It has been estimated that the stillage from the Puerto Rican rum distillers could produce about 54,000 tons annually of this fertilizer (4, p. 71).

As an aternate to the use of stillage for fertilizer, there is the possibility of establishing a land farm program which allows the stillage to be applied to the land purely to absorb the material. Such practices are used in Brazil where excessive amounts of stillage from large distilleries are piped onto very low fertility savanah soils.

The feasibility of land farming was investigated for the three distilleries in Puerto Rico (4, p. 67). Based on stillage transport by tanker trucks and pipeline using a maximum annual application rate, the land requirements for stillage disposal was 275 acres for Bacardí and 125 acres for Puerto Rico Distillers. Serrallés Distillery already disposes of its stillage by irrigation application. The site life was estimated to be a minimum of 25 years based on copper concentrations. If Cu was removed or reduced, the next limiting metal, Zn, would extend site life to 160 years. It is interesting to note that the Tamoio sugarcane mill in the state of Sao Paulo, Brazil, has applied stillage at rates of 100,000 gallons/acre yearly to sandy marginal land for over 15 years; stopped for two years, and then put the land back into cane.

2. Fuel

Stillage can be fermented anaerobically to produce methane gas that can be burned directly as a power source. Where the distillery is part of a sugarcane mill-sugar-alcohol complex, most of the energy needed for the distillery is supplied from the steam produced from the burning of the sugarcane bagasse. The Puerto Rican rum industry must seek external power sources, inasmuch as they are not an integral part of a sugarcane grinding operation. Thus, it that the Puerto Rican rum industry is more inclined to favor the use of stillage to produce methane gas for power and reduction of the BOD in the stillage rather than follow Brazilian distillers choice of using stillage as a fertilizer.

The Puerto Rico Distillery at Arecibo is presently evaluating the Anamet combined anaerobic/aerobic stillage treatment system (4, p. 75). For details see Fig. 7. The process differs from other biological systems in that:

(a) A closed tank is used for the aerobic stage rather than a pond.

(b) Methane is recovered as a fuel.

(c) The digester sludge with the anaerobic process is recycled and the waste-activated sludge from the aerobic step is recycled back to the digestor.

(d) Nitrogen stripping can be done for recovery of ammonium salts.

(e) Energy consumption is low.

(f) BOD reduction of up to 99% has been achieved allowing direct disposal of the stillage II effluent to the coastal waters without any harm to marine life.

The energy contained in a biogas mixture from the anaerobic fermentation [4/] of 1,000 gallons of stillage is equivalent to 0.52 barrels of fuel oil. Thus, the three Puerto Rican rum distillers producing 200 million gallons of stillage yearly could generate the energy equivalent to about 100,000 barrels of oil. The activated sludge from the methane production can be used for fertilizer.

Fusel oils and aldehyde heads from rum distillation columns may be recovered to a storage tank and then burned in the plant's boiler system. The Puerto Rico Distillery at Arecibo already recovers fusel and aldehyde heads and burns them as fuel. Bacardí expects to install a recovery system in 1980.

3. Animal Feeds

The use of concentrated and dry stillage has been evaluated as part of the rations for animal feeds for several years in Puerto Rico (28, 29). Results indicate that only small percentages (3-10%) can be used in a ration without causing nutritional problems.

The Bacardí distillery in Cataño installed a large pilot plant in 1977 to produce concentrated molasses stillage (CMS) to determine optimum operating conditions. Markets are limited in Puerto Rico. Assuming a 5% incorporation level into the feed ration, it was estimated that an annual theoretical maximum of 18% of the CMS produced by all three distillers could be utilized in Puerto Rico (4). The actual maximum may be significantly lower due to customer and manufacturer reluctance to accept a new product. Feeding tests suggest that CMS is technically less adequate than molasses as a feed ration, and that to balance this disparity CMS market prices would have to be about 50% lower than those for cane molasses.

4/ Using a 2-digestor system (27).

The production of unicellular protein from stillage by aerobic fermentation has been tried in several countries, but at present there are no large-scale commercial production of this product. Production of protein from stillage has also been evaluated in Puerto Rico on a laboratory scale (39, 40). The limitations of this product has been economic rather than technological. The unicellular protein has to compete with low-priced protein feed sources. Puerto Rico has no large-scale production of low-priced protein feed sources such as corn or soybeans. These protein sources are imported with 35,422 tons brought in for 1976 (41). The possibilities for animal feed protein production from stillage appears to be worth consideration. Based on an estimation of 60 pounds of unicellular protein from 1,000 gallons of stillage, the three distillers could produce 6,000 tons of protein feed from the 200 million gallons of stillage they produce yearly.

4. Others

There are other possible uses of stillage than for fertilizer, fuel, or feed for animals. Some of these other uses have potential in Puerto Rico even though they may be considered minor in a global evaluation. A brief description of these uses as applied to the Island are as follows:

(a) Recovery of saline soils can be accomplished by use of stillage. It is estimated that there are about 10,000 acres of soils affected by salinity (10). It would require about 326 billion gallons of stillage to reclaim the 10,000 acres using 326,000 gallons per acre (1-acre foot) as determined for these soils (13). Most of the saline soils are located on the south coast of the Island where the Serrallés distillery is also located.

