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Removal of heavy metals from water by capacitive deionization electrode materials derived from chicken feathers

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REMOVAL OF HEAVY METALS FROM WATER BY CAPACITIVE DEIONIZATION ELECTRODE MATERIALS DERIVED FROM CHICKEN FEATHERS

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A Dissertation Submitted in Partial Fulfillment of the Requirements for the Degree of Master's in Materials Science and Engineering at Nelson Mandela African Institution of Science and Technology

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ABSTRACT

Capacitive deionization (CDI) is an emerging desalination technology based on the principle of electrical double layer capacitors. When the voltage is applied to the surface of the electrodes, electrodes become oppositely charged and ions are adsorbed onto the electrode surfaces under the presence of the electric field, thus producing a purified stream of water. Once the electrodes are saturated with ions, adsorbed ions can desorb from the surface of the electrodes when the applied voltage is reversed or removed. Electrode materials play an important role in CDI performance. To date, the porous carbon derived from biomass shows a competitive advantage in CDI practical applications because of their low production costs, availability, good electrical conductivity, large specific surface areas, and environmental compatibility. In this study a high surface area porous carbons were synthesized from chicken feathers through pyrolysis and KOH activation; the KOH: CF ratio (R) and activation temperature (T_a) were variable parameters. The carbon samples synthesized were characterized by SEM, FTIR spectroscopy and nitrogen adsorption-desorption isotherms at 77 K and desalination experiments were performed by using potentiostat/galvanostat. All samples except the untreated carbon exhibited type IV isotherms demonstrating the existence of mesopores. The lead (Pb^{2+}) removal test was performed with a CDI cell containing the fabricated carbon electrode and 100 mgL^{-1} Pb $(NO_3)_2$ solution; the sample prepared with the ratio R of 1:1 and T_a = 800 K exhibited higher Pb^{2+} removal efficiency of 81% and electro sorption capacity of 4.1 mgg^{-1} at the electrode potential 1.2 V and flow rate 5 $mLmin^{-1}$. Therefore, chicken feather derived carbon (CF) is considered a promising CDI electrode material for the removal of heavy metals from wastewater.

DECLARATION

I, Tusekile Alfredy, do hereby declare to the Senate of Nelson Mandela African Institution of Science and Technology that this dissertation is my own original work and that it has neither been submitted nor being concurrently submitted for degree award in any other institution.

....25/03/2019...........

Tusekile Alfredy **Date**

Name and signature of candidate

The above declaration is confirmed

....25/03/2019...........

Dr. Yusufu Abeid Chande Jande **Date**

Name and signature of supervisor 1

....25/03/2019...........

Prof. Tatiana Pogrebnaya **Date**

Name and signature of supervisor 2

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CERTIFICATION

We hereby confirm that the dissertation entitled "Removal of Heavy Metals from Water by Capacitive Deionization Electrode Materials Derived from Chicken Feathers" submitted by Tusekile Alfredy to Nelson Mandela African Institution of Science and Technology, Tanzania in partial fulfillment of the requirements for the award of Master of Science degree in Materials Science and Engineering is an authentic work and has been done under our supervision.

....	25/03/2019 ....
Dr. Yusufu Abeid Chande Jande	**Date**
Name and signature of supervisor 1	

....	25/03/2019 ....
Prof. Tatiana Pogrebnaya	**Date**
Name and signature of supervisor 2	

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DEDICATION

I dedicate this work to my lovely husband Joseph Gregory, my daughter Glory and my son Geovann and the rest of my family members.

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LIST OF ABREVIATIONS AND SYMBOLS

AC	Activated carbon
BET	Brunauer-Emmett-Teller
CDI	Capacitive deionization
CF	Chicken feathers derived carbon
C_o	Initial concentration
EDL	Electric double layer
EDLCs	Electric double layer capacitors
FTIR	Fourier Transform Infrared spectroscopy
HCl	Hydrochloric acid
H_2O	Water
KOH	Potassium hydroxide
Pb^{2+}	Lead ions
$Pb(NO_3)_2$	Lead nitrate
PTFE	Polytetrafluoroethylene
PVA	Polyvinyl alcohol
PVDF	Polyvinylidinefluoride
R	Carbon mass ratio
SEM	Scanning Electron Microscope
t	Activation time
T_a	Activation temperature

CHAPTER ONE

INTRODUCTION

This chapter contains general introduction, objectives, research problems, justifications, hypotheses and significance of the study. The general objective was to investigate the performance of capacitive deionization (CDI) electrodes derived from chicken feathers carbon for removal of Pb^{2+} from water. The general objective was comprehended through three specific objectives, materials characterization and capacitive deionization testing of the electrodes.

1.1 Background information

Despite the fact that two-third of surface worldwide is covered by water, but only 3% can be used directly for domesticity, agriculture and industrial purposes (Gao *et al.*, 2015). Currently, access to clean and fresh water is becoming the most severe challenges facing the world due to the increase in human population and industrial activities (Schwarzenbach *et al.*, 2010; Elimelech and Phillip, 2011).

Presently on earth's surface, there are more than seven billion people globally (U.S. and World Population Clock, 2018). In accordance to 2009 World Water Development Report, by 2030, increasing demand for freshwater is about 64 billion m^3 a year and nearly half of the global population will be living in regions of high water stress, as the global population is expanding by 80 million people annually (Population-Institute, 2010).

It is well known that, as population increases definitely human activities such as agriculture, industries, as well as mining, also are expected to increase. As the result, the natural resources of water become contaminated with different contaminants, and heavy metals are among of the toxic pollutants of the global concern as supported by Mdegela *et al.* (2009). Unlike some organic pollutants, heavy metals are non-biodegradable and cannot be decomposed, it tends to accumulate in living tissues and cause diseases and disorders such as human kidney damage, brain, nervous and reproductive systems problems (Meena *et al.*, 2005; Ge *et al.*, 2012; Huang *et al.*, 2016; Bora *et al.*, 2017).

Amongst the toxic heavy metals, lead (Pb^{2+}) has been categorized as a serious hazardous due to its effect to the human body if consumed above the recommended limit (Yang *et al.*, 2014). In accordance with the World Health Organization (WHO), the drinking water quality should contain less than 0.01 mg/L (0.01 ppm) of Pb^{2+} (WHO, 2017). Table 1 shows different water sources in Tanzania that have a higher level of Pb^{2+} concentration than the recommended limit.

Table 1: Concentration of Pb^{2+} in some of the surface water sources in Tanzania

Water sample	**Pb^{2+} concentration (ppm)**	**Reference**
Mwanza gulfs of Lake Victoria	0.022	Ogoyi *et al.* (2011)
Winam gulfs of Lake Victoria	0.823	Ogoyi *et al.* (2011)
Kichangani swamp area in Morogoro	0.3	Mdegela *et al.* (2009)
Centre at Mindu dam in Morogoro	0.05	Mdegela *et al.* (2009)
Msimbazi river in Dar Es Salaam	0.142	Mwegoha (2010)

Therefore, the removal of this metal from water is a focal point of recent researches. Until now, several technologies such as reverse osmosis, membrane filtration, chemical precipitation, ion exchange resins, and distillation are used for water desalination (Guadalupe *et al.*, 2008; Huang *et al.*, 2016). Despite the fact that each method has a number of advantages, they have got some limitations. For instance, reverse osmosis and thermal processes are considered for large-scale water desalination but their commercial application requires high initial cost, huge manpower, high energy consumption and production of secondary waste water (Porada *et al.*, 2012; Huang *et al.*, 2016; Chen, 2017; Liu *et al.*, 2019).

Therefore, new, innovative and sustainable desalination methods that are less costly and environmentally friendly are required to overcome these challenges. CDI is a promising and rapidly growing technology due to the fact that, it is easy to operate, environmentally friendly and energy efficient for brackish water (<10 000 mgL^{-1}) purification (Suss *et al.*, 2015). It is reported to consume 0.4 kWh m^{-3}, for total dissolved solids (TDS) removal for brackish water desalination (Jande, 2015; Peng *et al.*, 2016).

Water desalination by CDI is based on the principle of the electric double layer capacitors (EDLCs), where the charged ions such as Pb^{2+} and NO_3^- can be adsorbed from the aqueous solution and electrostatically stored on the oppositely charged electrode surfaces (anode and cathode) through the application of voltage from an external power source (Baroud, 2018). Once the external voltage is removed, the trapped ions can be released back to the solution, which results in regeneration of the electrodes. Chemical and physical properties of materials used for the electrodes play vital roles in the capacitive behavior.

