

**ADSORPTIVE ABILITY OF BANANA STALKS BIOCHAR IN
GREYWATER REMEDIATION FOR REUSE: A CASE OF
UNIVERSITY OF EMBU, KENYA**

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DECLARATION

This thesis is my original work and has not been presented elsewhere for a degree or any other award.

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DEDICATION

I dedicate this work to my dear parents Mr. John Ndung'u Mutaha and Mrs. Rachael Wairimu Ndung'u for being the strongest pillar since I started this journey. I will remain indebted to you for all the support spiritually and academically.

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LIST OF ABBREVIATIONS

ANOVA	Analysis of Variance
APHA	American Public Health Association
ASTM	American Society for Testing Materials
BOD	Biochemical Oxygen Demand
BSP	Banana Stalk Powder
CEC	Cation Exchange Capacity
COD	Chemical Oxygen Demand
EC	Electrical Conductivity
FAO	Food and Agricultural Organization of the United Nations
FTIR	Fourier Transform Infrared
GWFH	Greywater from Female Hostels
GWHH	Greywater from Male Hostels
LSD	Least Significant Difference
K ⁺	Potassium ions
Na ⁺	Sodium ions
PW	Potable Water
SAR	Sodium Adsorption Ratio
SDG	Sustainable Development Goals
TDS	Total Dissolved Solids
TGA	Thermogravimetric Analysis

ABSTRACT

The global demand for fresh water resources has been increasing over the years due to population growth, urbanization, industrialization, climate change and the global warming effect. The available water resources have reduced over time leaving a water demand deficit. Recycling and reuse of greywater has been touted as one of the strategies to augment the existing water resources. This study characterized the greywater from students' hostels and prepared biochar from banana stalks for use as an adsorbent in greywater remediation. The study found significant differences in greywater quality based on source, day and time of sampling. The biochar produced through pyrolysis at pyrolytic temperatures of 300 °C, 400 °C and 500 °C showed the presence of hydroxyl and carboxyl surface functional groups responsible for adsorption of target contaminants. The biochars achieved varying percentage reduction of the selected parameters with biochar prepared at temperature 500 °C recording a significantly higher ($p \leq 0.05$) percentage removal of Na^+ , K^+ and COD at 65.43%, 89.36% and 60.60% when compared to 58.30%, 56.96% and 49.51% for biochar 400 °C, and 41.13%, 40.36% and 38.31% for biochar 300 °C, respectively. Biochar produced at temperature 300 °C exhibited a significantly higher ($p \leq 0.05$) reduction of BOD in the greywater of 71.28% as compared to biochars 400 °C and 500 °C which had 66.64% and 61.92% percentage reduction, respectively. Biochar 500 °C was found most suitable for Na^+ , K^+ and COD reduction while Biochar 300 °C most suited the BOD reduction. The results from this study showed that biochar produced from banana stalks can be used as an adsorbent for greywater remediation and the treated water was suitable for consideration in agricultural reuse under the test conditions.

CHAPTER ONE

INTRODUCTION

1.1 Background

Water is an essential natural resource whose availability supports economic and societal activities while also contributing to sustaining the natural ecosystem (Idris, 2017; Hanasaki *et al.*, 2013). Water scarcity has over time grown to be a major constraint to the socio-economic development and a threat to livelihoods in most parts of the world (Liu *et al.*, 2016). The global risk report of the world economic forum identified water supply crisis as the top 1 high impact risk of the current times (World Economic Forum, 2015).

The World Water Development Report (2018) further noted that water demand increases at a rate of 1% per year over the past decades as a result of growth in population, changing consumption patterns, economic development, climate change, among other factors. With the population forecasted to rise from the current 7.3 billion to 8.5 billion by 2030 and to 9.7 billion by 2050, there will be anticipated high water demand against finite water resources (UN, 2015; Schewe *et al.*, 2014; Gupta *et al.*, 2012). This situation calls for innovative technologies to bridge the existing water supply gap and meet the industrial, domestic and agricultural water needs (Kaluli *et al.*, 2011).

Technologies such as seawater desalination, exploitation of more distant surface waters and deeper ground water sources, wastewater and greywater recycling and reuse, are being considered (Ghaitidak & Yadav, 2013; Friedler *et al.*, 2005). Greywater has been identified as an alternative water source especially for water scarce countries since it has a high potential for recycling and reuse (Tsoumachidou *et al.*, 2017). Greywater comprises about 75% of all domestic wastewater and it emanates from bath tubs, showers, clothes washers, hand wash sinks and kitchen sinks (Laghari *et al.*, 2015; Leal *et al.*, 2011).

Although greywater recycling and reusing is a viable option of increasing water availability, Golda *et al.* (2014) notes that in developing countries, including Kenya, greywater treatment and reuse are still at infancy stage. Where this water is recycled and reused especially in institutions which produce huge volumes of greywater, this can go along way into improving food security, health, environmental integrity and the

achievement of sustainable development goal number six (6) which focuses on providing safe and adequate water and sanitation to all by 2030 (SDG, 2015).

Various methods used in greywater treatment include: screening, grit removal, sedimentation, ion exchange, multimedia filtration, adsorption and ultra-filtration, among others (Boyjoo *et al.*, 2013; Ghunmi *et al.*, 2011). Most of these methods involve huge capital cost, are power consuming and require highly skilled labor and this makes them not feasible for implementation in low and middle-income countries (Allen *et al.*, 2010). Therefore, there is need for low cost technologies for wastewater treatment. These technologies involve use of low-cost adsorbents that has led to identification of biochar as an alternative adsorbent for wastewater treatment.

Biochar is a porous, carbon residue derived from thermal conversion of biomass under limited oxygen and anaerobic conditions (Inyang & Dickenson, 2015). The biomass for biochar production includes: barley straw, almond shell, cashew nut shell, fruit juice residue, mango peel waste, ground nut shell, rice shell, rice straw, sugarcane bagasse, tree barks, giant reeds, among others (De Gisi *et al.*, 2016).

Agricultural residue such as banana waste is one of the biomass categories that can be used for conversion to biochar through thermochemical processes (Abdullah *et al.*, 2013). Due to their high lignin content, banana wastes produce high carbon content when pyrolyzed at high temperatures and the high carbon content improves their adsorptive ability against cationic, anionic and neutral pollutants (Ahmad & Danish, 2018; Abdullah *et al.*, 2013). They are further advantageous as a result of being readily available, low cost and when carbonized to useful biochar, the environment is protected from methane and carbon dioxide gas which would be produced if the wastes were dumped in wetlands or burned (Ahmad & Danish, 2018).

Low *et al.* (1995) studied the removal of metal from electroplating waste and synthetic solution using banana pith biochar, Anwar *et al.* (2010) used banana peels biochar to remove lead and cadmium from water, Memon *et al.* (2009) studied banana peels for the selective removal of Chromium (VI) from industrial wastewater, banana pseudo stem was studied for the removal of colour and Chemical Oxygen Demand from landfill leachate (Ab Ghani *et al.*, 2017), banana trunk activated carbon was studied for remediation of

methylene blue contaminated water (Abdullah *et al.*, 2013), while banana peduncle biochar was studied for the removal of Chromium (VI) (Karim *et al.*, 2015). This study focused on preparation, characterization and use of banana stalks biochar as an adsorbent for remediating selected chemical and biological contaminants in greywater from students' hostels at the university of Embu.

1.2 Problem Statement

Global, regional and local increase in population, increased rate of urbanization and the adverse effects of climate change have culminated to high water demand against finite water resources. This has created pronounced water scarcity which has directly impacted on food security, sanitation and environmental management. Institutions of higher learning have high student population who produce huge amounts of greywater. This wastewater is lost to public sewer systems adding load to the already stressed centralized sewer systems. This water can be recycled and reused to help ease the water demand burden.

Wastewater treatment technologies have been developed to counter water scarcity challenges among them; developing adsorbents for removing soluble pollutants in contaminated water (Wong *et al.*, 2018). Most of these adsorbents are considered expensive while others are prepared from scarce materials and therefore are not readily available to sustain the demand. There is need therefore, to develop low cost adsorbents from readily available materials especially from biomass materials for wastewater treatment. This study focused on preparation of a low-cost biochar adsorbent from readily available banana stalks biomass for greywater remediation.

1.3 Justification

Water pollution is a serious challenge to availability of pristine water resources. This has therefore contributed to reduced available water for domestic, agricultural and industrial use even as the demand keeps increasing with time. There is need to increase available water while also improving environmental integrity. Greywater constitutes 75% of domestic wastewater and therefore provides a significant amount of water that can be recycled and reused for non-potable water uses. Institutions of higher learning have huge

numbers of students whose daily operations result in production of huge volumes of greywater. It is therefore important to develop ways to recycle greywater for reuse purposes. Most of the technologies devised to remediate wastewater are expensive to acquire, maintain and require skilled personnel. As such, the need to develop a low-cost adsorbent that can be locally prepared and used to remediate polluted water remains to be satisfied.

1.4 Hypotheses

This research hypothesized that:

- i. There is no significant difference in quality of potable water and greywater effluent from students' hostels.
- ii. There is no significant difference in characteristics of the biochar prepared at different pyrolytic temperatures.
- iii. There is no significant difference in percentage reduction of the selected biological and chemical greywater pollutants by biochars prepared at different pyrolytic temperatures.

1.5 Objectives

1.5.1 General objective

To characterize potable and greywater and study the adsorptive ability of banana stalks biochar in greywater remediation.

1.5.2 Specific objectives

The specific objectives of the study were to:

- i. Determine the quality characteristics of potable water and greywater from University of Embu students' hostels.
- ii. Determine the characteristics of banana stalks biochar prepared at different pyrolytic temperatures.
- iii. Determine the percentage reduction of selected biological and chemical greywater pollutants by biochars prepared at different pyrolytic temperatures.

CHAPTER TWO

LITERATURE REVIEW

2.1 Global Water Situation

Many parts of the world have been experiencing increased scarcity of freshwater resources due to inexorable rise in demand for production, industries and domestic water use in urban and rural populations (Hoekstra *et al.*, 2012). The World Water Development Report (2019) showed that water use increases worldwide by about 1% per year since 1980s as a result of the combined effect of growth in population, socio-economic growth and change in consumption patterns. The Global water demand will increase continually at a similar rate until 2050, accounting for a 20 to 30% increase above the current water use (World Water Development Report, 2019). Countries with developing and emerging economies have been touted to experience vast majority growth in water demand (World Water Development Report, 2018).

Kenya being a developing economy in Africa, the WASH joint program report found that only 59% of Kenyans have access to basic water services and only 29% have access to sanitary services (WHO & UNICEF, 2019). This water scarcity challenge necessitates the need for strategies to augment the available fresh water resources. As such, water scarce regions should seek to close the gap between water demand and supply by undertaking water management solutions such as: reduction in sectoral water abstraction, increasing reservoir storage capacity, reduction in pollution and engaging wastewater treatment, recycling and reuse (Van Vliet *et al.*, 2017; Wada *et al.*, 2014; Kaluli *et al.*, 2011). Tsoumachidou *et al.* (2017) indicates that due to the high potential for recycling and reuse of grey wastewater, it is a promising alternative to replenish the available water resources in water scarce countries.

2.2 Potable and Greywater Characteristics

Potable water is water that is considered to be safe enough for consumption by humans with minimal risk of short term or long-term harm. This water is considered to be colourless, practically tasteless, odourless, free from physical, biological or chemical

contaminants (Meybeck *et al.*, 1996). Typically, this water is used for drinking, cooking, washing and toilet flushing. Greywater is the wastewater that emanates from bath tubs, showers, clothes washers and kitchen sinks. This water contains detergents, soaps, fats, grease, oils, hair, nutrients, cleansers salts, fabric softeners and other chemicals such as chlorides, borax, sodium and sulfates at elevated level (Laghari *et al.*, 2015). According to Ghaitidak & Yadav (2013), greywater can be categorized into light and dark greywater. Light greywater is the greywater from bathrooms and wash basins while dark greywater comprises of the kitchen sinks and laundry greywater. About 75% of all domestic wastewater produced is greywater (Leal *et al.*, 2011).

The chemical characteristics of greywater can be classified as organic or inorganic. The inorganic constituents include nutrients, metals, non-metallic elements, dissolved matter and gases. Organic constituents of greywater include aggregate and individual constituents. The aggregate constituents are used in characterizing the bulk of organic matter in the wastewater and include parameters such as chemical oxygen demand (COD), biochemical oxygen demand (BOD) and total organic carbon (TOC) (Tsoumachidou *et al.*, 2017).

The chemical, physical and microbiological characteristics of greywater from a specific dwelling are quite inconsistent. They are dependent on the number of occupants, their age distribution, lifestyle, living standards, water usage patterns, their social and cultural habits, the type of soaps or detergents they use, the quantity of household chemicals used and the storage period of the greywater before being used or disposed (Ghaitidak & Yadav, 2013; Matos *et al.*, 2012). Greywater from bathrooms contain soaps, shampoos, body care products, toothpaste, shaving wastes, skin, hair, lint, body fats, traces of urine and faeces (Noah, 2002). Greywater from laundry contains high concentrations of chemicals from soap powders such as sodium, phosphorous, surfactants and nitrogen, bleaches, solvents, oils, non-degradable fibers from clothing and paints. This therefore creates the need to characterize greywater since the above factors may not necessarily be the same in all considerations.

2.2 Wastewater Treatment Systems

Depending on the types of contaminants to be removed, wastewater treatments are divided into various levels which are preliminary, primary, secondary, tertiary and advanced (Ghaitidak & Yadav 2013). Preliminary and primary treatment deals with the removal of rags, sticks, oil, grease settleable and suspended particles that may cause operational problems. Secondary treatment involves removal of biodegradable organic matter while tertiary treatment includes removal of nutrients and disinfection. Advanced treatment includes removal of dissolved and suspended materials remaining after applying earlier treatment (Metcalf & Eddy, 2003).

Wastewater treatment at the preliminary and primary level engages technologies such as screening, filtration, sedimentation, centrifugation, gravity, coagulation and floatation methods. Screening removes solids such as cloth, wood, paper, among others, while filtration removes suspended solids, grease, oil and some bacteria by use of a medium with fine pores. During filtration, turbidity and colloidal matter of non-settleable type protozoan cysts and helminths eggs are removed. The protozoa are stopped in gravels, the bacteria in the medium gravel and viruses by the sand due to their relative size to that of the soil aggregates (Hijnen *et al.*, 2004). Centrifugation removes colloidal solids while sedimentation and gravity allow the suspended solids to settle under un-disturbed or semi-disturbed condition, thus removing the solids, grit and silts. Floatation involves skimming of the suspended solids, oils, grease and biological solids when they adhere to gas or air in the floatation process (Cheremisinoff, 2001; Nemerow & Dasgupta, 1991); Tchobanoglous & Burton, 1991).

