UNIVERSITI PUTRA MALAYSIA

DEVELOPMENT OF A HYBRID TECHNIQUE BY INTEGRATING ELECTROLYSIS WITH SAGO PALM BARK ACTIVATED CARBON TO TREAT LANDFILL LEACHATE

IQBAL KHALAF ERABEE

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By

IQBAL KHALAF ERABEE

Thesis Submitted to the School of Graduated Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

November 2017

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DEDICATION

To our great leader,

my lovely,

parents, husband, and dearest sons,

my brothers, sisters and closest friends,

I dedicate this work.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

DEVELOPMENT OF A HYBRID TECHNIQUE BY INTEGRATING ELECTROLYSIS WITH SAGO PALM BARK ACTIVATED CARBON TO TREAT LANDFILL LEACHATE

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November 2017

Chairman : Amimul Ahsan, PhD
Faculty : Engineering

In this study, a hybrid technique using electrolysis reactor and activated carbon (AC) adsorption was developed in order to decrease organic and inorganic contaminates which are considered important environmental concerns and an important issue to save the water environment. A sample of raw landfill leachate was collected from the Jeram Sanitary Landfill (JSL).The parameters studied were pH, chemical oxygen demand (COD), biochemical oxygen demand (BOD), total suspended solids (TSS), total dissolved solids (TDS), electrical conductivity, salinity, turbidity, phosphate, ammonia-nitrogen (NH_3-N), nitrate and some heavy metals, e.g. zinc (Zn), copper (Cu), sulphide (S), manganese (Mn) and hexavalent chromium (Cr (VI)).

The maximum removal efficiency for TDS, TSS, COD, BOD, salinity, turbidity, Zn and Mn were 78, 82, 94, 87, 82, 87, 87 and 93%, respectively using 60 V electrical potential at contact time (CT) of 120 min. Secondly, the adsorption process using modified AC with potassium permanganate ($KMnO_4$) showed that the removal of these parameters was proportional to the increase of CT (30-360 min) and adsorbent dose of AC (3-10 g/L). The optimum removal efficiencies of TSS, NH_3-N, Zn, Cu and sulphide were obtained as being equal to 91, 99 86, 100 and 57%, respectively, at CT of 240 min and 10 g/L adsorbent dose using AC-$KMnO_4$.

Two raw materials sago palm bark (SPB) and date pits were utilised as precursors for the preparation of AC by the physicochemical method using activation agents of zinc chloride ($ZnCl_2$), potassium hydroxide (KOH) and sulphuric acid (H_2SO_4). N_2 adsorption-desorption analysis was carried out for porosity characterization of the AC. Thermographimetric (TGA) analysis was also done for the two raw materials. The

prepared AC from SPB (AC-SPB) and date pits (AC-DP) showed the maximum surface area of 1737.72 m^2/g and 1443.45 m^2/g, respectively at 700°C activation temperature.

The maximal removal percentages using the dose of 10 mg/L and CT of 180 mins for BOD, COD, NH_3-N, Zn and Cr (VI) were 93, 95, 78, 78 and 85%, respectively using the prepared AC-SPB. The morphology of the AC was studied through the scanning electron microscopy (SEM) and Energy Dispersive X-Ray Spectroscopy (EDX) patterns. The surface groups present on the AC surface were determined by the Fourier Transform Infrared Spectroscopy (FTIR) analysis. Adsorption isotherms were determined by using Langmuir and Freundlich isotherms. A kinetic study was performed using the pseudo first order and pseudo second order adsorption. The adsorption isotherms of BOD, COD, NH_3-N, Zn, and Cr (VI) using the modified AC were well fitted to the Langmuir isotherm.

The values of final concentrations of heavy metals (Zn, Mn, Cu, sulphide) are (0.5, 0.17, 0.0, and 0.4 mg/L, respectively) lower than the standards for discharge of leachate required by Department of Environment (DOE, 2010), which equal to (2.0, 0.20, 0.20 and 0.50 mg/L), respectively.

