THE CONSTRUCTION AND ADSORPTION

EFFICIENCY OF A CARBON PRE-FILTER FOR THE

REMOVAL OF HEAVY METALS FROM DRINKING

WATER IN DEVELOPING AREAS

By

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Abstract: Pollution is one of the leading cause of death in the developing world. Unlike natural contaminants, heavy metals do not undergo biological decay and persist in the environment. Therefore, their remediation is considered a challenge. Further research has developed new, economical and safe technologies to provide household treatment devices like the Bio-sand water Filter (BSF) to procure decentralized quality drinking water. Although the BSF is effective on bacteria, it cannot remove heavy metals.

The objective of this study was to reduce mortality and illness caused by heavy metals in drinking water, specifically lead and cadmium, by focusing on field methods to efficiently produce and chemically treat coconut husk carbon without expensive equipment and use it as a pre-filter for BSF to remove heavy metals.

The carbon filter material was derived from widely available coconut husk processed by heating and chemically treated using an experimental method with commercial grade muriatic acid (31.45%) which increased the uptake capacity of the carbon. The surface area of the treated carbon was determined using the Quantachrome analysis. Acid treated carbon housed in a filter body was tested to pre-filter water for the BSF. Three different concentrations of aqueous solutions of cadmium and lead were prepared to test the efficacy of the pre-filter. The final concentrations of the heavy metal ions in solution were determined using Inductively Coupled Plasma (ICP) analysis.

From this study it was proved that the acid treated carbon pre-filter helps in reduction of heavy metals present in water and maintained their values in a range close to the permissible value of lead and cadmium in drinking water which is 0.05 mg/l and 0.005 mg/l respectively, recommended by World Health Organization (WHO).

This water treatment method may be implemented to improve the chemical quality of water and to make safe, potable water for people living in developing areas from a less expensive raw material like coconut husk.

Keywords: water, pre-filter, heavy metals, carbon

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CHAPTER I

INTRODUCTION

LACK OF CLEAN DRINKING WATER

It is estimated that 2.6 billion individuals around the globe need access to sanitation, with 1.1 billion lacking access to an enhanced drinking water source (United Nations 2014; World Health Organization 2010). Due to this individuals are at hazard for an assortment of medical issues.

Dominating medical issues connected with restricted or no entrance to clean drinking water, sanitation, and cleanliness, incorporate diarrheal maladies, helminthes, schistosomiasis, heavy metal poisoning, and trachoma. It is assessed that 90% of the 3.4 million demises every year ascribed to the absence of clean drinking water and poor sanitation are kids less than 5 years old (World Health Organization, 2017b).

The contamination of water with novel emerging organic compounds (EOC) is significant to public health (Pal and others 2014). Recent research has revealed that these compounds include pesticides and metabolites, surfactants, algae toxins, taste and odor compounds, (Richardson 2003; Shannon and others 2008).

All these have been shown to have toxic effects (Houtman 2010). Microbial contamination of drinking water also causes infectious diseases. Heavy metals from industrial used water also co-exists as contaminants along with the above (Kadirvelu 1998).

Exposure of human beings to drinking water contaminated with all these components is definitely a risk for human beings. So water treatment, source control and study of contaminants are of prime importance to provide safe and clean drinking water in households. Figure 1 below shows the environmental conditions in developing areas.

Picture courtesy: Dr. Timothy Bowser; Location: Tanzania (East Africa)

Figure 1. **Environmental conditions in developing areas**

REGULATIONS

Most countries have their own specific rules and regulations for the organization of wastewater and drinking water. The United States takes after guidelines set by the Environmental Protection Agency (EPA), while distinctive countries without legislative drinking water frameworks, tend to take after rules and regulations set by the World Health Organization (WHO).

BIO-SAND FILTERS

In poor countries, especially in rural areas, a lack of management and inferior infrastructure of centralized water supply is very common. Decentralized house hold treatment methods can be used to improve the chemical quality and microbial safety of drinking water in such situations. This would provide safe potable water for poor at the domestic level (Sobsey 2002). One such promising technology is the bio-sand filter containing some additional biological material as filtration media (Murcott 2002). It is an economical household water treatment system (HWTS) which has been in use for many years. The extensive literature survey shows that in poorly developing areas four household devices: the bio-sand filters (BSF), bucket filters (BF), ceramic candle filter (CCF) and silver impregnated porous pot filter (SIPP) are used (Mwabi and others 2011).

Point-of-use (POU) drinking water treatment allows people to improve the quality of water at home and take control of the safety of their drinking water (Sobsey 2002). One of the most promising affordable, decentralized emerging POU technologies is the bio sand filter (BSF), to reduce the concentration of enteric bacteria, viruses, protozoa and chemicals such as Fe in water (Elliott and others 2008).

HEAVY METALS

Heavy metals also referred to as the transition metals, belong to the d-block of the periodic table. Due to over population and expansion of industrial activities, heavy metal contamination in the water supplies has increased in recent years. The major source of contamination of natural water with heavy metals is industrial wastes (Harrison and Laxen 1980). The most common poisonous metals are Cd, Zn, Pb and Ni (Low and Lee 1991). These lethal metals go into the water bodies through waste water from metal plating and Cd-Ni battery industries, phosphate compost, mining and stabilizer amalgams, incorporating lead in petrol, mechanical effluents, and draining of metal particles from the dirt into lakes and waterways by corrosive rain. In addition, they emerge from the filtration of metals, for example purifying of minerals, the preparation of nuclear fuels, and electroplating (LifeExtension 1998). They hasten into soil, underground water, and surface water. Dissimilar to natural contaminants, heavy metals don't undergo biological decay and hence their remediation is considered a challenge.

To date, a couple of thousand publications have reported different aspects of heavy metals in drinking water, including the types and amounts of metals in drinking water, their sources, human introduction and their bioaccumulation, potential dangers and their expulsion from drinking water. Many developing nations are confronted with the test of lessening human introduction to heavy metals, basically because of their constrained monetary abilities to utilize propelled advances for heavy metal evacuation. (Chowdhury and others 2016).

Treatment processes like adsorption, precipitation and ion exchange have been used for removal of metals pollutants like zinc, arsenic, cadmium, mercury etc. Adsorption, using natural materials like coir pith carbon, (Kadirvelu and others 2001) immobilized biomass (Mohan and Pittman 2007) and silica activated carbon (Karnib and others 2014) have been reported to be economical and effective.

Lead (Pb) can influence the central nervous, renal, hematopoietic, cardiovascular, gastrointestinal, musculoskeletal, endocrinological, reproductive, neurological, developmental, and immunological frameworks (ATSDR 2015). Amid mental health, the level of lead in the blood (PbB) meddles with the trimming and pruning of neural connections, movement of neurons, and neuron/glia connections. Changes of any of these procedures may bring about the inability to build up fitting associations amongst structures and in the long run result in permanently altered functions (ATSDR 2015). Lead is radiated into the environment by the ignition of fossil fuels, the purifying of sulfide metals, and in lakes and streams by corrosive mine waste (Bodek and others 1998; Goyer and Chsolon 1972; Manahan 1984; Nordberg 1990). The drinking water quality level prescribed by World Health Organization (WHO) and American Water Works Association (AWWA) for lead is 0.05 mg/L (WHO 2004). The maximum allowable lead in drinking water has been set at a concentration of 15 ppb by the U.S. Environmental Protection Agency (EPA, National Primary Drinking Water Regulation, Washington, 2002).

Cadmium (Cd) is highly toxic and harms the kidneys and bones. It is best known for its relationship with itai–itai infection (Kasuya 2000). Cd amasses in people, bringing on erythrocyte obliteration, sickness, salivation, loose bowels, and muscular spasms, renal degradation, incessant pneumonic issues, and skeletal distortion (Mohan and Singh 2002). The major sources of cadmium discharge are the assembling of amalgams, batteries, colors and plastics. Mining and refining forms are additionally huge sources of cadmium (Tsezos 2001; Ho and Ofomaja 2006). In many ground waters that contain bicarbonate/carbonate anions, the aqueous speciation of cadmium incorporates many complexes with bicarbonate/carbonate. Cadmium carbonate can be a dissolvability control for some high alkaline environments that contain high cadmium pollution (Mohan and others 2007).

Above pH 11, all cadmium exists as its hydroxo-complex but cadmium ions tend to hydrolyze at pH values < 8 (Bodek and others 1998). Cd(II) prevails in fresh water at pH 6–8. $CdOH^+$, $Cd(OH)_2$, $Cd(OH)^-_3$, $Cd(OH)^{2-}_4$ likewise exist depending on the pH (Mohan and others 2006; Bodek and others 1998). The chlorocomplexes, $CdCl^+$, $CdCl_2$ and $CdCl^-_3$, prevails in ocean water and Cd(II) is present in very minute amounts (Bodek and others 1998), which ranges between 0.2–3.2 µg/L (Zhang and others 2015). The drinking water rule for Cd, suggested by the WHO and AWWA is 0.005 mg Cd/L.

ACTIVATED CARBON

Any solid substance can be used as an adsorbent. Adsorbents effortlessly draw in high molecular weight organics, low molecular weight and non-polar compounds and ions. Low molecular weight, highly polar compounds are not as readily adsorbed (Cecen 2011).

Activated carbon is one of the most commonly utilized adsorbents for wastewater and drinking water applications because of its large specific surface area, reaching up to approximately 1000 m^2/g (Hendricks 2006), capacity to expel organic compounds and its ability to grow bio-layer. Activated carbon helps in the removal of organic matter, especially natural organic matter (NOM), synthetic organic compounds (SOC), total organic carbon (TOC), as well as particulates, metal ions, odor, taste, and excess chlorine (Hendricks 2006). Removal was also demonstrated using isotherms like Freundlich or Langmuir, the former is used more commonly (Hendricks 2006).

Activated carbon is divided into two categories based on particle diameter. Adsorbent particles greater than approximately 0.3 mm in diameter are considered granular activated carbon (GAC) and diameters smaller than 0.3 mm are considered powdered activated carbon (PAC).