Secondary wastewater treatment involves the biological way of eliminating the insoluble and soluble pollutants by microbes using aerobic and anaerobic processes. In the aerobic process, organic matter is biodegraded through aerobic decomposition by the aerobic and facultative bacteria (Barragan *et al.*, 2007). In the anaerobic process, putrefaction occurs where the anaerobic and facultative bacteria convert complex organic matter into simpler organics based on nitrogen, carbon and Sulphur (Mohan *et al.*, 2007; Van der Zee & villaverde, 2005; Fux *et al.*, 2002). Aerobic treatment techniques include activated sludge process, aerated lagoons, waste stabilization pond, trickling filters, rotating biodiscs,

constructed wetland, among others. Anaerobic treatment techniques include contact beds, up flow anaerobic sludge blanket reactors, sludge digesters and anaerobic ponds (Arceivala & Asolekar 2006; Friedler *et al.*, 2005).

Tertiary wastewater treatment is used to obtain safe water for human use. Technologies in tertiary wastewater treatment include distillation, crystallization, solvent extraction, reverse osmosis, ion exchange, adsorption among others (Gupta *et al.*, 2012). Under distillation, water is heated up to 100 °C where the liquid vaporizes leaving pollutants behind, while in crystallization, the pollutants are removed by raising their concentrations to a point where they start to crystallize out. Solvent extraction engages the use of organic solvents such as benzene, hexane, acetone, among others, which are immiscible with water and have capacity to dissolve pollutants (Ahn & Ahn, 1997).

Reverse osmosis relies on the membrane development technology and involves use of pressure higher than osmotic pressure. In order to achieve filtration, various membranes are arranged in tubular, plate, disc, spiral and hollow fiber forms. Pollutants removal is determined by the solute's partition coefficient and the free energy of interaction between the membrane and the water (Tang *et al.*, 2006). Ion exchange technique involves the exchange of toxic with non-toxic ions in wastewater using an ion exchanger material. The material may be a cation or anion exchanger which have the capacity to exchange cations and anions respectively (Tarpeh *et al.*, 2017; Eom *et al.*, 2005; SenGupta, 1995).

The adsorption technique is a surface phenomenon based on the increase of particular components on a surface of a material or interphase between two phases. Common adsorbing materials include activated carbon, fly ashes, metal oxides, zeolites, biomass, among others (Gupta *et al.*, 2007; Gupta *et al.*, 2009). In the recent past, there has been growing interest in low-cost, efficient materials that can be used to remove organic and inorganic contaminants in wastewater (Braghiroli *et al.*, 2018). As such, the use of biochar for wastewater remediation has received much attention as a low-cost adsorbent and has been considered as a potential surrogate for activated carbon in wastewater remediation (Kearns *et al.*, 2014; Laird *et al.*, 2010). Biochar has shown great ability to immobilize organic and inorganic pollutants in soil and water systems (Inyang & Dickenson, 2015; Mohan *et al.*, 2014; Inyang *et al.*, 2011).

2.3 Pyrolysis Process

Pyrolysis is an inexpensive, robust technology that can result in thermochemical decomposition of organic materials into non-condensable syngas, condensable bio-oil and a residual solid co-product, biochar (Shaheen *et al.*, 2019; Ahmad *et al.*, 2014; Manya, 2012). The process of pyrolysis involves a thermochemical conversion technology that operates in the absence or limited oxygen and results in end products such as bio-oils, biochar and gases (Nanda *et al.*, 2016). Pyrolysis can be achieved through a number of modes available today which include slow pyrolysis, fast pyrolysis, flash pyrolysis, vacuum pyrolysis, intermediate pyrolysis and pressurized ultra-pyrolysis (Samanya *et al.*, 2012, Huber *et al.*, 2006). High yields of biofuels are obtained at the intermediate temperatures with faster heating rates and relatively short residence time. These are the characteristics and operating conditions for fast pyrolysis and flush pyrolysis.

On the other hand, the product biochar, is mostly obtained through a considerably favoured moderate temperature regime, slower rates of heating and a longer residence time which is representative of the slow pyrolysis and conventional pyrolysis (Nanda *et al.*, 2016). The slow pyrolysis operates at pyrolytic temperatures between 300 °C and 700 °C accompanied by a low heating rate of 0.1 °C/s and 1 °C/s, and a residence time of between 10 minutes and 100 minutes (Maschio *et al.*, 1992). During slow pyrolysis, an increase in peak temperature, which is the highest temperature reached during the process, may lead to an increase in content of carbon in biochars which is prominent in temperatures ranging between 400 °C and 500 °C. As such the biochar yields also decrease as the temperature rises during the pyrolysis process (Ahmad *et al.*, 2014; Manya, 2012).

Fast pyrolysis prefers pyrolytic temperatures of between 400 °C and 500 °C accompanied by a heating rate of 10 °C/s to 200 °C/s and a vapour residence time of 30 milliseconds to 150 milliseconds (Bridgwater, 1999). The process of flash pyrolysis of biomass and other materials is favoured at pyrolytic temperatures ranging from 400 °C to 600 °C with a high heating rate that is greater than 1000 °C/s and a vapour residence time lower than 100 milliseconds (Wagenaar *et al.*, 1993; Samolada & Vasalos, 1991). The bioconversion of materials by intermediate pyrolysis requires operating temperatures of around 500 °C with a moderate residence time of between 10 seconds to 20 seconds (Ahmad *et al.*, 2014). In

this intermediate pyrolysis the bio-yield is nearly 50% while biochar and gas yield are nearly 25% each.

Torrefaction is another pyrolysis process. This is a thermochemical treatment process which is carried out under atmospheric conditions, with an operating temperature ranging from 200 °C and 300 °C, under limited or no oxygen supply. This is also subjected to low heating rate and long residence time (Igalavithana *et al.*, 2017; Van Poucke *et al.*, 2016). Gasification is a promising technology for combining bio-energy production with resulting biochar (Hansen *et al.*, 2015). Gasification biochar generally may contain considerable amounts of minerals and recalcitrant carbon (You *et al.*, 2017; Wiedner *et al.*, 2013; Muller-Stover *et al.*, 2012). The gasification process is undertaken at much higher temperatures than pyrolysis and torrefaction. During the process, the energy in organic matter could be converted to combustible gases at temperatures ranging between 600 °C and 1000 °C producing biochar, water and condensable gas as minor products (Lee *et al.*, 2017; Oh *et al.*, 2017; You *et al.*, 2017).

Hydrothermal carbonization is an efficient method to produce biochar from biomass. In comparison to slow pyrolysis hydrothermal carbonization yields biochar with a higher amount of carbon and is enriched with O- containing functional groups and surface charge. It is further more acidic than biochar produced through slow pyrolysis (Tripathi *et al.*, 2016). However, a relatively higher energy is consumed in hydrothermal carbonization because it requires biomass with high moisture content (Garlapalli *et al.*, 2016; Malghani *et al.*, 2013).

2.4 Biochar Characterization

2.4.1 Spectroscopic characterization

Spectroscopic characterization of biochar, biomass and any other materials can be conducted using either of different spectroscopic techniques such as: Fourier Transform Infrared (FTIR) spectroscopy, X- ray Photoelectron Spectroscopy (XPS), synchrotron-based Near Edge X-ray Absorption Fine Structure (NEXAFS) spectroscopy or Raman spectroscopy (Shaheen *et al.*, 2019; Abdullah *et al.*, 2014). These technologies have been widely used to examine the surface chemistry of varying carbon materials including

biochar, and they provide qualitative information that can be used to show the functionalization and aging mechanisms of biochars (Shaheen *et al.*, 2019,)

Fourier Transform Infrared (FTIR) spectroscopy is a technique that has been widely used to examine the different surface functional groups on biomass and biochar. The FTIR spectra produced could be used to depict the possible changes in abundance of the surface functional moieties of biomass when compared with the product biochar (Zama *et al.*, 2017; Vaughn *et al.*, 2015; Ahmad *et al.*, 2012b). XPS as a spectroscopic technique has been used to delineate surface functional moieties, mineral elements and the sorption of pollutants/contaminants based on the elemental species bonding energies (Zama *et al.*, 2017; Singh *et al.*, 2014).

Near Edge X-ray Absorption Fine Structure Spectroscopy (NEXAFS) entails an element specific technique used to differentiate the highly variable and complex types of carbon material like the charcoal particles (Lehmann *et al.*, 2005). The NEXAFS spectroscopic technique is not quantitative in determining number of carbon bonds and it may be a complicated technique to be interpreted from a univocal solution of a mathematical approach. Therefore, the NEXAFS' spectrum features may vary according to the contour conditions such as the type of chosen reference material or even the data fitting software used (Lehmann *et al.*, 2005).

Raman Spectroscopic technique has also been used to evaluate the crystalline and the amorphous carbon structure in biochar material (Vithanage *et al.*, 2017; Parikh *et al.*, 2014; Mukome *et al.*, 2013; Wu *et al.*, 2009). However, the use of this technique in characterizing biochars and other adsorbents has relatively decreased possibly as a result of fluorescence that may arise during analysis by the polycyclic and graphillic aromatic compounds present in biochars and other sorbent materials (Chia *et al.*, 2012). As a result, the Raman spectroscopic technique is not a widely used technique.

2.4.2 Surface area

Surface area of biochar is a key factor in adsorption and it is directly proportional to adsorption (Pathak *et al.*, 2015.). The surface area of biochar material is a function of production feedstock and the operating conditions under which the biochar was produced,

principally, temperatures (Igalavithana *et al.*, 2017). The surface area can be determined using the Brunauer Emmett Teller (BET) method which calculates the biochar surface area by measuring the amount of N₂ (Nitrogen gas) adsorption on the biochar surface at low temperatures (77 K) (Igalavithana *et al.*, 2017; Brewer *et al.*, 2014; Keiluweit *et al.*, 2010). The carbon dioxide adsorption method has also been used to calculate the biochar surface area of biochar at relatively high temperatures (273 K) and it is shown to be more sensitive and it provides a more accurate biochar surface area measurements (Wang *et al.*, 2013; Kasozi *et al.*, 2010; Kwon *et al.*, 2005).

As increase in pyrolysis temperature has been evidenced to lead to an increase in the surface area of biochars. In a study done on biochar produced from the mulberry wood, it was noted that with the temperature increase from 350 °C to 550 °C, the resulting biochars increased in surface area from 16.5 m² g⁻¹ to 58 m² g⁻¹ (Zama *et al.*, 2017). Kim *et al.* (2012) also reported an increase in surface area of pine cone biochar from 2.9 m² g⁻¹ to 175 m² g⁻¹ as the pyrolysis temperature increased to above 300 °C. The findings by Zhang *et al.*, (2015) showed that the increasing pyrolysis temperatures led to decrease in cation exchange capacity of wood biochar which is explained by the decrease in volatile organic compounds and acid functional groups (Uchimiya *et al.*, 2013; Mukherjee *et al.*, 2011; Yuan *et al.*, 2011;). Variation in surface area as well as other physical properties is also affected by the difference in cellulose, hemicellulose and lignin content in biomass feedstock.

2.4.3 pH

The pH of biomass and biochar varies based on the source or type of feedstock. Pyrolysis is also a critical factor that affects the pH of biochar as evidenced by the results of pH 4.6 for biochars produced at temperatures below 200 °C, and 6.9 - 9.5 for biochars produced at temperatures of 400 °C to 600 °C, from oak wood (Zhang *et al.*, 2015). The increase of biochar pH with increasing temperature might be attributed the loss of volatile organic compounds and the increase in the basic cations in biochars produced at high pyrolytic temperatures (Shaheen *et al.*, 2019; Zhang *et al.*, 2015). In addition, the biochars produced at lower pyrolytic temperatures may have greater densities of acidic surface functional

groups such as carboxylic and phenolic groups, which may lead to decreasing biochar pH (Zhang *et al.*, 2015)

2.4.4 Thermogravimetric analysis

Thermogravimetric analysis of biomass is important while determining the potential of the biomass as pyrolysis feedstock and its thermal stability (Kabenge *et al.*, 2018). It is paramount in understanding the temperature regimes with the highest weight degradation of a material and thus the pyrolysis temperature required (Biswa *et al.*, 2016; Chaiwong *et al.*, 2013).

The biomass undergoes pyrolysis, the biomass experiences physical, chemical and molecular changes which in turn reduces the mass and volume of the material due to volatilization and shrinkages (Demessie *et al.*, 2015). Pyrolysis processes occur in three phases. The first phase involves feedstock/biomass dehydration which takes place in temperatures between 50 °C to 200 °C where sample moisture content is eliminated. The second phase entails devolatilization/thermal degradation of biomass where volatile matter including cellulose, hemicellulose and part of lignin are lost. This occurs at temperatures between 200 °C and 600 °C. The third phase involve the solid degradation of biomass and occurs between temperatures 600 °C and 900 °C (Biswa *et al.*, 2016; Chaiwong *et al.*, 2013; Sait *et al.*, 2012).

2.5 Use of Biochar in Water Treatment - Adsorption

Biochar is a porous, carbon residue derived from thermal conversion of biomass under limited oxygen and aerobic conditions (Inyang & Dickenson, 2015). Recently, biochar has been considered as a potential surrogate for activated carbon in environmental remediation and water treatment due to its low cost, relative abundance and comparative sorption abilities (Kearns *et al.*, 2014). Biochar has exhibited a greater ability and potential in remediation of contaminated waters than other adsorbents as it contains micro- and/or meso-porous structures, different surface functional groups which include carboxylic groups, hydroxyl groups, carbonyl groups, alcoholic and lactone groups, and inorganic mineral species (Rehrah *et al.*, 2016; Han *et al.*, 2013; Ronsse *et al.*, 2013). Activated or non-activated biochar or carbon is produced through thermo chemical processes which

include gasification, slow pyrolysis and fast pyrolysis. The physico-chemical properties of biochars produced from each of these processes can vary and are mostly influenced by process condition (reaction residence time and temperature); parent feed stock material and activation techniques (Inyang & Dickenson, 2015). Biochar can be produced through slow pyrolysis at moderate temperatures 350 °C – 800 °C and residence time ranging from minutes to days. Due to increased burn off of fixed carbon mass during pyrolysis, the surface area and pore volume of biochar generally increase with increasing temperatures and residence time (Zabaniotou *et al.*, 2008).

