PRODUCTION OF ADSORBENT FROM IRISH POTATO PEEL FOR THE

REMOVAL OF HEAVY METALS FROM EFFLUENTS

By

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i

DECLARATION

I declare that the work in the project report entitled PRODUCTION OF ADSORBENT FROM IRISH POTATO PEEL FOR THE REMOVAL OF HEAVY METALS FROM EFFLUENTS has been performed by me under the supervision of Dr. D.O. Agbajelola. No part of this project report was presented for any degree or diploma elsewhere at any institution to the best of my knowledge.

Mbaogu Hillary Chika

Name of Student

Signature

Date

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ABSTRACT

In this research work, Irish potato peel was used to produce adsorbent for the removal of heavy metals from industrial effluent. The Irish potato peel was washed, dried and crushed; the potato peel was then activated before it was carbonized at different carbonizing temperatures. Waste water was obtained and digested. The residual metallic ion concentrations were determined using an Atomic Absorption Spectrophotometer (AAS). The results obtained after contacting for 2 hours with industrial effluent, showed that the developed adsorbent achieved the aim of this work i.e. removal of heavy metal ions (Zinc and Iron). The adsorbent adsorbed more Iron ions than Zinc ions. The possibility of developing adsorbent from Irish potato peel to absorb metal ions as shown from the results is an opportunity of converting this material to valuable product.

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TABLE OF CONTENT

| | | Pag | e |
|--|----------|-----|---|
| Title Page | | i | |
| Declaration | | ii | |
| Certification | | iii | |
| Acknowledgement | | iv | |
| Abstract | • | v | |
| Table of Content | | vi | |
| List of Tables | | ix | |
| List of Appendices | | X | |
| Chapter One | | | |
| 1.0 Introduction | | 1 | |
| 1.1 Aims and Objectives | | 2 | |
| 1.2 Scope of Work | | 3 | |
| 1.3 Justification | | 3 | |
| Chapter Two | | | |
| 2.0 Literature Review | | 4 | |
| 2.1 Heavy Metals | | | |
| 2.1.1 Environmental And Health Effects Of Heav | v Metola | 4 | |
| 2.1.1 Effect of Iron on the Environment | y metals | 5 | |
| 2.1.1.2 Effect of Zinc on the Environment | | 5 | |
| 2.1.1.2 Effect of Zinc off the Environment | | 6 | |
| | | | |
| 2.2.1 Adsorption Mechanism | | 8 | |
| 2.2.2 Adsorption Isotherms | | 8 | |
| 2.3 Adsorbents | | 10 | |
| 2.3.1 Activated Carbon | | 10 | |
| 2.3.2 Carbon Nanotubes | | 11 | |
| 2.3.3 Bioadsorbent | | 11 | |
| 2.3.4 Characteristics and Requirement of Adsorbe | nts | 12 | |

vi

| 2.3.5 Activating Bioadsrbents | | | 12 |
|---|-----|-------|----|
| 2.4 About the Adsorbent | | | 13 |
| 2.5 Chemically Modified Plant/Fruit Waste | | | 14 |
| Chapter Three | | | |
| 3.0 Methodology | | | 15 |
| 3.1 Apparatus | | | 15 |
| 3.2 Methodology | | | 16 |
| 3.2.1 Sample Collection | | • | 16 |
| 3.2.1.1 Irish Potato Peel | | | 16 |
| 3.2.1.2 Waste Water | | | 17 |
| 3.2.1.3 Digestion of Waste Water | | | 17 |
| 3.2.1.4 Batch Experiment | | | 17 |
| 3.2.2 Characterization and Proximate Analysis | | | |
| Of Sample | | | 18 |
| 3.2.2.1 Ash Content Determination | | | 18 |
| 3.2.2.2 Pore Volume Determination | | • • • | 18 |
| 3.2.2.3 Determination of Volatile Content | | | 18 |
| 3.2.2.4 Determination of Moisture Content | | | 18 |
| Chapter Four | | | |
| 4.0 Results And Discussion | | | 20 |
| 4.1 Results | • • | | 20 |
| 4.2 Discussion of Results | | | 24 |
| Chapter Five | • | | |
| 5.0 Conclusion And Recommendation | | | 27 |
| 5.1 Conclusion | | | 27 |
| 5.2 Recommendations | | | 27 |
| References | | | 29 |
| Appendix | | | 32 |
| | | | |

i

LIST OF TABLES

Tables

| Table 3.1 List of Apparatus Used | 15 |
|--|----|
| Table 3.2 List of Materials Used | 16 |
| Table 4.1 Proximate Analysis of Irish Potato Peel | 20 |
| Table 4.2 Composition and Type of Adsorbent Produced | 20 |
| Table 4.3 Carbon Yield and Volatile Content of Adsorbent | 21 |
| Table 4.4 Ash Content of Adsorbent Produced | 21 |
| Table 4.5 Moisture Content of Adsorbent Produced | 22 |
| Table 4.6 Pore Volume of Adsorbent Produced | 22 |
| Table 4.7 Determination of the Adsorption Capacity of | |
| Samples (Absorbance) | 23 |
| Table 4.8 Determination of the Adsorption Capacity of | • |
| Samples (Mg/100 ml and Mg/L) | 23 |

page

24

Table 4.9 Effect of Particle Size on the Adsorption

of Heavy Metals

LIST OF APPENDICES

| Appendix | p | age | |
|--|-----|------|---|
| Appendix 1.0 Calculation of Proximate Analysis | | 32 | |
| Appendix 2.0 Calculation of Carbon Yield | | 32 | |
| Appendix 3.0 Calculation of Volatile Content | • | 33 | |
| Appendix 4.0 Calculation of Ash Content | | 33 | |
| Appendix 5.0 Calculation of Pore Volume | | 34 | |
| Appendix 6.0 Calculation of Moisture Content | | 34 | |
| Appendix 7.0 Calculation of the Concentration of Zinc in Mg/100 ml | | 35 | |
| Appendix 8.0 Calculation of the Concentration of Zinc in Mg/L | | 35 | |
| Appendix 9.0 Calculation of the Concentration of Iron in Mg/100 ml | Sec | . 36 | ý |
| Appendix 10.0 Calculation of the Concentration of Iron in Mg/L | | 37 | |

Chapter One

1.0 INTRODUCTION

Pollution is the addition of materials or energy to the environment that damages it, making it undesirable or unfit for life. In recent times mans' need for industrialization and civilization has helped make life easier but not without its disadvantages. Generation of wastes (including waste water) that pollute the environment is top on the list of these disadvantages.

