PRODUCTION AND CHARACTERIZATION OF ACTIVATED CARBON FROM BAOBAB FRUIT SHELL VIA CHEMICAL ACTIVATION FOR THE REMOVAL OF PHENOL

BY

RADHIA NEDJAI

A thesis submitted in fulfillment of the requirement for the degree of Doctor of Philosophy (Engineering)

Kulliyyah of Engineering
International Islamic University Malaysia

OCTOBER 2021

ABSTRACT

Palm oil mill effluent (POME) causes severe environmental pollution due to its high concentrated pollutants. One of the most effective treatment methods is the adsorption using the activated carbon (AC), which is considered as a solution to wastewater pollution problems but suffering from high cost due to its non-renewable sources. In this study, the potential of converting baobab fruit shells (BFS) into activated carbon was investigated as well as being used in treating POME for removal of the phenol. In this research, different chemicals ($ZnCl_2$, H_3PO_4, KOH) were used as activating agents for the preparation of AC from baobab fruit shell, which were impregnated (1:1) followed by the carbonized at temperature 500°C for 1 h. The effects of these chemicals on the performances of the prepared activated carbons (yield, iodine number, adsorption properties) were investigated. The BFS-ACs characterized to investigate by using the different analytical approaches such as FTIR, SEM, and XRD. The results indicated that the KOH in terms of adsorption and efficiency showed better results than the $ZnCl_2$ and H_3PO_4 for the removal of phenol, with a maximum adsorption capacity of 36.9 mg/g at a higher initial concentration (600 mg/L). The aim was to obtain optimum operating conditions for KOH-AC production for maximum phenol removal. KOH-AC samples were produced under varying the operating parameters of temperatures, activation times, and impregnation ratio using face centered central composite design (FCCCD) experimental design under response surface methodology (RMS). The optimum conditions to attain a removal percentage of 93.56 % were determined by employing the RSM. The results demonstrated that the activated carbon prepared at the activation temperature of 700°C for 60 minutes with an impregnation ratio of 1:2 showed the best adsorbent of phenol. KOH-AC was found comparable to the commercial-grade activated carbon. Characterization of the KOH-AC showed good quality adsorbent with highly active sites and well-developed pores with BET surface area of 1263.127 m^2/g. Furthermore, an optimization study for the adsorption conditions of the selected optimum parameters for KOH-AC production was investigated using the RSM. The determining factors such as contact time, AC dose, pH, agitation speed was initially screened using 2-level factorial approach. The screening revealed that the effect of the above parameters was significant. Furthermore, the impact of these four operating parameters was investigated using the FCCCD technique. The results presented the optimum conditions for phenol removal from aqueous solution were found to be contact time of 15 min, KOH-AC dose of 3 g/L, pH 2, and agitation speed of 250 rpm. Phenol adsorption behavior were described by the Redlich-Peterson (R-P) isotherm model as well as the pseudo-second-order kinetics. The maximum adsorption capacity of phenol (q_m) was 196.68 mg/g. An evaluation of the adsorption efficiency of the BFS based KOH-AC was examined in real wastewater as the palm oil mill final effluent (POME) using batch adsorption. It was found that BFS-AC is an efficient adsorbent for the removal of phenol from palm oil mill effluent (POME). Also, BFS-AC with bed height of 15 cm provided better eliminations of phenol with empty bed contact time (EBCT) of 9.9 minutes and carbon usage rate (CUR) of 1.74 g/L. The results obtained in this study have exposed the capability of BFS based AC in the removal of phenol and treating POME wastewater. Thus, this activated carbon can be a promising source for treating POME wastewater.