The surface area increases with increasing pyrolytic temperatures due to the escape of volatile substances such as cellulose and hemicelluloses and the formation of channel structure during pyrolysis (Kim *et al.*, 2013; Ahmad *et al.*, 2012a; Shen *et al.*, 2012). Decrease in pore size, internal pore structures formation and increased porosity due to the release of volatiles during carbonization could also be observed in biochars (Ahmad *et al.*, 2012a). Pyrolytic temperatures play a significant role in changing biochar characteristics. According to Kumar *et al.* (2011), biochar produced at high temperature show high pH, Cation Exchange Capacity (CEC) and surface area while biochar produced at low temperatures contain more active sites and exhibit stable carbon-oxygen complexes. Downie *et al.* (2009) further notes that at pyrolytic temperatures below 600 °C, biochar from slow pyrolysis may retain their parent feedstock chemistry, however as pyrolysis temperatures increase above 600 °C, aliphatic alcohol and acid functional groups can be converted to neutral or fused basic aromatic groups (Cheng *et al.*, 2008; Rutherford *et al.*, 2005).

Adsorption technique is a popular method in reducing the amounts of pollutants that enter water bodies and researchers are focusing on development of activated carbon from cheap sources to replace the costly commercial activated carbon (Wong *et al.*, 2018). Banana derived adsorbents have been studied for decontamination of heavy organic and inorganic pollutants considered soluble in water (Karim *et al.*, 2015). Banana fruit is one of the most popular and highly nutritional fruit crops cultivated in more than 130 countries and its total world production in 2016 was 144 million metric tons (Ahmad & Danish, 2018).

This avails a large volume of biowastes that can be utilized for development of useful biosorbents for treatment of wastewater.

Banana parts contain carbon rich organic compounds such as cellulose, hemicelluloses, pectin substances, chlorophyll pigments and lignin which can be carbonized to produce adsorbents for water soluble pollutants. Various parts of the banana plant have been studied as adsorbents against cationic, anionic and neutral pollutants (Ahmad & Danish, 2018; Abdullah *et al.*, 2013). This is due to their advantages which include being readily available, low cost and protecting the environment by preventing methane and carbon dioxide formation where the wastes are dumped in wetlands or burned (Ahmad & Danish, 2018).

Banana plant by-products have been studied for adsorption of pollutants in wastewater. Low *et al.* (1995) studied the removal of metal from electroplating waste and synthetic solution using banana pith biochar. The sorption was dependent on pH and concentration and this achieved maximum capacities of 8.55 and 13.36 mg/g for copper ions in electroplating waste and synthetic solution, respectively. Anwar *et al.* (2010) used banana peels biochar to remove lead and cadmium from water. The maximum amounts of lead and cadmium ions adsorbed were 5.71 and 2.18 mg/g of banana peel powder, respectively. Memon *et al.* (2009) further studied banana peels for the selective removal of Chromium (VI) from industrial wastewater. Banana pseudo stem was studied for the removal of color and chemical oxygen demand (COD) from landfill leachate. It achieved a color removal of 91.2% and COD removal of 83% (Ab Ghani *et al.*, 2017). Banana peduncle biochar at different pyrolytic temperatures was also studied for the removal of Chromium (VI). A mono layer adsorption capacity of 114 and 49 mg/g at 300 °C and 500 °C, respectively, was achieved (Karim *et al.*, 2015). This therefore indicates that banana plant parts have an adsorptive capacity for pollutants dissolved in water. The potential of banana stalks biochar in decontaminating greywater has not been studied.

2.6 Mechanisms of Pollutants Removal

2.6.1 Removal of inorganic pollutants in biochar

The potential of biochar adsorption mechanism for inorganic pollutants involves the combined effort of the various types of interaction which may include: surface sorption, complexation, electrostatic interaction, ion exchange and precipitation (Abbas *et al.*, 2018).

Surface sorption entails the formation of chemical bonds produced as a result of the diffusion of metal ions into the sorbent material's pores (Patra *et al.*, 2017). Elevated temperatures during the pyrolysis process always favours the surface area and pore volume in biochar. The ion exchange mechanism of adsorption of inorganic pollutants involves the exchange of cations and protons on biochar's surface with dissolved metals. This process is highly influenced by the functional groups on the surface of the biochar as well as the metal contaminant size. This ion exchange of metals occurs with the replacement of the positively charged ions on the surface of the biochar with the target metals. Bond characteristics, ionic radii and charge difference is a very important consideration in the ion exchange process (Krauskopf & Bird, 1967).

The electrostatic interaction mechanism is greatly dependent on the point of zero charge (Pzc) of biochar and the solution's pH. The charged metal ions and biochar have electrostatic interaction between them that in turn restricts the mobilization of heavy metals (Dong *et al.*, 2011; Mukherjee *et al.*, 2011). The precipitation mechanism of adsorption of inorganic pollutants is considered a major mechanism that is involved in the immobilization of inorganic contaminants interacting with the biochar. This process entails the formation of solids on either the surface or solution during the sorption process (Abbas *et al.*, 2018). The metal complexation mechanism entails multiatom arrangement where complexes interact with metal ligand. At low temperatures, functional groups of oxygen show high binding efficiency with heavy metals (Liu & Zhang, 2009; Mohan *et al.*, 2007). As established over a period of time, carboxyl group formation resulting from biochar surface oxidation increases the content of oxygen and thus the metal complexation increases respectively over the time (Harvey *et al.*, 2011).

2.6.2 Removal mechanism of organic pollutants in biochars

The mechanisms involved in the adsorption of organic pollutants on biochar is also a combination of different interactions. These interactions include: partitioning, pore filling, hydrophobic interaction, Electron Donor and Acceptor (EDA) interaction and electrostatic interaction. These interactions could be the major mechanisms involved in the adsorption of different organic pollutants onto biochar materials (Abbas *et al.*, 2018).

In the partitioning mechanism, the sorption of organic pollutants depends greatly on the characteristics of the carbonized and noncarbonized fractions of the biochar. This process starts with either diffusion of the sorbates into the pores of the sorbent or into the organic matter of the noncarbonized portion of biochar (Kasozi *et al.*, 2010; Chen *et al.*, 2008). As the process of partition continues, later on, the organic compounds solubilize within the matrix of the organic matter in order to enhance their sorption. Partitioning of organic compounds occurs on the amorphous phase of the carbon which contains the polyaromatic and aliphatic compounds such as sugars, ketones, phenols, among others (Keiluweit *et al.*, 2010).

The pore filling mechanism of organic pollutants adsorption is dependent on the presence of micropores (< 2 nm) and the mesopores (2-50 nm) on the surface of biochar (Hao *et al.*, 2013; Nguyen *et al.*, 2007). Based on the nature and type of the biochar, the pore filling adsorption mechanism may facilitate the vast sorption of both polar and the nonpolar organic contaminants. The electrostatic interaction (EI) adsorption mechanism is a critical mechanism in the sorption of ionic and ionizable organic compounds (Zheng *et al.*, 2013). The anionic sorbates always tend to bind with the positively charged biochar surface due to the alternate charges. The ionic strength and pH usually determine the fate of attractive and repulsive electrostatic forces in the process of sorption of the organic pollutants (Ahmad *et al.*, 2014). The pH of the solution controls the net charge on biochar surface and where the surface has a positive charge, mostly at low pH, the surface of the biochar grasps a net negative charge and vice versa (Mukherjee *et al.*, 2011).

Electron donor and acceptor (EDA) interaction mechanism normally occurs for the sorption of the aromatic compounds aided by the graphene-like surface of biochar (Abbas *et al.*, 2014; Spokas, 2010). At lower temperatures of less than 500 °C, the π aromatic

biochar system behaves as an electron acceptor, while at higher temperatures above 500 °C, the biochar acts like a donor through binding the retreating electron molecules (Zheng *et al.*, 2013; Sun *et al.*, 2012). Hydrophobic interaction mechanism involves both partitioning and hydrophobic mechanism in organic pollutants' adsorption. When compared to partitioning, hydrophobic interaction mechanism requires low hydration energy. This process is attributed in the sorption of both hydrophobic and neutral organic compounds by employing partitioning mechanism and hydrophobic interaction mechanism. (Zhu *et al.*, 2005; Murphy *et al.*, 1994)

CHAPTER THREE

MATERIALS AND METHODS

3.1 Study Site

This study was conducted in the University of Embu, Kenya. The university is located in the eastern region of the country, in Embu County at coordinates; 0.5156° S, 37.4560° E.

3.2 Sources of Water

The water considered for this study was: potable water (PW), greywater from male students' hostels (GWMH) and greywater from female students' hostels (GWFH) at the University of Embu. The male hostels had 11 rooms each accommodating 6 students. These were served by external showers and wash troughs. The female hostels had 28 rooms each accommodating 4 students. Female rooms had internal showers and wash basins.

3.3 Day and Time of Water Sampling

Water samples were collected and replicated in four days over a period of two weeks. In reference to semester dates and teaching timetable, the sampling times selected were morning and evening. These were the peak hours when most of the students would be in the halls of residence and engaging in activities that would lead to generation of greywater such as bathing, cleaning, laundry, among others. The peak time selected was between 6.00 – 7.30 am, when students were preparing for lectures and the day's activity, and 6.30 – 8.00 pm after lectures and the day's activities.

3.4 Collection of Water Samples

Potable water was collected from the main supply channels just before entry into the hostels. Three samples of 1000 mL were collected using 1000 mL glass beakers at intervals of 20 minutes during each sampling period. This was then mixed in a 5 liters water trough. The sampling was replicated in four days of study and twice every day, in the morning and evening.

Greywater samples from male and female hostels were also collected for the four days of study in the morning evening. The institution, University of Embu, has separate sewer system for grey and dark wastewater and it was therefore possible to collect the grey wastewater from the hostels' main effluent pipes. Three 20-liters samples were collected using 20-liters buckets, after every 20 minutes during the sampling period (morning and evening peak hours) and mixed in a 100-liters plastic container to make a composite.

3.5 Sample Preparation for Analysis

Three 100 mL samples were transferred into three 250 mL beakers for on-site determination of EC, TDS and pH. Determination of Sodium (Na^+) and Potassium (K^+) was conducted in the laboratory. For each water source, three 100 mL samples were taken from the composites of potable or greywater collected, and transferred into 100 mL plastic bottles. Samples for Na^+ analyses were refrigerated at a temperature of 4 °C, while samples for K^+ analyses were lowered to a pH below 2 using dilute Nitric acid and then refrigerated at temperatures of 4 °C. The analyses for the samples were carried out as outlined in subsection 3.12.

3.6 Banana Stalk Sample Collection and Preparation

Banana stalk biomass was collected from a banana plantation in Mitunguu-Meru, Kenya, located at geographical coordinates 0.1089° S and 37.7849 °E. The biomass was selectively collected from a single banana species, *Mussa acuminata*, within the plantation. The stalks were separated from the sheath and leaf parts of the banana plant. The sampled biomass was washed with distilled water to remove dirt on their surfaces. They were then sun dried to remove the water on the surfaces. The stalks were then sliced into small pieces (<2 cm) to increase the surface area for drying as well as to fit in crucibles during pyrolysis (Figure 3.1).

3.7 Determination of Banana Stalks' Moisture Content

Four samples of the banana stalks were taken from the whole sample and packed in khaki bags. These were weighed and the initial mass recorded before being introduced in the oven where the mass loss was monitored to constant mass. The moisture content was

determined using standard method for moisture analysis, method ASTM E871-82 (ASTM, 1998) using a conventional oven (Model: DHG-9053, China).

3.8 Pyrolysis of Banana Stalks

The dried banana stalks material from section 3.6 was packed into three crucibles each holding a mass of 35 g for pyrolysis using a muffle furnace (Model LH 15/14, Nabertherm, Germany). The muffle furnace was programmed for a temperature of either 300 °C, 400 °C or 500 °C and the temperature rate of rising was 10 °C/min. The holding temperature for the different pyrolysis temperatures was one hour and after the holding time, the furnace was programmed to cool down to room temperature. After the equipment was fully programmed, the crucibles containing the samples were introduced into the furnace for the respective pyrolysis temperature. The samples were removed from the furnace later after pyrolysis and cooling of the muffle furnace to room temperature. The yield collected after each pyrolysis process was determined. This was then packed into air-tight plastic ziplock bags for use in the activation process and later in adsorption experiment. This process was replicated seven times for biochar production at each pyrolytic temperature.

3.9 Activation of Banana Stalk Biochar

The biochars produced at temperatures 300 °C, 400 °C and 500 °C and packed in air-tight plastic bags were subjected to activation independently. The biochar was ground and graded using a mechanical shaker (Model JS14S, China) and the particle size retained by sieve with apertures of 1.18 mm (Figure 3.1) selected for activation. The material was impregnated with 1% phosphoric acid as the activating agent. The contact time of the biochar and the activating agent was five hours after which the material was rinsed with distilled water to a neutral pH equivalent to that of the distilled water. After achieving the required pH, the activated biochar was then dried in an oven (Model: DHG-9053, China) at 60 °C for 24 hours to remove the moisture content (Xu *et al.*, 2018). The samples were then packed in air-tight containers for use in the adsorption experiments within two days after preparation.



3.10 Characterization

3.10.1 Banana stalks characterization

The raw banana stalk powder (BSP) was characterized for FTIR spectroscopy (section 3.10.2.3) and thermal degradation to a maximum temperature of 500 °C in this study (section 3.10.1.1).

3.10.1.1 Thermogravimetric analysis (TGA) of the banana stalks powder

Thermogravimetric analyzer (model TGA-50 Shimadzu, Japan) was used in determining the thermal decomposition of the banana stalk material. A sample of 10 g of the dried banana stalks from section 3.6, was ground using a high-speed universal disintegrator (model FW80-I, China). From this, a sample of 1.864 g was placed into the aluminum sample holder. This was then slowly lowered into the instrument's furnace. The inert gas, Nitrogen, was introduced at a rate of 50 ml/min and heating commenced from a temperature of 24 °C (room temperature) to 500 °C. The weight loss was monitored using TGA-50 detector and recorded by the sensitive micro balance of the TGA. This was done according to ASTM method E1641

3.10.2 Biochar characterization

The biochars prepared at pyrolytic temperatures of 300 °C, 400 °C and 500 °C were characterized for Fourier Transform Infrared (FTIR) spectroscopy, yield and pH.