Wastewater varies so greatly in both flow and pollution strength because of its differing sources. So, it is impossible to assign fixed values to its constituents. In general, industrial wastewater may contain suspended, colloidal and dissolved (mineral and organic) solids. In addition, they may be either excessively acidic or alkaline and may contain high or low concentrations of colored matter. These wastes may contain inert, organic or toxic materials and possibly pathogenic bacteria.

In the last decades, the scientific community has become increasingly concerned about the potential public health impact of new environmental contaminants originating from industrial, agricultural and human activities. Industrialization in many regions has increased the discharge of industrial wastes (effluents), especially those containing heavy metals into natural water bodies or on land (Vilar et al., 2005).

According to (Ademoroti, 1996) metals with densities greater than 5 g/cm³ are referred to as heavy metals. The presence of these heavy metals in the environment is of important concern due to their toxicity and health effects on the human and living creatures (Itodo, 1979). Many industries such as oil refining, textile industry, metal plating facilities, electroplating, mining operations, pigments and dyes manufacturing, Fertilizer, and batteries produce heavy metals such as nickel, cobalt, zinc, chromium, copper, lead and cadmium, etc (Kiran et al., 2005).

Techniques such as chemical precipitation, evaporation, electro-deposition, ion exchange and membrane separation have been used to remove and recover metal ions from wastewater. However, these technologies are either ineffective or expensive when heavy metals are present in the wastewater at low concentrations (Popuri et al., 2009). Research interest into the production of adsorbent to replace costly conventional methods of wastewater treatment is attracting attention of scientists (El-sheriff et al., 2009).

1

Adsorption is the adhesion of atoms, ions, and molecules of a gas, liquid and dissolved solids to a surface. This process creates a film of the adsorbate (the molecules or atoms being accumulated) on the surface of the adsorbent. It differs from absorption, in which a fluid permeates or is dissolved by a liquid or solid. Adsorption techniques for wastewater treatment have become more popular in recent years owing to their efficiency in the removal of pollutants. Adsorption can produce high quality products while also being a process that is economically feasible (Allen, 2005). Generally adsorption is classified as physisorption (characteristic of the weak van der Waals forces) or chemisorption (characteristic of covalent bonding) and most recently biosorption.

Biosorption is an innovative technology that employs inactive dead biomass for the recovery of heavy metals from wastewater. These biomasses are generally wastes from households and industries that are in abundance and environmental friendly due to their biodegradability. The big advantages of biosorption are the low operating cost, minimization of the volume of chemical and / or biological sludge to be disposed of, and high efficiency in detoxifying very dilute effluents (Gabr et al., 2008). Several recent studies have utilized some inexpensive and local materials for the purpose of heavy metal removal such as rice husk, wheat bran, wheat husk, sawdust of various plants, bark of trees, groundnut shells, coconut shells, maize/corn cobs, sugarcane bagasse, apple and banana peels, etc., this research work will focus on the use of Irish potato peels for the same purpose.

1.1 Aim and Objectives

The aim of this work is to produce an adsorbent from Irish potato peel suitable for the removal of heavy metals from industrial effluents. This will be achieved through the following objectives:

- Preparation of the potato peel by washing, drying and crushing.
- Addition of activating agent (0.1M HCl) to the potato peel.
- Undertaking the carbonization of the activated potato peel.
- Performance of the analysis of the waste water for the presence of heavy metals.
- Checking the adsorption capacity of the produced adsorbent by mixing it with the waste water.

• Analyzing factors that could have affected the adsorption capacity of the adsorbent.

1.2 Scope of Work

The scope of this work is limited to the development and use of potato peel as an adsorbent for the removal of heavy metals from wastewater.

1.3 Justification

Heavy metals despite been useful in industries is now a threat to life as it impacts negatively on life. These are visible in contamination of water supply and water bodies posing danger to aquatic wildlife, diseases and epidemics as little amount of these metals are needed in the body and loss in soil fertility due its non-degradability. Nigeria, as a developing nation and even the world at large must look for a cost effective means handling this problem, especially waste water treatment. Bio-adsorbent production (potato peel in this case) is a cost effective approach to this issue. In the reduction of waste, this research work is relevant by converting waste into valuable product.

3

Chapter Two

2.0 LITERATURE REVIEW

Clean and fresh water is becoming a limited resource because of water pollution (Sarojini, 2003). In a bid to achieve industrialization, most developing nations have placed priority on matters relating to economic development, social and educational development, while issues relating to environmental safety and protection have been relegated to the background.

However, a few of these countries that are grouped among the less developed nations of the world have made landmark achievements towards their economic development via industrialization. This has given birth to a number of industries and factories, increased income as a result of exports and increased use of automobiles with inevitable problems of urbanization and population explosion (Ademoroti, 1996).

The search for better living standards has greatly increased the rural-urban migration. This has resulted in the clustering of large population of people in relatively small areas under poor sanitary conditions and heavy traffic movement (Olade, 1976). A typical example is Lagos (Nigeria) and most recently Abuja, (Nigeria).

2.1 Heavy Metals

The term heavy metal refers to any metallic chemical element that has a relatively high density and is toxic or poisonous at low concentrations. Examples of heavy metals include mercury (Hg), copper (Cu), cadmium (Cd), arsenic (As), chromium (Cr), zinc (Zn), thallium (Tl), and lead (Pb).

A heavy metal is a member of a loosely-defined subset of elements that exhibit metallic properties. It mainly includes the transition metals, some metalloids, lanthanides, and actinides. Many different definitions have been proposed, some based on density, some on atomic number or atomic weight, and some on chemical properties or toxicity. An alternative term for heavy metals is *toxic metals* (Olade, 1976).

Heavy metals are non-biodegradable i.e. they cannot be broken down into simple harmless substances (Sanjiro, 2003). Although these metals are released in low concentrations into the

4

Heavy metals are non-biodegradable i.e. they cannot be broken down into simple harmless substances (Sanjiro, 2003). Although these metals are released in low concentrations into the environment, they tend to accumulate in the environment and in living organisms and eventually reach toxic levels. This is known as bioaccumulation (Lenntech, 2004). Compounds accumulate in living things any time they are taken up and stored faster than they are broken down (metabolized) or excreted.

Living organisms require varying amounts of heavy metals. These amounts are in very minute quantities, to maintain body metabolism. Iron, cobalt, copper, manganese, molybdenum, and zinc are required by humans. Excessive levels can be damaging to the health. Other heavy metals such as mercury, plutonium, and lead are toxic metals have no known vital or beneficial effect on organisms, and their accumulation over time in the bodies of animals can cause serious illness.

