

**PREPARATION, CHARACTERISATION AND EVALUATION  
OF FIVE AGRICULTURE BY-PRODUCTS' ACTIVATED  
CARBONS USING HEAVY METALS AND DYE**

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## AUTHOR DECLARATION

This thesis is made of my original work, and no part of this thesis has been published previously by another person. I hereby declare that the work in this thesis is done by me under the supervision of Associate Prof. ChM. Dr. Mohd Sukri Hassan (Main Supervisor) and Dr. Syaza Azhari (Co-Supervisor), Faculty of Science & Technology (FST) of the Universiti Sains Islam Malaysia, Bandar Baru Nilai, Negeri Sembilan, Malaysia.

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## ABSTRAK

Akses kepada air minuman yang selamat adalah amat dititikberatkan di negara-negara membangun. Air permukaan adalah sumber air yang biasa digunakan. Keracunan logam berat telah menjadi ancaman besar kepada alam sekitar akibat air sisa industri. Plumbum, nikel, tembaga, dan kadmium adalah logam berat yang paling toksik dalam air tercemar. Ia mempunyai kesan berbahaya seperti barah paru-paru, kerosakan buah pinggang, gangguan mental dan saraf. Kaedah konvensional untuk menyingkirkan logam toksik dan pewarna dari air sisa seperti penggunaan karbon teraktif komersial sangat mahal untuk negara-negara dunia ketiga. Objektif utama kajian ini adalah untuk menilai kemampuan karbon teraktif yang dihasilkan daripada hasil buangan pertanian (sekam padi, sabut kelapa, tongkol jagung, kulit kayu neem dan kulit kayu *Moringa oleifera*) untuk menyingkirkan logam berat (tembaga (II), kadmium (II), plumbum (II) dan nikel (II)), pewarna (metilena biru). Karbon teraktif dihasilkan menggunakan kaedah berkarbonasi dalam keadaan lengai dengan pengaktifan kimia dalam nisbah berbeza pada suhu yang berlainan. Proses penyerapan dilakukan menggunakan pelarut asid (asid hidroklorik dan asid sulfurik) (0.1N) dan alkali (natrium karbonat). Asid hidroklorik adalah pelarut terbaik untuk menyerap bahan cemar yang dimuat dari penjerap. Plumbum disingkirkan sepenuhnya dari larutan tercemar menggunakan lima jenis karbon teraktif. Dalam proses penjerapan kumpulan, tembaga (97.00 %, 97.52 %, 64.49 %, 36.60 %, 98.24 %), kadmium (56.72 %, 54.16 %, 69.40 %, 41.48 %, 56.00 %) & nikel (92.22 %, 81.79 %, 83.35 %, 88.10 %, 82.30 %) disingkirkan dari larutan tercemar oleh karbon teraktif sekam padi, sabut kelapa, tongkol jagung, kulit kayu neem dan *Moringa oleifera*, masing-masing pada pH 6 menggunakan dos yang lebih rendah (0.025 g/100 mL) pada suhu bilik ( $25 \pm 1$  °C). Kuantiti maksimum pengambilan plumbum pada masa keseimbangan adalah (46.51, 43.08, 47.16, 51.57, 53.73 mg/g) menggunakan lima karbon teraktif. Kapasiti penjerapan tertinggi penjerap adalah kulit (sekam padi, sabut kelapa, tongkol jagung, kulit kayu neem dan kulit kayu *Moringa oleifera*). Karbon teraktif kulit kayu *Moringa oleifera* dihasilkan dengan suhu karbonisasi pada 700 °C dan nisbah impregnasi  $ZnCl_2$  dengan  $H_2SO_4$  hingga arang pada nisbah 5:1. Daripada penjerap yang dihasilkan dicirikan dengan menggunakan Mikroskop Pemindai Electron (SEM), Brunauer-Emmett-Teller (BET), Fourier-Transform Inframerah (FTIR) dan analisis proximate. *Moringa oleifera* menunjukkan hasil penjerapan terbaik kerana luas permukaan yang baiklah (439.23 m<sup>2</sup>/g) dan isipadu pori (0.189 cm<sup>3</sup>/g). Isoterm Freundlich Berpadanan baik dengan pekali regresi ( $R^2 \approx 1$ ) untuk data eksperimen daripada model Langmuir dan model D-R. Model tertib kedua juga berpadanan rapat ( $R^2 \approx 1$ ) dengan data eksperimen. Dari data FTIR, sebahagian besar karbon teraktif mengandungi kumpulan hidroksil, karboksil dan metoksi untuk menyerap logam berat dan pewarna. Eksperimen lebih lanjut, menunjukkan bahawa metilena biru dikeluarkan dari air tercemar dengan menggunakan semua karbon teraktif. Kuantiti maksimum pengambilan metilena biru ( $q_{max}$ ) ialah 108.94 mg/g, 107.71 mg/g, 107.71 mg/g, 109.34 mg/g, dan 108.12 mg/g menggunakan sekam padi, sabut kelapa, tongkol jagung, kulit kayu neem dan *Moringa oleifera* masing-masing.

