UNIVERSITI PUTRA MALAYSIA

***UTILIZATION OF BIOCHARS FROM WASTES OF THERMOCHEMICAL PROCESSES AS ADSORBENTS***

DALIA KHALID MAHMOUD

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# UTILIZATION OF BIOCHARS FROM WASTES OF THERMOCHEMICAL PROCESSES AS ADSORBENTS

By

**DALIA KHALID MAHMOUD**

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

**May 2016**

# DEDICATION

To those people who give happiness and hope through the darkness of the life

Especially

To

Beloved: Mother & Father

Brothers: Walid & Mahmoud

Sisters in law: Alyaa & Amani

Gorgeous nieces: Shahad, Tyba & Farah

Awesome nephews: Mustafa & Taha

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

**UTILIZATION OF BIOCHARS FROM WASTES OF THERMOCHEMICAL PROCESSES AS ADSORBENTS**

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**May 2016**

**Chairman : Associate Professor Mohamad Amran Bin Mohd Salleh**
**Faculty : Engineering**

Thermochemical conversion technologies generate large amounts of biochars as byproducts. Biochars are low value by-products that have received lesser attention and there is no valuable utilization method. On other hand, the removal of Methylene Blue (MB) dye from textile wastewater is environmentally important since MB could cause harmful effects. Kenaf core fibre char (KFC), palm oil empty fruit bunch char (EFBC) and *Leucaena leucocephala* wood char (LC), the by-products from gasification and pyrolysis processes at high temperature were evaluated as potential adsorbents in textile wastewater treatment in particular adsorption of MB. The adsorption of MB onto KFC, EFBC, LC and activated kenaf core fibre char (H-KFC), was studied in batch process. The adsorption was performed under different operating conditions including adsorbent dose, initial dye concentration and contact time onto KFC, H-KFC, EFBC and LC of 750 µm particle size. Further conditions were studied onto H-KFC including particle size, temperature and solution pH. Adsorption isotherm and kinetics of MB adsorption onto KFC, LC, EFBC (750 µm) and H-KFC (different particle size, solution pH and temperature) were also studied. The adsorption isotherm data for all adsorbents were considered to fit well to the Langmuir model. The Langmuir adsorption capacity of MB onto adsorbents (750 µm, at 30°C and solution pH=6.5) followed the trend of H-KFC>KFC>LC>EFBC. The maximum adsorption capacity of H-KFC was found increases with particle size, temperature and pH of solution (2.5- 8.5) increasing. The pseudo-second-order kinetic model described the adsorption experimental data of all adsorbents well. An analytical solution of the Langmuir kinetic model was obtained through derivation. It was found that there was a good agreement between predicted values of model and experimental data for all adsorbents. From the thermodynamic study of MB adsorption onto H-KFC(750 µm), it was found that the adsorption confirm the nature of chemisorption process, endothermic, increase the randomness of adsorbed species and more spontaneous nature at high temperature. The desorption study revealed that H-KFC(750 µm) could not be regenerated. The design of a batch adsorption study showed that H-KFC(750 µm) is more suitable to be used as adsorbent from an economical point of view. Analysis of Variance (ANOVA) results showed a

significant difference between the amount of dye adsorbed at different adsorbents at all concentrations. There were no significant differences between the data at different pH and temperature at all concentrations while the effect of particle size seems obvious at the initial dye concentration of 50 mg/L. ANOVA confirmed the result of batch adsorption design where the H-KFC(750 μm) was found to be more economically suitable than the other adsorbents. A Logical study for the comparison of chemical and physical properties of adsorbents showed that the better properties trend was followed H-KFC(750 μm)>KFC(750 μm) >EFBC(750 μm)>LC(750 μm).

