MANGANESE REMOVAL FROM SYNTHETIC WASTEWATER USING PYROLYZED CHICKEN FEATHER FIBER (PCFF)

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By

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I hereby declare that all corrections and comments made by the supervisor(s)and examiner have been taken into consideration and rectified accordingly.

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ABSTRAK

Kehadiran logam berat dalam air sisa menimbulkan pelbagai kesan negatif kepada kesihatan manusia dan alam sekitar. Pelbagai usaha giat dijalankan dalam menghasilkan bahan penjerap kos rendah untuk penyingkiran logam berat seperti kalsium, zink, magnesium, dan mangan dari air sisa. Karbon aktif sedia ada adalah tinggi harganya namun pelbagai usaha inovasi dan penyelidikan kini giat dijalankan ke arah penghasilan karbon aktif dari produk sisa semula jadi seperti penggunaan gentian bulu ayam. Langkah alternatif ini adalah lebih mudah, murah dan bersifat mesra alam untuk rawatan air sisa. Dalam kajian ini, penyingkiran logam berat dari air sisa sintetik kategori mangan dilaksana menggunakan gentian bulu ayam yang dipirolisis (PCFF). Gentian bulu ayam yang dipirolisis (PCFF) bertindak sebagai karbon aktif dalam rawatan air sisa dan dipirolisis menerusi suhu 195°C selama 5 jam di pirolisis pertama dan suhu 400°C selama 2 jam di pirolisis kedua. Manakala, penyediaan air sisa sintetik melibatkan penggunaan serbuk manganum sulfat menunjukkan reaksi tindak balas aktif berbanding penggunaan larutan piawai dengan kepekatan 1000 mg/L. Kajian ini menunjukkan peratusan penyingkiran mangan dari air sisa sintetik adalah sebanyak 33.55% pada pH 5 dan 0.8g PCFF, lebih tinggi berbanding peratusan penyingkiran mangan kajian terdahulu sebanyak 11.44% pada pH 5 dan 1.5g PCFF. Kajian ke atas karbon aktif telah dilaksanakan melalui SEM/EDX dan Raman Spektroskopi. Imej SEM menunjukkan permukaan PCFF-1 dan PCFF-3 adalah kasar berbanding permukaan PCFF-2 seterusnya menyediakan kawasan yang lebih luas bagi proses penjerapan. Walau bagaimanapun, keputusan Raman menunjukkan bahawa karbon aktif yang terhasil tidak mampu menjerap logam berat dalam air sisa dipengaruhi oleh faktor saiz liang karbon aktif, ketidaksesuaian karbon aktif untuk logam berat yang tertentu dan disebabkan oleh faktor caj permukaan karbon aktif.

ABSTRACT

The presence of heavy metal in wastewater pose to various negative impact on human health and environment. During the past decades, many attempt made to produce low cost adsorbent prior wastewater treatment for removing polluting ions such as calcium, zinc, magnesium, and manganese from industrial water systems. However, current activated carbon available are expensive but innovative effort and research now are moving forward towards the production of activated carbon with utilization of natural waste product such as chicken feather fiber. This alternative yet is a simple, low-cost, environmentally friendly for wastewater treatment. This study investigated the removal of heavy metal from synthetic wastewater using Pyrolyzed Chicken Feather Fiber (PCFF). PCFF act as an activated carbon derived from waste chicken feather produced from the two stage of pyrolysis method at 195°C for 5 hours and 400°C for 2 hours respectively.. The result obtained reveal that percentage removal of manganese in this study was 33.55% at pH 5 and 0.8g of adsorbent, higher than previous research with removal of 11.44% of manganese at pH of 5 and 1.5g of adsorbent. The activated carbon then are characterize by SEM/EDX and Raman Spectroscopy. SEM images showed that the surface of PCFF-1and PCFF-3 is rougher whereas the surface of PCFF-2 is smoother thus providing the larger surface area for the adsorption process. However, Raman analysis showed that produced activated carbon was unable to adsorb the pollutants due to the size of the pore presence in the activated carbon, unsuitability of activated carbon for the removal of selected heavy metal and due to surface charges.

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LIST OF SYMBOL AND ABBREVIATIONS

CF	Chicken Feather
CFF	Chicken Feather Fiber
PCFF	Pyrolyzed Chicken Feather Fiber
Mn	Manganese
T	Temperature
t	time
nm	Nanometer
µm	Micrometer
mm	Milimeter
cm	Centimeter
m	Meter
mg	Miligram
g	Gram
ml	Mililiter
L	Liter
h	Hour
rpm	Resolution Per Minute
mol	mole
C	Degree Celcius (°C)
K	Kelvin
%	Percentage
W	Weight of adsorbent
V	Volume of sample

