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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

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By

pr. George

Research Consul:

ssearch Associates, 2001 Glenridge Way

Agriculturei

?acer Park, Florida 32792

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?THE USE OF HIGH.

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?THE USE OF HICI

"EST MOLASSES DISTILLERY WASTES

George Sanvets, Ph.D. 1/

ABSTRACT

The Puerto Rican rum industry earns revenues of more than \$200 million yearly. A by-product of the rum production is distillery stillage, « mddy BREN liquid, a waste effluent from the making of alcohol. It has a high biological oxygen demand (BOD), equivalent to that of raw sewage. Yearly, 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 threatened 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 (6% K₂O, 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, ammonia sulfate, activated carbon, cement additive, corrosion inhibitor and agglutinin agent for animal feeds. They all present various advantages or 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 anaerobic 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.

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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 va

12 product {s 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 Environ
ment 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 asking 56 million gallons of alcohol for rum and gasohol with a residual 734 million gallons of stillage.

?the present rus industry and its future expansion for rum or gaschol production is treated by proper disposal of the stillage. The object of

this paper is to pr

int possible solutions to this probles.

1/ The waste effluent which will be referred to as stillage in this paper is ?leo known as distillery slops, distillery spent wash, distillery wastes and Vinasee (nosto in Spanish and vinhoto or vinhaca in Portuguese).

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SPILLAGE,

Source

Stillage is derived as a residual product in the production of alcohol by

fermentation. Its source is the effluent from =

1e bottom of the First

ita

tion coluan in the saking of alcohol.

Alcohol is made by fermenting a {lute solution of cane molasses with

yeast for one or two days and then distilling the fermentation "beet

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 any alcohols are removed.

?These waste products may become part of the waste stream, or may be recovered

?and used as solvents or fuels.

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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.

composition

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

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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 Cn. Chloride 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 carry:

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.

2. 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 413 gallons of stillage to be handled. The Puerto Rican rum industry produces the following volume of stillage daily (6

Company and Location Gallons Stillage/day

Bacardi, Cataño 300,000

Saccharat (formerly Schenley), Cataño 100,000

Puerto Rico Distillers, Arecibo 200,000

Destillerfa Sezrallés, Ponce 150,000

otal. ..s+s++ 710,000

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

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If the 70,000 acres of and to be slotted for sug

ane under the modern

agricultural plan of Puerto Rico (5) were to be used te produce high-test

olasses and bagasse ac advocated by CEER (2), the alcohol produced from this

acreage would be 56 million gallons which would give 734 million gations of

stillage to be disposed of yearly. Such large quantities of stillage, nore

than three tines the amount now produced, will place a challange of hov to

Aispose of it properly from environmental as well, es

.conouic 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 ppa (6). Stillage from the Puerto Rican rum industry has a BOD of 30,000 to 40,000 ppa (4, p. 21). Thus

one gallon of still

has a BOD equivalent to the sewage produced

by 1.5

persons. The dumping of 360,000 gallons of stillage daily into the coastal waters by the Bacardi 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

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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

alcohol production is threatened by

poor 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 CO₂ 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/ 295

Reverse osmosis (15% total solids) 9

Suggested processes of reducing the BOD of the stillage and/or recycling the stillage into commercial products will be presented in the following sections

of this report.

UTILIZATION

Stillage should not be considered as a waste material, but rather as a raw

material with possibilities for conve:

ion into useful products with potential

B/ This does not consider the effects of chimney enissions on air quality.

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economic value. Even if the soney genezated by the product utilization just covers Sts conversion costs or usage, it should be considered of beneficial value inasmuch as it eliminates @ pollutant from the environment.

?The possible processes and products for the utilization of stillage is

von in Figs 4.

A, Fertilizer

Seillage can be classified as a dilute Liquid orgenic fertilizer with «

igh content of K. It has about 90-93% water and 7-10% solids of which 752 i

organic. The 25% inorganic solids has about 63% K₂O 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

Fertilizer on

large commercial scale by the Brazilian 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

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increased by large applications of stillage (6). This action is short-lived

Af 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 settlage application.

Momerous experiments in Brazil have shown increases in cane tonna

pee

acre with the use of stitlage (12, 12). Increases up to 302 in cane production hhave been obtained, Also noted is the increase in nusber of ratoons per field when stillage is used on sandy soils.

