

**WASTE WATER MANAGEMENT IN NIGERIAN
INDUSTRIES: CASE STUDY OF NIGERIAN BREWERIES
PLC.**

BY

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FACULTY OF ENGINEERING
UNIVERSITY OF BENIN**

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**A PROJECT WORK SUBMITTED TO THE DEPARTMENT
OF CHEMICAL ENGINEERING IN PARTIAL
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AWARD OF BACHELOR OF ENGINEERING [B.ENG]
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CERTIFICATION

This is to certify that this project was carried out by **OKE PEACE**
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DEDICATION

I wholeheartedly dedicate this work to the Lord God Almighty who has kept me alive and immensely lavished His grace and bountiful blessings upon me, whom through his love, undeserved kindness, favour and strength, I am able to run this degree programme to a successful completion and came out with this project report.

To Him be all the glory, forever and ever, Amen.

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ABSTRACT

Brewers spent grain (BSG) is a by-product of the brewing industry that contributes to a large volume of wastes. The lignocellulosic nature of this waste, together with presence of functional components such as antioxidants, represents an attractive for the composite industry. In this work the use of the brewers spent grain was used to produce cellulose acetate membrane which was used for the treatment method of the brewery effluent (waste water).

The results obtained for the BOD are 16.65mg/l(untreated), then first to fifth treatment as 13.7mg/l, 11.16mg/l, 8,37mg/l, 5.58mg/l and 3.14mg/l respectively. It was observed to have a R value of 0.999 which was an indication of viability of treatment process as also ascertained with the results from the COD, TSS, TDS and PH test analysis as shown in this project work.

CHAPTER ONE

1.1 Background of Study

“Beer is the fifth most consumed beverage in the world apart from tea, carbonates, milk and coffee with an estimated annual world production exceeding 134 billion litres in 2002 (Fillaudeau et al., 2006). In the manufacturing of beer, various residues and by products are generated. The most common ones are spent grains, spent hops and surplus yeast which are generated from the main raw materials (Mussatto, 2009).

Recent advances in biotechnology ensures that brewer’s spent grain (BSG) is no longer regarded as a waste but rather a feedstock for producing several products. Based on this, it is an undeniable fact that BSG has its own potential for sustainable reuse through biotechnological processes. Thus, efficient recycling of BSG requires extensive Research and Development (R&D) work towards exploring newer applications and maximizing use of existing technologies for a sustainable and environmentally sound management. Finally, more insight is required for large scale utilization, which involves both laboratory and field experiments with proper control processes.

The first of the eight Nigerian Breweries which is the newly reconstructed multi-billion naria Lagos Nigerian Breweries PLC at No 1 Abebe Village Road, Iganmu Surulere local Government Area of Lagos State Nigeria. It occupies an

area of 123,290metre square which cover a total area of approximately 70hectares. The capacity at start up was about 20,000hl, which currently at 2012 to date capacity is 350 million liters. Nigerian breweries PLC and Heineken international jointly own the brew. It is a beverage company designed for the production of four (4) brands of beer (star, gulder, legend and heineken) and also three (3) brands of malt (Amstel malt, maltina and malta gold and maltex). They also produce three brands of soft drinks (climax, fayrouz and strongbow). The multi-billion naira Lagos Nigerian Brewery PLC manifest itself in plants/equipment, organization and work methods which include management that now takes us to the waste management aspect of the Lagos Nigerian Breweries.”

“The Lagos Nigerian breweries designed for the production of beverages deals with the problems of industrial management and industrial waste of which are

(1) solid waste such as spent grain, broken bottles, rupted can, food waste.

(2) waste water such as waste brewing water.

Spent grain: This is the solid particles (Raffinate) that is been filtered out from the mash filter which is sent to the spent grain silo for disposal or sent to the spent grain silo for spent grain boiler by the diverter to produce heat energy needed in the vessels for brewing operations. It takes about 2 hours to filter

680hl of mash which is the total volume of a brew and contains about 300kg of spent grain.

Broken bottles/rupted can: these are some destroyed containers that cannot be used for packaging as recommended by standard.

Food waste: These are waste materials from the canteen and from food waste derived from the various offices.

Waste brewing water: This is the waste water that are been recovered after the processing of the beer which must be treated or managed properly to allow for the right standard for disposal.

1.2 Problem Statement

Urban waste crises in Nigeria is attributed mainly to three factors, namely, rapid increase in urban population, heavy consumption pattern of urban dwellers and inefficiency of the authorities whose statutory responsibilities include efficient refuse management in cities.

As in the case of industrialization, there are so much reason to agglomerate lots of waste but the sustainability of the environment and economic development depends on the frame work of the management scheme.

It is on this premise that the motivation to carry out a study on the topic under review heightened with a view of finding out other new and effective means of waste management in Lagos Nigerian Breweries PLC”.

1.3 Aims & Objectives

The aim of the study is to review the industrial wastewater management framework in Nigeria using Lagos Nigerian breweries as a case study.

Objectives

1. To Produce cellulose acetate membrane from spent grain to be used for treatment of the brewery effluent.
2. To investigate the effectiveness of cellulose acetate membrane in the treatment of the effluent.
3. To determine the level of BOD, COD, TDS, TSS and PH reduction during the treatment process.

1.4 Significance of the Study

This project work is on the “general overview of wastewater management of industries in Nigeria, a case study of Lagos Nigerian Breweries PLC.

When the study is successfully carried out, it will fill the embarrassing lacuna created by the dearth of studies in the area of waste management in Nigerian industries.

It intends to bring about behavioural change of the workers and management of the breweries towards an improved attitude and approach to industrial waste management system.

It is finally hoped that the findings will be very useful to the Federal, State and Local Government in the formulation of policies and implementation programmes relating to waste disposal management.

1.5 Scope and Limitations of Study

It will not only be difficult but unrealistic to conduct a project work on all the Nigerian industries. For these obvious reasons, the scope of the study is then limited to waste management in Nigerian Breweries PLC, Iganmu Lagos.

The constraints ranges from lack of full co-operation in the supply of information by some staff of the Brewery for fear of reprisal.

Although there have been studies on Industrial waste management, there was scarcity of data/literature on the topic under review. The study to a large extent like every other human academic quest is faced with financial constraints.

Finally, there was no ample time for the project work because of the demand of other academic works.

CHAPTER TWO

2.0 LITERATURE REVIEW

“Human activities such as agricultural, industrial and domestic wastes discharge their waste containing pollutants. Brewing as an industrial activity is an intensive water consuming activity, besides utilizing a wide variety of chemicals. Expectedly, large volumes of effluent are discharged into water courses of brewery bearing communities, leaving in its wake a polluted water body and eco systems. Increasing concentration of these organic/non organic enriched brewery effluents in the water constitute a severe health hazard to both plants and animals, thus impeding the functionality of the ecosystem. The situation is typical of the brewery effluents receiving aquatic system in Nigeria, where much of water resources cannot be utilized without a form of treatment, following effluent discharges with negligible consideration for environmental control (Menkiti and Onukwuli, 2011).”

“The beer brewing process generates large amount of waste water effluent and solid waste that must be disposed off, or treated in the least costly way to meet strict discharge regulations set by government entities.

Brewing waste water typically has a high biochemical oxygen demand (BOD) from all the organic components (sugars, soluble starch, ethanol, volatile fatty acids, etc). Brewery wastewater usually has temperatures ranging from 25°C to

38°C. The pH levels can range between 2 and 12 and are influenced by the amount and type of chemicals used in cleaning and sanitation (e.g. caustic soda, phosphoric acid, nitric acid, etc). Nitrogen and phosphorus levels are mainly dependent on the raw material and the amount of yeast present in the effluent”.

2.1 BREWERY WASTE MANAGEMENT

2.1.1 Brewery Solid Wastes

“The most cost-effective method for significantly reducing effluent load of brewery waste water is to separate the solid wastes from the waste water itself. The equipment necessary includes holding vessels, tanker trucks that can haul away the material, bumps, and dedicated piping or hoses for transfer. Typical solid wastes include spent grains, trub, spent yeast, diatomaceous earth slurry from filtration, and packaging materials.