خلاصة البحث

تتسبب النفايات السائلة لمصانع زيت النخيل (POME) في تلوث بيئي شديد بسبب تركيزها العالي من حيث الملوثات. ومن بين أكثر طرق المعالجة فاعلية هي الامتزاز باستخدام الكربون المنشط (AC)، والذي يعتبر حلاً لمشاكل تلوث مياه الصرف الصحي، ولكنه يعاني من التكلفة العالية بسبب مصادره غير المتجددة. في هذه الدراسة، تمت دراسة إمكانية تحويل قشور فاكهة الباوباب (BFS) إلى كربون منشط وكذلك استخدامه في علاج POME من الفينول. في هذا البحث تم استخدام مواد كيميائية مختلفة ($ZnCl_2$، H_3PO_4، KOH) كعوامل تنشيط لتحضير AC من قشرة فاكهة الباوباب المشبعة (1: 1) ثم تفحمها عند درجة حرارة 500 درجة مئوية لمدة ساعة. تمت دراسة تأثير هذه المواد الكيميائية على أداء الكربون النشط المحضر (المحصول، عدد اليود، خواص الامتزاز). تم تمييز BFS-ACs بالتحقيق باستخدام نهج تحليلي مختلف مثل FTIR، SEM، وXRD. أشارت النتائج إلى أن KOH من حيث الامتصاص والكفاءة أظهر نتائج أفضل من $ZnCl_2$ وH_3PO_4 لإزالة الفينول، مع قدرة امتصاص قصوى تبلغ 36.90 مجم/جم عند تركيز أولي أعلى (600 مجم/لتر). كان الهدف هو الحصول على ظروف التشغيل المثلى لإنتاج KOH-AC لإزالة أقصى قدر من الفينول. تم إنتاج عينات KOH-AC وفقًا لمعايير التشغيل المتغيرة لدرجات الحرارة وأوقات التنشيط ونسبة التشريب باستخدام التصميم التجريبي (FCCCD). كما تم تحديد الظروف المثلى لتحقيق قدرة امتصاص 93.56مج/ج باستخدام منهجية سطح الاستجابة (RSM). أظهرت النتائج أن الكربون المنشط المحضر عند درجة حرارة التنشيط البالغة 700 درجة مئوية لمدة 60 دقيقة مع نسبة تشريب 1: 2 يمثل أفضل امتصاص للفينول. تم الكشف أن KOH-AC قابل للمقارنة مع الكربون المنشط التجاري. أظهر توصيف KOH-AC على أنه مادة ماصة عالية الجودة مع مواقع نشطة للغاية ومسام متطورة مع مساحة سطح BET تبلغ 1263.127 م^2/جم. علاوة على ذلك، تم دراسة التحسين لظروف الامتزاز للمعلمات المثلى المحددة لإنتاج KOH-AC باستخدام منهجية سطح

الاستجابة (RSM). تم فحص العوامل المحددة مثل وقت التلامس، وجرعة الكربون، ودرجة الحموضة، وسرعة التحريض في البداية باستخدام نهج عاملي ذو مستويين. أظهر الفحص أن تأثير المعلمات المذكورة أعلاه كان معنويا. علاوة على ذلك، تم التحقق من تأثير هذه المعلمات التشغيلية الأربعة باستخدام تقنيات التصميم المركب المركزي (CCD). عرضت النتائج الظروف المثلى لإزالة الفينول من المحلول المائي حيث وجد أن وقت التلامس 15 دقيقة، جرعة 3 جم/لتر من KOH-AC، درجة الحموضة تساوي 2، وسرعة التحريك 250 دورة في الدقيقة. يمكن وصف سلوك امتصاص الفينول بواسطة نموذج متساوي الحرارة Redlich-Peterson (R-P) بالإضافة إلى الخواص الحركية الزائفة من الدرجة الثانية. كانت السعة القصوى لامتصاص الفينول (q_m) 196.68مجم/جم. تم فحص تقييم كفاءة الامتزاز لـ KOH-AC القائم على BFS في النفايات السائلة النهائية لمصنع زيت النخيل (POME) باستخدام الامتزاز الدفعي. وجد أن BF-AC هو مادة ماصة فعالة لإزالة الفينول من النفايات السائلة لمصانع زيت النخيل (POME). كما يوفر BF-AC مع ارتفاع السرير 15 سم إزالة أفضل للفينول مع وقت ملامسة السرير الفارغ (EBCT) من 9.9 دقيقة ومعدل استخدام الكربون (CUR) 1.74 جم/لتر. كشفت النتائج التي تم الحصول عليها في هذه الدراسة عن قدرة الكربون المنشط القائم على BFS في إزالة الفينول ومعالجة مياه الصرف الصحي POME. وبالتالي، يمكن أن يكون هذا الكربون المنشط مصدرًا واعدًا لمعالجة مياه الصرف الصحي POME.