Heavy metals find their way into water bodies through emissions from waste incinerators and also effluents from factories, mining and smelting works, refineries and power plants.

2.1.1 Environmental and Health Effect of Heavy Metals.

To describe the environmental and health effect of heavy metals three metals namely; iron (Fe) and zinc (Zn) will be discussed.

2.1.1.2 Effect of Iron on the environment

Iron can be found in meat, whole meal products, potatoes and vegetables. The human body absorbs iron in animal products faster than iron in plant products. Iron is an essential part of hemoglobin; the red colouring agent of the blood that transports oxygen through our bodies.

Iron may cause conjunctivitis, choroiditis, and retinitis if it contacts and remains in the tissues. Chronic inhalation of excessive concentrations of iron oxide fumes or dusts may result in development of a benign pneumoconiosis, called siderosis, which is observable as an x-ray change. No physical impairment of lung function has been associated with siderosis. Inhalation of excessive concentrations of iron oxide may enhance the risk of lung cancer development in workers exposed to pulmonary carcinogens. When high concentrations of iron are absorbed, for example by haemochromatose patients, iron is stored in the pancreas, the liver, the spleen and the heart. This may damage these vital organs. Healthy people are generally not affected by iron overdose, which is also generally rare. It may occur when one drinks water with iron concentrations over 200 ppm.

Iron compounds may have a more serious effect upon health than the relatively harmless element itself. Water soluble binary iron compounds such as $FeCl_2$ and $FeSO_4$ may cause toxic effects upon concentrations exceeding 200 mg, and are lethal for adults upon doses of 10-50 g. A number of iron chelates may be toxic, and the nerve toxin iron penta carbonyl is known for its strong toxic mechanism. Iron dust may cause lung disease.

2.1.1.2 Effects of Zinc on the environment

Zinc is an essential element needed by the body and is commonly found in nutritional supplements. It is also one of the most common and hazardous element found in waste sites and exposure to it could be harmful. Zinc is one of the most common and abundant element in the Earth's crust and it is found in the air, soil and water. It is also present in all foods. (Lenntech, 2004)

Powdered zinc is explosive and may burst into flames if stored in damp places. Zinc has many uses in industry, among which are to coat steel as well as iron to prevent rust and corrosion. It can be mixed with other metals to form alloys; it can also be used to make dry cell batteries, paints, ceramics and other products such as rubber, cosmetics and drugs in pharmaceutical industries. (Lenntech, 2004)

Exposure to zinc could be through breathing (inhalation), eating or drinking substances containing zinc, or by skin contact. Factors affecting the level of harm due to contact with zinc include: the dose (how much), duration of contact and mode of contact. Zinc enters the air, water, and soil as a result of both natural process and human activities. Most zinc enters the environment as the result of mining, purifying of zinc, lead, and cadmium ores, steel production, coal burning and burning of wastes (Lenntech, 2004).

of zinc for several months may cause anemia, damage the pancrease and decrease levels of highdensity lipoprotein (HDL) cholesterol (Lenntech, 2004).

2.2 Adsorption

Adsorption is now recognized as an effective and economic method for heavy metal wastewater treatment. The adsorption process offers flexibility in design and operation and in many cases will produce high-quality treated effluent (Wang, 2011).

Adsorption is the adhesion of atoms, ions, and molecules of a gas, liquid and dissolved solids to a surface. This process creates a film of the adsorbate (the molecules or atoms being accumulated) on the surface of the adsorbent. It differs from absorption, in which a fluid permeates or is dissolved by a liquid or solid (Wang, 2011). It is a removal process where certain particles are bound to an adsorbent particle surface by either chemical or physical attraction. Adsorption is a process by which a gas, liquid, or solute (substance in solution) binds to the surface of a solid or liquid (called the adsorbent), forming a film of molecules or atoms (called the adsorbate). Adsorption is a consequence of attractive interactions between the surface of the adsorbent and the species being adsorbed. In the bulk of an adsorbent, all the bonding requirements (be they ionic, covalent, or metallic) of the constituent atoms of the material are fulfilled by other atoms in the material.

However, atoms on the surface of the adsorbent are not wholly surrounded by other adsorbent atoms and therefore can attract molecules of the adsorbate. The exact nature of the bonding depends on details of the species involved.

The adsorption process is generally classified as either physisorption (physical adsorption) or chemisorption (chemical adsorption). Weak interactions, characteristic of weak van der Waals forces, lead to physisorption; strong interactions, characteristic of covalent bonding, lead to chemisorption. In the former case, adsorbed molecules remain unbroken; in the latter case, the adsorbed molecules may or may not be broken up.

7

chemisorption. In the former case, adsorbed molecules remain unbroken; in the latter case, the adsorbed molecules may or may not be broken up.

2.2.1 Adsorption Mechanism

Sorption is transfer of ions from water to the soil i.e. from solution phase to the solid phase. Sorption actually describes a group of processes, which includes adsorption and precipitation reactions. Recently, adsorption has become one of the alternative treatment techniques for wastewater laden with heavy metals. Basically, adsorption is a mass transfer process by which a substance is transferred from the liquid phase to the surface of a solid, and becomes bound by physical and/or chemical interactions (Kurniawan et al., 2003). Various low-cost adsorbents, derived from agricultural waste, industrial by-product, natural material, or modified biopolymers, have been recently developed and applied for the removal of heavy metals from metalcontaminated wastewater. In general, there are three main steps involved in pollutant sorption onto solid sorbent: (i) the transport of the pollutant from the bulk solution to the sorbent surface; (ii) adsorption on the particle surface; and (iii) transport within the sorbent particle. Technical applicability and cost-effectiveness are the key factors that play major roles in the selection of the most suitable adsorbent to treat inorganic effluent.

The degree of adsorption is influenced by certain properties of the adsorbent and adsorbate, and conditions such as temperature and pressure. Generally, adsorption is enhanced at low temperatures and high pressures. In addition, it is favored by larger surface areas of the adsorbent and by greater attractive forces between the adsorbent's surface and the adsorbate.