Activated carbon for water treatment purpose was utilized for the first time in rapid filters in 1875, although the patents for its utilization in filtration techniques began appearing in the early 1900s, particularly for the control and expulsion of taste and odor. Powdered activated carbon (PAC) was the favored form of activated carbon until the 1960s when the Advanced Water Treatment Research Program was shaped by the U.S. General Health Association (Hendricks 2006; Cecen 2011). Trials on the use of activated carbon adsorption strategies to supplant natural/biological treatment in the 1930s were unsuccessful, however, systems were changed to current day techniques using both the technologies in succession (Hendricks 2006). Either order of technologies is possible and can proficiently treat the desired water source, however, the most ordinary setup uses natural/ biological treatment before activated carbon filtration due to the over-burdening of the activated carbon with organic films.

PROJECT OBJECTIVES

The lack of accessibility to clean drinking water for people living in underdeveloped areas in many parts of the world stemmed the inspiration for this research project. Heavy metal contamination of water due to urbanization and industrialization has been one of the major causes of deterioration of water quality in recent times. Heavy metals above the permissible limits can cause adverse health effects and can also be fatal in some cases. This study was initiated to identify an inexpensive way to remove heavy metals from water, to make it suitable for drinking.

The major objectives of this study are:

1. To use a naturally occurring, inexpensive agricultural waste product which is a widely available source of carbon as a biosorbent material.
2. To use a low cost and simple char generation method to produce the adsorbent.
3. To utilize easily available acids for the chemical modification of the biosorbent.
4. To use a low cost and simple method to construct the pre-filter system.

5. To test the removal efficiency of the chemically modified biosorbent as a pre-filter for the removal of heavy metals (lead and cadmium) from water.

In addition to the above, other research goals included:

1. Comparison of the surface area of the chemically modified biosorbent produced to the untreated biosorbent and commercially available granular activated carbon, Calgon carbon (F-300) by the BET (The Brunauer-Emmett-Teller) surface area analysis method.

2. Determination of the effect of initial metal concentration on the adsorption capacity of the pre-filter.

3. Determination of the saturation capacity of the pre-filter.

CHAPTER II

REVIEW OF LIETRATURE

DRINKING WATER QUALITY STANDARDS

Drinking water quality standards which define the quality parameters have been set up by a wide range of organizations everywhere throughout the world. In the United States, the Environmental Protection Agency (EPA) directs and makes laws about drinking water. For countries without an authoritative or regulatory structure for such standards, the World Health Organization (WHO) decides rules on the standards that ought to be accomplished.

ENVIRONMENTAL PROTECTION AGENCY

The EPA was established under President Nixon on 2 December, 1970, with the underlying reason for research, observing, setting benchmarks, and implementation (Environmental Protection Agency 1992). Currently, the EPA principally manages air and water contamination, waste, cleanup, toxics and different chemicals, and green advancements (Environmental Protection Agency 2012). The Safe Drinking Water Act (SDWA) which enforces regulations on drinking water was passed by Congress in 1974 to control public drinking water supply and was changed in 1986 and in 1996 in order to provide protection to water sources.

(Environmental Protection Agency 2012b). Water contaminants can be broken into six groups namely; disinfectants, disinfection byproducts, inorganic chemicals, microorganisms, natural chemicals, and radionuclides (Environmental Protection Agency 2009).

WORLD HEALTH ORGANIZATION

When diplomats met to shape the United Nations in 1945, they decided to set up a worldwide health organization. WHO (World Health Organization) was established on 7 April 1948, with its headquarters in Geneva, Switzerland (World Health Organization 2017a). The WHO has played many important roles in human health such as the eradication of small pox. Presently it is concerned about the following: communicable diseases, in particular HIV/AIDS, Ebola, malaria, tuberculosis; the mitigation of the effects of non-communicable diseases; sexual and reproductive health, development and aging; nutrition, food security and healthy eating; occupational health; substance abuse; and driving the development of reporting, publications, and networking. To help ensure general wellbeing of constituents, WHO has developed standards for air, water, waste and much more. Water criteria are divided into synthetic, microbial, and radiological features, each with its own particular set of standards.

PERMISSIBLE LIMITS OF LEAD AND CADMIUM IN DRINKING WATER

BY EPA AND WHO

"WHY LEAD AND CADMIUM?"

Many developing areas in India and Africa suffer from lead and cadmium poisoning with no meaningful remediation efforts. Hence, this project was taken up to work on a technique to reduce these heavy metals in water.

LEAD (Pb) STANDARDS

A growing toxin and dangerous even in tiny amounts, lead is associated with several ailments like laziness, loss of hunger, stomach torment, constipation, anemia, slow loss of motion in the muscles, and can also be lethal. Lead is used principally in the production of lead-acid batteries, solder and alloys. From a drinking water point of view, the universal use of lead compounds in plumbing fittings (like PVC pipes) and as solder in water dissemination systems is important (Moore 1988). Lead compounds leach into the water resulting in high lead concentration in drinking water.

If 1 in 10 tests of a public supply surpasses 15 µg/L, the USEPA prescribes treatment to evacuate lead and observing of the water supply for lead content (Environmental Protection Agency 1991). The drinking water standards set by EPA has two levels of protection: The maximum contaminant level goal (MCLG) is zero and is the level determined to be safe by toxicological and biomedical considerations, independent of feasibility; EPA's final rule establishes an action level, set at 15 µg/l or 0.015 mg/l (World Health Organization 2008).

The 1958 WHO International Standards for Drinking-water recommended the maximum allowable concentration of 0.1 mg/l for lead, based on health concerns. This value was lowered to 0.05 mg/l in the 1963 International Standards. Provisional tolerable weekly intake of 25 μg/l lead per kg body wt or 93.5 μg/kg body wt/day for all age groups was established (WHO 1993).

CADMIUM (Cd) STANDARDS

Cadmium is also a toxin of growing concern that is extremely lethal. Cadmium is known to promote renal arterial hypertension but high concentrations may cause liver and kidney damage, or even anemia, retarded growth, and death. Cadmium metal is utilized in the steel business, plastics and as a part of batteries. Cadmium is discharged to the earth in wastewater, furthermore,

diffused contamination is brought about by tainting from manures and nearby air contamination. Pollution in drinking-water may also be caused by impurities in the welds, zinc of galvanized pipes and some metal fittings. Food and smoking are the main sources of daily exposure to cadmium.

According to the EPA, the maximum contaminant level for cadmium in drinking water is 0.005 mg/L (ATSDR 2008). The 1958 WHO International Standards for Drinking-water did not include cadmium. The 1963 International Standards recommended a maximum allowable concentration of 0.01 mg/l, based on health concerns. A guideline value of 0.005 mg/l was recommended for cadmium in drinking-water, in the first edition of the Guidelines for Drinking-water Quality, published in 1984 (World Health Organization 2008).

BACKGROUND OF BIO-SAND FILTERS

The BSF developed by Dr. David Manz of the University of Calgary during the 1990 is a modification of slow sand filters (SSF) used throughout the world. This filter combines settlement, straining, filtration, removal of chemicals as well as removal of microorganisms to produce safe water. It is economical to construct, operate and maintain. It uses an active biological layer to devour pathogens and effectively removes Giardia and Cryptosporidium oocysts that are resistant to chlorine disinfection.

A bio-sand filter is constructed in various modified ways. It can be made from plastic bucket (Mwabi and others 2011) or concrete (Ali Baig and others 2011; Bowser and others 2016). Gravel, coarse sand, fine sand/ zeolite/ pinus bark biomass and a biological layer is used layer by layer (Elliott and others 2008; Mwabi and others 2011). Figure 2, shows the construction of a bio-sand filter housing using concrete.

Figure 2. **Construction of Bio-Sand Filter using concrete**

ACTIVATED CARBON

Activated carbon is a crude form of graphite with a random or amorphous highly porous structure with a broad range of pore-sizes. Wallis and others (1974) had warned against the use of charcoal filters after seeing an increase in bacterial densities even in an overnight period of no use when the water is stagnant because the charcoal beds concentrate both bacteria and organic nutrients that are present in water at low concentration. Carbon filters aid in removal of organic compounds from water but may be less effective in removing microbial contaminants (Snyder and others 1995). The efficiency of activated carbons as adsorbents has been reported by Cheremisinoff (2002). Selective adsorption using activated carbon was seen as the first line of defense for water treatment. Preparation of activated carbon from various sources is given in Table 1 below (Mohan and Pittman 2007). Although most commonly used are wood, coal, lignite and coconut shell.

Table 1**. Alternative feed stocks proposed for the preparation of activated carbons**

Bones	Lampblack
Bagasse	Leather waste
Bark	Municipal waste
Beat-sugar sludge	Molasses
Blood	Nut shells
Blue dust	News paper
Coal	Oil shale
Coffee beans	Olive stones
Coconut shell	Petroleum acid sludge
Coconut coir	Pulp-mill waste
Cereals	Palm tree cobs
Carbohydrates	Petroleum coke
Cottonseed hulls	Petroleum acid sludge
Corn Cobs	Potassium Ferro cyanide residue
Distillery waste	Rubber waste
Fuller's earth	Rice hulls
Fertilizer waste slurry	Refinery waste
Fish	Reffination earth
Fruit pits	Scrap tires
Graphite	Sunflower seeds
Human hairs	Spent fuller's earth
Jute stick	Tea leaves
Kelp and seaweed	Wheat straw
Lignin and lignite	Wood

Activated carbons have an advantage of being used for a wide range of contaminants (Bhatnagar and others 2013) due to its versatility (Cheremisinoff 2002). The credit to develop activated carbon commercially goes to Von Ostrejko. Reports by other scientists on the preparation of activated carbon are also available – rice husk (Srinivasan and Sorial 2011), coconut tree saw – dust (Kadirvelu and others 2001) and from agricultural by products.

