UNIVERSITI PUTRA MALAYSIA

ADSORPTION OF LEAD FROM AQUEOUS SOLUTION USING MODIFIED ACTIVATED CARBON PREPARED FROM PALM KERNEL SHELL

PAM ALOYSIUS AKAANGEE

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By

PAM ALOYSIUS AKAANGEE

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

March 2018

DEDICATION

To my dearest parents, Mr. Cletus Pam Amo and Mrs. Juliana Pam (late), for their love and prayers.

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

ADSORPTION OF LEAD FROM AQUEOUS SOLUTION USING MODIFIED ACTIVATED CARBON PREPARED FROM PALM KERNEL SHELL

By

ALOYSIUS AKAANGEE PAM

March 2018

Chairman : Associate Professor Abdul Halim Abdullah, PhD
Faculty : Science

Motivated by adsorbent/liquid-interactive interfacial character, a major study within this thesis examines lead (Pb) adsorption on activated carbon. Pb was chosen as a model because it is abundant in aqueous waste, a potential risk factor for both human health and the environment and the focus of prior scientific investigation. Convectional techniques for Pb removal in aqueous solutions have inherited limitations and collateral effects. Adsorption onto solid adsorbent especially activated carbon, is an excellent strategy to eliminate Pb(II) from solution due to the high efficiency of adsorption and sorption capacity. On this front, activated carbon (AC) was prepared by chemical treatment of palm kernel shell (PKS) using H_3PO_4 and later modified using citric acid (CA) and ethylenediaminetetraacetic acid (EDTA).

The respective physicochemical properties of the AC itself and the modified AC are identified using FTIR, FESEM, BET surface area and elemental analysis. The adsorption behavior of modified activated carbon is also put into context by calculations of interaction potentials through kinetics and isotherm models. Adsorption in batch method are benchmarked against adsorption in column studies, where the latter represents a real-world industrial situation and with potential for use in industries for water treatment. The activated carbon produced were mainly microporous in nature with BET surface area and pHpzc ranged from 548 m^2/g to 1560 m^2/g and 2 to 4.5, respectively. AC-600 2:1 exhibited the highest Pb(II) removal and was subjected to CA and EDTA modification. The adsorption process was endothermic and spontaneous in nature and the data fitted the pseudo-second order kinetics and Langmuir isotherm model. The maximum adsorption capacity of Pb(II) was in the sequence of AC-PKS (86.2 mg/g) < AC-CA (103.1 mg/g) < AC-EDTA (104.2 mg/g), at 25 ± 1°C and pH 5. Chemical reaction and mass transfer at the interface were the rate limiting steps during the adsorption of Pb(II). Both AC-CA and AC-EDTA showed good regeneration and reusability properties for Pb(II) adsorption. The result of the competitive adsorption studies, involving Pb(II), Cu(II) and Zn(II) showed strong antagonism in the multi-ions adsorption with the adsorbents showing more affinity towards Pb(II). The modified activated carbons also showed better adsorption performance in removing Pb(II) from electroplating wastewater than from the river water sample.

In the fixed-bed column studies, both the flow rate and bed height influenced the column performance; increase in flow rate resulted in early breakthrough and exhaustion time, though with less adsorption of Pb(II). On the other hand, increased bed height leads to extended exhaustion time with improved column performance. Thomas and Yoon-Nelson models successfully modeled the breakthrough curve for dynamic adsorption of Pb(II) on the modified activated carbon. In conclusion, the adsorbents can adsorbed Pb(II) in both batch and column studies. The modified ACs exhibited higher adsorption capacities due to OH, COOH, and NH complexation with the metal ions. The performance of these adsorbents, benchmarked against other low cost adsorbents, is promising. These findings illustrate that the metal ion adsorption at the ionic scale in aqueous environment is through ion exchange, π-π interactions or complexation reaction.