Increases in cane tonnage of up to 38% using still

fe 09 @ partially

reclained saline:

jodie soil were obtained in Puerto Rico (13). In Trinidad,

Increases of up to 45% in cane tonnage were shown on

first ratoon application

of stillage to a sandy loam (14).

Adverse effects as to cane quality have been reported even using large quantities of stillage per acre, up to 10,000 gallons. The principal effects of high rates of stillage application on cane quality is to retard maturation (probably due to excess N), lower the sucrose-percentage and increase the level of ash in the juice (15). High levels of ash in the cane juice interfere 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

é

minimize 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.

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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 2 tank trucks 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 9t

Less 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

disposing 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 port-

able 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 26 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 relative costs of the various systems as follows:

System Relative Application Cost:

Tank truck, gravity flow a

Tank truck, motor pump a

Tank truck pump powered by take-off 3

Tank truck delivering to tractor-tank 33

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Irrigation, overhead 46

Irrigation, furrow 96

Mineral fertilizer 300

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,

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 next 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

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 com

position is somewhat variable, thus as stilla

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, P, Ca, S and other fertilizer elements are

also supplied

by the stillage (Table 6).

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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 exist two commercial processes developed in Europe for the beet-silage 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 @ 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 bagass

No experience exists 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, hygroscopic stillage with 15%

superphosphate to make ϕ 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 @ vegetable potash made from distillery waste and containing

33% K₂O. Several proc:

jes exist today to produce potash ach fertilizer by

incineration of concentrated tillage (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

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usage, Products with up to 42% K₂O content have been obtained with a produ-

tion of 184 pounds K_2O per 1,000 gallons of stillage.

2. Fuel

Stillage can be fermented a

erobically to produce biogas! a mixture

of gas co

nsisting of 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 bacteria which catabolize the

substrate 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 methanogenic bacteria. The anaerobic fermentation can

occur at to temperature levels each favoring specific bacteria: mesophilic

phase (35-40%) and thermophilic phase (50-60°C). There is evidence that the

thermophilic phase can occur twice as rapidly as the mesophilic.

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 pps for K and 200 pps for S (26). The S as 80, favors formation of HS gas which inhibits the methanogenic 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 ethane production in an anaerobic fermentation can be

increased by using two digesters instead of one.

One digester give approx

imately 54% volume of methane gas in the biogas mixture coming from the

digester gives 77.4% methane volume in the biogas mixture. The caloric value

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of the biogas from one digester is 550 BTU's/cu. ft of gas and 790 BTU's/cu ft from the second digester giving 645 BTU's/cu ft for a mixture of the two digesters (27).

A schematic pre

intation 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 XI 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 recycling to the digesters 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 Feed

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

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

(©) Producing unicellular protein from aerobic fermentation of the natural stillage and using this material for the protein fraction in conventional

animal feed rations.

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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 content

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 22 for poultry

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 diagrammatically in Fig. 6. The cooled stillage (25°C) is fermented with agitation to insure good industrial production and produce a homogeneous mass of torula yeast. The cellular mass formed is washed and centrifuged to 90% moisture content, and then hot

is dried

to SX moisture content. Yields of 64 pounds of crude protein per 1000 gallons of stillage have been obtained, Reductions of up to 90x in BOD levels is also obtained in the residual stillage 11 effluent from the process. Also available from the alcohol-production process is saccharomyces yeast produced during the

vol

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

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Limits which require care and control including instrumentation and aseptic

conditions to avoid unwanted biological infections reducing or stopping

production.

4. oth

Although major attention has been given to use of stillage for fertilizer, animal food or fuel, there are other possible uses 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 and 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 content and prevention of crusting on

cement forms by addition of stillage in concentrate or power. 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.

(e) Tubification 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 6-20 times (32). The anticorrosive action is believed due to polymers, coloring matter, and reducing sugars present in the stillage,

(@) Glycerol can be obtained from concentrated stillage by extraction

with a solvent and then distillation (33). Preliminary trials in Brazil

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sue

revealed that the content of glycerol in their stillage was too low to make

such a recovery process economically viable.

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

(1) 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 (27):

(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 28%. 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

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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 5

stillage, the Alnoth process

(24) of molasses purification can be utilized to remove non-fermentable materials.

(a) 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 encrustation primarily due to the high amounts of calcium sulfate present in

the molasses. The purification of the mola:

1s by Almothers 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 in a precipitation tank and then removing the magnetite with the attached solids by means of high intensity magnetic fields. At present problems of aeration and destabilization exist (36).