2.2.2 Adsorption Isotherms

Adsorption may be described quantitatively by the use of isotherms. Isotherms are empirical relations which are used to predict how much solute can be adsorb by activated carbon (Perry, 1984). An isotherm shows the amount of adsorbate on the adsorbent as a function of its pressure (if gas) or concentration (if liquid) at constant temperature. It is basically imported to describe how solute (heavy metals), interact with adsorbent, and it is critical for optimizing the use of the adsorbent. The quantity of material adsorbed is nearly always normalized by the mass of the

a function of the partial pressure or concentration at a given temperature and the result expressed as an adsorption isotherm (Perry, 1984).

The five basic adsorption isotherms are: Freundlich isotherm, Langmuir isotherm, BET isotherm, the Kisliuk isotherm, and the Henderson-Kisliuk isotherm. However, the two isotherms used popularly in adsorption theory/process are the Langmuir isotherm and the Freundlich isotherm.

The Langmuir equation, which is valid for monolayer sorption on a surface containing a limited number of sites, predicting a homogeneous distribution of sorption energies, is expressed as

$$Cads = \frac{KlbCe}{(1+bCe)}$$
 2.0

Where,

Cads= amount of As (III) adsorbed ($\mu g. g^{-1}$)

Ce = equilibrium concentration of As (III) (μ g.L⁻¹)

K_I= Langmuir Constant (kg.kg⁻¹)

b = Langmuir Constant (m^3/kg)

The Freundlich isotherm is expressed as

$$Cads = KfCe 1/n 2.1$$

Where,

Cads = amount of As (III) adsorbed ($\mu g. g^{-1}$)

* **Ce = equilibrium** concentration of As (III) in solution (μ g. L⁻¹)

Kf = Freundlich Constant

1/n = Freundlich Constant

If 1/n < 1, bond energies increase with surface density,

If 1/n > 1, bond energies decrease with surface density and

If 1/n = 1 all surface sites are equivalent.

The Henderson Kisluick isotherm is used in a case where the adsorbate is strictly liquid and the adsorbent is solid. Although they are the most commonly applied isotherms in adsorption practices, and are used for a wide variety of adsorbates and adsorbents, they are not very suitable for adsorption in such cases (Kurniawan et al., 2003).

2.3 Adsorbents

Adsorbents are natural or synthetic materials of amorphous or microcrystalline structure used for adsorption. Adsorbents have been developed for a wide range of separations. Commercial materials are provided usually as pellets, granules, or beads, although powders are used occasionally. The adsorbent may be used once and discarded, or, as is more common, it is employed on a regenerative basis and used for many, many cycles. Adsorbents are generally used in cylindrical vessels through which the stream to be treated is passed. In the regenerative mode, two or more beds usually are employed with suitable valving to allow for continuous processing. Absorbents are used in applications requiring from a few ounces to over 2 million pounds in one plant. There are different types of adsorbents.

Agricultural wastes, industrial by-products and wastes and natural substances have been studied as adsorbents for the heavy metal wastewater treatment (Wang, 2011).

2.3.1. Activated Carbon Adsorbents

Activated carbon (AC) adsorbents are widely used in the removal of heavy metal contaminants. Its usefulness derives mainly from its large micropore and mesopore volumes and the resulting high surface area. A large number of researchers are studying the use of activated carbon for removing heavy metals. Nowadays, the depleted source of commercial coal-based activated carbon results in the increase of price. To make progress in heavy metals adsorption to activated carbon without the expense of decline in the pollutants adsorption, additives and composite activated carbon could be an option. Additives of alginate, tannic acid, magnesium, surfactants and activated carbon composite could be effective adsorbents for heavy metals. And searching and activated carbon composite could be effective adsorbents for heavy metals. And searching for activated carbon alternative from abundant and inexpensive sources is of concern. Converting carbonaceous materials into activated carbon for heavy metals remediation have been reported (www.scribd.com).

2.3.2. Carbon Nanotubes Adsorbents

Carbon nanotubes (CNTs) discovered in 1991, have been widely studied for their excellent properties and applications. As relatively new adsorbents, CNTs have been proven to possess great potential for removing heavy metal ions such as lead, cadmium, chromium), copper and nickel from wastewater. Carbon nanotube are classified into two (2); single-walled CNTs (SWCNTs) and (2) multi-walled CNTs (MWCNTs) (www.scribd.com)The mechanisms by which the metal ions are adsorbed onto CNTs are very complicated and appear attributable to electrostatic attraction, sorption-precipitation and chemical interaction between the metal ions and the surface functional groups of CNTs. The sorption capacities of metal ions by raw CNTs are very low but significantly increases after being ssoxidized by HNO₃, NaClO and KMnO₄ solutions.

2.3.3. Bioadsorbents

Biosorption of heavy metals from aqueous solutions is a relatively new process that has been confirmed a very promising process in the removal of heavy metal contaminants. The major advantages of biosorption are its high effectiveness in reducing the heavy metal ions and the use of inexpensive bioadsorbents. Bio-sorption processes are particularly suitable to treat dilute heavy metal wastewater. Typical biosorbents can be derived from three sources as follows:

(1) Non-living biomass such as bark, lignin, shrimp, krill, squid, crab shell, etc.;

(2) Algal biomass;

(3) Microbial biomass, e.g. bacteria, fungi and yeast.

2.3.4. Characteristics and requirements of adsorbents

Adsorbents are used usually in the form of spherical pellets, rods, moldings, or monoliths with hydrodynamic diameters between 0.5 and 10 millimeters (mm). For repetitive use at high temperatures, they need to have high abrasion resistance and high thermal stability. In addition, small pore diameters lead to higher exposed surface area and hence higher capacity for adsorption. Adsorbents must also have a distinct pore structure that enables rapid transport of vapors (New World Encyclopedia).

Activated carbon adsorbents are widely used in the removal of heavy metal contaminants. Its usefulness derives mainly from its large micropore and mesopore volumes and the resulting high surface area. A large number of researchers are studying the use of activated carbon for removing heavy metals (Wang, 2011). Activated Carbon uses the physical adsorption process whereby attractive Vander Waals forces pull the solute out of solution and onto its surface (Reynolds & Richards, 1996). Once the solute is bound to the carbon it is considered "removed" from the water.

Activated carbon adsorption proceeds through 3 basic steps

1. Substances adsorb to the exterior of the carbon granules

2. Substances move into the carbon pores

3. Substances adsorb to the interior walls of the carbon

Use of bio-adsorbent for waste water treatment solves two major problems i.e. the recycling of waste from food industry through processing and treatment of waste water.