Trub

Trub is slurry consisting of wort, hop particles and unstable colloidal proteins coagulated during the wort boiling.

Spent yeast

In brewing, surplus yeast is recovered by natural sedimentation at the end of the fermentation and conditioning . Only part of the yeast can be reused as new production yeast. Spent yeast is very high in protein and B vitamins, and may be given to livestock as a feeding supplement.

Diatomaceous Earth (DE) Slurry

Diatomaceous earth slurry from the filtration of beer also constitutes a very large category high in SS and BOD/COD. Different methods for regeneration are under development but presently they are not capable of totally replacing new diatomaceous earth.

Packaging Materials

Other solids waste include label pulp from the washing of returnable bottles, broken glass, cardboard, bottle caps, and wood that usually disposed of at sanitary landfills.

Spent Grains

Beer production results in a variety of residues, such as spent grains, which have a commercial value and can be sold as by products for livestock feed. The nutritional value of spent grain is much less than that of the same amount of dried barley, but the moisture makes it easily digestible by livestock. The spent grain as defined above are obtained through a process known as WORT separation.

After mashing, when the starch been broken down, the next step is to separate the liquid extract (the wort) from the residual undissolved solid materials (Spent Grains) found in the mash. Wort separation is important because the solids contain large amounts of protein, poorly modified starch, fatty material, silicates

and polyphenols (tannins). Brewer's spent grain (BSG) is the major by-product of the brewing industry, representing about 85% of the total by-product generated. BSG is a lignocellulosic material containing about 17% cellulose, 28% non-cellulosic polysaccharides, chiefly arabinoxylans and 28% lignin. BSG is available in large quantity throughout the year, but its main application has been limited to animal feeding”.

2.1.2 Brewery liquid waste [Wastewater]

“Wastewater is one of the most significant waste products of brewery operations. Even though substantial technology improvements have been made in the past, it has been estimated that approximately 3 to 10 liters of waste effluent is generated per liter of beer produced in breweries. The quantity of brewery wastewater will depend on the production and the specific water usage. (Kanagachandran, and Jayaratne 2006).

2.1.3 Characteristics of Brewery wastewater

Wastewater is mostly water by weight. Other materials make up only a small portion of wastewater, but can be present in large enough quantities that may require some pretreatment before discharging the wastewater into the sewage system. Some of these materials found in brewery wastewater are;

Water Quality- wastewater quality may be defined by its physical and chemical characteristics.

Physical Characteristics

Physical parameters include colour, odour, temperature, and turbidity. Insoluble contents such as total solids (TS), Oil and grease also fall into this category.

Chemical Characteristics

Chemical parameters associated with the organic content of wastewater include biochemical oxygen demand (BOD), chemical oxygen demand (COD), total organic carbon (TOC) and total oxygen demand (TOD).

2.1.4 Treatment of brewery waste water

Wastewater from a brewery may be discharged into either a waterway (rivers, streams or lakes), directly into a municipal sewer system, or into the municipal sewer system after the waste water has undergone some treatment.

2.1.4.1 Physical Treatment

Physical treatment is for removing coarse solids and other large materials, rather than dissolved pollutants. It may be a passive process, such as sedimentation to allow suspended pollutants to settle out or float to the top naturally. Some other processes like Flow Equalization, Screening, Grit removal, and Gravity sedimentation are also involved.

2.1.4.2 Chemical Treatment

Among the chemical treatment methods, pH adjustment and flocculation are some of the most commonly used at breweries in removing toxic materials and colloidal impurities.

2.1.4.3 Biological Treatment

After the brewery wastewater has undergone physical and chemical treatments, the wastewater can then undergo an additional biological treatment. Biological treatment of wastewater can be either aerobic (with air/oxygen supply) or anaerobic (without oxygen), which are discussed in more detail in the following sections. Generally, aerobic treatment has been applied for the treatment of brewery wastewater and recently anaerobic systems have become an attractive option.

Traditional process used in wastewater treatment: advantages and disadvantages (Volesky, 2001).”

PROCESS	ADVANTAGES	DISADVANTAGES
Precipitation and filtration	- Simple and low cost	- For high concentrations - Separation is difficult - Not very effective - Produces sludge
Biological oxidation and reduction	- Low cost	- When biological systems are used, the conversion rate is slow and susceptible to adverse weather conditions.
Chemical oxidation and reduction	- Mineralization enables metal recovery	- Requires chemicals applied to high concentrations. - Its very expensive.
Reverse osmosis	- Pure effluent (for recycling).	- Requires high pressures - Very expensive.
Ion exchange	- Very effective - Enables metal recovery.	- Responsive to the presence of particles, resins of high cost.
Adsorption	- Conventional sorbents (coal).	- Not effective for some metals.
Evaporation	- Pure effluent obtained.	- Requires an energy source - Expensive - Produces sludge.

For these reasons, alternative technologies that are practical, efficient and cost effective for low concentrations are being investigated.

2.1.4.4 Physiochemical Treatment

“Industrial waste water from certain industries don’t meet the standards for discharge and certainly not for reuse as process water. First of all the toxic components have to be removed from the waste water flow. Physiochemical waste water treatment is a frequently used technique in the area of waste water treatment. Physiochemical waste water treatment techniques are applied for the removal of heavy metals, oils and greases, suspended matter and emulgating organic substance, organic and inorganic components, toxic pollutants or high salt concentration, phosphorus. The physiochemical waste water treatment techniques are used as pretreatment, final treatment as well as specific treatment for waste water reuse as process water.

The physiochemical treatment techniques include flocculation, coagulation, flotation, neutralization, electroflotation, membrane technology (membrane filtration) NH₃ stripper-absorbers) etc. The treatment method selected depends in the first place on the size of the particles present in the wastewater. Solids visible to the naked eye can be removed by filtration or can be settled by sedimentation, or they can be separated by means of flotation techniques, this is in function of the density of the solids. The major problem however comes from fine suspended solids (particle size < 1µm). these are very stable and they all

have an electrostatic laden surface (usually negative), which means that they repel each other and will not clot together nor settle. This kind of fine suspended solids can only be removed by means of physiochemical techniques. For this purpose, chemicals are added (coagulants or flocculants) to accomplish the clotting or coagulation of the suspended solids, allowing to finally settle. A review of the above treatment method shall be discussed briefly.

Flotation:

Used for the separation of suspended solids, oils, greases and undissolved particle. This technique can be used as a preliminary treatment method for further purification, but at the same time the waste water treatment can be limited to the flotation technique only. The most used flotation technique is the DAF (Dissolve Air Flotation), by which compressed air is dissolved in the waste water to be treated. The released air forms tiny bubbles, adhering to the suspended matter (flakes) in the water (to which coagulants and/or flocculants have been added previously). This tiny bubbles and suspended solid flakes can now float to the surface, because of their now reduced density (by the dissolved air). The float layer formed that way is skimmed and collected in a container installed next to the DAF unit. The sludge can be further thickened or dewatered.

Neutralization:

Too acid or too alkaline waste water flows are neutralized until a pH value of approximately 7 is reached. The main reason to neutralize waste water is to prepare it for further treatment in a biological waste water treatment station or to meet the standards for discharge (e.g. Too acid waste water can damage the sewer system and waste water treatment plant). The most important objective of this technology include the following:-

- Apart from neutralization, the pH of the wastewater can be altered for several reasons e.g. a better control of the speed of chemical reactions, breaking down emulsions, conditioning, stabilization of raw wastewater, etc.
- Neutralization techniques can be used for a wide range of waste waters coming from almost any kind of industrial activities. This kind of installation uncomplicated can be fully automated and are thus maintenance friendly.