(b) Reverse osmosis for treatment of cane juice or stillage to remove

starches, dextrans, and other polysaccharides (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 un-

economical.

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(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.

(a) Continuous fermentation in vacuum gives better alcohol production

and reduces stillage volume by 0.2. 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 30x in BOD. Energy requirements are too high

for the reduction achieved.

(f) Flocculation and flotation of the stillage to remove solids can be

used, Flocculants 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. tert organics such as non-convencional poli-

electrolytes are being evaluated (38). At present the process is not econoaical.

(g) Yeast can be elinated from the fermenter "beer or wine" before distilation by centrifugation. The centrifuged yeast may then be recycled for reuse in the formenters, sold for animal feedetuff or land disposed. The tielLe~ Boinet technique can be used for yeast recovery (4, p- 23) shortening fernente-tion tine 40 to 80% with up to SE increase in alcohol yleld,

(hy Renoval of the unfermentable solide and dead yeast cells which settle to the botton of the fernenters to form a sludge (fermenter bottoms) can reduce suspended inorganic and organic saterisls which could becone part of the

stillage. Instead of discharging to local seuar drains, the sludge can be

renoved by a pump to a holding tank and then co land disposal.

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POTENTIAL USES OF STILLAGE

there are many potential uses of stillage in Puerto Rico rather than mere

dumping of thi

material into coastal waters which will be prohibited by 1983.

Technology exists for the development of most of the proce

18 and product:

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.

A. Fertilizer

Puerto Rico is a heavy user of commercial fertilizers, especially for

sugarcane, pineapples and vegetables. Yet, the use of stillage

is a fertilizer

is ignored except for the Serrallés run distillery in Ponce which for years

applies stillage

to its own nearby sugarcane fields by mixing with irrigation

water. The two largest run distillers do not own agricultural land, and thus

they have no fi

nal fertilizer market for the stillage they produce. The

Bacardi operation in Cataño has no agricultural lands nearby. However, the

Puerto Rico Distillery operation, west of Arecibo, is located in a large agricul-

rural 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

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.

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?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 fertilizer:

?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% K₂O 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 acid 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 alternate to the use of stillage for fertilizer, there is the possibility of

sibility 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 savannah 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 « maximum annual application rate, the land requirements for stillage disposal was 273 acres for Bacardi and 125 acres for Puerto Rico

Distillers. Serrallés Distillery already disposes of its

stillage by irriga-

tion application, The site life was estimated to be a minimum of 25 years

---Page Break---

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 Tanoio 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 fertilizer:

The Puerto Rico Distillery at Arecibo is presently evaluating the Anaset 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 digester.
- (d) Nitrogen stripping can be done for recovery of ammonium salts.
- (e) Energy consumption is low.
- (2) 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.

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The energy contained in a biogas mixture from the anaerobic fermentation ?/

of 1,000 gallons of stillage is equivalent to 0.52 barrels of fuel oil. Thus,

the three Puerto Rican run 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 éry stillage has been evaluated as

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 problem

The Bacardi distillery in Catatio installed @ 1a:

pilot plant in 1977 to

Produce concentrated molasses stillage (Q¥S) to determine optisus operating conditions. Markets are Limited in Puerto Rico. Assuming @ 5% incorporation evel into the feed ration, it was estimated that an annual theoretical naxisus of 18 of the GUS produced by all three distillers could be utilized in Puerto Rico (4). The actual maxisun may be significantly lover due to customer and

2 that

manufacturer reluctance to accept a new product. Feeding tests suggest

MS 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

Af Using @ 2-a4j

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?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 prot

from 1,000 gallons of stillage, che 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 stillas

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. Tt

is eavinated that the

fare about 10,000 acres of soils affected by salinity (10).

It would require about 326 billion gallons of stillage to reclaim the 10,000

using 326,000 gallons per acre (1-acre foot) as determined for these soils

23). Most of the saline soils are located on the south coast of the Island where the Serrallés distillery is also located.