C_o	Initial Concentration
C_e	Equilibrium Concentration
q_e	Adsorption capacity
COD	Chemical Oxygen Demand
SEM	Scanning Electron Microscope
EDX	Energy Dispersive X-ray
TEM	Transmission Electron Microscopy
FTIR	Fourier Transform Infrared Spectroscopy
BET	Brunaeur-Emmett-Teller (BET) Surface analysis
HWFM	High Width Full Man
G Band	Graphene Band
D Band	Diamond Band

CHAPTER 1

INTRODUCTION

1.1 Background

Pollution is an issue of concern in the modern world that we live in today. The main contributor to environmental pollution are industrial, agricultural and domestic activities. The increase use of heavy metals in industry has resulted in increased availability of metallic substances in natural water sources (Karnib *et al.*, 2014). These wastewater with heavy metal ions may discharged to the environment and can be adsorbed by living organism (Wang and Chen, 2009)(Barakat, 2011). Wastewater containing heavy metals has developed successful innovative process for the treatment purposes.

Heavy metals in the aquatic medium may originate from wastewater of many industries, such as battery manufacturing processes, electrical, electroplating, fertilizer, pesticides, mining, the production of paints and pigments and the ceramic and glass industries (Nguyen *et al.*, 2013). Heavy metal such as cadmium, nickel, lead, copper and zinc are well-known toxic and carcinogenic agents and when discharged into the wastewater represent serious threat to human population and the fauna and flora of the receiving water bodies (Srivastava and Majumder, 2008). Due to their hazardous effects, it can cause damage to many parts of human bodies even at low concentrations.

Nowadays, numerous methods proposed for efficient heavy metal removal from wastewaters, including chemical precipitation, ion exchange, adsorption, membrane filtration and electrochemical technologies. Among these techniques, adsorption offers flexibility in design and operation and in many cases it will generate high-quality treated effluent (Mittal *et al.,* 2013). A wide variety of agricultural waste and by products has been explored for the elimination of heavy metals (Nguyen *et al.*, 2013). The development of low cost adsorbents had been an attempt for the researcher to discuss the potential use of household, agricultural and industrial waste as an alternative for wastewater treatment to replace highly cost commercial adsorbent such as activated carbon, zeolites, silica. Recent years has shown the development of protein materials as promising candidate as an adsorbent due to its excellent biocompatibility and biodegradability (Ma *et al.*, 2016).

1.2 Problem Statement

Chicken feathers are a waste by product of poultry industry with more than $4*10^6$ ton per year in the worldwide. In general, some of them are pre-treated and used as animal feed with low nutritional value, the rest are disposed through landfills which will cause environmental problems (G,B *et al.,* 2016). Even though chicken feather has the potential for wastewater treatment but the utilization of natural resources such as chicken feather are still lack in many ways. This study is to improve the preparation method to ensure better removal efficiency of PCFF as an adsorbent.

Treatment method such as precipitation commonly used for treating industrial wastewater. However, the process are highly cost for operation and not environmental friendly method. Thus, adsorption has been the chosen method for removal of heavy metal in the wastewater. Expensive activated carbon has attracted the attention of many researchers to convert available waste into cheaper activated carbon as replacement for the current activated carbon. Due to that, this study aimed to look upon the potential of chicken feather as low cost adsorbent for partially used in order to enhance the removal of heavy metal for wastewater.

1.3 Research Objectives

The main aim of the study is to investigate the characteristic of Chicken Feather Fiber (CFF) after pyrolysis process with the process of converting the chicken feather to a cheaper and better alternative for wastewater treatment by utilizing adsorption method to remove polluting material. The objectives of this study are as follow :

1. To investigate the characteristic of the prepared Pyrolyzed Chicken Feather Fiber (PCFF) with different temperature.
2. To evaluate PCFF's adsorption performance by synthesizing, measuring and analysing the removal efficiency of the selected heavy metal.

1.4 Scope of Study

In this study, the adsorption performance of PCFF were observed by synthesizing, measuring and analysing the removal efficiency of the selected heavy metal. The efficiency of adsorbent evaluated through pyrolysis process on the chicken feather with different heating temperature, followed by the adsorption process for the removal of heavy metal ions in the prepared synthetic wastewater. The characterization of CFF was carried out using Scanning Electron Microscope (SEM) to study the adsorbent surface morphologies after pyrolysis process as well as the changes in morphological shape of CFF in enhancing the efficiency of adsorption process. The batch experimental study was conducted to determine the removal effectiveness of adsorbent on the heavy metal in the synthetic wastewater.

First step was choosing the best adsorbent by conducting the removal efficiency test on heavy metal using prepared PCFF with different heating temperature. Next, the study were focused on the identification of the best conditions on providing the best adsorption performance.

1.5 Summary of the thesis

There are total of five chapter in the thesis writing.

Chapter One **:** The introduction of the study including the background studies on environmental pollution and the problem statement as well as presents the objectives and scope of research.

Chapter Two : The Literature Reviews describing the review on wastewater contamination with major concern of heavy metal pollutant generally from industrial activity. This chapter also reviewing the characteristic of chicken feather as potential low-cost adsorbent. Besides, the activation method of chicken feather, treatment method and several application of chicken feather as adsorbent from past few years presented in this chapter.