## ABSTRACT

Access to safe drinking water is the most concern in developing countries. Surface water is a common source of usable water. Heavy metal toxicity has become a great threat to the environment due to industrial wastewater. Lead, nickel, copper, and cadmium are the most toxic heavy metals in polluted water. It has harmful effects such as lung cancer, kidney damage, mental and nerve disorder. Conventional methods for removing toxic metals and dyes from wastewater such as the use of commercial activated carbon are very expensive for third-world countries. The main objective of this study was to evaluate the ability of activated carbon produced from agricultural waste-products (rice husk, coconut coir, corn cobs, neem bark and *Moringa oleifera* bark) to remove heavy metals (copper (II), cadmium (II), lead (II) and nickel (II)), and dye (methylene blue). The activated carbons were produced using carbonized method in an inert state with chemical activation in varieties ratio at different temperatures. Desorption process were conducted using 0.1M acid (hydrochloric acid and sulphuric acid) and basic (sodium carbonate) solutions. Hydrochloric acid was the best solvent to desorb loaded contaminants from the adsorbents. The lead was absolutely removed from spiked aqueous solution in 5 mg/L using five types of activated carbon. In the batch adsorption process, copper (97.00 %, 97.52 %, 64.49 %, 36.60 %, 98.24 %), cadmium (56.72 %, 54.16 %, 69.40 %, 41.48 % 56.00 %) and nickel (92.22 %, 81.79 %, 83.35 %, 88.10 %, 82.30 %) were removed from spiked aqueous solution in 5 mg/L by activated carbon of rice husk, coconut coir, corn cobs, neem bark, and *Moringa oleifera* bark, respectively at pH 6 using lower dosage (0.025 g/100 mL) at room temperature ( $25 \pm 1$  °C). The maximum quantity of lead uptake at equilibrium time was (46.51, 43.08, 47.16, 51.57, 53.73 mg/g) using five activated carbons of rice husk, coconut coir, corn cobs, neem bark, *Moringa oleifera* bark, respectively. The highest adsorption capacity of adsorbents was *Moringa oleifera* bark. Activated carbon of *Moringa oleifera* bark was produced with carbonization temperature at 700 °C and impregnation ratio of ZnCl<sub>2</sub> with H<sub>2</sub>SO<sub>4</sub> to charcoal at ratio 5:1. The produced well adsorbents were characterized using Scanning Electron Microscopy (SEM), Brunauer-Emmett-Teller (BET), Fourier-Transform Infrared (FTIR) and proximate analysis. *Moringa oleifera* bark showed the best adsorption results due to well surface area (439.23 m<sup>2</sup>/g) and pore volume (0.189 cm<sup>3</sup>/g). Freundlich isotherm was well fitted with coefficient regression ( $R^2 \approx 1$ ) for experimental data than the Langmuir and D-R model. The second order model also was closely fitted ( $R^2 \approx 1$ ) with experimental data. From FTIR data, most of the activated carbon contained hydroxyl, carboxyl and methoxy groups to adsorb heavy metals and dye. A further experiment showed that methylene blue was greatly removed from spiked water using all activated carbons. The maximum quantity of methylene blue ( $q_{max}$ ) uptake were 108.94 mg/g, 107.71 mg/g, 107.71 mg/g, 109.34 mg/g, and 108.12 mg/g using rice husk, coconut coir, corn cobs, neem bark and *Moringa oleifera* bark, respectively.