2.1.4.5 Membrane Technology

Membrane processes have become more attractive for potable water production in recent years due to the increased stringency of drinking water regulations. Membrane processes have excellent separation capabilities and show promise for meeting many of the existing and anticipated drinking water standards. The surface water treatment rule (SWTR) and the anticipated ground water disinfection rule have led to the investigation of ultrafiltration UF and microfiltration MF for turbidity and microbial removal. According to a 1997 report by the national research council, most expert foresee that membrane filtration will be used with greater frequency in small systems as the complexities of conventional treatment processes for small system increases.

Membrane filtration techniques are used for very specific applications in the field of industrial waste water treatment. Membrane filtration is often applied as final step in the treatment of waste water, usually within the scope of water

recuperation or reuse. However, this technology is also used for the treatment of very specific waste water and process water flows, such as for the treatment of oil emulsions and other specific emulsions. Based on the separative capacity of the membranes, there are mainly four (4) different steps to be distinguished in the field of membrane filtration. They include micro-filtration (MF), ultra-filtration (UF), nano-filtration (NF) and reverse osmosis (RO).

Micro-filtration:

Separation of approximately 0.1 to 1 μ m size particles (big proteins, yeast cells, microorganisms etc.). The principle of micro filtration is physical separation. The extent to which particles are separated depends on the membrane pore size. Particles bigger than the membrane pore size will be completely separated, particles smaller than the membrane pore size will only be partly removed depending on the dirt layer on the membrane. Conventional pressures are usually between 0.1 and 3 bar. Possible applications in the field of waste water treatment are e.g. Microorganism separation from waste water, separation of oil/water emulsions, pretreatment of waste and process water prior to nanofiltration or reverse osmosis.

Ultra-filtration:

It involves a pressure driven separation of materials from water using a membrane pore size of approximately 0.002 to 0.1 μ m and an operating pressure of approximately 3 to 5 bar. UF is designed to remove suspended and

dissolved macromolecular solids from fluids. The commercially available modules are therefore designed to accept feedwaters that carry high loads of solids. UF will remove all microbiological species removed by MF (partial removal of bacteria), as well as some viruses (but not an absolute barrier to viruses) and humic materials.

UF will remove all microbiological species removed by MF (partial removal of bacteria), as well as some viruses (but not an absolute barrier to viruses) and humic materials. Disinfection can provide a second barrier to contamination and is therefore recommended. The primary advantages of low-pressure UF membrane processes compared with conventional clarification and disinfection (post chlorination) processes are:

- No need for chemicals (coagulants, flocculants, disinfectants, pH adjustment);
- Size-exclusion filtration as opposed to media depth filtration.
- Good and constant quality of the treated water in terms of particle and microbial removal.
- Process and plant compactness; and
- Simple automation.

Nano-filtration:

NF membranes have a nominal pore size of approximately 0.001 microns. Pushing water through these smaller membrane pores requires a higher operating pressure than either MF or UF. Operating pressures are usually near 600 kPa (90 psi) and can be as high as 1,000 kPa (150 psi). These systems can remove virtually all cysts, bacteria, viruses, and humic materials. They provide excellent protection from DBP formation if the disinfectant residual is added after the membrane filtration step. Because NF membranes also remove alkalinity, the product water can be corrosive, and measures, such as blending raw water and product water or adding alkalinity, may be needed to reduce corrosivity. NF also removes hardness from water, which accounts for NF membranes sometimes being called “softening membranes.” Hard water treated by NF will need pretreatment to avoid precipitation of hardness ions on the membrane. More energy is required for NF than MF or UF, which has hindered its advancement as a treatment alternative.

Process

NF membranes have been observed to operate on the principle of diffusion rather than sieving as with MF and UF membranes.

Operation and Maintenance

Operational parameters of membranes include the physical and chemical properties of the membrane, the pore size or molecular weight cut-off (MWCO), and configuration.

Reverse osmosis (RO)

RO systems are compact, simple to operate, and require minimal labor, making them suitable for small systems. They are also suitable for systems where there is a high degree of seasonal fluctuation in water demand. RO can effectively remove nearly all inorganic contaminants from water. RO can also effectively remove radium, natural organic substances, pesticides, cysts, bacteria, and viruses. RO is particularly effective when used in series. Water passing through multiple units can achieve near zero effluent contaminant concentrations. Disinfection is also recommended to ensure the safety of water.

Some of the advantages of RO are:

- Removes nearly all contaminant ions and most dissolved non-ions,
- Relatively insensitive to flow and total dissolved solids (TDS) level, and thus suitable for small systems with a high degree of seasonal fluctuation in water demand,
- RO operates immediately, without any minimum break-in period,
- Low effluent concentration possible,
- Bacteria and particles are also removed, and

- Operational simplicity and automation allow for less operator attention and make RO suitable for small system applications.
- Membranes are prone to fouling, and Produces the most wastewater at between 25–50 percent of the feed.

Process

RO removes contaminants from water using a semipermeable membrane that permits only water, and not dissolved ions (such as sodium and chloride), to pass through its pores. Contaminated is subject to a high pressure that forces pure water through the membrane, leaving contaminants behind in a brine solution.

Membranes are available with a variety of pore sizes and characteristics.

Equipment

Typical RO units include raw water pumps, pretreatment, membranes, disinfection, storage, and distribution elements. These units are able to process virtually any desired quantity or quality of water by configuring units sequentially to reprocess waste brine from the earlier stages of the process. The principal design considerations for reverse osmosis units are:

- Operating pressure,
- Membrane type and pore size,
- Pretreatment requirements, and
- Product conversion rate (the ratio of the influent recovered as waste brine water to the finished water).

For this research project the spent grain obtained from Nigerian Breweries Iganmulagos state, will be adopted for use as the membrane filter which closely mirrors the reverse osmotic process”.

2.2 CHARACTERISTICS OF BREWERS’ SPENT GRAIN

Chemical composition and physiochemical properties

“BSG basically consists of the husk-pericarp-seed coat layers that covered the original barley grain. Depending on the evenness of malting more or less starchy endosperm and walls of empty aleurone cells may also remain. The starch content will be negligible, and some residues of hops introduced during mashing will be present depending on the brewing regime used. Thus, the major components of BSG will be the walls of the husk-pericarp-seed coat, which are rich in cellulose and non-cellulosic polysaccharides and lignin, and may contain some protein and lipid. The husk also contain considerable amount of silica and much of polyphenolic components of the barley grain (Macleod, 1979). According to Kunze (1996), 25% of the minerals present in barley are present as silicate.

The chemical composition of BSG varies according to barley variety, harvest time, malting and mashing conditions, and the quality and type of adjunct added in the brewery process (Huige, 1994; Santos et al., 2003); but in general, BSG is considered as a lignocellulosic material rich in protein and fibre, which accounts for around 20 and 70% of its composition, respectively. The main component of these fibrous tissues are arabinoxylan, lignin (a polyphenolic micro molecule)

and cellulose (a linear homopolymer of glucose units). Protein and fibre are highly concentrated in spent grain because most of the barley starch are removed during mashing (Kissel and Prentice, 1979)".

2.3 CELLULOSE ACETATE MEMBRANE

"Cellulose acetate ($C_6H_7O_2(OH)_3$), basically a chain of glucose molecules, is a heavily useful industrial compound that is used in many important products used worldwide everyday. It is an acetate ester also known as cellon or rhodoid or zyl or zylonite.

Cellulose acetate is one of the most important esters of cellulose. Depending on the way it has been processed cellulose acetate can be used for great varies of application (e.g for films, membranes or fibers).

Modern polymer chemistry is highly proficient at modifying polymers to specifically aim in terms of mechanical or thermal stability and chemical compatibility (Arthanareeswaran, Thanikaivelan, Srinivasn, Mohan, and Rajendran, 2004. Oluranti Sadiku Agboola and Emmanuel Rotimi Sadiku, 2014)

Cellulose acetate and its derivatives are suitable as membrane materials because of the advantages such as moderate flux, high salt rejection properties, cost effectiveness, relatively easy manufacturing, renewable resource of raw material, and non-toxicity. It has been widely used for reverse osmosis (Perera et al, 2014), microfiltration (Perera et al, 2014) and gas separation (moghadassi, Rajabi, Hosseini, and mohammadi in press).