Chapter Three **:** The methodology for the overall experiment in the study. This chapter includes the preparation of chicken feather fiber, synthetic wastewater and preparation of an adsorbent for the batch adsorption studies as well as for the designation part. Next, this chapter also includes the list of material and equipment used in the study.

Chapter Four **:** This chapter are describing the experimental results, finding and the relevant discussion for the experimental and designation part including the result from all the analysis throughout the experiment such as SEM analysis, Raman spectroscopy and the adsorbent characterization.

Chapter Five **:** Last chapter consist of conclusion and recommendation of the thesis for improvisation in research work quality. This chapter also concludes the findings of the experiment by referring to the objectives as a guideline.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

The exposure to heavy metals may cause serious health problems even at very low concentration. Therefore, the removal of heavy metal from wastewater is extremely important. Numerous technologies developed to treat wastewater contaminated by heavy metal. However, adsorption is the widely used in wastewater treatment which were based on the physical interaction between metal ions and sorbents. This chapter includes the possible mechanism of adsorption and adsorption isotherm. Rapid advancement in solving environmental problems has become the interest of researcher to study the effectiveness of waste material to benefits to the society. This includes the used of chicken feather as raw material for the preparation of low-cost adsorbent. Besides, the mechanical, physical and thermal properties of chicken feather are knowledgeable in various application. In addition, chicken feather has capability in wastewater treatment including the removal of heavy metal (Sayed *et al.,* 2005), toxic dye removal (Mittal *et al.,* 2013) and removal of antibiotic residue from aquatic environment (Li *et al.*, 2017).

2.2 Heavy Metals in Industrial Wastewater

Excessive release of heavy metal into the environment due to industrialization and urbanization has contributed to great pollution in the wastewater. Unlike organic pollutants, the majority of heavy metal ions susceptible to biological degradation, it does not degrade into harmless ends product. Heavy metals are generally considered to be those whose density exceeds 5 g per cubic centimetre (Barakat, 2011). Exposure to some metals, such as mercury, lead, arsenic, cadmium. Chromium. Copper, Nickel and Zinc cause serious health effects and in extreme cases, death. Table 2.1 shows the type of heavy metal may present in the discharge water and the effects to human health.

Table 2.1 : The type of heavy metal that may be present in the discharge wastewater and the effects to human health (Babel and Kurniawan, 2003).

Type of Heavy Metal	Toxicity Impact Towards Human Health
Arsenic	Skin manifestations, visceral cancers, vascular disease
Cadmium	Kidney damage, renal disorder, human carcinogen
Chromium	Headache, diarrhea, nausea, vomiting, carcinogenic
Copper	Liver damage, Wilson disease, insomnia
Nickel	Dermatitis, nausea, chronic asthma, coughing, human carcinogen
Zinc	Depression, lethargy, neurological signs and increased thirst
Lead	Damage the fetal brain, disease of the kidneys circulatory system and nervous system
Mercury	Rheumatoid arthritis, and disease of the kidneys, circulatory system and nervous system.

2.3 Treatment of Heavy Metal

Considering legal limits and toxic effects to human health, the removal of heavy metal from water or wastewater is extremely important and mandatory. Methods for removing heavy metal ions mainly consist of physical, chemical, electrical, thermal and biological technologies (Ali *et al.,* 2012). Traditional method for heavy metal removal includes precipitation, ion exchange, membrane filtration, electroplating and adsorption (Banerjee, 2012). However, the low sorption capacities and efficiencies limit their application to remove heavy metal ions from wastewater. Adsorption technology with activated carbon are the most common method for wastewater treatment due highly effective technique for the removal of heavy metals and activated carbon has been widely used as an adsorbent (Hegazi, 2013). Table 2.2 shows the comparison of different technologies for removing heavy metals from waste water (Nguyen *et al.*, 2013).

Table 2.2 : Comparison of different technologies for removing heavy metals from waste water (Nguyen *et al.*, 2013)

Methods	**Disadvantages**	**Advantages**
Chemical precipitation	Large amount of sludge Extra operational cost for sludge disposal	Simple operation, inexpensive, can remove most of metals
Chemical coagulation	High cost Large consumption of chemicals	Sludge settling Dewatering
Ion exchange	High cost Less number of metal ions removed	High regeneration of material Metal selective
Electrochemical methods	High capital and running cost Initial solution pH and current density	Metal selective No consumption of chemicals Pure metals can be achieved
Adsorption using activated carbon	Cost of activated carbon No regeneration Performance depends upon adsorbent	Most of metals can be removed High efficiency (99%)
Biosorption	Early saturation, limited potential for biological process improvement, no potential for biological altering the metal valence state	Low cost, high efficiency, minimization of sludge, regeneration of biosorbents, no additional nutrient requirement, metal recovery
Membrane filtration	High operational cost due to membrane fouling	Small space requirement, low pressure, high separation selectivity
Electro dialysis	High operational cost due to membrane fouling and energy consumption	High separation selectivity
Photo catalysis	Long duration time, limited applications	Removal of metals and organic pollutant simultaneously, less harmful by-products