## الملخص

الحصول على مياه الشرب المأمونة هو الشاغل الأكبر في البلدان النامية. المياه السطحية هي مصدر شائع للمياه الصالحة للاستخدام. أصبحت سمية المعادن الثقيلة تشكل تهديداً كبيراً على البيئة بسبب مياه الصرف الصناعي. يعتبر الرصاص والنيكل والنحاس والكاديوم من أكثر المعادن الثقيلة سمية في المياه الملوثة. وله آثار ضارة مثل سرطان الرئة وتلف الكلى والاضطراب النفسي والعصبي. تعتبر الطرق التقليدية لإزالة المعادن والأصبغ السامة من مياه الصرف الصحي ، مثل استخدام الكربون المنشط التجاري ، مكلفة للغاية بالنسبة لبلدان العالم الثالث. كان الهدف الرئيسي من هذه الدراسة هو تقييم قدرة الكربون المنشط المنتج من منتجات النفايات الزراعية (قشر الأرز ، جوز الهند ، كوز الذرة ، لحاء النيم ولحاء المورينجا أوليفيرا) لإزالة المعادن الثقيلة (النحاس (II) ، الكاديوم (II) ، الرصاص (II) والنيكل (II)) ، صبغة (الميثيلين الأزرق). تم إنتاج الكربون المنشط باستخدام طريقة متفحمة في حالة خاملة مع تنشيط كيميائي في نسبة الأصناف عند درجات حرارة مختلفة. أجريت عملية الامتزاز باستخدام محلول 0.1 مولار حامض (حمض الهيدروكلوريك وحمض الكبريتيك) ومحاليل قاعدية (كربونات الصوديوم). كان حمض الهيدروكلوريك أفضل مذيب لامتصاص الملوثات المحملة من المواد الماصة. تمت إزالة الرصاص تماماً من المحلول المائي المسخن في 5 مجم / لتر باستخدام خمسة أنواع من الكربون المنشط. في عملية الامتزاز الدفعي ، النحاس (97.00% ، 97.52% ، 64.49% ، 36.60% ، 98.24% ، الكاديوم (56.72% ، 54.16% ، 69.41% ، 48.41% ، 56.00%) والنيكل (92.22% ، 81.79% ، 83.35% ، 88.10% ، 82.30%) من المحلول المائي المسخن في 5 مجم / لتر بواسطة الكربون المنشط من قشر الأرز ، جوز الهند ، كوز الذرة ، لحاء النيم ، ولحاء المورينجا أوليفيرا ، على التوالي عند درجة الحموضة 6 باستخدام جرعة أقل (0.025 جم) / 100 مل عند درجة حرارة الغرفة (25 ± 1 درجة مئوية). كانت الكمية القصوى لامتصاص الرصاص في وقت التوازن (46.51 ، 43.08 ، 47.16 ، 51.57 ، 53.73 ، مجم / جم) باستخدام خمسة ذرات من قشر الأرز ، جوز الهند ، كوز الذرة ، لحاء النيم ، المورينجا أوليفيرا ، على التوالي. كانت أعلى قدرة امتصاص للمواد الماصة هي لحاء المورينجا أوليفيرا. تم إنتاج الكربون المنشط لحاء المورينجا أوليفيرا بدرجة حرارة كربنة عند 700 درجة مئوية ونسبة تشريب من ZnCl<sub>2</sub> مع H<sub>2</sub>SO<sub>4</sub> إلى الفحم بنسبة 5:1. تم تمييز المميزات البئر المنتجة باستخدام الفحص المجهر الإلكتروني (SEM) ، Brunauer-Emmett-Teller (BET) ، Fourier-Transform

- Infrared (FTIR) التحليل التقريبي. أظهر لحاء مورينجا أوليفيرا أفضل نتائج امتزاز بسبب مساحة السطح العالية (439.23 م<sup>2</sup> / جم) وحجم المسام (0.189 سم مكعب / جم). تم تجهيز متساوي درجة حرارة Freundlich جيداً مع معامل الانحدار ( $R^2 \approx 1$ ) للبيانات التجريبية مقارنة بنموذج Langmuir و DR. كما تم ربط نموذج الدرجة الثانية ( $R^2 \approx 1$ ) مع البيانات التجريبية. من بيانات FTIR ، احتوى معظم الكربون المنشط على مجموعات الهيدروكسيل والكربوكسيل والميثوكسي لامتصاص المعادن الثقيلة والصبغة. أظهرت تجربة أخرى أن الميثيلين الأزرق كان تمت إزالته بشكل كبير من الماء المسنن باستخدام جميع الكربون النشط. بلغت الكمية القصوى لامتصاص الميثيلين الأزرق (qmax) 108.94 مجم / جم ، و 107.71 مجم / جم ، و 107.71 مجم / جم ، و 109.34 مجم / جم ، و 108.12 مجم / جم باستخدام قشر الأرز ، جوز الهند ، و أكواز الذرة ، و لحاء النيم ولحاء المورينجا أوليفيرا ، على التوالي.