To date, a wide range of materials such as carbon aerogel (Gabelich *et al.*, 2002; Hou *et al.*, 2013), activated carbon cloth (Huang *et al.*, 2016), activated carbon(AC) (Suss *et al.*, 2015; Feng *et al.*, 2018), carbon nanotubes (Tofighy and Mohammadi, 2010; Ma *et al.*, 2016), carbon nanofibers (Dong *et al.*, 2012; Wang *et al.*, 2012; Dong *et al.*, 2015; Wu *et al.*, 2015), nitrogen-doped carbon materials (Qian *et al.*, 2015), graphene (Qian *et al.*, 2015; Liu *et al.*, 2017), template mesoporous carbon (Zou *et al.*, 2008; Tsouris *et al.*, 2011) and the composite carbon materials (Dong *et al.*, 2014; Wu *et al.*, 2016; Ren *et al.*, 2018), have been used as electrode materials in CDI application. Though carbon aerogel and graphene met most of the specifications required for CDI applications, their practical application is hindered by their high cost (Machunda *et al.*, 2009) . Therefore more studies focusing on the development of renewable and less-cost electrode materials for CDI applications are needed (Lado *et al.*, 2016). Currently, various biomass wastes such as coffee residue, sugarcane bagasse, wood, coconut shells, rice husks, and chicken feathers attracted many types of research. These materials provide an alternative source for carbon precursor since they can be synthesized at very low cost, and possess high surface area, good electrical conductivity and suitable pore size distribution, wettability, chemical and electrochemically stability (Wang *et al.*, 2013; Yang *et al.*, 2015; Zhao *et al.*, 2015; Lado *et al.*, 2016; Liu *et al.*, 2019).

Approximately 24 billion chickens are slaughtered worldwide each year which brings to disposing of $1.8\times10^{9}\,kg$ of feathers waste. The huge amount of waste produced is either dumped directly to the environment causing soil and groundwater sources pollution or openly burnt to pollute the air causing health effects to human (Prasanthi *et al.*, 2016). Thus, studies were done to convert the feathers waste into materials for various commercial applications like adsorbents for water treatment (Guadalupe *et al.*, 2008; García-Sabido *et al.*, 2016) and electrode materials for EDLCs (Wang *et al.*, 2013; Zhao *et al.*, 2015). To the best of our knowledge, there have been no reported studies on the use of CF for CDI applications. Therefore, this study aims to evaluate chicken feather as a potential material for CDI electrodes, in particular for the removal of Pb^{2+} from the water.

1.2 Research problem

Various carbon materials have been investigated as CDI electrode materials for Pb^{2+} removal from water with high removal efficiency and electrosorption capacity. However, none of them was able to make cost effective electrode materials in terms of availability of raw materials and the method used for materials production. Hence, there is need to find renewable, easily available and effective electrode materials for CDI application. Chicken feathers are biodegradable, cost effective and renewable source of carbon that currently found several applications. It is used as electrode materials for supercapacitor as well as biosorbents for wastewater treatment. This is due to their interesting sorption properties such as high specific surface area, good pore structure (pore size and volume distribution), good electrical conductivity, wettability, chemical and electrochemically stability (Zhao *et al.*, 2015; García-Sabido *et al.*, 2016). However, no research has investigated the use of chicken feathers as electrode materials for CDI application. Therefore, this study aims to evaluate chicken feather as a potential material for CDI application, specifically for the removal of Pb^{2+} from the water.

1.3 Problem justification

Due to the fact that adequate clean and safe water demand in the world is constantly increasing, there is a need for searching alternative technologies which are efficient and environmental friendly for water treatment. CDI unit with electrodes derived from the renewable and easily available CF material is environmentally friendly and energy efficient technology for heavy metal removal from water specifically Pb^{2+}.

1.4 Objectives

The main objective of the study was to investigate the performance of CDI electrode materials derived from chicken feathers for removal of Pb^{2+} from the water.

The main objective was achieved by pursuing the following specific objectives:

(i) To synthesize and characterize carbon materials derived from chicken feather.
(ii) To fabricate CDI electrodes by using carbon materials derived from chicken feather.
(iii) To assemble CDI cell and test the electrosorption efficiency of different CDI electrodes based on the carbon materials derived from chicken feather for the removal of lead ions from water.

1.5 Research questions

(i) What are the properties of produced chicken feather carbon materials?

(ii) What is the electrosorption capacity (mg/g) of the CDI electrodes derived from chicken feather activated carbons?

(iii) What is the electrosorption efficiency of the fabricated CDI electrodes for the removal of Pb^{2+} from water?

1.6 The significance of the research

This research created knowledge in the use of chicken feather derived carbon as carbon precursor for CDI application specifically for the removal of Pb^{2+}. Chicken feathers are regarded as wastes which are either dumped directly to the environment or burnt. This lead to soil pollution, groundwater sources pollution also when openly burnt pollutes the air (Prasanthi *et al.*, 2016). Thus, converting into porous carbon is both an economical and environmentally friendly approach towards solving the water demand challenges facing the world on our time. It is reported that around 24 billion chickens are slaughtered worldwide each year which brings to disposing of $1.8\times10^{9}\,kg$ feather waste (Prasanthi *et al.*, 2016) that is sufficient to be used as raw material for large scale water treatment plantation.

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction

In order to overcome water scarcity and water quality issues that are the most challenges facing the global population currently, low cost and energy efficient desalination technologies become the focal point of recent researches. CDI is among of the promising and universally attracting methods because of its eco-friendliness, less energy consumption, and simplicity in regeneration and maintenance compared with other conventional techniques of desalination (Qichuan *et al.*, 2016; Gaikwad and Balomajumder, 2017; Wei *et al.*, 2017). However, the availability of CDI electrode materials with low-cost accompanied by the promising electrosorption efficiency hinders its practical applications. Thus, in order to improve the CDI performance, suitable CDI electrode materials which are renewable and easily available are needed to overcome the challenge. This review section describes different materials which have been used for CDI application specifically for Pb^{2+} removal and general challenges on CDI application.

2.2 Working principle of CDI technology

CDI is an electrosorption process in which ions are adsorbed onto the surface of porous electrodes under an electrical field. The positively charged ions such as calcium, magnesium, sodium, copper and lead adsorb onto the electric double layer (EDL) of negatively charged electrodes (cathode) while negatively charged ions such as sulfate, chloride, nitrate, cyanide, arsenate adsorb onto the EDL of positively charged electrode (anode) when aqueous solution is flowing between the porous electrodes. Once the adsorption capacity of the electrodes is achieved, adsorbed ions can desorb from the surface of the electrodes by either discharging the cell at 0.0 V or reversing the cell polarity (Marmanis *et al.*, 2013; Gaikwad and Balomajumder, 2017). The working principle of CDI technology is shown in Fig. 1.