Activated carbon can be modified to change the chemical and physical attributes, by different techniques as given in Figure 3 (Yin and others 2007).
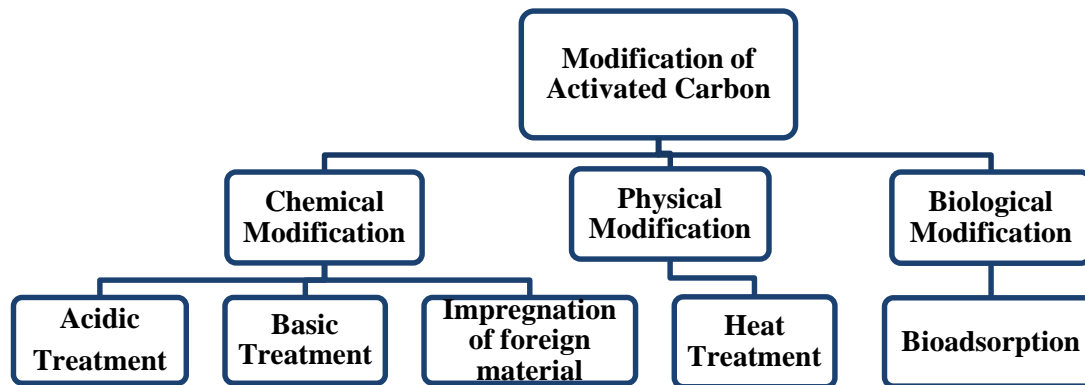
Figure 3. **Different modification techniques**

Table 2. **Advantages and disadvantages of modification techniques** (Yin and others 2007)

Modification	Treatment	Advantages	Disadvantages
Chemical characteristics	Acidic	Increases acidic functional groups on AC surface. Enhances chelation ability with metal species	May decrease BET surface area and pore volume Has adverse effect on uptake of organics May give off undesired SO_2 (treatment with H_2SO_4) or NO_2 (treatment with HNO_3) gases
	Basic	Enhances uptake of organics	May, in some cases, decrease the uptake of metal ions
	Impregnation of foreign material	Enhances in-built catalytic oxidation capability	May decrease BET surface area and pore volume

Table 2. **Advantages and disadvantages of modification techniques** (Yin and others 2007)

Physical characteristics	Heat	Increases BET surface area and pore volume	Decreases oxygen surface functional groups
Biological characteristics	Bio adsorption	Prolongs AC bed life by rapid oxidation of organics by bacteria before the material can occupy adsorption sites	Thick biofilm encapsulating AC may impede diffusion of adsorbate species

A review by Bhatnagar and others (2013) also gives the progress made in the modification of activated carbon. They stressed the importance of an acidic treatment (using HNO_3 and H_2SO_4) and a basic treatment (using inert H_2 and NH_3) and impregnation with metals like Ag and Cu. Acidic treatment of carbon is generally used in order to oxidize the porous carbon surface because it increases the acidic property, removes heavy metals because of the addition of acidic functional groups to the carbon surface and improves the hydrophilic nature of the surface. Basic (alkaline) treatment of AC produces positive surface charge which in turn is helpful to adsorb negatively charged species in higher amounts and is beneficial in enhancing the adsorption of especially organic species (like phenol) from water. The impregnation of AC with metals such as Ag, Cu, Al and Fe is gaining wide interest because of their significantly high adsorption capacity. Impregnated ACs have shown enhanced adsorption potential towards fluoride, cyanide and heavy metals like arsenic in water.

Also, microwave treatment and ozone treatment are few of the more novel methods that have been reviewed. Modification of AC by means of microwave radiation is gaining wide attention due to its capacity in heating at molecular level leading to homogenous and quick thermal reactions. One of the most widely used oxidants is ozone for the depuration of toxic organic compounds present in water.

Acidic treatment is favorable for higher uptake of metal ions and basic treatment is beneficial in enhancing the uptake of anionic species and organic based substances from aqueous solution (Yin and others 2007; Bhatnagar and others 2013).

COCONUT AS BIOSORBENT

Recently, biosorption techniques have emerged as a cost effective and proficient option for water and wastewater treatment. Biosorption techniques make use of naturally existing, horticultural waste materials which are less expensive, renewable and richly available. Various biosorbents have been evaluated for the elimination of different types of toxins from water (Davis and others 2003; Aksu 2005; Romera and others 2006; Volesky 2007; Vijayaraghavan and Yun 2008; Mathialagan and Viraraghavan 2008; Gadd 2009; Wase and Forster 1997).

Among a few of the horticultural waste products considered as biosorbents for water treatment, coconut has been of incredible significance as different parts of the tree (e.g. coir and shell) have been widely contemplated as biosorbents for the elimination of different sort of toxins from water. Coconut palm (Cocos nucifera) is an individual from the family Arecaceae (palm family) which is developed all through the tropical world. Coconut palms have been known as the "tree of life" as a result of the tremendous assortment of uses for fruit and byproducts. It is positioned seventh most critical vegetable oil crop on the planet (Bhatnagar and others 2010).

BIOCHAR PRODUCED FROM COCONUT HUSKS

AND

HEAVY METAL REMOVAL

There are various methods that have been reported for the production of biochar from coconut

husk and its chemical treatment to aid in the removal of heavy metals. The treated coconut

filaments give a superior execution than the unmodified fiber. De Sousa and others (2010)

functionalized the strands taken from coconut husks with the thiophosphoryl (P=S) group. This

resulted in the formation of stable complexes with Cd^{2+}, which were tested for their potential for

Cd^{2+} elimination. The adsorption isotherms for Cd^{2+} fitted well with the Langmuir display, with

binding capacities of 0.2–5 mmol/g at 25 °C (Bhatnagar and others 2010).

The adsorption of arsenic has likewise been considered utilizing coconut waste. Copper

impregnated coconut husk carbon (CICHC) was utilized for As (III) elimination (Manju and

others 1998). Most extreme adsorption limits were seen at pH 12.0. The adsorption limits

increased from 146.30 to 158.65 mg/g by raising the temperature from 30 to 60 °C which fitted

sensibly well to the Langmuir isotherm. Desorption contemplates uncovered that spent adsorbent

could be recovered and reused by 30% H_2O_2 in 0.5 M HNO_3 (Bhatnagar and others 2010).

Coconut husk has been investigated for the elimination of Cd(II), Cr(III) also Hg(II) particles

from fluid arrangements (Hasany SM and Ahmad 2006). It was found to expel the metal particles

existing at tiny levels from 84% to 96% in a single stage operation. The sorption information was

examined by applying diverse sorption isotherms. The estimations of the Freundlich constants,

1/n and Cm, were 0.92 and 52.6 mmol/g; 0.85 and 56.0 mmol/g; and 0.88 and 6.84 mmol/g for

Cd(II), Cr(III) and Hg(II) particles, respectively. The sorption process was observed to be

endothermic and unconstrained in nature with a weak bond arrangement between the metal

particles and coconut husk. Among different particles tried, just borate was found to lessen Cr(III)

sorption (<9%) while Hg(II) and Cd(II) sorption diminished to ~26% and ~35% within the sight

of thiosulfate and ascorbate particles, separately. A blend of ion exchange and surface complexation of metal particles was proposed as the conceivable mechanism for the sorption of Hg(II), Cd(II) and Cr(III) onto coconut husk (Bhatnagar and others 2010).

CHAPTER III

METHODOLOGY

The use of agricultural wastes like coconut husk for the removal of heavy metals is a green science technique for making our environment safe and clean (Kazemipour and others 2008). The heavy metal removal efficiency of the acid treated coconut husk carbon pre-filter was determined by preparing known concentrations of Pb and Cd solutions (500ml), passing them through the pre-filters at a fixed pH and at room temperature. The final concentrations of Pb and Cd in solution after passing through the pre-filter were determined using the ICP-OES technique. An attempt to activate and increase the surface area of the coconut husk carbon was made by treating it with muriatic acid (31.45%) solution and comparing the surface area to a commercially available activated carbon, Calgon Carbon (F-300) (Calgon Carbon Inc.).

CARBON SOURCE- COCONUT HUSK

Use of low cost adsorbents for water purification have become quite famous in the recent years (Johari and others 2016). High adsorption capacity of heavy metals from wastewater, has been exhibited by the residues from oil palm (Elaeis guineensis), rice (Oryza sativa L.) and coconut (Cocos nucifera L.) (Bhatnagar and others 2010; Johari and others 2013, 2014a, b; Sharma and others 2013; Song and others 2013).

Coconut husk, the rough outer shell or coating of the coconut is a generally accessible and bounteous natural material (Manju and others 1998). Activated carbon derived from coconut husk was utilized for the removal of phenols from water (Hitchcock and others 1983). In recent times, coconut husk has been used as an effective adsorbent material for removal of heavy metals from wastewaters.

PRODUCTION OF BIOCHAR FROM COMPRESSED COCONUT HUSK

"Why coconut husk?"

According to Kammen and Lew, "Half of the world's population uses biomass fuels for cooking. In 1992, 24 million tons of charcoal were consumed worldwide. Developing countries account for nearly all of this consumption, and Africa alone consumes about half of the world's production." (Kammen and others 2005). To state it more simply: charcoal is a common essential in the developing world. For the purpose of this study, we looked into another commonality in the developing nations, specifically: the coconut. Although people regularly consume coconut flesh, much of the coconut husk and shell are discarded. Considering that typical sources for biomass fuels result in deforestation, we turned to the coconut as a potential viable resource in the production of our biochar water filter.

The following step-by-step procedure for the production of coconut husk biochar is the result of one-on-one conversations with Mr. Marvin Collier, a self-proclaimed engineer-mountain-man from Stillwater, Oklahoma.

Step One: Acquiring Coconut Biomass

For the purpose of this experiment, compressed coconut fiber (used for animal bedding) was purchased from a local pet store. The husk was packaged as a solid briquette and as such, had to be broken down into smaller pieces in order to be effectively charred. We used a brand called EcoEarth (Figure 4).

Figure 4. **Compressed coconut fiber**

Step Two: Preparing the Coconut Biomass

We used a hammer and chisel, to pare down the compressed coconut into pieces no bigger than a US quarter and preferably no smaller than a pea (Figure 5). This allowed for faster burn-times and more evenly charred husk.

Figure 5. **Coconut husk pieces no bigger than a US quarter**

Step Three: Create an "Oven"

During our experiments, we made two different types of ovens: 1) a tin can with a tight fitting lid (Figure 6), and 2) a clean gallon-size paint can that was purchased from a local home improvement store. The only significant difference between these two "ovens" is reflected in the capacity of each container; cooking times tended to be nearly the same.