2.3.6 Activating Bio-Adsorbents

The process of activated carbon generation in bio-adsorbents begins with the selection of a raw carbon source. These sources are selected based on design specifications since different raw sources will produce activated carbon with different properties. Some of the more common raw sources include wood, sawdust, lignite, peat, coal, coconut shells, and petroleum residues

(AWWA, 1971). Sufficient activation for useful applications may come solely from the high surface area, though further chemical treatment often enhances the absorbing properties of the material. (www.googlesearch.com)

Activated carbon bio-adsorbents can be produced by one of the following processes:

2.3.6.1 Physical reactivation

The precursor is developed into activated carbons using gases. This is generally done by using one or a combination of the following processes:

a. Carbonization: Material with carbon content is pyrolyzed at temperatures in the range 600–900 °C, in absence of air (usually in inert atmosphere with gases like argon or nitrogen)

b. Activation/Oxidation: Raw material or carbonized material is exposed to oxidizing atmospheres (carbon dioxide, oxygen, or steam) at temperatures above 250 °C, usually in the temperature range of 600–1200 °C.

2.3.6.2 Chemical activation

Prior to carbonization, the raw material is impregnated with certain chemicals. The chemical is typically an acid, strong base, or a salt (phosphoric acid, potassium hydroxide, sodium hydroxide, zinc chloride, respectively). Then, the raw material is carbonized at lower temperatures (450–900 °C). It is believed that the carbonization / activation step proceeds simultaneously with the chemical activation. Chemical activation is preferred over physical activation owing to the lower temperatures and shorter time needed for activating material. (www.googlesearch.com)

2.4 About the adsorbent (Irish potato peels)

The potato specifically the Irish potato is a starchy, tuberous crop from the perennial *Solanum tuberosum* of the Solanaceae family (also known as the nightshades). The word potato may refer to the plant itself as well as the edible tuber. Potatoes yield abundantly with little effort, and adapt readily to diverse climates as long as the climate is cool and moist enough for the plants to

gather sufficient water from the soil to form the starchy tubers. Potatoes do not keep very well in storage and are vulnerable to molds that feed on the stored tubers, quickly turning them rotten. Potato plants are herbaceous perennials that grow about 60 cm (24 in) high, depending on variety.

2.5 Chemically Modified Plant/Fruit Waste

Pretreatment of plant/fruit waste can extract soluble organic compounds and enhance chelating efficiency (Gaballah et al.,). These methods of pre-treatment use different kinds of modifying agents such as base solutions e.g. sodium hydroxide, calcium hydroxide, sodium carbonate, etc., mineral and organic acid solutions e.g. hydrochloric acid, nitiric acid, sulfuric acid, citric acid, thioglycollic acid, tartaric acid, etc., organic compounds like ethylenediamine, formaldehyde, epichlorohydrin, methanol. It also uses oxidizing agents such as hydrogen peroxides, dye (reactive orange 13). All these are used for the purpose of removing soluble organic compounds, eliminating coloration of aqueous solutions and also increasing efficiency of metal adsorption. (Low et al., 2006; Baral et al., 2006; Abia et al., 2006)

Chapter Three

3.0 METHODOLOGY

3.1 Apparatus

The lists of apparatus used in this experiment are tabulated below.

Table 3.1: List of Apparatus Used

| Equipment | Source | Comment |
|----------------------------------|----------------------|--------------------------|
| 1. Funnels | Waft lab | 10 cm top inside diamete |
| 2. Electronic weighing balance | Waft lab | SWB model pm 2000 |
| 3. Sieve | Waft lab | < 2 mm mesh size |
| 4. Mortar and pestle | Waft lab | wood |
| 5. Oven | Waft lab | Gallenkamp |
| 6. Electric Furnace | Waft lab | Gallenkamp |
| 7. Filter Paper | Waft lab | Watman |
| 8. Desiccator | Waft lab | glassware |
| 9. Porcelain crucible | Waft lab | ceramic |
| 10. Petri dish | Waft lab | glassware |
| 11. Set of beakers and flasks | Waft lab | assorted sizes |
| 12. Spatula | Waft lab | steel |
| 13. Measuring cylinder | Waft lab | glassware |
| 14. Pipette | Waft lab | glassware |
| 15. Beakers | Waft lab | glassware |
| 16. Conical flask | Waft lab | glassware |
| 17. Measuring cylinder | Waft lab | glassware |
| 18. Plastic bottles | Minna Central Market | |
| 19. Air-tight storage containers | Minna Central M | arket |

Table 3.2: List of Materials Used

| | Materials | Source | Comment |
|----------|-------------------------------|--------------|--------------|
| <u> </u> | 1. Potato peels | Bosso | Raw material |
| | 2. Hydrochloric acid, (0.1 M) | Chemical lab | 100 ml |
| | 3. Distilled water | Waft lab | 50 litres |
| | 4. Pachloric acid mixture | Waft lab | 60 ml |

3.2 Methodology

3.2.1 Sample Collection and Preparation

3.2.1.1 Potato peel Sample

All the potato peels or biomaterials were collected from a local fish and chips making spot located in Bosso Estate, Minna, Niger State. The potato peels were washed with water and then distilled water to remove the dirt and was sun dried for two (2) days. After moisture content was highly reduced, it was crushed with a mortar and pestle in order to reduce its size.

After the biomaterial was collected, air-dried and their size reduced, 100 grams each of the dried powdered biomaterial sample was measured in six (6) different places for preparation of six (6) different adsorbents; three (3) samples of chemically activated and three (3) non chemically activated adsorbents at varied carbonizing temperatures.

A standard solution of 0.1 M HCl was prepared by measuring a required volume of acid of known concentration and topping it up to the 100 ml mark with distilled water. The required volume of acid was calculated and noted. Then three (3) of the previously weighed samples of 100 g were impregnated with the standard solution of the prepared 0.1 M HCl and each stirred properly to ensure thorough mixing of the solution with the biomaterial samples. This is the activation process. The remaining three (3) weighed samples of 100 g were not chemically activated.

The activated biomaterials were then placed into porcelain crucibles and labeled according to the carbonizing temperatures, and put in an electric furnace. The furnace was set at a temperature of 500 °C and two of the samples (one chemically activated and the other not chemically activated)

were left in the furnace for one hour to carbonize. After one hour, the carbonized material was removed and placed in a dessicator for about one hour and allowed to cool. This process was repeated for carbonizing temperatures of 600 °C and 700 °C.

The activated carbon of the potato peel sample was then washed with distilled water to free the bio-adsorbent of excess dried acid and oven dried at 60 °C for four hours. The final bio-adsorbent material obtained was stored in plastic air-tight storage containers ready for use.