However, Cellulose acetate is not suitable for more aggressive cleaning, has low oxidation and chemical resistances and poor mechanical strength. Hence the modification of Cellulose acetate gains importance (Alagumalai Nagendran and Doraiswamy Raju Mohan 2008). The performance of Cellulose acetate may be improved by mixing it with appropriate additives to fulfill new requirements and associated membrane properties (Sivakumar et al, 2000)”

Membrane filtration or separation is the most efficient way in separation and concentration treatments. The process is used to separate substances in fluid mixtures using the cellulose acetate as the permeable surface also known as membrane which is characterised to have different pore size that allows the lesser pore size fluid to pass through and retains the large pore size substances.

2.4 CELLULOSE ACETATE MEMBRANE PRODUCED FROM SPENT GRAIN

“Brewers spent grain (BSG) is the main by-product of the brewing industry. It represents almost the 85% of total wastes. To give an idea of the waste volume generation approximately 20kg BSG are generated for the production of 100litre beer end product. BSG is considered as lignocellulosic material since cellulose, hemicellulose (mainly arabinoxylose) and lignin represent almost 70wt% protein and then the other main component with a 20wt%.

The main uses of BSG is an ingredient for animal food, mainly for cattle. The, due to the high content on protein and fiber. It is also used for energy production. Another minor uses have been investigated during the last decades

biorefinery (POLYMER COMPOSITION., 38:40-47, 2017. 2015 Society of plastics Engineers)

However brewers spent grain (BSG) are upgraded by using it as a functional filler in polypropylene matrix to provide new environmentally friendly composite materials with balanced mechanical and thermal behaviour and improved antioxidant properties.(2015 Society of plastics Engineers)”

2.5 CITATION OF RELATED WORKS

“Lignocellulosic by-product derived from biofuel process were analyzed as functional fillers in polypropylene-based biocomposites. Corn cob, a byproduct yielded from corn harvesting for ethanol production, and sunflower hull, a byproduct of seed stripping for biodiesel production, can both be analysed as functional filler agents.(Micheal A. Fuqua, Venkata S. Chevali, Chad A. Ulven , 2012).

In another study the valorization of wastes from *Posidonia oceanic* (PO) has been carried out in order to obtain a fully biobased composite material in combination with a biobased polyethylene obtained from sugar cane as matrix. (B. Ferrero, V. Fombuena, O. Fenollar, T. Boronat, R. Balart 2014).

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 MATERIALS

“The materials and methods used in this project work will be divided into two sections. This sections includes the materials and method for the:

- The production of cellulose acetate membrane.
- The treatment of waste water (using micro-filtration process)

3.1.1 The production of cellulose acetate membrane.

Materials and Reagents

Spent grain obtained from the breweries, Hexane, Acetic anhydride, NaOH

Equipments

Beakers, Round bottom flask, Flat bottom flask, 250ml measuring cylinder, PH meter / Litmus paper, Air drying oven

3.1.2 The treatment of waste water (using micro-filtration process)

Materials and Reagents

Waste water [brewery effluent], Cellulose acetic membrane[produced from brewery spent grain], Filter paper , Cotton wool, Distilled water, Manganous Sulphate Solution, Alkaline Iodine Sodium Azide Solution, Sodium Thiosulphate Stock Solution, Standard Potassium Dichromate Reagent-Digestion Solution, Sulphuric Acid Reagent – Catalyst Solution, Standard Ferrous Ammonium Sulphate Solution, Starch Indicator, Sulphuric Acid, Ferroin Indicator.

Equipment

300ml glass stopper BOD bottle, 2ml syringe, Burrete / Retort Stand, Conical Flask, COD vials with stopper, COD Digester

3.2 METHODS

3.2.1 To prepare the cellulose acetate membrane;

1. Drying of spent grain and Grinding of spent grain to an acceptable particle size

Figure 3.1 dried spent grain



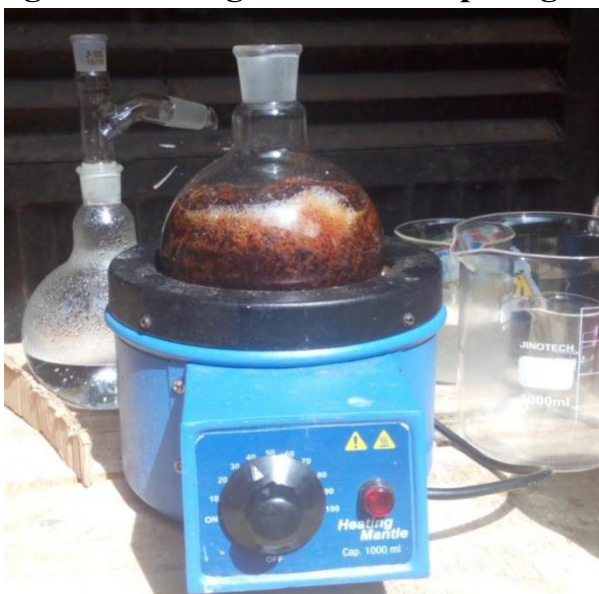
2. Dewaxing – This is the removal of the left over oil content in the spent grain using the soxhlet apparatus set up which comprises of boiling flask, extraction chamber, reflux condenser and heating mantle of cap 1000ml at 40c. Solution of n-hexane in the flat bottom boiling flask which with the help of the heat at 40c from the heating mantle evaporates into the spent grain that has been tied in a sack placed into the extraction chamber is dewaxed and then the vapour is cooled by the reflux condenser and refluxed back in the n-hexane solution leaving the spent grain dewaxed. The oil content can be recovered using the same soxhlet apparatus set up process. The spent grain after being dewaxed is loosened up from the sack into a tray dried at atmospheric temperature then put in the Electro-heater air dry oven to dry with set point at 105c.

Figure 3.2 dewaxing of spent grain



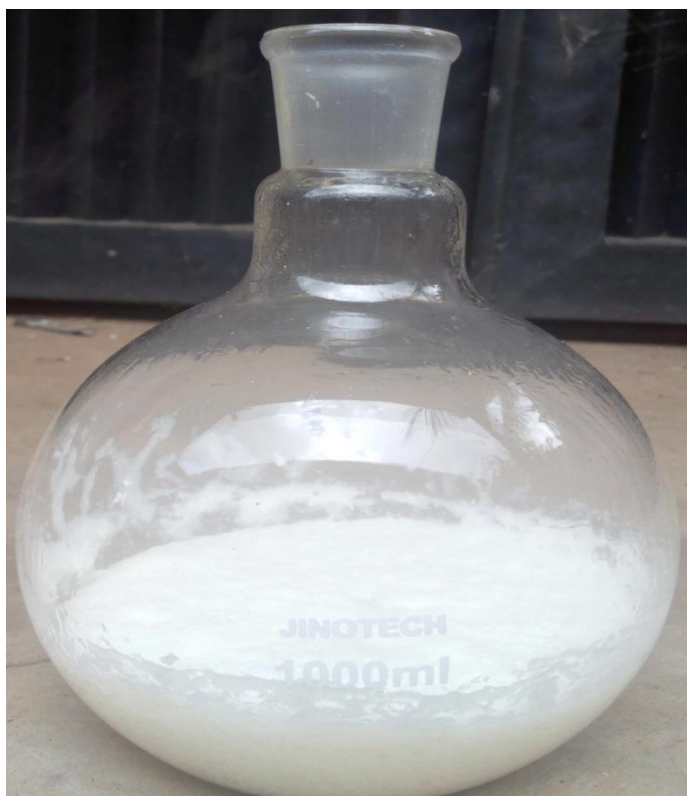
3. Delignification – This is the process of the removal of lignin that is present in the dewaxed spent grain. 20% conc NaOH solution was used.