When possible, the lid of the "oven" should be secured. To accomplish this, we drilled holes in the opposite sides of a can and inserted a metal dowel through the holes as shown in Figure 6. The purpose of this dowel was two-fold: (1) it allowed handling of the can while it was hot and (2) it ensured that the lid remained on the can while cooking.

Lastly, a small hole was punctured in the center of the lid using a hammer and screwdriver (Figure 7). Importantly, this hole allowed gases to escape during the cooking process.

Figure 6. **Charcoal oven made using a tin can with a tight fitting lid and a metal dowel**

Figure 7. **Small hole punctured in the center of the lid to allow gases to escape**

Step Four: Cooking the Coconut Husk

The tin was filled 1/3 with coconut fiber pieces described in step 2 (Figure 8). It was important

not to overfill the tin so that burn times were minimized and even cooking was ensured. After the

lid was firmly secured, the tin can was placed over direct flame as shown in Figure 9.

As the coconut husk began to char, white smoke poured from the hole in the top of the can. Every

3 to 5 minutes the can was agitated by shaking it with the metal dowel. The heating process was

continued for 15 to 17 minutes until no visible gases escaped from the tin.

Figure 8. **1/3 of tin filled with coconut fiber pieces**

Figure 9. **Heating the tin over direct flame using a jet-boil backpacking stove**

Step Five: Cool and Confirm Even Char

After the heating process, the tin was removed from the flame and cooled in the air at ambient temperature. The contents of the can were emptied to visually confirm that all coconut fiber has become blackened. If the charcoal production has been done correctly, the majority of the coconut fiber will be the size of coarse sand or pea gravel and evenly dark black throughout (Figure 10).

Figure 10. **Charcoal production of good size and quality**

TREATMENT OF CARBON

The charcoal produced as given in the earlier section was passed through standard sieve sizes of 20 and 30 using a sieving equipment (Sieve shaker, 1612B, Advantech Duratap Sieve, Seedburo equipment company, Illinois, USA) at the Bioengineering Laboratory at Oklahoma State University (Figure 11). Even though the finely powdered carbon has higher surface area per unit mass and hence higher metal uptake capacity when compared to granular carbon, it is difficult for bio-sand filters, or other filter materials, to efficaciously separate out the finely powdered carbon from the filtered water (Cobb and others 2012). Hence, it is suggested that the pieces of carbon should be approximately between 0.5 to 1 mm in size, analogous to standard sieves 20-30.

Figure 11. **Sieving Equipment**

The chemical treatment of the carbon is an important process that is purported to increase the metal uptake capacity of the carbon. About 25 g of the carbon that was sieved and separated was soaked in 625ml of Muriatic acid for 4 hours. After the soaking period, the adsorbent was separated using Whattman filter paper (P8 Grade, Fisherbrand, Pittsburg, USA) and rinsed with

deionized water. The washed, treated carbon was then placed in an oven to dry at 110° C
overnight (Nadeem and others 2006) (Figure 12).

Figure 12. **Chemical Treatment of carbon using muriatic acid**

SURFACE AREA ANALYSIS

After the chemical treatment of the carbon sample, the surface area was determined using a
Quantachrome Autosorb (model iQ AG) analysis Instrument (Quantachrome Instruments,
Boynton Beach, Florida) located at the School of Geology and Physics, University of Oklahoma
(Figure 13). A commercial granulated carbon, Calgon carbon (F-300, Pennsylvania, USA) was
purchased and its surface area was compared to the chemically treated carbon.

The specific surface area of activated carbon was measured using nitrogen adsorption data at 77
K with a Quantachrome surface area analyzer. It works on a flowing-gas technique in which the

analysis gas flows into a tube containing the adsorbent and into a balance tube at the same time and provides quick and precise sample analysis for solid materials (Anirudh and Sreekumari 2011; Nadeem and others 2006). Two samples at a time can be degassed/ analyzed. In our study degassing of the samples was carried out overnight at 30-50 degrees C.

The Brunauer-Emmett-Teller (BET) method is the most widely used procedure to determine the surface area of solid materials and involves the use of the following BET equation.

$$\frac{1}{W((P/P_0)-1)} = \frac{1}{W_m C} + \frac{C-1}{W_m C}\left(\frac{P}{P_0}\right)$$

Where W is the weight of gas adsorbed at a relative pressure, P/P_0, and W_m is the weight of adsorbate containing a monolayer of surface coverage. The term C is the BET constant, and is associated to the energy of adsorption in the first adsorbed layer and consequently, its value is an indication of the magnitude of the adsorbent/adsorbate interactions.

The BET equation involves a linear plot of $1/[W(P/P_0)-1]$ vs P/P_0 which for most solids, using nitrogen as the adsorbate, is confined to a limited region of the adsorption isotherm, generally in the P/P_0 range of 0.05 to 0.35. This linear region is moved to lower relative pressures for microporous materials (Washington University 2010). BET surface area of the sample was calculated using the BET isotherm equation by assuming the area of a nitrogen molecule to be 0.162 mm^2 (Anirudh and Sreekumari 2011).

Figure 13. **Quantachrome Autosorb Analysis Instrument**

PREPARATION OF THE METAL SOLUTIONS FOR TESTING

Analytical Reagents (AR) grade chemicals were purchased from Oklahoma State University (Stillwater) and EMD Millipore (California). About 1000 ppm stock solutions of cadmium and lead were prepared using cadmium (II) sulfate [$3CdSO_4.8H_2O$] and lead (II) nitrate [$Pb (NO3)_2$] salts respectively by dissolving proper amounts of each of the heavy metal salts in deionized water **(A.1.1)**. A total of three concentration strengths (2, 10, 30 ppm) of cadmium and lead were prepared from the stock solutions **(A.1.2)**. The pH of the solutions was adjusted to 7 by using 0.1 N NaOH and 0.1 N HCl solutions (Kazemipour and others 2008), which is in the range of recommended pH of drinking water.

CONSTRUCTION OF PRE-FILTER AND EXPERIMENTAL DESIGN FOR ADSORPTION STUDIES

Keeping in mind the three R's- Reduce, Recycle and Reuse, one of the goals of our project was to construct a pre-filter made out of materials that are inexpensive, widely available and reusable. Hence a 500 ml plastic soda bottle was used to house the treated carbon and made to work as a pre-filter because they are easier to handle and work, within a lab scale environment. Also, 500 ml is a commonly used standard for amounts of fluids in chemistry.

The base of the 500 ml, plastic soda bottle was horizontally cut at about 8 cm from the bottom and the remaining body of the bottle was used as a filter body. In this 5 grams of coconut husk was used to hold 25 grams of treated carbon which prevents the carbon from completely mixing up with the water being passed through and also helps in establishing the flow rate which was measured to be 75 ml/min. The cut base of the bottle was used to diffuse the water flowrate by making 11 holes using a nail and a hammer, it was then placed over the body of the bottle as shown in Figure 14. The diffuser kept the water from making flow channels and pathways through the carbon while pouring in water. A cardboard box was used to support the filter body by cutting circular holes to fit the mouth of the bottle. A clean 500 ml beaker was placed below the mouth of the bottle to collect the filtrate (Figure 15).

Three different concentrations of Pb and Cd solutions (2 ppm, 10ppm and 30 ppm) were chosen to test the efficacy of the filter over a range which covers low, medium and high concentrations of heavy metals when compared to the allowed permissible levels in drinking water.

500 ml of each of the above concentrations at room temperature and a flow rate of about 75 ml/min were poured through the filter body. The filtered water was collected in the beaker below (Figure 16).
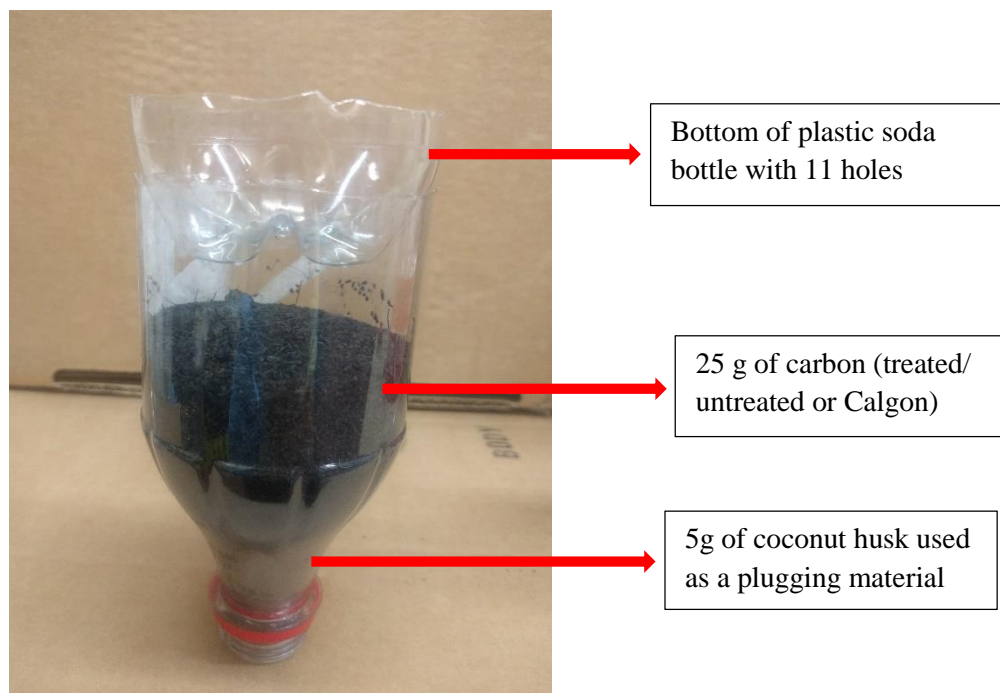
Figure 14. **Pre-filter model**

Adsorption of heavy metals by the acid treated charcoal was tested in batch experiments which were designed in triplicate. The average of the three trials was recorded and reported. Hence 19 of the above described experimental set-ups including one control (deionized water without any metal ions added) were organized for testing Pb and Cd treated water.