Finally, a pilot plant for the treatment of leachate was designed consisting of an electrolysis reactor with a pair of anode electrodes made from aluminum and a pair of cathode electrodes made from iron followed by secondary treatment using the activated carbon adsorption process. The dynamic adsorption behaviour was evaluated using the Thomas, Yoon-Nelson and the Adam's-Bohart models. The experimental results of the fixed-bed column showed increased removal capacity of NH_3-N from 14.33 to 20.81 mg/g. In addition, the Zn and Cr (VI) removal capacities were seen to increase from 7.56 to 8.167 mg/g and 10.5 to 13.23 mg/g, respectively.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PEMBANGUNAN SUATU TEKNIK HIBRID SECARA PENYEPADUAN ELEKTROLIS DENGAN KARBON TERAKTIF KULIT KAYU POKOK SAGU UNTUK MERAWAT LARUT RESAP TAPAK PENIMBUSAN TANAH

Oleh

IQBAL KHALAF ERABEE

November 2017

Pengerusi : Amimul Ahsan, PhD
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Di dalam kajian ini, teknik hibrid menggunakan rawatan elektrolisis dan penjerapan karbon teraktif untuk mengurangkan jumlah pencemaran bahan organik dan bukan organik yang tinggi yang dianggap sebagai salah kebimbangan utama alam sekitar dan yang merupakan isu penting untuk menyelamatkan persekitaran air. Sampel larut resap mentah tapak pelupusan penimbusan tanah yang diambil dari Tapak Pelupusan Sanitari Jeram (JSL) dirawat dengan teknik. Parameter yang dikaji ialah pH, permintaan oksigen kimia (COD), permintaan oksigen biologi (BOD), jumlah pepejal terampai (TSS), jumlah pepejal terlarut (TDS), kekonduksian, kemasinan, kekeruhan, fosfat, ammonia-nitrogen (NH_3-N), nitrat dan beberapa logam berat, contohnya zink (Zn), kuprum (Cu), sulfida, mangan (Mn) dan kromium heksavalen (Cr (VI)).

Kecekapan penyingkiran maksimum untuk TDS, TSS, COD, BOD, kemasinan, kekeruhan, Zn dan Mn adalah 78, 82, 94, 87, 82, 87, 87 dan 93%, masing-masing, menggunakan potensi elektrik 60 V selepas masa sentuhan 120 minit. Kedua, proses penapisan menggunakan AC diubahsuai dengan kalium permanganat ($KMnO_4$) menunjukkan bahawa penyingkiran parameter-parameter ini adalah berkadar dengan peningkatan masa sentuhan (30-360 min) dan dos penjerap AC (3-10 g/L). Kecekapan penyingkiran optimum TSS, NH_3-N, Zn, Cu dan sulfida diperoleh sebagai sama dengan 91, 99 86, 100 dan 57%, masing-masing, selepas 240 min dan 10 g/L dos penjerap menggunakan AC-$KMnO_4$.

Dua bahan mentah iaitu kulit kayu pokok sagu (SPB) dan biji kurma telah digunakan sebagai prapenanda untuk penyediaan AC dengan kaedah fizikokimia menggunakan agen pengaktifan zink klorida ($ZnCl_2$), kalium hidroksida (KOH) dan asid sulfurik (H_2SO_4). Analisis penjerapan-nyaherapan N_2 telah dijalankan untuk pencirian keliangan AC. Analisis termografimetrik (TGA) juga telah dilakukan untuk kedua-dua bahan mentah tersebut. AC yang disediakan dari SPB (AC-SPB) dan biji kurma (AC-DP) menunjukkan kawasan permukaan maksimum 1737.72 m^2/g dan 1443.45 m^2/g masing-masing pada suhu pengaktifan 700^oC.

Peratusan penyingkiran maksimum dilihat bagi dos 10mg/L dan masa sentuhan 180 minit untuk BOD, COD, NH_3-N, Zn dan Cr (VI) adalah 93, 95, 78, 78 dan 85%, masing-masing menggunakan AC-SPB yang disediakan.

Morfologi AC dikaji melalui pemeriksaan kemikroskopan elektron imbasan (SEM) dan corak Kespekroskopan X-Ray Serakan Tenaga (EDX). Kumpulan-kumpulan permukaan yang hadir di permukaan AC ditentukan oleh analisis Kespektroskopian Inframerah Penjelmaan Fourier (FTIR). Isoterma penjerapan telah ditentukan dengan penggunaan isoterma Langmuir dan Freundlich. Satu kajian kinetik dianalisis dengan menggunakan order pertama pseudo dan order kedua pseudo.

Isoterma penjerapan BOD, COD, NH_3-N, Zn, dan Cr (VI) yang menggunakan karbon teraktif diubahsuai sesuai dengan isoterma Langmuir.

