# WASTE TYRE POWDER-BASED ACTIVATED CARBONS BY $CO_2$ ACTIVATION FOR METHYLENE BLUE AND PHENOL REMOVAL

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*To my beloved parents, Zaiton Binti Saat and Mohd Shaid bin Tasiran as well as my relatives for their limitless love and encouragement*

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## ABSTRACT

The development of industries in Malaysia has led to water pollution. Adsorption is an effective technique in the treatment of wastewater as it utilizes low cost adsorbent, no sludge generation, and simple to operate. Pyrolysis waste tyre powder is a suitable precursor of activated carbon due to its high carbon content, less commercial value and abundantly available. This work aims to evaluate the adsorption properties of activated carbons prepared from pyrolysed waste tyre powder for the removal of positively charged methylene blue and negatively charged phenol from water. The activated carbons were prepared by physical activation using carbon dioxide at activation temperatures of 900 to 1000 ℃ for 2 to 8 h. The resultant activated carbons were modified with hydrofluoric acid followed by nitric acid, and were characterized for Brunauer–Emmett–Teller surface area, pH value at the point of zero charge, scanning electron microscope, functional groups and thermal gravimetric analysis. The adsorption of methylene blue and phenol were studied at varying concentrations (5 - 200 mg/L), contact times (5 min - 72 h) and temperatures (30 - 60 $^{o}$C). The isotherm, kinetics and thermodynamics models were employed to describe the adsorption data. The specific surface area of activated carbons increased with activation temperature and time. The maximum adsorption of methylene blue is 132 mg/g. The oxidized activated carbon showed an increase of methylene blue adsorption from 102 mg/g (bulk) to 107 mg/g. However, the phenol adsorption decreased from 48 to 39 mg/g after oxidation. The equilibrium data of methylene blue adsorption fitted well with the Langmuir and Redlich-Peterson models, while that of phenol adsorption obeyed the Freundlich isotherm. The kinetics data of both model pollutants could be described by the pseudo-second-order model. The rate-limiting step in the adsorption of methylene blue and phenol could be dominated by pore diffusion. The positive enthalpy change and entropy change indicate that the adsorption of methylene blue is endothermic and spontaneous at high temperatures, while the phenol adsorption is exothermic and spontaneous at low temperatures. In conclusion, the activated carbons prepared are feasible to be used as an adsorbent.

# ABSTRAK

Perkembangan industri di Malaysia telah membawa kepada pencemaran air. Penjerapan merupakan teknik yang efektif dalam rawatan air kerana kos penjerap yang rendah, tiada penghasilan enap cemar, dan mudah dikendalikan. Serbuk tayar terpirolisis sesuai untuk menghasilkan karbon teraktif kerana kandungan karbon yang tinggi, kurang nilai komersial dan boleh didapati dengan mudah dan banyak. Penyelidikan ini bertujuan untuk menilai sifat-sifat penjerapan karbon teraktif yang disediakan daripada serbuk tayar terpirolisis untuk penyingkiran metilena biru bercas positif dan fenol bercas negatif daripada air. Karbon teraktif disediakan secara pengaktifan fizikal menggunakan karbon dioksida pada suhu pengaktifan 900 hingga 1000 ℃ selama 2 hingga 8 jam. Karbon teraktif yang terhasil diubahsuai dengan asid hidrofluorik diikuti oleh asid nitrik, dan dicirikan dengan luas permukaan Brunauer–Emmett–Teller, nilai pH pada titik sifar caj, mikroskop elektron pengimbas, kumpulan berfungsi dan analisis gravimetrik haba. Penjerapan metilena biru dan fenol telah dikaji pada kepekatan berbeza (5-200 mg/L), masa sentuh (5 min - 72 jam) dan suhu (30-60 $^{o}$C). Model isoterma, kinetik dan termodinamik digunakan untuk menggambarkan data penjerapan. Luas permukaan tertentu karbon teraktif meningkat dengan suhu pengaktifan dan masa. Penjerapan maksimum terhadap metilena biru adalah 132 mg/g. Karbon teraktif menunjukkan peningkatan penjerapan metilena biru dari 102 mg/g (pukal) ke 107 mg/g. Bagaimanapun, penjerapan fenol menurun daripada 48 ke 39 mg/g selepas pengoksidaan. Data keseimbangan penjerapan metilena biru bersesuaian dengan model Langmuir dan Redlich-Peterson, manakala penjerapan fenol mematuhi isotherma Freundlich. Data kinetik bagi kedua-dua model bahan cemar boleh dijelaskan melalui model pseudo-tertib kedua. Langkah menghad kadar dalam penjerapan metilena biru dan fenol boleh didominasi oleh resapan liang. Perubahan positif entalpi dan entropi menunjukkan bahawa penjerapan metilena biru adalah endotermik dan spontan pada suhu yang tinggi, manakala penjerapan fenol mewakili penjerapan eksotermik dan spontan pada suhu yang rendah. Kesimpulannya, karbon teraktif yang disediakan boleh digunakan sebagai penjerap.