3.2.1.2 Waste water Sample

The wastewater was collected at the PPMC depot along Minna - Suleja road, Niger state. It was carefully bottled in a plastic container and taken to the laboratory for analysis.

3.2.1.3 Digestion of Wastewater Sample

About 10 ml of wastewater solution was measured into an 80 ml beaker and 10 ml of concentrated Pachloric acid was added. The mixture was then heated on the heating mantle until the solution turned almost colorless. Thereafter the beaker was rinsed with water, filtered and transferred quantitatively to 100 ml volumetric flask and topped up with distilled water to the. 100 ml mark. The reason for the dilution before analysis was to reduce organic matter interference and convert metal to a form that can be analyzed by AAS.

The waste water solution was tested for the presence of heavy metals/metal ions using an atomic adsorption spectrophotometer.

3.2.1.4 Batch Experiment

About 1 g of the bio-adsorbent sample was measured and put in a conical flask and 50 ml of wastewater solution was added. The mixture was allowed a contact time of two hours and then was filtered and the residue separated from the filtrate. The filtrate obtained was bottled in plastic containers, labeled and the metal ions in the filtrate were determined using the atomic adsorption, spectrophotometer (AAS). The procedure was repeated for the different adsorbents produced to determine the effect of temperature/chemical activation on the effectiveness of the adsorbent.

3.2.2 Characterization of developed adsorbent and proximate analysis of sample

3.2.2.1 Ash content determination

0.5 g of the adsorbent was weighed and put in a crucible. The crucible was put in an electric furnace set at 800 °C. The sample was left in the furnace until it ashen, then withdrawn from the furnace and placed in a dessicator to cool. The ash obtained was weighed and the ash content was determined by calculations.

Ash Content = $\frac{W \times 100\%}{2}$

3.1

3.2

3.2.2.2 Pore volume determination

0.5 g of the adsorbent was weighed and boiled in 20 ml of distilled water for 10 minutes. The sample was withdrawn after boiling and dried on a filter paper. The sample was reweighed and the difference in weight was determined and the pore volume calculated. The pore volume was

calculated using the formula:

Pore volume $(cm^3/g) = \Delta W/\rho_{water}$

Where,

 ΔW is the change in weight of the sample

 ρ_{water} is the density of water (1 g/cm³)

3.2.2.3 Determination of Volatile content

The mass of the activated Irish potato peel before carbonization was weighed and the mass of the final adsorbent produced after carbonization was reweighed to determine the weight difference caused by the escaped volatile content (VC).

 $VC = \frac{mass \ before \ carbonization-mass \ after \ carbonization}{mass \ before \ carbonization} \times 100\%$ 3.3

Where:

 W_0 = is the weight of the non-volatile matter retrieved from the furnace

3.2.2.4 Determination of moisture content

2.0 g of the adsorbent was dried continuously in an oven. The drying sample was removed and reweighed after 24 hours and the mass in grams, Wc was obtained. The ratio of the change in weight, (2.0- Wc), to the initial weight, (2.0), expressed in percentage, gave the moisture content.

3.4

Moisture content= $\frac{2.0 - Wc \times 100\%}{2.0}$

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Chapter Four

4.0 RESULTS AND DISCUSSION

4.1 Results

The results of the experiments conducted are tabulated below.

Table 4.1: Proximate Analysis of the Irish Potato Peel

| Properties | Values |
|------------------|---------|
| Moisture content | 4.5 % |
| Ash content | 11 % |
| Volatile content | 84.47 % |
| Pore volume | 1.8 |

Table 4.2: Samples of Adsorbent Produced

| Sample | Carbonizing Temp (°C) | Concentration of HCl |
|--------------------|-----------------------|--|
| A ₁ | 700 | 0.1 M |
| A ₂ | 600 | 0.1 M |
| A ₃ | 500 | 0.1 M |
| B ₁ | 700 | |
| B ₂ | 600 | and the second sec |
| B ₃ | 500 | |
| | | |

| Sample | Initial mass (g) | Final mass (g) | Volatile | Yield (%) |
|----------------|----------------------|---------------------|-------------|-----------|
| | Before carbonization | after carbonization | content (%) | |
| A ₁ | 100 | 10.25 | 89.75 | 10.25 |
| A_2 | 100 | 12.11 | 87.89 | 12.11 |
| A ₃ | 100 | 17.01 | 82.99 | 17.01 |
| B ₁ | 100 | 15.53 | 84.47 | 15.53 |
| B ₂ | 100 | 21.54 | 78.46 | 21.54 |
| B ₃ | 100 | 18.60 | 81.40 | 18.60 |

Table 4.3: Carbon Yield and the Volatile Content of the Adsorbents Produced

Table 4.4: Ash Content of the Adsorbent Produced

| Sample | Initial mass (g) | Final mass (g) | Ash content (%) |
|----------------|------------------|----------------|-----------------|
| A ₁ | 0.5 | 0.04 | 8 |
| A ₂ | 0.5` | 0.06 | 12 |
| A ₃ | 0.5 | 0.07 | 14 |
| B ₁ | 0.5 | 0.04 | 8 |
| B ₂ | 0.5 | 0.05 | 10 |
| B ₃ | 0.5 | 0.08 | 16 |
| | | | |

20

CERTIFICATION

This is to certify that this project report entitled PRODUCTION OF ADSORBENT FROM IRISH POTATO PEEL FOR THE REMOVAL OF HEAVY METAL FROM EFFLUENTS by Mbaogu Hillary Chika (2006/24230EH) meets the requirement for the partial fulfillment of the award of Bachelor of Engineering (B.Eng) degree in Chemical Engineering, Federal University of Technology, Minna.

Dr. D.O. Agbajelola Project Supervisor

Date

Dr. M.O. Edoga

Head of Department

External Examiner.

