Concept of Waste to Wealth
- A Study on Alcohol Industry

By

K. S. Jhansi Pradhiba

A DISSERTATION SUBMITTED TO THE AVINASHILINGAM INSTITUTE FOR HOME SCIENCE AND HIGHER EDUCATION FOR WOMEN - DEEMED UNIVERSITY, COIMBATORE - 641 043 IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF

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April 1998

Certified As Bonafide Research Work

S. Sivakamasundari
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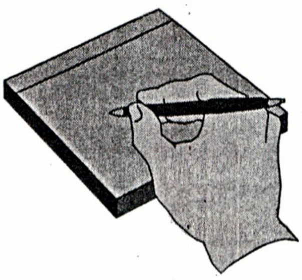

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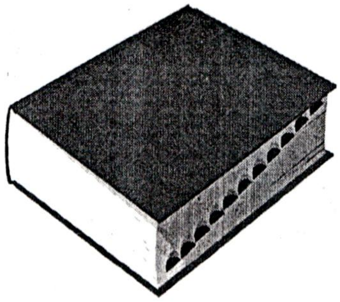

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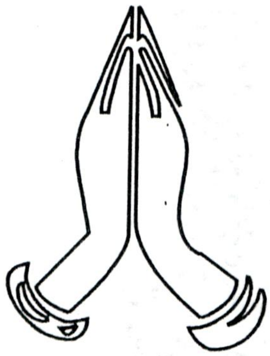


Introduction

INTRODUCTION

Human activities are mainly responsible for modifications and deterioration of the environment. In the process of development man's relationship with the ecosystem has changed gradually and some major modifications have been brought about in the environment which are becoming detrimental to human society. Our industries, agriculture, mining and growth of cities have expanded and their output and wastes are polluting the unpolluted air, water, and soil (Vyas et al, 1982).

Industrialisation and urbanisation are world wide phenomenon. More and more people will be living in larger and larger cities. These high density communities will pose a special challenge in the provision of potable water, clean, air, waster disposal, transportation and recreational space. (Trivedi and Raj, 1992)

Industry, the second largest sector of Indian economy has sustained the momentum of growth during last decades. It is because of the fact that industrialisation in our development planning process has been emphasised with a view of making India a modern and competitive in which all sectors of society and economy are able to flourish (Chaurasia, 1992).

It is undoubtedly true that industrialisation has boosted up the national economy but in turn has adversely affected the total environment.

Industries can conveniently be classified into two groups viz (i) Dry process industries (ii) wet process industries. Dry process industries do not use water in their process. While wet process industries use water either as raw material or in their process or for both. Only a little quantity of water is observed in the process and the rest being discharged as effluent. The solid wastes produced in these industries are not given importance as they are either recycled, dumped or disposed of to remote place (Manivasakam, 1997).

Today, the sugar industry is the third largest industry in the country and ranks next to Iron and steel, and cotton industries. In India, there are about 380 sugar industries of which 4.1 million tons of sugar produced. annually. (Pandey and Carrey, 1994).

Sugar is prized not for its sweet taste alone for its supplies about 13% of energy required for existence. As a result sugar production and refining is a huge industry with the total world production of 95.8 x 10^6t (Austin, 1983)

The sugar recovery from sugarcane in modern mills leaves substantial quantities (average 4% of sugarcane used) of molasses containing 45 to 50% of sugar that cannot be taken out as such. It is also a problem material to handle and dispose of but the sugar contained in it can be readily and efficiently converted into ethyl alcohol and other products.

Molasses which is the by-product of the sugar industry is by and large, the main raw material for the production of ethyl alcohol in many countries. The alcohol produced by the fermentation of molasses can be the best raw material for the manufacture of chemicals. The production of alcohol from different raw materials depend upon the availability of raw materials in large quantities on a long term basis. (Manohar Rao, 1983).

In India there are about 210 distilleries producing around 6500 million litres of alcohol every year generating 130 million litre of waste water every year. (Shinde 1984).

After recovery of alcohol the spent wash left behind is another waste product of distilleries which is highly acidic. The spent washis the largest effluent stream from distilleries, the volume of which varies from 14-22 tonnes of the volume of the rectified spirit produced (Pandey & Carney,1994).

The spent wash from distilleries carry a high organic load and cause severe fouling of the atmosphere. Discharge of spent wash on land results in pollution of ground water and charring of vegetation and crops. Stagnation of the effluents on land results in obnoxious conditions in the region. (Agarwal,1996)

Today it is technically possible to recover atleast two-third of industrial wastes. The reverse of industrial effluents and solid wastes can reduce the size of waste to small fraction. Reed, straw, corn cob and stalk such as coir fibre, coconut husk, groundnut husk and bagasse are obtained as industrial wastes. (Misra et al., 1987).

Much Municipal, Industrial and agricultural trash can be neither reused nor repaired and consequently must be reduced to raw materials suitable for remanufacture.

Agriculture wastes can be reused as well, when sugar is extracted from cane the remaining fibrous stalks are well suited for paper production (Turk, et al., 1984).

Utilisation of these wastes apart from solving the problems of disposal and dumping would improve the agricultural economy considerably.

The disposal and utilisation of wastes are also becoming of great importance in highly industrialised areas because of

(i) The ever-increasing difficulties in disposing of great accumulation of wastes from cities in a sanitary and orderly manner.

(ii) The ever increasing threat to soil fertility due to the great decrease in the amount of animal manure.

(iii) The ever increasing intensive demands being put on the agricultural lands to produce more food.

India has a vast potential of manural resources and the organic wastes for recycling are quite varied. The major wastes are live stock and human wates; green manures, urban and rural wastes, agro indsutries by products, marine wastes and tank silt.

If these organic wastes are partially decomposed by bacteria, worms and other living organisms, a valuable soil conditioner is produced. This process is called composting. (Turk et al., 1984).

The principle of production of farmyard manure from cattle shed wastes has been extended for the preparation of composts in which a variety of carbonaceous and nitrogenous wastes like straw, paddy, sugarcane trash, bagasse and other agricultural as well as industrial wastes of all sorts can be used to produce manures. (Gaur & Sadasivam, 1992)

No doubt, being organic substances, these agro industrial waste materials can increase the fertility of land after being dumped. (Misra et.al., 1987)

As the plant residues used in the preparation of artificial manures are poor in nitrogen, available inorganic nitrogen must be introduced for the purpose of bringing about active decomposition.

The demand of our soils with regard to nitrogen is far greater than the indigenous production of commercial fertiliser. hence it is worth while to examine the possibilities of utilising nitrogen waste products like distillery effluents (Bajpai and Dua, 1972.)

The reuse of waste water for agricultural irrigation offers many attractive benefits, including reduced pollution of water sources, increased water supply for productive fertilisers and macro nutrients to maintain soil fertility.

The use of industrial waste water from urban centre makes it possible to conserve limited water resources for economically beneficial agricultural irrigation. Waste water irrigation returns vital nutrients to the soil that would be expensive to add in other forms (Shuval et al., 1986).

Waste water irrigation coupled with large interseasonal storage reservoirs or year round land application can be a cost effective means of preventing or reducing water pollution.

The benefits of waste water irrigation depends on 3 main factors viz., (i) the quantity of water (ii) the quantity and type of nutrients (N,P,K) and (iii) predictability of the flow.

Infact a waste is essentially a resource although out of place. It may be in the form of energy or matter. Being not available at its right place such a resource would create economic problems, while its being available at some wrong place give rise to ecological problems (Saxena, 1990).

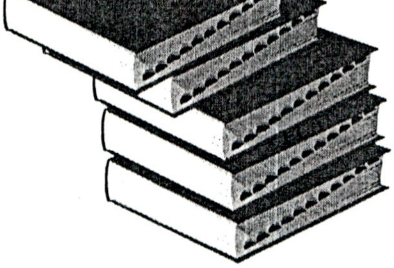
Thus the present study is an attempt to

(i) Assess the physcio-chemical characteristics of the effluent from alcohol industry.

(ii) To utilise spent wash along with pressmud in agriculture for its manurial value.

(iii) In a view to reduce the consumption of chemical fertilisers.

CHAPTER 2



Review of Literature

REVIEW OF LITERATURE

As human needs increase and civilisation changes, more and more finished products of different types are required. Accordingly a large number of industries born and grown in every country (Manivasakam, 1997).

Distillery industry is one of the major industry where ethyl alcohol is manufactured. Research studies have been done on the various aspects of alcohol production such as the process involved, source, nature and effects of the effluent, ways of waste prevention and methods of their treatment and disposal. The following is the review of literature pertaining to the present work **"Concept of waste to wealth - A study on alcohol industry"** is discussed under following leadings.

2.1 BRIEF INTRODUCTION OF ALCOHOL INDUSTRY
- 2.1.1 Manufacture of industrial alcohol
- 2.1.2 Sources, Characteristics and effects of spentwash
- 2.1.3 Biomethanisation

2.2 DISTILLERY SPENT WASH - AN USEFUL MANURE
- 2.2.1 Agricultural utilisation

2.3 RECOVERY OF BY-PRODUCTS FROM SPENTWASH

2.4 UTILISATION OF SUGAR INDUSTRY PRESSMUD IN AGRICULTURE.

2.1 BRIEF INTRODUCTION OF ALCOHOL INDUSTRY

Raw Materials

Due to limited availability of non-renewable fossil fuels, production of ethanol from renewable plant resources has become important. Ethanol is manufactured synthetically from petrochemicals or biologically through the process of fermentation by micro organisms. Although economically produced world wide by catalytic hydration of ethylene from petroleum, industrial alcohol obtained through yeast fermentation of sugar is being competitive in the international market mainly as a consequence of rising oil prices (Bilgrami & Pandey, 1992).

