

**TREATMENT OF WASTE WATER FOR IRRIGATION USING  
ACTIVATED CARBON AND BIOCHAR FROM CROP RESIDUE**

**BY**

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**THIS DISSERTATION IS SUBMITTED TO THE UNIVERSITY OF  
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## DECLARATION

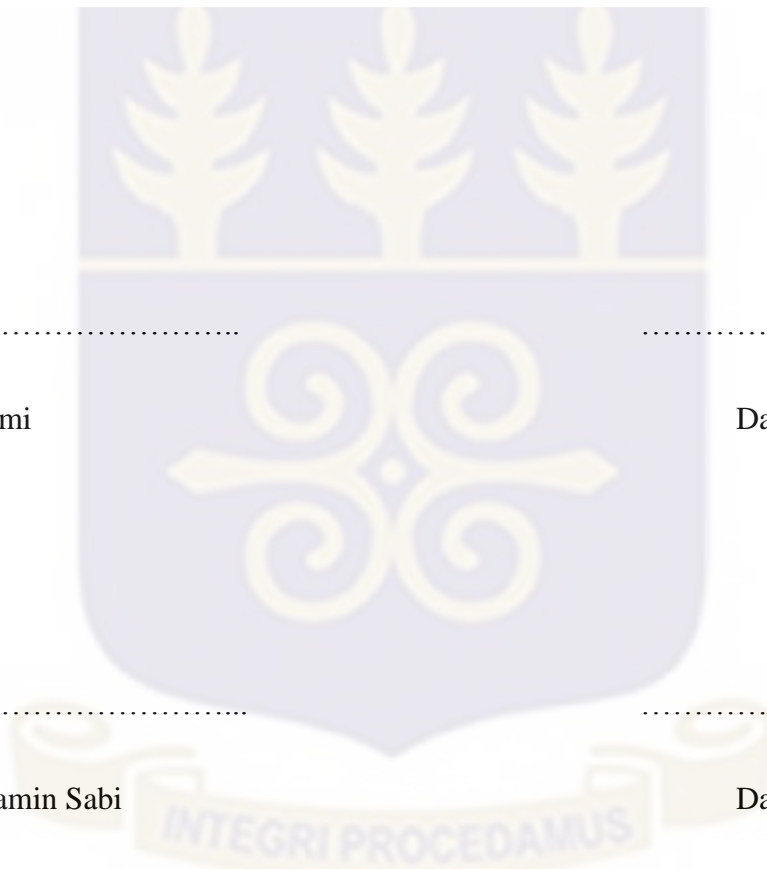
I, Bello Aisha Wunmi, the author of this research hereby declare that this work, titled “Waste water treatment for irrigation using activated carbon and biochar from crop residue”, with the exception of cited references is my own work produced from research under supervision in the Department of Agricultural Engineering, University of Ghana Legon. This work has not been presented for the awarded of any degree in any University nor any format prior to this. Submitted in July 2017.

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## ABSTRACT

Sustainability of urban vegetable farming has engaged many people for its economic benefits. The challenge to improving vegetable yield is water scarcity during dry season. Many farmers have therefore resorted to using waste water for irrigation, with the potential of contaminating crops. Contaminants in waste water are of biologically and physicochemically origin. This research aimed at treating waste water contaminated with heavy metals using activated carbon and biochar.

Cost effective technology was applied to produce biochar and activated carbon from crop residue. Six heavy metals were initially tested in the waste water and the adsorbents. Three of the heavy metals, i.e. Zn, Mn, and Cr were detected in the adsorbents only while the other three, namely Iron Fe, Ni and Pb were detected only in the waste water. Biochar and activated carbon therefore served as adsorbents and was used to decontaminate the Fe, Ni and Pb contained in the waste water. Waste water for vegetable irrigation was sampled from Korle-Bu backyard gardens. Treatments used were 100 g and 200 g each of biochar and activated carbon. One litre of sampled waste water was filtered through each amount of the adsorbents in a Randomized Complete Design.

There was significant difference in Fe decontamination between the Control (untreated waste water) and Treated Waste water at  $P \leq 0.05$  using LSD test but between the different adsorbents, there were no significant differences in Fe decontamination. There was significant difference in Ni decontamination between the control and 100 gram activated carbon. Ni was completely decontaminated by the 100 grams activated carbon. Finally, there was no significant difference in Pb decontamination between the control and all adsorbent treatments. In general, all adsorbents contributed to heavy metal decontamination in the waste water although insignificant in some cases. It was therefore concluded that activated carbon and biochar can be used for heavy metals decontamination in waste water hence recommended for treatment of waste water for irrigation.

## **DEDICATION**

I dedicate this dissertation to my kids, Yazid and Yumn Udoma for their patience during my studies in this prestigious University of Ghana, Legon.



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My gratitude goes first to God Almighty who made it possible for me to complete my studies in good health and ensured that I overcame all the obstacles in my way during this period.

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I also thank the technicians, Mr P. Owusu and Mr S. C. Brah of Ecological Laboratory (ECOLAB) of the Institute of Environment and Sanitation Studies (IESS), University of Ghana for their support in laboratory work and operation of the Atomic Adsorption Spectrometer (AAS).

Biochar and charcoal for preparing activated carbon were obtained from University of Ghana Forest and Horticultural Crops Research Centre (FOHCREC), Kade and transported to ECOLAB with the help of a loyal colleague and friend, Mr A. Yakubu. Special thanks to him for this assistance.

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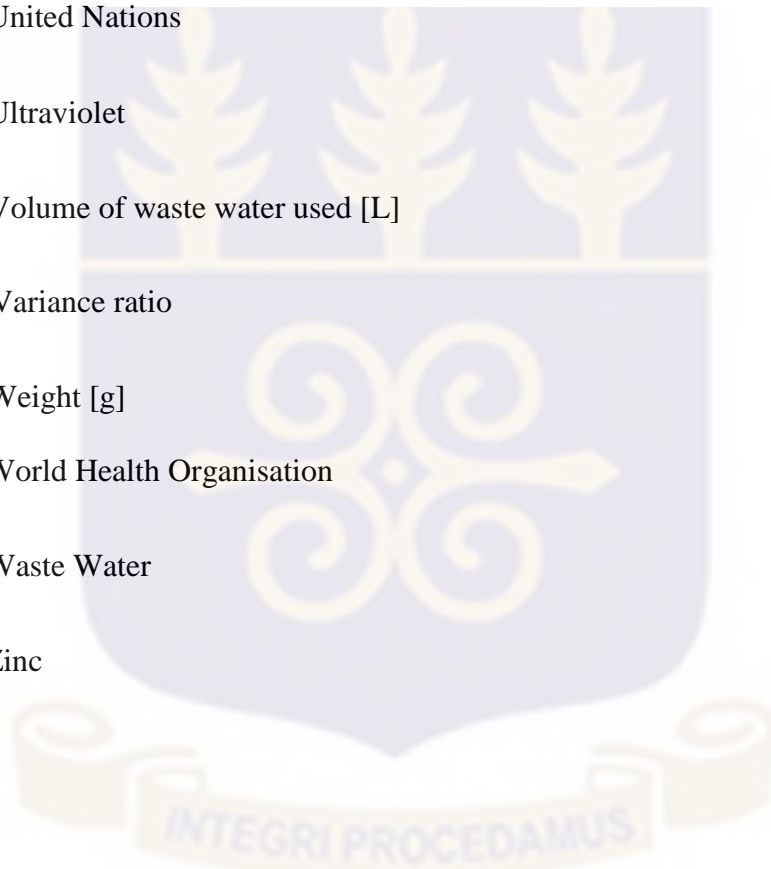
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## LIST OF SYMBOLS AND ABBREVIATIONS

AAS	- Atomic Adsorption Spectrometer
ANOVA	- Analysis of Variance
As	- Arsenic
Cd	- Cadmium
C <sub>f</sub>	- Concentration of trace element in waste water after treatment [mg/L]
C <sub>i</sub>	- Concentration of trace element in waste water before treatment [mg/L]
CO <sub>2</sub>	- Carbon dioxide
COD	- Chemical Oxygen Demand
Cr	- Chromium
CRD	- Complete Randomized Design
Cu	- Copper
DoF	- Degree of freedom,
ECOLAB	- Ecological Laboratory
EC <sub>w</sub>	- Electrical Conductivity of water
F pr.	- Frequency probability
FAO	- Food and Agriculture Organisation
FAOSTAT	- Food and Agriculture Organization Statistic
FDA	- Food and Drugs Authority

Fe	- Iron
GAC	- Granular Activated Carbon
GDP	- Gross Domestic Product
GSA	- Ghana Standard Authority
Hg	- Mercury
IESS	- Institute for Environment and Sanitation Studies
IWMI	- International Water Management Institute
KBTH	- Korle-bu Teaching Hospital
LSD	- Least Significance Difference
MBR	- Membrane Bioreactor
Mg	- Magnesium
Mn	- Manganese
MS	- Mean Squares
Ni	- Nickel
Pb	- Lead
pH	- Hydrogen Ion Concentration
$q_c$	- Amount of heavy metal adsorbed [mg/g]
R	- Percentage of heavy metal adsorbed (%)
SS	- Sum of Squares,

SSA	- Sub-Saharan Africa
T	- Temperature (°C)
TDS	- Total Dissolved Solids
TSS	- Total Suspended Solids
UG	- University of Ghana
UN	- United Nations
UV	- Ultraviolet
V	- Volume of waste water used [L]
V.r	- Variance ratio
W	- Weight [g]
WHO	- World Health Organisation
WW	- Waste Water
Zn	- Zinc



## CHAPTER ONE

### INTRODUCTION

#### 1.1 Background

Demand for food is increasing in most arid and semi-arid parts of the world as a result of adverse weather conditions that affect rainfall for crop production especially in the dry seasons. Irrigation is used to remedy crop failure due to drought in the dry seasons by small scale vegetable farmers globally. On the other hand, urban vegetable farmers face numerous challenges in finding unpolluted water sources in the cities for irrigation (Obuobie et al., 2006).

Vegetable farming is gaining popularity both in the urban and peri urban areas due to its short term in income generation. Many of these vegetable farmers in the cities of Accra, Lagos and other parts of Sub-Saharan Africa (SSA) use waste water flowing in nearby drains (gutters) to irrigate their vegetable crops due to water scarcity. Waste water used for irrigation is health threatening since it is a potential source of contaminants to vegetable crop grown for human consumption.

FAO data based on imputation methodology for the year 2014 ranked China the world's largest producer of vegetables with an average production of 325,979,466 tonnes, Nigeria ranked fourth largest producer with 6,681,688 tonnes and Ghana emerged one hundred and fifty - first (151) on the global ranking chart with a production of 10,749 tonnes (FAOSTAT, 2014).

Vegetables are grown in both rural and urban areas with the rural areas usually having larger cultivated land area for commercial purposes. Most vegetables are susceptible to harsh climatic condition such as heavy rain, humidity and atmospheric temperature and thus influenced the distribution of the various vegetable grown across the country.

Vegetables do well in savannah areas as insect and pest attack are minimal compared to the forest areas of the country. Field grown vegetable crop suffer severe insect and pest infestations in the forest belt making farmers in the forest zone apply agro-chemicals frequently for insects and pest control.

In Ghana, two forms of urban vegetable crop farming exist, i.e. the open space for urban market purposes and the backyard garden solely for home consumption by the farmer and his family (Drechsel et al., 2006). Different varieties of vegetables are produced in Ghana for both local and export markets.

The most common vegetables grown in Accra include lettuce, cabbage, spring onions, carrot, cauliflower, green beans, hot pepper, garden eggs, tomatoes, okra, green pepper and cucumber. Both the exotic and local varieties are grown by different farmers in back yard gardens, environ domes and the field where only few are grown in environ dome mostly by institutions for research purposes.