2.4 Adsorption Process

Adsorption is a mass transfer process involves the accumulation of substances at the interface of the two phases, such as, liquid-liquid, gas-liquid, gas-solid or liquid-solid interface forming a film of adsorbate (Jorgensen, S.E. ,1979). The adsorbate is the substance being adsorb and the adsorbing material is termed as an adsorbent. The properties of the adsorbate and adsorbents depend upon their constituents. Generally, sorption is the transfer of ions from solution phase to the solid phase. 'Sorption actually describes a group of process which includes adsorption and precipitation reactions' (Rathoure, Ashok K. 2018).

There are four main steps involved in adsorption process of pollutant onto solid sorbent. This step involves bulk solution transport, film diffusion transport, internal pore transport and adsorption process. The transport of the adsorbate from bulk solution to the boundary layer of adsorbent known as bulk solution transport. Film diffusion transport means the transport by molecular diffusion through the stationary liquid film. Next, the substance are being transport through the pores to available adsorption sites along the adsorbent surface through internal pore transport and bond are formed between the particle and adsorbent at the adsorption sites.

2.4.1 Mechanism of Transport

In general, there are two types of adsorption mechanisms, physisorption and chemisorption. Physisorption is described when the interaction between the solid surface and the adsorbed molecule has a physical nature. Besides, desorption and interruption studies on the adsorbent can further indicated through chemisorption or physisorption.

In this context, activated carbon resulted into physisorption interaction (Ali *et al.,* 2012). However, it forms weak interactions with the surface due to Van Der Waal forces. As they controlled by the weak forces, the results are reversible. On the other hand, chemisorption described the attraction forces between adsorbed molecules and the solid surface due to the chemical bonding. Chemisorption occurs only as a monolayer and the presence of stronger forces caused substances chemisorbed on the solid surface to be hardly removed (De Gisi *et al.*, 2016).

2.4.2 Factors Affecting Adsorption Process

The adsorption process are influenced by several factors such as dosage of adsorbent, initial concentration of adsorbent, surface area of adsorbent , pH, contact time and temperature. One of the most important factors is the surface area of adsorbent. The efficiency of adsorption are slightly depending on the internal surface of the adsorbent at given temperature (Ali *et al.,* 2012). Another important factor is the dosage of adsorbent. It will determine the adsorption capacity under given operating condition. As the adsorbent dose increased, the active site of adsorbent were exposed are higher thus increase the rate of adsorption (Paper, 2016). However, the optimum dosage of adsorbent must be into detail consideration if will be applied in real scale of wastewater treatment. Next is the pH changes as it enhance the uptake of heavy metal ions in the solution. Increase in pH results in the decreasing of adsorption ability (Li *et al.*, 2017). The pH determine the surface charge of the adsorbent and the degree of ionization of the adsorbate. However, high initial pH could precipitated out the heavy metal ions and this are against the purpose of sorption process (Abdel Salam *et al.,* 2011).

Apart from that, the contact time also affect the adsorption process. Rapid heavy metal uptake was due to the binding site available within the adsorbent, since the adsorbent surface area influenced the adsorption process. However, the heavy metal uptake rate will decreased as the available binding sites were being used up and clogged by metal ions (Paper, 2016). Besides, rate of adsorption might also affected by the temperature changes during the adsorption process. The amount of metal being adsorbed was enhanced as the temperature increased (Mittal *et al.*, 2013).

2.5 Adsorbent

There are several adsorbents such as activated carbon, alumina, silica and zeolites were used in the purpose for purification of water (Saleh, T.A and Gupta, V.K, 2016). In recent years, activated carbon has been frequently used as an adsorbent for the removal of heavy metal (Hegazi, 2013). Activated carbon has shown its capability as potential adsorbent for the removal of various organic and inorganic contaminants presents in aquatic environment due to its large surface areas that range from 500 to 1500 m^2/g (Karnib *et al.*, 2014). However, activated carbon remain as an expensive material despite of its extensive use in water and wastewater treatment industries. Therefore, economic and environmental friendly method has developed interest for the production of low cost adsorbent as an alternative in wastewater treatment. In general, for an adsorbent to be termed as a low cost adsorbent, it shall fulfil criteria such as little processing requirement, abundant in nature, or is a by-product or waste material form any other industry (Hegazi, 2013). In addition, sustainable solution for wastewater treatment has introduced several low-cost by-products transformed from agricultural, household and industrial to become as potential adsorbent (De Gisi *et al.*, 2016). Figure 2.1 shows the division of low-cost adsorbents.