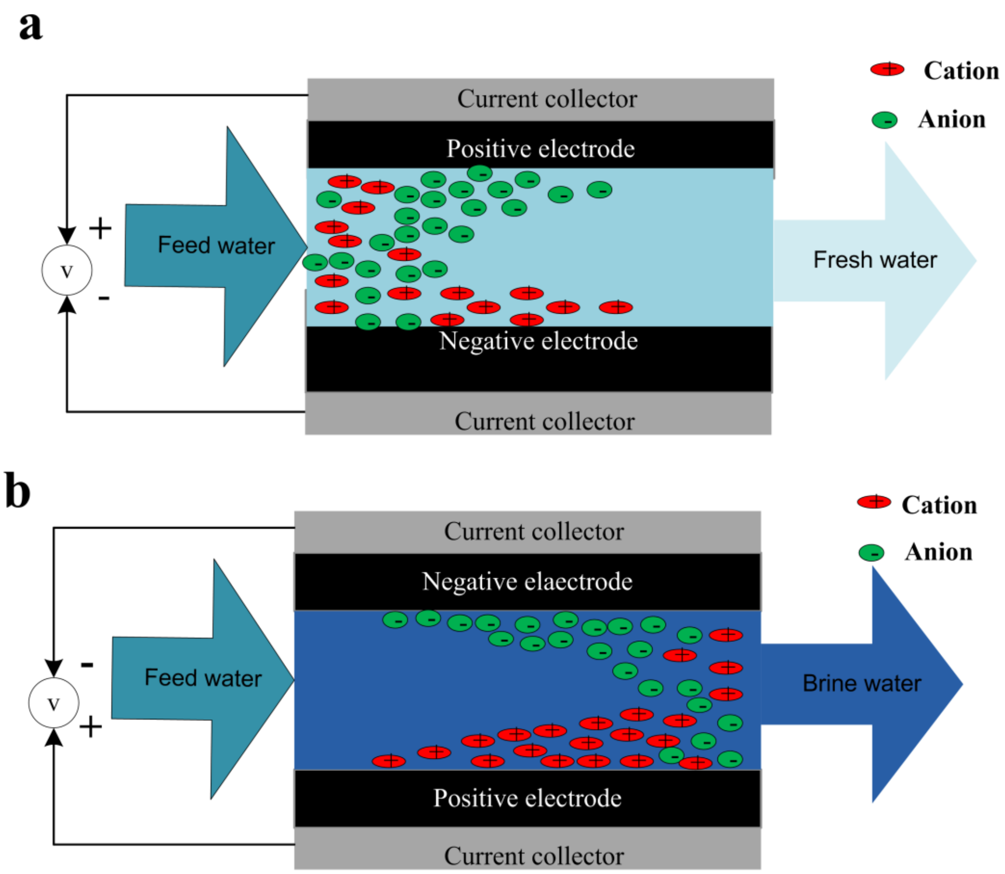

Figure 1: Schematic representation of the basic principle of CDI (a) Adsorption (b) Desorption.

2.3 Electrode materials for CDI application specifically for lead removal

It is well known that CDI performance usually depends on the physical and structural properties of the electrode materials such as high specific surface area, good pore structure (pore size and volume distribution), good electrical conductivity, wettability, chemical and electrochemically stability (Liu *et al.*, 2019). Relatively high specific surface area and better conductivity contribute to a better adsorption capacity (Gu *et al.*, 2015). Currently, effort and focus in CDI researches have been put into the development and synthesis of better electrode materials to improve the CDI performance. Up to now various kinds of carbon-based materials such as activated carbon cloth (Huang *et al.*, 2016), 3D graphene (Liu *et al.*, 2017), activated carbon (AC) (Xu *et al.*, 2014), Fe_3O_4/porous graphene nanocomposite (Bharath *et al.*, 2017), carbon nanotube (Yang *et al.*, 2014), O- doped BN nanosheet (Chen *et al.*, 2017), activated carbon with an ion-exchange membrane (Dong *et al.*, 2018). have been studied as CDI electrode materials for lead removal from aqueous solution.

Among these, graphene electrodes met most of the specification requirements for a CDI cell, but the high-cost limit graphene for practical use as an ideal electrode material in CDI technology, hence other literature has been dedicated to investigating the production of low-cost CDI electrodes to promote CDI technology (Marmanis *et al.*, 2013). Table 2 below shows a summary of the performance of various CDI electrode materials used for lead removal. The parameters considered are removal efficiency, initial concentration, voltage, time, the volume of the solution, flow rate, electrosorption capacity, size of the electrode and mode of operation.

Table 2: Performance of reported CDI electrode materials for the removal of lead ions from aqueous solution

Material	RE (%)	EC (mgg^{-1})	C_O (mgL^{-1})	Volt. (V)	Time (min)	Volume (mL)	Flow rate ($mLmin^{-1}$)	Approach /Mode	Size of electrodes	Reference
AC cloth	43	17.8	165.6	1.2	120	60	0	Batch	1 × 1 $inch^2$	(Huang *et al.*, 2016)
3D graphene	99.9	6.99	20	1.4	60	35	40	Batch	115 × 7 × 1 mm^3	(Liu *et al.*, 2017)
AC with ion-exchange membrane	80	0.18	1	1.2	60	1000	23	Single- pass	4.2 ×4.2 $inch^2$	(Dong *et al.*, 2018)
Carbon nanotube (activated by air plasma)	*-	3.40	10	0.45	5	200	4	Batch	30 ×50 mm^2	(Yang *et al.*, 2014)
Fe3O4/porous graphene nanocomposite	90	460	9.9	1.2	5	150	4	Batch	80 × 30 × 0.3 mm^3	(Bharath *et al.*, 2017)
O- doped BN nanosheet	*-	220	600	1.2	20	100	50	Batch mode	2 ×2 cm^2	(Chen *et al.*, 2017)

Note; *- Data not found, RE is removal efficiency, *C*o is initial concentration and EC is electrosorption capacity

Recently, researchers focused on biomass and agricultural wastes as the emerged possible option for carbon precursor because of its abundant raw materials, relatively low manufacturing cost, high specific area, good electrical conductivity and suitable pore size distribution (Wang *et al.*, 2013; Thambidurai *et al.*, 2014; Lado *et al.*, 2016).

Biomass is an important raw material for the production of valuable carbon materials because it is available in low cost and is an environmentally friendly renewable resource (Wang *et al.*, 2013), but they suffer from low electrical conductivity which hinders their electrosorption capacities. To improve electrical conductivity of the AC electrode, the conductivity-enhancing materials like carbon black and graphite powder may be added to the AC (Thamilselvan *et al.*, 2016), while the polymeric binders such as polyvinyl alcohol (PVA), polyvinylidene fluoride (PVDF) or Polytetrafluoroethylene (PTFE) are also important as it provides structural integrity such as good wettability, thermal stability and mechanical strength of the AC.

Several studies reported that activation process plays a great role in porosity development and surface functional groups of any kind of raw material. Activated carbon for CDI application is produced from various biomass materials such as rice husks, bamboo, sawdust, pine needles, coffee beans, cow dung, coconut shells, wood, sugarcane bagasse, and tea waste by subjecting it to either physical activation or chemical activation (Lado *et al.*, 2016; Gaikwad and Balomajumder, 2017). Due to the several possible raw materials and activation methods, AC can vary in quality, and thus suitable AC should be chosen based on their surface area, pore size and structure, and other relevant properties. AC electrodes can be treated to increase their electrosorption efficiency by modifying their properties according to the application.

Tea waste biomass activated carbon has been employed as CDI electrodes for Cr and fluorine removal, the percent removal were found 88.5% and 85.20% for 10 mg L^{-1} mix feed solution respectively (Gaikwad and Balomajumder, 2017), also it is reported that the carbonized eggshell membrane has been used as electrodes for supercapacitors and showed a high specific capacitance of 297 F/g and excellent reversibility with cycling efficiency of 97% after 10 000 cycles in 1 M KOH (Li *et al.*, 2012). Moreover activated carbons derived from jackfruit peels with chemical activation method using phosphoric acid as activating agent were studied and it is reported that the BET surface areas and total pore volumes of the carbons produced at temperature 450 and 550 $^{\circ}$C were in the range of 907–1260 m^2/g and

0.525–0.733 cm^3/g, respectively (Prahas *et al.*, 2008). Also activated carbons recycled from bitter-tea and palm shell wastes were used as the CDI electrode for the treatment of salt water, it possessed a promising specific surface area and the electrosorption efficiency of the produced electrodes increased up to 40% (Chen *et al.*, 2018). These few studies justify that abundant and environmentally friendly bio-waste materials can be evaluated for CDI electrode for water desalination purposes. In this study, CF was used as CDI electrode materials for lead removal from aqueous solution.