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

PENJERAPAN PLUMBUM DARI LARUTAN AKUEUS MENGGUNAKAN KARBON TERAKTIF DIUBAHSUAI YANG DISEDIAKAN DARIPADA TEMPURUNG KELAPA SAWIT

Oleh

ALOYSIUS AKAANGEE PAM

Mac 2018

Pengerusi : Profesor Madya Abdul Halim Abdullah, PhD
Fakulti : Sains

Didorong oleh sifat antara muka penjerap/cecair yang interaktif, kajian ini mengkaji penjerapan plumbum (Pb) daripada larutan akueus pada karbon aktif. Pb dipilih sebagai model kerana ia banyak terdapat dalam sisa akueus, faktor risiko yang berpotensi kepada kesihatan manusia dan alam sekitar, dan adalah fokus penyelidikan saintifik sebelum ini. Teknik konvensional untuk penyingkiran Pb dalam larutan akueus mempunyai batasan dan kesan sampingan. Penjerapan kepada penjerap pepejal terutamanya karbon teraktif, merupakan strategi yang sangat baik untuk menyingkirkan Pb(II) daripada larutan kerana kecekapan dan kapasiti penjerapan yang tinggi. Berdasarkan ini, karbon teraktif (AC) telah disediakan dengan mengenakan rawatan kimia terhadap tempurung kelapa sawit (PKS) menggunakan H_3PO_4 dan kemudian diubah suai menggunakan asid sitrik (CA) dan asid etilenediaminetetraasetik (EDTA).

Ciri fizikokimia AC dan AC diubahsuai masing-masing telah dikenal pasti menggunakan FTIR, FESEM, luas permukaan BET dan analisis unsur. Tingkah laku penjerapan karbon teraktif diubah suai juga dimasukkan ke dalam konteks dengan mengira potensi interaksi melalui model kinetik dan isoterma. Kaedah penjerapan kelompok menjadi tanda aras terhadap penjerapan dalam kajian turus, di mana kajian turus mewakili keadaan sebenar di industri dan dengan potensi untuk digunakan dalam industri untuk rawatan air. Karbon teraktif yang dihasilkan adalah berliang mikro dengan luas permukaan BET dan pHpzc berkisar masing-masing antara 547.8 m^2/g hingga 1559.9 m^2/g dan 2 hingga 4.5. AC-600 2:1 mempamerkan penyingkiran Pb(II) tertinggi dan digunakan dalam pengubahsuaian dengan CA dan EDTA. Proses penjerapan adalah endotermik dan spontan dan data penjerapan mematuhi tindak balas tertib pseudo kedua dan model isoterma Langmuir. Kapasiti penjerapan maksima

Pb(II) adalah dalam urutan AC-PKS (86.2 mg/g) < AC-CA (103.1 mg/g) < AC-EDTA (104.2 mg/g) pada 25 ± 1°C dan pH 5. Tindak balas kimia dan permindahan jisim di antara muka dianggap sebagai langkah penentu kadar semasa penjerapan Pb(II). Kedua-dua AC-CA dan AC-EDTA mempamerkan sifat kebolehjanaan semula dan kebolehgunaan semula yang baik bagi penjerapan Pb(II). Hasil kajian penjerapan kompetitif yang melibatkan Pb(II), Cu(II) dan Zn(II) menunjukkan antagonisme yang kuat dalam penjerapan pelbagai ion dengan penyerap yang menunjukkan afiniti yang lebih terhadap Pb(II). Karbon teraktif yang diubahsuai juga menunjukkan prestasi penjerapan yang lebih baik dalam menyingkirkan Pb(II) dari air sisa industri penyaduran daripada dari sampel air sungai.