# TABLE OF CONTENTS

<u>ITEM</u>	<u>PAGE</u>
AUTHOR DECLARATION	ii
AUTHOR DECLARATION AND COPYRIGHT	iii
ACKNOWLEDGMENT	iv
ABSTRAK	v
ABSTRACT	vi
الملخص	vii
TABLE OF CONTENTS	ix
LIST OF TABLES	xiv
LIST OF FIGURES	xvi
LIST OF ABBREVIATION	xx
CHAPTER 1	1
INTRODUCTION	1
1.1 Background of Study	1
1.2 Problem Statement	3
1.3 Significance of Study	5
1.4 Objectives of Research	6
1.5 Scope of Research	6
CHAPTER 2	7
LITERATURE REVIEW	7
2.1 Introduction	7
2.2 Sources of Wastewater	7
2.3 Sources and Effects of Heavy Metals to Human body & Environments	9
2.4 Environmental Pollution and Health Problems	12
2.5 Methods of Wastewater Treatment	12
2.5.1 Adsorption	13
2.5.1.1 Physical adsorption	14
2.5.1.2 Chemisorption	14
2.6 Adsorption of Activated Carbon	14

2.7 Adsorbents from Agricultural by-Products	16
2.7.1 Rice Husks	16
2.7.2 Coconut Coir	17
2.7.3 Corn Cobs	18
2.7.4 Neem Tree	19
2.7.5 <i>Moringa oleifera</i>	20
2.8 Preparation of Activated Carbon	21
2.8.1 Carbonization	21
2.8.2 Activation Step	22
2.8.2.1 Physical Activation	22
2.8.2.2 Chemical Activation	23
2.8.2.3 Physico-chemical Activation	25
2.8.3 Ash in Activated Carbon	26
2.9 Characterization of Activated Carbon	27
2.9.1 Physical Properties	27
2.9.1.1 Carbon Yield	28
2.9.1.2 Activated Carbon Structure	29
2.9.1.3 Pore and Surface Area Characteristics	29
2.9.1.4 Surface Morphology	30
2.9.2 Chemical Properties	30
2.9.2.1 Proximate Analysis	30
2.9.2.2 Surface Functional Groups	31
2.10 Adsorption Factors	31
2.11 Adsorption Equilibrium Models	32
2.11.1 Langmuir Isotherm	32
2.11.2 Freundlich Isotherm	33
2.11.3 Dubinin – Radushkevich (D-R) isotherm	34
2.12 Adsorption Kinetics Models	35
2.12.1. Pseudo-First-Order Kinetic Model	35
2.12.2. Pseudo-Second-Order Kinetic Model	36
2.12.3 Intra-Particle Diffusion	37
2.13 Adsorption Mechanism of Activated Carbon	38
2.14. Regeneration and Desorption of Used AC	40
2.14.1. Regeneration	40