Figure 15. **Batch Experimentation set up**

Figure 16. **Pouring water through the filter bodies**

SATURATION CAPACITY OF ACID TREATED COCONUT HUSK CARBON PRE-FILTER

In order to obtain the saturation capacity of the acid treated coconut husk carbon, three portions, each 500 ml of 2 ppm, 10 ppm and 30 ppm Pb or Cd solutions (one at a time) were passed sequentially thrice through three different pre-filters for each concentration. After each step, the removal efficiency (%) was calculated.

REMOVAL EFFICIENCY DETERMINATION

After filtration, the cadmium and lead ions remaining in the solution were analyzed using the Inductively Coupled Plasma Optical Emission Spectroscopy technique (ICP-OES) at the Soil, Water and Forage lab at Oklahoma State University. The removal efficiency percent (RE%) of the activated carbon pre-filter on each cation was determined using:

$$RE\% = \left[\frac{C_0 - C_e}{C_0}\right] \times 100$$

where C_0 and C_e are the initial concentration and final concentration at equilibrium (after passing through the treated carbon pre-filter) of cation solution (mg/L), respectively (Kazemipour and others 2008).

INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROSCOPY TECHNIQUE (ICP-OES)

The residual concentrations of Pb and Cd in solution after passing through the pre-filters were determined using the Inductively Coupled Plasma Optical Emission Spectroscopy Technique (ICP-OES) which is a very well defined method to analyze metal elements found even in traces. It uses the emission spectra of a sample to identify and quantify the elements present. The OES instrument has the capacity to measure the relative amounts of up to 60 elements in a single

sample run in less than a minute. The instrument analyses the samples three times and averages all three readings. The ICP-OES analysis for this study was done at the Soil, Water and Forage analytical lab, Oklahoma State University, Stillwater using the Spectroblue ICP-OES spectrometer (SPECTRO Analytical Instruments, Germany) (Figure 17).
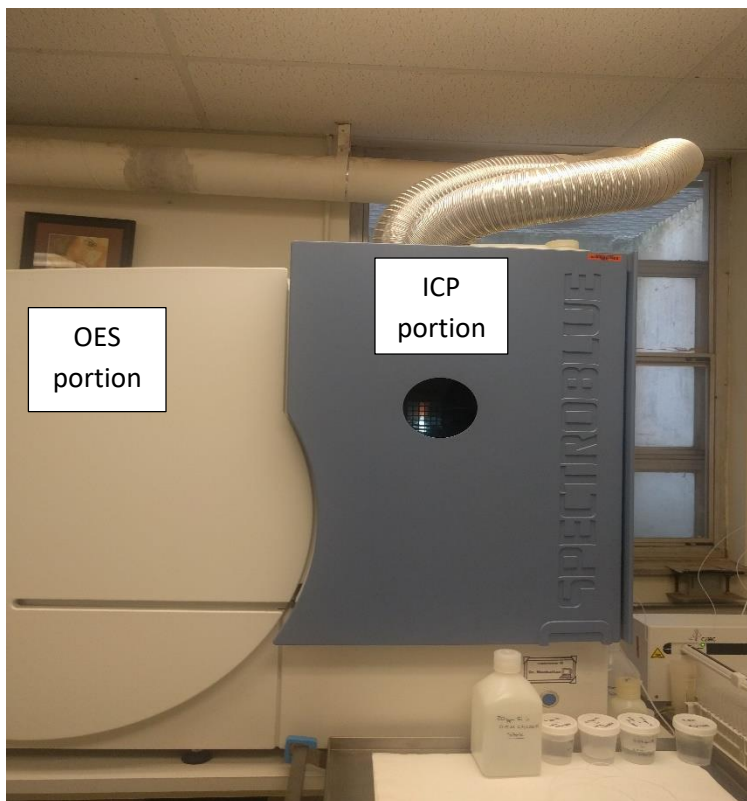
Figure 17. **The ICP-OES at the Soil, Water and Forage lab in Oklahoma State University**

The Environmental Protection Agency (EPA) standard protocol for ICP-OES sample analysis was followed (Environmental Protection Agency 1994). After calibration of the instrument following the standard protocol, ~5ml of sample solutions containing the residual heavy metals after passing through the pre-filters were introduced into the plasma, then nebulized into the core of an inductively coupled argon plasma. The temperatures were maintained at 9000 K so that the nebulized solution was vaporized, and the analyte species were atomized, ionized and thermally excited (Figure 18).
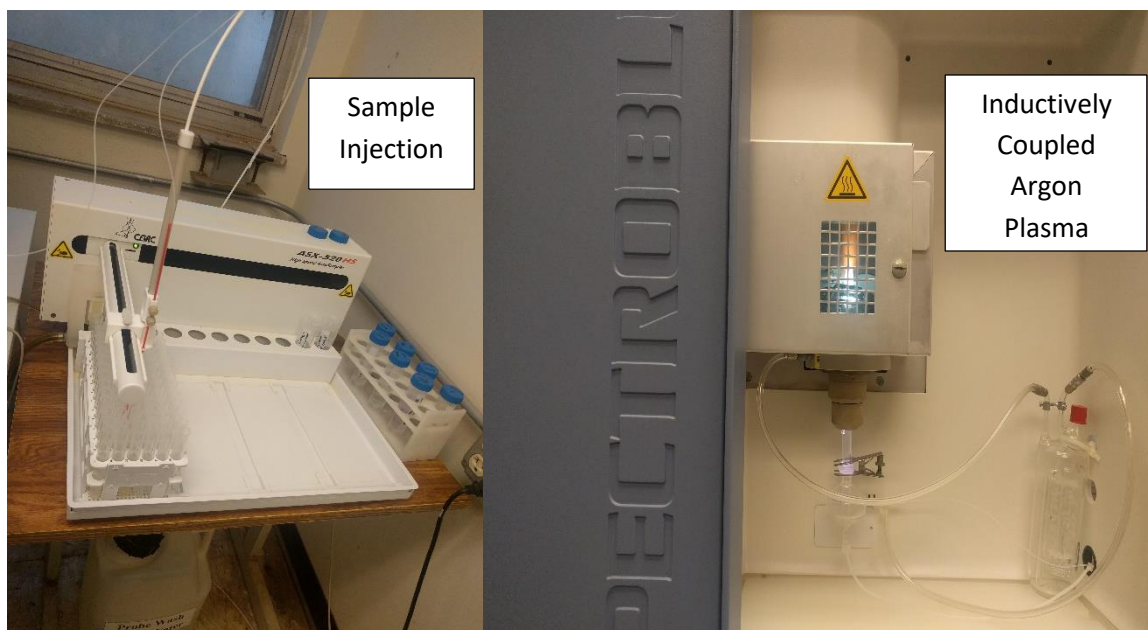
Figure 18. **Samples Injection and Inductively Coupled Argon Plasma**

This allowed the detection and quantification of the samples with an optical emission spectrometer (OES), which measured the intensity of radiation emitted at the element-specific, characteristic wavelength (for Pb – 220.353 nm and for Cd – 214.438 nm) from thermally excited Pb and Cd ions. The intensity thus measured was converted to elemental concentration by comparison with the calibration standards.

Note: The reported concentration values for Pb and Cd in this study are rounded at two 2 significant digits, to be consistent with the accuracy and precision of the ICP-OES results for the standard solutions prepared. The standard error bars are shown in the graph and the values for the same are given in Appendix **A.2.**

CHAPTER IV

RESULTS AND DISCUSSION

The results of this study indicated that the use of the plastic soda bottle as the pre-filter body in the reduction of Pb and Cd in drinking water was a fairly good system.

COST OF PRE-FILTER MADE FROM COCONUT HUSK VERSUS STANDARD BRITA FILTER

The materials used for constructing the acid treated coconut husk pre-filter are all reusable waste products which are obtained for free. The only cost incurred in constructing the pre-filter for this study was the acid used for treating the carbon (neglecting the cost of labor) and the fuel for heating the coconut husk. The total cost of a pre-filter is almost half when compared to a Brita filter which clearly indicates that the former is more economical (Table 3).

Table 3. **Cost Comparison between coconut husk pre-filter and Brita standard Filter**

Filter	Parts and cost ($)	Total Cost ($)
Coconut husk pre-filter	Coconut Husk – 0 Plastic Soda Bottle- 0 Muriatic acid – 4/ 40gallons	4
Brita Filter	Brita standard filter – 7.99/40gallons	7.99 (brita.com)

The other advantage of using the acid treated coconut husk pre-filter is that the charcoal can be easily removed from the filter, dried in the sun and then reused for cooking purposes. This is not a possible option while using a Brita filter, because the carbon is permanently closed in the filter body.

REMOVAL OF Pb AND Cd METAL IONS BY THE PRE-FILTER AND EFFECT OF INITIAL METAL CONCENTRATION

The data in Table 4 shows the effect of different concentrations of Pb and Cd (one at a time) on the removal efficiency of the acid treated coconut husk pre-filter. From Table 4 and Figure 19, it can be inferred that at low heavy metal concentration, the removal percentage was high and gradually decreased with the increase of heavy metal concentration, this trend was also reported by Erdem and others (2004) and Wu and others (2008). According to El-Ashtoukhy and others (2008), at low initial metal concentrations, the metal ions will be adsorbed by specific sites of the carbon, while with increasing metal concentrations, the specific sites get saturated and the exchanges sites are filled resulting in decreased removal efficiency.

At 2 ppm of heavy metals, the removal efficiencies of the pre-filter were 79.83 % and 82.33 % for Pb and Cd respectively. The removal percentage for cadmium was higher than lead at all

concentrations. On the contrary, the highest heavy metal concentration (30 ppm) led to the lowest

removal efficiency of the coconut husk pre-filter (27 % and 43 % for Pb and Cd respectively).

Table 4. **Removal efficiencies of Pb and Cd with different Initial Metal Concentrations using the treated coconut husk pre-filter**

Heavy metal	Initial Metal Concentration (C_0) (ppm)	Final Metal Concentration (C_e) (ppm)	Removal Efficiency (%)
Lead (Pb)	2	0.40	80
	10	5.5	45
	30	22	27
Cadmium (Cd)	2	0.35	82
	10	4.7	53
	30	17	43

Figure 19. **Removal efficiencies of Pb and Cd with different initial metal concentrations using the treated coconut husk carbon pre-filter**

Removal efficiencies of Pb and Cd were also calculated with an initial metal concentration of 30 ppm using the untreated coconut husk carbon and Calgon Carbon (F-300) to see how efficiently the pre-filters would work at the highest concentration level in this study. We had limited resources available and 30 ppm was the worst case scenario. The results are as shown in Table 5, Figure 20 and Figure 21.