3.10.2.1 Determination biochar yield

The yield of biochars prepared at the different pyrolytic temperatures were determined. This involved comparing the initial recorded mass of the dry banana stalks before pyrolysis in the muffle furnace and the final output from the furnace after pyrolysis. The following formula was used in calculating the percentage yield at each pyrolytic temperature as outlined by Karim *et al.* (2015):

$$\% Yield = \frac{Ma}{Mb} * 100$$

Where, Ma was the mass of the output (biochar) after pyrolysis, and

Mb was mass of the dried banana stalks before pyrolysis

3.10.2.2 Determination of biochars' pH

The pH of biochars 300 °C, 400 °C or 500 °C was determined by placing one gram of the non-activated biochar in a 100 mL beaker and adding 20 mL of distilled water. The solution was stirred for 10 minutes and allowed to stand before the reading was taken following the method by Karim *et al.* (2015) using a pH meter (model HI 98128 HANNA, Romania). This test was conducted in triplicates and an average of the readings recorded.

3.10.2.3 Fourier transform infrared spectroscopy (FTIR) analysis

Analysis for the functional groups present in the banana stalk material (BSP) and biochars was conducted using an FTIR machine (Model Jasco FT/IR-4700, Japan). The analysis was done in two phases. Phase one included the FTIR analysis of the banana stalks powder (BSP) material and the activated biochars 300 °C, 400 °C and 500 °C. This phase was used in monitoring the changes in functional groups as a result of pyrolysis at the different pyrolytic temperatures used in the study. Phase two of the FTIR analysis entailed the analysis of activated biochars 300 °C, 400 °C and 500 °C after adsorption of pollutants in greywater used in the study. This phase showed the changes in abundance and presence of functional groups on the biochar material as a result of adsorption.

Analysis for the functional groups present on the samples was conducted using an FTIR machine (Model Jasco FT/IR-4700, Japan) in attenuated total reflectance (ATR) mode. The scans were conducted from a wavelength of 4000 cm^{-1} to 500 cm^{-1} at a scan rate of 50 conducted at a resolution of 4 cm^{-1} . The infra-red (IR) spectra for each sample was obtained. The samples were analyzed in triplicates.

3.11 Adsorption Study.

The adsorption ability of the banana stalks biochar was evaluated for removal of the selected biological and chemical pollutants. This process involved packing the respective biochars into glass columns and passing contaminated greywater through the biochar. A biochar particle of size retained by sieve with apertures of 1.18 mm was chosen for this study and a glass column of diameter of 120 mm chosen. The ratio of the column diameter

to biochar particle diameter in this study was above the recommended minimum ration of 20:1 column diameter to particle diameter ratio (Arbuckle & Yen-Hu, 1990). An equal biochar mass (8 g) was considered for biochars 300 °C, 400 °C and 500 °C. The activated biochar was weighed using the analytical balance and then packed into the glass columns (Figure 3.2). The columns were packed in triplicates for each pyrolytic temperature. After packing, greywater was introduced into each of the columns progressively, for adsorption of the target pollutants at a constant flow rate of 0.3 l/h. The filtrate from the column was tested for the target pollutants in order to establish the percentage removal after adsorption. These tests were done in triplicates.



Figure 3.2: Illustration of the columns in which biochar was packed

To determine the percentage removal of the selected greywater pollutant, the influent water to the system was characterized and the final effluent from the columns characterized as described in section 3.12. The percentage removal was calculated as:

$$\% \text{ Removal} = \frac{IC - FC}{IC} * 100$$

Where, IC is the initial concentration of the greywater characteristic

FC is the final concentration of the greywater characteristic

3.12 Analysis of Water Samples

The physicochemical characteristics of the sampled water was conducted in accordance with the standard methods for the examination of water and wastewaters (APHA, 2005). The characterization was done in two phases. Phase one involved the characterization of potable water (PW), greywater from the male students' hostels (GWMH) and greywater from female students' hostels (GWFH). In this phase, physical and chemical water characteristics were considered and these included the concentration levels of sodium ions (Na^+), potassium ions (K^+), electrical conductivity (EC), total dissolved solids (TDS) and pH. This phase compared the variation in quality for the three water sources and the variation in quality for the different sampling days and time.

Phase two involved testing the performance of the biochar systems in reducing the levels of the selected biological and chemical greywater pollutants. The selected biological parameters were biochemical oxygen demand (BOD) and chemical oxygen demand (COD) while the chemical parameters considered were the levels of Na^+ and K^+ ions concentrations. The levels were tested before introduction into the biochar containing columns and after passing through the column. The samples were analyzed in triplicates and the parameters were determined as follows:

3.12.1 Determination of electrical conductivity (EC)

The Electrical Conductivity (EC) was determined using a conductivity meter (ECscan 40 conductivity tester Bante Instruments, China). This was calibrated using the manufacturers calibration standards. These tests were done on-site during the sampling times according to method 2510B (APHA, 2005).

3.12.2 Determination of total dissolved solids (TDS)

The total dissolved solids were determined using the total dissolved solids meter (ECscan 40 TDS tester Bante Instruments, China). The tests were done onsite and the readings were taken in triplicates.

3.12.3 Determination of pH

The pH was determined using a pH meter (model HI 98128 HANNA, Romania). The meter was calibrated using the respective calibration buffer standards. For every measurement done, the meter was re-calibrated before reading the next sample. This was done in triplicates according to method 4500 - H⁺ B of APHA (2005).

3.12.4 Determination of sodium (Na⁺) and potassium ions (K⁺)

The standard calibration curves for Na⁺ and K⁺ determinations were prepared using analytical grade sodium and potassium salts. The calibration standards ranged from 0 ppm to 100 ppm. The greywater samples were filtered using a filter paper (Whatman No. 1) to remove burner-clogging particulate matter and then diluted using distilled water to a dilution factor of 10 due to the anticipated high concentrations of Na⁺ and K⁺ ions. The potable water used as a control was not subjected to dilution. The concentrations were then determined using a flame photometer (model FP640, Unimedsume Company, UK) in triplicates. The standard methods section 3500-Na B and 3500-K B were used for Na⁺ and K⁺ determination, respectively (APHA, 2005).

3.12.5 Determination of biochemical oxygen demand (BOD₅) and chemical oxygen demand (COD)

Determination of BOD₅ and COD of untreated and treated greywater samples was conducted according to methods 5210B and 5220C of the APHA standard methods for determination of water and wastewaters (APHA, 2005).

3.13 Data Collection, Analysis and Presentation

The physicochemical characterization data for the different treatments, the source of water, was collected and organized in MS Excel spreadsheet. The data was then analyzed for Analysis of Variance (ANOVA) using the Statistical Analysis Software (SAS) version 8. The means were separated using the Tukey's studentized range (HSD) at p= 0.05. The quantitative data collected for objectives two and three where the treatments were the biochars prepared at different pyrolytic temperatures, was organized in the MS excel spreadsheets. Statistical analysis was conducted using the Statistical Analysis Software

(SAS) version 8 and means separated using the Tukey's studentized range (HSD) at $p=0.05$. The results for the different analyses was presented in tables, graphs and figures for interpretation.

CHAPTER FOUR

RESULTS

4.1 Characteristics of Potable Water

The mean concentrations of Na⁺, K⁺, EC, TDS and pH for the potable water were 11.87 mg/L, 2.23 mg/L, 110.73µs/cm, 66.41 mg/L and 7.23 respectively, (Table 4.1). The results further show that the concentration levels of Na⁺, K⁺, EC, TDS and pH varied from day to day while the levels at the evening sampling times were significantly higher as compared to the morning sampling time except for K⁺ and pH.

Table 4.1 Physicochemical characteristics of potable water at the University of Embu

Potable Water (PW)	Na ⁺ (mg/L)	K ⁺ (mg/L)	EC (µs/cm)	TDS (mg/L)	pH
Day					
Day 1	11.85 ^b	2.23 ^{ab}	109.45 ^a	65.57 ^a	7.26 ^a
Day 2	11.91 ^a	2.21 ^b	111.68 ^a	67.13 ^a	7.23 ^{ab}
Day 3	11.85 ^b	2.25 ^a	109.32 ^a	65.48 ^a	7.20 ^b
Day 4	11.86 ^{ab}	2.22 ^b	112.48 ^a	67.47 ^a	7.22 ^{ab}
MSD	0.0375	0.0332	9.986	6.240	0.046
Time					
Morning	11.85 ^b	2.21 ^a	106.48 ^b	63.95 ^b	7.23 ^a
Evening	11.90 ^a	2.23 ^a	114.98 ^a	68.88 ^a	7.22 ^a
LSD	0.0197	0.017	5.256	3.285	0.024
Source Mean	11.87	2.23	110.73	66.41	7.23

*Values with similar alphabet in a column represent mean values that are not significantly different at $p \leq 0.05$

4.2 Greywater Characteristics

The results (Table 4.2) show the mean values of Na⁺, K⁺, EC, TDS and pH levels in male and female students' hostels greywater as affected by day and time of sampling for each source, as well as the overall mean variation based on the source of greywater.

Table 4.2 Physicochemical characteristics of greywater drawn from the male and female University students' hostels

Days (GWMH)	Na⁺ (mg/L)	K⁺ (mg/L)	EC (µs/cm)	TDS (mg/L)	pH
Day 1	328.35 ^a	43.01 ^{ab}	270.78 ^a	170.87 ^a	7.24 ^{ab}
Day 2	337.46 ^a	42.69 ^{ab}	182.17 ^c	109.12 ^c	7.81 ^a
Day 3	292.39 ^{ab}	49.90 ^a	182.93 ^c	109.63 ^{bc}	7.69 ^{ab}
Day 4	255.18 ^b	34.02 ^b	228.13 ^b	137.14 ^b	7.15 ^b
MSD	53.663	11.89	35.933	27.689	0.616
Days (GWFH)					
Day 1	285.34 ^b	40.41 ^b	210.83 ^a	147.13 ^a	7.99 ^a
Day 2	283.00 ^b	42.50 ^b	177.67 ^b	118.66 ^b	7.72 ^{ab}
Day 3	285.83 ^b	43.66 ^{ab}	175.65 ^b	110.92 ^b	7.27 ^b
Day 4	298.70 ^a	46.46 ^a	202.47 ^a	121.40 ^b	7.92 ^a
MSD	9.182	3.818	18.036	19.965	0.477
Time (GWMH)					
Morning	226.34 ^b	39.37 ^a	168.63 ^b	105.54 ^b	7.31 ^b
Evening	380.35 ^a	45.44 ^a	263.37 ^a	157.84 ^a	7.64 ^a
LSD	28.245	6.260	18.913	14.574	0.324
Time (GWFH)					
Morning	188.95 ^b	43.41 ^a	133.68 ^b	90.33 ^b	7.23 ^b
Evening	387.49 ^a	43.11 ^a	249.63 ^a	158.72 ^a	8.23 ^a
LSD	4.833	2.010	9.493	10.508	0.251
Source					
Male hostels' greywater	303.35 ^a	42.40 ^a	216.00 ^a	131.69 ^a	7.47 ^a
Female hostels' greywater	288.22 ^a	43.26 ^a	191.65 ^b	124.53 ^a	7.73 ^a
LSD	19.033	3.856	12.346	9.545	0.269

*Values with similar alphabet in a column represent means that are not significantly different at $p \leq 0.05$

From the results in Table 4.2, the mean Na⁺, K⁺, EC, TDS and pH of greywater from the male hostels was 303.35 mg/L, 42.40 mg/L, 216.00 µs/cm, 131.69 mg/L and 7.47, respectively. The female hostels' greywater recorded mean values of 288.22 mg/L, 43.26 mg/L, 191.65 µs/cm, 124.53 mg/L and 7.73 for the same parameters respectively. There was no significant difference ($p \leq 0.05$) in Na⁺, K⁺, TDS and pH for both sources except for EC. There was significant difference ($p \leq 0.05$) in quality of greywater for both sources from day to day. The results also show that the quality of water sampled in the evening was significantly higher ($p \leq 0.05$) in Na⁺, EC, TDS and pH concentration levels for both GWMH and GWFH when compared to the morning concentration levels. The K⁺ ions

concentrations remained significantly not different in the morning and evening sampling times for both the male and female greywater sources.

4.3 Banana Stalks and Biochar Characterization

The results for banana stalks and biochar characterization are as follows:

4.3.1 Moisture content analysis

The results for the moisture content of the sampled banana stalks was 84.09%, 84.15%, 84.92% and 84.52%, for the four samples considered, while the overall mean moisture content of the banana stalks was recorded as 84.44%.

4.3.2 Determination of biochar pH

The pH of biochar pyrolyzed at different temperatures are as shown in table 4.3

Table 4.3 The pH of biochars pyrolyzed at temperatures 300 °C, 400 °C and 500 °C

Biochar	Ph
Biochar 300°C	6.45 ^c
Biochar 400° C	8.07 ^b
Biochar 500°C	10.18 ^a
MSD	0.0167

The pH of biochars 300 °C, 400 °C and 500 °C were 6.45, 8.07 and 10.18, respectively (Table 4.3). As per the results, as the pyrolytic temperatures increased, the biochar pH became more alkaline.

4.3.3 Biochar yield analysis

The results of biochar yields produced at different pyrolytic temperatures are as outlined in Figure 4.1.