Raw materials from which ethyl alcohol may be prepared are (i) starchy materials (ii) hydrocarbon gases (ii) cellulosics materials such as agricultural residues and waste sulphite liquors (iv) Fruits [mainly for alcoholic beverages) (v) Molasses (Chakkrabarty, 1981).

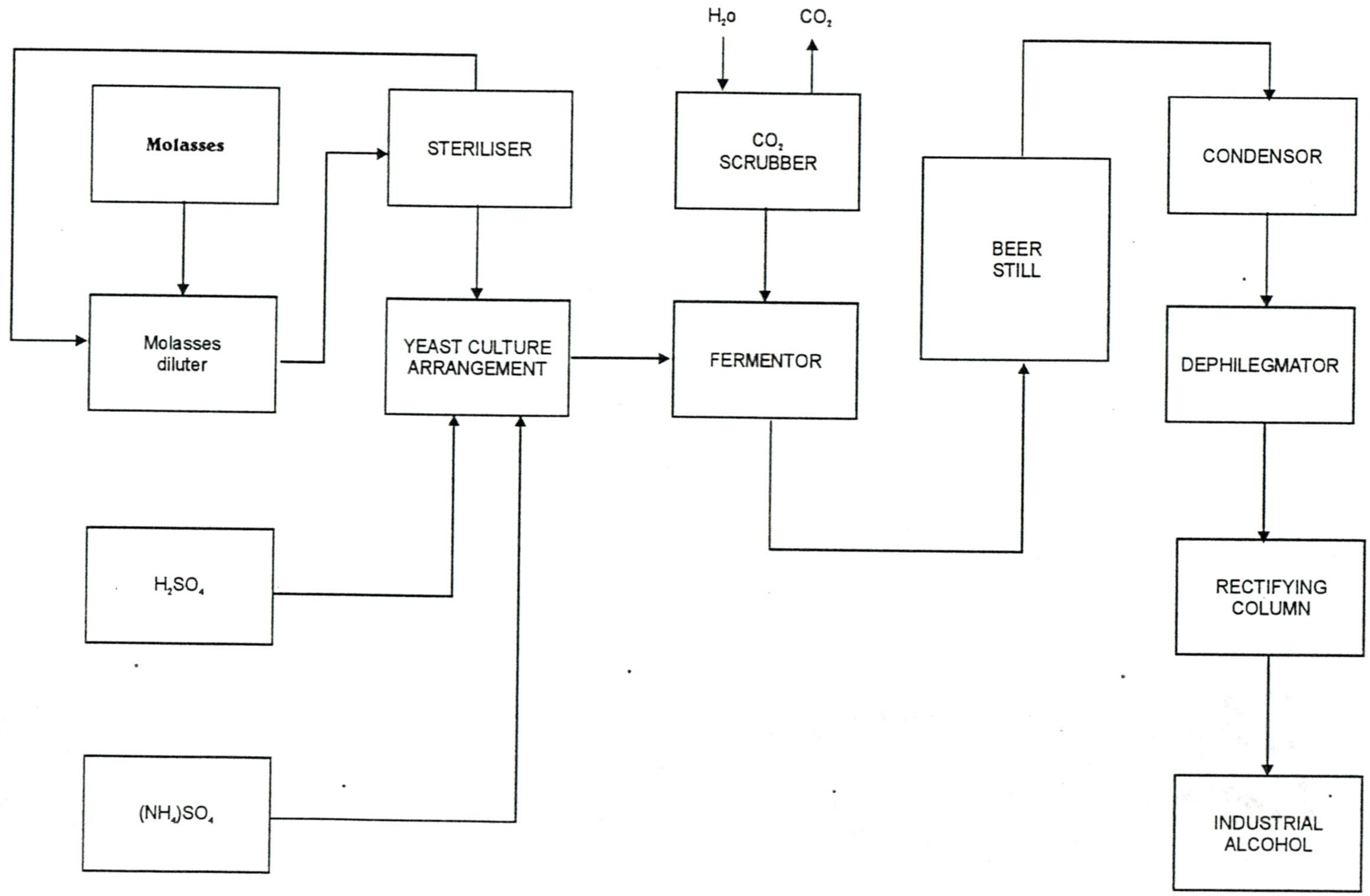
Different raw materials like Wheat, Maize, Potato, Cassava, rye etc have also been used for preparation of wort, which is then distilled to get alcohol (Mitra, 1985).

Of all the sugar bearing raw materials used for the production of alcohol, molasses is a very common and widely used commodity. Though there are various types of molasses, cane molasses is the most common type of molasses for the production of alcohol (Manohar Rao, 1983).

2.1.1 Manufacture Of Industrial Alcohol.

The process of manufacture of alcohol from molasses is mainly for the production of industrial alcohol. The molasses received from the sugar factories are first diluted with water to have a sugar concentration of 15% to 17%. The pH of the diluted molasses is adjusted to lie between 4.0 to 4.5 to suppress bacterial action and multiplication of wild yeasts. Nutrient supplements in the form of Ammonium sulphate or urea are added as a source of nitrogen. Then selected culture of yeast is inoculated into the mask for fermentation. (Manivasakam, 1997)

FIG.1 FLOW SHEET FOR THE MANUFACTURE OF INDUSTRIAL ALCOHOL



Fermentation

In the distillery, mollasses is pumped to overhead tank from where weighed quantities are lead through a continuous diluter to a fermentation tank. The specific gravity of molasses is 1.09-1.10. Readymade yeast cells are introduced into the fermentors. After the addition of nutrients of required quantities, the mixture is allowed to ferment under controlled conditions of temperature and pH for about 48 hours. Within this period 7-8 % of alcohol is formed.

During fermentation considerable amount of heat and carbondioxde are evolved. In order to prevent the temperature of fermentation raising about 97°C the fermentation vats cooled.

The following reactions takes place during fermentation.

$$C_{12}H_{22}O_{11} + H_2O \text{ ------------> } \underset{\text{d-glucose}}{C_6H_{12}O_6} + \underset{\text{d-fructose}}{C_6H_{12}O_6}$$

$$C_6H_{12}O_6 \text{ ------------> } 2\,C_2H_5OH + 2CO_2$$

Yeast sludge containing 30% of solids settles down in the fermentation vats and constitutes a major source of waste. The liquid is then distilled for the recovery of alcohol. The residues which are known as spent wash which is hot when discharged constitute important source of waste water from molasses distillery (IS:8032-1976)

Distillation

The fermented liquor is pumped to an overhead tank and allowed to flow from the top through two columns in succession. In the degasifying column., it is freed from carbon dioxide and other gases. As it passes down the second column., it is heated by injection of steam, where all alcohol is stripped, leaving a liquid free from alcohol. The liquid spent wash is discharged from the bottom of the column. The alcohol vapours issuing out from the second column are led to the base of rectifying column and gets rectified under reflux action to 95 to 99% alcohol, which is tapped, cooled and received in the spirit cylinder. The carbondioxide gas is collected washed and compressed into cylinders to aerated water industry.

Uses Of Alcohol

Duty paid pure ethyl alcohol is used only for medicinal, pharmaceutical, flavouring and beverage purposes. Duty free alcohol is low priced and hence its consumption has increased enormously and the field of application has also widened. Alcohol is second only to water in solvent value and is employed nearly in all industries. It is also used as the raw material for making numerous chemicals, such as acetaldehyde, ethyl acetate, acetic acid, ethylene dibromide. glycols, ethyl chloride etc. It may also be used as the raw material for butadiene and styrene. In countries with poor petroleum supply, it is economical to mix absolute alcohol with gasoline in order to conserve the petroleum products. Such alcohol is known as power alcohol (Chakrabarty, 1983).