Dalam kajian turus lapisan tetap, kedua-dua kadar aliran dan kedalaman lapisan mempengaruhi prestasi turus; peningkatan dalam kadar aliran menyebabkan masa penembusan dan penepuan yang awal tetapi dengan penjerapan Pb(II) yang berkurangan. Sebaliknya, peningkatan dalam kedalaman lapisan melanjutkan masa penepuan dengan peningkatan prestasi turus. Model Thomas dan Yoon-Nelson berjaya memodelkan lengkung penembusan untuk penjerapan dinamik Pb(II) pada karbon teraktif yang diubahsuai. Kesimpulannya, penjerap dapat menjerap Pb(II) dalam kedua-dua kajian kelompok dan turus, AC diubahsuai mempamerkan penjerapan yang lebih tinggi disebabkan oleh pengkompleksan OH, COOH, dan NH dengan ion logam. Prestasi adsorben, yang ditanda aras dengan adsorben kos rendah yang lain, adalah memberangsangkan. Penemuan ini menggambarkan penjerapan ion logam, pada skala ionik, dalam persekitaran berair adalah melalui pertukaran ion, interaksi π-π atau tindak balas pengkompleksan.

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I certify that a Thesis Examination Committee has met on 13 March 2018 to conduct the final examination of Pam Aloysius Akaangee on his thesis entitled "Adsorption of Lead from Aqueous Solution using Modified Activated Carbon Prepared from Palm Kernel Shell" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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Name of Member of Supervisory Committee: Professor Dr. Zulkarnain Zainal

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LIST OF ABBREVIATIONS

AAS	Atomic Absorption Spectrometer
AC	Activated carbon
AC-500 1:1	Activated carbon synthesized at 500 ºC H_3PO_4 to precursor ratio of 1:1
AC-500 2:1	Activated carbon synthesized at 500 ºC H_3PO_4 to precursor ratio of 2:1
AC-600 1:1	Activated carbon synthesized at 600 ºC H_3PO_4 to precursor ratio of 1:1
AC-600 2:1	Activated carbon synthesized at 600 ºC H_3PO_4 to precursor ratio of 2:1
AC-600 3:1	Activated carbon synthesized at 600 ºC H_3PO_4 to precursor ratio of 3:1
AC-600 1:2	Activated carbon synthesized at 600 ºC H_3PO_4 to precursor ratio of 1:2
AC-700 1:1	Activated carbon synthesized at 700 ºC H_3PO_4 to precursor ratio of 1:1
AC-700 2:1	Activated carbon synthesized at 700 ºC H_3PO_4 to precursor ratio of 2:1
ART	Attenuated Total Reflectance
BJH	Barrett-Joyner-Halenda
BET	Brunauer-Emmet-Teller
EDTA	Ethylenediaminetetraacetic acid
FESEM	Field Emission Scanning Electron Microscopy/Microscope
FTIR	Fourier Transform Infrared Spectroscopy
pHpzc	pH of point of Zero Charge
PKS	Palm kernel shell
NCA	Non-conventional adsorbents

C_t	Liquid phase concentration at time (t)	mg/L
C_b	Breakthrough concentration	mg/L
C_e	Liquid phase equilibrium concentration	mg/L
C_0	Initial liquid phase concentration	mg/L
C_{se}	Equilibrium concentration	mg/L
K_f	Freundlich capacity factor	$(mg/g)(L/mg)^{1/n}$
K_L	Langmuir constant	L/g
K_T	Thomas model constant	L/min. mg
k_1	First-order adsorption rate constant min-1	min^{-1}
k_2	Second-order adsorption rate constant	g/mg. min
m	Mass of adsorbent	g
n	Freundlich isotherm exponent	
q	Adsorbed capacity of bed	mg/g
q_e	Adsorbed phase equilibrium concentration	mg/g
q_m	Maximum adsorbed phase concentration	mg/g
q_o	Thomas model bed capacity	mg/g
R^2	Correlation coefficient	
t	Time	min
t_b	Breakthrough time min	min
m/s	U Superficial fluid velocity	m/s
V_b	Breakthrough volume of packed bed	L