Urban vegetable production is mainly practiced by urban dwellers living under peasant economy in the cities. Vegetable growing areas in Accra include Dzorwulu/plant Pool, Marine drive (Ghana Independence Square area), La (behind Burma Camp) and Korle-bu Hospital residence backyards (Obuobie et al., 2006). Small holder vegetable farms can also be found around Opeibea (Airport residential area) and Okponglo - Legon around the University of Ghana and opposite the premises of Ghana Standard Authority (GSA) at Shiashi, Accra.

Vegetable production is very lucrative and resourceful but the challenge of expanding farm size due to land tenure and water scarcity in urban cities of developing countries is a hindrance to increasing production. Meanwhile, agricultural workforce accounts for about 52 percent of the

total populace contributing 22 percent of Ghana's Gross Domestic Product (GDP) (Gonzalez et al., 2016).

Basically there are two sources of water for farming namely storm water (rainfall) and irrigation sources of water. Precipitation or rain-fed agriculture is losing its effectiveness and economic value due to scarce and irregular patterns in rainfall for crop farming. Many at times the amount of rainfall is insufficient for crop production even in the rainy-seasons in Sub Saharan Africa. To supplement the insufficient precipitation for crop production especially in vegetable farming, irrigation has always played an important role and very effective for ensuring continuous crop production throughout the year. This is possible provided there is water available for irrigation throughout the year.

In the absence of rainfall source of water, urban vegetable farmers' alternative source of water for irrigation are nearby drains (gutters), dugout wells and or pipe borne water. The former made up of waste water from homes, public institutions, the industries as well as storm water runoff into such drains is widely practiced. In the city of Accra, irrigation water for urban vegetable production is mainly sourced from drains especially in the dry spell seasons (Obuobie et al., 2006). Such act of sourcing drain water for irrigating vegetables by some farmers in urban Accra is increasing along with the increasing demand for fresh vegetables. The types of irrigation system practised by these urban vegetable farmers are the watering can irrigation systems and the use of water hose connected from stand- pipe or pumped from a dugout pond. Furrow irrigation, sprinkler irrigation and drip irrigation are rarely practiced by these urban vegetable farmers.

Waste water could be any undesired water source physically or chemically contaminated with substances that have adverse effects on human health upon consumption. It could also appear unpleasant in terms of odour and colour for human consumption. It is usually produced from

domestic sources, storm water runoff, municipal and industrial effluent which flow into main drains. Common contaminants in waste water are heavy metals (trace elements), acids & bases, microorganisms, bacteria, metalloids, poisonous elements, faecal matter and so on.

More than 800 million farmers are engaged in urban agriculture and not less than 3.5 million ha of land is irrigated with waste water globally (Qadir et al., 2010). According to Drechsel et al. (2006), West African cities have less than 10% of the generated waste water collected in piped sewage systems for treatment.

There are different forms of contaminants in waste water including dozens of heavy metals. The anticipated heavy metals in a given waste water proposed for treatment using activated carbon and biochar in this research are Zinc (Zn), Iron (Fe), Nickel (Ni), Chromium (Cr), Manganese (Mn) and Lead (Pb). Treatment was carried out only after the given heavy metals were tested and detected in the waste water. Heavy metals commonly found in waste water include Arsenic (As), Zinc (Zn), Mercury (Hg), Cadmium (Cd), Chromium (Cr), Manganese (Mn), Iron (Fe), Copper (Cu), Magnesium (Mg) and Lead (Pb).

While the intake of certain amount of most heavy metals is unsafe for human health, World Health Organisation (WHO) of the United Nations (UN) and some countries have defined threshold concentrations of the various heavy metals in food substances allowable and safe for consumption. On the other hand, some of these heavy metals such as Iron (Fe), Magnesium (Mg) and Zinc (Zn) are micro nutrients that play important roles in both human being and crop.

Waste water or contaminated water can be treated and used for irrigation, domestic, industrial and other purposes. Several methods and technologies have been applied in treating contaminated or waste water whereby the level of treatment depends on standards and the end user's preferences. Among the methods used in treating waste water, the scientific methods include the use of filtration

membranes, crop residue, chemicals, biochar, treatment plants, bioremediation and activated carbon. The activated carbon and biochar method is economically viable as compared to other scientific methods and applied in this research to treat waste water contaminated with heavy metals for irrigation purpose.

Activated carbon also called activated charcoal is a more effective and advanced form of charcoal which has been used in ancient times and still in existence (Çeçen, 2014). It is produced from pyrolysis of biomaterials usually carbon rich materials or carbonaceous materials. Carbonization and activation are the two stages involved in its preparation. It is used primarily for removal of odour and extracting heavy metals and harmful chemicals from contaminated water and other media. It can be produced from coconut shell, sawdust, wood, bitumen, coal, crop waste etc. Two types are available, namely thermal and chemical activated carbon. Activated carbon can be produced from all natural organic materials rich in carbon (Çeçen, 2014) but only few can be commercially used (Marsh and Rodriguez-Reinoso, 2006).

Biochar is produced from thermal decomposition of plant biomass in partial or total absence of oxygen to produce char, carbon dioxide (CO<sub>2</sub>) and combustible gases intended specifically for application to soil (Sohi et al., 2010). Two types of biochar produce are high pyrolysis biochar and low pyrolysis biochar. Biochar has been reported to conserve water and nutrient in the soil and the extent of conservation is proportional to the amount applied into the soil. In other words, the effectiveness of biochar depends on its quantity and particle sizes. Biochar can be produced from rice husk, corncob and saw dust among other biomaterials.

Heat treatment of carbon rich materials increases pore space of the respective material. This leads to increase in surface area of the end product after carbonization. It is this charged surface area of the carbonized material that attract and adsorb molecules of other substances. The science of

adsorption of elements to surface of carbonized materials are the principles behind the use of activated carbon and biochar as adsorbents for heavy metals decontamination.

## **1.2 Problem statement**

Most Urban vegetable farmers have resorted to the use of waste water for irrigating their crops due to water scarcity and high cost of applying treated pipe-borne water. Cultivated vegetable crops when irrigated with waste water are exposed to all forms of contaminants including heavy metals and microorganisms. Heavy metals contamination of vegetable crops from waste water irrigation is as dangerous as microorganism contamination and it is a potential threat to human health. The act of using waste water by some urban vegetable farmers for irrigation is increasing on daily bases. There is therefore the need to intervene with waste water treatment in order to reduce the widespread of heavy metals contamination on vegetable crops.

State regulations and control measures have not been successful in stopping farmers from the use of waste water in vegetable irrigation in various urban areas especially in the city of Accra. Little has been done by Food and Drugs Authority (FDA) and Ghana Standard Authority (GSA) documented to creating public awareness on measures to curtail vegetable contamination through waste water irrigation. Appreciable research work have reported the use of waste water in irrigating vegetable crops in various cities of Ghana but none has proposed a simple mechanism to treat such waste water of heavy metals for irrigation. As the control of such urban vegetable farmers to desist from using waste water is less enforced by state authorities and stake holders, it is necessary and important to propose and research on cost effective techniques of treating waste water for irrigation.

Plant residue such as corn cob, pruned branches, rice husk, saw dust and litter are mostly used in smokehouses and as firewood in our homes for cooking, an act that can potentially cause environmental pollution and ozone layer depletion. Activated carbon and biochar for waste water treatment can be prepared from these plant residues that pollute the environment when used in smokehouses and as firewood in cooking or when improperly disposed. Activated carbon and biochar have the properties of adsorbing and retaining biological and chemical elements hence can be used to filter heavy metals in waste water for irrigation purpose.

### **1.3 Aim and objectives**

This research aimed at assessing the potential of varied quantities of activated carbon and biochar to treat waste water contaminated with heavy metals.

The following objectives helped achieve the overall aim of the research:

- a. To eliminate or ameliorate heavy metals contained in waste water used for vegetables irrigation.
- b. To vary activated carbon and biochar amounts to assess its effectiveness in heavy metals decontamination from waste water.
- c. To reduce environmental pollutant sources by converting rice husk (plant residue) into biochar for decontaminating heavy metals from waste water.
- d. To apply simple and cost effective technology by using crop residue to produce adsorbents for heavy metals decontamination in waste water.

## CHAPTER TWO

### LITERATURE REVIEW

#### 2.1 Waste water treatment and irrigation

International and local research works carried out on water and sanitation has gained overwhelming attention. For example the World Health Organisation (WHO) of the United Nations (UN) and some major countries have invested in research work to alleviate health challenges emanating from food contamination. Though much attention had been given to water sanitization for human consumption, many people have also raised concern over food crop or vegetable contamination. In other words, contaminated vegetable is equally dangerous as contaminated water for consumption. The most alarming issue of concern is that, cultivated crop contamination could result from the use of contaminated water for irrigation. Another source of vegetable crop contamination is the soil or growth media in which the crops are cultivated.

Vegetable contamination in urban Accra and beyond is mostly as a result of using waste water from nearby drains (gutters), dugout and mini ponds for irrigation. These contaminants range from biological to physicochemical parameters. Appreciable research work including (Lente et al., 2014; Akrong et al., 2012; Qadir et al., 2010; WHO, 2006; Amoah et al., 2007) have reported plausible results on evidence of waste water used for vegetable farming in urban cities of West Africa and other parts of the globe. Consumption of biological and or physicochemically contaminated vegetables has also been reported but no research work was done on treating such waste water to eliminate heavy metals for vegetables irrigational purpose in Ghana.

In Ghana, the city of Accra has recorded no research work done in waste water treatment to eliminate or decontaminate heavy metals for vegetable irrigation purpose. Even the current waste

water treatment plant at the University of Ghana, Legon is not clearly indicated as to its propensity to decontaminate heavy metals in waste water for food crop irrigation purpose. This is because waste water could contain either physicochemical substances, e.g. heavy metals, oil suspension etc., and biological constituents such as bacteria, faecal coliforms, pathogens and so on or both.

Fresh water for agricultural crop production is progressively becoming exhausted and inaccessible by the global community. Waste water used for urban vegetable irrigation is on the increase and cannot be overlooked. Agriculture being the largest consumer of fresh water in the world is equally contributing to waste water production. This is largely as a result of runoff that carry along chemicals and fertilizers applied on rain-fed and irrigated farms into nearby fresh water resources.

Treatment of waste water for irrigation purposes is commendable and will gain more attention as long as water scarcity for food crop production persist on the global community. In this regard, several methods of waste water treatment have been researched and proposed to increase the availability of uncontaminated water for agriculture and human consumption. This also contributes to increment of sanitize irrigation water for sustainable agricultural crop production. Suitable method of waste water treatment lies in using the most economic and viable technology. Feasibility of economic and technical factors of the methods of removing heavy metals from aqueous solution may limit the implementation of the respective methods (Sari and Tuzen, 2008).

## **2.2 Urban vegetable farming in Ghana**

Vegetable farming has evolved from peasant farming to the commercial scale and attracting much workforce due to its economic returns. Nevertheless its lucrateness has engaged exuberant young men in Ghana especially in the urban cities to earn their living. In Ghana, the larger vegetable

quantities are obtained from rural areas. Current mining activities have polluted possible irrigation water resources with heavy metals in most of these rural Ghanaian communities thereby reduced irrigation water availability. Lack of storage facilities for bumper harvest in the rainy season have also rendered most vegetable produce waste on the farm and market gates. Urban vegetable farming is an alternative source of income and practiced alongside rural vegetable farming with the male folk dominating in the venture. Selected vegetable production (tonnes) from 1986 to 2011 in Ghana has been reviewed in (Gonzalez et al., 2016).

Gonzalez et al. (2016) found out that Ghana's vegetables on the international market is remarkable, nevertheless the urban middle class residents and financially sound people demand high quality vegetable products with more emphasis on food safety. The GhanaVeg Program's recent report on the progress, challenges, opportunities and way forward in the vegetable industry in Ghana has been well elaborated in Gonzalez et al. (2016). However the report did not mention heavy metals contamination of vegetables nor proposed any method of treating waste water contaminated with heavy metals for irrigation.