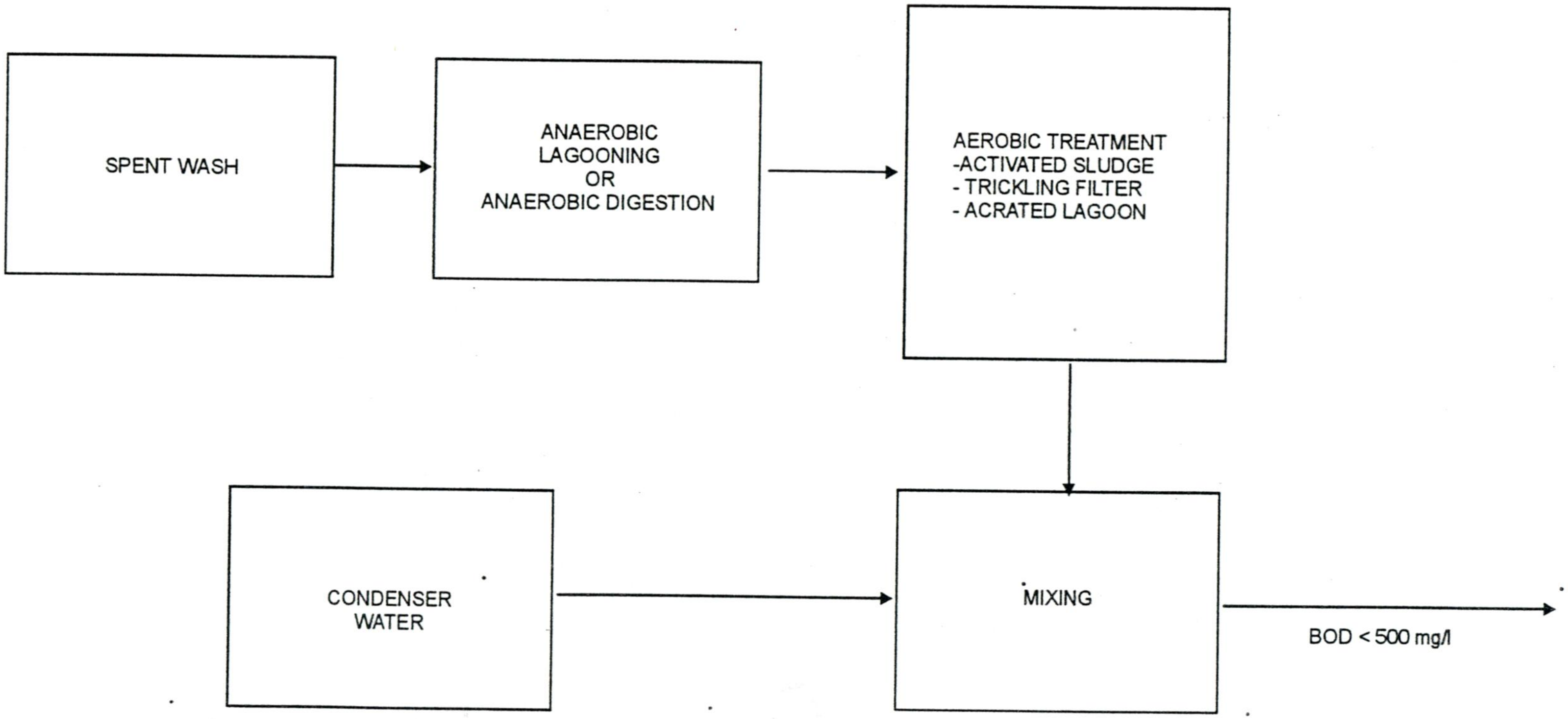
2.1.2 Sources Characteristics And Effects

The main source of waste from distilleries are

(i) Equipment and floor washing
(ii) Sludge waste from fermentation tank
(iii) Spent wash from distillation column (Agarwal , 1996)

10(a)

FIG.2 FLOW SHEET FOR THE TREATMENT OF SPENTWASH FOR DISPOSAL ON LAND



The spentwash is the major waste in molasses distillery. However some of the distilleries combine yeast sludge with spent wash stream. Insome distilleries the yeast sludge is sold out as manure (Manivasakam, 1997)

Spent wash resulting from cane molasses based alcohol industries is one of the most polluting industrial effluents . This waste is characterised by high BOD, high dissolved solids, and inorganic solids (Varma et al., 1979).

The colour of the spent wash and mixed waste is reddish brown and contains several organic and inorganic matters (Subbha Rao, 1980).

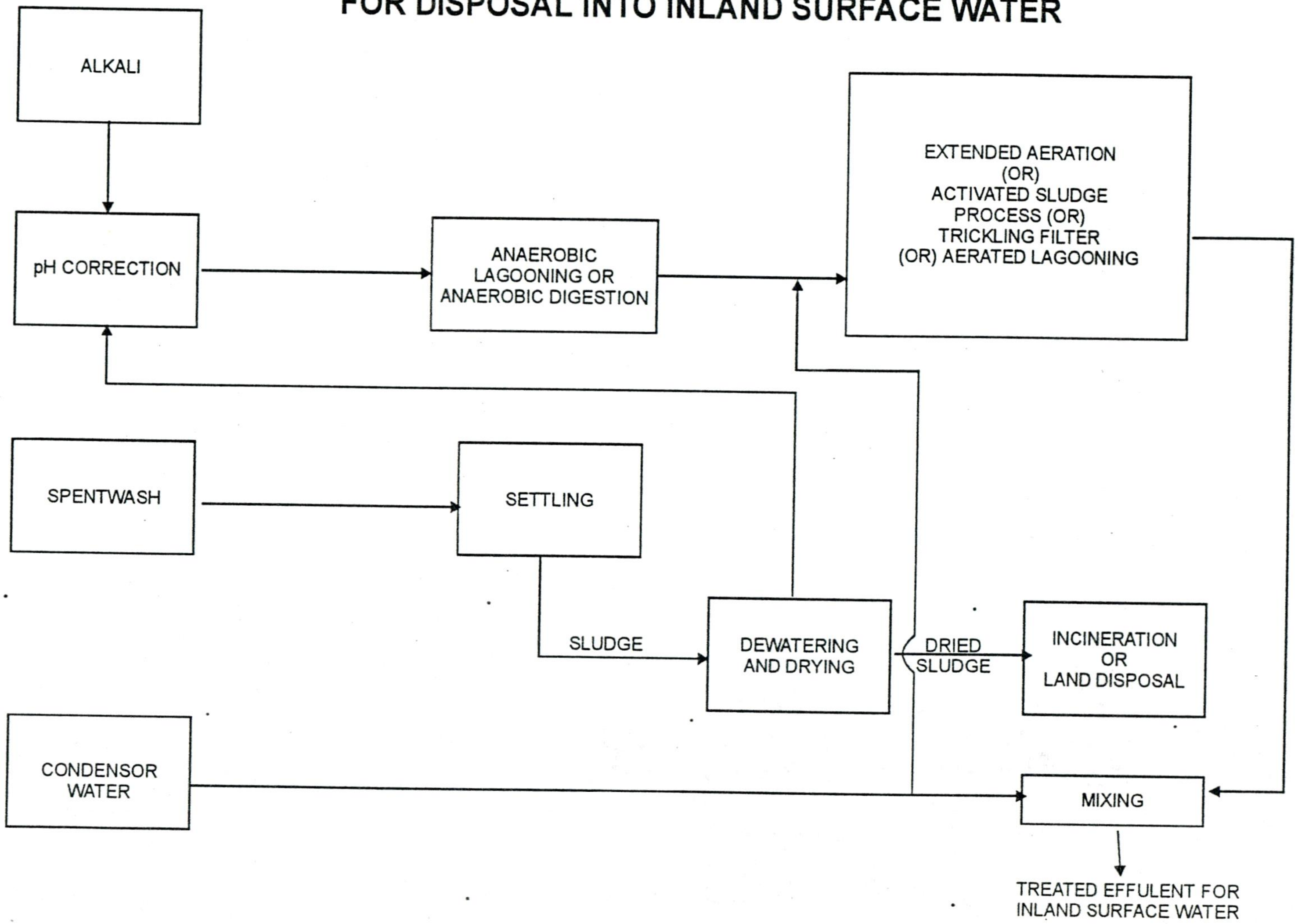
The waste water produced in a distillery carry a high organic load and cause severe fouling of the atmosphere. In addition when these waste water find their way into a river they cause severe depletion of oxygen content of water and this leads to deterioration of the water quality. It lowers the pH of the stream and also causes discoloration of water.

Discharge of spent wash on land results in ground water pollution. Stagnation of these effluents on land results in obnoxious conditions in this region.

Sharma et al., (1973) observed that discharge of spentwash into fresh water system resulted in the precipitation of ferric ions, which affected the respiratory activity of the fist by choking their gills through fine deposition. Suspended solids present in the effluent disturb the osmo - regulation and resulted in suffocation even in the presence of adequate dissolved oxygen. (Haniffa and Sundaravadanam, 1984) David and Ray (1996) reported high quantity of iron salts in effluent cause asphyxiation in fish resulting in reduced oxygen consumption..

FIG.3

FLOW SHEET FOR THE TREATMENT OF SPENTWASH FOR DISPOSAL INTO INLAND SURFACE WATER



Hart et al., (1947) found that the distillery waste should be diluted 16 to 20 times for its safe disposal into the fresh water resources.

Average composition of gas from spentwash (Chakrabarty et al.,1982)

GAS COMPOSITION	AVERAGE CONCENTRATION
Methane	55%
Carbondioxide	44%
Hydrogen sulphide	30-35 mg/cft
Hydrogen	Rest
Calorific value	530 BTo/cft

The biogas produced from spentwash can be entirely utilised for raising steam in the boiler of the production plant. It has been estimated that utilising the biogas generated in the digesters, the distillery will be in a position to save about 60% of their coal requirement (Chakrabarty et al., 1983)

Ghosh et al., (1962) studies that, on sulphite fermentation of twenty times diluted distillery liquor plus 0.1% urea and 5% gypsum powder , the yield of sulphide was observed to be 44.7 % and 32.6% of the mass of total a carbon present in the change at retention periods of 12 days and 6 days respectively. On the basis of the study a scheme was proposed in which treatment of 5000 litres of waste should produce 75000 litres of total gas composed of 45000 of methane, 6000 litres of hydrogen and 22500 litres of carbondioxide; 25.5 Kg of elemental sulphur and 21 kg of potash could also be recovered.

Minas For Distillery Effluents

Minimum national standard for distillery finalised that BOD level of 3000 mg/l to be achieved after anaerobic treatment. Resultant effluent to be diluted with water to bring BOD to 500 and used on land for irrigation.

Secondary two stage aerobic treatment was to be provide to bring down BOD 100 mg/litre, for application on land, and to 30 mg./litre for discharge into water courses.

2.1.3 Distillery Waste Treatment

Bio-Gas Plant - Present Method Of Treatment

The system works on fixed film biomethanisation process. The organic matter present in spent wash of a distillery are primarily non -sugars, highly putrisicible but are a rich source for the production of biogas. To obtain optimum production of gas, it is necessary to feed the waste to a digested at a controlled rate, under anaerobic conditions. The methonogenic bacteria which is mainly responsible for the release of methane as a metabolic product, will utilise the nutrients from the effluent and excrete biogas. Mixing temperature, loading and pH have great influence on the production of Biogas.