(&) Retarding of the setting of cement or prevention of crusting on forms

dition of stillage concentrate or powder provides a potential market in the

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

(©) Glycerol can be obtained from the extraction and distillation of

concentrated stillage. The amount of glycerol used in Puerto Rico is not available in the Literature, The residue from the distillation can be pyrolyzed 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 "hive" 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 sugar cane 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 Bacardi Distillers to help the rum industry solve this pollution problem. Bacardi has initiated a pilot plant for producing OHS 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

treatment expenditures and the effect on price increases of the rum, Economic

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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 large model plant (such as Bacaréf) 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 size and direct answer to the solution of the stillage disposed 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 ALCONOL AND STILLAGE FROM VARIOUS
SOURCES 1/

Fementable Production, Volumetric relation,

Source materials, % gallons/ton _stillage/alcohol

Sweet potatoes 16 23 2

cassave 25 a a

cerca} grain 38 14s, a

Sugar beets 50 96 7

Sugarcane, juice 10 20 2

Sugarcane, black~ 35 96 2

strap mole:

oe 120 aw

Sulfite Liquor 2 3B 96

Hood 6 58 36

/ Derived from (42, 43)

2/ Gallon alcohol/gallon of sulfite Liquor

---Page Break---

~ 2

TURLE 2, THE CHEMICAL COMPOSITION (F TOTAL WEIGHT) OF STILLAGE FROM RDI AND ANHYDROUS

im neoner 2

tee Nolasser Cane juice

?Total solide 8.55 (7.69-8.15) 4 8.15 (4.60-8.75) 237

Organic solide - 6.00 (3.70-6.59) 2.00

Grade protein 0.47 (0.38-.56) 0.75 (.38-.83) oa

ors 6.23)

Carbon (0) 2.89 793.00) La a

Porassive (K) 6 0.65 (.68-.75) oo

sulfur (504) at 66 (37-8) 8

calciua (ca) a2 +26 (.26-.37) 05

atorine (C2) 2 3 0

?Witropen (8) 0.08 (0.06-.09) AY a2 03

Magoesiun (ig) ase +06 (.06-.10) ol

Phosphorus (P) sors (.012-.014) 4008 (009-017) 0008

AU derived from Cy pe 2.

BU derived from (44, table 2).

BY derived from (46).

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TABLE 3, CHEMICAL COMPOSITION OF PUERTO RICAN MOLASSES,

1976-75 1)

een

suger, Z

Ash, ©

Potassin (RK), %

Calcium (Ca), %

Nitrogen (N), %

Magnesium (Mg), %

Sodium (Na), %

Phosphorus (P), %

Iron (Fe), ppm

Manganese (Mn), ppm

Nickel (Ni), ppm

Copper (Cu), ppm

Zinc (Zn), ppm

AJ Derived from (45, table 2).

Content in molasses

58.6

10.45

3.33

0.78

32

3

an

08

280

50

a7

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ous

TUMLE 4, TOXIC POLLUTANTS (PARTS PER BILLION) 10 PUERTO RICAN mM DISTILLERY

sritiace 2

sacorat Pocete Rice Distiaery

Teen Undiluted Conbined 2! ?Undiluted Combined Average

loorgange

Beryilin (Be) 2 a6 6 10 au

ccadniue (C2) * as e 10 8

Onroeiua (Ce) 230 29 170 ns 268

copper (cu) 3850 7200 3520 430 ass

end (Pb) a0 3380 0 03 a6

wereury (ig) 8 5 ? ? 5

Mieke) (21) 600 os 0 s sto

siiver (he) © © s 10 4s

cyanise 3 ? 4 > 7

Pens 8 a %6 ?

Bie (2 ethyl Sed) 10 10 10 20 20

?phthalare

Tolutene - 10 - -

no - -

- 252 - 1.

Benzene - 2 - -

A Atcer (Ly ps 19).

2) With other waste waters from the distillery.

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TAMLE 5. COWPARISON OF STILLAGE-FERTILIZER APPLICATION

system Advantages Disadvantages

?Tank rack, Easy operation and Poor control of application

low flow maintenance rates

caer) Slow distribution time

Contacts cost

(Can't use rainy weather

for steep slopes

Application

tines faster prcater foe! consumption

Ualfore application con woe aay weather

or steep slopes

Lets soft conpection

Better nobility op

cen be used in rainy

?veather

Vetocity.conporable

Tecigation, oy infetat Sovestnent igh Labor conte co prepare

Tetons co'be'vsed co Soil crorion by Mgh

Tilste ceidloge BEDS Setnectacnce ests for

sod ines

tion, ow labor coats eo

overhead prepore lend

ess foil erorion

Eesy fo install end

enove fren field

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{USLE 6, QUANTITIES OF FERTILIZER ELDONTS SUPPLIED BY DIFFERENT RATES

OF sriuiace aPOLTeATION 2/

Fertilizer applied, pounds/acre

fate applied, ns

pallons/scre NF, %0 aD MeO 3

500 2 1 » 2 7 ?

