



Faculty of Engineering

**OPTIMIZATION STUDY ON PREPARATION OF ACTIVATED CARBON
FROM ORANGE PEEL USING MICROWAVE INDUCED KOH
ACTIVATION**

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OPTIMIZATION STUDY ON PREPARATION OF ACTIVATED CARBON FROM
ORANGE PEEL USING MICROWAVE INDUCED KOH ACTIVATION

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A dissertation submitted in partial fulfilment
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Dedicated to my beloved parents and my family who always bestow me sustainable motivations and encouragements

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ABSTRACT

The prolific use of commercial activated carbon (AC) is restricted despite AC has been known to be one of the most effective adsorbent due to its high cost since the precursor is usually non-renewable. Hence, this study aims to investigate the feasibility of producing AC from orange peel (OP) since the high demand of orange fruit in food processing industry is resulting in high output of fruit peels as waste which contain high carbon source with low ash content, great adsorption capacity as well as with considerable mechanical strength. This research focuses on the preparation of AC from OP using KOH impregnation followed by microwave - induced activation. The effects of three preparation variables (microwave power, irradiation time and KOH:char impregnation ratio) on Methylene Blue (MB) adsorption and iodine adsorption were investigated. Based on the central composite design, mathematical models were developed to correlate the three preparation variables to the two responses. From the analysis of variance (ANOVA), the most influential factor on each experimental design response was identified. Two quadratic models and 3-D graphs were obtained for the influential factors of each responses. The optimization study with an initial MB concentration of 200 mg/L showed that the AC produced with impregnation ratio of 1.62, microwave power of 655.74 W and irradiation time of 5.77 minutes exhibited the highest MB and iodine adsorption uptake of 134.86 mg/g and 1147mg/g respectively. SEM analyses revealed that the prepared AC consisted of uniformly developed pores. FTIR spectrum showed the presence of various functional groups on the surface of the raw OP and prepared AC where there was some shift of adsorption bands occurred due to the evaporation of volatile materials present in the raw OP when heat was supplied to the samples. . Batch adsorption studies of MB on the prepared AC were conducted to evaluate the equilibrium isotherms, kinetics and adsorption mechanism. The adsorption of MB was best fitted by the Langmuir isotherm model and best described by the pseudo-second order kinetic model. The maximum monolayer adsorption capacity of the AC on MB was 144.93 mg/g. The OP based AC was shown to be a promising adsorbent for the removal of pollutant from aqueous solutions.

ABSTRAK

Penggunaan karbon aktif komersil (AC) yang produktif dibatasi walaupun AC dikenalpasti sebagai salah satu penyerap yang paling berkesan kerana biaya yang tinggi sejak pendahulu biasanya tidak dapat diperbaharui. Oleh itu, kajian ini bertujuan untuk mengkaji kelayakan pengeluaran AC dari kulit jeruk (OP) kerana permintaan tinggi buah jeruk dalam industri pemprosesan makanan mengakibatkan keluaran buah-buahan yang tinggi sebagai bahan buangan yang mengandungi sumber karbon yang tinggi dengan kandungan abu yang rendah kapasiti penyerapan serta kekuatan mekanikal yang besar. Penyelidikan ini memberi tumpuan kepada penyediaan AC dari OP menggunakan KOH impregnation diikuti dengan pengaktifan induksi gelombang mikro. Kesan daripada tiga pemboleh ubah penyediaan (kuasa gelombang mikro, masa penyinaran dan KOH: nisbah penyerapan char) pada penyerapan Methylene Blue (MB) dan penyerapan iodin diselidiki. Berdasarkan reka bentuk komposit pusat, model matematik telah dibangunkan untuk mengaitkan tiga pemboleh ubah penyediaan kepada dua respon. Dari analisis varians (ANOVA), faktor yang paling berpengaruh terhadap setiap tindak balas reka bentuk eksperimen telah dikenalpasti. Kajian pengoptimuman dengan kepekatan awal MB sebanyak 200 mg / L menunjukkan bahawa AC dihasilkan dengan nisbah impregnasi sebanyak 1.62, kuasa gelombang mikro 655.74 W dan masa penyinaran selama 5.77 minit mempamerkan pengambilan MB dan penyerapan penyerapan iodin tertinggi iaitu 134.86 mg/g dan 1147mg/g masing-masing. Analisis SEM mendedahkan bahawa AC yang disediakan terdiri daripada liang-liang yang dikembangkan seragam. Spektrum FTIR menunjukkan kehadiran pelbagai kumpulan berfungsi pada permukaan OP mentah dan AC yang disediakan di mana terdapat beberapa pergeseran band penyerapan yang berlaku disebabkan oleh penyejatan bahan tidak menentu yang hadir dalam OP mentah apabila haba dibekalkan kepada sampel. Kajian penyerapan batch MB pada AC yang disediakan telah dijalankan untuk menilai isoterma keseimbangan, kinetik dan mekanisme penyerapan. Penyerapan MB paling sesuai dengan model isoterm Langmuir dan yang paling digambarkan oleh model kinetik pesanan pseudo-kedua. Kapasiti penyerapan monolayer maksimum AC pada MB adalah 144.93 mg/g.