CHAPTER 1

INTRODUCTION

1.1 Background Studies

The release of heavy metals into the environment by anthropogenic activities is becoming a worldwide problem. Industrial activity, domestic effluents, and discharge of wastewater remain the main pathway these species can enter natural waters (Ray & Shipley, 2015). Among these metals, lead (Pb) is highly distributed and principally found in aqueous waste (Saka & Mas, 2012). Industries, especially ceramic and glass, acid battery manufacturing, ammunition, metal plating and finishing, have been implicated for discharging lead contaminated water without adequate treatment to the surroundings (Chowdhury, 2012). The current threshold level of lead concentrations in drinking water is 0.05 mg/L (United States Environmental Protection Agency, 2011). It is essential, therefore, that the level of lead in drinking water should be less than this threshold. Repeatedly exposure to lead ions through drinking water could have adverse health consequences such as damage to the central nervous system, kidney, hemopoiesis, heart and blood vessels, and internal secretion system of human beings (Gong *et al.*, 2015). Lead is also bio-cumulative through food or water (Alothman *et al.*, 2016; Chen *et al.*, 2015); hence, the urgent elimination of Pb from water sources is vital before bioaccumulation and bioconcentration through the food chain take place. The numerous conventional methods, utilized to remove Pb(II) from aquatic recipients, include ion-exchange, membrane filtration, chemical precipitation, solvent extraction, electrochemical treatment, etc. (Chen *et al.*, 2015). However, applications of these techniques on large scale become difficult due to some technoeconomic reasons, such as generation of large volume of toxic sludge, exorbitant cost of maintenance and operation, complicated treatment procedure, time taking, and production of secondary pollution (Amad, *et al*, 2011; Chen *et al.*, 2015), due to addition of chemicals. In addition, these methods could be ineffective, particularly for metal solutions with concentrations of up to 100 mg/L (Azizi *et al.*, 2016). Hence, the protection of water resources requires technologies that can efficiently eliminate micro-pollutants in a fast and cost effective manner (Njoku *et al.*, 2014). Relatively, adsorption process is attracting much interest in water treatment in recent times because it is operationally flexible, non-harmful by products generation and cost effective technique (Álvarez-Torrellas *et al.*, 2016).

Consequently, successful attempts by different researchers have been made to develop various novel adsorbents of different sources of low cost and abundant materials (Njoku *et al.*, 2014). Although these materials have proven to be efficient to some extent (Soltani *et al.*, 2013), activated carbon (AC) stands out as the most used and efficient adsorbent due to its exceptional surface chemistry and porosity (Njoku *et al.*, 2014). Quite often, coal and lignite are the precursors for production of commercial activated carbon by steam activation (Vanderheyden *et al.*, 2016), which is expensive and in addition, brings about diminution of fossil fuel materials (Kong *et al.*, 2016).

Consequently, many researchers have studied the feasibility of cheaper substitutes for the preparation of AC in order to remove heavy metals from industrial wastewaters, either by physical or chemical means, using different starting materials, such as rice straw (Adinaveen *et al*, 2015), sunflower seeds (Donaldson *et al*, 2012), coconut shell (Buah & Williams, 2012), fox-nut shell (Kumar and Jena, 2015), pine cone (Carlos and Hu, 2016), *Typha orientalis* leaves (Anisuzzaman *et al*., 2015) etc.

Taking into account the important role structural variations the starting material contributes in the development of porosity of the carbon, it is essential to choose a suitable precursor (Álvarez-Torrellas *et al.*, 2016). Out of the numerous agricultural wastes investigated as adsorbents for the elimination of pollutants, oil palm– derived agricultural wastes, have an amazing removal response for pollutants, including heavy metals (Amad *et al*., 2011).