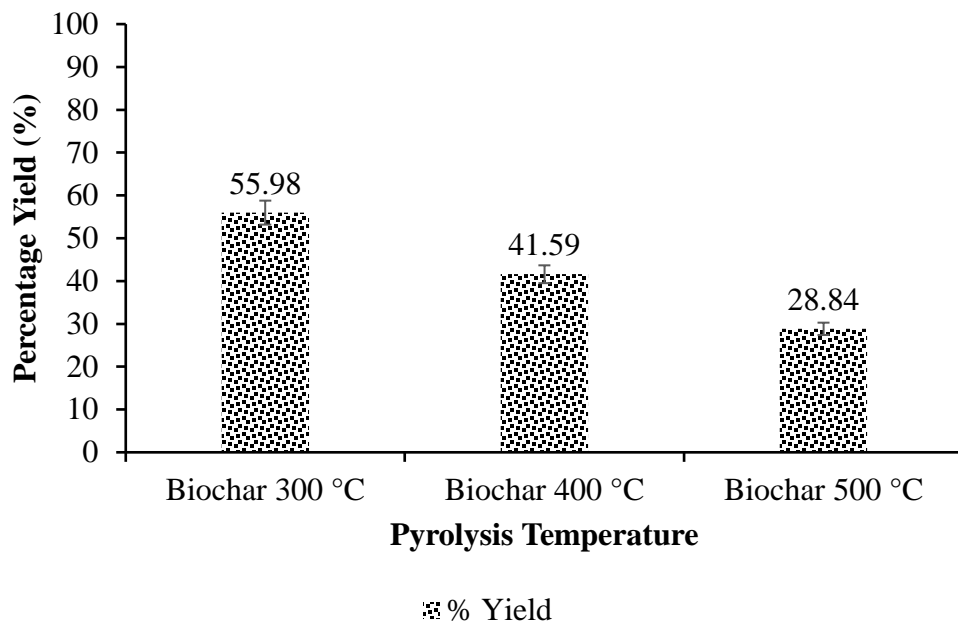
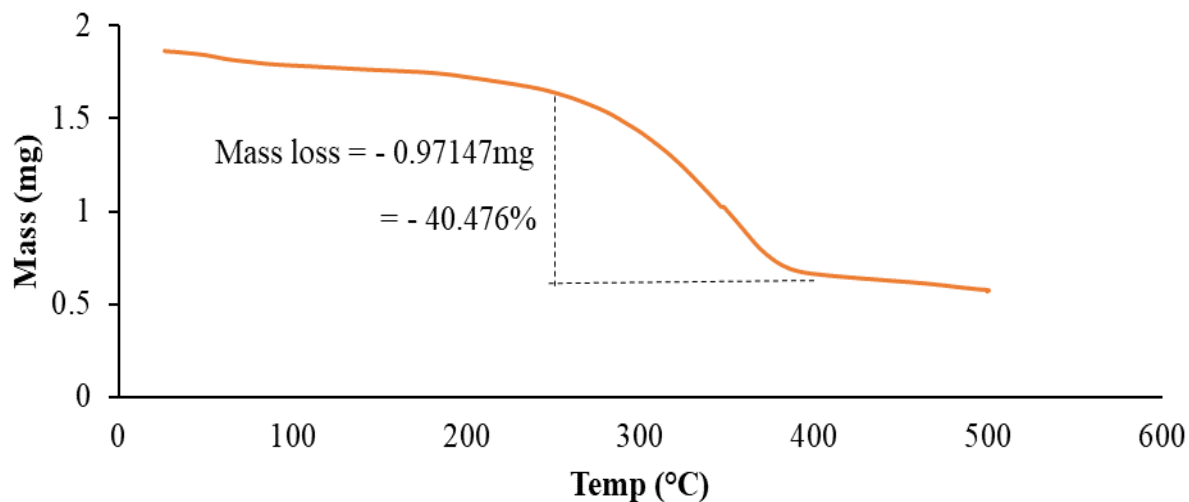


Figure 4.1: Percentage (%) yield of biochars at pyrolytic temperatures 300 °C, 400 °C and 500 °C

The yield of biochar decreased with increase in pyrolytic temperatures. As shown in the results (Figure 4.1) the yield of biochar at 300 °C was significantly higher ($p \leq 0.05$) than biochar 400 °C and biochar 500 °C, at 55.98%, 41.49% and 28.84%, respectively.

4.3.4 Thermo gravimetric analysis (TGA)

The results of the thermal gravimetric analysis of dry banana stalks powder are as shown in figure 4.2. The figure shows the thermal degradation curve of the banana stalks sample from room temperature to a temperature of 500 °C.



— Thermal Decomposition Curve

Figure 4.2: Thermogravimetric (TG) curve for the decomposition of Banana Stalks powder from room temperature to 500 °C

From the results (Figure 4.2), from a temperature of 0 °C to 200 °C there was moderate decrease in mass of the sample. As the temperature increased, the banana stalks powder (BSP) experienced a significant mass loss of over 40% at temperatures between 250 °C and 400 °C.

4.3.5 Fourier transform infrared analysis

The results of phase one FTIR analysis are as shown in Figures 4.3, 4.4, 4.5 and 4.6 while phase two FTIR analysis is shown by Figures 4.7, 4.8 and 4.9

4.3.5.1 FTIR - phase one

The infrared spectra depicted in Figures 4.3, 4.4, 4.5 and 4.6 shows the surface functional groups present in raw banana stalks, activated biochar pyrolyzed at 300 °C, activated biochar pyrolyzed at 400 °C and activated biochar pyrolyzed at 500 °C, respectively.

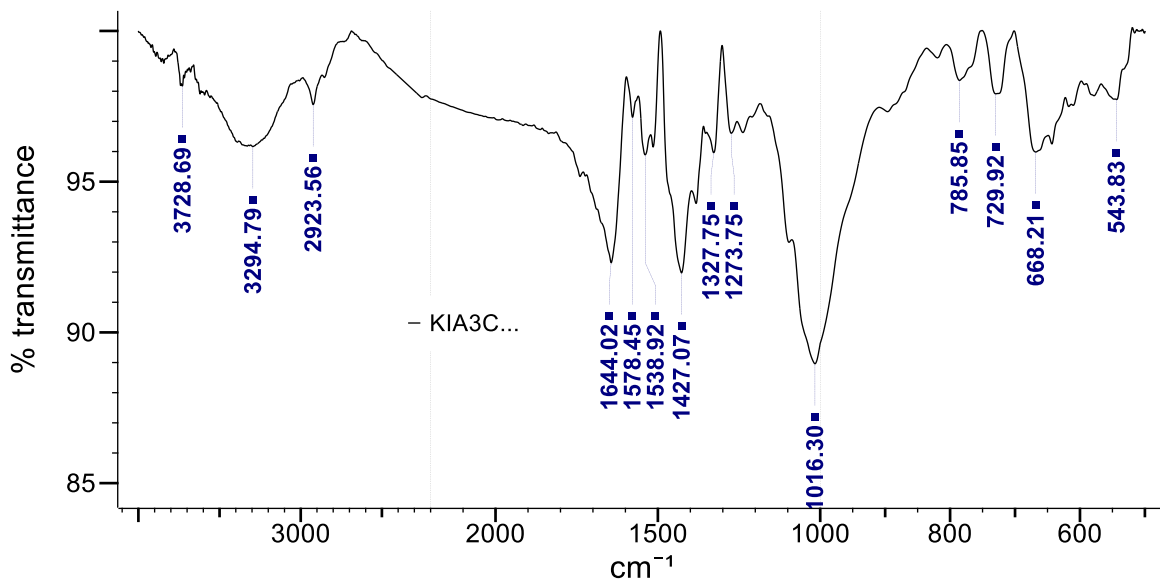


Figure 4.3: FTIR spectrum of raw banana stalks powder before pyrolysis (BSP)

The raw banana stalks FTIR showed peaks at wavenumbers 3294 cm^{-1} and other peaks at 2923 cm^{-1} , 1644 cm^{-1} , 1538 cm^{-1} , 1427 cm^{-1} and 1016 cm^{-1} . These peaks correspond to O-H, C-H stretching, C=O, N-O, O-H bending and C-O surface functional groups, respectively.

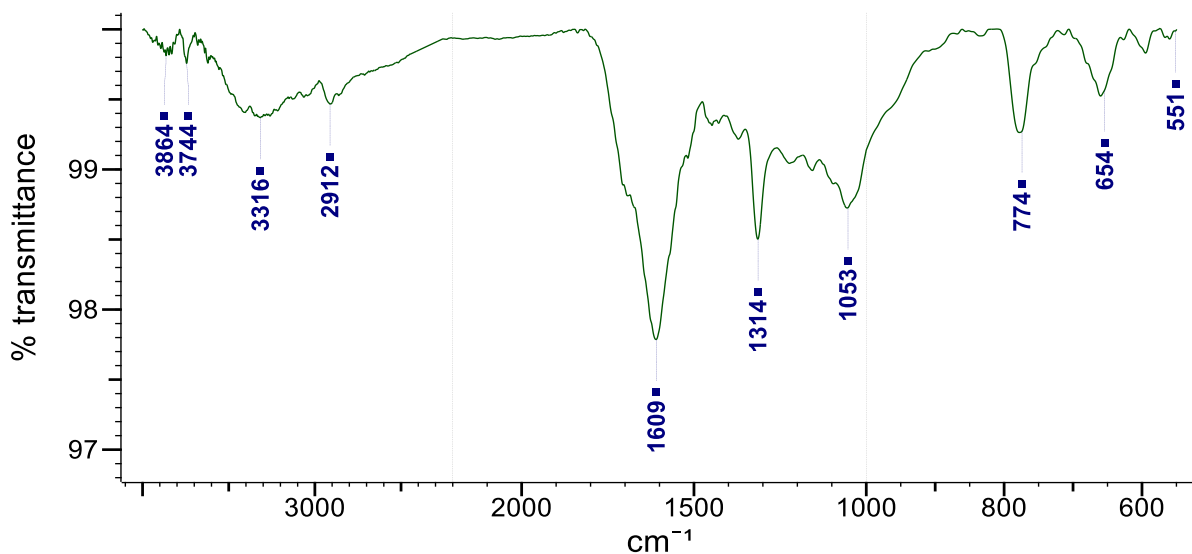


Figure 4.4: FTIR spectrum of banana stalks pyrolyzed at Temperature 300 °C (Biochar 300 °C)

The peaks identified on biochar 300 °C were at wavenumbers 3316 cm^{-1} , 2912 cm^{-1} , 1609 cm^{-1} , 1314 cm^{-1} and 1053 cm^{-1} . These peaks correspond to carboxylic O-H, aldehyde C-H, conjugated C=C, amine C-N and stretching C-O groups, respectively.

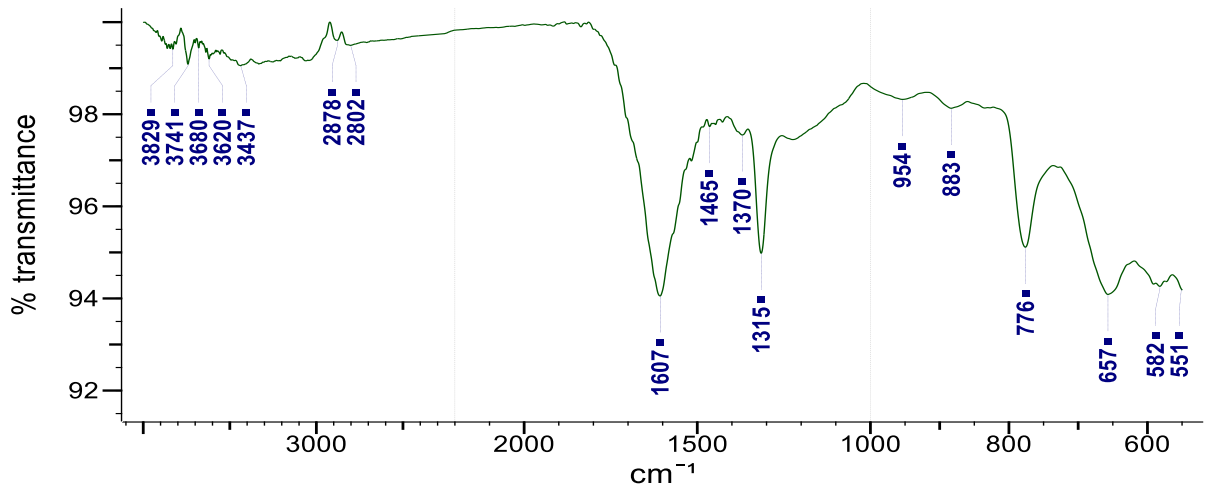


Figure 4.5: FTIR spectrum of banana stalks pyrolyzed at temperature 400 °C (Biochar 400 °C)

The peaks identified on the surface of biochar 400 °C were at wavenumbers 3620 cm^{-1} , 2878 cm^{-1} , 1607 cm^{-1} and 1315 cm^{-1} . The peaks correspond to free O-H, C-H, conjugated C=C and amine C-N groups, respectively.

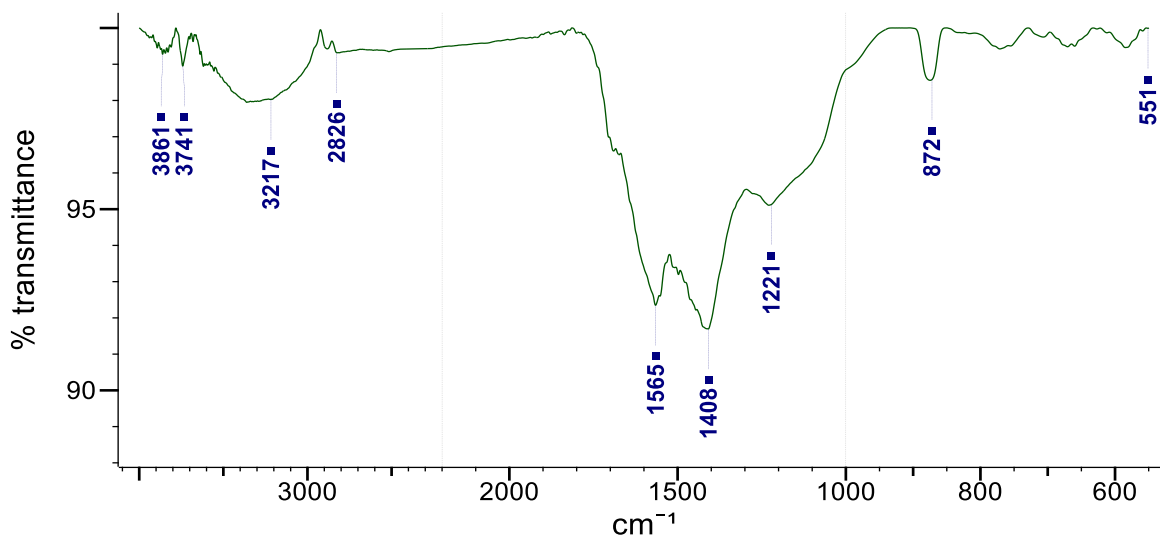


Figure 4.6: FTIR spectrum of banana stalks pyrolyzed at temperature 500 °C (Biochar 500 °C)

On biochar 500 °C, the identified surface functional groups were the O-H functional group at 3217 cm^{-1} , a nitro NO_2 at 1565 cm^{-1} , a C-O stretching group at 1221 cm^{-1} and a peak at 1408 cm^{-1} which could be attributed to S=O stretching sulfate/sulfonyl chloride or O-H bending alcohol or carboxylic acid group.