Waste water used for vegetable irrigation in Ghana has been confirmed to be one of the potential sources of heavy metal and biological contaminants (Lente et al., 2014; Akrong et al., 2012; Keraita and Drechsel, 2004; Cornish et al., 1999).

### **2.3 Irrigation in urban vegetable farming**

Irrigation is the application of water to crop either in the absence of natural precipitation or as a means of supplementing the insufficient and unpredictable precipitation in crop production. The vital factors determining crop growth and yield performance are water and nutrient. Although lack of arable lands in some parts of the world is a hindrance to sustainable crop production, water

scarcity is a dominant factor contributing to unsustainability in agricultural crop production in Ghana and many arid and semi-arid parts of the world.

Irrigation has the potential to boost crop production in dry seasons but the challenge of accessing fresh water in Accra for vegetable irrigation has forced many farmers to resort to waste water for irrigating their vegetable farms.

There are many vegetable farmers in urban Accra who make use of irrigation especially in the dry season due to high returns from vegetable trading during dry seasons. Only few of these urban vegetable farmer owns about 1 ha of land for cultivation. The majority of farms are under a land area of 0.01 -0.02 ha per farmer for cultivation and the maximum of 2.0 ha are possible in the peri-urban areas (Obuobie et al., 2006). Recent urban infrastructural development is seizing and making use of the available land reserved and allocated for such vegetable farms limiting farmers to smaller plots of land for cultivation compared to 10 – 20 years ago.

The only water resource readily available at all time of the year is waste water flowing in the open drains basically from domestic waste water source (grey and black water). The defiant urban vegetable farmers use such waste water flowing in the open drains for vegetable irrigation during “water scarce” situation when there are no alternative source of water.

Heavy metals are everywhere on earth and can be found not only in waste water but soil and other growth media. Therefore vegetable contaminated by heavy metals is not only via irrigated waste water but soils or growth media are potential sources as well.

## **2.4 Sources of irrigation water**

According to FAOSTAT (2015) approximately 70 percent of the world's total fresh water is used for agricultural purposes. Irrigated agricultural farming covers about 20 percent of the total cultivated land contributing 40 percent to world food production (FAOSTAT, 2015). Fresh water for irrigation in urban Accra is not only inaccessible but expensive. Urban water withdrawn for consumption constitute 15 percent to 25 percent while the rest is returned as waste water to urban hydrologic system (Qadir et al., 2010).

Waste water may be produced from industrial effluent, municipal, landfill leachate, domestic and agricultural sources. The type of waste water from urban drainage system (gutters) used by some vegetable farmers in the city is a combination of all the possible sources which has higher chances of contaminating the vegetable crops grown. As a result of population increase with more industries sited in urban cities, waste water production increases concurrently. Asano et al. (2007), Lazarova and Bahri (2005) and Qadir et al. (2007a) also attributed increase in waste water generation in developing countries to the increasing urban population and residents seeking higher standards of living. The increased populace contributes to the conversion of larger amount of fresh water through domestic, commercial and industrial usage into waste water.

The most common sources of water for vegetable irrigation in urban Accra are pipe borne, manually dug-out well, streams and open drains (gutters) (Akropong et al., 2012). None of the above mentioned water sources are tested for health threatening contaminants and treated by the various vegetable farmers before used for irrigation in the Accra Metropolis.

Ghana's waste water production from all the possible sources was estimated by Agodzo et al. (2003) to have increased from 36 percent in the year 2000 and predicted to about 45 percent in the year 2020. Waste water is commonly discharged into natural water bodies at a raw state or slightly

treated making such water bodies vulnerable to pollution (Qadir et al., 2010). Some urban vegetable farmers in Accra will continue to use waste water for irrigation because there are no strict applicable national regulations mandating and guarding against the use of waste water for vegetable production in the city.

Other vegetable farmers presumed of harnessing nutrients in waste water made up of kitchen waste matter as well as black water for use as fertilizer without considering other potential pollutants in the waste water. The act of using waste water with the intention of tapping nutrients for vegetable production is detrimental to human health and the natural environment (Qadir et al. 2007 b).

According to Qadir et al. (2010), the WHO upon its quest to fight against the use of waste water for irrigation, revised its guidelines in 2006 (WHO, 2006) suggesting the use of untreated waste water in the production of only industrial or non-consumable food crops. The reason was attributed to the fact that it was difficult to impose a ban or improvise an alternative source of vegetable production in the affected areas for urban markets. Technical and financial intervention in waste water management in developing countries require a short-term strategies and effective solutions to prevent negative impact of waste water irrigation on human health and the environment (IWMI, 2006; WHO,2006).

## **2.5 Contaminants in waste water used for irrigation**

Most aspects of the pertinent literature including (Amoah et al., 2007; Akrong et al., 2012; Drechsel et al., 2006) have reported the use of contaminated waste water for vegetable irrigation in Accra, Ghana. The contaminants in such waste water are basically biologically or physicochemically based. Plausible research works of (Drechsel et al., 2006a; Keraita et al., 2003; Amoah et al., 2007) and other researchers have shown much attention to biological contaminants

in waste water more than the chemical constituents (heavy metals) in Ghana. Drechsel et al. (2006a) observed that major health concern has been limited to microbiological pathogen from domestic sources of waste water in Ghana and throughout the sub region. According to Keraita et al. (2003) the microbiological organism contaminants in waste water for irrigation are mostly beyond WHO permissible limits and hence the need to treat waste water.

Biological contaminants in waste water vary from living organism to bio-waste of other organisms. These include faecal coliform, total coliform, bacterial, algae, worms, food debris, pathogens etc. Amoah et al. (2007) investigated for total and faecal coliform and helminthic egg contaminants in waste water used for irrigating lettuce in urban Ghana. He attributed lettuce contamination to manure application and post-contaminated soil beside irrigation water.

Heavy metals are ubiquitous on the earth surface and are obtained naturally from the earth crust through weathering of some parent rocks and volcanic eruptions. Their dispersal is usually through sediment transport in runoff water and Aeolian dust carried from point sources to different parts of the earth. Heavy metals could be contaminants based on the type and the assimilation requirement by the crop. Zinc (Zn) and Manganese (Mn) for instance are heavy metals but important micro nutrients for the crop.

Though heavy metals are naturally occurring metals, anthropogenic activities have increased their widespread in our environments. Lente et al. (2014) observed heavy metal contamination in sample irrigation water used for vegetable production in some areas of Accra. This was not limited to the irrigation water alone but the soil and edible parts of the vegetable grown were also confirmed contaminated with heavy metals.

Heavy metal decontamination from water and waste water resources is an important issue pertinent to our environment and at economic point of view (Abdel Ghafar et al., 2013). Treatment of

irrigation water contaminated with heavy metals has been an issue of concern to Ghanaian populace. Keraita and Drechsel (2004) and Cornish et al. (1999) reported that heavy metal pollution of irrigation water in most situations does not exceed irrigation standards but Obuobie et al. (2006) drew exceptions to mining areas of Ghana. Irrespective of the heavy metal sources and quantity defined as safe limit under irrigation standards, its accumulation in the human system could be health threatening and it is necessary to decontaminate even the very minimum concentration.

## **2.6 Waste water treatment for irrigation**

Many technologies have been used in treating waste water and the extent of treatment is highly dependent on the end user's preference. Examples include the use of sand and charcoal filters (Çeçen, 2014), application of membrane filters, treatment plants and the use of low cost adsorbent from crop residue (Wan Ngah and Hanafiah, 2008). Thus waste water could be treated for domestic use, industrial, municipal and or irrigation purposes. Depending on the aim of a study, waste water could be treated to eliminate biological contaminants or physicochemical contaminants or both. In addition to chemical and biological contaminants in waste water treatment for domestic reuse, odour and colour are usually given equal consideration in the treatment process while this is not the case for irrigation purposes.

Zahid et al. (2016) in their study treated municipal waste water for the reduction of Chemical Oxygen Demand (COD), Total Dissolved Solids (TDS), Total Suspended Solids (TSS), colour and turbidity to improve its quality for irrigation. Their study presented an important information but did not consider treating chemical contaminants such as the heavy metals.

Selected methods of heavy metals decontamination from aqueous solution e.g. waste water include, reverse osmosis, ion exchange, adsorption on activated carbon, membrane filtration and chemical precipitation (Yu et al., 1999; Turker, 2012; Haque et al., 2013).

In the light of treating waste water for agricultural purposes, it is observed that Ghana, Nigeria and many other developing countries have done less in implementation and practices. There are many water treatment companies for domestic use in Ghana out of which none of them treat waste water for agricultural use. High cost of treating waste water for agricultural purpose could be a factor refraining farmers from patronizing the available technologies.

There is no waste water treatment to decontaminate heavy metals for agricultural crop production in the larger part of Accra. Literature has emphasized on waste water production, its use in irrigating vegetable crops, vegetable contamination and the health implications of waste water irrigation of vegetable. Little has been done to propose a simple inexpensive technology for heavy metals decontamination from waste water for irrigation purpose in urban areas in Ghana. The technology of preparing biochar and activated carbon using plant residue in this research is a cost effective method of treating waste water of heavy metals for irrigation purposes.

## **2.7 Agro by-products for waste water treatment**

Various materials have been applied in heavy metal adsorption from contaminated waste water, as reviewed in Kumar (2006) who concluded that low cost adsorbents are highly beneficial for commercial purpose in the future. He emphasized on agricultural products and by-products as low cost sorbents for decontaminating heavy metals in water and waste water.

Demirbas (2008) reviewed heavy metal adsorption onto agro-based waste materials and reported the technology was inexpensive and an alternative to existing technologies. Abdel Ghafar et al. (2013) on the other hand carried out an experiment on Cd and Pb removal using biosorption mechanism and based on their research findings, they concluded that *Anabaena sphaerica* biomass can serve as a low cost biomass for heavy metals decontamination from waste water.

Similarly, Buasri et al. (2012) also converted corn cob to heavy metal ion biosorbent for waste water treatment where they successfully used the agricultural by-product in adsorbing Zn (II) from an aqueous media (waste water). They also concluded that the agricultural by-product, i.e. corn cob could serve as an alternative low cost biosorbent for heavy metal ions decontamination from aqueous media.

Activated carbon and biochar can be produced from low cost agricultural products and by products such as plant biomass and residue. Available materials used to produce activated carbon for waste water treatment include wood shavings, corncob, coconut husk and bituminous coal. Crop residue such as rice husk, corncob, cocoa pods and saw dust can also be used to produce biochar.

Methods of treating waste water include the use of activated carbon (Farahmand, 2016), biochar (Huggins et al., 2016) as well as *Moringa oleifera* cake residue as an agricultural by-product (Eman et al., 2014). Fazal et al. (2015) also used Membrane Bioreactor (MBR) to treat waste water among others. Living organism, fresh water alga (*Anabaena sphaerica*) biomass was used to remove Cd (II) and Pb (II) from an aqueous solution through biosorption mechanism in a research work by Abdel Ghafar et al. (2013) in Egypt.

Availability and cost efficiency in acquiring raw material for adsorbents in waste water treatment of heavy metals informed the choice of biochar and activated carbon in this study. Research on agro-based waste for heavy metal adsorption in waste water treatment was well reviewed by

Demirbas (2008). Heavy metals decontamination from waste water has also been carried out by (Wahi et al., 2009; Li et al., 2009).

Among the advantages of selecting biochar and activated carbon for waste water treatment include its availability and low cost in Ghana. Moreover converting crop residues e.g. rice husk, corncob, corn straw, sawdust etc. is a form of reducing their pollution effect on the environment.

The contribution to knowledge on the subject matter, i.e. waste water treatment is the idea of measuring the efficacy of varying quantity of the adsorbent used for treatment i.e. biochar and activated carbon. In other words the effectiveness of biochar and activated carbon on heavy metal adsorption depends on quantity used and the degree of contamination of the aqueous solution, waste water in this case.