Nilai kepekatan akhir ;ogam berat adalah 0.5,0.17, 0.0 dan 0.4 mg/L lebih rendah daripada piawaian untuk pelepasan larut resap yang dikehendaki oleh jabatan persekitaran (DOE, 2010) yang untuk Zn, Mn, Cu dan sulfida bersamaan dengan (2.0, 0.2, 0.2, dan 0. Mg/L) masing-masing.

Akhirnya, kilang perintis untuk rawatan resap direka terdiri daripada reaktor elektrolisis dengan sepasang elektrod anod diperbuat daripada aluminium dan sepasang elektrod katod diperbuat daripada besi diikuti dengan rawatan sekunder menggunakan proses penjerapan karbon teraktif. Tingkah laku penjerapan dinamik dinilai menggunakan model-model Thomas, Yoon-Nelson dan Adam's-Bohart. Keputusan eksperimen turus dasar-tetap menunjukkan peningkatan kapasiti penyingkiran NH_3-N dari 14.33 ke 20.81 mg/g. Selain itu, kapasiti penyingkiran Zn dan Cr (VI) dilihat meningkat dari 7.56 ke 8.167 mg/g dan 10.5 ke 13.23 mg/g, masing-masing, apabila ketinggian dasar ditingkatkan dari 5 ke 15 cm.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

AC	Activated Carbon
AC-DP	Activated Carbon made from Date Pits
AC-Heating	Activated Carbon Modified with Heating
AC-HNO_3	Activated Carbon Modified with Nitric Acid
AC-$KMnO_4$	Activated Carbon Modified with Potassium Permanganate
AC-SPB	Activated Carbon made from Sago Palm Bark
AOP	Advanced Oxidation Processes
BOD_5	Biochemical Oxygen Demand (mg/L)
COD	Chemical Oxygen Demand (mg/L)
Cr (VI)	Hexavalent Chromium (mg/L)
CT	Contact Time (min)
Cu	Copper (mg/L)
DC	Direct Current (A)
EC	Electrical Conductivity (µS/cm)
EDX	Energy Dispersive X-Ray Spectroscopy
FTIR	Fourier Transform Infrared Spectroscopy
GAC	Granular Activated Carbon
JSL	Jeram Sanitary Landfill
Mn	Manganese (mg/L)
MSW	Municipal Solid Waste
N	Nitrogen (mg/L)
NH_3-N	Ammonia Nitrogen (mg/L)
NTU	Nephelometric Turbidity Units

PAC	Powdered Activated Carbon
ppm	Parts per million
RO	Reverse Osmosis
rpm	Revolution per minute
RT	Retention Time (min)
SBR	Sequential Batch Reactor
SEM	Scanning Electron Microscopy
SS	Suspended Solids (mg/L)
TDS	Total Dissolved Solids (mg/L)
TGA	Thermographimetric analysis
TKN	Total Kjeldahl Nitrogen (mg/L)
TOC	Total Organic Carbon (mg/L)
TSS	Total Suspended Solid (mg/L)
Zn	Zinc (mg/L)

CHAPTER 1

INTRODUCTION

1.1 Background

The extreme growth rate of population and methodical development of the technological and industrial sectors have been supplemented with the swift creation of industrial and municipal solid wastes that has the capability of polluting the environment. These wastes are termed as one of the gravest ecological issues faced by the world. In Malaysia, the annual population growth rate has touched 2.4 percent and the rate of creation of municipal solid waste (MSW) too has risen intensely. Consequently, huge amounts of waste, such as commercial, industrial, and agricultural by-products, are despatched to landfills year on year (Akinbile et al., 2012). According to the Census data for 2012, at present, Malaysians are producing around 5,781,600 tonnes of solid waste every year. Considering the overall waste creation of 0.9 kg/capita/day, the volume of solid waste is likely to rise to double digits as the nation takes a step toward becoming an advanced economy by 2020. The MSW sources in the country differ for every local authority area according to the size of the city and economic norms. In Malaysia's southern and central regions, the proportion of wastes is as follows: 36.73 percent is household waste, 28.34 percent is industrial and construction, and 34.93 percent is waste from other sources (Abdullah, 1995).