4.3.5.2 FTIR - phase two

The FTIR spectra depicted in Figures 4.7, 4.8 and 4.9 shows the surface functional groups present in activated biochar at 300 °C, 400 °C and 500 °C, respectively, after adsorption

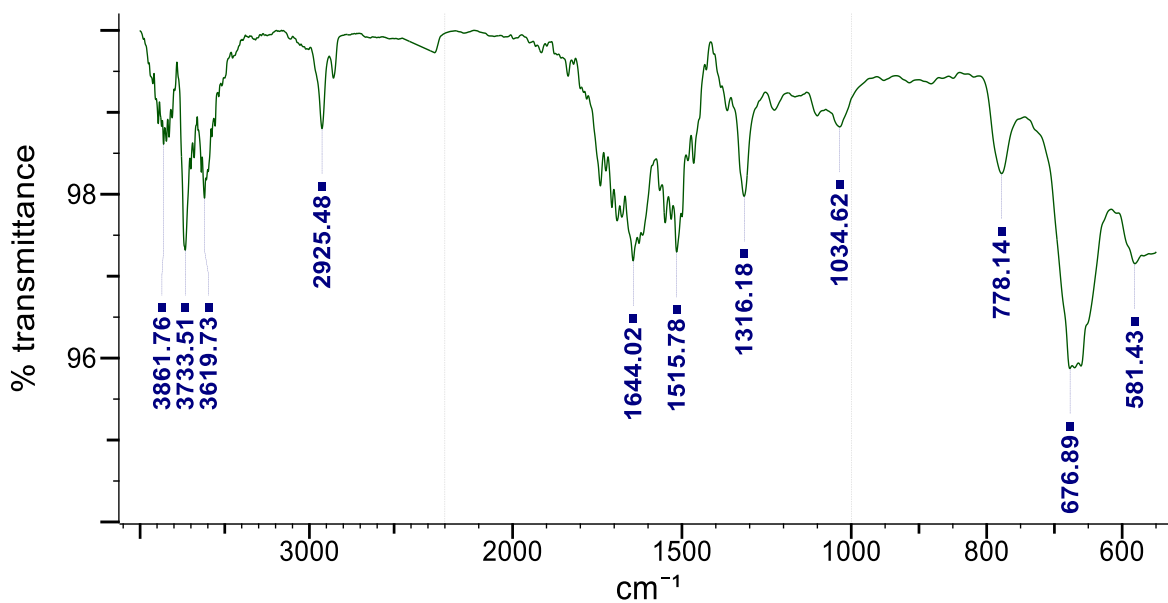


Figure 4.7: FTIR spectrum of biochar 300 °C after adsorption

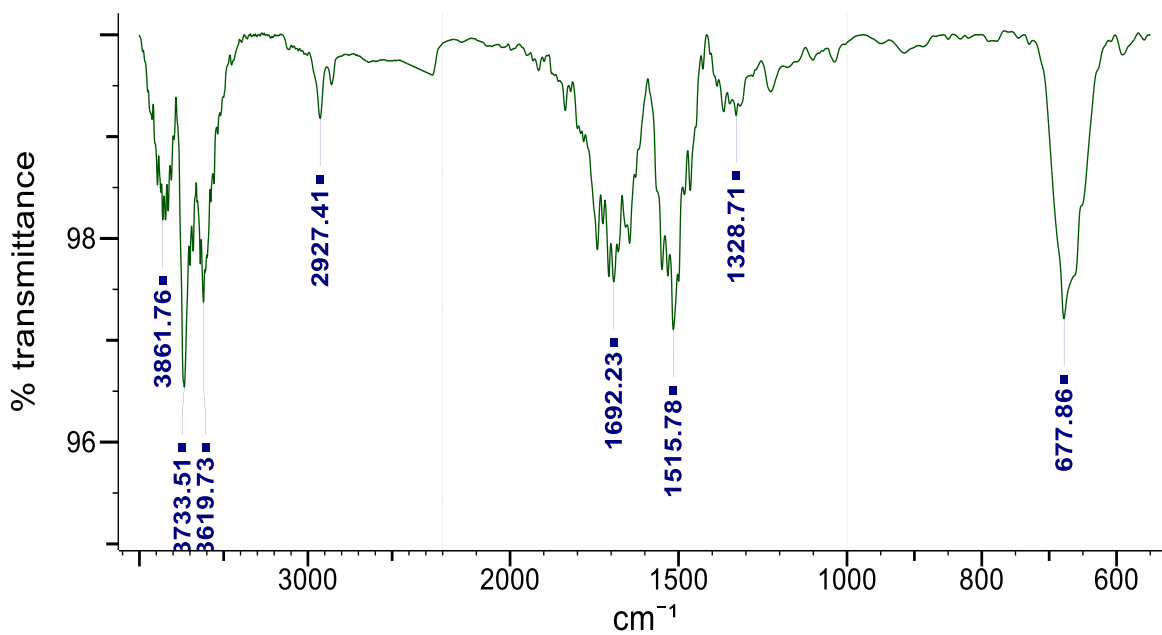


Figure 4.8: FTIR spectrum of biochar 400 °C after adsorption

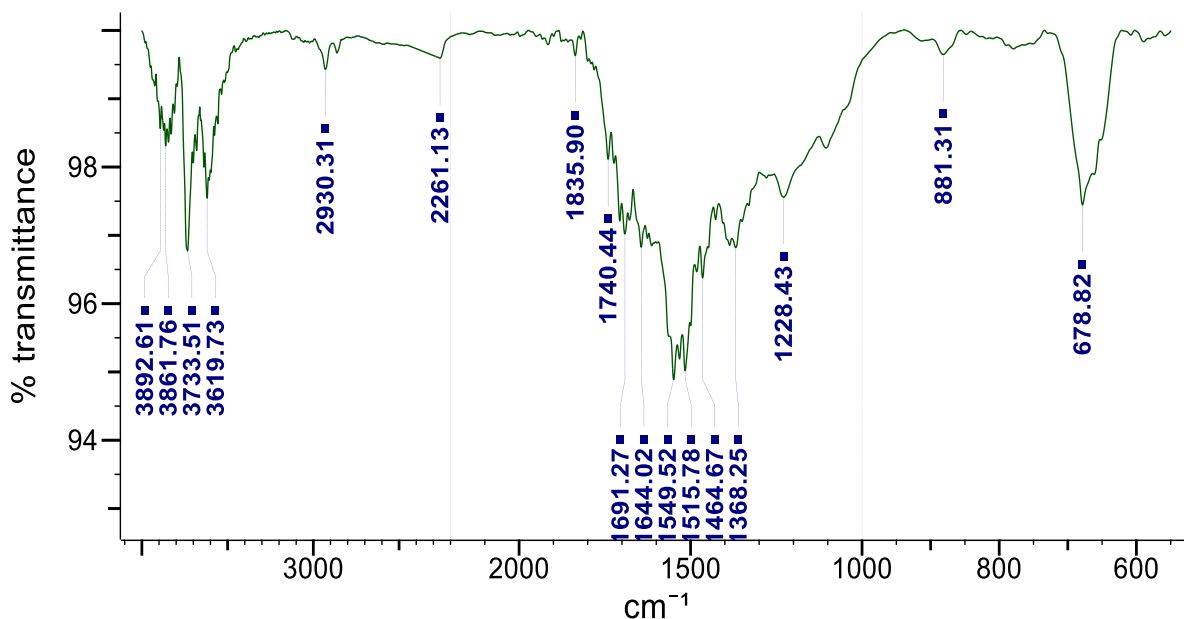


Figure 4.9: FTIR spectrum of biochar 500 °C after adsorption

As depicted in Figure 4.8, 4.9 and 4.10, the peaks at 1034 cm^{-1} , 1228 cm^{-1} , 1316 cm^{-1} , 1328 cm^{-1} , 1336 cm^{-1} , 1515 cm^{-1} , 1549 cm^{-1} , 1644 cm^{-1} , 1691 cm^{-1} , 2925 cm^{-1} , 2927 cm^{-1} , 2930 cm^{-1} and 3619 cm^{-1} surface functional groups were observed. The carboxyl and hydroxyl surface functional groups were attenuated after adsorption.

4.4 Adsorption Study

The results of the initial and final concentration levels of the study parameters are as shown in Table 4.4. From the results, the final concentration levels reduced with biochar 500 °C recording higher removal of Na^+ , K^+ and COD than biochar 400 °C and biochar 300 °C, respectively. In the case of BOD, biochar 300 °C recorded the highest reduction followed by biochar 400 °C and 500 °C. The percentage reduction is as shown in Figure 4.10 and Figure 4.11.

Table 4.4 The initial and final concentration levels of the study parameters

	Na+ (mg/L)	K+ (mg/L)	COD (mg/L)	BOD (mg/L)
Initial concentrations	286.98	42.50	616	416.67
Final concentrations				
Biochar 300 °C	168.94	25.35	380.01	119.67
Biochar 400 °C	119.67	18.29	311.02	139.0
Biochar 500 °C	99.21	4.52	242.7	158.67

4.4.1 Na⁺ and K⁺ percentage reduction

The results of the percentage reduction in concentration levels of Na⁺ and K⁺ in greywater after adsorption in biochar 300 °C, biochar 400 °C and biochar 500 °C, are as shown in Figure 4.10. The results depict a comparative analysis of the three types of biochars' performance in reducing the target chemical pollutants.

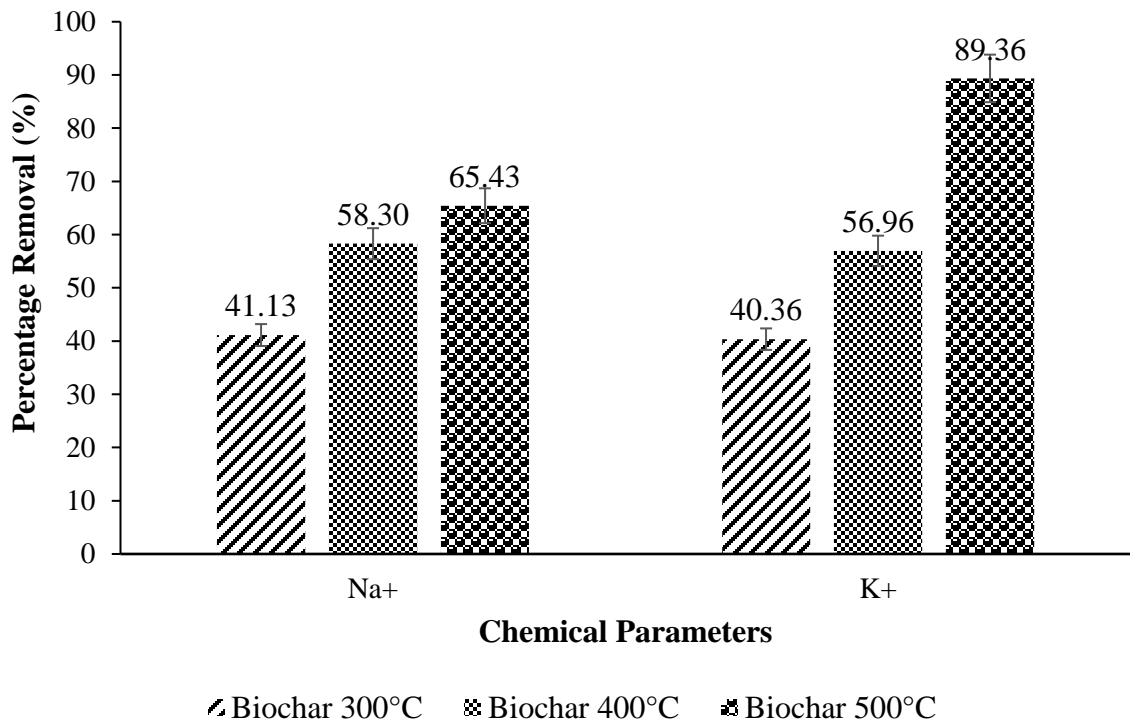


Figure 4.10: Percentage (%) removal of Na⁺ and K⁺ by biochars 300 °C, 400 °C and 500 °C

The initial levels of Na⁺ and K⁺ in the greywater was recorded as 286.98 mg/L and 42.59 mg/L. The percentage reduction in the levels of the cations in greywater was calculated and the results are as shown in Figure 4.10. The results showed a significant difference ($p \leq 0.05$) in the performance of biochar 300 °C, biochar 400 °C and biochar 500 °C in reducing the Na⁺ and K⁺ levels in greywater. Biochar 500 °C removed a significantly higher ($p \leq 0.05$) percentage of Na⁺ when compared to biochar 300 °C and biochar 400 °C while biochar 300 °C performance was significantly lower ($p \leq 0.05$) than biochar 400 °C and 500 °C. A similar trend was recorded for removal of K⁺ in the greywater where biochar 500 °C recorded a significantly higher ($p \leq 0.05$) percentage removal than biochar 400 °C and 300 °C. The performance of biochar 400 °C was also significantly higher ($p \leq 0.05$) than biochar 300 °C in respect to the percentage removal of K⁺ ions.

4.4.2 COD and BOD percentage reduction

The results of the percentage reduction of COD and BOD after adsorption in biochar 300 °C, biochar 400 °C and biochar 500 °C are as shown in Figure 4.11. The results show a comparative performance of the three types of biochar in reducing the target biological pollution indicators.