(b) Retarding of the setting of cement or prevention of crusting on forms by addition of stillage concentrate or powder provides a potential market in the

Island, inasmuch as cement is used almost exclusively in its construction program. Research is needed to determine rates and forms of stillage needed.

(c) Glycerol can be obtained from the extraction and distillation of concentrated stillage. The amount of glycerolused in Puerto Rico is not available in the literature. The residue from the distillation can be pyrolized to obtain activated carbon and ammonium sulfate, both of commercial value in the Island. The activated charcoal is used in the rum industry for producing "white" rum, and the ammonium sulfate is a nitrogen fertilizer used in mixed fertilizers for most crops.

ECONOMIC CONSIDERATIONS

The rum industry is an important and growing source of government revenues contributing about $213 million or 12.7% of general fund receipts in Puerto Rico for 1978. The industry employed 1,463 individuals in 1977. The chief secondary contributions of the industry are its support of Puerto Rican sugarcane and molasses production and bottle and carton manufacturing.

The entire rum industry is threatened by its inability to dispose properly of its stillage. The EPA has ruled that this dumping must stop unless the stillage is properly treated before 1983 (1). Investments have been initiated by the Puerto Rican government to build a $5 million waste-treatment plant for the Puerto Rico Distillers Corporation and an outfall pipe for Bacardí Distilleries to help the rum industry solve this pollution problem. Bacardí has initiated a pilot plant for producing CMS and Puerto Rico Distillers has a pilot plant to use stillage for methane production for fuel.

Economic studies were developed using model distillery plants of small, medium and large size to determine their potential to deal with waste-water treatemtn expenditures and the effect on price increases of the rum. Economic input analyses indicated that price increases of 2 to 8% would be needed for treatment of stillage and still maintain pre-control levels of profitability (4). The study indicated that a medium model plant (such as Puerto Rico Distillers) at average efficiency could afford up to $17 million for stillage treatment investments, and a large model plant (such as Bacardí) could afford up to $40 million for stillage treatment investments. At high efficiency and less stringent tax assumption, the medium model could afford $25 million and the large $50 million for stillage treatment investments.

There is no one simple and direct answer to the solution of the stillage disposal problem. Investment will be required in the agro-industrial sector of sugar and alcohol production, as well as the government, to insure reduction in the damage to the environment caused by dumping this material into coastal waters. Considering the source of the stillage, technology selected, and financial resources available, the commercialization of stillage can give a return for the investment.

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TABLE 1. PRODUCTION OF ANHYDROUS ALCOHOL AND STILLAGE FROM VARIOUS SOURCES 1/

Source	Fermentable materials, %	Production, gallons/ton	Volumetric relation, stillage/alcohol
Sweet potatoes	16	29	12
Cassava	25	41	13
Cereal grain	58	146	11
Sugar beets	50	96	7
Sugarcane, juice	10	20	12
Sugarcane, blackstrap molasses	55	96	12
Sugarcane, high-test molasses	68	120	12
Sulfite liquor	2	3 2/	98
Wood	6	58	36

1/ Derived from (42, 43)

2/ Gallon alcohol/gallon of sulfite liquor

TABLE 2. THE CHEMICAL COMPOSITION (% TOTAL WEIGHT) OF STILLAGE FROM RUM AND ANHYDROUS ALCOHOL DISTILLERIES.

Item	Rum	Anhydrous alcohol 3/	
	Molasses	Molasses	Cane juice
Total solids	8.55 (7.6-9.5 1/	8.15 (4.60-8.75)	2.37
Organic solids	-	6.00 (3.70-6.59)	2.00
Crude protein (N x 6.25)	0.47 (0.38-.56) 1/	0.75 (.38-.83)	0.19
Carbon (C)	2.53 (2.73-3.43) 1/	1.82	.61
Potassium (K)	0.6 2/	0.65 (.48-.75)	.10
Sulfur (SO_4)	.3 2/	.64 (.37-.81)	.06
Calcium (Ca)	.2 2/	.26 (.26-.37)	.05
Chlorine (Cl)	.2 2/	.3	.01
Nitrogen (N)	0.08 (.06-.09) 1/	.12	.03
Magnesium (Mg)	.15 2/	.06 (.06-.10)	.01
Phosphorus (P)	.013 (.012-.014) 1/	.009 (.009-017)	.0004
pH	4.6		

1/ Derived from (4, p. 21).

2/ Derived from (44, table 1).

3/ Derived from (46).

TABLE 3. CHEMICAL COMPOSITION OF PUERTO RICAN MOLASSES, 1974-75 1/

Item	Content in molasses
Sugar, %	58.6
Ash, %	10.45
Potassim (K), %	3.33
Calcium (Ca), %	0.76
Nitrogen (N), %	.52
Magnesium (Mg), %	.51
Sodium (Na), %	.11
Phosphorus (P), %	.06
Iron (Fe), ppm	280
Manganese (Mn), ppm	50
Nickel (Ni), ppm	47
Copper (Cu), ppm	30
Zinc (Zn), ppm	7

1/ Derived from (45, table 2).