Table 5. **Removal Efficiencies of Pb and Cd with 30 ppm Initial Metal Concentrations using untreated coconut husk carbon pre-filter and Calgon carbon (F-300) pre-filter**

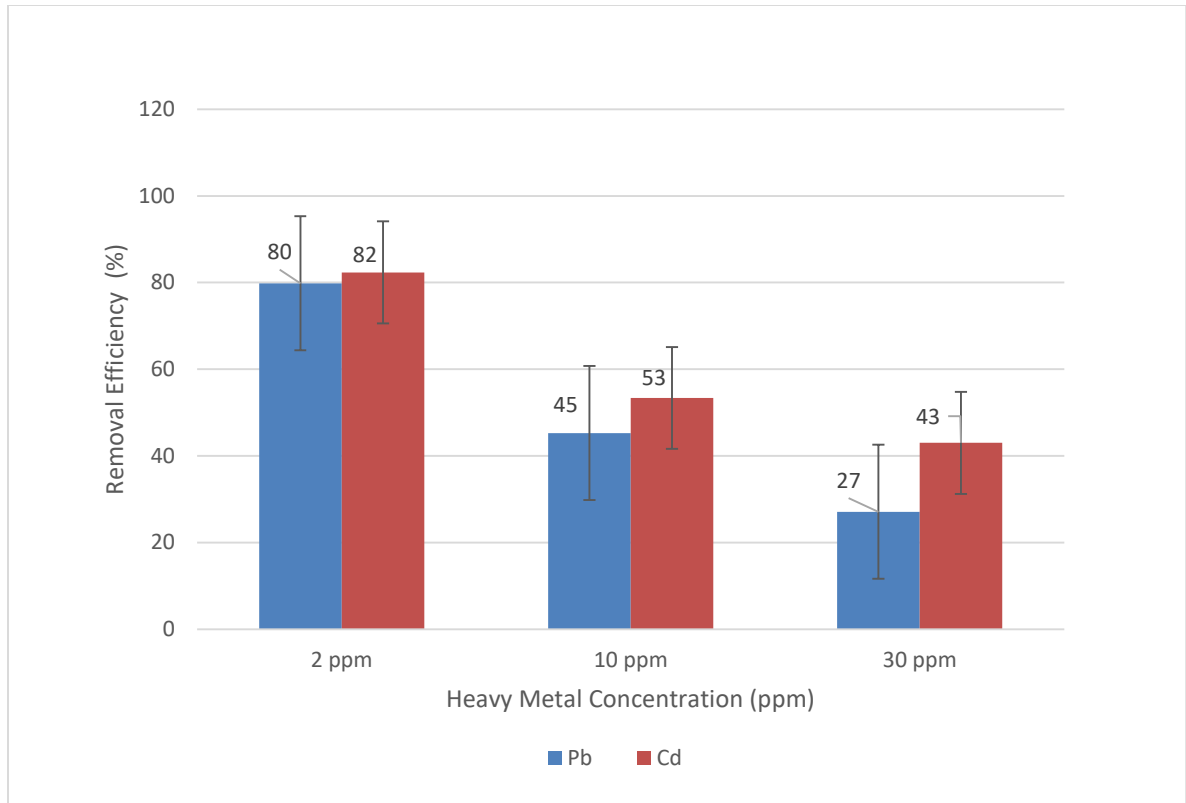| Type of Carbon	Heavy Metal	Initial Metal Concentration (C_0) (ppm)	Final Metal Concentration (C_i) (ppm)	Removal Efficiency (%)
Untreated Coconut Husk Carbon	Pb	30	28	6.9
	Cd	30	27	11
Calgon Carbon (F-300)	Pb	30	15	51
	Cd	30	15	49
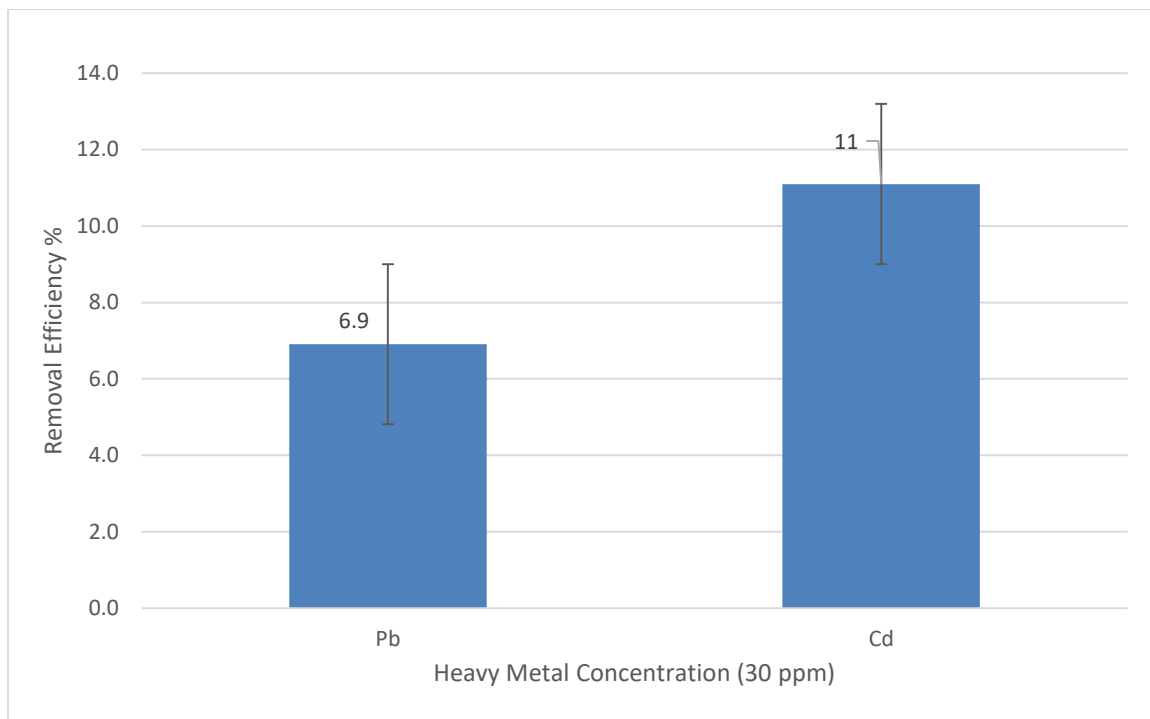
Figure 20. **Removal efficiencies of Pb and Cd with 30 ppm initial metal concentrations using untreated coconut husk carbon pre-filter**
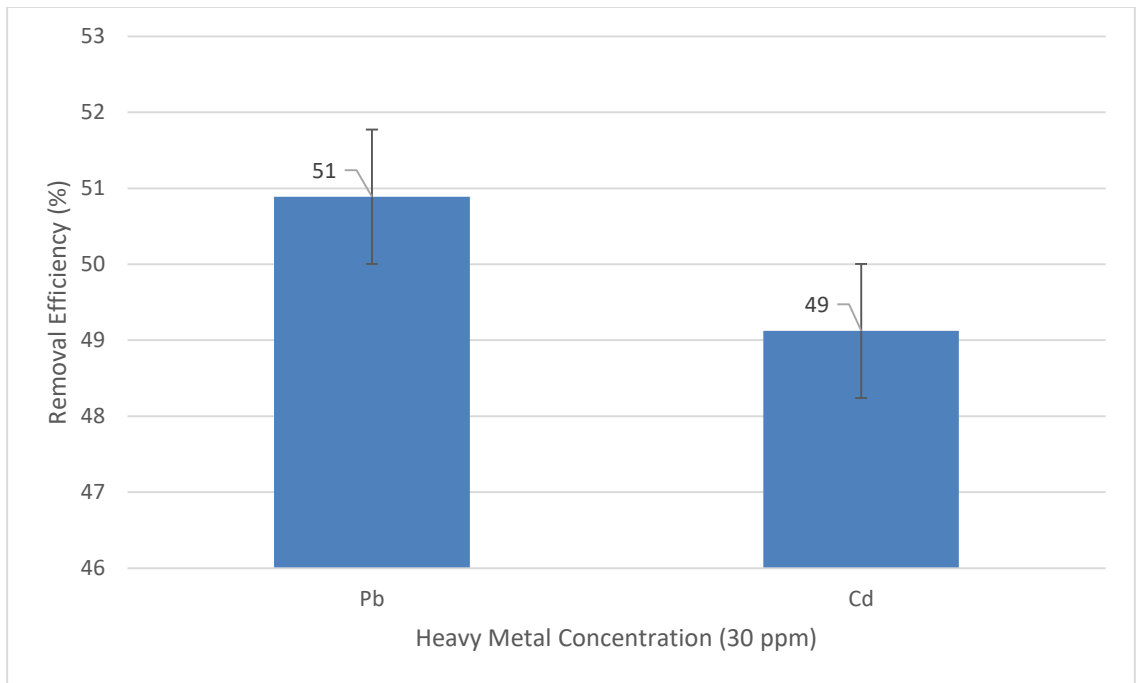
Figure 21. **Removal efficiencies of Pb and Cd with 30 ppm initial metal concentrations using Calgon carbon (F-300) pre-filter**

According to the literature review, acid treatment of charcoal helps in facilitating the chelation of metals and from the above tables and graphs, it is clear that the muriatic acid treatments helped in the removal of heavy metals when compared to the untreated coconut husk carbon. This is because acids help in increasing the acidic functional groups on the surface of the adsorbent, which enhances the metal chelation ability (Yin and others 2007).

The commercially available activated carbon- Calgon carbon (F 300) showed better removal efficiency for Pb than Cd. It can also be inferred that the acid treated coconut husk carbon pre-filter was almost as equally effective as the commercially available carbon pre-filter for an initial concentration of 30 ppm for Cadmium. The removal efficiency of Cd using the treated carbon pre-filter was calculated to be 43 % while that of Calgon carbon was 49 %.