Figure 3.3 Delignification of spent grain



4. Bleaching –This is a process of whitening the cellulose to give a clear membrane. It is achieved by heating the cellulose with H₂O₂. This whitening or complete bleaching is obtained at about 6mins emitting H₂O₂ gas. It is then allowed to cool.

Figure 3.4 Bleaching of spent grain



5. Acetylation – This process was achieved by using 2.84g of bleached pulp dispersed in 49.7ml of acetic acid stirred at 55c for one hour. Then mixture of 0.57ml of concentrated H₂SO₄ and 14.2ml of acetic anhydride was gradually added to the acetic acid pulp mixture keeping temperature at 60c. 35ml of methyl chloride was also added to help dispersing of the pulp. The resulting mixture kept in water bath at a temperature of about 80c for one hour with occasional stirring until a clear solution is obtained which confirms complete acetylation that comprises of triacetate and

diacetate precipitate. This precipitate is then washed thoroughly until it is neutral to litmus.

6. Drying – The precipitate is dried in an oven. After drying it is known as and can now be used as a CELLULOSE ACETIC MEMBRANE of a micro-filtration process used as a physio-chemical method of waste water treatment of brewery effluent.

Figure 3.5 Drying of spent grain with an air drying oven



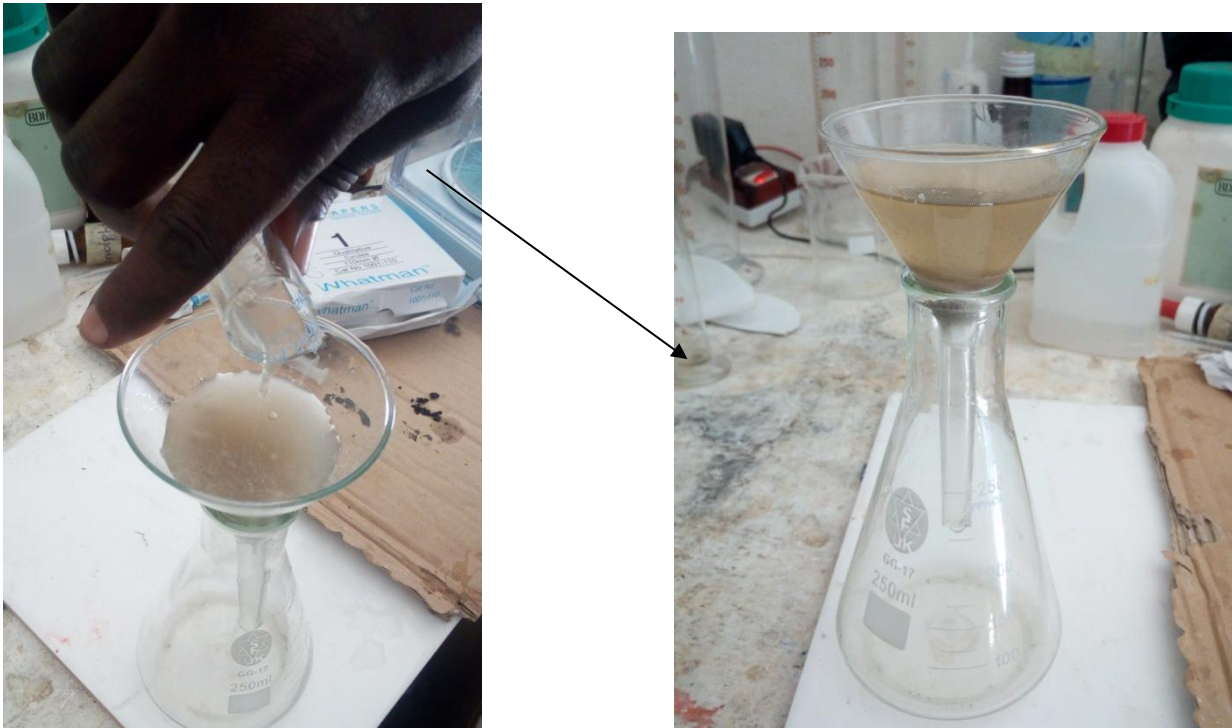
Figure 3.6 Dried Cellulose Acetate Membranes



3.2.2 To treat waste water [Using micro-filtration process]

The Waste Water treatment is a physio-chemical process. It is being treated using the micro-filtration process using a conical flask with a glass funnel on it that helps filter the waste water which on passing through the cellulose acetate membrane using a cotton wool as a membrane support where the solids or other non fluid substance are removed leaving the treated water into the conical flask as shown below:

Figure 3.7 Micro filtration process



This process is then being confirmed with the various test measures put to check the success level of treatment that is administered to the brewery effluent as it varies with repeated times of treatment. Some of the test administered are-;

- Biological Oxygen Demand [BOD and BOD5] test
- Chemical Oxygen Demand [COD] test
- Total Dissolved Solid [TDS] test
- Total Suspended Solid [TSS] test
- PH value test

Procedure for brewery effluent BOD test -;

- The BOD bottles were measured to ascertain its volume before being used for the test.

- The BOD bottles were filled with 10ml of sample together with making it up to the rim with distilled water to be tested and avoided any kind of bubbling or trapping of air bubbles.
- 2ml of manganese sulphate was added to the BOD bottle by inserting the 2ml syringe of manganese sulphate solution just below the surface of the liquid content of the BOD bottle.
- 2ml of alkali-iodide-azide reagent was added in the same manner. If the reagent is added above the sample surface, you will introduce oxygen into the sample
- On adding these two reagents, if there is presence of oxygen, a brownish-orange cloud of precipitate or floc will appear. It is allowed to settle for sufficient time in order to react completely with oxygen.
- 2ml of concentrated sulphuric acid was added with the aid of the 2ml syringe held just above the surface of the sample. Carefully place the stopper and invert several times to dissolve the floc.
- Fix the burette to the retort stand, Rinse and fill the burette with sodium thiosulphate.
- Measuring 203ml of the solution formed from the BOD bottle, it is transferred to a conical flask with titration started immediately after the transfer of the content to the conical flask using starch solution of 1-2 drops as indicator.
- The end point of titration was the first disappearance of the blue colour to colourless. The volume of sodium thiosulphate solution used up to activate the disappearance of the blue colour to colourless was noted.

These same procedures were all repeated for samples of BOD5 [five days BOD test] and other varied BOD samples as well as Blank samples which is used as a guide or standard to values obtained to be dissolved oxygen values.”

Procedure for brewery effluent COD test

- Two COD vials with stopper were used [one for the sample and one for the blank solution].
- 2ml of the sample is put in one of the COD vial and the other COD vial is for the blank to which 2ml distilled water was added.
- 7ml of potassium dichromate reagent digestion solution was added and 5ml of sulphuric acid reagent- catalyst solution to each of the two COD vials. COD vials are hot now.
- Tubes are capped tightly, then the COD vials were placed into a blocked COD digester at 150c and is set for 2hours. At set time 2hours the digester automatically switches off. The vials were removed and allowed to cool to room temperature.
- The burette was fixed to a retort stand then rinsed and filled the burette with the ferrous ammonium sulphate solution and adjusted the burette reading to zero for titration.
- Then transferred the contents of the blank sample to a conical flask, then added a few drop of ferroin indicator which turns the solution bluish green in colour.
- The titration of the bluish green solution with the ferrous ammonium sulphate solution was carried out.
- The end point of the titration is the appearance of reddish brown colour. The volume of ferrous ammonium sulphate solution added to the blank sample to activate the colour change was recorded.
- The same titration process was carried out on the other vial which contained the sample solution of which on putting the ferrous indicator which turns the solution green in colour but also on end point of titration it appears reddish brown colour. The volume of ferrous ammonium sulphate solution is also recorded for further calculation.

During these procedures some process conditions were adjusted to fit our environmental conditions and attain the expected results which were

- Increment and reduction in volumes of reagents and sample used to ascertain a desired result.
- Reduction in temperature and set time of COD digester for the test process.
- Then these same procedures were all repeated for samples of varied treatment time duration.