TABLE 4. TOXIC POLLUTANTS (PARTS PER BILLION) IN PUERTO RICAN RUM DISTILLERY STILLAGE 1/

Item	Bacardí		Puerto Rico Distillery		Average
	Undiluted	Combined 2/	Undiluted	Combined	
		Inorganic			
Beryllim (Be)	12	16	16	10	14
Cadmium (Cd)	78	85	62	10	59
Chromium (Cr)	190	269	470	125	264
Copper (Cu)	3850	7200	5520	450	4255
Lead (Pb)	1110	1350	940	103	876
Mercury (Hg)	8	5	4	4	5
Nickel (Ni)	600	675	830	53	540
Silver (Ag)	60	60	51	10	45
Cyanide	3	4	14	7	7
		Organic			
Phenol	59	68	41	96	66
Bis (2 ethyl hexl) phthalate	10	10	10	10	10
Tolulene	-	10	-	-	
Carbon tetrachloride		210	-	-	
Naphtalene	-	252	-	10	
Benzene	-	29	-	-	

1/ After (4, p. 19).

2/ With other waste waters from the distillery.

TABLE 5. COMPARISON OF STILLAGE-FERTILIZER APPLICATION

System	Advantages	Disadvantages
Tank truck, gravity flow (TTGF)	Easy operation and maintainance	Poor control of application rates Slow distribution time Compacts soil Can't use rainy weather or steep slopes
Tank truck, motor pump or power take off (TTMP)	Application rate 3-4 times faster than TTGF Uniform application	Bigger capital investment & greater fuel consumption Can't use rainy weather or steep slopes
Tank truck delivery to tractor tank	Less soil compaction Better mobility on terrain Can be used in rainy weather Velocity comparable to TTMP	High investment costs Need fleet tank trucks for field delivery
Irrigation, furrow	Low initial investment Allows other waste waters to be used to dilute stillage Ideal for steep terrain	High labor costs to prepare land Soil erosion by high flow High maintainance costs for pumps and pipes
Irrigation, overhead	Low labor costs to prepare land ess soil erosion Better application rate control Easy to install and remove from field	High investment costs for pipes and pumps Labor costs high to move pipes

TABLE 6. QUANTITIES OF FERTILIZER ELEMENTS SUPPLIED BY DIFFERENT RATES OF STILLAGE APPLICATION 1/

Rate applied, gallons/acre	Fertilizer applied, pounds/acre					
	N	P_2O_5	K_2O	CaO	MgO	S
500	3	1	30	12	7	4
750	5	2	45	18	11	6
1,000	7	3	60	23	14	8
2,000	13	5	120	47	28	17
3,000	20	8	180	70	43	25
4,000	26	10	240	93	57	33
5,000	33	13	300	117	71	42
7,500	50	19	450	175	106	63
10,000	67	25	600	233	142	83

1/ Based on stillage from molasses with an average compostion of 0.08% N, 0.03% P_2O_5, 0.72% K_2O, 0.28% CaO, 0.17% MgO and 0.1% S.

TABLE 7. POTENTIAL FERTILIZER PRODUCTION FROM STILLAGE IN PUERTO RICO

Stillage source	Product	Amount, thousand tons	Fertilizer elements, tons 3/		
			N	P_2O_5	K_2O
Rum distilleries yearly production, 1978	Natural stillage	200 1/	670	250	6,000
	Concentrate 2/	117	670	250	6,000
	Powder	71	670	250	6,000
	Potash ash	18	0	0	6,000
Total alcohol conversion of high-test molasses from 70,000 acres sugarcane	Natural stillage	733 1/	2,500	920	22,000
	Concentrate	427	2,500	920	22,000
	Powder	260	2,500	920	22,000
	Potash ash	67	0	0	22,000

1/ Million gallons

2/ 60% total solids

3/ Based on average composition of stillage given in table 6, footnote 1.