Anaerobic digestion of distillery waste showed gas yield of 0.56 m^3 and 0.11m^3 per kg of BOD reduction in 12 days detention time, the gas containing about 60 percent methane.

2.2 DISTILLERY SPENT WASH - AN USEFUL MANURE

2.2.1 Agricultural Utilisation

In India several distilleries located in villages and small town, use their spent wash for irrigation after dilution with water (IS:8032-1976). Pretzman

(1958) tried to utilise the diluted spent wash for irrational purposes. Singh (1961) used neutral spent wash because of its acidic nature and observe that after use the soils showed a marginal decrease in pH.

Excellent results have been obtained for getting bumper crops of cane, wheat, paddy, barley, etc. The distillery effluents, in addition to other nutrients contain potassium and calcium salts mainly which are used for the irrigation of the sugarcane fields. (Manohar Rao, 1983).

The disadvantage of this method is that it requires enormous dilution water and also leads to contamination of irrigation channels and ground water. It also results in foul smell when an overdosage of waste water is applied. Besides, it creates gradual death of soil when the land is continuously irrigated (Agarwal, 1996).

In Brazil, the effluents are transported in tankers and sprayed in the sugarcane field. In Australia, the latest method adopted is to pump the distillery effluents into a pond in the midst of sugarcane field and force it through a high pressure pump in the form of a jet and sprayed on the entire field uniformly. By this a part of the volatiles and water are evaporated and the concentrated effluent is spread uniformly on the soil, which is turned over by deep ploughing thereby the nutrients are uniformly mixed in the soil. Similarly, the effluent used for furrough irrigation is also mixed up in the soil by deep ploughing immediately after the application of the effluent on the soil. (Manohar Rao, 1983).

In one of the patented processes, the distillery effluent is concentrated and the concentrated mass is mixed up with bagasse or some similar fibrous agricultural residue and burnt in a furnace to raise steam. The residue left over is a mixture of NPK fertilizer.

2.3 RECOVERY OF BY PRODUCTS FROM SPENT WASH

By products from spentwash include potash, riboflavin, and other B vitamins particularly B12. yeast powder of pharmaceutical grade and animal feed can be obtained from the yeast sludge as it contains dead yeasts, proteins, vitamins, fats and carbohydrates (Manivasakam,1997).

Recovery of by-products from grain distillers and using them as a cattle feed, solves the disposal problems of the distilleries and brings in revenue. (IS:8032-1976).

Boruff (1952) have reported on the commercial production of riboflavin and other 'B' vitamins and an antibiotic from spentwash.

Some distilleries in India are practising recovery of dried yeast powder of pharmaceuticals quality from spent wash - yeast sludge mixture. (IS:8032-1976)

Bhaskaran (1962), reported that the sludge resulting from another aeration of distillery waste contained high concentration of vitamin B12 of the order of 800 μg/Kg of dry sludge and this can be used as a supplementary animal or poultry feed.

Potash Recovery:

Reich (1945) described a process wherein by dilution, acidfication and heating of molasses prior to fermentation about 88% calcium salts could be precipitated as calcium sulphate. Activated carbon and salts of potassium and sodium could be obtained by suitable treatment of spent wash.

Chakkrabarty and Bhaskaran carried out a pilot study to recover potassium salts from spent wash of a molasses distillery in India. In this process, raw spent wash was neutralised with lime and evaporated to about 75% solids in a forced circulation evaporator under vacums. Thick liquor was burnt in an incinerator where the spent wash was converted to a substance called 'spent wash coke", which burnt itself under the grates of the incinerator and turned into ash, thereby supplying necessary heat required for the incoming thick liquor. The ash was collected and leached with water, by which all the soluble potassium and sodium salts dissolved in it leaving behind iron, calcium and other impurities in the residue. The solution was alkaline due to the presence of potassium carbonate, which was filtered and neutralise with sulphuric acid and further concentrated in the evaporator. Potassium salts in the form of chloride and sulphate were crystallised from the concentrated solution in the crystalizer.

It has been claimed that a distillery in India producing on an average about 3220 kl of spent wash per day could recover about 3.86 tonnes of potash (K.[illegible]) or about 5.7 tonnes of potassium sulphate and 1.27 tonnes of potassium chloride per day.

TABLE - 2

COMPOSITION OF EXTRACTED POTASSIUM SALTS FROM SPENT WASH

	PERCENT
Potassium Chloride (KCl)	16.5
Potassium sulphate (K_2SO_4)	73.5
Sodium salts	5
Moisture	5

2.4 UTILISATION OF PRESSMUD, SUGAR INDUSTRY WASTE IN AGRICULTURE

The gravity of environmental degradation because of faulty agriculture has set attention on ecologically sound, viable and sustainable farming systems. One such system which will help to overcome the problems of soils and environmental degradation is organic farming - which aims at co-operating rather than confronting with the nature (Gunjal, 1991).

Organic farming is a production system which avoids or largely excludes the use of synthetically compounded fertilisers, pesticides, growth regulators and livestock feed additives and rely upon crop residues, animal manure's and aspects of biological pest control to maintain soil productivity and tilth.

In simpler terms organic farming is defined as farming without chemicals which is only a misconception.

Compost

The maintenance of organic matter in the soil is one of the fundamental problems of agriculture and good farming goes hand in hand with maintenance of organic matter with soil at a satisfactory level. All organic wastes may be collected and used for augmenting the organic matter suppliers. They cannot be used directly as manure in most cases and required to be decomposed before application. The process of decomposing organic wastes are called composting and the decompost materials is called compost (Singaram, 1994).

Aerobic Composting

At present the majority of methods in used are aerobic. They insist on securing thorough aeration of the mass during the process of composting. It is possible that such intensely aerobic methods are specially adopted to deal with resistance types of waste materials which are poor in nitrogen and undergo decomposition slowly. But the indiscriminate application of the same treatment often leads to heavy losses of organic matter and nitrogen (Yawalkar et al, 1972).

An intensive aerobic treatment has the advantage that it shortens the period of composting and the method would prove useful in cases where the refuses has to be disposed of quickly. But a slow fermentation process is preferred by the farmers as the manure is required in particular seasons, only which may be once or twice in a year.

Press mud is a "WASTE" product obtained during sugar manufacture and it constitutes about 4% of the crushed cane. It contains organic matter, sulphur and phosphorous and other nutrients in varying amounts depending upon the materials used by the sugar factory.

Press mud is known variously as milo, filter mud, mill mud, filter press or scum (Alexander, 1972). A factory with 2000 million tonnes of cane crushing capacity produces 60-70 of pressmud perday (Mariappan et al., 1993).

Most of the pressmud is usually dumped on the factory premises where it accumulates like hillocks (Samuel and landrau, 1955). Pressmud is spread into sugarcane fields and incorporated in the soil as a conditioner (Bawaskar et al., 1982).

Alexander et al., (1972) note that presumed, though an industrial waste contains fairly high quantities of macro and micro nutrients. Its value as fertiliser is attributable to its phosphate content and appreciable amounts of Ca, Mg, S & K.

Narwal et al., (1990) noticed that pressmud is a good source of plant nutrient containing 43.2% of organic carbon, 3.08% of N and 0.62% of P. It may serve as an economical and ecofriendly fertilizer supplementing costly chemical fertilizers.

In India press mud produced is of two types carbonation and sulphitation pressmud depending upon the method of clarification followed by the sugar factory.

Manurial Value Of Press Mud

Sulphitation press mud contains about 1.0-3.1% N, 0.6-8.6 % P and 0.3-1.8% K in addition to secondary and micronutrients on dry weight basis. It is estimated that sulphitation press mud in India had an annual potential of supplying 7,992-24,775 tonnes of Nitrogen, 4,795-24,770 tonnes of phosphorus and 2,398 - 14,875 tonnes of potassium besides secondary and trace elements. Potential nutrient supplies from pressmud reported by Tambhenar (1992) are of much high order (60,000 t of N; 105,000 t of P_2O_5 and 1,00,000 t of K_2O)

Utilisation Of Pressmud

Pressmud has been used mainly as

(i) source of plant nutrients

(ii) an ameliorant for acidic, and saline - sodic soils

(ii) as a medium for raising sugarcane seedlings

(iv) as a carrier for leguminose inocuolants

A year old pressmud is superior to new or fresh mud for crops. Sulphitation pressmud is more effective as it contains sulphur (Chand et al., 1977). Pressmud, when applied directly to the land, there is considerable bacterial activity and the bacteria utilise the nitrogen in the soil for their growth and the crop following is starved of nitrogen for a period temporarily.

After the decomposition of press mud, the bacteria, die out and the nitrogen taken from the soil is returned to it as bacterial protein and it becomes available for the use of crops in course of time. In view of this drawback the filter mud can be composted and applied to the land (Subbiah Mudaliar, 1960).

Press mud has also been suggested to be used as a manure along with distillery spentwash than pressmud alone according to (Thopale et al., 1991) when applied directly it is likely to burn the plant like fresh organic manure. It has therefore to be cure for 6-7 months. The addition of distillery spentwash partially cures the pressmud and hastens its decomposition to about 3 months.