EFFECT OF ACID TREATMENT ON BRUNAUER–EMMETT–TELLER (BET)

SURFACE AREA OF CARBON

It has been reported that any chemical modification of the biochar should help in activating the carbon and hence increasing the surface area. In this study, the muriatic acid treated coconut husk carbon along with untreated coconut husk carbon and the commercial carbon were sent to School of Geology and Physics, University of Oklahoma for carrying out the BET surface area analysis using the Quantachrome technique. As seen in Table 6, the acid treatment was not successful in the activation of the carbon and hence there was not much difference in the surface area before and after treatment. The Calgon carbon (F-300) on the other hand has a very large surface area as compared to the treated and untreated carbon.

Table 6. **The BET surface area values**

Adsorbent	BET Surface Area (m^2/g)
Untreated Carbon	29.16
Treated Carbon	31.55
Calgon Carbon (F-300)	703.72

SATURATION CAPACITY OF ACID TREATED COCONUT HUSK CARBON PRE-

FILTER

The saturation capacity helps us to determine the reusability of the same filter over a period of time as shown in Table 7 and Figure 22.

Table 7. **Effect of sequential use of the adsorbent to remove the Pb and Cd solutions (500 ml aliquots) using the acid treated coconut husk carbon pre-filter**

Heavy Metal	Initial Metal Concentration (C_0) (ppm)	Final Metal concentration (C_e) (ppm)	Removal Efficiency (%)
Pb	2	0.45	78
	2	1.3	38
	2	1.4	32
	10	5.4	47
	10	6.9	31
	10	7.8	22
	30	22	27
	30	24	21
	30	24	19
Cd	2	0.35	83
	2	0.80	60.
	2	0.90	55
	10	4.8	52
	10	5.9	41
	10	6.7	33
	30	17	42
	30	22	27
	30	22	27

Figure 22. **Effect of sequential use of the adsorbent on the Pb and Cd solutions (500 ml aliquots) using the acid treated coconut husk carbon pre-filter**

From Table 6 and Figure 20, it is clear that when the heavy metal solutions (in 500 ml aliquots) were passed sequentially thrice through the same pre-filter, the removal efficiency of the filter reduces due to the quick exhaustion of the adsorption sites on the adsorbent. The rate of percent removal is higher in the beginning due to a larger surface area of the adsorbent being available for the adsorption of the heavy metals. Cadmium again shows a better removal efficiency when compared to Lead when re-using the acid treated pre-filter.

Saturation capacities of the untreated coconut husk carbon pre-filter and the Calgon carbon (F-300) pre-filter were also determined in the same way using 500 ml aliquots of 30 ppm of Pb or Cd to see how efficiently the pre-filters would work at the highest concentration level in this study. We had limited resources available and given that 30 ppm was the worst case scenario, we did not repeat the test at other concentrations of heavy metals. The results have been shown in Table 8, Figure 23 and Table 9, Figure 24.

Table 8. **Effect of sequential use of the adsorbent on the Pb and Cd solutions 9500 ml aliquots) using untreated coconut husk carbon pre-filter**

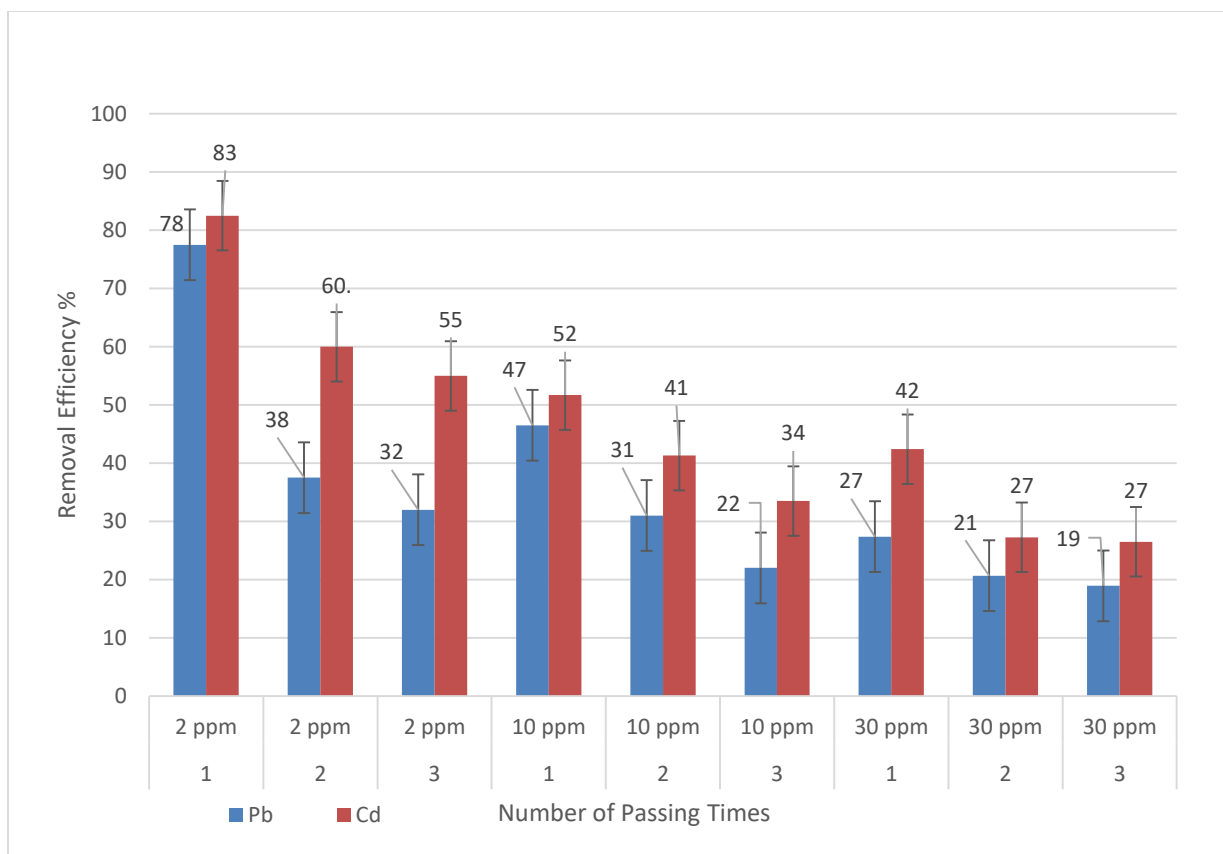| Heavy Metal	Initial Metal Concentration (C_0) (ppm)	Final Metal Concentration (C_e) (ppm)	Removal Efficiency (%)
Pb	30	27	9.1
	30	29	3.4
	30	30.	-0.070
Cd	30	26	12
	30	27	9.5
	30	28	5.7
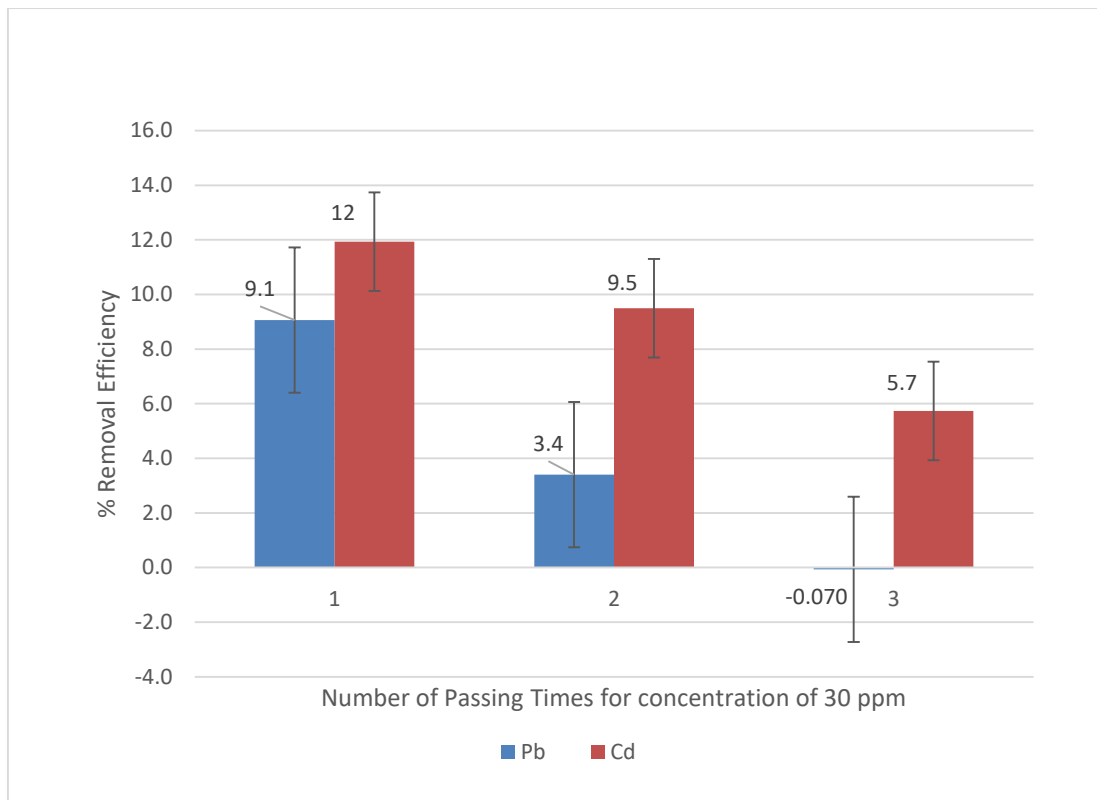
Figure 23. **Effect of sequential use of the adsorbent on the Pb and Cd solutions (500ml aliquots) using untreated coconut husk carbon pre-filter**

Table 9. **Effect of sequential use of the adsorbent on the Pb and Cd solutions (500 ml aliquots) using Calgon carbon (F-300) pre-filter**

Heavy Metal	Initial Metal Concentration (C_0) (ppm)	Final Metal Concentration (C_e) (ppm)	Removal Efficiency (%)
	30	16	48
Pb	30	16	48
	30	15	49
	30	16	46
Cd	30	17	43
	30	17	43

Figure 24. **Effect of sequential use of the adsorbent on the Pb and Cd solutions (500 ml aliquots) using Calgon carbon (F-300) pre-filter**

From the above results, we can deduce that the acid treated coconut husk carbon is more efficient than the untreated carbon for the removal of heavy metals (Pb and Cd) even when reused. While the commercially available carbon, Calgon (F-300) was more efficient than the acid treated coconut husk carbon for the removal of heavy metals (being slightly better for Pb than Cd). Also, the removal efficiencies stay almost the same even on re-using the pre-filter because the Calgon carbon has a much larger surface area when compared to the untreated or acid treated coconut husk carbons, therefore it has more adsorption sites available and a greater saturation capacity.