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

**UTILISASI BIOCHAR DARIPADA SISA PROSES TERMOKIMIA SEBAGAI PENJERAP**

Oleh

**DALIA KHALID MAHMOUD**

**Mei 2016**

**Pengerusi : Profesor Madya Mohamad Amran Bin Mohd Salleh**
**Fakulti : Kejuruteraan**

Teknologi penukaran termokimia menjana begitu banyak biochar sebagai produk akhir. Biochar merupakan produk akhir bernilai rendah yang telah menerima perhatian yang agak kurang dan tidak terdapat kaedah utilisasi yang berkesan. Namun, penyingkiran pencelup Biru Metilen (MB) daripada sisa air tekstil adalah penting dari segi persekitaran disebabkan MB mungkin membawa kesan yang memudaratkan. Char Gentian teras Kenaf (KFC), Char tandan Kosong Kelapa Sawit (EFBC) dan Char kayu Petai Belalang/ Leucaena Leucocephala wood Char (LC), produk akhir dari proses gasifikasi dan pirolisis pada suhu yang tinggi telah dinilai sebagai penjerap berpotensi dalam rawatan sisa air tekstil terutamanya dalam penyerapan MB. Penyerapan MB ke atas KFC, EFBC, LC dan Char Gentian teras Kenaf yang diaktifkan (H-KFC) telah dikaji dalam proses kelompok. Penyerapan telah dijalankan di bawah kondisi operasi yang berbeza termasauk dos penjerap, konsentrasi pencelup awal dan masa kontak ke atas KFC, H-KFC, EFBC dan LC, 750 µm saiz partikel. Kondisi seterusnya telah dikaji ke atas H-KFC termasuk saiz partikel, suhu dan larutan pH. Penyerapan Isoterm dan kinetic MB ke atas KFC, LC, EFBC (750 µm) dan H-KFC ( saiz partikel yang berbeza, larutan pH dan suhu) juga telah dikaji. Penyerapan data isoterm bagi semua penjerap telah dikira sebagai amat sesuai dengan model Langmuir. Kapasiti penyerapan Lanhmuir MB ke atas penjerap (750 µm, pada 30°C dan larutan pH=6.5) mengikut trend H-KFC>KFC>LC>EFBC. Maksimum kapasiti penyerapan H-KFC didapati telah meningkat dengan peningkatan saiz partikel, suhu dan pH pada larutan (2.5-8.5). Model kinetik tertib pseudo kedua menerangkan data eksperimental penyerapan bagi semua penjerap adalah jelas. Larutan analitikal bagi model kinetik Langmuir telah diperoleh melalui derivasi. Kajian ini mendapati bahawa terdapat persetujuan yang baik antara jangkaan nilai model dengan data eksperimental bagi semua penjerap. Daripada kajian termodinamik mengenai penyerapan MB ke atas H-KFC(750 µm), didapati bahawa penyerapan menyamai sifat proses pengkimiaerapan, endoterma, meningkatkan kerawakan spesis terserap dan sifat spontan yang lebih banyak pada suhu tinggi. Kajian penyaherapan menunjukkan bahawa H-KFC(750 µm) tidak dapat dijanakan. Reka bentuk kajian penyerapan kelompok menunjukkan bahawa H- KFC

(750μm) adalah lebih sesuai digunakan sebagai penjerap daripada sudut pandangan ekonomi. Analisis varians (ANOVA) menunjukkan bahawa terdapat perbezaan yang sigfnifikan antara bilangan pencelup yang dicelup pada penjerap yang berbeza paad semua konsentrasi. Tidak terdapat perbezaan yang signifikan antara data pada pH yang berbeza dan suhu pada semua konsentrasi manakala kesan saiz partikel adalah ketara pada awal konsentrasi pencelup 50mg/L. ANOVA mengesahkan dapatan mengenai reka bentuk penyerapan kelompok, H-KFC(750 μm) didapati lebih sesuai dari segi ekonomi daripada penjerap lain. Kajian logikal bagi perbandingan sifat kimia dan fizikal penjerap menunjukkan bahawa sifat trend adalah lebih baik seperti berikut H-KFC(750μm)>KFC(750μm)>EFBC(750 μm)>LC(750 μm).