Press Mud Composting

Pressmud composting can also be done by mixing with distillery effulent which is also a rich nutrient source. Pressmud from sugar factory is formed into windrows of 100 metres high x 3 metres width x 1 metre height of concrete floor. Fresh press mud with a moisture content of 75 percent is worked with aerotiller to reduce the moisture below 50 percent. It is mixed with additives containing waste materials like composted coir pit h 5 percent, sugar cane trash 2.5 percent and phosphorus is added as rockphosphate 2.5 percent in the beginning. The compost culture containing thermophilic microbes bacillus species and pseudomnoas species are inoculated with the materials at 1.5 litres ton of pressmud. Then the distillery effluent is sprayed over the windrows periodically at the ratio of 1:25 and the windrows are pulverized and aerated regularly. The heap is worked with aerotiller and reformed. A temperature of 60 to 70°C is formed and after 4 to 5 days, next dose of effluent is added when the moisture content goes below 50 percent. Again the materials is worked with aerotiller and reformed. The composting of pressmud and effluent are thus added, worked and reformed. The entire addition of effluent is completed in about 1.5 to 2 months period. The superphosphate at the rate of 2.5 percent is added to the heap and mixed. The hot heap is kept undisturbed for curing for a period of 15 days.

Then the micronutries like $FeSO_4$ and $ZnSO_4$ at the rate of 20 Kg per ton of pressmud is added for enrichment of these micronutrients and allowed for composting pressmud for yield application it is further enriched with biofertilizer viz, Azotobacter, Azospirillam and phosphobacterium at 2 Kg/ton of the composted material This enhance free nitrogen fixation and solubilization of native and added phosphorus. The final product has the moisture of 80 percent for enriching the pressmud, cultures of trichoderma viride or pleurotus species should be added per tonne of pressmud. Urea, cowdung or well decomposed farmyard manure were added as additives, increases the activities of both added microbial culture and native flora of pressmud. The presmud thus decomposed may be used as a starter for subsequent enrichment of pressmud. (Padmanaban et al, 1993).

Carbonation of pressmud has also been attempted to be used as distempers and portland cement but so far none of the products have found industrial usage due to the inferior quality of the products made.

Pressmud is mainly used as a fertilizer but the process has been now developed to manufacture was out of it which is very much used in boot and paint polishes (Mathur, 1981).

Biocomposting of pressmud helps in recyling of agricultural wastes and also instead of dumping the wastes in one particular area as the input came from different fields viz., about 20,000 acres for sugar factory the recycling also needs to be done to the same extent of 20,000 acres, area to become more scientific and judicious. This type of recycling is more valid in days to come since the concept of organic farming and sugar production are in the anvil.

CHAPTER 3

Materials and Methods

MATERIALS AND METHODS

Industry has always been and continues to be the prime cause of economic development all over the world (Niyate, 1993) with gradual industrialisation and urbanization the problem of pollution is increasing as the effluent from all the industries find their way into rivers.

Usually the effluent from different industries are let out on land for irrigation purposes or into surface waters like rivers, canals, tanks etc. But this method of disposal is highly objectionable as the effluent find their way into drinking water. Hence drastic treatment of the effluent is concerned.

The present study was aimed to assess the physcio-chemical characteristics of the distillery effluent and the NPK value of the pressmud.

3.1 CHARACTERIZATION OF DISTILLERY SPENT WASH

3.1.1 Selection of the industry

Effluent from industries manufacturing pesticides, chemicals, fertilizers, paper and drugs etc. may contain constituents which are dangerous to human health.

So far as the distillery is concerned, the disposal of spentwash has found to be neither satisfactory nor a successful one. The present method of treatment is biomethanisation which is found to be more economical. Moreover valuable by products can be recovered from distillery spentwash and can also be used as manure.

3.1.2 Collection of the Sample

Sufficient volume of sample for physcio-chemical analysis was collected from the industry in polythene carbouys to carry out all determinations. The sampling point was chosen carefully so that a representative sample of the water to be tested is obtained.

The effluent was collected at two areas, one from the primary outlet and the other from the secondary outlet of the treatment plant installed at the premises of the industry.

Three samples of raw and composted pressmud was collected at the site of compost yard and analysed for the major nutrients (NPK) present in it.

3.1.3 Physical Properties

Colour: Colour of the samples were noted at the time of receipt of the samples.

Odour: Odour was felt at the time of receipt of the samples by normal sniffing at room temperature to identify whether pleasant or unpleasant.

3.1.4 Techniques Used For Chemical Analysis

1. **pH** - Electronic pH meter was used.
2. **Total suspended solids** - Filtration technique was used
3. **Total dissolved solids** - Evaporation technique were followed.

4. Biochemical Oxygen Demand (B.O.D)

Biochemical Oxygen Demand is a measure of oxygen required when the waste is decomposed by bacteria. Water saturated with dissolved oxygen was added to the sample to be tested and kept at a constant temperature of 20°C for 5 days in a closed incubation bottle. The B.O.D was calculated from the difference in dissolved oxygen.

5. Chemical Oxygen Demand

COD signifies the amount of oxygen required to oxidise the organic matter. The sample reflexed for a hour in strongly acidic solution with a known excess of potassium dichromate. After digestion, the remaining unreduced $K_2Cr_2O_7$ was titrated with ferrous ammonium sulphate to determined the amount of $K_2Cr_2O_7$ consumed and the oxidizable organic matter was calculated in terms of oxygen equivalent.

6. Chloride

Estimation of chloride was performed with the help of titrating method against silver nitrate using potassium chromate as indicator. The flesh red precipitate of silver chromate was found to be the end point.

7.Sulphate

Estimation of sulphate was done by Turbidimetric method. i.e., the sulphate ion precipitate in an acidic medium with Barium chloride formed Barium sulphate crystals of uniform size. Light absorbance of Barium Sulphate suspension was measured using spectrophotometre and the sulphate ion concentration was determined by comparison of the reading with the standard curve.

8. Total Nitrogen

Total nitrogen is the sum of ammonia nitrogen and organic nitrogen. The classical kjeldhal methodis used to determine the total nitrogen content.

9. Phosphate

Phosphate was estimated by calorimetric method.

10. Potassium

Potassium was found in appreciable concentrations is spent wash. Potassium was best determined by flame photometric method.

3.2 CHARACTERISATION OF PRESSMUD

1. Total Nitrogen

Total nitrogen is the sum of ammonia nitrogen and organic nitrogen. The classical kjeldhal method is used to determine the total nitrogen content.

2. Phosphate

Phosphate was estimated by calorimetric method.

3. Potash

Potassium was best determined by flame photometric method.

CHAPTER 4

Results and Discussion

RESULTS AND DISCUSSION

Even though mineral fertilisers are required to achieve food production targets, the limitation is the availability of fossil fuel makes the cost of fertilisers enormously high. Use of inorganic fertilisers alone creates infertility, unfavourable soils, physical and biological conditions, complementary use of available renewable sources of plant nutrients like organics and biologics is of great importance for soil health.

Sewage and other waste water from industries in the form of effluents are rich in organic matter and other elements, which when discharges on land or water causes pollution. The organic matter in the waster water can be recycled and the pollution due to it is effectively reduced. Similarly the distillery effluent also posses a high organic load which can be used as a manure after dilution.

4.1 CHARACTERISTICS OF THE UNTREATED EFFLUENT

4.1.1 Physical Properties

TABLE -3

CHARACTERISTICS	RESULTS
Colour	Dark brown
Odour	Highly Dangerous

The effluent appeared dark brown in colour. The odour was highly disagreeable which is the characteristics odour of the molasses, used as the raw material in the manufacture of alcohol.

4.1.2 Chemical Properties

TABLE 4

CHARACTERISTICS	RESULTS
pH	3.9
Total suspended solids	63,000
Total dissolved solids	35,600
Biochemical oxygen demand	40,000
Chlorides (as cl)	4,240
Sulphates (as SO_4)	4,400
Ammoniacal Nitrogen	977
Potassium	10,200

The value except pH are expressed in mg/l.

The pH of the effluent was found to be 3.9 which is acidic in nature. The total solids highly exceeded the limits for discharge into inland waters.

The biochemical oxygen demand was extremely high. Likewise the values of chlorides, sulphates and nitrogen was far above the limits. The potassium content of the effluent was also too high but that makes the effluent usable for irrigation.

Some of the specific tolerance limits for the effluent discharges into inland surface waters prescribed by Indian standards are given in Table:5

4.1.3 Specific Tolerances For Effluents Of Distilleries

Table :5

CHARACTERISTICS	TOLERANCE LIMITS
Colour and odour	Absent @
Suspended solids	100 mg / l
Biochemical oxygen demand	30 mg / l

No requirement have been laid down in the Indian standard for colour and odour but it is recommended that as far as practicable, colour and unpleasant odour should be absent in the effluents.

Thus the present study is an attempt to utilise the treated effluent in combination with pressmud as a manure in Agriculture.

4.2 SPENTWASH CHARACTERISTICS AFTER PRIMARY TREATMENT

4.2.1 Physical properties

Table 6

CHARACTERISTICS	RESULTS
Colour	Dark brown
Odour	Highly Disagreeable

4.2.2 Chemical Properties

Table : 7

CHARACTERISTICS	RESULTS
pH	7.6
Total suspended solids	1,500
Total dissolved solids	35,386
Biochemical oxygen demand	580
Chemical oxygen demand	30,800
Total nitrogen	120
Potassium	9,300
Phosphate	13.5

All values except pH are expressed in mg / l

The physical properties of the effluents showed no appreciable change after primary treatment.

Considering the chemical properties, the pH was found to be 7.6. The suspended solids has been reduced to 1500 mg/l, so also the BOD to 580 mg/l. But the chemical oxygen demand was found to be still higher.