Procedure for brewery effluent TDS test

- 1ml of the waste water sample [brewery effluent] was collected into a beaker then weighed the sample.
- The content was heated to dryness the weighed sample in the beaker on the electronic heater and then weighed the beaker to get the Total dissolved solid percentage difference.

The procedure was repeated for the waste water sample [brewery effluent] as it undergoes a continuous batch wise process treatment. It was retreated for about four consecutive times which gave it a total of five runs.

Procedure for Brewery Effluent TSS Test

- 1ml of the waste water sample was measured into a beaker that was weighed and then used the mass obtained together with the known volume to calculate for the Density.
- The waste water sample was filtered with a filter paper in a funnel into a conical flask and then 1ml of the filtered sample was weighed and then the density calculated.
- The percentage differences of the two densities were calculated.

The procedure was repeated for the waste water sample [brewery effluent] as it undergoes continuous batch wise process treatment.

Procedure for Brewery Effluent PH Test

- Some volumes of the waste water sample [brewery effluent] were put in a beaker, then the litmus paper was inserted in the sample.
- Colour changes were observed after some seconds that was compared with the colour codes that are used as guide or standard to the PH value of sample solutions shown on the pack containing the litmus paper.

The procedure was repeated for the waste water sample [brewery effluent] as it undergoes continuous batch wise process treatment.

CHAPTER FOUR
RESULTS AND DISCUSSIONS

4.1 RESULTS

Table 4.1 Table of results obtained.

RUNS	COD	BOD(mg/l)	TSS (%)	TDS (%)	PH
1(Untreated)	262	16.65	4.86	59.41	8.0
2	85	13.70	4.04	45.92	8.0
3	70	11.16	3.06	30.69	8.0
4	20	8.37	2.02	22.77	7.5
5	20	5.58	1.01	10.10	7.5
6	20	3.14	1.01	5.05	7.5

Chemical oxygen demand (COD)

Biological oxygen demand (BOD)

Total suspended solid (TSS)

Total dissolved solid (TDS)

Hydrogen ion concentration (PH)

Table 4.2 Standard result Of FEPA and WHO.

Parameters	FEPA	WHO
COD	N/A	N/A
BOD	30	N/A
TSS	30	N/A
TDS	2000	1000
pH	6-7	N/A

Table 4.3 Standard range value for BOD

BOD VALUES	VIABILITY
1-3	Very Good
4-6	Good
7-9	Poor
10 and above	Very Poor

4.2 RESULT ANALYSIS

Table 4.4 COD ANALYSIS

Runs	COD
1	262
2	85
3	70

4	20
5	20
6	20

*Table 4.4 Result of COD Analysis
Reduction capacity vs runs*

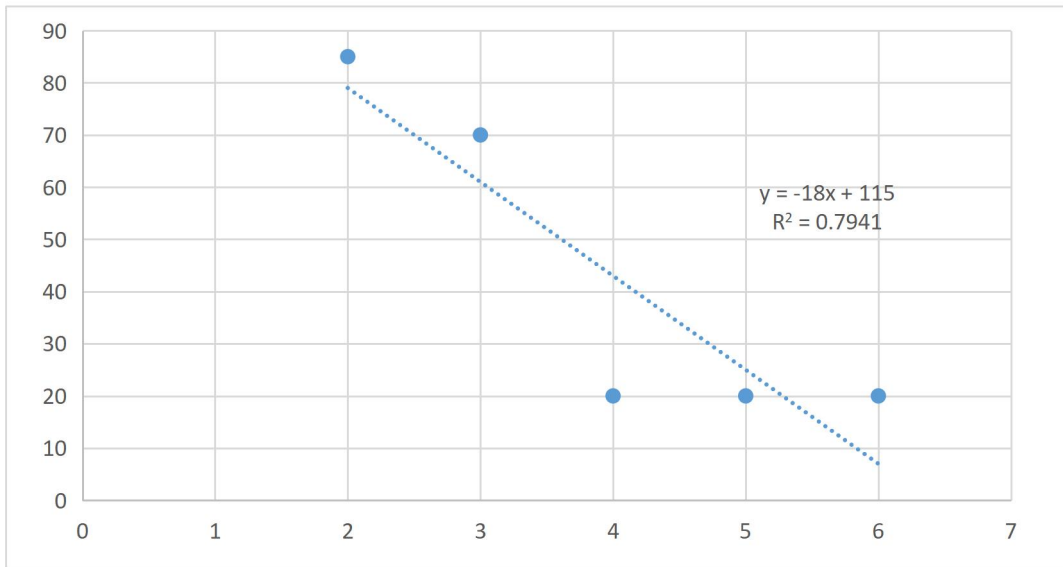


Fig 4.1: graph of COD reduction capacity vs runs

From the graph plotted, the equation of line was obtained as

$$Y = -18x + 115$$

Y = Reduction Capacity

X = Runs

It was observed from figure 4.1, that the reduction capacity (level) is inversely proportional to the runs (number of treatment) administered to the sample.

Basically it deduces that an increase in numbers of treatment, will lead to a corresponding decrease in the amount of chemical oxygen demand present.

Table 4.5 Result of BOD Analysis

Runs	BOD (mg/l)
1	16.65
2	13.7
3	11.16
4	8.37
5	5.58
6	3.14

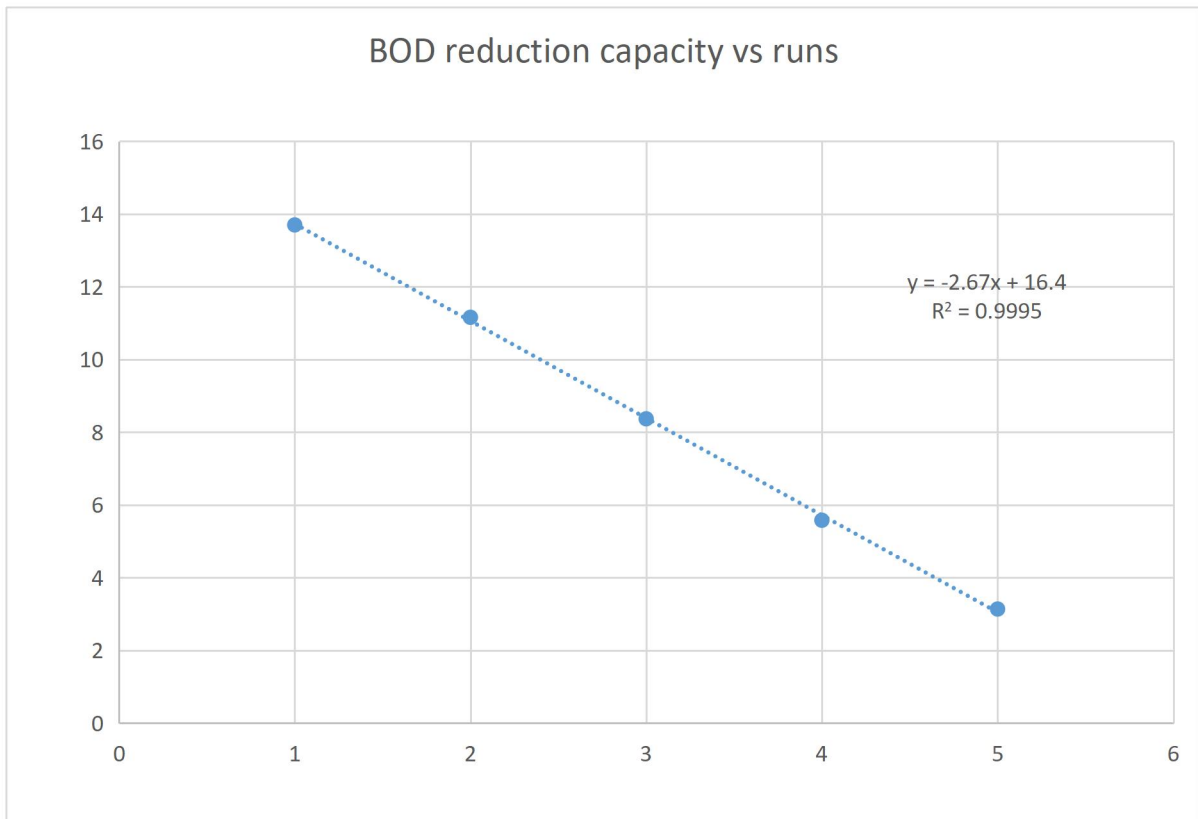


Fig 4.2: BOD reduction capacity vs runs

From the graph plotted, the equation of the line was gotten as

$$Y = -2.67x + 16.4$$

Y= Reduction Capacity

X= Runs

It was observed from figure 4.2, that the R^2 value obtained was 0.999 which explains that the continuous batch treatment process used lead to appreciable values of decreasing BOD which portray efficiency in treatment process. An increase in runs, will lead to decrease in the BOD values.