2.14.2. Desorption of Organics and Heavy Metals	41
<b>CHAPTER 3</b>	<b>42</b>
<b>PREPARATION OF ACTIVATED CARBONS FROM FIVE AGRICULTURAL WASTE PRODUCTS AND STUDIES OF HEAVY METALS REMOVAL</b>	<b>42</b>
3.1 Introduction	42
3.2 Materials and Methods	42
3.2.1 Reagents	42
3.2.2 Preparation of the Adsorbents	43
3.2.3 Instrument	46
3.2.4 Batch Studies of Adsorption of Heavy Metals on AC	47
3.2.5 Influence of Initial Concentration	48
3.2.6 Influence of Contact Time	48
3.2.7 Influence of Adsorbent Dosage	49
3.2.8 Influence of pH	49
3.2.9 Equilibrium studies	49
3.2.10 Kinetic Studies	50
3.2.11 Desorption Studies	50
3.2.12 The Point of Zero Charge (pHpzc)	51
3.3 Results and Discussion	52
3.3.1 Influence of Initial Concentration	52
3.3.2 Influence of Contact Time	54
3.3.3 Influence of Adsorbent Dose	60
3.3.4 Influence of pH	61
3.3.5 Adsorption Isotherms	65
3.3.6 Adsorption Kinetics	71
3.3.7 Desorption Studies	75
3.4 Conclusions	83
<b>CHAPTER 4</b>	<b>84</b>
<b>CHARACTERIZATION OF THE PRODUCED ACTIVATED CARBONS</b>	<b>84</b>
4.1 Introduction	84
4.2 Materials	84
4.3 Methods	84

4.3.1 Proximate Analysis of the Activated carbon	84
4.3.1.1 Determination of Moisture Content	85
4.3.1.2 Determination of Volatile Matter Content	85
4.3.1.3 Fixed Carbon	87
4.3.2 Surface Morphology	87
4.3.3 Surface Chemistry	88
4.3.4 Experimental Setup for Furnace	88
4.3.5 Carbonization	89
4.3.6 Chemical Impregnation	89
4.3.7 Pyrolysis for Activation	90
4.3.8 Washing	91
4.3.9 Surface Area and Porosity Measurement	91
4.4 Results and Discussion	92
4.4.1 Characterization of the activated carbon	92
4.4.1.1 Proximate Analysis	92
4.4.1.2 Ultimate Analysis	93
4.4.1.3 Surface Area and Porosity	94
4.4.1.4 Surface Morphology	98
4.4.1.5 Surface Functional Groups	99
4.4.1.6 Adsorption Isotherm of the two best Activated Carbon	106
4.5 Conclusion	108
CHAPTER 5	109
ADSORPTION OF METHYLENE BLUE BY THE PREPARED FIVE ACTIVATED CARBONS	109
5.1 Introduction	109
5.2 Materials and Chemicals	109
5.2.1 Sample of Agricultural By-Products	109
5.2.2 Chemicals	109
5.3 Adsorption Experiments	110
5.3.1 Effect of Initial Adsorbate Concentration	111
5.3.2 Effect of Contact Time	111
5.3.3 Effect of Solution pH	111
5.3.4 Effect of Adsorbent Dosage	111

5.4 Results and Discussion	112
5.4.1 Effect of Initial adsorbate Concentration	112
5.4.2 Effect of Contact Time	114
5.4.3 Effect of pH Solution	115
5.4.4 Effect of Adsorbent Dosage	116
5.4.5 Adsorption Isotherms of Methylene Blue	117
5.4.6 Adsorption Kinetic Studies	121
5.5 CONCLUSION	124
CHAPTER 6	125
GENERAL DISCUSSION AND CONCLUSION	125
6.1 Conclusions of the Thesis	125
6.2 The Used of Agricultural Waste Convert into Activated Carbon	125
6.3 Evaluation of the Prepared Adsorbents in Batch	126
6.4 Efficiency of the Prepared Adsorbents on Removal of Heavy Metals	128
6.5 Efficiency of the Prepared Adsorbents on Removal of Methylene Blue	128
6.6 Conclusion	129
6.7 Scope for Future Research	130
REFERENCES	131
APPENDIX	1522
LIST OF PUBLICATIONS	155