Date

Date

| Sample | Initial mass (g) | Final mass (g) | Moisture content (%) |
|----------------|------------------|----------------|----------------------|
| A ₁ | 2.0 | 1.99 | 0.50 |
| A ₂ | 2.0 | 1.99 | 0.50 |
| A ₃ | 2.0 | 1.94 | 3.00 |
| B ₁ | 2.0 | 1.97 | 1.50 |
| B ₂ | 2.0 | 1.96 | 2.00 |
| B ₃ | 2.0 | 1.89 | 5.50 |
| | | ₽.e. | e |

Table 4.5: Moisture Content of the Adsorbent Produced

Table 4.6: Pore Volume of the Adsorbent Produced

| Sample | Initial mass (g) | Final mass (g) | Pore Volume (cm ³ /g) |
|----------------|------------------|----------------|----------------------------------|
| A ₁ | 5.0 | 3.20 | 1.80 |
| A ₂ | 5.0 | 3.15 | 1.72 |
| A ₃ | 5.0 | 3.50 | 1.67 |
| B ₁ | 5.0 | 3.31 | 1.69 |
| B ₂ | 5.0 | 3.30 | 1.60 |
| B ₃ | 5.0 | 3.55 | 1.50 |
| | | | |

| Sample | Zinc | Iron | |
|----------------|-------|-------|---|
| A ₀ | 0.048 | 0.021 | |
| A ₁ | 0.022 | 0.000 | · |
| A ₂ | 0.027 | 0.000 | |
| A ₃ | 0.031 | 0.001 | |
| B ₁ | 0.026 | 0.000 | |
| B ₂ | 0.029 | 0.001 | |
| B ₃ | 0.031 | 0.003 | |
| | | | |

Table 4.7: Determination of the Adsorption Capacity of Samples (Absorbance)

Where, A_0 is the initial absorbance of the metals in the effluent before adsorption.

| Sample | Zinc | Zinc | Iron | Iron | |
|-----------------------|--------------------|---------------------------|-------|--------|--|
| | (100 ml of Digest) | (mg/L) (100 ml of Digest) | | (mg/L) | |
| | | | | | |
| A_0 | 1.266 | 12.66 | 1.240 | 12.40 | |
| A_1 | 0.580 | 5.80 | 0.000 | 0.00 | |
| A ₂ | 0.712 | 7.12 | 0.000 | 0.00 | |
| A_3 | 0.818 | 8.18 | 0.060 | 0.60 | |
| B ₁ | 0.686 | 6.86 | 0.000 | 0.00 | |
| B ₂ | 0.765 | 7.65 | 0.060 | 0.60 | |
| B ₃ | 0.818 | 8.18 | 0.176 | 1.76 | |

 Table 4.8: Determination of the Adsorption Capacity of Samples

Where, A_0 is the initial concentration of the metals in the effluent before adsorption.

| Sample | Particle size (µm) | Zinc | Iron | |
|--------|--------------------|-------|--------|--|
| A1 | 750 | 0.022 | 0.000 | |
| | 850 | 0.024 | 0.0005 | |
| B1 | 750 | 0.026 | 0.000 | |
| | 850 | 0.027 | 0.001 | |

Table 4.9: Effect of Particle Size on the Adsorption of Heavy Metals (Absorbance)

4.2 Discussion of Results

From the results obtained in the proximate analysis in Table 4.1, the Irish potato peel was found to have a moisture content of 4.5 %, volatile content of 84.47 %, ash content of 11 % and a pore volume of 1.8 cm^3 /g.

The Table 4.2 shows the type of adsorbent produced and the conditions in which they were produced.

From Table 4.3; where the results of the carbon yield and the volatile content were presented. It was observed that as the carbonization temperature increased, the carbon yield decreased and the volatile content increased. In the same Table 4.3 it was observed that the samples, in which the activating agent was added, had lesser carbon yield and higher volatile content than the samples that the activating agent was not added.

Table 4.4 presents the results of the ash content characterization and it shows that, the ash content of the produced adsorbent decreased with increase in carbonization temperature from 500 °C to 700 °C.

In Table 4.5, the moisture content of the adsorbents produced was noticed to decrease with increase in carbonization temperature from 500 °C to 700 °C. A further look at the table reveals that the chemically activated samples had lesser moisture content as against the non-chemically activated samples carbonized at the same temperature.

24

The pore volume as presented in Table 4.6 increased with increase in carbonizing temperatures (500 °C to 700 °C) and it was observed that the addition of activating agent greatly increased the pore volume.

The result of the adsorption capacity in absorbance, mg/100 ml of digest and mg/L of the samples in Tables 4.7 and 4.8 respectively, showed that increase in the carbonizing temperatures from 500 °C to 700 °C favoured the adsorption capacities of the samples. Also, it was noted that addition of the activating agent increased the adsorption capacities of the samples.

Adsorption capacity of the two best samples from the chemically activated and the nonchemically activated samples was analyzed using different particle sizes. In Table 4.9, it was observed that adsorption capacity of the samples increased with smaller particle size.

Effect of activating agent

It was noted that the addition of the activating agent to the Irish potato peel sample, increased the pore volume, the adsorption capacity of the adsorbent produced and hence, the removal of metal ions from the waste water sample. That is to say, the addition of the activating agent (0.1 M HCl) prior to carbonization is proportional to the quantity of metal ions removed. For instance, from the Tables 4.7 and 4.8 the sample A_1 adsorbed more than sample B_1 which was carbonized at the same temperature with A_1 but with no addition of activating agent.

Effect of Temperature

It was noted that as the carbonizing temperature increased from 500 °C to 700 °C, the yield in Table 4.3, the ash content in Table 4.4 and the moisture content of the adsorbent in Table 4.5 decreased. It was noted that as the temperature increased from 500 °C to 700 °C, the volatile content in Table 4.3 and the pore volume of the adsorbent in Table 4.6 increased.

It was also noted that as the temperature increased from 500 °C to 700°C, the adsorption capacity of the samples increased as seen in Tables 4.7 and 4.8. It can be said that the relationship between the carbonization temperatures and the adsorption capacity of the adsorbent is proportional.

Effect of particle size

The adsorption capacity varied with change in particle size. It was seen that the amount of metal ions adsorbed decreased with increasing particle size. The smaller particle size provides a shorter

diffusion path, thus allowing the adsorbent to penetrate deeper into the effluent more quickly, resulting in a higher rate of adsorption For instance, in sample A₁, the absorbance of zinc ions before treatment was 0.048 and after treatment, it changed to 0.022 mg/L at a particle size of 710 μ m. At a particle size of 850 μ m, the absorbance of zinc ions before, and after treatment were 0.048 and 0.024 respectively as in Table 4.9.

Chapter Five

5.0 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

This research work was successfully carried out and a bioadsorbent was developed from Irish potato peel. The results obtained from the experiments carried out show that this bioadsorbent was developed by activating with HCl and is best carbonized at a temperature of 700 °C. The adsorbent produced, showed more removal for the iron ions than the zinc ions. The heavy metal pollution level in the water was successfully reduced and utilization of waste biomaterial was achieved at an affordable and relatively cheap cost.