2.4 Chicken feather material for water treatment

The management of the huge amount of waste generated by poultry processing industries is a serious challenge facing the agriculture industries globally. Thus, the scientific convention of feather waste as renewable materials for various applications such as electrode materials for supercapacitor and biosorbents for wastewater treatment is both economic and environmental benefits. Several studies have been conducted to investigate chicken feather as a potential material for numerous applications due to its interesting properties such as high surface area, high capacitance and several reactive functional groups (Sun *et al.*, 2009; Zhao *et al.*, 2015). Chicken feather has been used as biosorbents for the removal of chromium ions from aqueous solution and the removal efficiency was approximately to 90% at 6 pH and the sorbent dose was 100 mg (Sun *et al.*, 2009). Chicken feathers were tested as biosorbents for colour removal of synthetic coloured water and it removed organic dye up to 80% (García-Sabido *et al.*, 2016). Chicken feather treated with alkaline solution were used to remove zinc and copper ions from waste water and the maximum uptake of zinc and copper ions was approximately to 0.16 and 0.14 $mmolg^{-1}$ respectively, where by the initial concentration was 5 $mgml^{-1}$(Al-Asheh and Banat, 2003). Chicken feathers were used as biosorbents for Pb removal from aqueous solution and the removal efficiency and sorption capacity was 8.3 mg/g and 81.6% respectively (Guadalupe *et al.*, 2008). Also chicken feathers were treated with NaOH and NaOCl to prepare a sorbents that were used to Ca^{2+}, Mg^{2+}, and Fe^{3+} and Mn^{2+} ions from contaminated water. Chicken feather treated with NaOH removed 93.38% Ca^{2+}, 72.55% Mg^{2+}, 97.13% $Fe^{3+}$and 95.66% Mn^{2+}, while chicken feather treated with NaOCl removed 92.14% Ca^{2+}, 71.85% Mg^{2+}, 94.26% Fe^{3+} and 93.57% Mn^{2+}, within 20-30 min respectively (Sayed *et al.*, 2005). Hence, these few studies justify that abundant and environmentally friendly bio-waste materials derived from chicken feather can be used as CDI electrode material for water desalination purposes. To the best of our knowledge, there have been no reported studies on the use of CF for CDI applications particularly for Pb^{2+}removal from water. In this study, CF was used as CDI electrode materials for lead removal from aqueous solution.

CHAPTER THREE

MATERIALS AND METHODS

3.1 Materials and reagents

Chicken feathers were collected from Kilombero poultry market in Arusha region, Tanzania. The chemicals including potassium hydroxide (KOH), lead nitrate ($Pb(NO_3)_2$), ethanol and hydrochloric acid (HCl) were of analytical reagent grade, Polytetrafluoroethylene (PTFE, 60 wt % dispersion in H_2O) and carbon black were purchased from Sigma Aldrich. Distilled water was purchased from Arusha Technical College, Arusha, Tanzania. All chemicals were used without any modification.

3.2 Preparation of chicken feather activated carbon

Chicken feathers were washed thoroughly with distilled water and then dried at 90 °C for 12 h in the oven, ground to powder and carbonized in a horizontal tube furnace (CTF 12/65/550) at 400 °C for 1 h at a heating ramp rate of 5 °C min^{-1} in the presence of nitrogen. The carbonized carbon char was chemically activated by KOH in the weight ratio KOH to the carbon (KOH: CF) of 1:1, 2:1, 3:1 and 4:1 at 600 °C, 700 °C and 800 °C under nitrogen flow for 3 h at a heating rate of 5 °C min^{-1}. The tube furnace was allowed to cool naturally at room temperature before activated products are removed out. The samples were washed by 1 M HCl to remove KOH and by hot distilled water to remove the residual KCl till neutral pH was attained and finally dried up in an oven at 100 °C for 12 h. The materials produced were denoted as CF and CF-R-T_a-t representing the chicken feather derived carbon and activated carbon derived from chicken feather respectively. The letters R, $T_{a,}$ and t represent KOH to carbon mass ratio, activation temperature and activation time (in hours) respectively. On the other hand, the untreated carbon was labeled as CF-400, 400 denoted carbonization temperatures. Figure 2 summarized the preparation process of activated porous carbon derived from chicken feathers.

Figure 2: Schematic illustration of the synthesis of chicken feather derived carbon.

3.3 Characterization of materials

The textural properties of the prepared CF samples; BET surface area, pore diameter and pore size distribution, were obtained from physical N_2 adsorption-desorption isotherm measurements at liquid nitrogen temperature 77 K by a surface area and porosity analyzer Quantachrome NovaWin ©1994-2013, Quantachrome Instruments v11.03. The BET surface area was calculated by using the Brunauer-Emmet-Teller (BET) equation at the relative pressure (p/p_0) between 0.05-0.35 while the pore size distribution was obtained by Barret-Joyner –Halender (BJH) method. Prior analysis, samples were degassed at 120 °C for 3 h. The morphology of the samples was studied by a field emission scanning electron microscope (FE-SEM) (JSM-7600F Thermo NORAN System 7) while the functional groups analysis was conducted by Fourier transform infrared (FTIR) spectroscopy.

3.4 Fabrication of CDI electrodes

The electrodes were prepared by mixing activated CF powder, carbon black, and Polytetrafluoroethylene (PTFE) dispersion in H_2O suspensions 60 wt%, at a weight ratio of

8:1:1 in ethanol. The mixture was stirred for 2 h to ensure homogeneity and dried at 80 ºC to form a dough-like paste, then the paste formed was pressed to required thickness and cut into squares of the size of 3 cm×3 cm, then dried at 50 °C for 12 h in the oven before testing to remove the remaining solvents. The fabrication process of the CDI electrode is presented in Fig. 3.

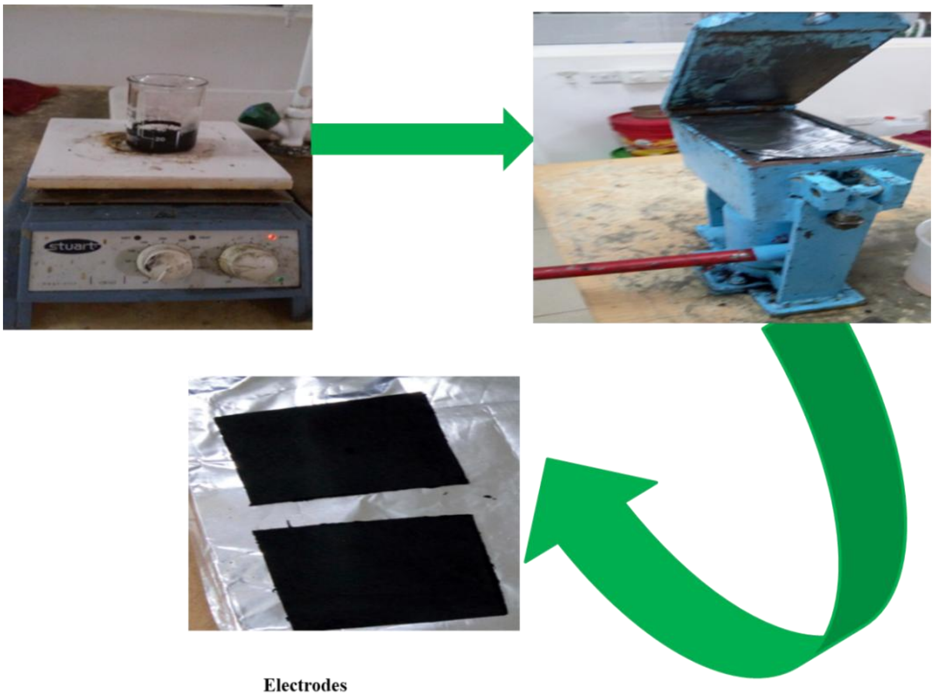

Figure 3: Schematic illustration of the fabrication of the CDI electrodes.

3.5 Capacitive deionization testing

The CDI performance of the prepared activated carbon was evaluated by using two electrodes configuration, a working electrode (cathode), and a counter electrode (anode) as shown in Fig.4. Batch experiments were conducted to investigate the electrosorption of Pb^{2+} on the electrodes prepared from CF. The fabricated electrodes weighing 0.6 g were placed in the CDI cell with $Pb(NO_3)_2$ solution, the volume of the solution was 30 mL and initial concentration C_o = 100 mgL^{-1}. Desalination experiments were performed with operating parameters of 1.2 V applied voltage, for 2 h adsorption time by using potentiostat/galvanostat (Vertex.1A.EIS) (1A/10V/1MHz EIS, Ivium Technologies, The Netherlands, equipped with Iviumsoft electrochemistry software). The conductivity of the solution was continuously monitored by a conductivity meter (GMH 3400 series) after every 5 min. The Pb^{2+} solution concentrations were determined by using the calibration curve.