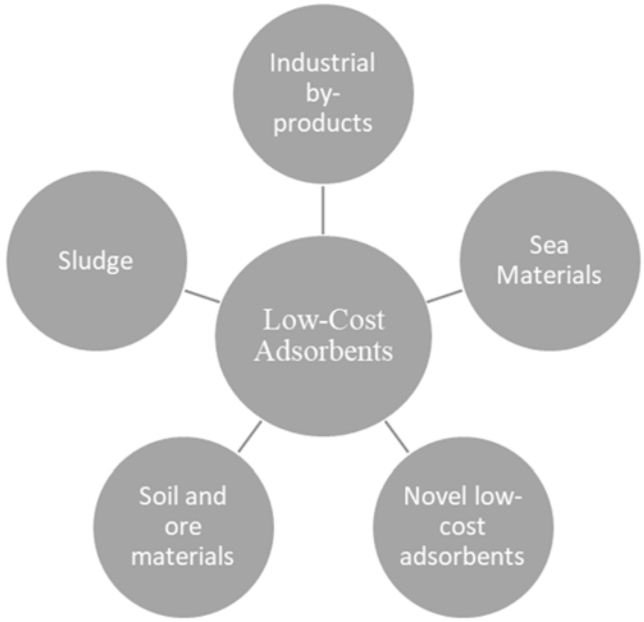

Figure 2.1 : The division of low-cost adsorbents (De Gisi *et al.*, 2016)

2.6 Chicken Feather As An Adsorbent

In recent years, the large amount of chicken feather disposed by various poultry based industry as a waste is a strict solid-agricultural dispose problem. Most CFs are usually burned or dumped to landfilled which lead to environmental pollution (Reddy, 2015). However, instead on remains abundant to nature, due to unique characteristic of chicken feather, it has engrossed research on expanding materials from it to be used in many application. The presence of keratin fibres and carbonizing organic materials in chicken feather has effectively contributed to one of the most widely used adsorbent to remove heavy metal contaminant in wastewater (Abdel Salam *et al.,* 2011). Besides, usage of chicken feathers for sorption also showed an excellent result for the removal of dyes, antibiotic residues and other organic toxic compounds (Li *et al.*, 2017).

2.6.1 Chicken Feather Composition And Morphological Structure

Chicken feather structure consist of a central carbon linked to functional groups (amine, $-NH_2$, and carboxylic acid, -COOH), and the hydrogen atom and the group R (sulphur). The feathers are one of the natural resources, renewable, low-cost and supported by the hollow structure which has low-density (0.8 g/cm^3) compared with wool (1.3 g/cm^3) and cellulose fibers (1.5 g/cm^3). It also act as thermal insulators. The feather mainly posed high flexibility and hydrophobic. Furthermore, these feathers has features of fibrous, good compressibility and quite resilience when used in composite, textile product and films (Belarmino *et al.*, 2012).

Another unique structure of chicken feather is the fibers, primarily made up of three separate units. The central axis of feather called rachis with up to 7cm in length attached to the calamus. Calamus is the secondary structures comprise of bards usually 1 to 4.5 cm depending on its location in the rachis. Typically, it is longer when near to the base compared to the tip (Belarmino *et al.*, 2012). Figure 2.2 shows the photograph of a chicken feather showing rachis, calamus and barbs. The third structure of chicken feather is the barbules with length of 0.3 – 0.5 mm, connected to the barbs and has hooks in its ends (Reddy and Yang, 2007). Figure 2.3 shows the micrograph of the secondary and tertiary structure of chicken feather showing barb and barbules.

Figure 2.2 : Photograph of a chicken feather showing rachis, calamus and barbs (Belarmino *et al.*, 2012)

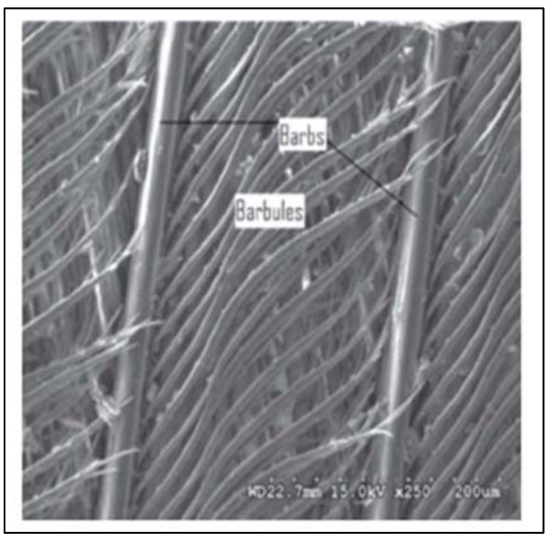

Figure 2.3 : Micrograph of the secondary and tertiary structure of chicken feather showing barb and barbules (Reddy and Yang, 2007)

2.6.2 Chicken Feather Properties

The most interesting properties of chicken feather is keratin. The feather is composed of about 90% of keratin which high in mechanical strength. The keratin has a-helix and B-pleated sheet structure comprising about 20 amino acids, mainly cysteine. Besides, Feather keratin also known for its structural protein, characterized by high cysteine content and a significant amount of hydroxyl amino acids, especially serine (Meyers *et al.*, 2008) . Figure 2.4 shows the a-helix and b-pleated sheet structure of keratin.