## **2.8 Sorbent adsorption capacity**

Materials rich in carbon after heat treatment have different surface area from the original. The state of the adsorbent e.g. granular, powdery or pellets come with different surface areas as their binding sites for ions and molecules. In general, all carbon-rich substances increase in pore space when heated at high temperatures. It is these pore spaces that serve as binding sites for ion exchange during adsorption, chemisorption, physisorption and biosorption processes. The most important factor of each adsorbent material is its adsorption capacity (Kyzas and Kostoglou, 2014).

Biochar and activated carbon are carbonaceous materials which have been treated with high temperature range of 200 °C and above to reduce the carbon content in their molecular pore spaces. Depending on the pyrolytic temperature used, there are two types of biochar, namely low pyrolysis (below 400 °C) and high pyrolysis (400 °C and above) temperature biochar (Kameyama *et al.*, 2014). The low pyrolysis temperature biochar are more reactant and can easily be degraded by soil

microbes while high pyrolysis temperature biochar is recalcitrant in soil and other media (Brewer *et al.*, 2011).

Activated carbon is prepared when carbonaceous materials are exposed to high temperature range of 400 °C to 600 °C in a process termed carbonization (Bansal and Goya, 2005) and further activated with either temperature (700 °C and above) or chemical like  $K_2CO_3$  and KOH (Tay *et al.*, 2009). A research was also done by Hesas *et al.* (2013) in preparation of activated carbon from crop waste using phosphoric acid ( $H_2PO_4$ ) as a chemical activating agent. This leads to increase in pore space and surface area of the carbonized material. The difference between biochar and activated carbon is the activation process in preparing activated carbon whereas scientifically they are all charred materials.

Biochar and activated carbon are among other adsorbents with different adsorption capacities based on many factors like surface area, temperature of the aqueous solution containing the heavy metal, time to establish equilibrium in adsorption process, particle size of the sorbent and sorbent concentration. Experiments carried out with varying biosorbent amounts showed that metal ion adsorption increased directly proportional to the different amount of biosorbent applied (Buasri *et al.* 2012).

While previous studies had reported the efficacy of adsorbent in decontamination to proportionate its quantity applied, this research applied two different quantities (100 g and 200 g) of both biochar and activated carbon to ascertain the degree of heavy metal decontamination in waste water. The objectives of the study will therefore ensure the application of a known quantity of adsorbent for effective heavy metal decontamination. This will inform the general public on the economics of the technology applied in treating waste water contaminated with heavy metals for irrigation purpose.

## CHAPTER THREE

### MATERIALS AND METHODS

#### 3.1 Experimental site

Urban waste water used for vegetable irrigation was sourced and collected from the Korle-Bu Teaching Hospital (KBTH) backyard gardens. The Korle-Bu Teaching Hospital is located in Accra Metropolitan assembly on Latitude 5°32' 9.71" N and Longitude 0° 13' 23.20" W along the Gulf of Guinea coastal belt in West Africa. Accra covers a land area of about 230 km<sup>2</sup> to 240 km<sup>2</sup> (Obuobie et al., 2006). Accra's mean monthly temperature ranges from 24 °C to 28 °C and a bimodal rainfall pattern with an annual rainfall of 810 mm according to Obuobie et al. (2006). But a study conducted by Logah et al. (2013) indicated a decline in rainfall amount over the last 30 years. Open space farming is practiced under small scale in most areas of Accra Metropolis. Major small holder farms where waste water, among other water sources, are used for vegetable crop production in the Greater Accra region was mapped by Kufogbe et al. (2005) in Figure 3.1.

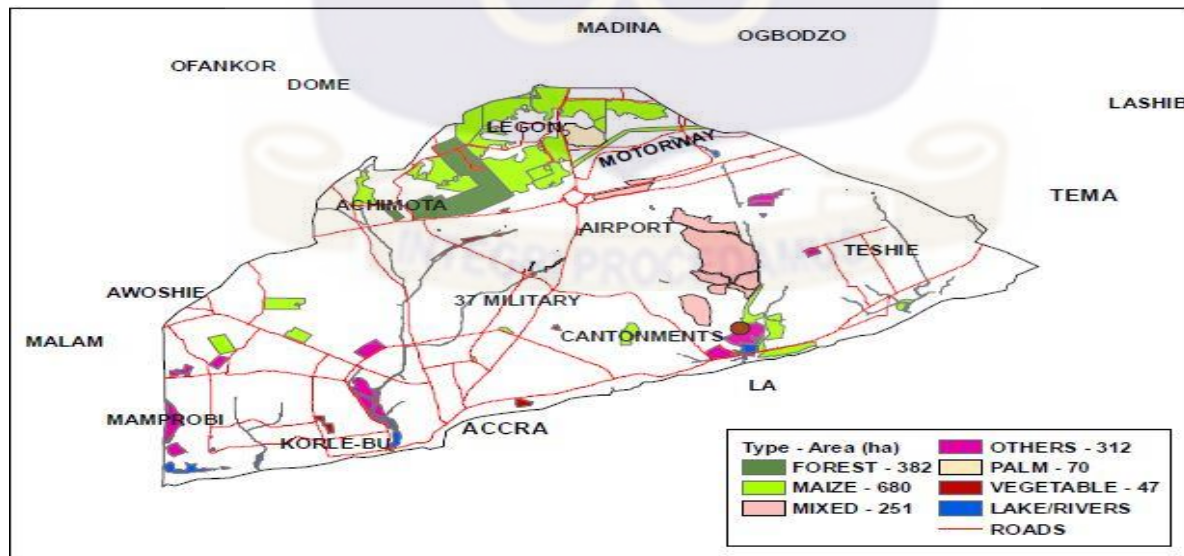


Figure 3.1 Map of small-holder farm sites in Greater Accra region.

The experimental site under this study, Korle-Bu Teaching Hospital backyard gardens where waste water are used for vegetable irrigation is labelled 'A' with green highlight as the legend (Figure 3.2). It currently covers less than 10 ha of land area. The present land size is prone to reduction for the hospital's infrastructural development. The cultivated site is basically used for vegetable farming throughout the year with few cereal (maize) cultivated in the rainy season. It is managed by the various farmers under rain-fed agricultural system of farming in the wet seasons of the year and supplemented by irrigation during off season (dry season). Irrigation water for vegetable production in the area is sourced from open drains that carry waste water (grey and black water) from the hospital departments and its staff residents usually diverted into hand dug out wells. Few farmers who are well-resourced use mechanical pumping machines to extract water from underground water resources on the farm site for irrigation. Waste water used for irrigation on the farm was sampled and transported to Ecological Laboratory (ECOLAB) of the Institute for Environment and Sanitation Studies (IESS) of the University of Ghana, Legon for analysis.

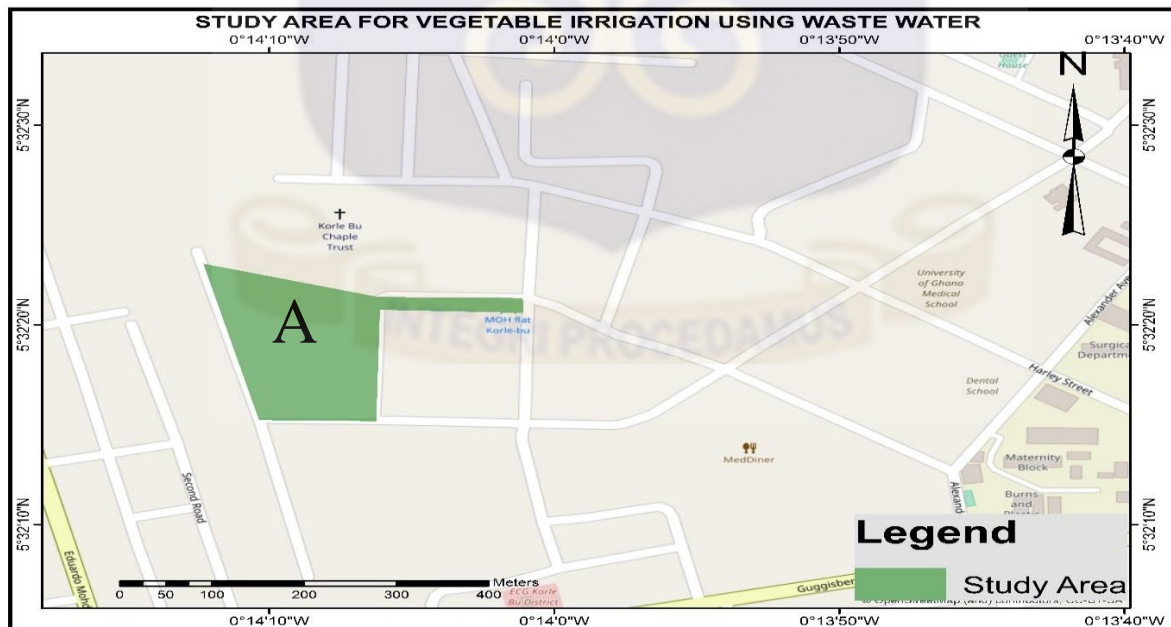


Figure 3.2 Map of Korle-Bu Teaching Hospital showing study area, A (highlighted green).

### 3.2 Experimental materials

Materials used under the study were economical and sourced locally. Biochar for instance was produced from plant residue i.e. rice husk. The firewood (rubber plant) used in the biochar preparation turned into charcoal. The charcoal was eventually used to produce the activated carbon. Classification of the experiment materials is given in (Table 3.1).

Table 3.1 Materials for research

Material	Quantity	Access point
Activated carbon	2 kg	FOHCREC
Biochar	2 kg	FOHCREC
Container for sampling waste water (4 L container)	5 pcs	ECOLAB
Waste water sampled	20 L	Korle-bu backyard gardens
Watering can for collecting waste water from source	1 piece	Korle-bu backyard gardens
Filter cloth	0.25 m <sup>2</sup>	ECOLAB
Catch can for collecting waste water filtrate (adsorbate)	15 pcs	ECOLAB

Note that each container for sampling waste water had a cork and measured four litres (4 L) in volume. Catch cans for collection of filtered waste water was obtained by cutting a one and half litres (1.5 L) Voltic natural mineral water bottle bisectionally. Fine woven cloth was used as filter.

Activated carbon and biochar prepared were used as adsorbent for adsorbing heavy metals in waste water sampled. Waste water was to be treated for irrigation purpose. Voltic Mineral water plastic bottles were used for packing adsorbent materials to form a filter bed. It also served as catch cans for collecting filtered waste water (Plate 3.3) after treatment.



Plate 3.3 Set-up for waste water treatment (filtration) using biochar and activated carbon.

Waste water source from open drains for vegetable irrigation on the study site (Plate 3.4) was sampled from Korle-Bu Teaching Hospital backyard gardens (Plate 3.5). Containers for sampling waste water, filter bed preparation and catch can used for collecting filtrate were all cleaned thoroughly with detergent and distilled water before and after use.



Plate 3.4 Open drain for sourcing waste water for irrigation.



Plate 3.5 Korle-Bu backyard gardens.