It will be disastrous if the developing countries make the same mistakes as the developed nations by sacrificing the environment at the expense of economic development. Because of the apparent lack of accurate information on the levels of heavy metals in the environment, it is most likely that pollution from point sources is more serious than documented. It is therefore essential to establish pollution standards and effective monitoring before environmental deterioration sets in.

The use of Irish potato peel as a bio-adsorbent is scientifically viable and should be explored (, further to treat waste water and other effluents from chemical process industries to treat heavy metals, reduce water pollution (and environmental pollution as a whole), as well as deliver safer and more purified water for consumption, domestic and industrial or commercial purposes.

5.2 Recommendation

It was observed during the course of experimental studies that there are lots of necessary equipments for carrying out this project that are either in short supply or unavailable. It is recommended that necessary equipment should be made available in the laboratories to allow for simultaneous work to be carried out, thereby conserving time and minimizing errors due to variations in experimental conditions.

Further research into the use of potato peel should be conducted to discover which parameters could be manipulated to give maximum yield and higher percentage efficiency of removal, and

to determine at which temperature the Irish potato peel will not adsorb more (optimum temperature).

The issue of power supply is one that must be tackled with urgency and determination, because power outage was a major source of concern during the course of this work. A steady power source should be acquired for the laboratories as well as back-up alternatives.

The use of waste bio-adsorbent materials should be thoroughly explored in Nigeria, as an alternative to activated carbon which is more expensive. This would favor our struggling economy. More research efforts should be channeled towards developing this aspect of technology by setting up programs that would encourage private individuals and corporate bodies to venture into the use of this technology. Research centres in universities and related institutions should also be encouraged to undertake systematic studies on specific problems related to environmental pollution and should be well equipped with the means to do so, as the prevailing effects of global warming and other environmental problems in our society and world at large is reflected in the incessant and heavy rains experienced late into the month of November. It is only by the coordination of efforts of government agencies and research-oriented institutions that the limited resources within these countries can be most efficiently utilized and possibly a permanent solution can be profilered.

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30

APPENDIX

1.0 Calculation of the proximate analysis

Calculation of moisture content

Moisture content = $\frac{\text{Initial weight} - \text{Final weight}}{\text{Initial weight}} \ge 100\% = \frac{2 - 1.91}{2} \ge 100\%$

Moisture content = 4.5%

Calculation of ash content

Ash content = $\frac{\text{Weight of ash}}{\text{Initial weight of sample}} \ge 100\% = \frac{0.055}{0.5} \ge 100\%$

Ash content = 11%

Calculation of volatile matter

Volatile matter = initial weight – final weight = 100 - 15.53

Volatile matter = 84.47%

Calculation of pore volume

Pore volume = initial weight – final weight = 5 - 3.52

Pore volume = 1.48

2.0 Calculation of the yield

 $Yield = \frac{final weight(m)}{initial weight (M)} X 100\%$

For sample A₁,

Yield = $(10.25/100) \times 100\% = 10.25\%$

For sample A₂,

 $Yield = (12.11/100) \times 100\% = 12.11\%$

For sample A₃,

Yield = $(17.01/100) \times 100\% = 17.01\%$

For sample B₁,

Yield = (15.53/100) x 100% = 15.53%

For sample B₂,

 $Yield = (21.54/100) \times 100\% = 21.54\%$

For sample B₃,

Yield = (18.60/100) x 100% = 18.40%

3.0 Calculation of volatile content

Volatile content (loss of weight) = Initial weight – Final weight

For sample A₁,

Volatile content = 100 - 10.25 = 89.75g

For sample A₂,

Volatile content = 100 - 12.11 = 87.89g

For sample A₃,

Volatile content = 100 - 17.01 = 82.99gFor sample B₁,

Volatile content = 100 - 15.53 = 84.47g

For sample B₂,

Volatile content = 100 - 21.54 = 78.46gFor sample B₃,

Volatile content = 100 - 18.60 = 81.40 g

4.0 Calculation of the ash content

Ash content = $\frac{\text{weight of ash}}{\text{weight of initial sample}} \ge 100\%$

For sample A₁,

Ash content = $(0.04/0.5) \times 100\% = 8\%$

For sample A₂,

Ash content = $(0.06/0.5) \times 100\% = 12\%$

For sample A₃,

Ash content = $(0.07/0.5) \times 100\% = 14\%$

For sample B₁,

Ash content = $(0.04/0.5) \times 100\% = 8\%$

For sample B₂,

Ash content = $(0.05/0.5) \times 100\% = 10\%$

For sample B₃,

Ash content = $(0.08/0.5) \times 100\% = 16\%$

5.0 Calculation of pore volume

Pore volume (V) = $\frac{(DW)}{\rho water}$ in cm³/g

Where:

 ρ water = 1g/cm³

DW = initial weight of sample – final weight of sample

For sample A₁,

 $V = (5 - 3.2) = 1.8 \text{ cm}^3/\text{g}$

For sample A₂,

 $V = (5 - 3.28) = 1.72 \text{ cm}^3/\text{g}$

For sample A₃,

$$V = (5 - 3.37) = 1.67 \text{ cm}^3/\text{g}$$

For sample B₁,

 $V = (5 - 3.31) = 1.69 \text{ cm}^3/\text{g}$

For sample B₂,

$$V = (5 - 3.40) = 1.60 \text{ cm}^3/\text{g}$$

For sample B₃,

 $V = (5 - 3.50) = 1.50 \text{ cm}^3/\text{g}$

6.0 Calculation of Moisture Content

Moisture content = $\frac{\text{Initial weight} - \text{Final weight}}{\text{Initial weight}} \ge 100\%$

For sample A₁,

Moisture content $=\frac{2.0-1.99}{2.0} \times 100\% = 0.5\%$

For sample A₂,

Moisture content $=\frac{2.0-1.99}{2.0} \times 100\% = 0.5\%$

For sample A₃,

Moisture content $=\frac{2.0-1.94}{2.0} \times 100\% = 3.0\%$

For sample B₁,

Moisture content $=\frac{2.0-1.97}{2.0} \times 100\% = 1.5\%$

10.0 Calculation of the Concentration of Iron in mg/L.

Since a dilution factor of ten was used during the digestion of the wastewater, therefore

Conc. in mg/L = conc in $\frac{mg}{100ml} \times 10$

For sample $A_0 = 12.40$

For sample $A_1 = 0.00$

For sample $A_2 = 0.00$

For sample $A_3 = 0.60$

For sample $B_1 = 0.00$

For sample $B_2 = 0.60$

For sample $B_3 = 1.76$