4.3 SPENTWASH CHARACTERISTICS AFTER SECONDARY TREATMENT

Physical Properties

Table 8

CHARACTERISTICS	RESULTS
Colour	Brown
Odour	Disagreeable

The physical properties of the finally treated effluent has shown only a very small change.

4.3.2 Chemical Properties

Table 9

CHARACTERISTICS	RESULTS
pH	7
Total suspended solids	325
Total dissolved solids	20,730
Biochemical oxygen demand	300
Chemical oxygen demand	12,800
Total nitrogen	11.5
Potassium	5,000
Phosphate	12.5

All values except pH are expressed in mg/l

After secondary treatment the value of BOD has reduced considerably, but the dissolved solids and COD was still higher than the prescribed limits.

The average percentage of N, P_2O_5 and K_2O in the raw and composted pressmud are given in the tables below.

4.3.2 Percentage Of Major Nutrients In Raw Pressmud And Composted Pressmud

TABLE 10

PROPERTIES	RAW PRESSMUD	COMPOSTED PRESSMUD
N	1.05 %	1.5 %
P_2O_5	1.40 %	1.7 %
K_2O	0.6 %	2.7 %

TABLE 11

PROPERTIES	RAW PRESSMUD	COMPOSTED PRESSMUD
N	1.09 %	1.8 %
P_2O_5	1.5 %	2.1 %
K_2O	0.75 %	2.9 %

TABLE 12

PROPERTIES	RAW PRESSMUD	COMPOSTED PRESSMUD
N	1.3 %	2.1 %
P_2O_5	1.5 %	2.3 %
K_2O	0.75 %	3.2 %

The amount of major nutrients present in composted pressmud was higher when compared to that of the raw pressmud.

4.5 PERCENTAGE OF MAJOR NUTRIENTS IN SYNTHETIC FERTILIZERS

TABLE 13

PROPERTIES	PERCENTAGE
N	21.2 %
P_2O_5	20.5 %
K_2O	0.6 %

Eventhough the NPK content of synthetic fertilisers was found to be higher, they may have an adverse effect in soil. Continuous application of mineral fertilisers resulted in lower yield besides declining soil health; while continuous use of composted manure alone has significantly enhanced the yield of crops.

FIG. 4

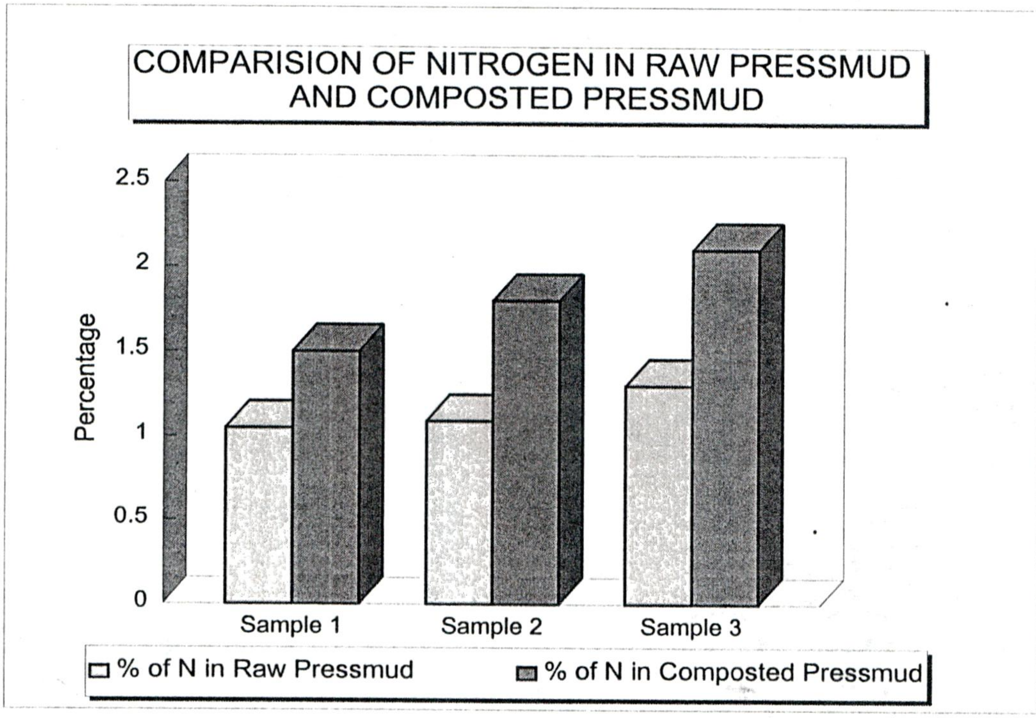

COMPARISION OF NITROGEN IN RAW PRESSMUD AND COMPOSTED PRESSMUD
2.5
2
1.5
1
0.5
0
Percentage
Sample 1
Sample 2
Sample 3
% of N in Raw Pressmud
% of N in Composted Pressmud

FIG. 5

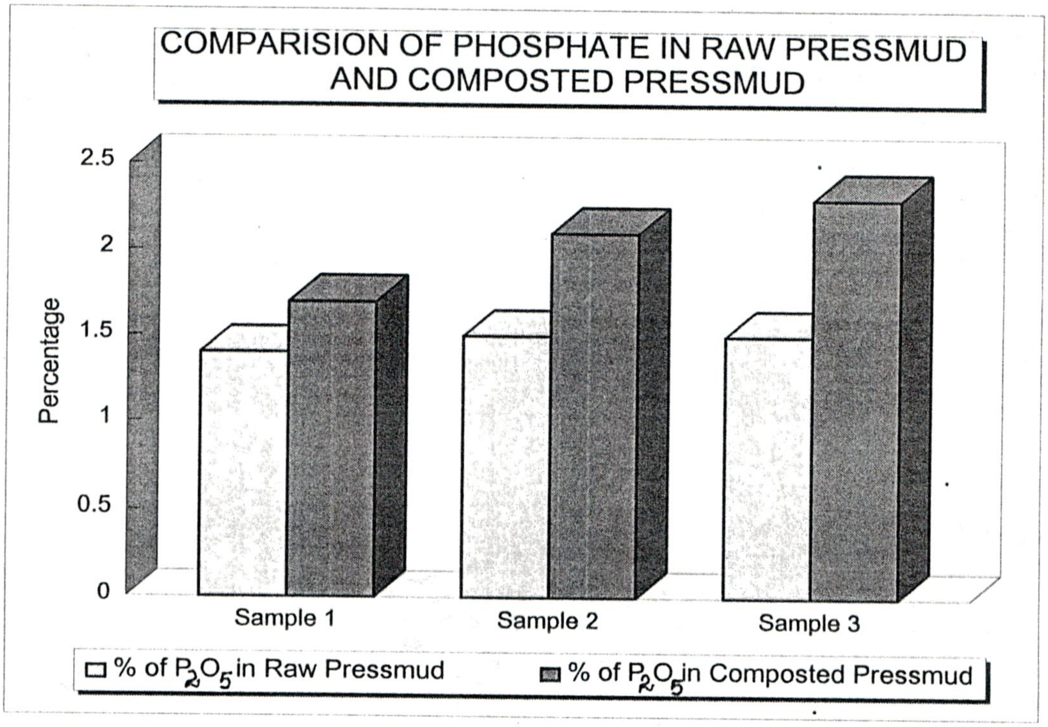
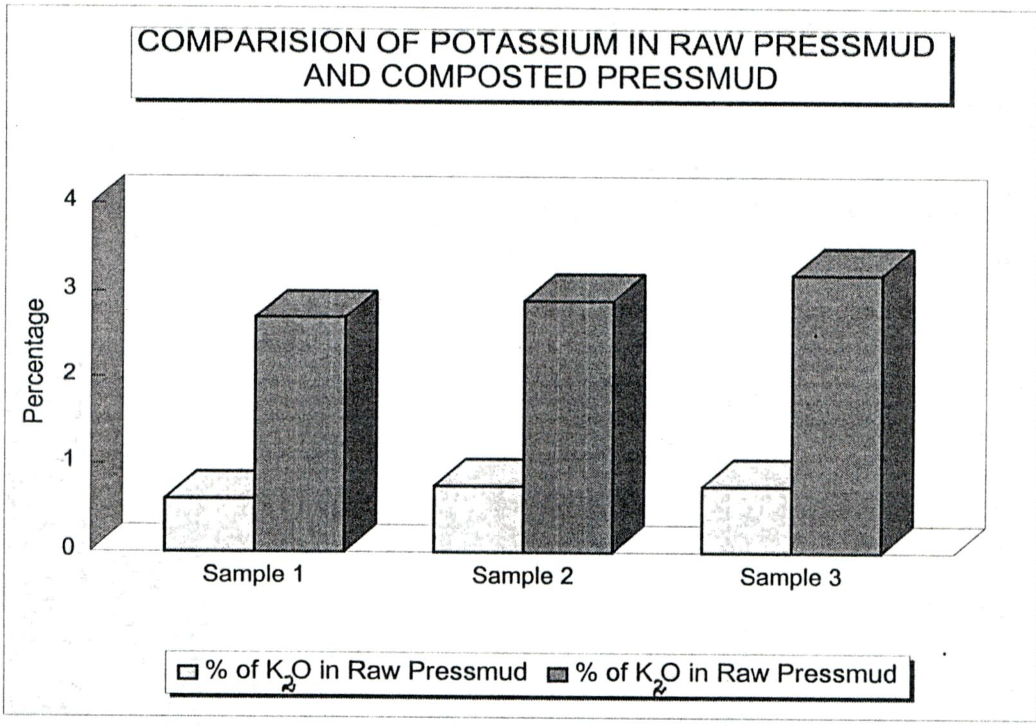

COMPARISION OF PHOSPHATE IN RAW PRESSMUD AND COMPOSTED PRESSMUD
2.5
2
1.5
1
0.5
0
Percentage
Sample 1
Sample 2
Sample 3
% of P_2O_5 in Raw Pressmud
% of P_2O_5 in Composted Pressmud

FIG.6


COMPARISION OF POTASSIUM IN RAW PRESSMUD AND COMPOSTED PRESSMUD
Percentage
4
3
2
1
0
Sample 1
Sample 2
Sample 3
% of K2O in Raw Pressmud
% of K2O in Raw Pressmud

The use of composted pressmud has recovered 20% of chemicals in organic fertilisers; while the prolonged use of inorganic fertilisers has diminished the inherent fertility of the soil.