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NOMENCLATURE

%	:	Percent
°C	:	Degree Celsius
cm ⁻¹	:	Per centimetre
g	:	Gram
mg/g	:	Milligram per gram
mL	:	Milli litre
rpm	:	Revolution per minute
W	:	Watt
min	:	minute

ABBREVIATIONS

AC	:	Activated Carbon
ANOVA	:	Analysis of Variance
BET	:	Brunauer-Emmett-Teller
CCD	:	Central Composite Design
FTIR	:	Fourier Transform Infrared Spectroscopy
GAC	:	Granular Activated Carbon
H ₃ PO ₄	:	Phosphoric Acid
HCl	:	Hydrochloric Acid
IUPAC	:	International Union of Pure and Applied Chemistry
KOH	:	Potassium Hydroxide
MB	:	Methylene Blue
OP	:	Orange Peel
RSM	:	Response Surface Methodology
SEM	:	Scanning Electron Microscope

CHAPTER 1

INTRODUCTION

1.1 Environmental Pollution: Current Situation and Problems

Environmental pollution is considered as a global phenomenon as it can cause adverse effects on human health, plants, animals and exposed materials. Industrialization and urbanization are attributing greatly to the pollution issues due to the huge amount of organic and industrial effluents released to the environment which increases water, land and air pollution problems manifold (Rajan & David, 2014). The discharge of effluent which contain harmful chemicals and compounds that attributes to water pollution is a major concern due to their adverse effect to many forms of life since large quantity of effluent is discharged into the water bodies. The presence of dye effluent is highly toxic in nature due to its high suspended solid, COD, dye and chemicals along with high concentration of heavy metals such as Cu and Cd (Tan, et al., 2008). Furthermore, the contaminated surface and the ground water by the untreated industrial effluent is no longer suitable for irrigation and drinking (Rajan & David, 2014).

Industries such as textile, leather, paper, plastics, etc., are some of the sources for dye effluents discharge. According to Wong et al. (2004), the global dye consumption of the textile industry has reached the amount of 107 kg/year and it is estimated 90% of this ends up on fabrics. Consequently, about 1,000,000 kg/year of dye are discharged into waste

streams by the textile industry. In this case, Methylene Blue (MB) is a widely used substance for dyeing cotton, wood and silk which has its harmful effects including permanent injury to the eyes of human and animals. Besides, upon inhalation, it can cause short breath or difficulty in breathing while ingestion through the mouth causes a burning sensation and may cause vomiting, mental confusion, nausea and methemoglobinemia profuse sweating (Chen et al., 2007).

Beside water pollution, another serious problem in environmental issue is the air pollution. Greenhouse gas (GHG) emission which contributes to global warming is the most worrisome situation all over the world as it can cause climate changes. According to (Scientific Malaysian, 2017) Malaysia's total GHG emission was recorded as 290.23 Mt CO₂eq. Beside GHG, some other pollutants like volatile organic compounds, sulphur dioxide and nitrogen dioxide act as pre-cursors to others air pollutants. For example, NO_x and SO₂ can react in the atmosphere and lead to particulate matter (PM) compounds formation. The sources of each pollutant most of the time linked with fuel combustion and industrial activities; pollutants are released as by-products of these processes (Cohen et al., 2015).

Malaysia has gained benefits from the significant development during recent years. However, there is a price to pay behind it. The speedy development has increased the severity of the environmental pollution problems like discharge of pollution causing matters into environment. In Malaysia, the Environmental Quality Act 1974 (amended 1985) is the law that is directly related to the environmental pollution and the Department of Environment is the authority to monitor and enforce the act (EQA, 2015).

1.2 Activated Carbon

Over the centuries, activated carbon (AC) is a commonly used adsorbent for wastewater treatment especially to purify the effluents from an industry. The abundant use of AC as adsorbent is due to certain characteristics such as tremendously high surface area with various micropore volumes, high capacity of adsorption, fast adsorption kinetics as well as the ability of regeneration (Sircar et al. 2008). AC can be extracted from a variety of carbonaceous materials such as petroleum pitch, agricultural waste and even by using waste tires. In this case, the production of AC is highly depending on the availability of the raw