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| | | |
|---|---|---|
| AC | - | Activated Carbon |
| BET | - | Brunauer-Emmett-Teller |
| EC | - | Elemental Carbon |
| FESEM | - | Fourier Transform Infrared Spectroscopy |
| FTIR | - | Field Emission Scanning Electron Microscope |
| LLE | - | Liquid–Liquid extraction |
| MB | - | Methylene Blue |
| MSDS | - | Material Safety Data Sheet |
| PAH | - | Polytetrafluoroethylene |
| PTFE | - | Polycyclic Aromatic Hydrocarbons |
| TGA | - | Thermogravimetric Analysis |
| UV-Vis | - | Ultraviolet-Visible Spectroscopy |
| V | - | Volume of Solution |
| W | - | Mass of Activated Carbon |
| WAC | - | Waste Activated Carbon |
| WTP | - | Waste Tyre Powder |
| WTP-AC | - | Waste Tyre Powder Based Activated Carbon |

# LIST OF SYMBOLS

| | | |
|---|---|---|
| $\lambda$ | - | Wavelength |
| % | - | Percent |
| $\Delta G^o$ | - | Gibbs Energy |
| $\Delta H^o$ | - | Enthalpy |
| $\Delta S^o$ | - | Entropy |
| $^oC$ | - | Degree Celcius |
| $C_o$ | - | Initial Concentration |
| $C_e$ | - | Equilibrium Concentration of Solution |
| $cm^3$ | - | Centimeter Cube |
| $CaCl_2$ | - | Calcium Cloride |
| $CO_2$ | - | Carbon Dioxide |
| CO | - | Carbon Monoxide |
| g | - | Gram |
| h | - | Hour |
| $H_2O$ | - | Water |
| $H_2SO_4$ | - | Sulphuric Acid |
| HF | | Fluoric Acid |
| $HNO_3$ | | Nitric Acid |
| K | | Kelvin |
| Kt | | Kilo Tonne |
| KOH | | Potassium Hydroxide |
| L | | Liter |
| $m^2$ | | Meter Square |
| mg | | Miligram |
| min | | Minute |
| NaCl | | Sodium Chloride |
| NaOH | | Sodium Hydroxide |
| NaOCl | | Sodium Hypochlorite |
| $O_2$ | | Oxygen |
| ppm | | Parts Per Million |

| | | |
|---|---|---|
| $R^2$ | | Coefficient Values |
| $N_2$ | | Nitrogen |
| $ZnCl_2$ | - | Zinc Chloride |

# CHAPTER 1

# INTRODUCTION

## 1.1 Introduction

Waste in the form of solid, liquid and gases can harm the environment and living creatures. The sources of waste are mostly from the industrial-based manufactured goods. Before the wastes are discharge to the environment, proper treatment must be taken into serious consideration. Although the zero disposals target seems nearly impossible, there are many alternatives and ways to minimize the negative impacts of the pollution (Scharff *et al*., 2007).

The tyre manufacturing industry is one of the major industries that contributes in the waste production. Generally, there are two strategies that have been applied for decades in the waste management of scrap tyres, i.e., disposal and recycle. The scrap tyres are disposed through incineration or dumped in landfills. In a regular basis, the scrap tyres are burnt to reduce the available the landfills space. On the other hand, the recycling of scrap tyre is aimed to recover various valuable parts of tyre, reconstruct the tyre, and for the energy production (Torretta *et al*., 2015).

There are a numbers of environmental and public health effects from the open dumping of waste tyres. The dumping areas could become the breeding sites for flies and mosquitoes, consequently will potentially cause human-related diseases such as dengue fever. Problem also arises when the tyres are burnt, forming thick black smoke that quickly spreads and subsequently causes air pollution. The air pollutants from tyre combustion include CO, $CO_2$, $SO_2$, suspended and fine particles (PM2.5), elemental carbon (EC) and polycyclic aromatic hydrocarbons (PAH) (Downard *et al*., 2015), that can lead to the potential increase risk of cancer (Singh *et al*., 2015).

The recycling of scrap tyres could be the sole solution to minimize the environmental pollutions and public health-related problems. The used tyres are not abandoned, but are reconstructed. The end-of-life tyres that can no longer be used in road are undergo materials or energy recovery (Torretta *et al.*, 2015). The tyres are ground into small pieces for materials recovery and for the reconstruction based on the physical characteristics. Normally, the particles are low in weight, high in drainage capacity, relatively compressible and have a low thermal conductivity. In the energy recovery process, the scrap tyres are pyrolysed to produce low-grade fuel. The recycle and recovery processes offer a lot of advantages, mainly on the minimization of the hazardous emissions. Apart from the energy efficiency and product versatility, the energy produced can be integrated within the units in process (Hita *et al.*, 2016). Nevertheless, the pyrolysis of scrap tyre inevitably produces secondary waste that is the residue of carbon tyre powder (pyrolysis waste tyre powder).