### 3.2.1 Waste water sampling

Waste water (grey water and black water) was obtained early in the morning when farmers were on site with their farm activities at 7:00 am on 17<sup>th</sup> March 2017, i.e. late dry season. Precautionary measures in handling experimental samples from the field to laboratory for analysis was strictly adhered to in order to avoid adulteration of the physicochemical properties of the sampled waste water. The following safety guidelines were observed to ensure accurate results;

Plastic containers for fetching waste water from the open drain on the farmers' field were thoroughly cleaned with detergent and rinsed with distilled water. Watering can used by farmers for on farm vegetable irrigation was used to fetch raw waste water from the dugout well recharged by open drain water source. All five plastic containers measuring 4 L each was then filled with waste water drawn with the watering can. At very cold temperatures e.g. freezing point or ice temperature, chemical reaction of elements are minimal and serves as a means of transporting aqueous solution such as waste water without chemical reaction of element content of the solution. To achieve transporting waste water that contained heavy metals from experimental site to laboratory without alteration, there was the need to prevent chemical reaction of the elements including heavy metals in the sampled waste water. A millilitre (1 mL) of Nitric acid ( $\text{HNO}_3$ ) was introduced into the sampled waste water as an alternative to cold transport of waste water without any chemical reaction of the constituents. The Nitric acid when introduced into the waste water will prevent chemical reaction of any cation content of the waste water. Hence heavy metal conservation in the waste water from sampling site to laboratory for analysis was achieved by introducing Nitric acid into the waste water. The former, cold transport of waste water involves the use of ice block to preserve the samples at cold temperature in a tight fitted container usually ice chest to prevent ionic reactions. Black polythene sheet was wrapped to cover totally each

container containing the sample waste water to prevent interaction with Ultraviolet (UV) Rays from the sun's radiations. Waste water in containers wrapped with the black polythene bags were quickly transported to ECOLAB. It was kept in a refrigerator at 5 °C for 4 days in the laboratory while biochar and activated carbon were under preparation.

### **3.2.2 Biochar preparation**

Rice husk biochar used for the study was prepared at high pyrolytic temperature, i.e. temperature above 500 °C using plant residue, rubber plant (*Ficus elastica*) as source of heat energy. Experimental biochar was prepared at the University of Ghana Forest and Horticultural Crops Research Centre (FOHCREC) in Kade following the steps below;

Dry biomass of rubber plant sourced from its pruned branches was setup on the floor as firewood. A galvanized barrel was cut open at both ends serving as a hollow tube. The barrel was perforated all around its circumference to create vents for heat exchange and partial oxygen circulation during burning. It was then erected to enclose the firewood on the floor (Plate 3.6). Firewood was lighted and allowed to burn for about ten minutes.

Dried rice husk was heaped around the mounted perforated galvanized barrel while the firewood burnt inside the barrel (Plate 3.7). Heat energy produced from burning firewood was conducted through the walls of the barrel to the heaped rice husk around the hollow perforated galvanized barrel. Heating process continued while the rice husk surrounding the dented galvanized barrel turned into charred material. After charring water was used to quench and cool the end product, i.e. rice husk biochar.



Plate 3.6 Fire wood enclosed in barrel.



Plate 3.7 Charring heaped rice husk around barrel.

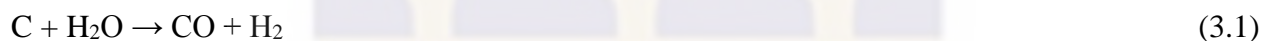
Charcoal produced as a result of using the rubber plant firewood in biochar preparation was used to prepare the activated carbon in a laboratory.

### 3.2.3 Activated carbon preparation

In order to prepare activated carbon, two process are involved namely carbonization and activation (Béuchel et al., 2000). Carbonization is the heat treatment or pyrolytic decomposition of rich-organic material such as wood or coal at higher temperature range of 400 °C to 600 °C in partial or absence of oxygen (Bansal and Goya, 2005). Activation on the other hand is the further application of high temperature of 700 °C and above to carbonized material to further displace the remaining carbon content of the material to enrich and enhance its porosity. Oxidative treatment is used to achieve this activation process (Bansal and Goya, 2005; Marsh and Rodriguez-Reinoso, 2006).

These steps were applied in activated carbon preparation under the study:

Charcoal produced from rubber plant residue served as carbonized material since the material (firewood) was burnt at a temperature higher than 500 °C under partial oxygen during the biochar production. Granular char produced was crushed into smaller particles sizes before activation. Activation involved placing granular char in furnace for further heat treatment. Thermal activation was done using furnace at a temperature of 800 °C in Ecological Laboratory of the Institute for Environment and Sanitation Studies of the University of Ghana within six hours. Carbon was partially combusted in activation process according to equations 3.1, 3.2 and 3.3 proposed by B uchel et al. (2000) referenced in  e en (2014).



Activation leads to extended and high surface area product that promote effective adsorption capacity of the activated carbon produced ( e en, 2014). Alternatively, chemical activation can be applied using  $\text{K}_2\text{CO}_3$  and  $\text{KOH}$  to form activated carbon (Tay et al., 2009).

Prepared activated carbon was pulverised in the laboratory and sieved to a particle size of 2 mm. Similarly, rice husk biochar was also sieved to a particle size of 2 mm. Biochar and activated carbon produced at the University of Ghana Forest and Horticultural Crops Research Centre and Ecological Laboratory of Institute for Environment and Sanitation Studies are showed in Plate 3.8 and Plate 3.9.



Plate 3.8 Sample rice husk biochar prepared.

Plate 3.9 Charcoal after biochar preparation.

### 3.3 Experimental material characteristics

The efficacy of experimental material in treating waste water depends on its physical and chemical properties among others. The adsorption potential of biochar and activated carbon depends on their surface area, pore space, carbon content, particle size, and chemical constituent. Total Dissolved Solids (TDS), Hydrogen Ion Concentration (pH), Electrical Conductivity (EC) and selected chemical elements including heavy metals composition of adsorbent (biochar and activated carbon) and waste water were measured.

The physical parameters, TDS, pH, EC and the selected heavy metals for the study were measured in waste water, biochar and the activated carbon. Heavy metals measured were Zinc (Zn), iron (Fe), Nickel (Ni), Chromium (Cr), Manganese (Mn) and Lead (Pb). Some of these elements that were detected in the waste water were also measured in the biochar and Granulated Activated Carbon (GAC). The reason for measuring was to ascertain their presence in the experimental

biochar and GAC. That was a guide serving as a deterrent on the use of an adsorbent i.e. biochar and GAC containing same elements as that in the waste water to be treated. As a precaution, it was not advisable to use biochar and GAC as an adsorbent to eliminate Zn in waste water when the biochar and GAC already contained Zn.

### **3.4 Determination of heavy metals in experimental material**

Ideally, one cannot pinpoint the exact heavy metals and their concentration levels in any media rather an assumption of the specific heavy metals presence are tested for certainty because heavy metals are ubiquitous. Six heavy metals including Cr, Zn, Pb, Ni, Mn, and Fe were initially tested in the experimental waste water, biochar and activated carbon and their concentrations noted before treatment was done.

#### **3.4.1 Determination of heavy metals in waste water**

Experimental waste water was tested for the presence of the selected heavy metals using the following procedure;

Waste water in corked bottle was shook thoroughly to unsettle any particles. A given volume, one litre was filtered and sampled into each of three different measuring cylinders serving as three replicates. Samples in measuring cylinders were analysed in the laboratory using Atomic Adsorption Spectrometer (AAS). All six heavy metals listed under the study were tested using the AAS at ECOLAB in all the three replicates and their presence and concentrations noted. The particular heavy metals detected in the waste water was proposed for decontamination using

biochar and activated carbon based on the principle that the biochar and activated carbon did not contain the particular heavy metals detected in the waste water.

### **3.4.2 Determination of heavy metals in biochar and activated carbon**

In order to use an adsorbent material like biochar and activated carbon to decontaminate a given heavy metal e.g. Lead (Pb) in an aqueous solution, it was necessary to ensure that the adsorbent material did not contain the given heavy metal to be decontaminated. Hence the prepared biochar and activated carbon were also initially tested for the presence and concentrations of the selected heavy metals in this study. This was achieved through digestion of the adsorbent materials. Digestion of experimental activated carbon and biochar was therefore carried out in ECOLAB to determine the presence and concentrations of the selected heavy metals following the procedure below;

Ten grams (10 g) each of biochar and activated carbon were sampled and air-dried for eight hours. After drying, the samples were sieved to 2 mm particle size. One gram (1 g) each of the activated carbon and biochar were weighed separately into digestion tubes. Ternary mixture made up of 3 ml of Perchloric acid ( $\text{HClO}_4$ ) and 4 ml Nitric acid ( $\text{HNO}_3$ ) were added to the 1 g of the activated carbon and biochar in a digestion tube. The contents were then placed and heated on a hot plate for about 10 minutes. It was cooled under room temperature and 5 mL distilled water was added into each digested sample. Each of the solutions were filtered into 100 ml volumetric flask. Distilled water was added to the 100 ml mark and corked. The content in the volumetric flask was thoroughly shaken to ensure evenly mixture of the concentrated solution. The aqueous solutions of digested activated carbon and biochar in the volumetric flask were then measured, filtered and analysed for heavy metals content using the Atomic Adsorption Spectrometer.

### 3.5 Experimental treatments

Two treatment levels for each of biochar and activated carbon were used in the study. Each level of the biochar and activated carbon treatments were combined with 1 L of untreated waste water in a filtration set up. Biochar treatments were 100 g and 200 g and the activated carbon treatments were 100 g and 200 g respectively.

First treatment combination consisted of 100 g biochar with 1 L of waste water. Second treatment combination was made up of 200 g of biochar and 1 L of waste water. The third treatment level was given as 100 g activated carbon combined with 1 L of waste water. Fourth treatment combination had 200 g activated carbon and 1 L of waste water. This was replicated three times and results tabulated. Filtrates from each of the four treatment levels were collected and analysed for concentrations of the given heavy metals. Results were then compared with the control to determine any difference in heavy metals concentrations. Treatment combination at the four different levels is summarised in Table 3.2.

Table 3.2 Treatment combinations

	Adsorbent type			
	Biochar		Activated Carbon	
Treatment amount (g)	100	200	100	200
Waste water amount (L)	1	1	1	1
Treatment level	1	2	3	4

Note that the table above represents one experimental set-up which was replicated three times.

### 3.6 Physicochemical properties of treatments

Temperature plays an important role in chemical reactions in aqueous solution. Some elements are susceptible to volatility at high temperatures. Temperature also influences the adsorption capacity of an adsorbent material herein given as rice husk biochar and activated carbon. Temperature of waste water and filtrate of adsorbents were measured alongside their chemical properties in the laboratory

Meanwhile irrigation water may not be the only source of crop contamination in terms of heavy metals. Growth media sometimes can possibly contribute to heavy metals contamination in irrigated agriculture. Physicochemical parameters determined were Hydrogen ion concentration (pH), Electrical Conductivity of water ( $EC_w$ ) and Total Dissolved Salts (TDS). EC and TDS are the two basic quality index for irrigation water assessment and are used to define salinity level of irrigation water. Electrical conductivity is influenced by the dissolved salts (TDS) in a solvent and hence EC is directly related to TDS. This relation still hold whether the solvent in question is an unadulterated water or waste water. Estimating TDS from  $EC_w$  is made with caution as the conversion factor depends not only on salinity level but composition of the water is also considered (Grattan, 2002). TDS was determined from  $EC_w$  using a relation given by Grattan (2002) with a conversion factor dependent on the level of  $EC_w$  as equation 3.4 and equation 3.5.

$$TDS = 640 * EC_w \quad \text{When } EC_w < 5 \text{ dS/m} \quad (3.4)$$

$$TDS = 800 * EC_w \quad \text{When } EC_w > 5 \text{ dS/m} \quad (3.5)$$

Where;

TDS is the Total Dissolved Salts (mg/L),

$EC_w$  is the Electrical Conductivity of water (dS/m),

640 and 800 are conversion factors based on  $EC_w < 5 \text{ dS/m}$  and  $EC_w > 5 \text{ dS/m}$ .

Values of  $EC_w$  and TDS determined were compared to United Nations Food and Agriculture Organisation (FAO) standards for irrigation water quality (Ayers and Westcot, 1985) (Table 3.3).

pH was similarly interpreted using the FAO guidelines in Ayers and Westcot (1985).