Moreover the application of composted manure was more easier as no technical skills required and also it was found to be economical.

Application of nutrients through organic sources was found to reduce insect infestation when compared to inorganic fertiliser application (Eigenbrode and pinmetal, 1988).

This is due to enhancing the nature enemy activity against pests and reducing this susceptibility of host by consuming soil nitrogen and restricting luxury uptake of nitrogen by the crop.

The addition of organics improves the properties of soil. It acts as a source of plant minerals which are not present in the chemical fertilisers. It improves soil aeration and the cat ion exchange capacity. Organic matter increases water holding capacity and nutrient holding power. It also acts as a source of carbon for the multiplication of number of micro organisms. More the micro organisms and their activities resulted in increased mineralisation. More the mineralisation leads to more availability of plant nutrients. It not only increases the soil fertility but also the productivity. Hence organic materials are the saviour of soil environment.

In conclusion it can be emphasised that organic farming not only helps in recyling of organics coupled with environmental protection but also sustains the soil health physically, biologically and chemically with quality improvement of crops in long run.

CHAPTER 5

Summary and Conclusion

SUMMARY AND CONCLUSION

Industrialisation, no doubt makes a nation supporting and provides more avenues for employment. However, the variety of wastes generated during the process possess numerous environmental problems.

There is now a global awakening on pollution control through the recovery of reusable matter from waste effluents, salvaging of recovery of usable materials and by-products could contribute towards saving of expenditure on one hand and pollution control abatment on the other. Reuse of waste water within the industry would help in minimization of freshwater requirements and at the same time reduces the volume of the waste water for final treatment before discharge (Mc.Laughlin et al., 1992).

Today most of rivers receive million of litres of sewage, wastes, industrial and agricultural wastes containing substances varying in characteristics from major nutrients to ones of high toxicity.

The spent wash of Distilleries rich in organic matter is discarded into water bodies resulting in environmental pollution. Thus the present study was an attempt to compost pressmud, a sugar industry waste by mixing with distillery waste water which was found to be a rich nutrient source and used as a manure.

The physico-chemical characteristics of the effluent were studied and the amount of major nutrients present in the composted pressmud was analysed.

The summary and conclusion arising out of the study are, the effluent was brown in colour and has a disagreeable odour. The pH was found to be 7.0 and had slightly objectionable levels of BOD and COD. The dissolved solids was also higher than the limits. Nitrogen and phosphate was found to be in considerable amounts and the level of potassium was high but that can be used up by utilising the effluent for irrigation after dilution.

The composted pressmud possessed a higher value of the major nutrients (N,P,K) than the raw pressmud and their application increased the yield of crops thereby saving 20% of chemicals in organic fertilisers.

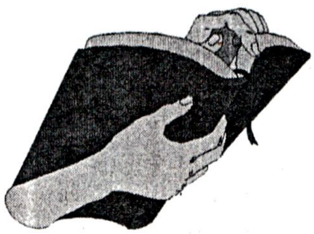
India has planned for replacing 15% fertilisers input through Biofertilizers and the high price of factory made inputs, environmental degradation, depletion of soil organic matter, nutrient deficiencies and ground water pollution have made us to reorient our studies towards lesser use of synthetic chemicals. Combined use of biofertilisers and reduced doses of chemical fertilisers would not only give higher yield but help to maintain soil health and reduce pollution problems created by the application of high dose of chemical fertilisers.

SCOPE FOR FUTURE STUDIES

1. Citric acid can be manufactured by aerobic fermentation of crude sugar by a special strain of **Aspergillus Niger**.

2. The process of composting pressmud with distillery effluent can be improved such that it can be done in a short period of time.

3. The practice of nutrient recycling can be re-emphasized.



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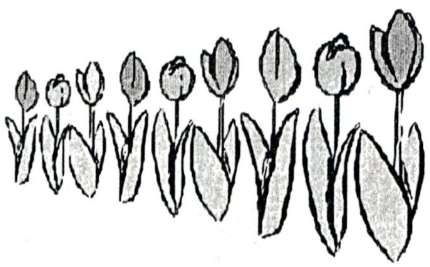

Appendices

APPENDIX I

DETERMINATION OF P^H

ELECTROMETRIC METHOD

Principle

P^H is a term used universally to express the intensity of the acid or alkaline of a solution.

Reagents : Buffer solution with P^H 4,7 and 9.

Procedure : The electrode systems were calibrated against standard with the help of P^H meter

APPENDIX II

DETERMINATION OF TOTAL SUSPENDED SOLIDS FILTRATION METHOD

Principle

A well mixed sample is filtered through a weighed standard whatmann filter paper and the residue retained in the dired to costant weight at 103°C to 105°C. The increase in the weight of the filter paper represents the total suspended solids.

Procedure

25 ml of sample was filtered through a funnel using weighed whatmannfilter paper. It was kept in oven for one hour at 105°C and in dessicator for 45 minutes.

Calculation

$$\text{Total suspended solids} = \frac{(W_2 - W_1) \times 1000 \times 1000}{\text{Volume of sample}} \text{ mg/liter}$$

W_1 - Initial weight of filter paper

W_2 - Final weight of filter paper

1000 for conversion of gram to milligram

1000 for conversion of milli litre to litre.

APPENDIX III

DETERMINATION OF TOTAL DISSOLVED SOLIDS EVAPORATION METHOD

Principle

A well mixed sample is filtered through a standard whatman filter paper No: 30 and the filterate is evaporated to dryness in a weighed dish and dried o constant weight at 180°C. The increase in dish weight represens the total dissolved solids.

Procedure

25 ml of sample was filtered through funnel using whatmann flter paper. Filterates was collected in a weighed silica bowl and it was kept in a water bath for drying after drying. It was kept in an oven for two hours at 180°C and then in dessicator for 45 mts.

$$\text{Total suspended solids} = \frac{(W_2 - W_1) \times 1000 \times 1000}{\text{Volume of sample}} \text{ mg/liter}$$

W_1 - Initial weight of bowl

W_2 - Final weight of bowl

1000 for conversion of gram to milligram

1000 for conversion of milli litre to litre.

APPENDIX IV

DETERMINATION OF BIOCHEMICAL OXYGEN DEMAND STANDARD TITRATION METHOD

Principle

The dissolved oxygen content of the sample is determined before and after 5 days at 27°C. Samples devoid of oxygen or containing less amount of oxygen are diluted several times with special type of dilution water saturated with oxygen inorder to provide. Sufficient amount of oxygen.

Reagents

1. Phosphate buffer solution : 8.5 of patassium dihydrogen phosphate and 1.7 gm of ammonium chloride was dissolved in 500 ml disilled water and made upto 1 litre.

2. Magnesium sulphate solution : 22.5 of mangesium sulphate was dissolved in distilled water and made upto 1 litre.

3. Calcium chloride solution: 27.5 gm of anhydrous calcium chloride was dissolved in distilled water and made upto 1 litre.

4. Ferric chloride: 0.25 gms of ferric chloride was dissolved in distilled water and diluted to 1 litre.

5. Manganese sulphate : 450 of manganese sulphate was dissolved in 1 litre.

6. Alkali iodide Azide reagent:

500 gm sodium hydroxide, 135 gm sodium lodide are dissolved in one litre distilled water. To this log sodium azide in 40 ml was added.

7. Sodium thiosulphate - 0.025 N

8. Sodium patassium dichromate solution - 0.025 N

9. Potassium iodide : 2 gm potassium Iodide in 150 ml distilled water.

Standardisation

10 ml potassium Iodide, 9 ml sulphuric acid and 20 ml potassium dichromate solution were taken and the mixture was titrated against 0.025 N thio using starch indicator. The end point was the disappearance of blue colour.

Preparation of Dilution water

Good quality of distilled water was aerated with a supply of clean compressed air, after bringing the temperatre to 26° C. 1 ml of each calcium chloride solution, magnesium sulphate solution ferric chloride solution and phosphate buffer solution were added.

Pretreatment

1. Sample was neutralised with sulphuric acid using p^H meter

2. Samples were allowed to stand for 1 to 2 hours, to remove residual chlorine.

Dilution technique

Suitable volumes of the sample were measured directly into bottles of known capacity with a pipette and the bottles are fitted with sufficient dilution water to permit insertion of the stopper without leaving air bubbled.

Blank was prepared using dilution water alone. Blank and diluted samples are kept in a incubator for 5 days in dark at 27°C.

Procedure

After 5 days the bottles were removed from the incubator. 1 ml of manganese sulphate solution, 1ml of alkali iodide azide solutions were added. Bottles were stoppered carefully to exclude air bubbles and it was mixed by inverting the bottles. The precipitate settles leaving a clear supernated liquid. After 2minutes 2 ml conc. sulphuric acid was added. It was restoppered and mixed by gentle invertion until dissolution was completed 100 ml sample was taken and titrated against thio using starch indicator. The end point was the appearance of blue colour.