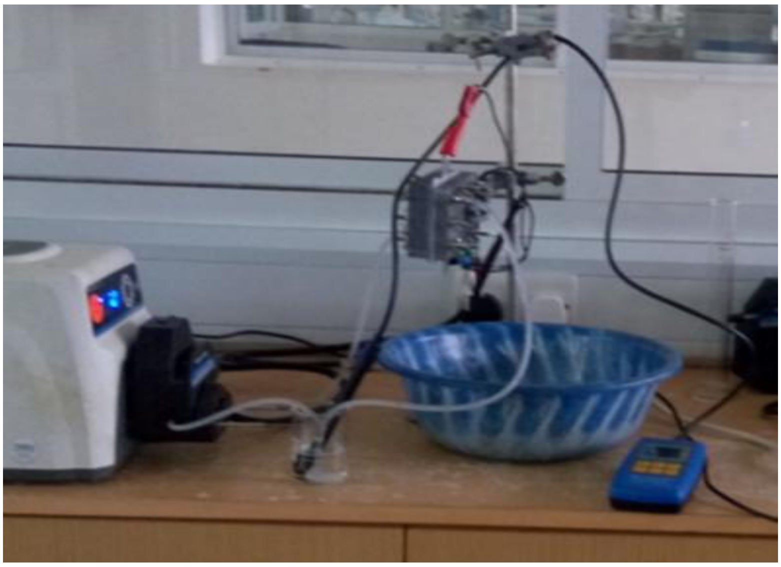

Figure 4: Schematic diagram of the CDI unit for Pb^{2+} removal.

The removal of the Pb^{2+} ions by the CF activated carbon samples were characterized via removal efficiency (*RE*) and electrosorption capacity (*EC*) as shown in Eqs. (1) and (2):

$$RE = \frac{C_o - C_f}{C_o} \times 100\% \quad (1)$$

$$EC = \frac{(C_o - C_f)}{m} \times V \quad (2)$$

where C_o and C_f represent the initial and final concentrations (mgL^{-1}) of the Pb^{2+} ions respectively; *V* is the total volume of solution (mL), and *m* is the mass of electrode material (g).

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Introduction

This chapter introduces and discusses the characterization of the prepared electrode materials and CDI performance. It describes the effect of the varied parameters which include activating agent ratios *R* and activating temperature, T_a. The electrodes were prepared by using the carbon materials activated at different amount of KOH: CF ratios and temperature and then tested in the CDI unit to evaluate the lead removal efficiency and its adsorption capacity of each prepared electrode.

4.2 Nitrogen adsorption isotherms of the samples

The textural properties of the CF samples; BET surface area (S_{BET}), micropore surface area (S_{micro}), mesopore surface area (S_{meso}), pore diameter (D_p), micropore volume (V_{micro}), mesopore volume (V_{meso}) and pore volume (V_T), are analyzed regarding the amount of KOH during activation as well as activation temperature and presented in Table 3.

Table 3: Textual characteristics of CF activated carbon sample

Carbon sample	**S_{BET} (m^2g^{-1})**	**S_{micro}(m^2g^{-1})**	**S_{meso}(m^2g^{-1})**	**S_{micro}/S_{meso}**	**D_p (nm)**	**V_{micro}(cm^3g^{-1})**	**V_{meso}(cm^3g^{-1})**	**V_{micro}/V_{meso}**	**V_T (cm^3g^{-1})**
CF-400	642	230	412	0.56	3.3	0.06	0.47	0.13	0.53
CF-4-600- 3	1101	505	596	0.85	3.8	0.20	0.84	0.24	1.04
CF-4-700-3	1666	814	852	0.96	3.5	0.30	1.17	0.26	1.47
CF-1-800-3	1642	868	774	1.12	3.5	0.39	1.03	0.38	1.42
CF-2-800-3	1808	646	1162	0.56	3.4	0.34	1.21	0.28	1.55
CF-3-800-3	1951	812	1139	0.71	3.6	0.41	1.37	0.30	1.78
CF-4-800-3	2481	972	1509	0.64	3.2	0.49	1.49	0.33	1.98

The BET surface area of untreated carbon (CF-400) was approximately 642 m^2g^{-1} but increased to 1642 m^2g^{-1} for CF-1-800- 3 after activation with KOH at 800 ºC. This increase in surface area can be attributed to KOH etching which creates more pores on the carbon materials. There was a further increase in surface area up to 2481 m^2g^{-1} for the CF-4-800-3. These results show that the ratio KOH: CF also has an effect on surface area; significant increase from 1642 m^2g^{-1} for R = 1 (CF-1-800-3) to 2481 m^2g^{-1} for R = 4 (CF-4-800-3) is observed while keeping constant the activation temperature at 800 ºC. Furthermore, the surface area increases from 1101 to 2481 m^2/g as activation temperature rises at constant KOH to carbon impregnation ratio of 4. The pore size of all carbon samples ranges from 3.2 to 3.8 nm which is a clear indication of the mesopore type of the samples. Availability of mesopores should favor the electrosorption capacity of activated carbon as reported in the literature (Liu *et al.*, 2013). From Table 3, one can see there is positive correlation between BET SSA and pore volume. Pore volume increased with increasing the activation temperature, as well as the amount of KOH. During activation the increase in the amount of KOH to the sample is anticipated to widen the pores thus giving a larger pore volume resulting in increased surface area. Furthermore, the linear relationship between the total pore volume together with mesopores volume was observed. As the KOH: CF and activation temperature increase then the total pore volume and mesopore volume also increases.

To further quantify porosity of the CF samples, the N_2 adsorption-desorption isotherms were determined; the effects of the KOH: CF ratio (Fig. 5a) and activated temperature (Fig. 5b) were investigated. One can see that materials have both micropores and mesopores. The isotherms for the CF samples exhibit apparent hysteresis loops between relative pressure of 0.4 - 0.8. According to the IUPAC classification, these curves exhibit type IV isotherms, showing that the materials are abundant with mesopores. It can be noted that all samples demonstrated large amount of non-uniform mesopores as the absorbed N_2 volume keeps increasing significantly. Figures 6a and 6b show the pore size distribution as calculated using the BJH method. The plots depict that more pores were distributed at 4 nm which agrees well with the isotherms that indeed the samples have pores in the mesopores region. The presence of mesopores is very important for ions transfer, and they also act as an ion reservoir in the electrode

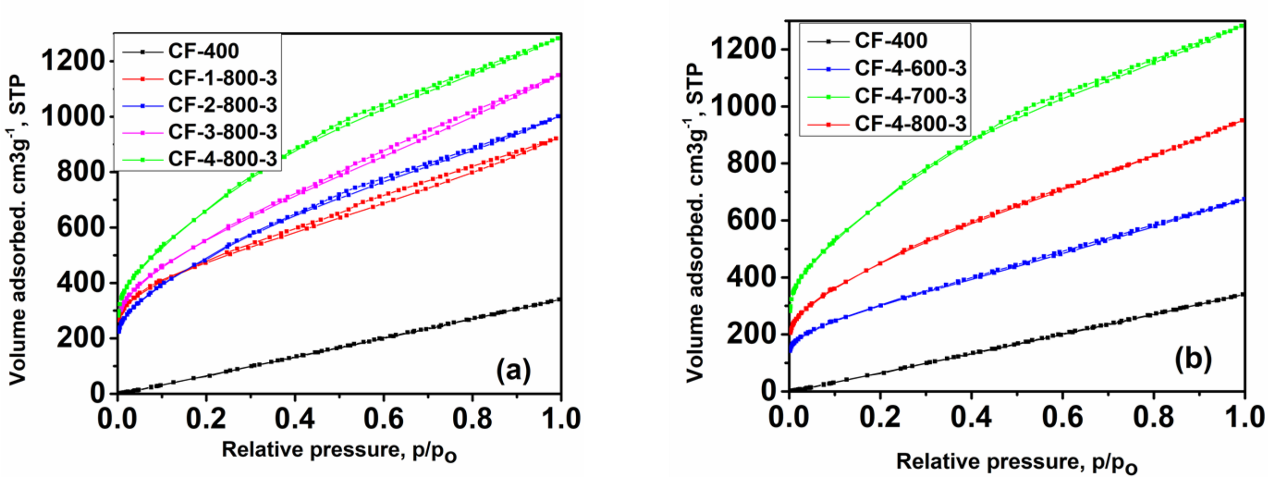

Figure 5: Nitrogen adsorption-desorption isotherms of CF activated carbon samples: (a) Impact of KOH: CF ratio; (b) Impact of activation temperature.