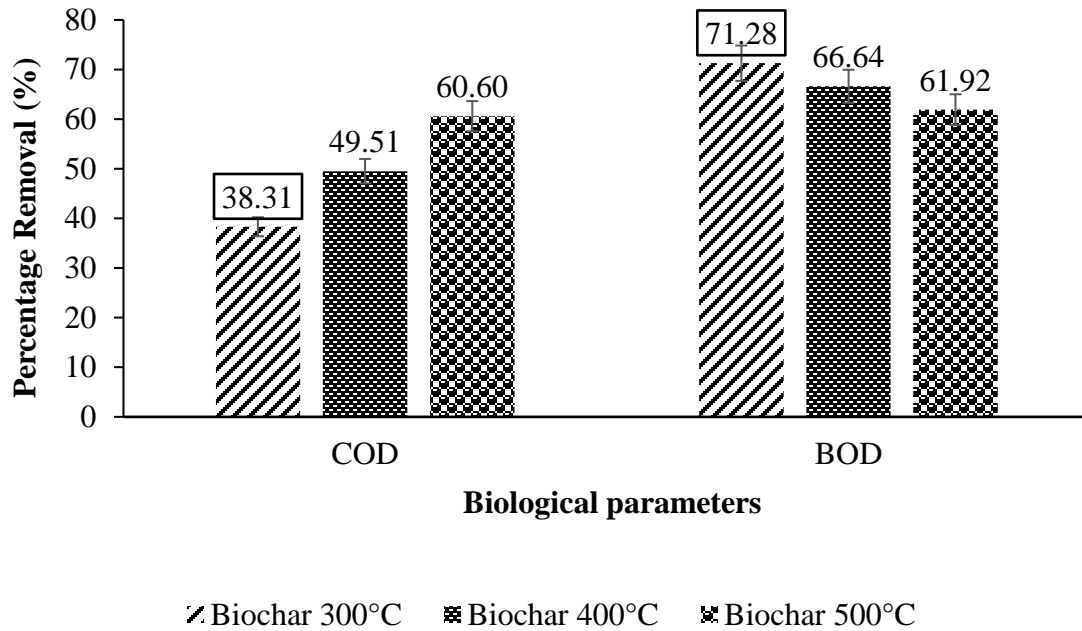


Figure 4.11: Percentage (%) removal of COD and BOD by Biochars 300 °C, 400 °C and 500 °C

The initial levels of COD and BOD₅ in greywater were recorded as 616 and 416.67 mg/L respectively. The recorded percentage removal of the biological parameters in the greywater for the different biochar systems are as shown in Figure 4.11. According to the results, biochar 500 °C had significantly higher ($p \leq 0.05$) percentage reduction of the chemical oxygen demand (COD) in the greywater as compared to biochar 400 °C and 300 °C. Biochar 400 °C was also significantly higher ($p \leq 0.05$) in the percentage reduction of the COD levels in the greywater as compared to biochar 300 °C. In the case of biochemical oxygen demand, the performance of the biochar systems also varied significantly ($p \leq 0.05$) with the highest percentage removal rate recorded for biochar 300 °C. Biochar 500 °C recorded a significantly lower ($p \leq 0.05$) percentage removal of the BOD in greywater as compared to biochar 400 °C and biochar 300 °C.

CHAPTER FIVE

DISCUSSION, CONCLUSION AND RECOMMENDATIONS

5.1 Discussion

5.1.1 Potable water characteristics

The results (Table 4.1) revealed that the Na^+ , K^+ , EC, TDS and pH levels in the sampled water were within the acceptable WHO quality levels for drinking water of 50 mg/L, 10 mg/L, 1000 $\mu\text{s}/\text{cm}$, 600 mg/L and 6.5-8.5, respectively (WHO, 2017). The results further showed that EC and TDS for the different days were not significantly different ($p \leq 0.05$). The Na^+ levels were not significantly different for days 1, 3 and 4 while day 2 was not significantly different from day 4. The K^+ and pH levels were not significantly different for days 1, 2 and 4 while day 3 was not significantly different from day 1 for K^+ and days 2, 3 and 4 for pH ($p \leq 0.05$). The small variation noted for Na^+ , K^+ , and pH had an LSD of 0.0375, 0.0332 and 0.046, respectively.

Apart from the pH, the concentration levels of Na^+ , K^+ , EC and TDS were slightly higher in the evening as compared to morning levels. This could be because of the water treatment process that takes place during the day where coagulants and disinfectants used in the process may cause anionic and cationic variation during the day. The potable water generally had slight variations in quality over the sampling period while the concentration levels of the selected physicochemical water quality parameters remained within the recommended standards for drinking water (WHO, 2017).

5.1.2 Greywater characteristics

The results (Table 4.2) revealed that the Na^+ , K^+ , EC and TDS levels from both GWMH and GWFH sources were significantly higher ($p \leq 0.05$) compared to their levels in the potable water (Table 4.1). The high levels are attributed to the detergents used by students and dirt introduced as water is being used in the hostels while bathing, washing or during laundry. This water generally contains soap precipitates, cosmetics, hair, lint, detergents, textile chemicals, among others (Laghari *et al.*, 2016). The soaps and detergents, mainly sodium and potassium salts, explain the high levels of Na^+ and K^+ in greywater. The other

anions and cations present in detergents contribute to the high EC and TDS of greywater when compared to potable water. The GWMH and GWFH pH was also slightly more alkaline compared to the potable water.

The results (Table 4.2) also indicate that Na^+ , K^+ , TDS and pH levels in both GWMH and GWFH were statistically similar ($p \leq 0.05$). This may indicate that the detergents used by the students are similar as well as their water use patterns. The GWMH had higher EC levels as compared to GWFH indicating presence of additional anions and cations in male greywater. This could be because most male students are involved in gaming activities as compared to female students. The GWFH, though not significantly different ($p \leq 0.05$) to GWMH, recorded a more alkaline pH as compared to the GWMH. This could be due to the use of more cosmetics and other personal care products. The recorded pH means of 7.47 and 7.73 for greywater drawn from the male and female hostels, respectively, closely corroborate with a mean of 7.49 recorded in students' hostels in India (Ghaitidak & Yadav, 2016).

The GWMH and GWFH TDS values of 131.70 and 124.53 mg/L, respectively, recorded in this study were slightly lower but within close range to 140 and 161 mg/L TDS recorded in schools' hostels in Kuwait and in Thiruvananthapuram, Kerala, respectively (Alsulaili & Hamoda, 2015; Pillai & Vijayan, 2012). The Na^+ ions level recorded in this study are higher than those of 220 and 140 mg/L reported from student hostels in Jordan and India, respectively (Ghunmi *et al.*, 2008; NEERI, 2007). This could be attributed to the difference in detergents and other personal care products used by the students in their respective domains. The K^+ ions levels were also higher than those reported by Ghunmi *et al.* (2008). The EC and TDS recorded in this study were lower compare to the reported means of 489 $\mu\text{s}/\text{cm}$ and 304 mg/L in a study involving student hostels in India (Ghaitidak & Yadav, 2016). The differences in Na^+ , K^+ , TDS and EC levels could probably be attributed to differences in the cosmetics used by tertiary level students in Africa and Asia.

5.1.2.1 Effect of sampling day on greywater quality

The results presented in Table 4.2 reveal that the Na^+ in GWMH was not significantly different ($p \leq 0.05$) for days 1, 2 and 3 while day 3 and 4 were also not significantly

different ($p \leq 0.05$). The mean K^+ concentration for days 1, 2 and 4 were not statistically different ($p \leq 0.05$), while the mean values on day 1, 2 and 3 were also not statistically different ($p \leq 0.05$). The highest EC mean of 270.78 $\mu\text{S}/\text{cm}$ was recorded on day 1 and was significantly higher ($p \leq 0.05$) compared to those recorded on day 2, 3 and 4. The lowest EC was on day 2 and was not statistically different ($p \leq 0.05$) from that of day 3. The TDS of greywater from the male source had daily variations with day 1 recording a significantly higher ($p \leq 0.05$) mean value when compared to day 2, 3 and 4 (Table 4.2). It was also noted that the TDS values on days 2 and 3, and 3 and 4, respectively, were not statistically different ($p \leq 0.05$). The GWMH pH value was highest on day 2 and was significantly higher ($P \leq 0.05$) than the one recorded on day 4, although it was not significant different from the values recorded on day 1 and 3.

The GWFH also revealed daily variations for the different physicochemical characteristics studied. The results (Table 4.2) indicate that that the Na^+ and K^+ levels were significantly higher ($p \leq 0.05$) on day 4 as compared to values recorded on day 1, 2 and 3 all three being not significantly different. The EC levels on day 1 and 4 are not statistically different ($p \leq 0.05$) while the values recorded on days 2 and 3 are not significantly different ($p \leq 0.05$). The TDS levels were not significantly different ($p \leq 0.05$) on days 2, 3 and 4 and values on all three days were significantly lower ($p \leq 0.05$) compared to day 1. The highest pH in female greywater of 7.99 was recorded on day 1 and was not significantly different ($p \leq 0.05$) from values recorded on day 2 and 3 (Table 4.2). It was also noted that the pH values recorded on days 1, 3 and 4 were not significantly different ($p \leq 0.05$).

Generally, the levels of the selected physio-chemical greywater characteristics for both GWMH and GWFH varied from day to day. Characteristics of greywater may vary based on many anthropogenic factors such as population, inhabitants living styles, nature of occupants and water usage patterns, among others (Laghari *et al.*, 2015). As such, the variations noted in this study indicate that though the personal care products as well as laundry products used by the students may be similar, the quantities may vary from day to day as well as their water usage patterns. The daily activities that students engage in may vary from day to day as well as the number of students engaged in activities such as gaming or even laundry. This may lead to the daily variations in the pollutants loads as

observed in this study. Shankhwar *et al.* (2015) indicated that fluctuations in greywater discharge in all days of the week impacts on the overall variation in greywater composition.

5.1.2.2 Effect of sampling time on greywater quality

The results in Table 4.2 revealed that for both GWMH and GWFH the sampling time had a significant impact on the levels of Na⁺, EC, TDS and pH, while the K⁺ levels for samples drawn in the morning and evening were not significantly different ($p \leq 0.05$). The levels of Na⁺, EC, TDS and pH recorded in the evening were significantly higher ($p \leq 0.05$) when compared to levels recorded in the greywater sampled in the morning (Table 4.2). This is indicative of the days' activities which include movement to lectures, day time activities and evening gaming activities which lead to increased sweating and dirt which in turn increase pollutants load during bathing in the evening.

The personal care products applied in the morning as students start their day's business are washed in the evening and thus may increase the anions and cations present in the greywater. Some students tend to do laundry in the evening after classes which in turn increases the pollutants load in the evening greywater. In the morning, the concentrations were lower indicative of a relatively low pollutants load due to less to zero activity during the night, less use of personal care products used for the nights as well as less laundry in the morning hours. A study by Eriksen *et al.* (2009) also noted an increase in conductivity from the morning to late afternoon. The potassium (K⁺) ions concentration levels were not significantly different ($p \leq 0.05$) in the morning and evening. In soaps and detergents making, sodium salts are more commonly used as compared to potassium salts and therefore the levels of potassium ions are generally lower compared to sodium ions. Other researchers have also noted temporal variations in pollutants loads (Shankhwar *et al.*, 2015; Li & Zhang, 2007;).

5.1.3 Biochar characterization

5.1.3.1 Moisture content

The banana stalks recorded a mean moisture content of 84.44%. This indicates that the dry mass yield of banana stalks after eliminating the moisture content was around 15.56% of the total feedstock mass. These results closely corroborate with a study by Kabenge *et al.* (2018) where the moisture content of banana pseudo stem, leaves and banana peels recorded a moisture content of 87%, 78% and 72%, respectively.

5.1.3.2 Biochar pH

The results in Table 4.3 shows that the pH of the biochars pyrolyzed at different temperatures were significantly different ($p \leq 0.05$). The pH of biochar 500 °C was significantly more alkaline than both biochar 400 °C and biochar 300 °C, while biochar 400 °C was also significantly more alkaline than biochar 300 °C ($p \leq 0.05$). The increase in biochars pH may be as a result of loss of volatile organic compounds as well as the increase in basic cations in biochars produced in high temperatures of pyrolysis (Shaheen *et al.*, 2019, Zhang *et al.*, 2015). The alkali pH of the biochars could also be as a result of the alkali salts separation from the organic matrix in the feedstock (Ahmad *et al.*, 2012a). Biochars produced at lower temperatures may have greater densities of acidic functional groups such as carboxylic and phenolic groups, which may account for the decreasing biochar pH (Zhang *et al.*, 2015).

The trend of increasing pH with increasing pyrolytic temperature, observed in this study, broadly corroborates with a similar pH trend reported by Zhang *et al.* (2015). In their study on sludge biochar, they recorded a pH of 6.2, 7.5, and 8.1 at pyrolytic temperatures of 300 °C, 400 °C and 500 °C, respectively. The slight difference of their reported pH at similar pyrolytic temperatures to the ones used in this study, could be as a result of the difference in the parent material used in producing biochar in both studies. In another study, a banana peduncle pyrolyzed at 500 °C had a pH of 10.1 (Karim *et al.*, 2015) which was similar to the pH of 10.18 for banana stalks pyrolyzed at 500 °C in this study.

5.1.3.3 Biochar yield

The results in Figure 4.1 revealed that the biochar yield was significantly different ($p \leq 0.05$) at the three pyrolytic temperatures studied. The yield of biochar prepared at a temperature of 300 °C was significantly higher ($p \leq 0.05$) than those prepared at temperatures 400 °C and 500 °C. The yield at temperatures 500 °C was significantly lower than the yield at temperatures 400 °C ($p \leq 0.05$). This indicates that as the pyrolytic temperature increased from 300 °C to 400 °C and finally to 500 °C, for this study, the yield of biochar decreased with increasing pyrolytic temperatures. This can be as a result of the loss of volatile organic compounds experienced at high pyrolytic temperatures.

These results concur with Nanda *et al.* (2016) who reported that biochar prepared at lower temperatures have higher yields and more volatile compounds when compared to biochar prepared at higher temperatures which exhibit low yields and less volatiles but increased surface micro-porosity. This is further in line with Novak *et al.* (2009) who noted that the rise in pyrolysis temperatures decreases the biochar yield due to the decomposition of cellulose, hemicellulose and some parts of lignin in feedstock. The yield of biochar pyrolyzed at 300 °C by Karim *et al.* (2015) had a yield of 66% which is slightly higher than 55.98% in this study. The difference could be due to the difference in feedstock material as well as the heating rate in the two studies. The yield of bagasse biochar was noted to substantially decrease from 77% to 32% when temperatures increased from 250 °C to 400 °C (Ding *et al.*, 2014), depicting a similar trend to the one reported in this study.

5.1.3.4 Thermogravimetric analysis

As depicted in the TG curve (Figure 4.2), the banana stalks experienced mass loss as the temperature increased. The biomass, thus, underwent physical, chemical and molecular changes which in turn reduced the mass and volume of the material due to volatilization and shrinkages (Demessie *et al.*, 2015). In this study the banana stalks had been oven dried to constant mass before being introduced in the TGA furnace and thus the mass loss was significantly low from 0 °C to 200 °C. Significant mass loss was noted at temperatures between 200 °C and 400 °C with the greatest mass loss of over 40% occurring at temperatures between 250 °C and 400 °C.