CHAPTER V

CONCLUSION AND RECOMMENDATIONS FOR FUTURE WORK

CONCLUSION

Urbanization and industrialization have led to the depletion of clean drinking water in developing areas where people lack access to potable water due to the dearth of resources. Use of agricultural waste products for the production of charcoal and activation of the same for the adsorption of heavy metals is a well-established technique used for treating contaminated waters. In this study, we used a readily available agricultural waste byproduct- coconut husk.

The conclusions of this study are listed in numerical order and correspond to the objectives listed in the introduction.

1. Coconut husk was identified as a low cost and widely available biosorbent material.

2. A simple, low cost char generation method was identified and tested for this study. The only requirements were a tin can and a cook stove.

3. The biosorbent was chemically modified using muriatic acid. Muriatic acid is an inexpensive material that is widely available in countries where concrete is used for construction. Muriatic acid improved the function of the biosorbent by adding acidic functional groups to the surface of the carbon and aiding in the chelation of the metal ions present in water.

4. The pre-filter body was built from recycled materials (plastic soda bottle) and met all requirements of the process: retained biosorbent, low cost, simple construction, controlled flow-rate and helped in removal of the heavy metals (Pb and Cd) from water.

5. The removal efficiency of the biosorbent as a pre-filter for the removal of heavy metals was proven to be very effective when the biosorbent was treated with acid especially at low concentration (2 ppm) of heavy metals. This is because acid treatment facilitates the metal chelation and at low concentrations the heavy metals are adsorbed by specific sites on the surface of the carbon, while increasing the concentration results in saturation of all the available sites present.

The char generation method, the muriatic acid treated coconut husk carbon production and the construction of pre-filter was very simple, quick, easy and inexpensive. The muriatic acid treated coconut husk carbon was fairly effective adsorbent and cost effective for the removal of Pb and Cd ions from aqueous solutions, especially at low concentrations of 2 ppm.

The surfaces area of the acid treated coconut husk chars (31.55 m^2/g) in this research was more than an order of magnitude less when compared to the commercially available activated carbons like Calgon carbon (F-300) (~ 700 m^2/g). Though the treatment with acid did not help in activating the charcoal and increasing the surface area, it did help in increasing the acidic functional groups on the surface of the adsorbent and increasing the metal chelation ability of the adsorbent.

The acid treated coconut husk carbon had a better removal efficiency for cadmium compared to lead. The initial metal concentration plays an important role in determining the removal efficiency of the pre-filter. At lower initial concentration (2 ppm), the removal efficiencies were greater but as the initial concentrations were increased the removal efficiencies decreased because of the quick exhaustion of the adsorption sites.

Also re-using the acid treated carbon resulted in reduced removal efficiency when compared to the commercial carbon (Calgon, F-300) as the commercial carbon has larger surface area, hence greater number of adsorption sites available for heavy metals to be adsorbed. Therefore, the Calgon carbon (F-300) has a better saturation capacity.

Using coconut husk to produce bio-char provides a less expensive raw material than the commercial carbon as well as a renewable one. The cost of removal is expected to be quite low as the adsorbent (coconut husk) is available in abundance and is cheap. Also, the cost of construction of the pre-filter is negligible as we are recycling plastic soda bottles to use as the filter body. Use of the pre-filter could help maintain the Pb and Cd in a range that is close to the levels set by WHO and EPA. It can be used in conjunction with a bio-sand filter to remove organisms and compounds that cannot be removed by biological treatment alone, that might possibly include pesticides, insecticides, and herbicides.

RECOMMENDATIONS FOR FUTURE WORK

There is still some scope of improvement for increasing the removal efficiency as well as increasing the surface area of the coconut husk biochar which will help in exploiting it commercially. Recommendations for future studies have been streamlined below.

1) Due to the inability of the muriatic acid to increase the surface area of the charcoal, steam activation or some other chemical or physical treatment methods maybe employed to help increase the surface area. This might help in improving the removal efficiency further and potentially improve filter performance to more regularly meet the WHO and EPA drinking water standards.

2) Muriatic acid maybe re-used after soaking the charcoal in it by using filtration method, which would help in reducing the cost of the pre-filter.

3) Testing the pre-filter by varying parameters like flow rate, pH, contact time and the amount of adsorbent used should also be studied.

4) Adsorption isotherm models like Langmuir and Freundlich can be constructed to determine the adsorption capacities of the filters.

5) Bacterial studies on the pre-filter can also be carried out to study the growth of bacteria on the carbon filters as well as determine the formation of biofilms and the life of a single pre-filter.

6) Testing for other obtainable agrarian waste byproducts as a raw carbon material for the production of activated carbon should be investigated for developing areas where coconut husk is not available.

7) The polluted waters contain many organic and inorganic impurities, the physico-chemical key parameters like pH and the initial metal concentration will also vary, field testing the pre-filter with the actual source of water should be conducted, to determine its practical removal efficiency.

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APPENDICES

A.1 HEAVY METAL SOLUTION PREPRATION

A.1.1. Preparation of 1000ml Standard Stock Solution

1) Lead Nitrate ($Pb(NO)_3$) – Dissolve 1.598 g in 100 ml DI water. Dilute to 1L in a volumetric flask with DI water. 1ml of this solution contains 1mg of Pb (Protocol according to Environmental Protection Agency).

2) Cadmium Sulfate Hydrate – Dissolve 2.282 g in 100 ml DI water. Dilute to 1L in a volumetric flask with DI water. 1ml of this solution contains 1mg of Cd (Protocol according to Environmental Protection Agency).

A.1.2. Dilution of Stock solutions to Prepare the required Concentrations of Pb and Cd Solutions

1) 1000 ml of 2 ppm Pb/ Cd solution –Take 2 ml (Pb/ Cd) from stock solution and dilute to 1000 ml using a volumetric flask.

2) 1000 ml of 10 ppm Pb/ Cd solution –Take 10 ml (Pb/ Cd) from a stock solution to dilute to 1000 ml using a volumetric flask.

3) 1000 ml of 30 ppm Pb/Cd solution – Take 30 ml (Pb/ Cd) from a stock solution to dilute to 1000 ml using a volumetric flask.

A.2 Inductively Coupled Plasma- Optical Emission Spectrometry (ICP-OES) Results

A.2.1. Standard Stock and Working solution ICP-OES Results

ppm	Lead	Cadmium
1000	988.88	990.83
2	1.83	1.95
10	9.58	9.62
30	29.49	29.89

A.2.2. 1CP-OES Results for 30 ppm Pb and Cd Solutions Passed through Untreated Carbon Samples

Pb	Cd
27.28	26.42
28.12	26.72
28.38	26.87
Average	**Average**
27.93	26.67
Standard error	**Standard error**
0.57	0.23

A.2.3. 1CP-OES Results for 30 ppm Pb and Cd Solutions Passed through Calgon Carbon (F-300)

Pb	Cd
15.54	16.25
14.68	14.98
13.98	14.56
Average	**Average**
14.73333	15.26333
Standard error	**Standard error**
0.78	0.88

A.2.4. 1CP-OES Results for Pb and Cd Solutions Passed through Treated Carbon

ppm	Pb	Cd
2	0.45	0.35
	0.39	0.32
	0.37	0.39
Average	0.40	0.35
Standard error	0.04	0.35
10	5.35	4.83
	5.42	4.56
	5.65	4.6
Average	5.47	4.66
Standard error	0.16	0.15
30	21.79	17.28
	22.01	16.95
	21.82	17.08
Average	21.87	17.10
Standard error	0.12	0.17

A.2.5. Effect of sequential use of the adsorbent on the Pb and Cd solutions (500 ml aliquots)

using untreated coconut husk carbon pre-filter

Heavy Metal	Initial Metal Concentration (C_0) (ppm)	Final Metal Concentration (C_e) (ppm)	Removal Efficiency (%)
	30	27.28	9.07
Pb	30	28.98	3.40
	30	30.02	-0.07
Standard error	1.38		
	30	26.42	26.42
Cd	30	27.15	27.15
	30	28.28	28.28
Standard error	0.94		

A.2.6. Effect of sequential use of the adsorbent on the Pb and Cd solutions (500 ml aliquots)

using Calgon carbon (F-300) pre-filter

Heavy Metal	Initial Metal Concentration (C_0) (ppm)	Final Metal Concentration (C_e) (ppm)	Removal Efficiency (%)
Pb	30	15.54	48.20
	30	15.63	47.90
	30	15.22	49.27
Standard error	0.22		
Cd	30	16.25	45.83
	30	16.98	43.40
	30	17.01	43.30
Standard error	0.43		

A.2.7. Effect of sequential use of the adsorbent on the Pb and Cd solutions (500 ml aliquots) using acid treated coconut husk carbon pre-filter

Heavy Metal	Initial Metal Concentration (C_0) (ppm)	Final Metal concentration (C_e) (ppm)	Removal Efficiency (%)
	2	0.45	77.50
	2	1.25	37.50
	2	1.36	32.00
Pb	Standard error	0.50	
	10	5.35	46.50
	10	6.90	31.00
	10	7.80	22.00
	Standard error	1.24	
	30	21.79	27.37
	30	23.80	20.67
	30	24.32	18.93
	Standard error	1.34	
	2	0.35	82.50
	2	0.80	60.00
	2	0.90	55.00
	Standard error	0.29	
Cd	10	4.83	51.70
	10	5.87	41.30
	10	6.65	33.50
	Standard error	0.91	
	30	17.28	42.40
	30	21.82	27.27
	30	22.05	26.50

VITA

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