APPROVAL PAGE

The thesis of Radhia Nedjai has been approved by the following:

_____
Nassereldeen Ahmed Kabbashi
Supervisor

_____
Ma'an Alkhatib
Co-supervisor

_____
Md. Zahangir Alam
Co-supervisor

_____
Mohammed Saedi Jami
Internal Examiner

_____
Abdurahman Hamid Nour
External Examiner

_____
Mubarak Mujawar
External Examiner

_____
Radwan Jamal Yousef Elatrash
Chairman

DECLARATION

I hereby declare that this thesis is the result of my own investigations, except where otherwise stated. I also declare that it has not been previously or concurrently submitted as a whole for any other degrees at IIUM or other institutions.

Radhia Nedjai

Signature …… Date ……..

INTERNATIONAL ISLAMIC UNIVERSITY MALAYSIA

DECLARATION OF COPYRIGHT AND AFFIRMATION OF FAIR USE OF UNPUBLISHED RESEARCH

PRODUCTION AND CHARACTERIZATION OF ACTIVATED CARBON FROM BAOBAB FRUIT SHELL VIA CHEMICAL ACTIVATION FOR THE REMOVAL OF PHENOL

I declare that the copyright holder of this thesis is jointly owned by the student and International Islamic University Malaysia.

By signing this form, I acknowledged that I have read and understand the IIUM Intellectual Property Right and Commercialization policy.

Affirmed by Radhia Nedjai

……. ……..
Signature Date

"Dedicated first to Almighty ALLAH then to my beloved parents & family for their endless love and support"

ACKNOWLEDGEMENT

First and foremost, I would like to extend my heartfelt gratitude to Almighty ALLAH for giving me strength, perseverance and all the means to make this study a reality. Moreover, the accomplishment of this thesis is made possible with the valuable support from many people who had a direct and indirect contribution to this work.

It is my utmost pleasure to dedicate this work to my dear parents, sister, and brothers. Words cannot express my gratitude to them for their encouragement and endless emotional support. My gratitude goes to my beloved husband for his prayers, support, understanding, and endurance.

To Prof. Dr. Nassereldeen Ahmed Kabbashi, my supervisor, who with his valuable guidance, suggestions, and support made this thesis possible. I am also grateful to my co-supervisors, Prof. Dr. Md. Zahangir Alam and Assoc. Dr. Ma'an Alkhatib, for their valuable explanations which helped me to clarify some obscure parts in this study, and for their kind support and meaningful contributions.

Once again, we glorify Allah for His endless mercy on us one of which is enabling us to successfully round off the efforts of writing this thesis, Alhamdulillah.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

°	Degree
%	percentage
cm	centimeter
mg/g	milligram per gram
l	liter
cm^3	cubic centimeter
Kg	kilogram
g	gram
h	hour
mg	milligram
min	minute
nm	nanometer
°C	degree Celsius
v/v	volume per volume
w/v	weight per volume
ml	milliliter
mm	millimeter
mt	metric tone
g/L	gram per liter
1/n	The Intensity Parameter in Freundlich Isotherm
C_0	Initial Concentration
C_t	Concentration at time t
K_1	Rate constant of pseudo-first-order
K_2	Rate constant of pseudo-second-order
K_F	Freundlich Isotherm Constant
K_L	Langmuir Adsorption Constant
q_e	Equilibrium Adsorption Capacity
q_m	Maximum Adsorption Capacity
q_t	Adsorption Capacity at time t
T	Temperature
ppm	parts per million
rpm	revolutions per minute
R^2	Coefficient of determination
sec	second
H_{MTZ} (δ)	Height of Mass Transfer Zone
Z	Height of the adsorption column
V_E	Throughput volume to exhaustion
V_B	Throughput volume to breakthrough
f	Fractional capacity of the active adsorption zone
EBCT	empty bed contact time
V_f	Volume occupied by adsorbent media including porosity volume
A_f	Adsorbent area available for flow
Q	Flow rate to adsorber
L	Adsorbent or media depth

v	Superficial flow velocity
V	volume of liquid passed through the column
V_b	represents the effluent volume at breakthrough point
V_x	volume of effluent at exhaustion point
t_b	Column Breakthrough Time
t_x	Column Exhaust Time
t_δ	Time to Exhaust Mass Transfer Zone
CUR	carbon usage rate
K_H	Halsey isotherm constant
n	Freundlich constant
n_H	Halsey isotherm constant
A	Redlich-Peterson isotherm constant
B	Redlich-Peterson isotherm constant
wt	weight