The choice of a perfect and viable approach for regulating the clearance of huge amounts of MSW in a cost-effective manner, which could evade ecological damages, is quite challenging because of the different deliberations involved (Umar et al., 2010). In fact, landfilling, i.e. disposing the solid waste at landfill facilities, is terms as the most commonplace and sustaining approach of solid waste management that aids in a viable disposal and eradication of residue wastes through separation, recycle and incineration, in advanced as well as emerging economies (Baldasano et al., 2003). Thanks to its simplicity of operation, technical viability, minimum supervision and low operating outlay, sanitary landfilling is today the most preferred approach for disposing of solid waste. In the majority of the nations, landfilling is the most suitable approach for getting rid of MSW, considering the technology utilisation and capital outlay (Renou et al., 2008). In Malaysia, landfills are the preferred approach for disposing of MSW (Manaf et al., 2009; Yusof et al., 2009), and the majority of the sites are open dumping zones (Manaf et al., 2009). Such landfills are preferred because they are comparatively inexpensive and most commonplace for treating solid waste that has a high proportion of organic elements (Ngoc and Schnitzer, 2009). Use of landfills has significantly subjected river water to pollution from leachate, making appropriate leachate management necessary. In Malaysia, there are 261 landfills, and over 80 percent of them practise open dumping or controlled tipping. This can be ascribed to the lower operation and upkeep costs as against other established methods such as incineration and advanced landfill mechanisms (Halim et al., 2010). World over, up to 95 percent of the total MSW gathered is disposed through landfills (Kurniawan, et al., 2006). In a contemporary sanitary landfill, its structure and design are the most vital factors. Usually, the structure comprises a quarried site that has several "*cells*" that are distinct yet linked in order to operate as a unified system. Figure 1.1 depicts the preferred constituents of a well-regulated landfill disposal site (Knox, 2000).

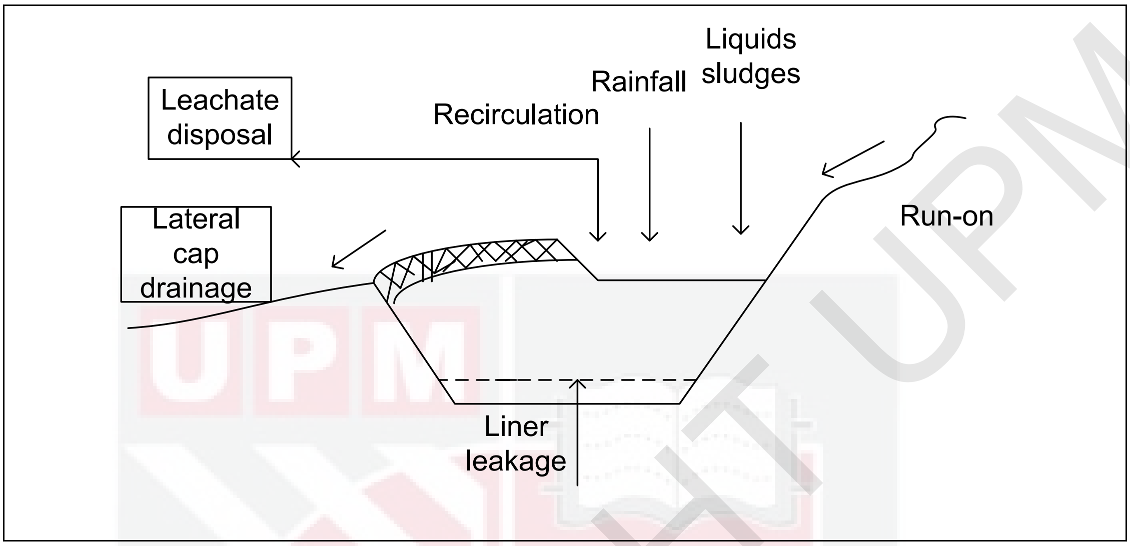

Figure 1.1 : Schematic landfill cross-section, depicting characteristic constituents of a water budget of a landfill (Knox, 2000)

When the water circulates through landfill deposits, the waste pollutes the water, leading to leachate. This leachate comprises biologicals, soluble chemicals, and metal ions (for example, iron). Leachate is acidic as well as anoxic, with a distinct stench. Even though the bacteria will reduce the waste to a condition that would will be comparatively harmless if it escapes into the environment, it might take several hundreds of years. If a landfill does not have a technique of gathering and cleansing the leachate, it will continue to enter and pollute groundwater streams.

Treatment of landfill leachate can be done through different chemical, physical, and biological treatment approaches. System designs for treating active leachate include biological procedures for the elimination of labile organic components and nitrogen, flocculation and coagulation and physical settling and filtrations for the elimination of phosphorus, and recalcitrant organic as well as inorganic components. Moreover, mechanisms for treating active leachate can comprise air stripping for nitrogen elimination, and physiochemical procedures like ion exchange and sorption (Deng and Englehardt, 2007).