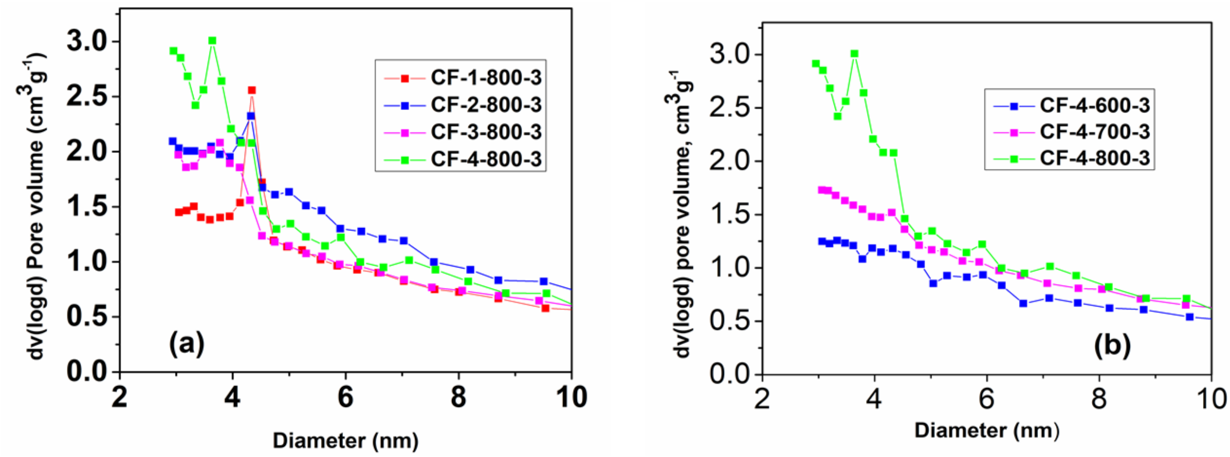

Figure 6: Pore size distribution calculated from adsorption-desorption isotherms using BJH method for activated carbon samples: (a) Impact of KOH: CF ratio; (b) Impact of activation temperature.

4.3 Morphological analysis

To gain further insight into the morphology of CF samples the SEM micrographs were taken for the carbon activated at 1:1 and 4:1 KOH: CF ratios, as shown in Fig. 7a and 7b, respectively. SEM images of Fig. 7a exhibit irregular pore sizes; the flake-like and graphitic structure of the carbon with a rough surface is generated by KOH etching that brings this high porosity observed on the SEM micrographs which are paramount for CDI applications. Nevertheless the shape of the sample treated at higher ratio 4:1(Fig. 7b), was completely changed that showed several types, such as flakes and blokes with smooth or wavelike surface. It is because that chicken feather melted completely and charred into charcoal.

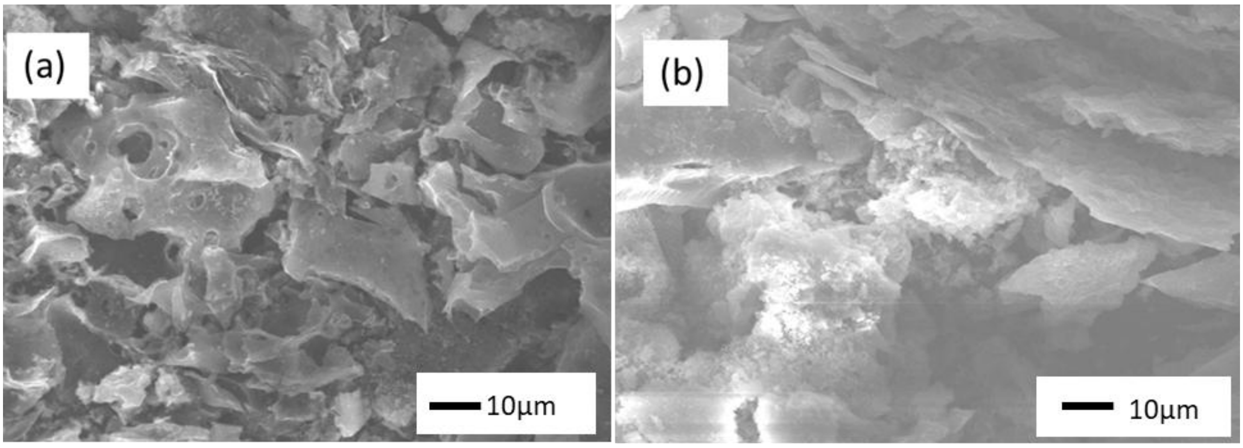

Figure 7: SEM images for CF samples: (a) CF-1-800-3 and (b) CF-4-800-3.

4.4 Fourier Transform Infrared spectroscopy analysis

FTIR analysis was used to identify the functional groups present in the AC surfaces. The FT-IR spectra of the untreated carbon (CF-400) and activated samples were measured to compare the presence of surface chemical functional groups at various processing stages of CF (Figure 4). Peaks resulted at ~1000 cm^{-1}, 1700 cm^{-1}, and 2200 cm^{-1} show that for untreated carbon, only bonds C-O, C=O and C≡C exist. These double and triple bonds might come from aromatic carbons in the chicken feather precursors. After activation with KOH, the new O-H and C-H bonds were introduced. The stretching vibrations of O-H and C-H and C-O were observed at 3569-3635 cm^{-1}, 2904-2939 $cm^{-1,}$ and 1041-1191 cm^{-1}, respectively. The presence of peak observed at ~1550-1700 cm^{-1} was assigned to C=C stretching vibrations. All activated carbon showed similar functional groups apparently because they were subjected to the same KOH chemical treatment. The oxygen-containing functional groups identified on the CF surfaces play a great role in metal sorption whereby during adsorption they bind with lead ions hence improve the lead removal efficiency. The presence of hydroxyl and carbonyl functional group enhances the wettability of the material which favors ionic adsorption during water treatment since it affects the total areas and ions removal rate.

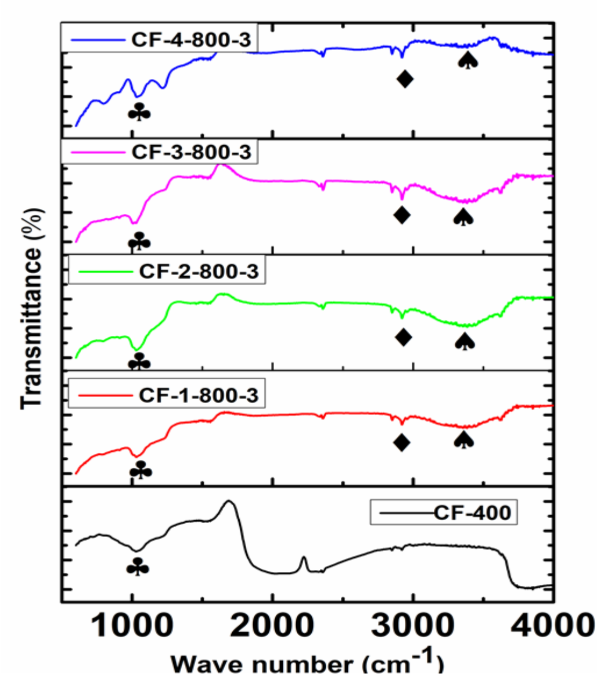

Figure 8: FTIR spectra of the untreated carbon (CF-400) and CF activated samples; where (♠) O-H, (♦) C-H, and (♣) C-O

4.5 Desalination experiments

The CDI electrosorption experiments were carried out with different CF electrodes to study the effects of activating agent to carbon ratio *R* and activating temperature T_a in CDI performance. The electrical conductivity of the $Pb(NO_3)_2$ solution versus operating time was measured with untreated CF-400 and different activated CF electrodes; results are shown in Fig. 9. The decrease in conductivity with time is observed for all CF electrodes and saturation state is seen within less than 2 hours of the adsorption stage. The conductivity decreases from initial value of 81.3 µS cm^{-1} (100 mg/L) to 19.7 µS cm^{-1} (18.6 mg/L), 28.0 µS cm^{-1} (29.4 mg/L), 31.4 µS cm^{-1} (33.8 mg/L), and 35 µS cm^{-1} (38.5 mg/L) for CF-1-800-3, CF-2-800-3, CF-3-800-3 and CF-4-800-3 electrodes, respectively, hence the largest change occurs for the smallest KOH:CF ratio, *R* = 1 (Fig. 9a). Increase in activation temperature T_a of the CF samples results in more effectual desalination (Fig.9b) as final conductivity of the $Pb(NO_3)_2$ solution drops from 69 µS cm^{-1} (82.8 mg/L), (T_a = 600 K) to 35 µS cm^{-1} (38.5mg/L) (T_a = 800 K). At the same time, practically no change in conductivity is seen for the sample CF-400 prepared without KOH treatment.