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I certify that a Thesis Examination Committee has met on 27 May 2016 to conduct the final examination of Dalia Khalid Mahmoud on her thesis entitled "Utilization of Biochars from Wastes of Thermochemical Processes as Adsorbents" 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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This thesis was submitted to the Senate of the 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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**TABLE OF CONTENTS**

# LIST OF TABLES

## LIST OF FIGURES

## LIST OF ABBREVIATIONS AND ACRONYMS

| | |
|---|---|
| MB | Methylene blue |
| KFC | Kenaf core fibre char |
| H-KFC | Activated kenaf core fibre char |
| EFBC | Palm oil empty fruit bunch char |
| LC | *Leucaena leucocephala* wood char |
| FTIR | Fourier Transforms Infrared Spectroscopy |
| TGA | Thermal Gravimetric Analysis |
| BET | Brunauer-Emmet-Teller |
| CHNS/O | Ultimate analysis |
| SEM | Scanning Electron Microscopy |
| ANOVA | Analysis of Variance |
| NATIP | National Timber Industry Policy |
| FFB | Palm oil Fresh Fruit Bunch |
| CPO | Edible crude palm oil |
| EFB | Empty fruit bunches |
| PK | Palm kernel |
| POME | Palm oil mill effluent |
| CDM | Clean Development Mechanism |
| QRëC | Quality Reagent Chemical |
| UITM | MARA University of Technology |
| BJH | Barret-Joyner-Halena |
| SAE | Sum of Absolute Error |
| AE | Absolute Error |

| | |
|---|---|
| V. BET | Value of BET Surface Area |
| D.C=O | Detect of C=O Functional Group |
| V. O. | Value of Oxygen Content |
| V. H. | Value of Hydrogen Content |
| V. N. S. | Value of Nitrogen and Sulfur Content |

# CHAPTER ONE

# INTRODUCTION

## 1.1 Biochar from Agricultural Sources

New and renewable fuels are the major alternatives to conventional fossil fuels. Biomass in the form of agricultural residues is considered the most popular among new renewable energy sources due to its wide potential and numerous uses. Agricultural wastes such as rice husk, fruit peels and corncob are low cost resources and are available as sources of biomass energy and precursory materials of biochars. Biochar, derived from biomass, is increasingly recognized as an environment friendly sorbent to abate organic pollutants (Chen & Chen, 2009).

There are two basic categories for conversion technologies, namely thermochemical and biochemical processes, where combustion, carbonization/pyrolysis, gasification and liquefaction are considered as thermochemical conversion processes.

Pyrolysis of biomass consists of heating the material in the absence of air to produce solid, liquid or gaseous products (Özçimen & Karaosmanoğlu, 2004) while gasification is a form of pyrolysis that can be carried out at high temperatures to optimize gas production (Demirbas, 2005). High temperature carbonized biochar is stable solid, without toxicity and difficult to decompose in nature (Lin & Hwang, 2009).

The application of thermochemical processes will produce three products, which are liquid products (wood tar, tar, oil, pyrolytic oil), gas products (wood gas, pyrolytic gas) and solid products (char, charcoal) (Klass, 1998; Özçimen & Ersoy-Meriçboyu, 2008).

Kenaf core fibre, palm oil empty fruit bunch and *Leucaena leucocephala* wood are great interest as popular main sources for bio-fuel in Malaysia. Kenaf Natural Fiber Industries Sdn. Bhd. (Matrade Wilayah Timur, 2011) processes kenaf fibers and cores and uses the produced core as feeds in pyrolysis at 1000 °C. Future Nrg Company has installed pilot scale gasifier units using EFB as fuel to generate electricity. Malaysian Biotechnology Corp (Biotechcrop) has started research on the *Leucaena* for biofuel production; where the idea is to use *Leucaena,* with wood as an energy source to produce steam and electricity to run the factories (Heng, 2013).

The conversion of biomass into liquid and gas fuels by gasification and pyrolysis is promising technology. It is anticipated that a large amount of chars will be available. Therefore, any use with a value in excess of its fuel value is welcome (Mohan *et al*., 2007b; Boateng *et al*., 1992).

Carbonaceous materials used as adsorbents are well developed internal porosity and there is possibility of modifying their textural and surface properties by simple treatments (Pérez-Mendoza *et al*., 2006). Therefore, char utilization for adsorption is a bonus advantage and important due to the need for a low cost source of carbon for different small scale industries in waste water treatments (Lin & Hwang, 2009).