The electrochemical technique for treating wastewater has drawn significant attention, primarily due to the simplicity of operation and the higher efficacies obtained through the usage of electrocatalytic electrodes with a longer lifetime (Comninellis and Pulgarin, 1993; Lamy, 1984). The electrolysis process functions on the fundamental of reductive or oxidative chemistry. It entails comparatively modest equipment along with an ambient temperature as well as pressure. The main process in electrolysis is the exchange of ions and atoms along with addition or elimination of electrons from an outside source (Cho et al., 2010).

The adsorption process is utilised as a phase of assimilated chemical–physical–biological process for treating landfill leachate (Morawe et al., 1995; Geenens et al., 2001), or concurrently with a biological process (Loukidou and Zouboulis, 2001; Kargi and Pamukoglu, 2003). Powdered or granular activated carbon is the most often utilised adsorbent. Carbon adsorption enables 50–70 percent elimination of ammonia nitrogen as well as COD (Amokrane et al., 1997). The objective of activated carbon adsorption is to (i) warrant final polishing level by eliminating lethal heavy metals or organics (ii) aid microorganisms.

1.2 Problem Statement

The express rate of industrialisation, urbanisation, and population expansion has driven generation of huge volumes of solid waste. Malaysia has 261 landfills, and the volume of municipal solid waste (MSW) has been increasing over the years. According to the national strategic plan on management of solid waste, the rate of waste management rises by 3.59 percent every year; this estimate is derived from the predictions of population growth for the period 2002–2020 (NSWMD, 2013; Abumutha et al., 2011). These lead to a rise in the amount of landfill. The gathering of solid waste at landfill sites might lead to water contamination particularly in the rainy season through the leachate produced by the rainfall and the flowing of surface water into the landfill. Consequently, this leachate can detriment the quality of ground water through different composition contents, including high COD, high BOD, ammonia nitrogen, suspended solids, cyanide, mercury, zinc, lead, copper and other heavy metals. These cause greater complex composition of chemicals, which is the key issue in the administration of landfill leachate (Nora, 2006). Landfill leachate management has emerged as one of the key focus areas for environment management. Today, the key emphasis in the process of leachate treatment is enhancing the treatment through certain advanced techniques, and that too in a cost effective and effectual manner.

Nowadays, many technologies are used to treat landfill leachate such as biological processe s and physical-chemical treatment but they are totally inefficient for the toxic naure of stabilized leachate. However, satisfactory results cannot be achieved using a sole treatment for landfill leachate (Kargi et al., 2003; Oller et al., 2011); this is due to frequent nutrient imbalances like carbon conten, high ammonium nitrogen and low phosphourus (Zhao et al., 2012). Futhermore, a high cost of chemical, sludge disposal, generation of sludge are some of problems, which hamper the individual use of coagulation-flocculation in leachate treatment. These problems have led to the realization that there is an urgent need to think of alternative method easy in use and economical in cost for treatment of landfill leachate.

The electrolysis method is the most extensively deployed approach in environmental safeguard and wastewater treatment. This can be attributed to its ease of use and economical cost as well as efficiency in eliminating the most common existing contaminants from wastewater. This process is termed as an alternate approach towards breaking down contaminants that resist biological dilapidation (Reddy et al., 2003). When direct current is used to treat leachate, several chemical and physical processes occur. These include anodic oxidation and cathode decline of impurities existing in leachate (Probstein et al., 1993).

Activated carbon is termed as an effective adsorbent for wastewater contaminants due to its high adsorption capacity pertaining to high surface area and pore volume. Furthermore, the raw material utilised for producing activated carbon is an inexpensive material with a high amount of carbon (Bansal et al., 1988). This includes wood, rice husk, oil palm shell, coconut shell, and saw dust.

This study represents a novel hybrid technique through developing low cost treatment process by integrating electrolysis, aeration and adsorption. The adsorption process was conducted in this study, using highly porosity characterizations activated carbon from highly cellulosic agricultural waste used for the first time as an adsorbent, which is sago palm bark. This surface area precursor leads to increase the removal efficiencies for most of parameters (BOD, COD, NH_3-N, Zn and Cr (VI)) and contributes in reduction treatment cost and saves energy, and helps in protecting the environment.