Malaysia is one of the largest producers and exporter of palm oil products, including crude palm oil (CPO) and crude palm kernel oil (CPKO) (Poudel *et al.*, 2016). The concomitant waste products from the milling industrial process of the palm oil produce, include palm trunks, palm mesocarp fibre (PMF), empty fruit bunches (EFB), palm fronds, spikelets, palm kernel shells (PKS) and palm oil mill effluent (POME) and other waste products (Poudel *et al.*, 2016; Sani *et al.*, 2015). Out of these industrial waste products, only 60% of the palm fibers and shells are put into use as the boiler fuel to generate steam and electricity in the mills (Shafie *et al*, 2012). In addition, most of the power plants used the steam turbine, and it is observed that only a few power plants in palm oil mill industry use the waste of their products to generate electricity (Shafie *et al.*, 2012). These highlight the need to transform the remaining waste biomass by exploring new economical applications. The pollution of aquatic ecosystems in Malaysia has emerged as a major ecological problem concurring with rapid industrial development and urbanization (Al-Shami *et al.*, 2010). Palm kernel shell has demonstrated to be a promising material for the preparation of activated carbon (Lim *et al*, 2010; Xu et al., 2017). As a result, this research work explored the use of PKS activated carbon for surface attachment as a potential strategy to develop an economically feasible technique to efficiently remove Pb(II) from aqueous solution.

AC, can basically be fashioned in two ways: physical and chemical methods. Among these methods of AC preparations, the chemical method is more often applied than the physical activation (Mussatto *et al.*, 2010). H_3PO_4 is a common chemical activating agent, often used for preparing AC from a variety of precursors due to the relatively lower, environmental and toxicological constrains compared to $ZnCl_2$, and lower working temperature compared with KOH or NaOH (Li *et al.*, 2015).

To that effect, the new study attempts to prepare activated carbon from PKS using H_3PO_4 and to find out whether the improved surface chemistry of AC- PKS with citric acid and EDTA salt, will give efficient impact on Pb(II) removal from aqueous solutions in batch process. In addition, to explore the potential use of these adsorbents as cost effective and efficient alternative in an industrial situation, by using continuous adsorption studies in column. Although this work was initially design to be focused on the removal Pb(II) in both synthetic and real wastewater, it was latter extended to Zn and Cu in situations where the initial concentration of these metals in real samples were deemed to exceed the permissible levels. Various characterization methods were employed to identify and verify the existence of surface functional groups of the activated carbons and textural characteristics using nitrogen adsorption isotherm, Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM) and Boehm titration.

1.2 Problem Statement and Justification

Pollution of water sources due to dumping of untreated wastewater and chemical wastes directly into rivers, lakes and drains, has been of great concern over the last decades (Feng and Guo, 2012). Unlike the organic pollutants, industrial wastewater containing heavy metals are non-biodegradable, persistent and recalcitrant in the environment and can bioaccumulate in living tissues of humans, causing disorders, such as cancer; thus the need for their removal before discharge (Liang *et al.*, 2009).

Removal of heavy metals from wastewater pose a serious challenge, hence a wide range of methods has been explored. Adsorption by using AC, which is the widely used adsorbent in treatment of wastewater, has proven to perform very well for the removal of heavy metals, however, the challenge remains the high cost of the commercial available AC, hence there is need for alternative cheaper methods. The search for low cost and easily available adsorbents has led to investigation of agricultural waste materials as potential adsorbents, due to their availability, eco-friendly, simple processing method with less time, renewable in nature and high metal removal efficiency due to their distinctive chemical make-up (Dawood *et al.*, 2016). Especially, the adsorbents obtained from PKS have spurred great interest in the adsorption of heavy metal ions from aqueous solutions (Hesasa *et al*., 2013). This is because of the particular physicochemical properties of the PKS, such as high carbon content, internal porous structure, less ash (Rafatullah *et al.*, 2013), high specific surface area, different chelating groups, eco-friendly and cost-effective, which are appropriate properties of a suitable precursor for synthesis of high-grade AC (Lim *et al*, 2010). More importantly, when this AC is chemically modified, exhibits improved interfacial region, selectivity and enhanced adsorption capacities (Albishri & Marwani, 2016), but the high efficient utilization of the adsorbent capacity and ease of scaling up the process in column, makes it a preferred material for the purpose of industrial wastewater treatment. Citric acid and EDTA, polycarboxylic and aminopolycarboxylic acids are often used as chelating agents, because of their strong metal complexing properties.