Table 4.6 TSS ANALYSIS

Runs	TSS	
	(mg/l)	(%)
1	0.05	4.86
2	0.04	4.04
3	0.03	3.06
4	0.02	2.02
5	0.01	1.01
6	0.01	1.01

TSS reduction capacity vs runs

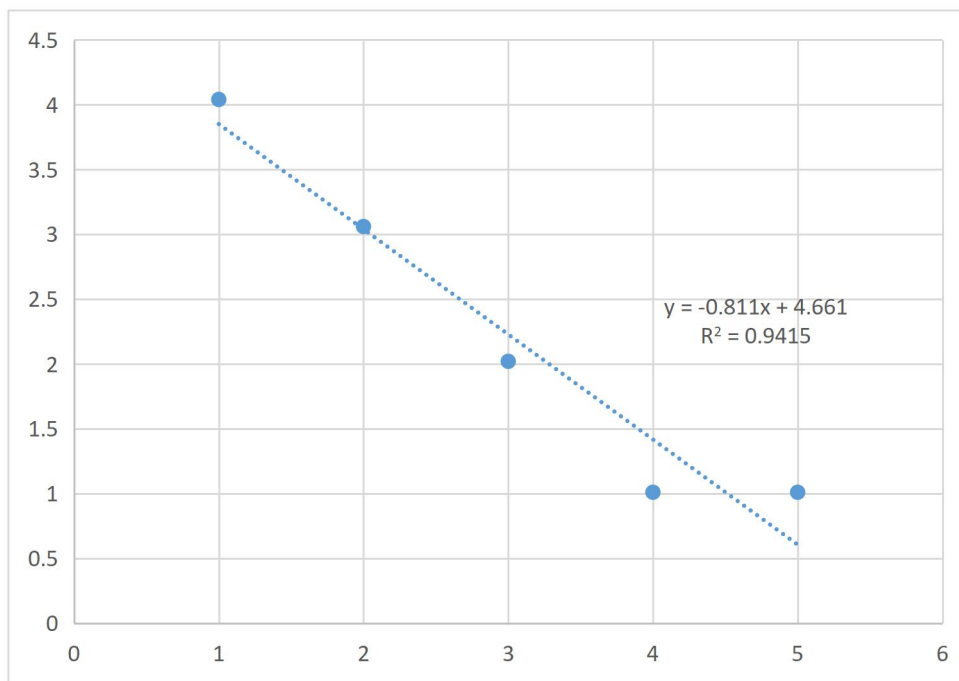


Fig 4.3: graph of TSS reduction capacity vs runs

From the graph plotted, the equation of the line was gotten as

$$Y = -0.811x + 4.661$$

Y=TSS reduction capacity

X=Runs

From figure 4.3, it was observed that the reduction capacity is inversely proportional to the runs that is an increase in the number of runs (treatment}, will lead to decrease in the amount of total suspended solid in the solution.

Table 4.7 TDS ANALYSIS

Runs	TDS	
	(mg/l)	(%)
1	0.41	59.41
2	0.53	45.92
3	0.70	30.69
4	0.78	22.77
5	0.89	10.10
6	0.94	5.05

TDS reduction capacity vs runs

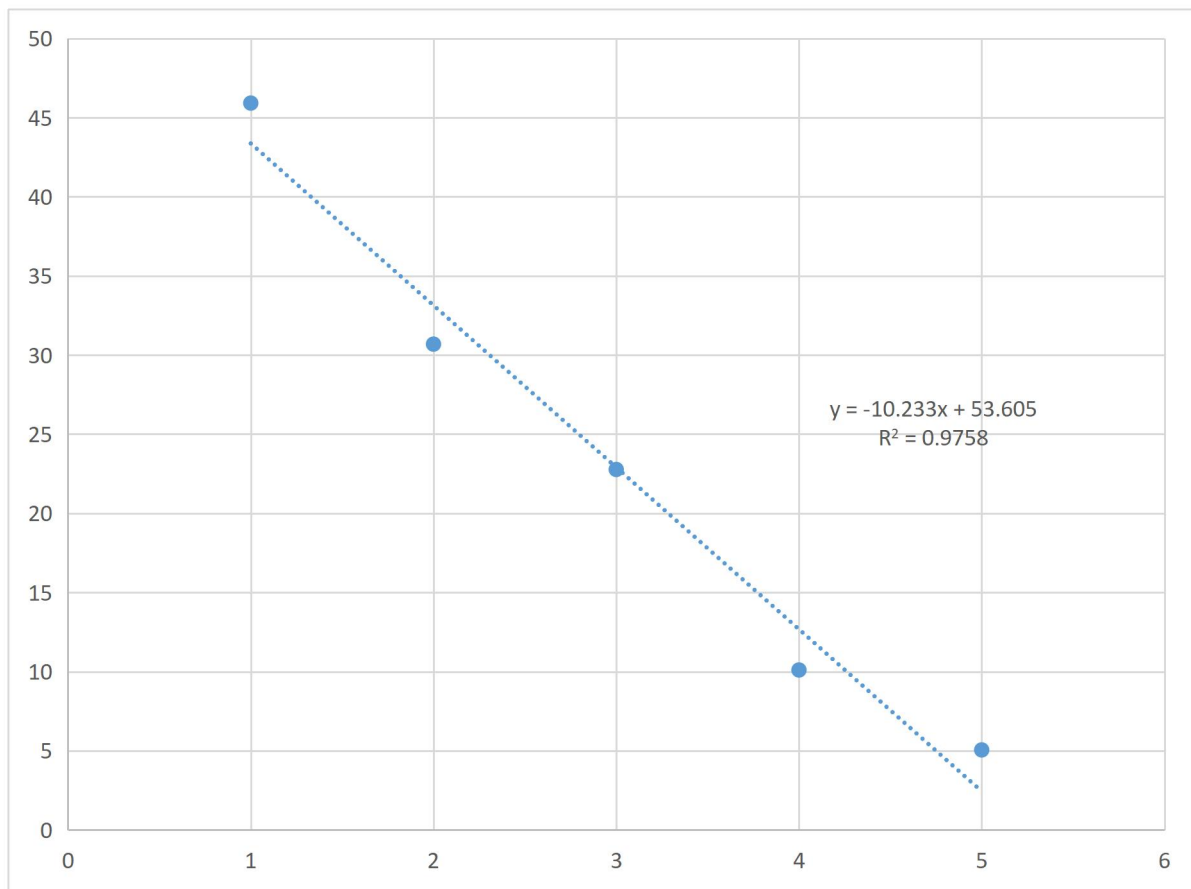


Fig 4.4: TDS reduction capacity vs runs

From the graph plotted, the equation of the line was gotten as

$$Y = -10.23x + 53.60$$

Y = TDS reduction capacity

X = Runs

From figure 4.4, it was observed that the reduction capacity is inversely proportional to the runs that is an increase in the number of runs (treatment), will lead to decrease in the amount of total dissolved solid in the solution

Table 4.8 PH ANALYSIS

RUNS	1	2	3	4	5	6
pH	8	8	8	7.5	7.5	7.5

4.3 DISCUSSION

It was observed from the analysis that reduction capacities were improved as there was continuous treatment of the brewery waste water.