These results were in tandem with a report that documented that major mass loss in biochars occur in temperatures between 200 °C and 400 °C (Asadullah *et al.*, 2007). The results of a study by Sulaiman & Abdullah (2014) closely corroborates with this study. The study noted moisture loss of the banana pseudo stem to around 150 °C while at temperatures 300 °C and 350 °C, the material experienced maximum degradation rate. Another study by Ding *et al.* (2014) on the thermal degradation of bagasse noted that the yield dropped from 77% to 32% as the pyrolysis temperatures increased from 250 °C to 400 °C.

5.1.3.5 FTIR analysis

5.1.3.5.1 FTIR – Phase one

The raw banana (Figure 4.3) showed a broad band at around 3294 cm^{-1} and other peaks at 2923 cm^{-1} , 1644 cm^{-1} , 1538 cm^{-1} , 1427 cm^{-1} and 1016 cm^{-1} . The broad band corresponds to the presence of an O-H functional group while the other peaks corresponds to C-H stretching, C=O, N-O, O-H bending and C-O functional groups, respectively. These results corroborate with a study by Dos Santos *et al.* (2019) on functional groups present in raw banana fiber who noted the presence of O-H, C-H stretching, O-H bending, C=O and C-O surface functional groups. The presence of O-H, C-H, C-O, C=O functional groups was also reported in banana peel FTIR spectrum (DeMessie *et al.*, 2015).

Biochar 300 °C (Figure 4.4) exhibited peaks at 3316 cm^{-1} , 2912 cm^{-1} , 1609 cm^{-1} , 1314 cm^{-1} and 1053 cm^{-1} . These peaks correspond to O-H, aldehyde C-H, conjugated C=C, amine C-N and stretching C-O groups, respectively. The C-H and C-O surface functional groups experienced a shift in peaks after pyrolysis at 300 °C. In biochar 300 °C, the band shift from 2923 cm^{-1} to 2912 cm^{-1} , and 1016 cm^{-1} to 1053 cm^{-1} , for the C-H and C-O groups was observed. A similar trend was observed in a different study of banana fiber pyrolyzed at 300 °C which showed the presence of O-H, C-H stretching, C=C and C-O groups in biochar (Dos Santos *et al.*, 2019; Milani *et al.*, 2016). A shift in functional group band after pyrolysis was also confirmed by Li *et al.* (2016), where they reported a shift in C-O peaks after increasing pyrolysis temperature.

Biochar 400 °C (Figure 4.5) showed peaks at 3620 cm⁻¹, 2878 cm⁻¹, 1607 cm⁻¹ and 1315 cm⁻¹. The peaks correspond to free O-H, C-H, conjugated C=C and amine C-N groups, respectively. A band shift was also noted for the C-H group from 2923 cm⁻¹ to 2878 cm⁻¹. Biochar 500 °C (Figure 4.6) exhibited a broad O-H functional group at 3217 cm⁻¹, an aldehyde C-H stretching group at 2826 cm⁻¹, a nitro NO₂ at 1565 cm⁻¹, a C-O stretching group at 1221 cm⁻¹ and a peak at 1408 cm⁻¹ which could be attributed to S=O stretching sulfate/sulfonyl chloride or O-H bending alcohol or carboxylic acid group. A band shift for the C-O group from 1016 cm⁻¹ to 1221 cm⁻¹, carboxylic O-H group from 3294 cm⁻¹ to 3217 cm⁻¹, the O-H bending from 1427 cm⁻¹ to 1408 cm⁻¹, was observed on biochar 500 °C. The presence of O-H, C-H, C=C functional groups in biochar 400 °C and 500 °C was also reported by Dos Santos *et al.* (2019).

After pyrolysis of the banana stalks, the hydroxyl O-H stretching vibrations and the C-O stretching vibrations were attenuated. This indicates that most of the O₂-containing functional groups such as polysaccharides diminished after pyrolysis (Shaheen *et al.*, 2019; Kloss *et al.*, 2012). The functional group C-O which was pronounced in the raw banana stalks FTIR spectrum was noted to have significantly reduced in intensity at biochar 300 °C and was missing in both biochar 400 °C and 500 °C. This change could be attributed to the loss of C-O or C-OH functional groups as a result of dehydration and rearrangement of molecules at high pyrolytic temperatures (DeMessie *et al.*, 2015; Ding *et al.*, 2014).

Rutherford *et al.* (2005) and Bagreev *et al.* (2001) found that high pyrolysis temperatures enhance dihydroxylation and gives rise to pore formation as a result of con-current development of fused-ring structures which was consistent with an increasing surface area of the studied biochar. Pyrolysis therefore can lead to alternation of carbon to oxygen, hydrogen to carbon and carbon to nitrogen ratios and may modify functional groups leading to instances such as increase in aromatic C=C or a decrease in O-H and C-H (DeMessie *et al.*, 2015) a case observed on biochar 300 °C and biochar 400 °C in this study.

5.1.3.5.2 FTIR - phase two

Figures 4.7, 4.8 and 4.9 shows the functional groups of biochar 300 °C, 400 °C and 500 °C, respectively, after greywater was passed through the material for adsorption. On biochar 300 °C, (Figure 4.7). It was noted that the O-H group disappeared while the aldehyde C-H and conjugated C=C groups were attenuated. A band shift for C=C group from 1609 cm⁻¹ to 1644 cm⁻¹ was also observed. On biochar 400 °C, the peak intensity of free O-H was reduced after adsorption (Figure 4.8). The peak also experienced a band shift 3620 cm⁻¹ to 3619 cm⁻¹. The observed change in peak intensity could be attributed to adsorption of cations and anions on the surface of the biochar which was further indicated by the reduction in concentration levels of the monitored greywater characteristics (Section 5.1.4).

The decrease in peak vibrations was also reported by Ding *et al.* (2014), who reported a decrease in the carboxyl functional group and a shift of the C=O group vibrational frequency, after lead sorption on biochar. Karim *et al.* (2015) reported a band shift for biochar 300 °C for the O-H and the C-O surface functional groups after adsorption. For biochar 500 °C, the broad O-H surface functional group disappeared in the FTIR spectra of the adsorbent (Figure 4.9). This could be attributed to the adsorption of the ions in the greywater and this is further in tandem with the higher adsorption efficiency of biochar 500 °C as compared to biochar 300 °C and 400 °C. Amin *et al.* (2018) noted that the presence of O-H, N-H and S-O functional groups, aliphatic groups and aromatic rings in banana peel were all responsible for adsorption.

5.1.4 Adsorption study

The results in Figure 4.10 showed that the Na⁺ levels in the greywater was reduced by 41.13%, 58.30% and 65.43% by biochars 300 °C, 400 °C and 500 °C, respectively. A similar trend was observed for K⁺ ions with 40.36%, 56.96% and 89.36% by biochars 300 °C, 400 °C and 500 °C, respectively. A percentage decrease of 38.31%, 49.51% and 60.60% for biochars 300 °C, 400 °C and 500 °C, respectively, was recorded for COD levels (Figure 4.11). The percentage BOD percentage reduction was highest at 71.28% for

biochar 300 °C, followed by 66.64% for biochar 400 °C and 61.92% for biochar 500 °C (Figure 4.11).

The observed reductions in the levels of Na⁺, K⁺, COD, and BOD (Figures 4.11 and 4.12) in greywater from the student's hostels after being treated using biochar agrees with studies conducted on the ability of biochar to reduce contaminants in a range of wastewaters. Manyuchi *et al.* (2018) notes that biochars have high surface area that allows them to act like bio filters in wastewater treatment thus allowing for pollutants removal in the wastewater. The chemical activation of biochars 300 °C, 400 °C and 500 °C using phosphoric acid before being used in adsorption of the study parameters in this study enhanced the adsorptive capacity of the biochars by increasing the active functional groups, external surface area and pore size on the adsorbents' surface (Pathak *et al.*, 2015; Dawood & Sen, 2012).

As noted in the results of Na⁺, K⁺ and COD, the adsorption ability of biochar 500 °C was significantly higher ($p \leq 0.05$) followed by biochar 400 °C and lastly biochar 300 °C. The high percentage removal for biochar pyrolyzed at higher pyrolytic temperatures can be attributed to the increased surface area and micro-porosity of the biochar as a result of being carbonized at high pyrolytic temperatures (Dawood & Sen, 2012). As the temperature rises, more volatiles are lost and the carbon content increases and thus the adsorption ability of the material increases with pyrolysis temperature. The BOD removal was highest at biochar 300 °C and lowest at biochar 500 °C. Ding *et al.* (2014) notes that high adsorption of biochars prepared at low temperatures is as a result of the key role of the present surface functional groups while for biochars prepared at higher pyrolytic temperature, their efficiency is as a result of abundant pore spaces playing a dominant role through intra-particle diffusion.

The percentage reduction of Na⁺ and K⁺ ions for biochar 300 °C and Biochar 400 °C was relatively the same but a significant difference ($p \leq 0.05$) in the percentage reduction in biochars 500 °C at 65.43% and 89.36% for Na⁺ and K⁺, respectively, was recorded and thus more of K⁺ was adsorbed at biochar 500 °C. This can be as a result of the difference in the ionic radius of the two metals with potassium having a higher ionic radius of 1.38 Å compared to 1.02 Å of sodium metal. Komnitsas *et al.* (2016) studied the adsorption of

lead and copper metals on biochar and noted a higher percentage adsorption for lead metal as compared to copper metal with the ionic radius of the two being 1.19 Å and 0.73 Å, respectively. The adsorption on the biochar could also be attributed to the difference in surface net charge of the sorbent material leading to the specific selectivity of the sorbent material towards the cations as well as the electrostatic and complexation mechanisms (Laube & Reza, 2016; Samsuri *et al* 2013; Xu *et al.*, 2013; Tong *et al.*, 2011;).

Biochar made from municipal biowaste achieved a COD reduction of 90% which was attributed to the biochar's adsorptive ability and its specific high surface area (Manyuchi *et al.*, 2018). The difference in the retention time, pyrolysis contact time, biochar biomass as well as the specific surface area may be the cause of the difference in reduction potential when compared to the potential achieved in this study. Hernandez *et al.* (2007) reported a 40% reduction in COD anaerobically using the UASB reactor and noted that the low percentage removal could be as a result of the presence of surfactants in the greywater which have a negative effect in anaerobic processes (Elmitwalli *et al.*, 2001).

After the greywater was treated using the banana stalks biochars, there was a significant reduction in the final concentration levels of the study parameters when compared to the initial levels before treatment (Table 4.4). The level of K^+ reduced significantly at biochar 500 °C to levels within drinking water quality standards which stands at a recommended 10 mg/L (WHO, 2017). The levels of Na^+ were significantly reduced by biochar 500°C to acceptable levels for agricultural reuse which stands at below 920 mg/L (FAO, 1985). For agricultural reuse, reduction of soluble salts level in irrigation water is fundamental to controlling the soil salinity challenges (Quirk, 1986). Salinity may result from excess soluble salts and dominance of exchangeable sodium in the soil exchangeable complex which may result to difficulty in plants to extract water from the soil, nutrient imbalances, toxicities and reduction of water infiltration where sodium levels are high (Paz *et al.*, 2020; Kotuby-Achacer *et al.*, 2000; Bernstein, 1975). Thus, the reduced levels of the K^+ and Na^+ for the treated greywater rendered the water fit for agricultural reuse under the test conditions.

5.2 Conclusions

This study concludes that the quality of potable water was within WHO standards for quality drinking water. The physicochemical parameters monitored were within the acceptable ranges of 50 mg/L, 10 mg/L, 1000 $\mu\text{s}/\text{cm}$, 600 mg/L and 6.5 to 8.5 for Na^+ , K^+ , EC, TDS and pH, respectively. The quality of greywater from both male and female hostels had significantly higher levels of concentration for the same parameters when compared to potable water. The levels of Na^+ and K^+ in the greywater were high for both male and female greywater sources and therefore the need to reduce their concentration levels where the water was to be considered for long term agricultural use due to anticipated soil salinity challenges that would result.

The biochars prepared at different pyrolytic temperatures exhibited significant difference for the properties characterized. The pH became more alkaline with increase in pyrolytic temperatures with biochar 500 °C recording the most alkaline pH at 10.18. The mass decomposition increased with increasing temperature with highest decomposition rate experienced at temperatures between 250 °C and 400 °C. The yield of the biochars reduced significantly with biochar prepared at pyrolytic temperature of 500 °C recording a yield of 28.84% compared to a yield of 55.98% recorded at pyrolytic temperature 300 °C and 41.59% at pyrolytic temperature 400 °C. The present surface functional groups on the surface of the biochars reduced with increase in pyrolytic temperatures where some reduced in intensity, experienced a band shift or disappeared with the increase in pyrolytic temperature.

For the percentage removal of target pollutants in greywater, biochar 500 °C exhibited the highest adsorptive ability for reduction of Na^+ , K^+ and COD concentration levels, followed by biochar 400 °C and lastly biochar 300 °C. The reduction of BOD levels in the greywater was recorded to be highest in biochar 300 °C, followed by biochar 400 °C and lowest reduction was recorded in biochar 500 °C. After the adsorption process, the quality of treated water was within the levels for agricultural reuse with biochar 500 °C reducing the Na^+ and K^+ ions significantly to levels that would not affect the soil's salinity over long term use of the water.

5.3 Recommendations

5.3.1 Recommendations from this study

This study recommends that:

- i. Embu Water and Sanitation Company Ltd. (EWASCO) should maintain the quality of potable water supplied to the university of Embu. The greywater from students' hostels should be treated to reduce the levels of pollutants to palatable levels for reuse.
- ii. The University of Embu and other interested institutions can use pyrolytic temperature 500 °C in order to produce quality biochar product for use as an adsorbent.
- iii. Due to the adsorptive ability shown by the banana biochar, the University of Embu and other interested parties should consider using banana stalks to prepare biochar for use in greywater remediation.

5.3.2 Recommendations for further studies

This study recommends:

- i. Further study to optimize the performance of the banana stalks biochar and investigate the adsorption capacity of the material.
- ii. Further research on use of banana biochar in greywater remediation to guide in policy formulation.
- iii. A cost benefit analysis to be carried out to determine the economic viability of using the material as an adsorbent.

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