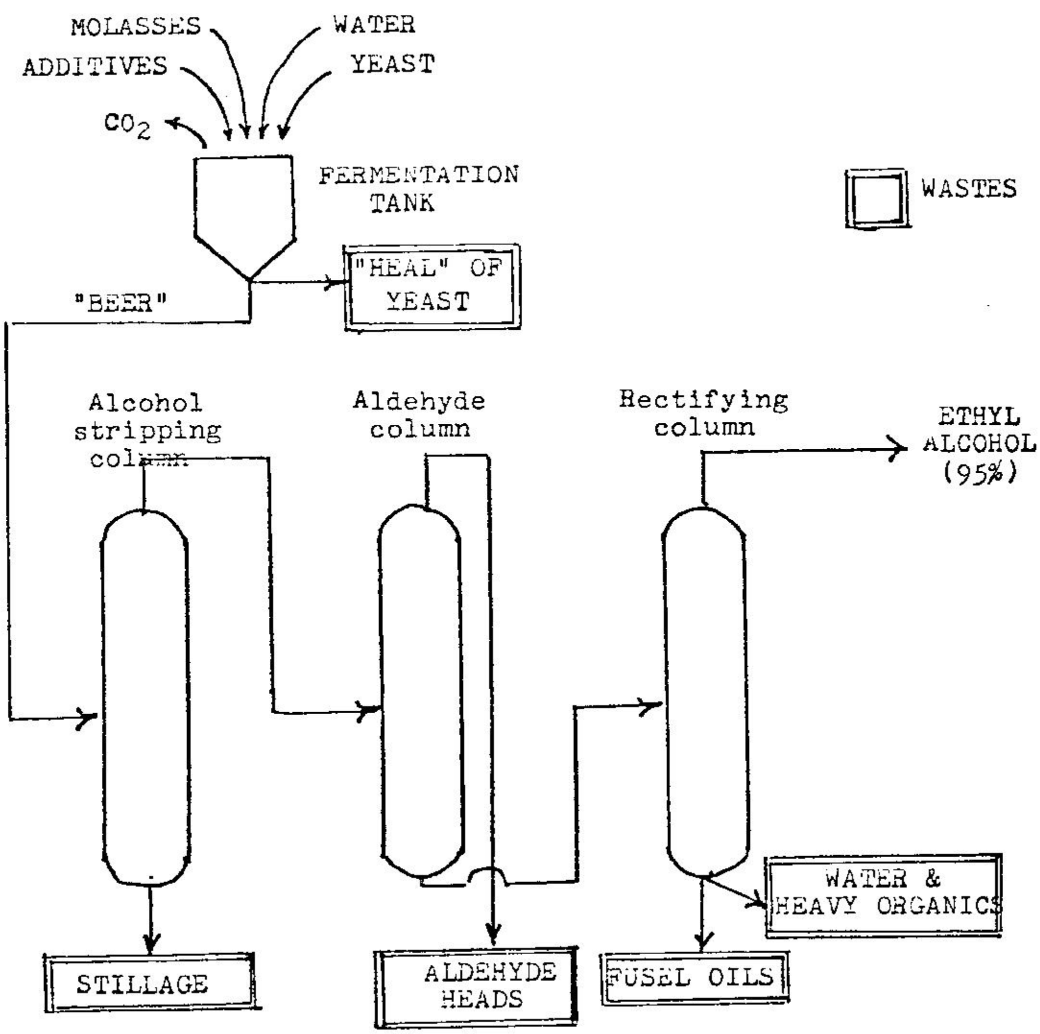

Fig. 1. Flow diagram of a typical rum distillery (4, p. 15).