## LIST OF TABLES

<b>Table 2.1</b>	A list of some activated carbon produced from agricultural waste using physical activation method	23
<b>Table 2.2</b>	A list of some agricultural activated carbon prepared using chemical activation technique	25
<b>Table 2.3</b>	A list of some application of physic-chemical activation technique in preparation of activated carbon	26
<b>Table 3.1</b>	Isotherm factors for the uptake of Cu (II), Cd (II), Pb (II) and Ni (II) onto rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	70
<b>Table 3.2</b>	Kinetics factors for the uptake of Cu (II), Cd (II), Pb (II) and Ni (II) onto rice husk, coconut coir, corn cobs, neem bark and <i>Moringa oleifera</i> bark	75
<b>Table 3.3</b>	Adsorption desorption table with different chemical	79
<b>Table 3.4</b>	Desorption efficacy with 0.1N HCl during several cycles	82
<b>Table 4.1</b>	The impregnation ratio (IR) using varieties chemical and char	90
<b>Table 4.2</b>	The activation temperature applied for 1 hour of each natural material	91
<b>Table 4.3</b>	Proximate analysis of all adsorbents	93
<b>Table 4.4</b>	Weight percentage for the elements in coconut coir	94
<b>Table 4.5</b>	Surface area and pore characteristics of the prepared activated carbons	95
<b>Table 4.6</b>	Comparison of FTIR band positions of raw rice husk before and after metal ions activation in wave number ( $\text{cm}^{-1}$ ).	101
<b>Table 4.7</b>	Comparison of FTIR band positions of raw coconut coir before and after activation in wave number ( $\text{cm}^{-1}$ )	102
<b>Table 4.8</b>	Comparison of FTIR band positions of raw corn cobs before and after activation in wave number ( $\text{cm}^{-1}$ )	103

<b>Table 4.9</b>	Comparison of FTIR band positions of raw neem bark before and after activation in wave number ( $\text{cm}^{-1}$ )	104
<b>Table 4.10</b>	Comparison of FTIR band positions of raw <i>Moringa oleifera</i> bark before and after activation in wave number ( $\text{cm}^{-1}$ )	105
<b>Table 4.11</b>	Infrared assignment of functional groups on the prepared activated carbon surface (Adapted from Ahmad et. al., 2013)	106
<b>Table 5.1</b>	Separation factor, $R_L$ (Ahmad & Alrozi, 2011)	118
<b>Table 5.2</b>	Isotherm constants for adsorption of methylene blue by all activated carbon	121
<b>Table 5.3</b>	Adsorption kinetics parameters for methylene blue adsorption	124

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## LIST OF FIGURES

<b>Figure 2.1</b>	Rice paddy and husk	16
<b>Figure 2.2</b>	Coconut tree and coir	18
<b>Figure 2.3</b>	Corn cobs and tree	19
<b>Figure 2.4</b>	Neem tree and bark	19
<b>Figure 2.5</b>	<i>Moringa oleifera</i> tree and bark	20
<b>Figure 2.6</b>	Adsorption mechanism of different types of the pollutants	38
<b>Figure 2.7</b>	Regeneration process of activated carbon	40
<b>Figure 3.1</b>	Raw materials of rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	46
<b>Figure 3.2</b>	Influence of initial concentration on the adsorption capacity of (a) lead, (b) nickel, (c) copper, and (d) cadmium for rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	53
<b>Figure 3.3</b>	Influence of contact time of (a) lead, (b) nickel, (c) copper, and (d) cadmium for rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	55
<b>Figure 3.4</b>	Adsorption capacity in 5 mg/L of (a) lead, (b) nickel, (c) copper, and (d) cadmium for rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	56
<b>Figure 3.5</b>	Influence of adsorbent dose in 5 mg/L of (a) lead, (b) nickel, (c) copper, and (d) cadmium for rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	60
<b>Figure 3.6</b>	The final pH against initial pH plots for rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	61
<b>Figure 3.7</b>	Influence of pH plots in 5 mg/L of (a) lead, (b) nickel, (c) copper, and (d) cadmium for rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	62

