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

BY

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# 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 $ZnCl_2$ و$H_3PO_4$ 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:

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# 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 ……………………..

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Signature Date

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

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| | |
|---|---|
| ° | 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 |
| $t_\delta$ | 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 |