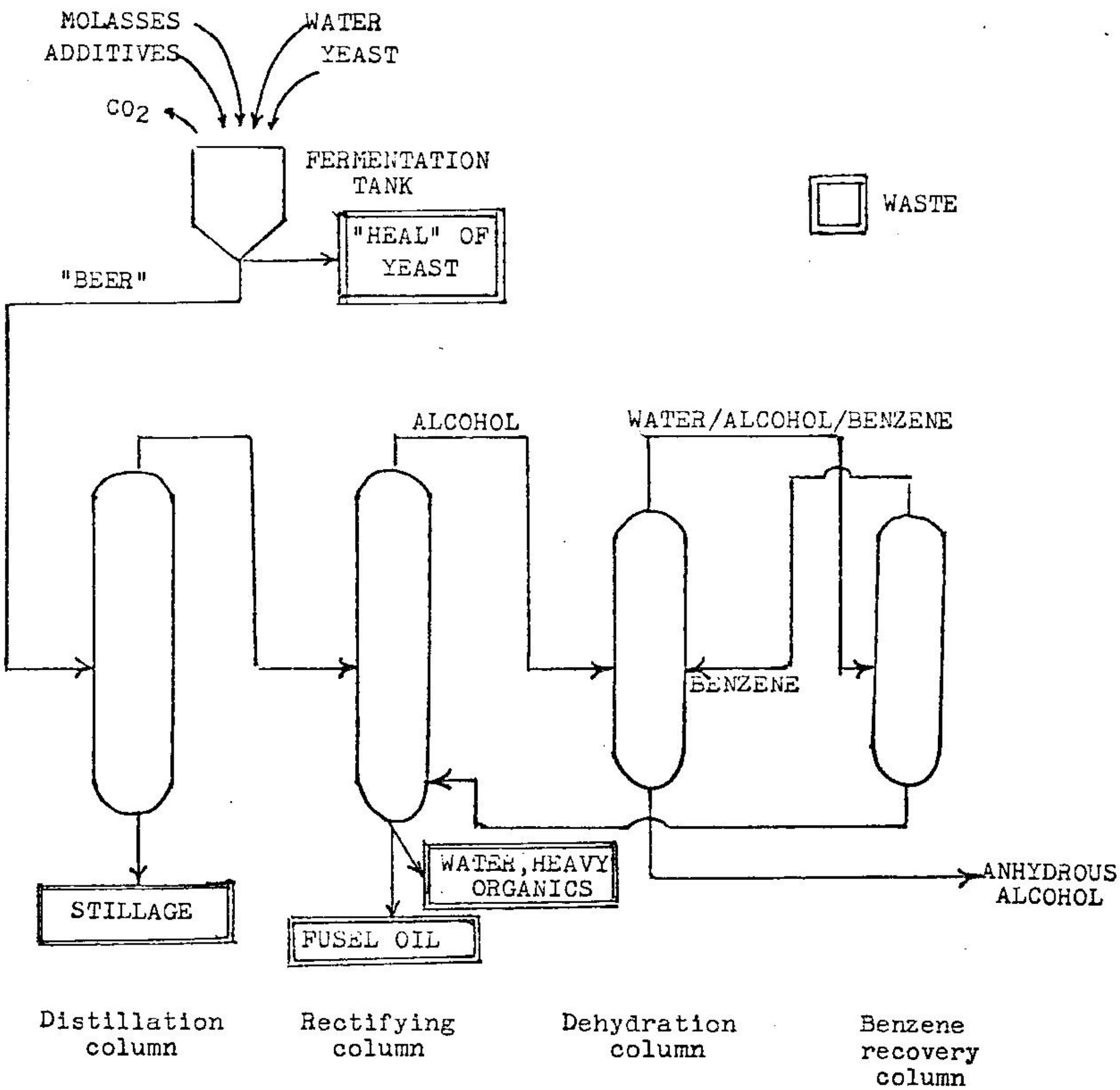

Fig. 2. Flow diagram of a typical anhydrous alcohol distillery.

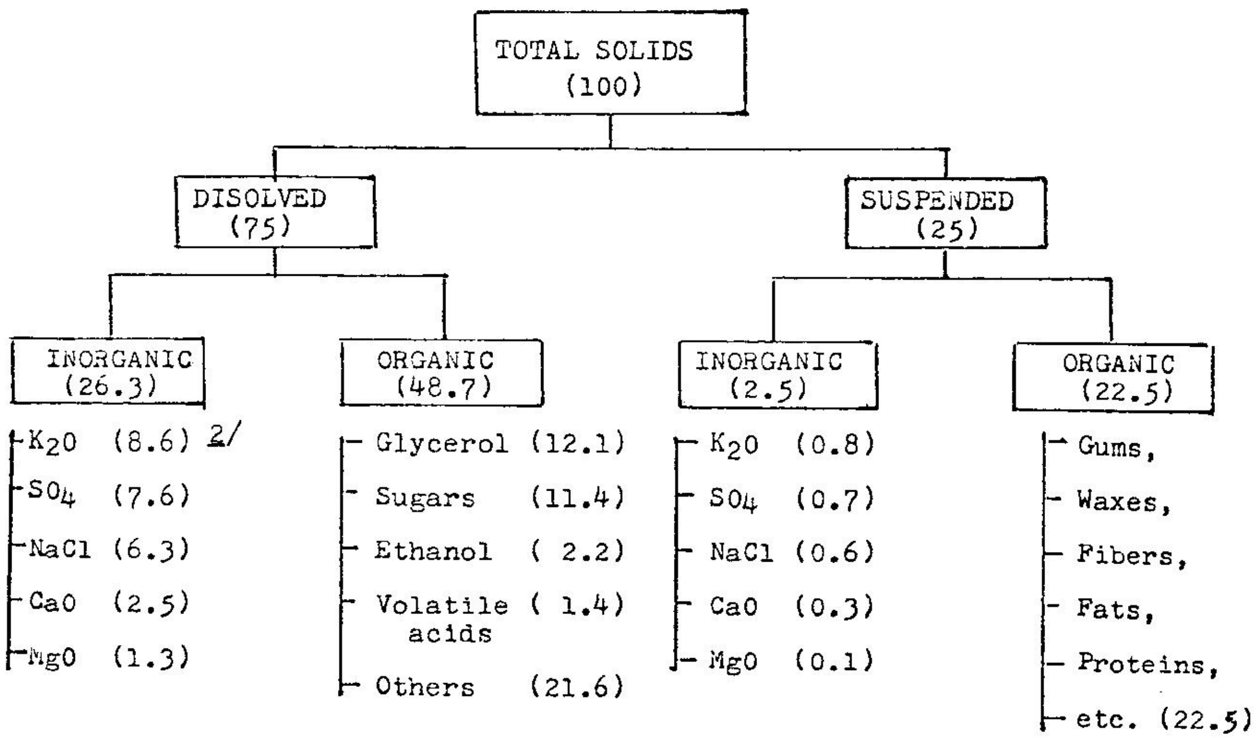

Fig. 3. Distribution of stillage solids on a dry-weight basis 1/

1/ Hypothetical composition based on stillage from anhydrous alcohol production (45).

2/ Numbers in parenthesis are % of total solids.