In this work, biochars derived from kenaf core fibre, oil palm empty fruit bunch and *Leucaena leucocephala* (petai belalang) wood were used as adsorbents for textile wastewater treatments in particular adsorption of methylene blue (MB).

## 1.2 Textile Industry and Colored Effluents

In the present century, increasing population, consumption of natural habitat and industrialization has contributed to economic growth but at the same time has led to negative environmental impact and an increase in environmental problems such as air and water pollutions. This has caused catastrophes that destroy the ecologic system and therefore clean resources have exhausted rapidly (Erdoğan *et al*., 2005; Tham *et al*., 2011). Industries have a large potential to cause lake, streams and river pollution. The nature of pollution varies from industry to industry and also from plant to plant (Mohan *et al*., 2008), where industries like textile, chemical, refineries, plastic and food processing plants produce wastewaters accompanying with a palpable content of organics such as phenolic compounds with strong color. Since the typical dyeing process consists of desizing, scouring, bleaching, dyeing, finishing and drying operations (Mohan *et al*., 2007a), colored effluents produced from textile industries are considered as one of the main problems regarding textile wastewaters (Hameed *et al*., 2007a).

Malaysia has a long history of producing textiles, where fabrication colored textiles grow continuously day by day according to the growth of the population and export demands, providing a new dimension to Malaysian economic. Textile industries use dyes or pigments to color their final products (Hameed *et al*., 2007b); therefore, the main pollution in textile wastewater comes from the dyeing and finishing processes.

Wastewater generated from textile processing industries contains high amount of dissolved solids, suspended solids, un-reacted dyestuffs (color) and other auxiliary chemicals (Rajkumar *et al*., 2007). These chemicals and dyestuffs make the removal of dyes a great concern from an environmental point of view.

Dyes represent a special problem in wastewater since they are intensely colored. Most dyes are difficult to decolorize due to their synthetic origin and complex structure. The discharge of dye wastewater in the environment has serious environmental effects and furthermore, it is aesthetically unfavorable (Demirbas *et al*., 2008). Dyes have different compounds with unknown environmental behavior where dyes of several varieties are used in textile industry such as acidic dyes, basic dyes, reactive dyes, direct dyes, disperse dyes, solvent dyes, sulfur dyes and vat dyes. Basic dyes may impart toxicity to aquatic life, thus causing harmful effects such as allergic dermatitis, skin irritation, cancer and mutations (Karagozoglu *et al*., 2007).

## 1.3 Wastewater Treatment

Wastewater treatment is one of the major problems of everyday life. Water pollution may cause severe problems, and in order to reduce these problems, many countries and environmental protection authorities are continually restricting the use of chemicals and emphasising on the discharge limits. Therefore, wastewaters must be treated carefully before being discharged as effluents especially those containing toxic materials such as dyestuff, organic pollutants and metal ions (Chan *et al*., 2012a).

The public demand for waste discharge of free of color receiving waters and the strict color standards have made the decolorization as a top priority of variety of industrial wastes. Unfortunately, the decolorization of colored wastes is considered as a difficult and challenging task because of the complicated color-causing compounds (Hao *et al*., 2000). One of the main problems faced the textile industry is the removal of color, where the removal of synthetic dyes represents a great concern from an environmental point of view (Hameed *et al*., 2007b). This target is desired because the legislation requirements have become stricter and the water quality used for recycling is taken into account (Ferrero, 2007). As synthetic dyes are chemically stable, traditional methods have often proven to be ineffective in removing these species from the environment.

A number of different technologies have been applied for the treatment of dye effluents, and these include coagulation, flocculation, advanced oxidation processes and adsorption. Such methods need to be economically viable and environmentally friendly while, at the same time, the final concentrations of contaminants in the treated effluents must comply with the standards imposed by governmental regulatory agencies (Vieira *et al*., 2012).

## 1.4 Adsorption Technique

Adsorption has been extensively used in industrial processes for either separation or purification since it holds promise in the treatment of wastewater, as it is easy to handle, inexpensive, simply designed, and provides sludge-free cleaning operations (Dizge *et al*., 2008; Gupta *et al*., 2000).