The lead removal efficiencies of CF samples calculated by Eq. (1), are represented in the bar diagrams (Fig. 10). The CF-1-800-3 electrode displays higher removal efficiency (81%) toward Pb^{2+} compared to other electrodes as shown in Fig.10a. These results indicate that as an activating agent to carbon ratio increases, the removal efficiency decreases which is irrespective of the increased BET surface area (Table 3). This may be due to the etching phenomenon of activating agent KOH which distorts the pores as the amount increases. Thus, the active surface for adsorption of the ions becomes smaller as compared to the BET surface area. Therefore struggles for making electrode with high surface areas are still not so important as removal efficiency depends on other factors as opined for example by Seo *et al.* (2010). Figure 10b shows the behavior of electrodes with CF-4-T_a-3 samples prepared at different activation temperatures: the CF-4-800-3 exhibits higher removal efficiency (61%) of Pb^{2+} as compared to other samples. These results show that the removal efficiency increases with an increase in activation temperature. This is due to the fact that as activation temperature increases the surface area also increases as proved by BET results (Table 3), thus increasing the adsorption sites.

The electrosorption capacities *EC* of the CF samples calculated by Eq. (2) are presented in Fig. 11a and 11b. The electrode prepared with the CF-1-800-3 exhibits maximum *EC* = 4.1 $mg \cdot g^{-1}$ on the removal of the Pb^{2+} ions which is in agreement with the highest removal efficiency for the same sample (Fig. 10a) as well as highest electrosorption capacity (Fig. 11a). It is worth to notice that the sample of maximum electrosorption capacity CF-1-800-3 doesn't possess the largest surface area as shown in Table 3. It seems that an increase in the amount of activating agent might bring to enlarging the specific surface area of the sample but not always results in the best adsorptivity. Therefore no direct accordance is observed between the BET surface area and the electrosorption capacity of samples. This is also observed by Wang *et al.* (2010)

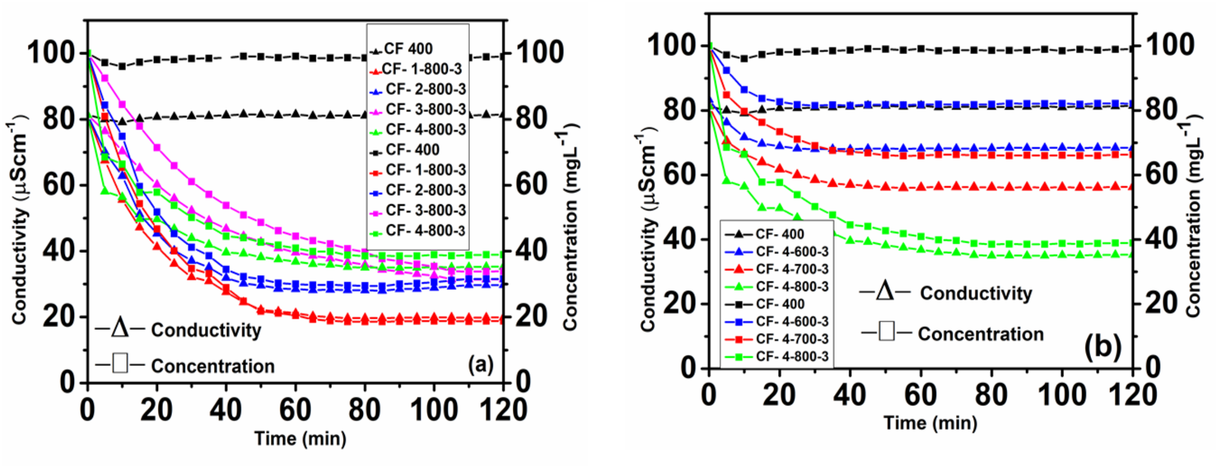

Figure 9: Desalination experiment with the CF carbon electrodes: (a) Impact of KOH: CF ratio; (b) Impact of activation temperature.

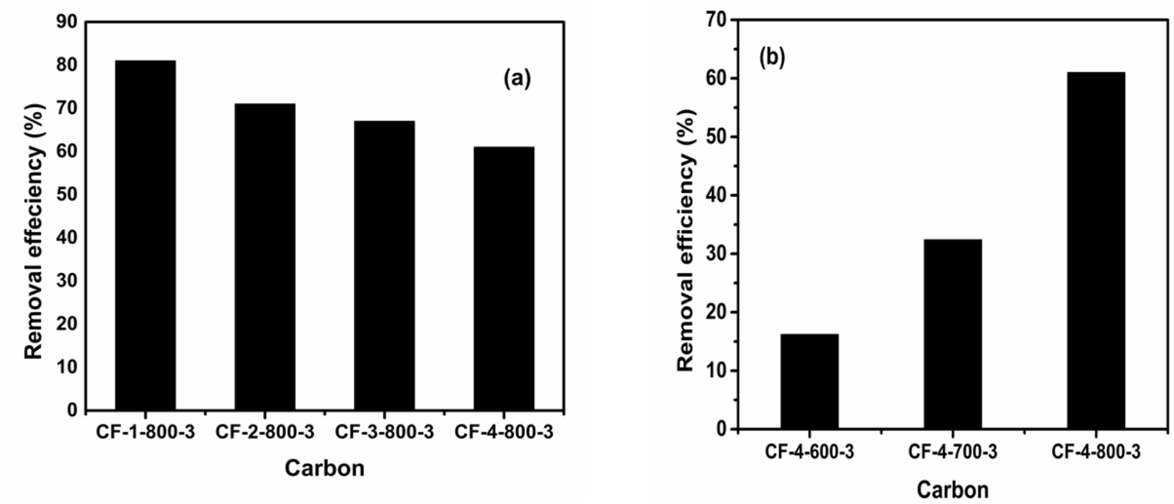

Figure 10: Lead removal efficiency of CF electrodes: (a) Effect of KOH: CF ratio; (b) Effect of activating temperature.

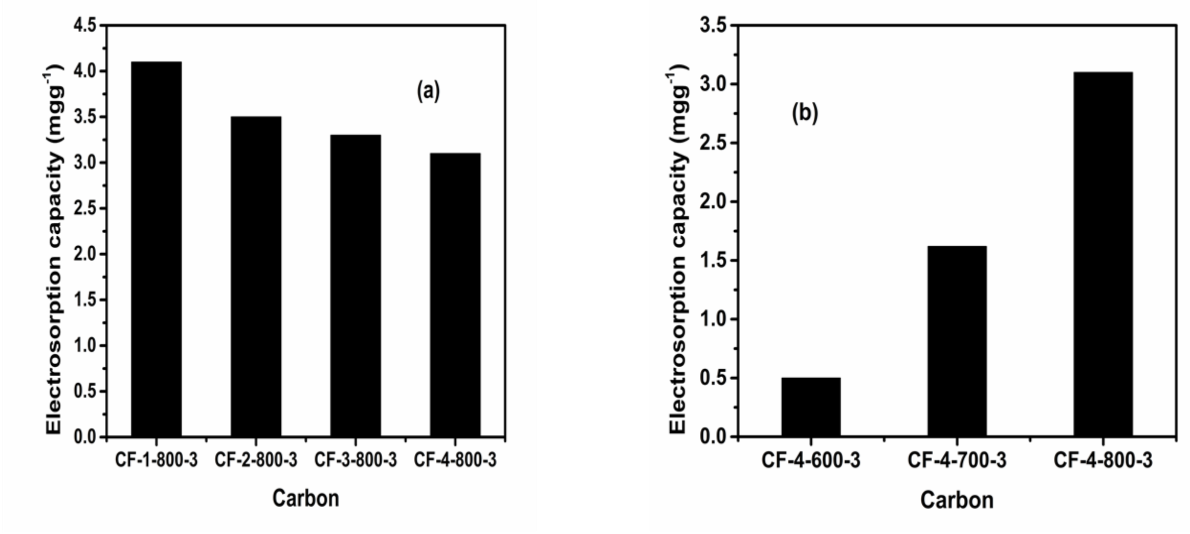

Figure 11: Electrosorption capacity of lead by CF electrodes: (a) Effect of KOH: CF ratio; (b) Effect of activating temperature.