Table 3.3 Guidelines for interpretations of irrigation water quality (FAO)

Potential irrigation problem	Units	Degree of restriction on use		
		None	Slight to moderate	Severe
Salinity problem				
$EC_w$	dS/m	< 0.7	0.7 – 3.0	> 3.0
TDS	mg/L	< 450	450 - 2000	> 2000

Results obtained under this study were interpreted using the designations “None, Slight to moderate and Severe” as a degree of restriction on use for irrigation. It was based on the FAO guidelines for irrigation water quality interpretation in Ayers and Westcot (1985).

### 3.7 Heavy metal decontamination in waste water

Heavy metals that were present in the waste water but undetected in the biochar and activated carbon were certified and listed for decontamination. The certified heavy metals in waste water were then subjected to decontamination using biochar and activated carbon as adsorbents. Final concentrations of the given heavy metals were measured in the filtrates from the different amounts

of the biochar and activated carbon after treatment. Results were then compared to the untreated waste water serving as control.

Heavy metals decontamination using different amounts of biochar and activated carbon as a means of treating waste water was carried out in the laboratory (ECOLAB) following the outline below;

An amount of 100 g and 200 g each of activated carbon and biochar was measured and packed in a filtered and corked plastic bottles as (W). 1 litre volume of the waste water to be treated was measured (V). Measured sample waste water (control) whose initial concentration had been measured as ( $C_i$ ) was poured on the 100 g and 200 g of each adsorbent packed in the inverted plastic bottles respectively. The content was stored for infiltration and adsorption of heavy metals onto the surface of the adsorbents. A contact time of 24 hours was allowed to ensure optimum adsorption. The cork of the inverted plastic bottles (Volic mineral water bottle) were gently loosen to drain filtrates into catch cans after the 24 hours. Filtrate (treated waste water) in each catch can was sampled and tested for final concentrations of the heavy metals using AAS machine as ( $C_f$ ) (Appendix A, Figure A11).

The amount of heavy metals adsorbed by the activated carbon and biochar in each case, i.e. adsorption capacity,  $q_c$  (mg/g) was calculated using equation (3.6) (Abdel-Ghani et al., 2015).

$$q_c = \frac{C_i - C_f}{W} * V \quad (3.6)$$

Where;

$q_c$  is Amount of heavy metal adsorbed [mg/g],

$C_i$  is Concentration of trace element in waste water before treatment [mg/L],

$C_f$  is Concentration of trace element in waste water after treatment [mg/L],

W is Weight of adsorbent (activated carbon or biochar) [g],

V is Volume of waste water used [L].

Percentage heavy metals removed or decontaminated by adsorbent, R (%) was also calculated using equation (3.7) (Abdel-Ghani et al., 2015):

$$R = \frac{C_i - C_f}{C_i} * 100 \quad (3.7)$$

Where;

R is the percentage of heavy metal adsorbed (%),

$C_i$  is Concentration of trace element in waste water before treatment [mg/L],

$C_f$  is Concentration of trace element in waste water after treatment [mg/L].

### 3.8 Experimental design and data analysis

Four treatment combination levels for both biochar and activated carbon filtrates were replicated three times and one level of waste water serving as the control was also replicated three times. Fifteen (15) samples in all were analysed in a Complete Randomized Design (CRD) with three (3) replications. Data was processed with the help of Microsoft excel (2013 edition) and GenStat (GenStat 12<sup>th</sup> edition). Analysis of Variance (ANOVA) was done to determine any significant difference in heavy metal adsorption capacity of the different adsorbent amounts using Least Significance Difference (LSD) test.

## CHAPTER FOUR

### RESULTS

#### 4.1 Physicochemical properties of experimental materials

Biochar, activated carbon and waste water in general have individual principal characteristics in terms of physicochemical properties. Irrigation water, irrespective of the source contains some appreciable amounts of chemical elements alongside physical and biological constituents. Quality of irrigation water depends tremendously on these constituents but measured on standards. Thus irrigation water quality is characterized by its biological, physical and chemical constituents. The concentration or level of irrigation water contamination by these constituents are the indicators of health concern when used on food crops.

Waste water used for this study and the corresponding treated waste water using biochar and activated carbon at varying quantities were analysed. Temperature (T), Hydrogen ion concentration (pH), Electrical Conductivity ( $EC_w$ ) and Total Dissolved Solids (TDS) measured are given in table 4.1. Carbonized materials herein given as biochar and activated carbon contained high amount of potash ( $K_2O$ ), i.e. source of potassium (K) because it was prepared under high pyrolysis temperature. Huggins et al. (2016) also observed high concentrations of macronutrients (Ca, K, and Mg) and low concentrations of metals (Cr, Cd, As, Cu, Zn and Pb) in a similar study. This potassium (K) and the other elements such as Magnesium (Mg) from the Magnesium oxide ( $MgO$ ) content of the carbonized materials accounts for increase in TDS in aqueous solution.  $EC_w$  is directly proportional to TDS according to equation 3.4 hence the rise in  $EC_w$  with TDS in the biochar and activated carbon filtrates as well.

Table 4.1 Physical properties and Total Dissolved Solids content of treatments

Treatment	pH	EC <sub>w</sub> (dS/m)	TDS (mg/L)	T (°C)
WW (control)	8.14	1.14	729.6	29.8
100 A	8.10	1.24	793.6	29.4
100 B	8.06	2.38	1523.2	29.4
200 A	7.99	1.40	896.0	29.2
200 B	7.91	2.62	1678.8	29.6

Key: A is Activated carbon (g), B is Biochar (g) and WW is Waste water (L).

#### 4.2 Quality of irrigation water based on FAO standards

Electrical Conductivity of water and Total Dissolved Solids measured under this study was used to characterize quality of treated waste water for irrigation purpose using FAO standards as a guide. FAO irrigation water standards published by Ayers and Westcot (1985) provides the guideline for interpreting irrigation water quality based on the degree of restriction for use.

Results of EC and TDS obtained under this study were interpreted after comparison with FAO standards (Table 4.2). EC<sub>w</sub> and TDS values of all the treatments under this study are categorized as slight to moderate for use in irrigation based on FAO standards. Note that absolute figures of FAO standards for interpretation of result are given in Table 3.3.

Table 4.2 Comparison of current study results with FAO standard for irrigation water quality

Treatment	Current study EC <sub>w</sub> (dS/m)	Interpretation based on FAO standards	Current study TDS (mg/L)	Interpretation based on FAO standards
WW	1.14	Slight to moderate	729.6	Slight to moderate
100 A	1.24	Slight to moderate	793.6	Slight to moderate
100 B	2.38	Slight to moderate	1523.2	Slight to moderate
200 A	1.40	Slight to moderate	896.0	Slight to moderate
200 B	2.60	Slight to moderate	1678.8	Slight to moderate

Note that dS/m = mS/cm.

### 4.3 Heavy metals in experimental material

Levels of all six heavy metals namely Zn, Fe, Cr, Mn, Pb, and Ni were analysed in the waste water, biochar and activated carbon before treatment was carried out. Three out of the six heavy metals namely Zn, Mn and Cr were detected in the aqueous solutions of digested biochar and activated carbon. Results were tabulated for the different treatments of biochar and activated carbon used (Table 4.3). Meanwhile, these same heavy metals detected in the biochar and activated carbon were not detected in the waste water.

Table 4.3 Heavy metals levels in biochar and activated carbon treatments

Treatment	Mean concentration of heavy metal (mg/L)		
Concentration (g/L)	Zn	Mn	Cr
100 A	0.381	0.862	1.373
100 B	0.294	1.248	1.256
200 A	0.387	0.955	1.350
200 B	0.347	1.351	1.233

Note that 100 A means 100 g/L of Activated Carbon solution, 100 B means 100 g/L solution of Biochar and same definition for 200 A and 200 B respectively.

#### 4.4 Statistical analysis of heavy metal decontamination

Three heavy metals detected in the waste water and not in adsorbents were Fe, Ni and Pb. These heavy metals were adsorbed and decontaminated by the biochar and activated carbon after treatment. The amount decontaminated was analysed statistically to find any significant difference in adsorbate concentration after treated with the different quantities of adsorbents.

The three heavy metals decontaminated from the waste water were Iron (Fe), Nickel (Ni) and Lead (Pb). The various concentrations of the heavy metals after treatment with varying quantities of activated carbon and biochar were statistically analysed at 95 percent confidence level ( $P \leq 0.05$ ). Table 4.4 shows the result of adsorbent significant level in heavy metal decontamination from waste water. ANOVA tables for each heavy metal decontaminated are given in Appendix B.

Values recorded in the table are mean concentrations of the respective heavy metals after treatment.

Table 4.4 Amount and type of adsorbent effect on heavy metals decontamination

Treatment	Mean concentration of heavy metal (mg/L)		
Concentration (g/L)	Fe	Ni	Pb
0 WW	0.6310 b	0.6737 b	0.1990 a
100 A	0.0853 a	- 0.1447 a	0.1570 a
100 B	0.1693 a	0.3463 b	0.1280 a
200 A	0.0087 a	0.4740 b	0.0857 a
200 B	0.1643 a	0.6370 b	0.0307 a
<b>LSD<sub>0.05</sub></b>	<b>0.2135</b>	<b>0.4315</b>	<b>0.4170</b>

Note: 0 WW is waste water without any adsorbent, i.e. no Biochar/activated. A and B attached to the 100 and 200 under treatments concentration represent Activated carbon and Biochar. Typical range of adsorbate (Fe, Ni and Pb) mean concentration values attached with common alphabet in the same column shows no significant difference at  $P \leq 0.05$  LSD using Duncan's multiple range test. Likewise, concentration values attached with uncommon alphabets within the same column shows significant difference at  $P \leq 0.05$  LSD using Duncan's multiple range test.

#### 4.5 Heavy metal adsorption after waste water treatment

While the study aimed to treat waste water contaminated with heavy metals, results obtained after treatment indicated some level of heavy metal decontamination but not totally eliminated in most

treatments. These concentrations varied with varying amount of adsorbent type and amount used. Final concentration the heavy metals treated under the study were measured in all treatments i.e. 100 g and 200 g each of the adsorbents. Concentrations of the heavy metals treated using activated carbon and biochar were compared to the control treatment. A treatment that does effective decontamination are those whose heavy metal concentrations are lower than that of the waste water designated as the control in the corresponding graphs (Figures 4.1 to 4.3).

#### 4.5.1 Effect of adsorbent type and quantity on Fe decontamination

Treatment of waste water for Fe decontamination showed a positive trend with respect to the carbonaceous materials. It was observed that the concentration of Fe in the waste water (blue bar) after treatment with activated carbon and biochar generally reduced. Reduction in Fe concentration was also observed to occur in the activated carbon treatment more than the biochar at the various amounts (Figure 4.1).

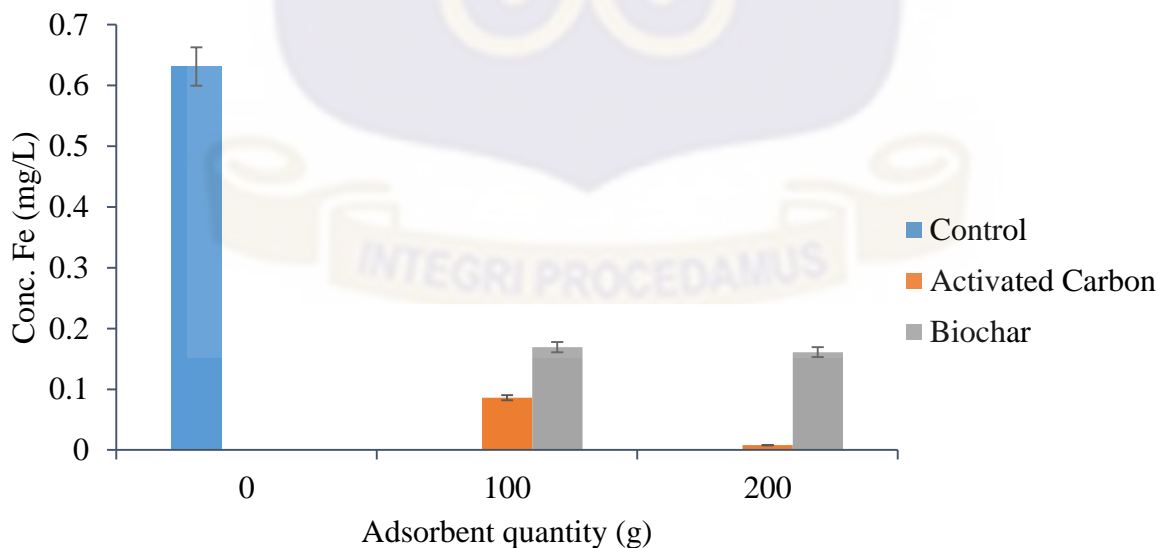


Figure 4.1 Concentration of Fe after waste water treatment.