Adsorption using activated carbon is considered very effective in the reduction of color, adsorbable organic halides and the non-biodegradable fraction of the pulp bleaching wastewater (Shawwa *et al*., 2001). Furthermore, adsorption using activated carbon has been found superior compared to other techniques in water re use methodology because of its simplicity of design and its ability for adsorbing wide range of different types of adsorbates efficiently (Ahmad *et al*., 2007). Although the adsorption with activated carbon appears to be the best prospect for elimination of dyes, this adsorbent is expensive and difficult to regenerate after use (Dolphen *et al*., 2007). Therefore, it is better to find cheap, commercially available materials as its possible replacements (Gupta *et al*., 2003). Studies have reported that biochar can be a suitable environmental substitute for activated carbon in the organic contaminants treatment (Inyang & Dickenson, 2015).

## 1.5 Adsorbate

For this research, Methylene blue (MB) was chosen as a model as many previous researchers have studied the adsorption of methylene blue dye as it is a basic dye that is widely used in the textile industry. The cationic dye MB is the most commonly used material for dyeing cotton, wood and silk (Hameed *et al*., 2007c). Although MB dye is not regarded as acutely toxic, it has various harmful effects (Özer *et al*., 2007).

The adsorption of MB has been used for a long time for the evaluation of the absorption properties of activated carbon, particularly liquid phase carbons (Ghasemi & Asadpour, 2007). A number of studies have been published on the removal of MB from aqueous solution by different types of adsorbents such as adsorption of MB using indian rose wood (Garg *et al*., 2004), coir pith carbon (Kavitha & Namasivayam, 2007), biomass of baker's yeast (Yu *et al*., 2009), perlite (Doğan *et al*., 2004), jute processing waste (Banerjee & Dastidar, 2005), natural zeolite (Han *et al*., 2007), fuel oil fly ash (Andini *et al*., 2008), castor seed shell (oladoja *et al*., 2008) and steam activated bituminous coal (El Qada *et al*., 2006).

## 1.6 Problem Statement

From the point of view of reducing the sources of global warming and sustainable resource management, biomass is attracting attention as a renewable energy resource to substitute the current fossil fuel resources (Spokas *et al*., 2009). Using biomass as a fuel offers certain advantages, in terms of energy, environment, society and economy (Kaushal *et al*., 2010). The used of biomass as an energy resource can be efficiently achieved through the thermochemical conversion technology such as gasification, pyrolysis and combustion (Mohammed *et al*., 2011a).

Thermochemical conversion technologies generate a large amount of biochar as by-products which are considered as lower value by-products. In biomass conversion technologies, most attention has been focused on the liquid product (bio-oil). Biochar, as a low value by product has received lesser attention, and there is no valuable utilization method for it except direct combustion for heat production (Liu & Zhang, 2009). Therefore, char produced at high temperature (1000 °C) through thermal processes (high gas production) has the potential to be used for environmental applications such as adsorbents in treating textile wastewaters while most studies simply produce activated carbon from different biomass sources to be used as adsorbents but did not focus on byproducts of industrial wastes.

The environmental issues related to the color presence in effluents are a continuing problem for dyestuff manufacturer, dyers, finishers and water companies due to the increasing of stringent color consent standards enforced by regulatory bodies to reduce the quality of color in effluents and water sources. Most dyes that are used for coloring materials are toxic to aquatic organisms and prevent re-oxygenation in receiving waters by cutting off sunlight penetration (Senthilkumaar *et al*., 2006a).

Methylene blue dye (MB) is basic dye intensely used in textile industry. Acute exposure to MB may cause some harmful effects, such as increase in heart rate, vomiting, shock, jaundice, cyanosis, quadriplegia and tissue necrosis in human (Vadivelan & Kumar, 2005). Therefore, removal of MB dye from process effluent becomes environmentally important (Theydan & Ahmed, 2012).

Adsorption technique has been proven to be an excellent method for dyes removal and commercially activated carbon is popular and effective dye sorbent. The high cost of activated carbon and the problems related with its regeneration, has prompted the search for alternate adsorbents (Amin, 2008). Thus, attention has been focused on the development of low cost adsorbents that can be applied in wastewater treatment. Demand for activated carbon keeps increasing thus, the source of materials must be expanded to a variety of biomass sources including wastes to meet the demand.