- Figure 3.8** Langmuir plot for (a) nickel, (b) cadmium, and (c) copper adsorption of rice husk, coconut coir, corn cobs, neem bark, and *Moringa oleifera* bark 66
- Figure 3.9** Freundlich plot for (a) nickel, (b) copper, and (c) cadmium adsorption of rice husk, coconut coir, corn cobs, neem bark, and *Moringa oleifera* bark 67
- Figure 3.10** D-R plot for (a) lead, (b) nickel, (c) copper, and (d) cadmium uptake of rice husk, coconut coir, corn cobs, neem bark, and *Moringa oleifera* bark 69
- Figure 3.11** First order graph for (a) lead, (b) nickel, (c) copper, and (d) cadmium uptake of rice husk, coconut coir, corn cobs, neem bark, and *Moringa oleifera* bark 71
- Figure 3.12** Second order graph for (a) lead, (b) nickel, (c) copper, and (d) cadmium uptake of rice husk, coconut coir, corn cobs, neem bark, and *Moringa oleifera* bark 73
- Figure 3.13** Intraparticle diffusion of (a) lead, (b) nickel, (c) copper, and (d) cadmium adsorption for rice husk, coconut coir, corn cobs, neem bark, and *Moringa oleifera* bark 74
- Figure 3.14** Desorption efficiency using 0.1N different solvents for lead of all AC 76
- Figure 3.15** Desorption efficiency using 0.1N different solvents for nickel of all AC 77
- Figure 3.16** Desorption efficiency using 0.1N different solvents for copper of all AC 77
- Figure 3.17** Desorption efficiency using 0.1N different solvents for cadmium of all AC 78
- Figure 3.18** Desorption efficacy with 0.1N HCl during several cycles for lead of rice husk, coconut coir, corn cobs, neem bark, and *Moringa oleifera* bark 80
- Figure 3.19** Desorption efficacy with 0.1N HCl during several cycles for nickel of rice husk, coconut coir, corn cobs, neem bark, and *Moringa oleifera* bark 80
- Figure 3.20** Desorption efficacy with 0.1N HCl during several cycles for copper of rice husk, coconut coir, corn cobs, neem bark, and *Moringa oleifera* bark 81

<b>Figure 3.21</b>	Desorption efficacy with 0.1N HCl during several cycles for cadmium of rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	81
<b>Figure 4.1</b>	EDX analysis of the coconut husk and its derived char at 700°C.	94
<b>Figure 4.2</b>	Pore size distribution of rice husk and <i>Moringa oleifera</i> bark	97
<b>Figure 4.3</b>	SEM micrograph of <i>Moringa Oleifera</i> bark (a)-mag.-1000x and (b)-mag.-30,000x before and (c)-mag.-1000x and (d)-mag.-30,000x) after activation	98
<b>Figure 4.4</b>	FTIR spectra for before and after activation on rice husk	101
<b>Figure 4.5</b>	FTIR spectra before and after activation on coconut coir	102
<b>Figure 4.6</b>	FTIR spectra before and after activation on corn cobs	103
<b>Figure 4.7</b>	FTIR spectra before and after activation on neem bark	104
<b>Figure 4.8</b>	FTIR spectra before and after activation on <i>Moringa oleifera</i> bark	105
<b>Figure 4.9</b>	Adsorption isotherm of rice husk and <i>Moringa oleifera</i> bark	107
<b>Figure 5.1</b>	Effect of initial concentration on methylene blue removal by rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	113
<b>Figure 5.2</b>	Adsorption capacity on methylene blue (50 mg/L) removal by rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	114
<b>Figure 5.3</b>	Effect of pH change for methylene blue (5 mg/L) of rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	116
<b>Figure 5.4</b>	Effect of dosage for methylene blue (5 mg/L) of rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	117
<b>Figure 5.5</b>	Langmuir adsorption for methylene blue (50 mg/L) of rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	119
<b>Figure 5.6</b>	Freundlich adsorption for methylene blue (50 mg/L) of rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	120
<b>Figure 5.7</b>	Dubinin-Radushkevich plot for methylene blue adsorption of rice husk, coconut coir, corn cobs, neem bark, and <i>Moringa oleifera</i> bark	120

- Figure 5.8** Pseudo-first-order of methylene blue (50 mg/L) kinetic adsorption for rice husk, coconut coir, corn cobs, neem bark, and *Moringa oleifera* bark 122
- Figure 5.9** Pseudo-second-order of methylene blue (50 mg/L) kinetic adsorption for rice husk, coconut coir, corn cobs, neem bark, and *Moringa oleifera* bark 123
- Figure 5.10** Intra-particle diffusion of methylene blue (50 mg/L) for rice husk, coconut coir, corn cobs, neem bark, and *Moringa oleifera* bark 123

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## LIST OF ABBREVIATION

AC	Activated Carbon
AAS	Atomic Absorption Spectroscopy
BET	Brunauer Emmett Teller
CC	Coconut Coir
CCB	Corn Cob
FTIR	Fourier-Transform Infrared Spectroscopy
SEM	Scanning Electron Microscopy
RH	Rice Husk
MO	<i>Moringa Oleifera</i>
NB	Neem Bark
UV	Ultra-Violet Spectroscopy
UNICEF	United Nation International Children's Emergency Fund
WHO	World Health Organization