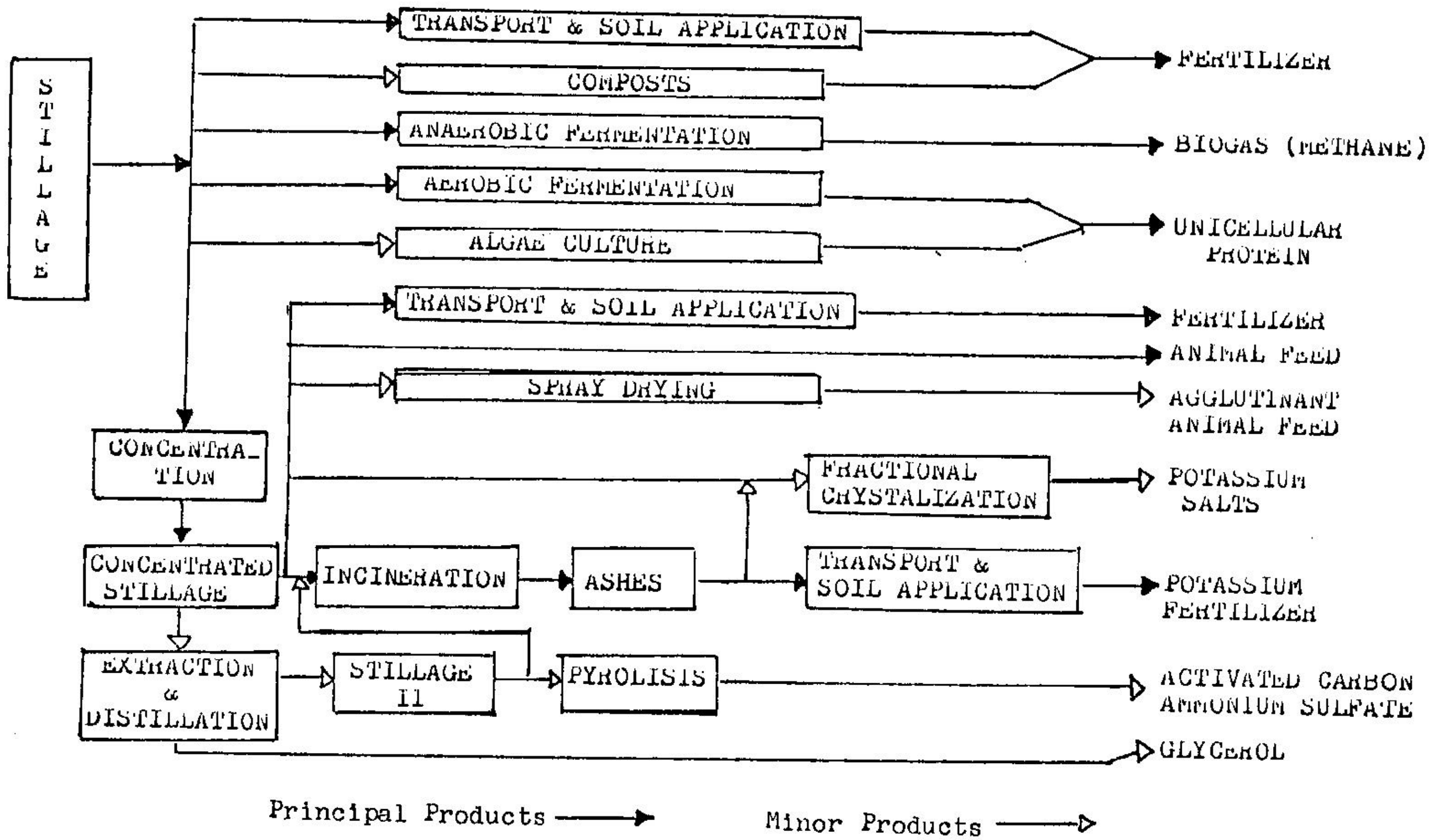

Fig. 4. Processes and products for stillage utilization.