The type of activated carbon contractor which used in this study is the up flow mode (fluidized bed mode). The main advantages of this type of flow is the carbon can be continuously removed from the bottom of the contactor as it is exhausted. Fresh make-up carbon can than be added to the top of contactor at the same rate. This process will eliminate the need to shutdown the contactor after exhaustion occurs. Up flow fluidized beds also minimize clogging and unintentional filtration. Moreover, the design of pilot plant system-integrating electrolysis, aeration and adsorption treatment have not been studied until now. It is hoped that this type of system will help in decreasing the concentrations and increasing the removal efficiencies of various pollutants in landfill leachate which leads to discharge unpolluted solution to the groundwater and stream water and which also helps in solve one of the most important environmental problems.

1.3 Objectives

The main aim of this study is to develop a hybrid technique by integrating electrolysis with activated carbon (AC) to treat landfill leachate. The specific objectives are:

i) To examine the electrolysis process performance through a laboratory analysis of raw and treated landfill leachate using a standard technique.
ii) To investigate the effect of different modification methods of commercial AC on the porosity characterizations of AC and examine the effect of sorption efficacies of modified AC to decrease pollutant concentrations.
iii) To produce microporous AC from two distinct raw materials: date pits and sago palm bark, and examine the impact of different chemical activating agents, such as $ZnCl_2$, H_2SO_4, and KOH on the porous attributes of AC.
iv) To assess the efficacy of prepared AC in eliminating heavy metals and organic contaminates from landfill leachate and compare the outcomes with commercially available AC.
v) To develop a lab scale pilot plant by integrating electrolysis reactor with AC adsorption for landfill leachate treatment.

1.4 Scope of the Study

This study offers an improved solution for landfill leachate treatment. The leachate was collected from the Jeram Sanitary Landfill (JSL). The aim is to augment the removal efficacies of various parameters such as salinity, pH, electrical conductivity, turbidity, TDS, TSS, BOD, COD, nitrate, ammonia-nitrogen, and heavy metals such as Mn, Zn, Cu, and sulphide. The process of treatment is split into two parts: (i) the primary treatment involves deploying the electrolysis process at different contact times as well as voltages; (ii) the secondary treatment involves utilising a filter of AC with various contact times, to ascertain the removal efficacies of organic contaminants and heavy metals in samples of leachate. The study involved utilising various modification techniques on the commercial AC produced from coconut shell. Furthermore, an effectual microporous adsorbent is developed from three distinct raw materials, namely date pits and sago palm bark.

1.5 Thesis Composition and Structure

This thesis contains seven chapters. Here is a short description of the contents of each chapter.

Chapter 1 offers the general introduction. It offers information on the issues of municipal solid waste management in Malaysia, i.e. the creation of landfill leachate, and elucidates the means of treatment. The chapter then outlines the key research objective and the research structure stated in the thesis.

Chapter 2 offers a review of prior literature which elucidated the most commonly utilised biological and physical-chemical treatment approaches; the attributes of landfill leachate; aspects which impact the creation of leachate; and electrolysis, an AC technique.

Chapter 3 discusses the methodology of experimental work and outlines the approach and materials utilised in landfill leachate analysis and treatment. This is split into four parts: (i) the scrutiny of landfill leachate samples, which would be sourced from the study area; (ii) deploying the electrolysis process for treating the samples; (iii) the treatment phase that uses activated carbon; and (iv) devise a pilot plant for landfill leachate treatment through integrated electrolysis with activated carbon.

Chapter 4 contains two key sections of results, the first section represents the results and discussions of raw leachate analysis and the second section includes the results and discussions of the electrolysis treatment, pertaining to results achieved from the relevant laboratory experiments concerning their effect on improving the quality of leachate. The outcomes of both experiments are centred on 13 key leachate parameters. Both experiments assessed the likelihood of treating the quality of leachate in an easy, inexpensive, and effectual manner through the electrolysis process.

Chapter 5 includes two parts of results; the first one discusses leachate treatment through commercial AC modified with three distinct techniques. Moreover, the second part concludes the results of preparation method of AC from two raw materials and elucidates the process of chemical activation of AC with different chemical agents. In addition, abstracts the using of prepared AC from sago palm bark for adsorption of heavy metals from landfill leachate sample.

Chapter 6 includes the results of landfill leachate treatment with a hybrid technique by integrating electrolysis with activated carbon through design a pilot plant. In addition, it includes the estimation analysis of the pilot plant and evaluates dynamic behavior of the fixed-bed adsorption results using the Thomas, Yoon-Nelson and Adam's-Bohart models.

Chapter 7 offers a summary and concludes the thesis by emphasising the results of the study concerning its application in leachate treatment in JSL. Certain guidelines for future research are discussed at the end of the chapter.

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