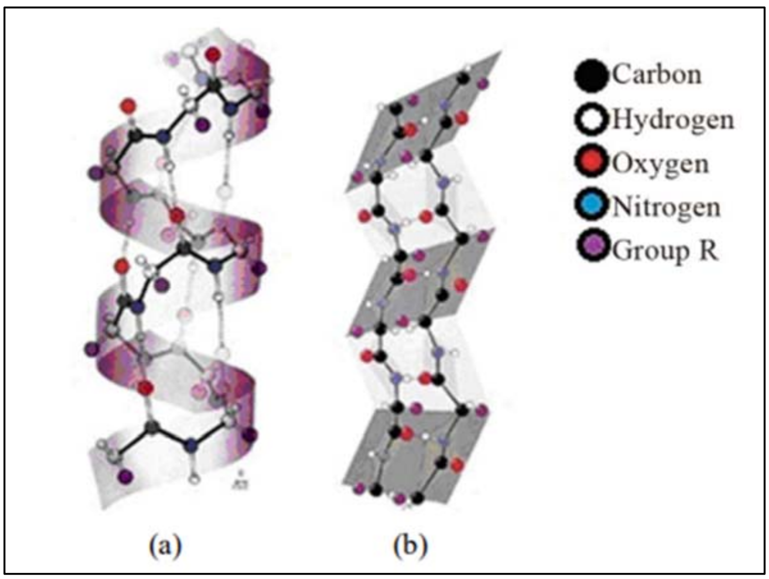

Figure 2.4 : The (a) a-helix and (b) B-pleated Sheet structure of Keratin (Meyers *et al.*, 2008)

The structure and properties of chicken feather especially barbs is beneficial as natural protein fiber. The uniqueness of the barbs is their low density, availability as well as low-cost makes barbs useful and preferable fiber for various application such as textiles and composites industry. In addition, the presence of honeycomb structures allows the barb to have low density, provides air and heat insulating properties unlike any other natural fiber (Reddy and Yang, 2007).

2.6.3 Activation Method

The chicken feather was subjected to undergo activation process to enhance the adsorption capacity and refine the structure of hen feather (Mittal *et al.,* 2013). There are two types of activation method such as chemical and thermal treatment for chicken feather as one of the potential low-cost adsorbent. In order to obtain better quality of chicken feather, activation process in necessary to applied. This is to enhance the structural of chicken feather fiber with micro porosity, high pore volume as well as surface area in the range of 100-2000 m^2/g (Senoz, 2011).

2.6.3.1. Chemical Treatment

The purpose of chemical treatment is to enhance the chicken feather fiber by modifying the reactive site to ensure the effectiveness during adsorption process. The chicken feather as adsorbent material washed, cut into small pieces and activated using hydrogen peroxide (Mittal *et al.,* 2013). Besides, another alternatives includes washing chicken feather using water and hair drying shampoo and rinsed several time then kept in an open atmosphere for 24 hours results in complete dry, clean and less sticky appearance (G,B *et al.,* 2016). Next, the fibers were cleaned under running water and dried followed by soaked in the solution prepared with 6% of sodium hydroxide to distilled water. Chemical treatment using NaOH helps to remove moisture content from the fibers, improving strength of the adsorbent material (Reddy *et al.*, 2014).

2.6.3.2. Thermal Treatment

The chicken feather was subjected to undergo thermal treatment by two-step of pyrolysis is to enhance the porous nature of chicken feather fiber and its quality into high temperature resistive material (Senoz and Wool, 2010). The pyrolysis process is an irreversible reaction by thermal treatment with the absence of oxygen. The first step of pyrolysis at 195°C which is below the keratin melting point is to investigate the effect of isothermal heat treatment towards the structure as well as mechanical strength for the fibers. As for the second step of pyrolysis at 300°C in order to obtained the microporous material with a narrower pore distribution which less than 1nm for the use of hydrogen adsorption (Senoz and Wool, 2010). (Senoz and Wool, 2010) suggested the temperature of 215°C and 400-450°C for the first and second step of pyrolysis respectively. However leads to unsatisfactory result during previous research. Some modification made by adjusting the thermal heat treatment temperature to improve the efficiency of heavy metal removal.

2.7 Application of Chicken Feather

One of the characteristic of chicken feathers, it contain about 90% of protein (Keratin) and are cheap and renewable source of protein fiber. Thus, the high content of keratin has necessitate interest in researcher to explore the potential of chicken feather for various application. The chicken feather able to remove tetracycline (antibiotic residue), organic dyes, heavy metal ions from wastewater due to their large surface area and several reactive functional group. Besides, keratin extracted from chicken feather also used in the feasibility study of transform the disposable chicken feather into useful protein material (Ma *et al.*, 2016).