750 5 2 se u ?

1,000 7 2. wm oD u ?

2,000 a sm 4 2 oo

3,000 2» rt nc) 3s

4,000 % 10 os 78

3,000 3s no

1,500 so ts

19,000 os mss

1/ Sased on s2itlage fron molasses with an average coupotion of 0.08% By

O38 F05, 0.72% Kj0, O.28T CaO, On17% MgO and OIE 5.

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TABLE 7. POTENTIAL FERTILIZER PRODUCTION FROM STILLAGE 16 PUERTO AICO

Fereitizer etenente, cons 2/

suittape source Prodect??_shouandtone N05 AP

Rum distilleries ?Katural 200 3/ 670 230 6,000

Natptquattita, ibe

te

concentrate an? oo 6,000

roster a 0 1506000

otach a ? ° © Go

otal stcohel ??Nagurat rs soma ,e00

seg

MET oe cocenteate 500,000

Soret nr

mereane rovter mo seo mm ,o00

Fotash ash « ° © 22,0

AJ MG150n gations

BU 60% coral sotids

12) maned on average conpes

tion of stiliage given fn rable 6, footnote 1,

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MOLASSES. wanes

NC

?PERMELTATION

TANK

wastes

TA

AST

Alcohol, Aldehydes, Acetic Acid

Essential oils

Stripping column, etc.,

(os)

wines

EADS

Figs 1s Flow diagram of a typical rum distillery (, p. 15).

---Page Break---

=a.

MOLASSES

cea

ADDITIVE: \, YEAST

aK

EANENTARION

"TANK

waste

EAL" OF]

<> ?veast

ALCOHOL, ATA /ALCOHOL /BENZENE

Distillation ? Aetifying Dehydration Benzene

coluaa, column ?column Pecovery

?coluan

Fig. 2.

Flow diagram of © typical anhydrous alcohol distillery.

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-40-

?TOTAL SOLIDS

0)

DisoLveD ?SUSPENDED

(25) (25)

f i T

Hee) ?ORGANIC ?INORGANIC OAGANTE

(265) (e.7) 5) (22.5)

20 (8.6) 2/ [> Glycerol (12.2) p20 (0.8) [- Gums,

\$y (7.6) Sugars (11.6) F505 (0.7) + Waxes,

Frac (6.3) f tnenot (2.2) f maca (0.6) + Pivers,

cao (2.5) Woratize (144) f cao (0.3) } Fats,

Fag (2.3) lgo (0.2) |. Proteins,

ae 7

Fete, (22.5)

Fig. J. Distribution of etillage solide one ary-Ketgnt basis 2/

2/ Hypothetical composition based on etillage from anhydrous alcohol production (45).

2/ Wasbers in parenthesis are £ of total solids.

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?worsuaritan eBetta@ sos syonpoad puv sessscosg *y ?9g

e? Honpoag s01Ty, + er0mpous redrourag

TT

erarvonnaty{ anvFieze

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STILLAGE.

coonrxe WATER ?of

Coons

|

|?f Je_____

PESKEWTATION

suectazcray ETHAN

|?+ sivas,

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L__, sornzce

am ???>| i

BOILER

Process

WATER

fa sre

Fig. 5. Process of stean generation with biogase (methane) using anaerobic fermentation of etillage.

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SIILLAGE

WATER foo tig

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?PaOTEIN

Fig. 6, Process for the production of unicellular protein ty

use of aerobic fermentation,

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Fig. 7.

?SOLUELE

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Acte forming

enzymes auazotic

4 ?STEP

ORGANIC ACIDS + My + HS

Methane forming

?bacteria

¥

METHANE + CARBON DIOXIDE

sae aa

?ARRORTA BsDUCHION

-RERAINING

+O +NrP

AEROBIC

?STEP

/acrivaTeD

002 + H2O ?SLUDGE

CLEANED WASTE WATER

Aranet system for treating tillage using a combined

anaerobic/aerobic process (4, p. 75).

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