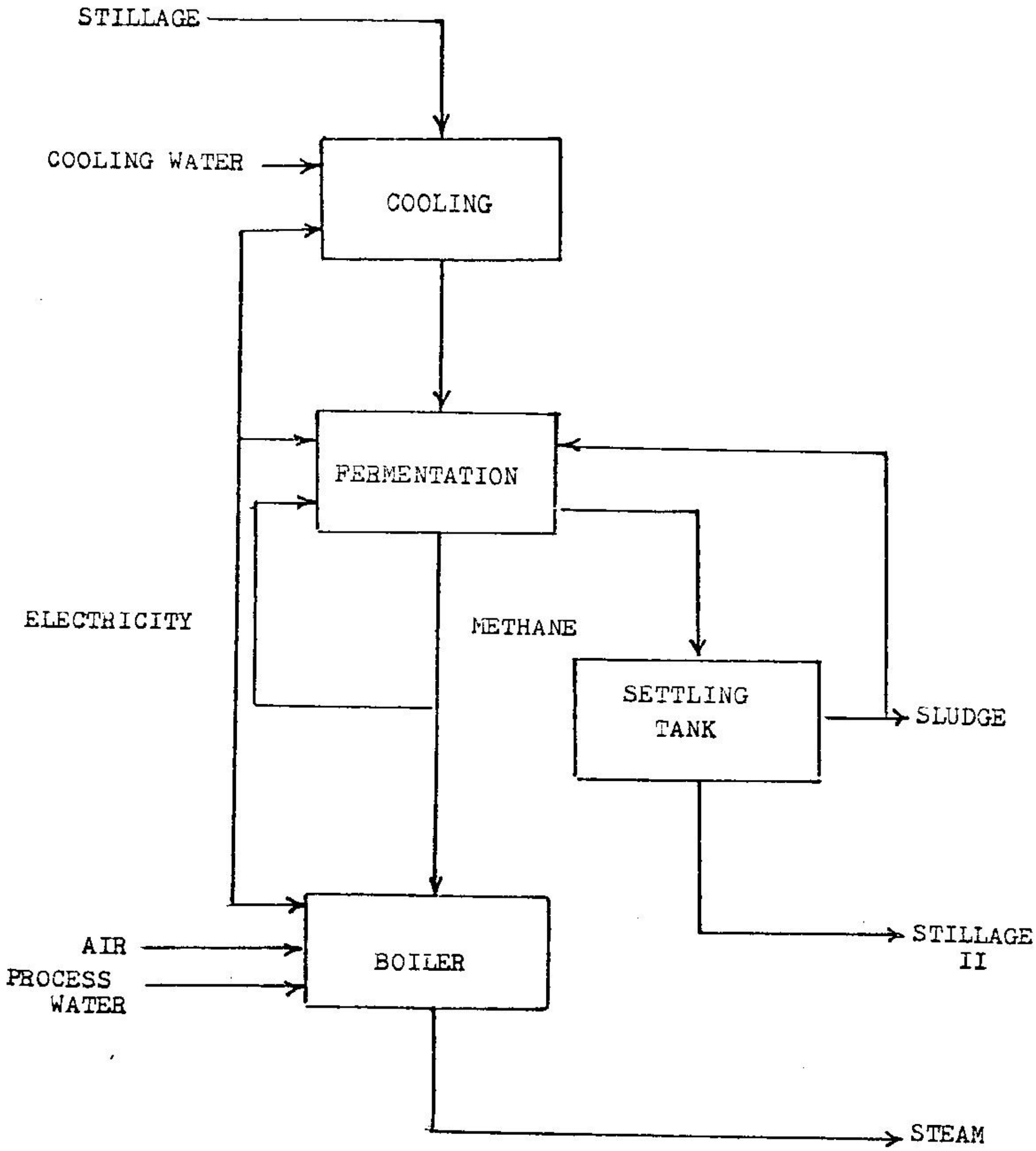

Fig. 5. Process of steam generation with biogass (methane) using anaerobic fermentation of stillage.