#### 4.5.2 Effect of adsorbent type and quantity on Ni decontamination

Nickel was also decontaminated in the waste water. Its concentration in all the treated waste water reduced with respect to the control. The concentration of Ni after treatment with 100 g activated carbon was nil indicating its complete decontamination. On the other hand 200 g activated carbon reduced the concentration of Ni after treatment but not completely decontaminated (Figure 4.2).

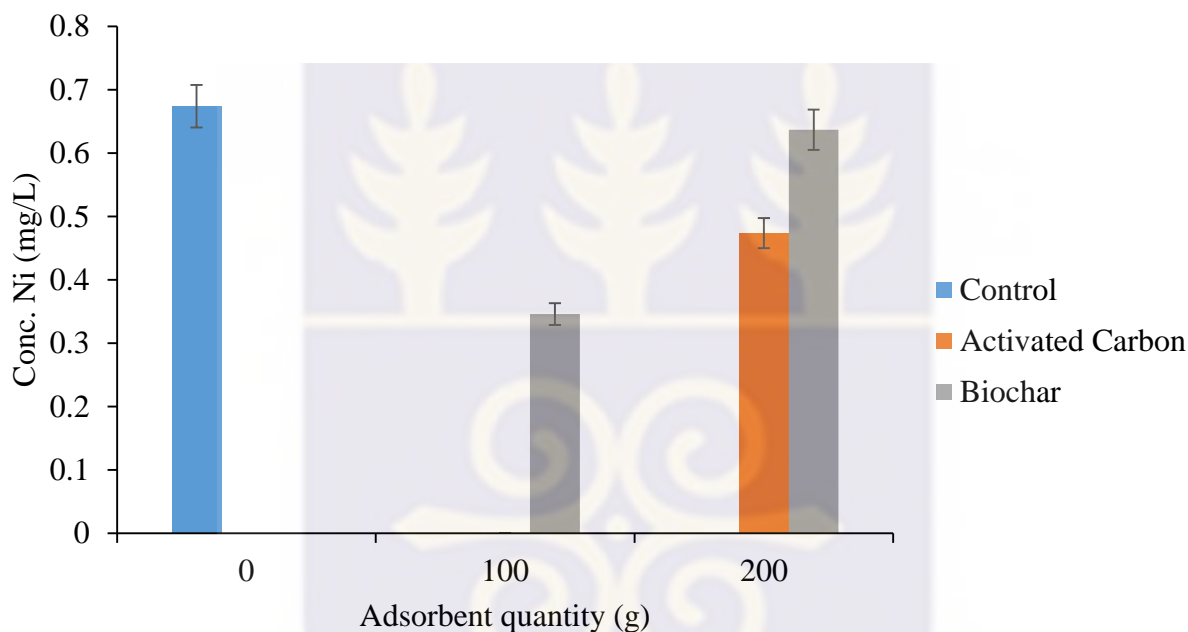


Figure 4.2 Concentration of Ni after waste water treatment.

#### 4.5.3 Effect of adsorbent type and quantity on Pb decontamination

Lead (Pb) concentration in the waste water was reduced from 0.199 mg/L to 0.031 mg/L in the 200 g biochar and 0.128 mg/L in the 100 g biochar. Similarly, it reduced from 0.199 mg/L in waste water to 0.157 mg/L in the 100 g activated carbon and 0.086 mg/L in the 200 g activated carbon respectively after treatment. In this case the biochar in general for the first time was observed to

perform better in decontaminating Pb than the activated carbon. The 200 g biochar produced the best decontamination (Figure 4.3). Biochar was more effective in Pb decontamination as compared to the activated carbon though it could not completely eliminate Pb from the waste water.

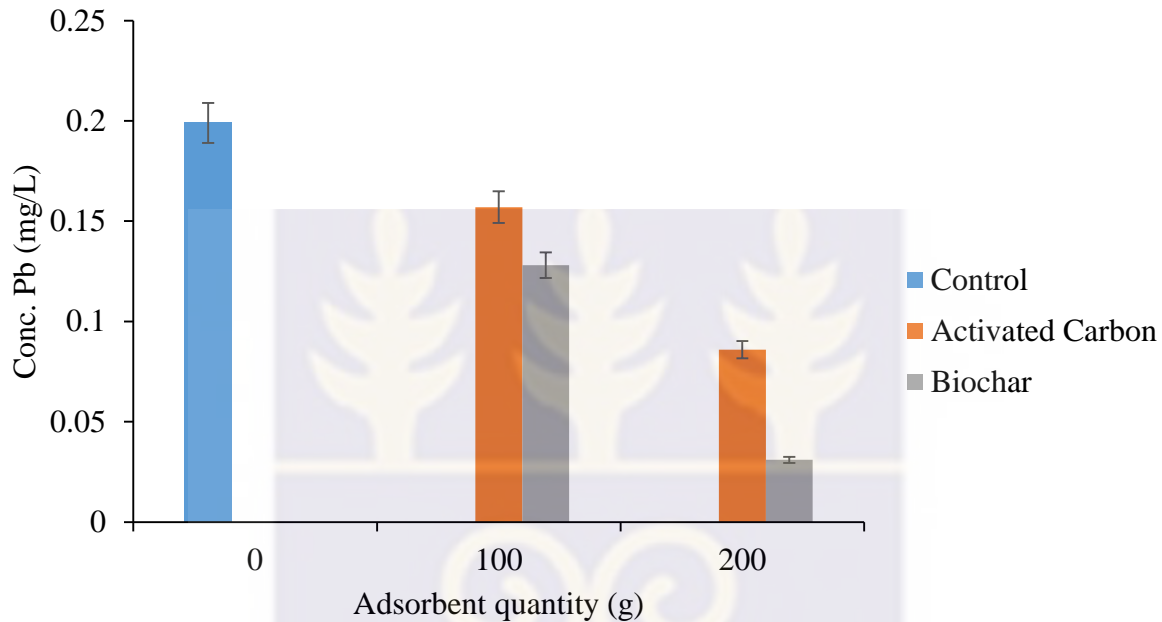


Figure 4.3 Concentration of Pb after waste water treatment.

#### 4.6 Adsorption capacity of biochar and activated carbon on heavy metals

Activated carbon and biochar adsorption of heavy metal and other organic substances have been reported in literature (Oleszczuk et al., 2012; Sud et al., 2008; Lemley et al., 1995). In this study, heavy metals in waste water were not completely decontaminated except Nickel in a 100 g activated carbon treatment. In general the adsorbent reduced the concentrations of the heavy metals after treatment based on the amount of adsorbent applied.

#### 4.6.1 Determination of adsorption isotherm

Amount of heavy metals adsorbed by the biochar and activated carbon in the various concentration after waste water treatment was calculated using equation 3.6 and mean concentration values of heavy metals from Table 4.4, page 41. The fraction of heavy metals adsorbed against the concentration of solution (adsorption isotherm) are given in Table 4.5.

Table 4.5 Amount of heavy metal decontaminated

Treatment Concentration (g/L)	Mean adsorbate, Q (mg/g)		
	Fe	Ni	Pb
100 A	0.00546	0.00674	0.00042
100 B	0.00462	0.00328	0.00071
200 A	0.00311	0.00100	0.00056
200 B	0.00233	0.00018	0.00084

Note that control treatment 0 WW served as reference for measuring change in heavy metal concentration in the biochar and activated carbon treatments at their respective levels. 100 A denotes concentration of adsorbent, activated carbon in this case at 100 g/L. 100 B denote 100 g/L of biochar serving as an adsorbent. Similarly, 200 A and 200 B denote concentration of Biochar respectively. Adsorption isotherm which is a plot of the amount of adsorbate against the concentration of biomass (adsorbent) are normally used to determine equilibrium concentration for optimum adsorption efficiency of an adsorbent.

#### 4.6.2 Percentage adsorption of heavy metals by Activated carbon and biochar

Percentages of heavy metals adsorbed (Table 4.6) were calculated using equation 3.7. Nickel was the only element that was completely decontaminated when 100 grams activated carbon was used. Percentage Fe decontaminated by the two different adsorbents was higher compared to the other heavy metals. In general, the percentage of Fe filtered by the adsorbents after waste water treatment was higher than the other heavy metals. The amount of Fe and Pb adsorbed by the adsorbents was directly proportional to the quantity of adsorbent used. Similar scenario of adsorption capacity of adsorbent was observed under Pb decontamination. This has also been observed by Wahi et al. (2009) when an adsorbent (activated carbon) produced from palm oil empty fruit bunch was used to remove Mercury, Lead and Copper from Aqueous solution. On the other hand Ni adsorption rate by the two different adsorbents was inversely proportional to the amount of adsorbent used.

Table 4.6 Typical ranges of Fe, Ni and Pb level decontaminated

Treatment	Percentage heavy metal adsorbed, R (%)		
	Fe	Ni	Pb
100 A	86.48	100	21.11
100 B	73.17	48.60	35.68
200 A	98.62	29.64	56.93
200 B	73.96	5.44	84.57

R is percentage heavy metal filtered by adsorbent from waste water.

Variation in the typical ranges of adsorbate were due the efficacy of the type and the quantity of adsorbent used in each case. In summary, result indicated higher percentage decontamination of

Fe in most of the adsorbent treatments but no 100 percent decontamination was observed. Instead a 100 % decontamination was achieved when Ni was totally adsorbed in a 100 g activated carbon.



## CHAPTER FIVE

### DISCUSSION

#### 5.1 Chemical properties of filtrate on irrigation standards

Irrigation water quality is measured by several factors including biological organisms and physicochemical constituents. Other water characteristics such as colour and odour are usually overlooked under irrigation perspective. Globally, fresh water is scarcely available for irrigation purposes, which has resulted in compromising the use of alternative water resource whose quality is guided by some standards in order to ameliorate crop contamination.

It could be seen from this study that pH of all the treatments was approximately 8 raising concern of alkalinity. pH ranged from 7.91 to 8.14 and are in the acceptable range for irrigation purpose (Duncan et al., 2009; Yemiyahu et al., 2009). According to Ayers and Westcot (1985), FAO standards for irrigation water quality with pH of 6.5 – 8.4 is a normal range and hence safe for irrigation purpose. Biochar and activated carbon prepared under high pyrolysis temperature contain potash in ample quantity which could contribute to alkalinity in aqueous solution. Both the biochar and activated carbon contributed to reducing the initial waste water pH but increased the  $EC_w$ .

Electrical conductivity is a measure of salt content of an aqueous solution or salinity. High EC values of irrigation water signifies high salt content and hence not favourable for irrigation. Electrical conductivity has a direct relationship with Total Dissolved Solids (TDS) given by equation 3.4 and equation 3.5 on page 33. EC and TDS also have influence on valence of heavy metals in aqueous solution. This can either decrease or increase the combining power of the heavy metal. The usual range in irrigation water for EC is 0 -3 dS/m and 0 – 2000 mg/L for TDS (Ayers

and Westcot (1985). This range of values satisfied study results where  $EC_w$  measured in untreated waste water and the treated waste water ranged from 1.14 – 2.62 dS/m.