4.6 General discussion of the results

The effect of activating temperature on the CF samples is summarized in Table 4. The results show a direct relationship between activation temperatures and BET surface area; the BET surface area increases as activation temperature increases; this result is supported by Zhao *et al.* (2015). Also, it was observed that the electrode materials with high BET surface area exhibit high removal efficiency as well as electrosorption capacity.

Table 4: Relationship between the CF-4-Ta-3 samples textural properties and adsorption performance of the electrodes: effect of activating temperature Ta.

T_a (°C)	S_{BET} (m^2g^{-1})	D_{BJH} (nm)	V_T (cm^3g^{-1})	RE (%)	EC (mgg^{-1})
600	1101	3.8	1.04	16.2	0.5
700	1666	3.5	1.47	32.4	1.6
800	2481	3.2	1.98	61	3.1

The results summarized in Table 3 show the effect of activating agent ratio on the CF samples; the results revealed that there is a linear relationship between the activating agent ratio and the BET surface area. The surface area is increasing with increase in the amount of KOH. During activation the increase in the amount of KOH to the sample is anticipated to widen the present pores thus giving a larger pore volume so as increasing the surface area; as supported by Enock *et al.* (2017). However, the higher is ratio R, the larger the surface area but the lower the sorption ability. It means in CDI, the high surface area is not the only factor that contributes to the good electrosorption capability of the electrode materials. It depends on other factors like the number of electrosorption sites and electrical double layer overlapping effect this means that for good electrosorption capacity there should be a balance between the number of electrosorption sites and EDL overlapping effect. Moreover, pore size and structure of the electrodes enhances the removal efficiency, so, if the pore size of the hydrated ion is compatible with the size of the pores of the sample then adsorption of the ions could be improved (Largeot *et al.*, 2008). Furthermore appropriate pore size distribution between micropore and mesopore is required to improve the CDI performance as supported by Seo *et al.* (2010) and (Wang *et al.*, 2010).

Table 5: Relationship between the CF-R-800-3 samples textural properties and adsorption performance of the electrodes: effect of the KOH: CF ratio (*R*).

R	**S_{BET} (m^2g^{-1})**	**D_{BJH} (nm)**	**V_T (cm^3g^{-1})**	**RE (%)**	**EC (mgg^{-1})**
1	1642	3.5	1.42	81	4.1
2	1808	3.4	1.55	71	3.5
3	1951	3.6	1.78	67	3.3
4	2481	3.2	1.98	61	3.1

CHAPTER FIVE

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

To conclude, the main objectives of this study were achieved as mentioned in Chapter 1. This dissertation focuses on the development of porous carbon derived from chicken feather for CDI application.

Chicken feather derived carbon was prepared through carbonization followed by KOH activation at different KOH:CF ratios and different temperatures. Also in this study, the capacitive deionization performances for Pb^{2+} removal from dilute aqueous $Pb(NO_3)_2$ solutions using electrodes derived from chicken feathers were investigated. Experimental results don't show a direct relationship between KOH: CF ratio and the removal efficiency, as well as BET surface area while the linear relationship was observed between activation temperature and removal efficiency, as well as BET surface area. Electrosorption capacity and removal efficiency do not depend on BET surface area only rather other factors need to be considered. Generally, results of this work show that electrode materials derived from chicken feather can be suitable for removing heavy metals such as Pb^{2+} from aqueous solution using CDI technique.

5.2 Recommendations

Based on the findings of this study, it is proposed that future work should consider the following;

(i) Despite the achievements in the synthesis of chicken feather derived carbon materials for CDI applications, there is a need for optimization of carbonization and activation temperature as well as the KOH: carbon ratio.

(ii) In this study, the varied parameters were activation temperature and activating agent ratio, but we would wish to recommend investigating the impact of other parameters like activating time, operational voltage, approach, and flow rate.

(iii) The porous carbon synthesized can be tested further for efficiency in water purification with reference to fluoride removal, salt removal and removal of other heavy metals.

(iv) Further characterization of the CF is required to investigate the other factors that contribute to higher electrosorption capacity.

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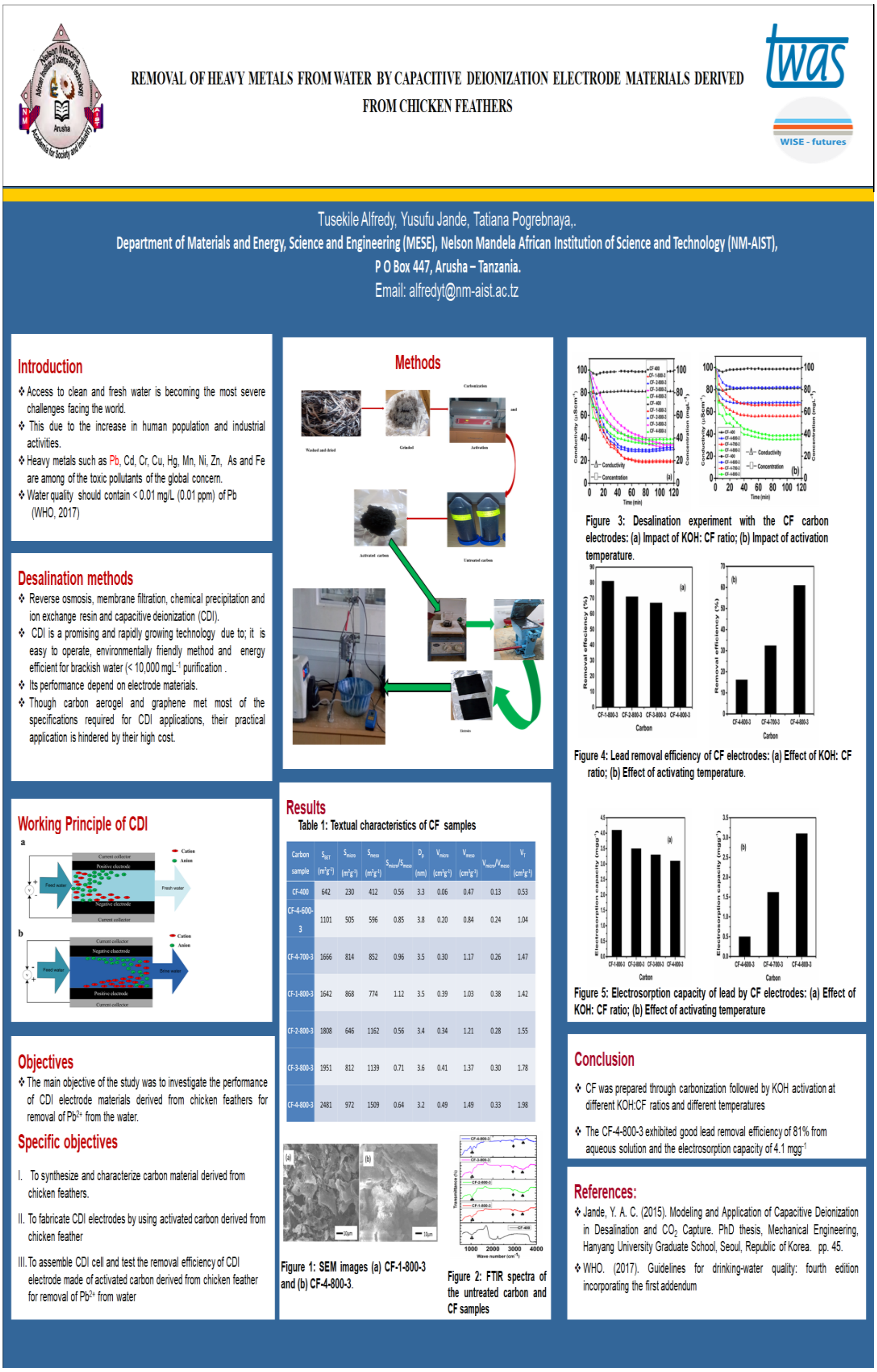
RESEARCH OUTPUTS

Poster presentation



Accepted manuscript

Dear MSc student Alfredy,

I am pleased to tell you that your submission "Removal of Lead Ions from water by Capacitive Deionization Electrode Materials Derived from Chicken Feather" (JWRD-D-18-00074R1) has now been accepted for publication in Journal of Water Reuse and Desalination.

It was accepted on 14 Mar 2019.

You will receive a separate e-mail with details on how to pay the Article Processing Charge, and you will hear from IWA Publishing soon regarding the publication of your paper.

Thank you for submitting your work to Journal of Water Reuse and Desalination.

With best wishes

How Yong Ng

Editor

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