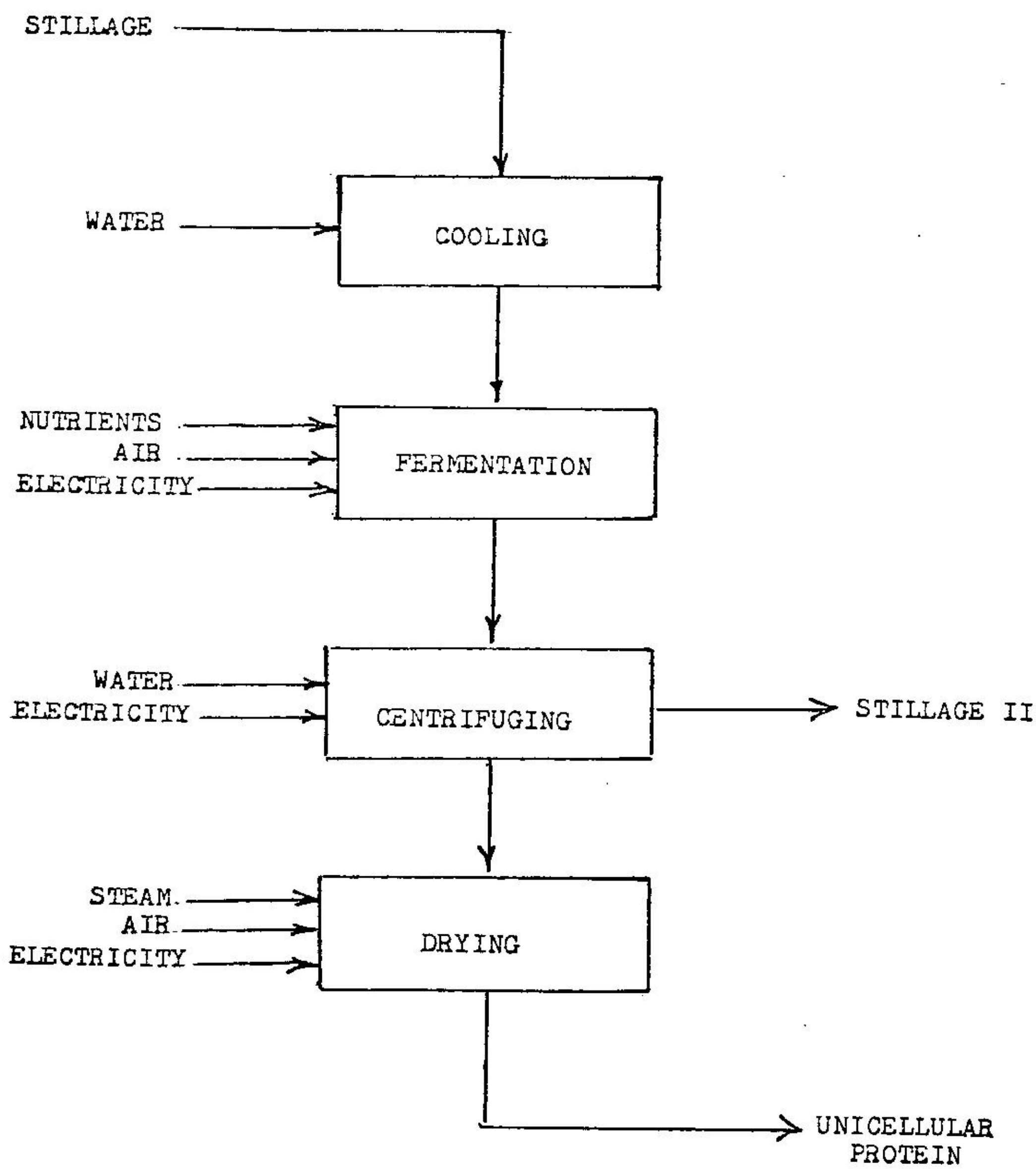

Fig. 6. Process for the production of unicellular protein by use of aerobic fermentation.

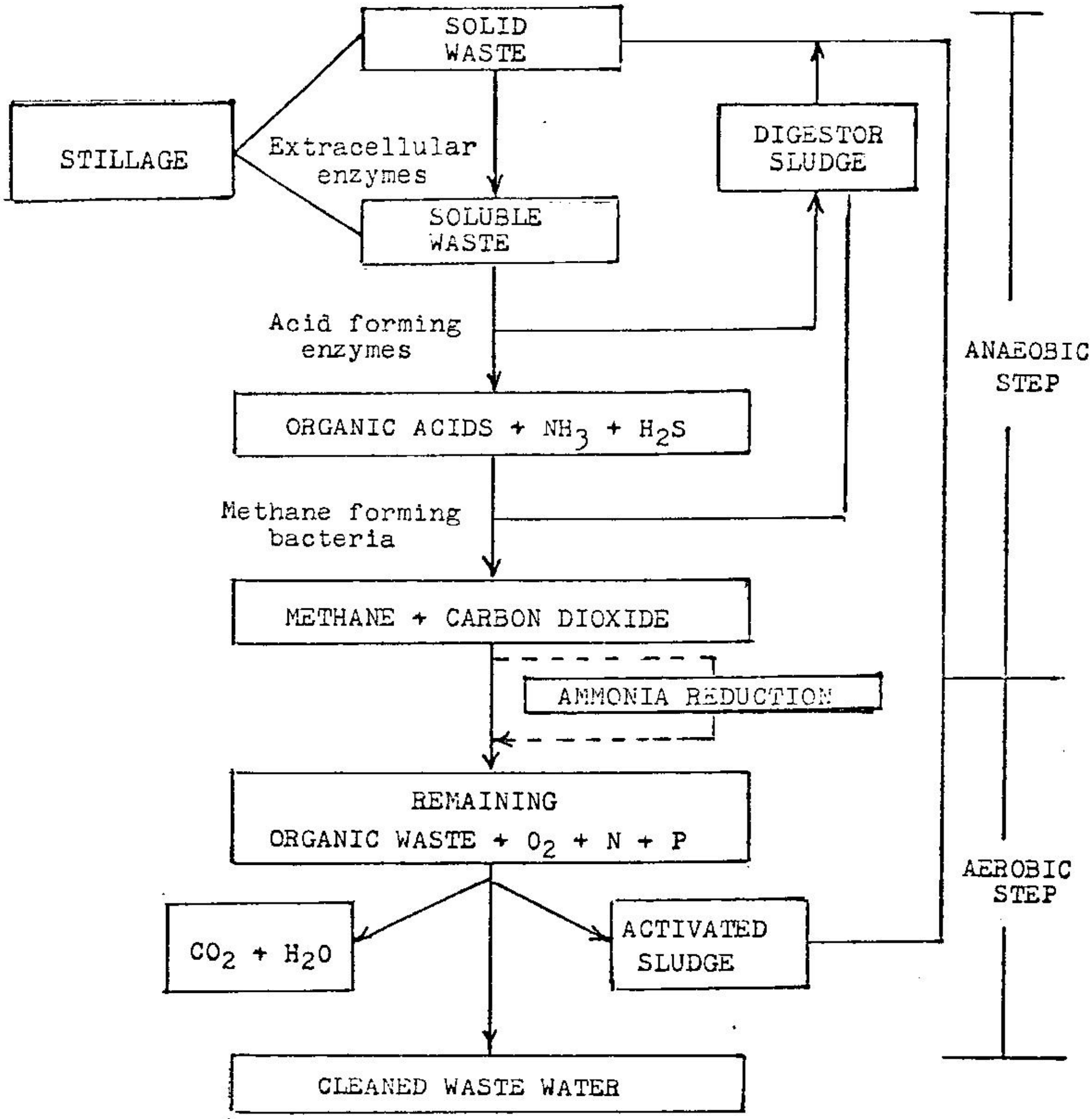

Fig. 7. Anamet system for treating stillage using a combined anaerobic/aerobic process (4, p. 75).