Calculation

$$\text{Biochemical oxygen demand} = \frac{(B-S) \times 1000}{\text{Volume of sample}} \times N \text{ mg/l}$$

B - ml thio consumed by blank

S - ml thio consumed by sample.

APPENDIX V

DETERMINATION OF CHEMICAL OXYGEN DEMAND

CLOSED REFLUX METHOD

Principle

COD is measure of oxygen consumed during the oxidation of the oxidsable organic matter by a strong oxidizing agent. Organic matter are oxidised by boiling a mixture of chromic and sulphuric acid smaples was refluxed in strong acid solution with a known excess of potassium dichromate. After digestion the remaining un reduced potassium dicromate is titrated with ferrous ammonium sulphate, the amount o potassium dichromate was determined and the amount of oxidisable organic matter is calculated interms of oxygen equivalent.

Reagents

1. Standard potassium dichromate solution (0.25N)

Dissolved 12.25 gms of potassium dichromate in distilled water and made upto 1 litre.

ii. Silver sulphate

iii. Conc.Sulphuric acid

iv Ferroin indicator

v. Standard ferrous ammonium sulphate [0.25N]

Procedure

(a) Standardisation : 10 ml of potassium dichromate of strength 0.25 was taken and 30 ml of concentrated sulphuric acid was added, looked and titrated against ferrous ammonium sulphate using ferroin indicator. The end point was the appearance of bluish green to reddish brown coloured.

(b) 20 ml of sample. 20 ml of 0.25 N potassium dichromate was taken in refluxin flask and 30 ml of conc.sulphuric acid was added, allowed to cool and a pinch of silver sulphate was added. The flask was attached to the condenser and turned on cooling water. The refluxing was done for 2 hrs. The reflex condenser was disconnected and the flask was cooled, diluted with distilled water and titrated against ferrous ammonium sulphate using ferroin indicator. The end point was the appearance of bluish green to reddish brown. Blank was carried out as same mentioned above

Calculation

$$\text{COD mg/l} = \frac{(B-S) \times \text{Nomality of FAS} \times 8 \times 1000}{\text{Volume of sample}}$$

B - ml FAS consumed by blank

S - ml FAS consumed by sample.

APPENDIX VI

ESTIMATION OF CHLORIDE
ARGENTOMETRIC METHOD

Principle

In a neutral or slightly alkaline solution, potassium chromate can indicate the end point of the silver nitrate titration of chloride. Silver chloride is precipitated quantitatively, before red silver chromate is formed.

Reagents

1. Potassium chromate indicator solution

2. Standard silver nitrate solution 2.395 gm silver nitrte was dissolved in distilled water it was diluted to 100 ml. It was titrated against 0.0141 sodium chloride solution. p^H was adjusted to 7 to 10 using sulphuric acid. 1 ml of potassium chromate indicator was added. It was titrated against standard silver nitrate. The end point was the appearance of pinkish yellow.

Procedure

10 ml of the sample was taken in a conical flask. To adjust P^H range of 7 to 9.5, sulphric acid was added. It was titrated against standard silver nitrate solution using potassium chromate indicator with constant stirring until a slightest perceptible reddish colouration persist. Blank was conducted by placing 10 ml chloride free distilled water instead of sample.

Calculation

$$\text{Chloride} = \frac{(A-B) \times \text{Normally} \times 35.45 \times 1000}{\text{Volunme of sample}}$$

A - ml of silver nitrate solution for sample

B - ml of silver nitrate for blank

35.45 - Atomic weight of chlorine

APPENDIX VII

ETIMATION OF SULPHATE

TURBIDIMETRIC METHOD

Principle

Sulphate ions are precipitated as barium sulphate crystals of uniform size in acid medium. Light absorbed by the precipitate is measured using a spectrophotometer.

Reagents

1. Conditioning reagent: Dissolved 75 g sodium chloride in 30 ml is distilled water and added 30 ml conc., HCl or 100 ml of 95% alcohol. Added 50 ml glycerol and mixed well.

2. Barium chloride: Crystals were taken

3. Standard sulphate solution: Dissolved 147.9 mg anhydrous sodium sulphate distilled water and made upto 1000 ml. i.e., 1.0 ml = 100 $\mu g.SO_4^{2-}$.

Procedure

Standard

1. In 250 ml conical flasks 5,10,15,20,25,30,40 ml of sulphate solution was taken and diluted to 100 ml.

2. Added 5 ml conditioning reagent and mixed well using magnetic stirrer. 0.5g of barium chloride crystals were added and stirred and poured some of the solution to adsorption cell of the photometer and the optical density was measured using a spectrophotometer at a wavelength of 420 m.

3. Blank determination was carried on the reagents used.

4. Calibration graph was prepared by relating optical density against μg of sulphate.

Sample: Sulphate quantity of sample was taken in 250 ml conical flask and diluted to 100 ml and proceeded as done for standards and from the calibration graph, the mg of sulphate equivalent to the optical density was measured.

Calculation

Sulphate as SO_4^{2} = mg/l = $MgSO_4$ x 100 / ml sample taken for estimation.

APPENDIX VIII

ESTIMATION OF NITROGEN

KJELDHAL METHOD

Principle

The method involves distilling the sample with alkaline potassium permanganate solution and determining the NH_3 liberated which serves as an index of the Nitrogen status.

Procedure

30 ml of sample was taken in a dry kjeldahl flask; added 20 ml of distilled water followed by the addition of 100 ml of 0.32% potassium permanganate solution (16 mg/lit) and 100 ml of 2.5% Sodium hydroxide solution. Distilled the contents at a steady rate and collected the liberated ammonia in a conical flask containing 20ml of 2% boric acid. Collected 100 ml of the distillate in 30 minutes. Titrated with N/50 sulphuric acid (0.6 ml in 1 litre of water) with double indicator. (Methyl red and Bromo Cresol green dissolved in alcohol). A blank was run without the sample.

Calculation

$$\text{Nitrogen 1} = R \times 0.00028 \times \frac{1000}{20} \times \frac{1000000}{1000}$$

$$= R \times 14 \text{ Kg / ac.}$$

$$R = \text{Titre value}$$

APPENDIX - IX

ESTIMATION OF PHOSPHATE

COLORIMETRIC METHOD

Principle

Phosphate is estimated colorimetrically by adding ammonium molybdate and reducing the Molybdenum Phosphate complex in acidic medium. The intensity of blue colour on reduction provides a measure for the concentration of phosphate in the best solution.

Preparation Of Solutions

12 mg of ammonium molybdate (AR) was dissolved in 250 ml of distilled water. 0.291 mg of antimony potassium tartrate (AR) was dissolved in 100 ml of distilled water. Both these solutions in 100 ml added to 1000 ml of 5N sulphuric acid and thoroughly mixed; made upto 2 litres with distilled water thus forming reagent A. Reagent B was prepared by dissolving 1.056 gm of ascorbic acid (AR) in 200 ml of reagent A.

Procedure

To 5 g of sample, 50 ml of reagent B was added, followed by the addition of activated carbon. The contents were shaken for 30 minutes and filtered through Whatman No.1 filter paper. The filterate was collected discarding the turbid solution.

Colour Development

5 mg of the filtrate was acidified with 5N sulphuric acid, diluted upto 20 ml in a 25 ml; volumetric flask; 4 ml of reagent B was added and made upto the mark. After 10 minutes measured the intensity of the blue colour in a photoelectric colorimeter using 730-840 nm filter (red filter).

Standard Curve For Phosphate

4.393 mg of AR potassium dihydrogen ortho phosphate was dissolved in 1 litre distilled water which gives 1000 ppm phosphate solution. From that prepared solutions for 0.05, 0.1, 0.15,0.2 upto 0.5 ppm dilution. Added 5 ml Olsen extractant, developed colour and read the colour in the colorimeter. Plotted the curve taking the colorimetric reading on the vertical axis and amount of phosphorous in the horizontal area.

Calculation

$$\text{Phosphate} = R \times \frac{50}{5} \times \frac{25}{5} \times \frac{10^6}{10^6}$$

$$= R \times 50 \text{ kg lac.}$$

APPENDIX X

ESTIMATION OF POTASSIUM

FLAME PHOTOMETRIC MERTHOD

Principle

When sample is treated with neutral ammonium acetate, the ammonium ion replaces potassium ion of the sample and bring into the solution. The estimation of potassium is carried out with the help of flame photometer.

Preparation Of Solutions

Ammonium acetate - 77 mg was dissolved in 900 ml of distilled water and adjusted the pH to 7 with ammonia or acetic acid and made upto 1 litre with distilled water.

Procedure

To 5 gm of sample added 25 ml of extractant shaken for 5 minutes and filtered through Whatman No.1 filter paper. Measured the amount of potassium in the filtrate in the Flame photometer.

Standard Curve For Potassium

1.9074 gm of potassium chloride (AR) was dissolved in 1 litre which gave 1000 ppm potassium stock solution. Dilutions were done to get 10,20,30... 100 ppms. The reading was taken in the flame photometer. Plotted the curve taking the flame photometer readings in the vertical axis and the amount of potassium in the horizontal axis.

Calculation

$$\text{Potassium} = \frac{\text{Rx Vol.of the extractant} \times 10^6 \text{ Kg/ac}}{\text{Wt of sample taken} \times 10^6}$$

R = ppm of potassium in the extractant