TDS obtained after waste water treatment was in the range of 729.6 – 1678.8 mg/L. This means even the untreated waste water was safe for irrigation in relation to salinity problems. The high potassium content of the biochar and activated carbon prepared at high pyrolysis temperatures accounted for the increase in TDS and  $EC_w$  over the control. Based on FAO standards for irrigation water quality  $EC_w$  and TDS determined before and after waste water treatment in the study were in the acceptable range for use in irrigation. Study results for TDS and  $EC_w$  were therefore interpreted following FAO standards (Ayers and Westcot, 1985) as slight to moderate for irrigation use. While the interpretation of irrigation water quality is necessary, it is equally important to note that best results are dependent on the choice of use of the water and the type of crop to irrigate as different crops have different degree of tolerance for salinity.

Temperature also plays a key role in chemical reaction of elements. At higher temperature heavy metals tend to react effectively as compared to lower temperatures. All aqueous solutions from the treatment were at room temperature between 29 °C and 30 °C and that explains the low adsorption of the given heavy metals though others were significantly adsorbed after treatment.

## **5.2 Heavy metal decontamination by activated carbon and biochar**

Waste water treated with activated carbon and biochar did not produce desired results in all cases per the objective of the research. While some treatments significantly contributed in heavy metal decontamination, others were insignificant.

The first heavy metal determined in the waste water for treatment was Fe. Its mean concentration detected in the waste water before treatment was 0.6310 mg/L. Any adsorbent treatment that produced mean concentration less than the 0.6310 mg/L after filtration therefore showed a positive result and was said to have decreased the concentration of Fe in the waste water. Results after waste water treatment indicated that the concentration of Fe was reduced in all the treatments applied (100 g Activated carbon, 100 g Biochar, 200 g Activated carbon and 200 g Biochar). At  $P \leq 0.05$  through LSD test, there was significant difference in Fe decontamination between the control (untreated waste water) and all the other treatments made up of different quantities of biochar and activated carbon. There was no significant difference in Fe decontamination among the different adsorbent types used in different quantities statistically. Numerically, there were differences in Fe decontamination among the adsorbent types in varied quantities (Table 4.4, page 41). The most effective treatment that showed significant difference in treating waste water contaminated with Fe was the 200 g activated carbon followed by the 100 g activated carbon. This means the activated carbon in general produced the most desired result in terms of Fe decontamination in the waste water.

The second heavy metal tested and decontaminated in the waste water was Nickel (Ni). Its initial concentration in the waste water was 0.6737 mg/L. The waste water was subjected to treatment in the different adsorbents made up of activated carbon and biochar at 100 g and 200 g each. Numerically, Ni concentration decreased in all the treatments. There was a significant difference in Ni decontamination between the control (waste water) and the 100 g activated carbon treatment. There was also a significant difference between the 100 g activated carbon and all other adsorbent treatment levels under Ni decontamination. This was the only analysis in the experiment where an adsorbent completely decontaminated a heavy metal (Ni) in aqueous solution. Thus in the 100 g

activated carbon treatment, Ni was beyond traceable limits and was said to have completely decontaminated the heavy metal in the waste water. Wahi et al. (2009) reported similar results for 100 percent removal of Lead and Mercury from aqueous solution at low adsorbent dosage.

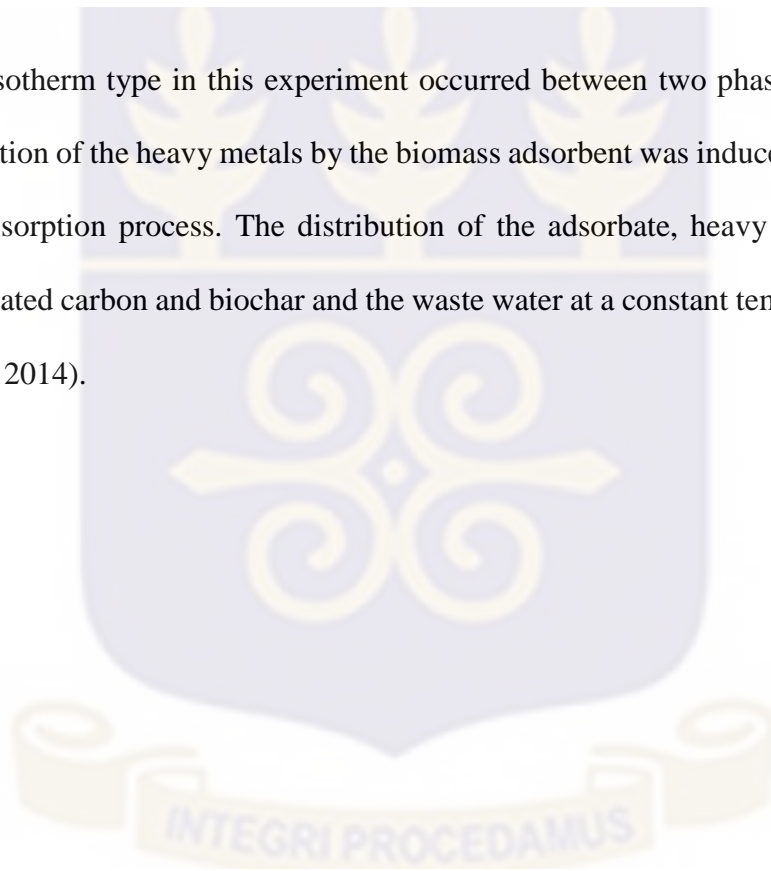
The final selected heavy metal decontaminated in the waste water was Lead (Pb). The concentration of Pb in the waste water analysed with the Atomic Adsorption Spectrometer was 0.199 mg/L. Filtrate from the biochar and activated carbon treatments also tested positive with varying concentrations of the heavy metal Pb. Initial concentration before treatment was 0.199 mg/L in the waste water. Difference in Pb concentration between the waste water and all other treatment was recorded and analysed statistically. There were no significant difference in Pb decontamination among the control and other treatments at  $P \leq 0.05$  through LSD test. Amount of Pb decontamination was directly proportional to adsorbent application rate. In other words, the higher the amount of adsorbent used, the higher the decontamination effect. Thus 200 g activated carbon and 200 g biochar performed better than the 100 g activated carbon and 100 g biochar in Pb decontamination although statistically insignificant.

### **5.3 Percentage concentration of heavy metal adsorbed during waste water treatment**

Table 4.5 and table 4.6 summarized the quantity and percentage of heavy metal decontamination in waste water. Suitable result in terms of heavy metal decontamination was recorded for Fe when higher percentage decontamination was observed under the majority adsorbent treatment. Percentage Fe eliminated in waste water ranged from 74 percent to 99 percent. It was only Ni which was 100 percent removed by activated carbon and was said to be totally eliminated or decontaminated from the waste water.

Biochar and activated carbon has been reported to retain nutrients (Foereid, 2015). These nutrients are classified into macro nutrients and micro nutrients which include some heavy metals. It has also been reported that the extent of nutrient retention is proportional to the amount of activated carbon and biochar used (Brantley et al., 2015). It was evident from this study that activated carbon and biochar retained the respective heavy metals. It was also observed at one instance under this research that the amount of heavy metal adsorbed was directly proportional to the amount of adsorbent used.

The adsorption isotherm type in this experiment occurred between two phase i.e. liquid – solid interface. Adsorption of the heavy metals by the biomass adsorbent was induced by van der Waals forces in a physisorption process. The distribution of the adsorbate, heavy metals in this case between the activated carbon and biochar and the waste water at a constant temperature defines an isotherm (Çeçen, 2014).



## CHAPTER SIX

### CONCLUSION AND RECOMMENDATIONS

#### 6.1 Conclusion

Waste water used for vegetable irrigation at Korle – Bu Teaching Hospital backyard garden was treated to decontaminate heavy metals using activated carbon and biochar. Two different quantities of activated carbon and biochar as adsorbents were applied in the waste water treatment.

Based on the research findings, the following conclusions were made in the study:

- Biochar and activated carbon contributed to treatment of waste water contaminated with heavy metals for vegetable irrigation at the study area. The extent of treatment varied with the type of heavy metal, adsorbent type and amount of adsorbent used.
- Heavy metal decontamination was directly proportional to the quantity of adsorbent used in most cases.
- The activated carbon produced more favourable results than biochar in terms of percentage heavy metals decontaminated.
- Conversion of the plant residue into activated carbon and biochar was viable as it reduces environmental pollution potential of the raw material if not well disposed.
- The technology applied under the study in treating waste water was simple, cost effective and viable.

## 6.2 Recommendations

From the study results and conclusions, the following recommendations were made:

- Activated carbon and biochar are recommended for treating heavy metal contaminated waste water used for irrigation.
- Based on treatment result from the individual adsorbents, it is recommended that further research should be carried out to compare the combine effect of activated carbon and biochar in decontaminating heavy metals in waste water.
- Rice husk biochar and activated carbon from rubber plant residue were used as adsorbents for waste water treatment hence it is recommended that different plant residue should be used to prepare activated carbon and biochar for similar study.
- 100 g and 200 g adsorbent quantities were used in current study and it is recommended that different amount of adsorbents (biochar and activated carbon) be used in a similar study to ascertain the effect of adsorbent quantity on absorbability.
- It is recommended that biochar and activated carbon be tested for the presence of the specific heavy metals to be decontaminated in the waste water before use. This will help prevent further contamination of waste water by the adsorbents as heavy metals are ubiquitous.
- Study on the kinetics of adsorption by the adsorbents (activated carbon and biochar) is also recommended for future experiments.

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## APPENDICES

### Appendix A Photographs showing irrigation waste water treatment experiment



Figure A1 Site for waste water irrigation (Korle-Bu Teaching Hospital backyard gardens).



Figure A2 Plastic containers (4 liters) filled with sample waste water from experimental site.



Figure A3 Dented hollow galvanized barrel. Figure A4 Firewood for biochar pyrolysis.



Figure A5 Biochar preparation (pyrolysis).



Figure A6 Biochar prepared.



Figure A7 Charcoal for activated carbon preparation.



Figure A8 Grinding machine used in reducing particle size of activated carbon.



Figure A9 Weighing biochar and activated carbon for waste water filtration experiment.



Figure A10 ECOLAB technician setting up Atomic Adsorption Spectrometer (AAS).



Figure A11 Determining heavy metal concentration in treatments using AAS machine.



**Appendix B ANOVA tables for heavy metal decontaminated after waste water treatment**

Table B1 ANOVA of Fe decontaminated after waste water treatment with different adsorbents

Source of variation	DoF	SS	MS	V.r	F. pr
Treatment	4	0.7111	0.1778	13.83	0.001
Replication stratum	2	0.0012	0.0006	0.05	
Error	8	0.1028	0.0129		
Total	14	0.8152			

DoF means Degree of freedom, SS is Sum of squares, MS is Mean Squares, V.r is Variance ratio and F pr. is Frequency probability.

Table B2 ANOVA of Ni decontaminated after waste water treatment with different adsorbents

Source of variation	DoF	SS	MS	V.r	F. pr
Treatment	4	1.5261	0.3815	7.27	0.009
Replication stratum	2	0.2779	0.1389	2.65	
Error	8	0.4201	0.0525		
Total	14	2.2241			

DoF means Degree of freedom, SS is Sum of squares, MS is Mean Squares, V.r is Variance ratio and F pr. is Frequency probability.

Table B3 ANOVA of Pb decontaminated after waste water treatment with different adsorbents

Source of variation	DoF	SS	MS	V.r	F. pr
Treatment	4	0.1042	0.0261	0.53	0.717
Replication stratum	2	0.2159	0.1080	2.20	
Error	8	0.3924	0.0490		
Total	14	0.7125			

DoF means Degree of freedom, SS is Sum of squares, MS is Mean Squares, V.r is Variance ratio and F pr. is Frequency probability.