On this front, limited research has been observed for the removal of Pb(II) using PKS based AC with surface modification with citric acid and EDTA in treatment of Pb(II) in aqueous solution in fixed bed column operation, and that has brought to the fore an exciting interest in undertaking this present research. Knowing that metals can co-exist in wastewater, co –adsorption of heavy metals is also of interest in this research work.

This is expected not only to develop cheaper materials for water treatment but also to provide a solution for clean water especially in industrial establishments.

1.3 Hypothesis

Adsorption of heavy metals to solid adsorbents is strongly governed by the physicochemical properties of the adsorbents and selectivity of the metals to the adsorption systems. Because of the high surface area and well developed pore structure of AC (Njoku *et al.,* 2014), it is prone to adsorb heavy metals from aqueous solutions. It is therefore hypothesized in this work that the level of Pb(II) would decreased as aqueous solution containing Pb(II) ions is treated using AC.

1.4 Significance of the Research

Because of the growing human population coupled with rapid industrial development and urbanization, there is a tremendous demand and pressure on natural resources, sometimes with a negative connotation of accompanying depletion of these resources on an industrial scale. For sustainable development to be achieved, efficient and cost-effective use of such resources most be sustained. Therefore, there is need to continually monitor anthropogenic activities with respect to their nature, volume, direct harmful effects, potential impacts on the environment as well as develop and applied cost effective and efficient technics for remediation where these activities become pollutional in nature. In that context, studying alternatives for wastewater treatment and management is important. This study involves minimization of environmental pollution through remediation of toxic metal ions from industrial wastewater using activated carbon from palm shells. The findings of this research will be useful in developing an efficient alternative means of water remediation in place of the conventional ones. The technique, if applied in treatment of metal ions containing effluent from various industrial establishments before discharged, will prevent the release of hazardous metal ions to water-bodies, which can poisoning aquatic as well as human life.

In addition, the utilization of PKS, as an agro-industrial by product for the production of an important commercial product like activated carbon, will also serve as an economic solid waste-management measure in the palm oil industry, and minimized the hazardous emissions that usually accompanied the burning of these agricultural wastes.

1.5 Aim and Objectives of Research

The main aim of this research is to evaluate the potential of converting palm kernel shells, a low-cost agro-industrial by-product, to commercial-grade activated carbon using H_3PO_4 as the chemical activation reagent with the following specific objectives:

(i) To improve the selectivity and binding properties of the produced activated carbon by modifying with Citric acid and EDTA
(ii) To determine the adsorption capacity of the adsorbents for metal ions uptake using different parameters such contact time, operational temperature, initial ion concentration and pH
(iii) To evaluate the data obtained using kinetic models, equilibrium adsorption isotherms and thermodynamic parameters.
(iv) To apply the developed and modified activated carbon for the removal of Pb(II) from wastewater through single and competitive mode with Cu(II) and Zn(II) ions.
(v) To perform column studies to investigate the Pb uptake characteristics of modified AC under different flow rates and depth.