2.7.1 Rapid Removal of Tetracycline (TC)

The release of antibiotic residue into the aquatic environment might lead to health issues and caused toxicity to aquatic organisms. Tetracycline (TC) is one of the most widely used antibiotics, often detectable in surface water and groundwater. However, complete removal of TC residue through traditional biological treatment is difficult due to the anti-bacterial characteristic. Thus, new reliable approach related to TC removal begins to develop. The technique used in the present study for the removal of tetracycline using multi-layered graphene-phase biochar (MGB) derived from disposed chicken feather (Li *et al.*, 2017).

2.7.2 Dye Treatment

Textile industry is one of the largest consumer of water in the world. Processing ton of textiles estimated to consume about 80-100 m^3 of water that released into the environment (Fu and Wang, 2011). Although the toxicity of the dye is less widely known but even the trace amount of the dye present in living system may cause several health problem to human and animal. The effect of released textile by-product or toxic dye into the environment has become major concern as it may cause serious health problems such as skin and eye irritation, and damage to respiratory system due to its toxicity. The chicken feather used as an adsorbent for the adsorption of hazardous dye from the aqueous solution. The efficiency of hazardous dye removal is dependent on several parameters such as pH, contact time, adsorbate concentration, amount of adsorbent and temperature. Overall, results showed the adsorption technique can be successfully applied for the removal of toxic and hazardous dye from aqueous solution (Mittal *et al.*, 2013).

2.7.3 Heavy Metal Removal

Removal of some polluting metals such as lead, ion, copper, cadmium, calcium, zinc, magnesium, mercury and nickel in wastewater using chicken feather are studied and use widely in wastewater treatment. The chicken feather used as adsorbent material for the adsorption of heavy metal in wastewater. In addition, the efficiency of heavy metal removal by activated chicken feather is higher as in comparison between several technologies such as chemical precipitation, ion exchange and others. From previous research has proven the ability and potential of chicken feather as natural resources to be low-cost adsorbent due to the unique characteristic of chicken feather itself. The important parameters in metal sorption is dependent on the pH, dosage of adsorbent, initial adsorbate concentration and temperature (Sayed *et al.,* 2005).

2.8 Summary

From the literature review, it can conclude that environmental pollution are associated with the presence of heavy metals in the water. Based on the literature review, the adsorbent used for heavy metal removal reviewed mainly focusing on their pre-treatment, modification, bio-sorption performance, their evaluation as well as potential application in the future. The literature survey indicated that low-cost adsorbent have shown their equal or better adsorption capacities compared to conventional adsorbents which is expensive. Therefore, utilizing natural resources or waste material as a replacement of traditional adsorbents is highly recommended for the removal of heavy metal. Chicken feather are abundant and usually end up in the landfill. However, chicken feather shown its excellent performance to be used as raw material for the preparation of low-cost adsorbent.

This study will focus on utilizing disposed chicken feather as raw material for the preparation of low-cost adsorbent to remove selective heavy metal such as Zinc and magnesium from synthetic wastewater. The treatment method in this study includes chemical and thermal treatment with some modification based on the recommendation from the previous study to for better adsorption of heavy metal. Previous study on chicken feather fiber as low-cost adsorbent has define clear result on the heavy metal removal. However, the production of COD leaching has become the major concern during the study. Therefore, this study will emphasize not only on investigating the characteristic of chicken feather fiber after the two steps of pyrolysis but also to improve the efficiency of heavy metal removal with less production of COD leaching. Improvising Pyrolyzed Chicken Feather Fiber (PCFF) in terms of environmental values is the major concern in this study to ensure it has the capability to be use as low-cost adsorbent.

CHAPTER 3

METHODOLOGY

3.1 Introduction

The experimental study is divided into three stages. The first stage is the preparation of Pyrolyzed Chicken Feather Fiber (PCFF) followed by the selection of best adsorbent of PCFF with different heating temperature and the last stage is to study the removal efficiency of heavy metal ions in the prepared synthetic wastewater.

This chapter discusses in details all the experimental procedure carried out in this study. The flow chart in Figure 3.1 illustrate the summary of flow of study including the preparation of Pyrolyzed Chicken Feather Fiber (PCFF), the preparation of synthetic wastewater, the selection of best adsorbent for batch adsorption studies as well as the experimental design using Design Expert program version 6.0.6 and the analysis on the characteristic of chicken feather fiber. The materials and equipment used in the study are listed in this chapter.