Kenaf core fibre, oil palm empty fruit bunch and *Leucaena leucocephala* are currently being used as feeds for pyrolysis and gasification processes that generate chars that can be used as adsorbents for MB dye removal from wastewaters.

As there is a need to understand the favorable adsorbent from practical and economical point of views and also the effect of factors on adsorption process, statistical analysis is used to achieve these purposes. A familiar method to evaluate adsorbents and the effect of factors on adsorption process is by measuring the adsorption capacity at equilibrium using isotherm study, neglecting the deep comparison at all initial dye concentrations with respect to time.

Physical and chemical characterizations of adsorbents can be used to determine the better adsorbent. As, there are not enough understanding on the relationship between chemical and physical properties of adsorbents and their adsorption capacities, there is need for theoretical expression for the physical and chemical characterizations of adsorbents for comparison purposes. This has created a need for function formulas that can make an in-depth comparison between adsorbents of each property.

## 1.7 Research Objectives

i. To evaluate biochars (KFC, H-KFC, LC and EFBC) generated from the by-products of gasification and pyrolysis processes, as potential adsorbents of methylene blue (MB).
ii. To study the adsorption of MB using biochar-based adsorbents in batch system under different operating conditions and to study the adsorption isotherm, kinetic on all adsorbents and the thermodynamic on H-KFC (750 μm).
iii. To evaluate the adsorbents by comparing chemical and physical properties of adsorbents using Logical Functions.

## 1.8 Thesis Layout

The thesis consists of five chapters, where each chapter represents an important section for general construction of the thesis.

**Chapter 1** provides an introduction to biochar from agricultural sources, textile industry and colored effluents, wastewater treatment methods, adsorption technique, adsorbate (methylene blue (MB)). It is also states the problem statement and research objectives.

**Chapter 2** presents the literature review which includes brief information on biomass as renewable energy sources, adsorbents, production of biochar and biochar as adsorbent. The literature review also presents general information on dye effluents from textile industry, dyes classification, dyes health impact, basic (cationic) dyes, methylene blue (MB) dye, the methods of dyes removal, adsorption and its types, adsorption by activated carbon and adsorption by biochar. In addition, this chapter provides brief information on activation methods of adsorbents and a review of factors that affects adsorption, isotherms models, kinetics models and thermodynamic parameters determination, desorption process, batch adsorption design and a summary.

**Chapter 3** presents the experiment conducted part and the technical details involved. It gives a general description of batch adsorption experiment, adsorbents preparation, equipments used in batch experiment, materials, and a description of the analysis techniques performed for characterization of adsorbents. This chapter also explains the experimental procedure and describes the factors that affect the adsorption process. The schematic of the parameter estimation procedure of Langmuir kinetic model and the procedure of comparison between the chemical and physical properties of adsorbents using Logical study are also described.

**Chapter 4** displays the experimental results and presents the discussion. This chapter reveals the results of characterization of adsorbents, and discusses the comparison of adsorbents and the effects of different factors on batch system adsorption. It is also gives the results of the equilibrium, kinetics, the error analysis test, mechanism, thermodynamic, desorption and design of batch sorption studies. The derivation of Langmuir kinetic model and its application to the experimental data are also presented in this chapter. Analysis of Variance ANOVA used to validate the results of amount of dye adsorbed onto adsorbents, amounts of dye adsorbed onto H-KFC (750 μm) of different particle sizes, amount of dye adsorbed onto H-KFC (750 μm) at different pH solution, amount of dye adsorbed onto H-KFC (750 μm) at different temperatures and amount of adsorbents required to remove 90% of MB solution at different initial dye concentration are also included. The multiple regression analysis to study the relationship between effective parameters and maximum adsorption capacity and the logical study for comparison of chemical and physical properties of adsorbents are also included in this chapter.

**Chapter 5** presents the overall conclusions based on the findings obtained in the Results and Discussion **(Chapter 4)**. Recommendations for future studies are also given in this chapter.

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