Bukit Batu Brickmills is a brick factory located in Johor that employs the pyrolysis of scrap tyres in the production of low-grade fuel for in-house use. The pyrolysis process produces carbon powder as a side product of no commercial value to the company. The particles are easily suspended in air because of the micron-sized and low density. Consequently, this may cause respiratory problems and other health implications to the nearby localities and public. At present, the waste is handled through incineration and landfilling. Yet, it is also necessary to find other means of cheap and sustainable environment to overcome the secondary pollutions associated with the waste management approaches.

Previous studies reported the synthesis of activated carbons from pyrolysis of tyre powder by chemical activation using zinc chloride, potassium hydroxide, sodium hydroxide and calcium chloride for dyes removal (Zaini *et al*., 2014; Li *et al.*, 2010). In general, the findings show a relatively poor performance of the activated carbons for wastewater treatment. Hence, a new activation strategy is worth to be explored to enrich the body of knowledge so as to improve the characteristics and performance of activated carbon. The physical activation of pyrolysis tyre powder has not been regularly reported. Thus, the characteristics of activated carbons and the adsorptive properties by physical activation are still lack in literature.

This study is aimed at evaluating the adsorptive characteristics of physically-activated pyrolysis waste tyre powder for water pollutants removal. The activated carbons were prepared by $CO_2$ activation at different temperatures and activation times. The $CO_2$ activation was used because the preliminary work has revealed that the adsorption performance from this activation is better compared to chemical activation (Shaid *et al.*, 2017). The activated carbons were also modified by hydrofluoric acid and nitric acid, and characterized for physical, chemical and adsorptive properties. The performance of activated carbons was evaluated by methylene blue dye and phenol adsorption from water to study their behaviours towards the positively charged and negatively charged molecules, respectively. The isotherm, kinetics and thermodynamics were studied to ascertain the feasibility of activated carbons for industrial applications.

## 1.2 Problem Statement

The amount of scrap tyres is increasing gradually every year. Some industrial sectors have taken proactive effort and responsibility to manage the abundance of waste tyres through pyrolysis to produce low-grade fuel. However, the process inevitably produces secondary waste, namely pyrolysis waste tyre powder. For environmental and economic sustainability, it is decided to reuse the waste carbon powder instead by converting it into activated carbon. In previous works, chemical activation of the exact same pyrolysis waste carbon powder using potassium hydroxide, calcium chloride, zinc chloride and sodium hydroxide yields activated carbons with low uptake of target water pollutants (Zaini *et al.*, 2014; Baral and Jha 2012). Therefore, the improved adsorptive properties of activated carbons are sought through physical activation using $CO_2$. The effectiveness of activated carbons as adsorbent for water treatment was assessed based upon the adsorption of methylene blue dye and phenol from water.

## 1.3 Objectives

i. To synthesize and characterize activated carbons from pyrolysis waste tyre powder by CO2 activation.

ii. To evaluate the adsorption properties of methylene blue dye and phenol by activated carbons.

iii. To analyze the isotherm, kinetics and thermodynamics from the adsorption data.

## 1.4 Scopes of Study

The pyrolysis waste tyre powder was obtained from Bukit Batu Brickmills, Johor. The activated carbons were prepared $CO_2$ activation at temperatures 900 $^oC$ and 1000 $^oC$ for 2 to 8 hours. The post treatments of activated carbons are de-mineralization and oxidation using hydrofluoric acid ($H_2F_2$) and nitric acid ($HNO_3$), respectively. The activated carbons were characterized based on BET surface area, $pH_{PZC}$, and Fourier transform infrared (FT-IR) spectroscopy, Boehm titration and thermal gravimetric analysis.

Methylene blue dye and phenol were used for the adsorption studies. The adsorption was performed at varying concentrations (5 mg/L-200 mg/L), contact times (5 minutes- 72 hours) and temperatures (30 $^oC$-50 $^oC$). The isotherm, kinetics and thermodynamics models were employed to describe the adsorption data. The isotherm models are Langmuir and Freundflich equations, while the kinetic models are pseudo-first-order and pseudo-second-order equations. The thermodynamics properties, i.e., Gibbs energy ($\Delta G$), enthalpy ($\Delta H$) and entropy ($\Delta S$) were investigated through the effect of temperature on methylene blue dye and phenol adsorption.

## 1.5 Significance of Study

This research was carried out to give further understanding on the contribution of pyrolysis waste tyre powder-based activated carbons by $CO_2$ activation for the removal of methylene blue dye and phenol from water. Also, the problems related to the management of pyrolysis waste tyre powder can be reduced by converting it into activated carbons with sufficient performance.

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