SUMMARY OF RESEARCH METHODOLOGY

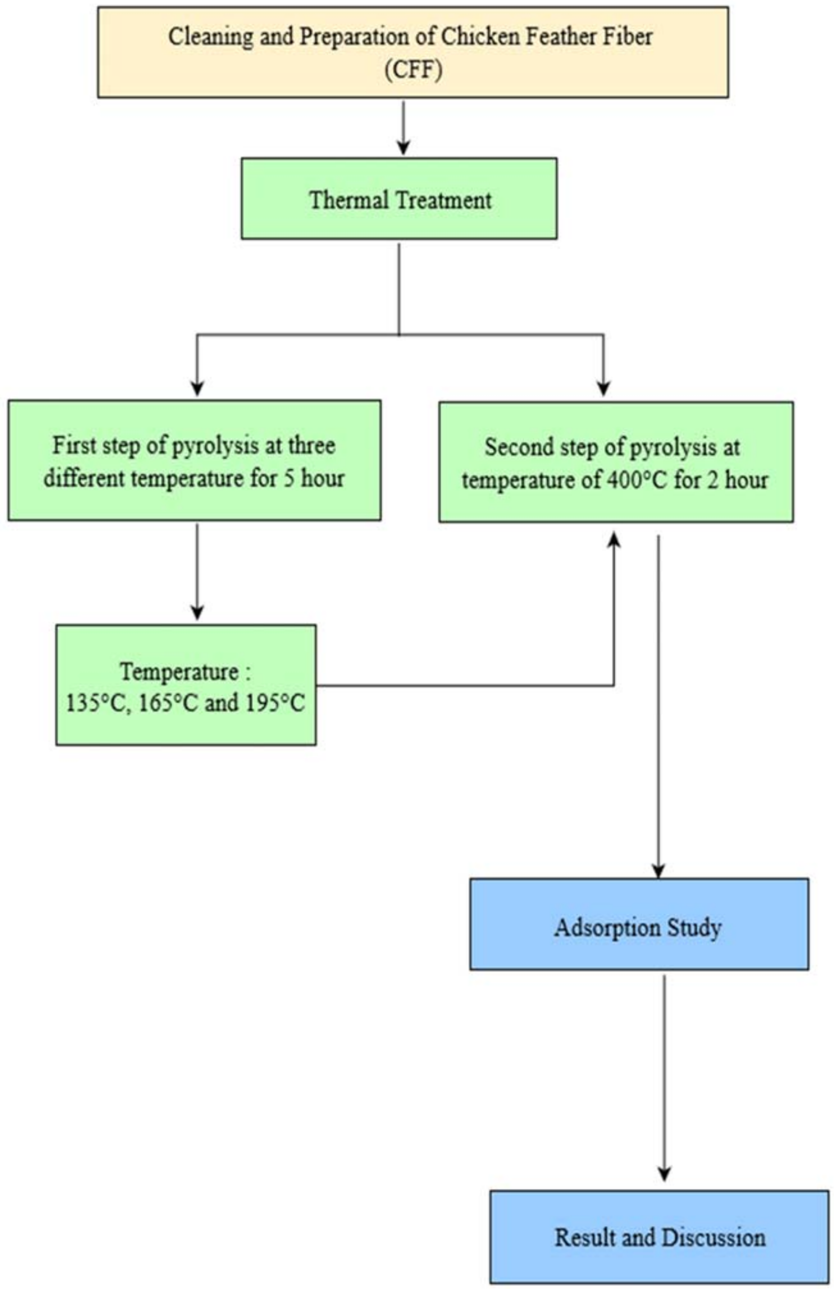

Figure 3.1 : Flow chart for research methodology

3.2 Materials

The main material used in this study was the chicken feather collected from Ayamplus Food Corporation Sdn Bhd located in Nibong Tebal about 4 km away from USM Engineering Campus.

Two different concentration of Ethanol of 95% and 70% were used in the preparation and cleaning of chicken feather. For the preparation of synthetic wastewater, chemical were used in this study. Other chemical such as N/50 Sodium Hydroxide (NaOH) used in the study for pH adjustment of the wastewater sample. Next, gas substance such as nitrogen gas and argon gas were used in this study for pyrolysis process as inert gas as well as for the ICP analysis respectively.

Measuring device used in this study were DR2800 spectrophotometer and Inductive Couple Plasma (ICP) optical emission spectrometer. These two instrument function to measure the concentration of heavy metal ions and also COD in the treated sample. For efficient agitation of sample in conical flask, Orbital Shaker SK-600 Lab Companion were used during the experiment. Next, COD reactor and FAC-350 Oven. Digital balance were used to weight the PCFF adsorbent for treatment. Lastly, H1 284 pH meter for measuring and adjusting pH of the prepared sample.

Figure 3.2 : Orbital Shaker SK-600 Lab Companion

Figure 3.3 : COD Reactor

3.3 Methodology

3.3.1 Preparation of Manganese Synthetic Wastewater

In this study, the removal efficiency of heavy metal focused on Manganese. Volume of 50 mg/L of manganese were prepared from manganese sulphate with atomic weight 54.983 g/mol. As for the preparation, manganese sulphate were diluted with distilled water in a volumetric flask separately then swirled to mix. Next, 200 ml of synthetic wastewater solution was treated by certain weight of PCFF. The calculation for the preparation of the solution from this compound $MnSO_4.H_2O$ are shown in Appendix A.