The result of the COD were obtained as follows for each treatment starting from the first to the last, (fifth) treatment as 85, 70, 20, 20 and 20 respectively which shows from the result that there was appreciable decrease of the COD level at the first to the third from where it became constant and shows no visible change which could tell a point of optimisation of the COD treatment on the brewery waste water.

Other parameter of treatment that was considered such as the BOD there was a very visible reduction as each runs of treatment from first to last, (fifth) were administered obtaining values of 13.70mg/l, 11.16mg/l , 8.37mg/l , 5.58mg/l , 3.14mg/l respectively which from its R value on the graph with value of 0.999 giving a clear indication of a very good treatment method for this brewery waste water.

On the same trend with the BOD parameter, the TSS and TDS values obtained from first to last(fifth) treatment runs were 4.04% , 3.06% , 2.02% , 1.01% , 1.01% and 45.92% , 30.69% , 22.77% , 10.10% , 5.05% respectively showed an appreciable reduction trend, also the R values obtained were 0.941 and 0.975 respectively which were seen to be appreciable values that also indicated a good treatment method for the brewery waste water. The hydrogen ion concentration(PH) values obtained from first to last(fifth) values were 8 , 8 ,

8 , 7.5 , 7.5 , 7.5 respectively also showed that the treatment method used is a good treatment method for the treatment of brewery waste water.

The result from the BOD values as obtained are 16.65mg/l as the raw waste water sample collected from the Lagos Nigerian breweries plc and then in the orders of treatment(runs) were 13.70mg/l , 11.16mg/l , 8.37mg/l , 5.58mg/l and 3.14mg/l respectively shows a better and in depth form of parameter test method since it has to do with difference in BOD results as with BOD(five days) result to give a comprehensive analysis with good result and showed encouraging treatment method. Therefore more biochemical degradation had occurred which implies reduced biochemical oxygen demand of the waste water that encourages aquatic lives and environmental sustainability.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

Brewers spent grain (BSG) is a waste from the brewing industry that represents high volumes. Its lignocellulosic nature provides attracting properties for composite manufacturing with polymer matrices. It is not only inert filler but also acts as functional filler that provides antioxidant properties due to phenolic and free radical scavenging compounds. Its lignocellulosic properties gave rise to the production of the Cellulose Acetate Membrane used for the filtration process and treatment method of the brewery effluent (waste water). Therefore the aim of this study was achieved. The following conclusions were made;

- The brewery effluent(waste water) treatment method showed good results as indicated from the parametric test measures used such as the BOD , COD , TSS , TDS and PH .
- The Cellulose Acetate Membrane produced from the brewers spent grain was successfully used for the brewery effluent treatment giving an appreciable result of reducing values of parameters at every subsequent treatment.
- From the result analyzed the increase of number of treatments (runs) , the more the reduction capacity of the brewery effluent (waste water) .
- The result from the BOD values as obtained are 16.65mg/l as the raw waste water sample collected from the Lagos Nigerian breweries plc and

then in the orders of treatment(runs) were 13.70mg/l , 11.16mg/l , 8.37mg/l , 5.58mg/l and 3.14mg/l respectively shows a better and in depth form of parameter test method since it has to do with difference in BOD results as with BOD(five days) result to give a comprehensive analysis and encouraging result. Therefore more biochemical degradation had occurred obtaining good reduction capacity values.

5.2 RECOMMENDATION

- The use of Cellulose Acetate Membrane in the treatment of waste water should be used in other industrial waste water.
- It is cost effective and an eco-friendly technology that is economically more advantageous than other treatment alternatives and as such, various industrial plants should be encouraged to employ this method.
- More research facilities should be implemented and built by the government and private individuals so as to carry out more research on not just Cellulose Acetate Membrane from spent grain from but other Lignocellulosic natured material.
- The use of components of Cellulose Acetate Membrane for the treatment of waste water should be further investigated as it is a viable and environmental friendly method of waste water treatment.

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APPENDICES

CALCULATIONS

(i) Determination of the Chemical Oxygen Demand in the given water sample is tabulated below:-

Table 4.1 COD Test values

SI No	Volume of Blank (ml)	Volume of Sample (ml)	Volume of 0.1N FAS Blank (ml)	Volume of 0.1N FAS Sample (ml)
1	5	5	14.20	1.10
2	5	5	4.50	0.25
3	1	1	2.70	2.00
4	1	1	3.90	3.70
5	1	1	4.70	4.50
6	1	1	4.30	4.10

From the test result above on the table I calculated the COD values using this formular below :

$$\text{COD} = \frac{(A-B) \times N \times 1000}{V_s}$$

Where :

A = Volume of Ferrous Ammonium Sulphate for Blank

B = Volume of Ferrous Ammonium Sulphate for Sample

N = Normality of Ferrous Ammonium Sulphate

V_s = Volume of sample used

Therefore the values of the COD treatment were obtained starting with the second run to help for easy identification of the change observed from the first run as indicated untreated and then recorded as shown in the table below:

RUNS	COD
1(Untreated)	262
2	85
3	70
4	20
5	20
6	20

(ii) Determination of the Biochemical oxygen Demand

Table 4.2 (a) BOD Test values

SI No	Volume of Blank (ml)	Volume of Sample (ml)	Volume of Titrant Blank (ml) $\text{Na}_2\text{S}_2\text{O}_3$	Volume of Titrant Sample (ml) $\text{Na}_2\text{S}_2\text{O}_3$	Dissolved Oxygen Blank (mg/l)	Dissolved Oxygen Sample (mg/l)
1	327	333	0.40	1.10		
2	327	274	0.60	1.10		
3	328	279	0.60	0.80		
4	327	279	0.50	0.60		
5	274	279	1.70	1.90		
6	274	285	1.50	1.60		

Table 4.2: (b) BOD (5days) Test values

SI No	Volume of Blank (ml)	Volume of Sample (ml)	Volume of Titrant Blank (ml) Na ₂ S ₂ O ₃	Volume of Titrant Sample (ml) Na ₂ S ₂ O ₃	Dissolved Oxygen Blank (mg/l)	Dissolved Oxygen Sample (mg/l)
1	328	324	0.20	0.40		
2	285	314	0.10	0.10		
3	328	274	0.30	0.10		
4	327	319	0.30	0.10		
5	327	314	0.10	0.10		
6	327	331	0.10	0.10		

(iii)Table 4.4Calculation of Total Suspended Solids (TSS)

SI No	Volume of Sample (ml)	Volume of Filtrate (ml)	Mass of sample (g)	Mass of Filtrate (g)	Density of Sample (g/ml)	Density of Filtrate (g/ml)	TSS (percentage difference in density) (%)
1	1	1	1.03	0.98	1.03	0.98	4.85
2	1	1	0.99	0.95	0.99	0.95	4.04
3	1	1	0.98	0.95	0.98	0.95	3.06
4	1	1	0.99	0.97	0.99	0.97	2.02
5	1	1	0.99	0.98	0.99	0.98	1.01
6	1	1	0.99	0.98	0.99	0.98	1.01

(iv) Calculation of Total Dissolved Solids (TDS)

Table 4.5: TDS test values

SI No	Volume of Sample (ml)	Mass of Sample (g)	Mass of Sample (after dryness) (g)	TDS (Percentage dissolved solids) (%)
1	1	1.01	0.6	59.41
2	1	0.98	0.45	45.92
3	1	1.01	0.31	30.69
4	1	1.01	0.23	22.77
5	1	0.99	0.1	10.10
6	1	0.99	0.05	5.05

Table 4.6: pH test values

SI No	1	2	3	4	5	6
pH	8.0	8.0	8.0	7.5	8.0	8.0