1.6 Scope of work

The scope of this work includes the development of 8 kind of adsorbents using PKS (AC-700 2:1, AC-700 1:1, AC-600 3:1, AC-600 2:1, AC-600 1:1, AC -600 1:2, AC-5002:1 and AC-500 1:1). Out of these, the optimum adsorbent (AC-PKS) was chosen and applied in subsequent experiments. Other categories of adsorbents were developed and used at different stages of this study, including AC-EDTA and AC-CA, obtained by modifying AC-PKS with EDTA and citric acid, respectively. These adsorbents were employed in both batch (synthetic, electroplating and river wastewater) and column experiments (limited to synthetic wastewater). The AC-PKS, was used only for batch adsorption experiments (synthetic wastewater). The kinetic and isotherm experiments were investigated through batch mode using synthetic wastewater. The isotherm data were fitted by Freundlich and Langmuir models, while the pseudo first order, pseudo second order and the intraparticle diffusion models, described the data from the kinetic studies. The effect of column operating parameters and the breakthrough curves were modeled through Thomas and Yoon-Nelson. Reusability studies using batch mode were restricted to synthetic wastewater, while competitive adsorption studies, were focused on Pb, Cu and Zn in single, binary and ternary system. The real water sample used were collected from Sungai Balok, Kuantan Pahang, and also from an electroplating plant in Selangor, while synthetic wastewater was made from $Pb(NO_3)_2$. Figure 1.1 summarized the entire task and scope of this research.

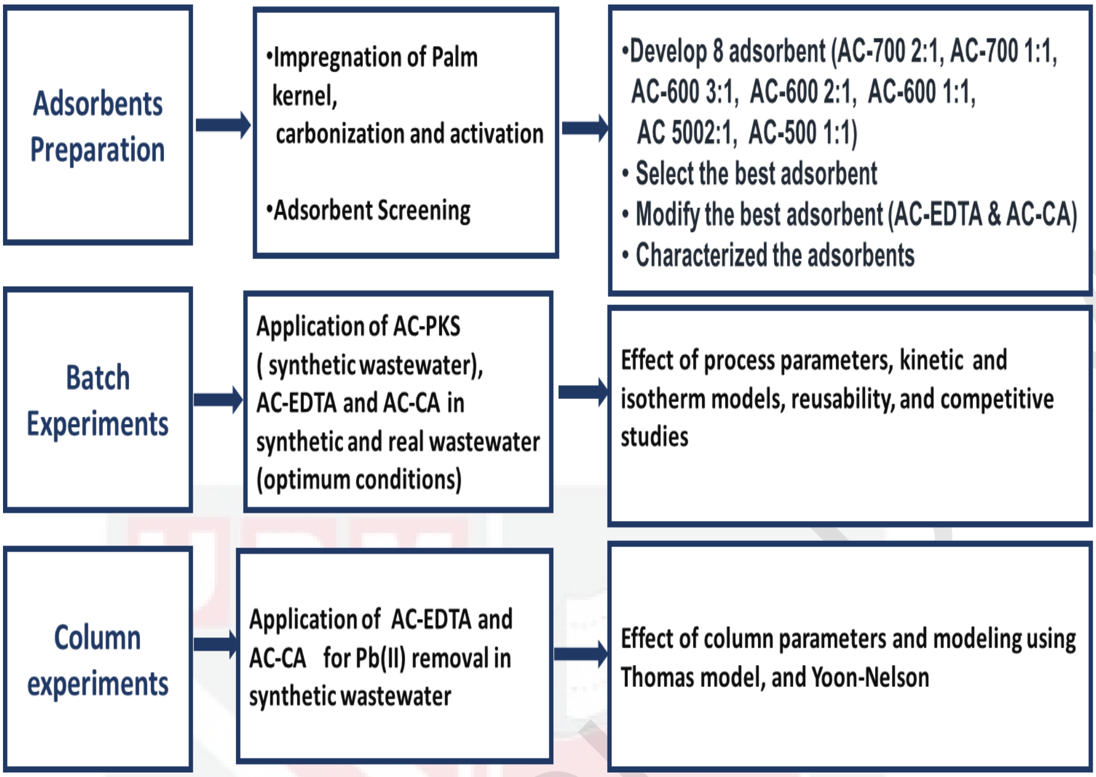

Figure 1.1 : The main scope of this Research

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