

REMOVAL OF MALACHITE GREEN BY MIXTURE OF TEXTILE SLUDGE
AND SAWDUST CHEMICALLY PRODUCED ACTIVATED CARBON

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ABSTRACT

The growing Malaysian textile industry has resulted in generation of large quantity of textile sludge and its disposal problem has become a critical environmental issue. Textile sludge based activated carbon (TSAC) derived using potassium hydroxide and potassium iodide chemical activation is a promising route for removal of dyes and it can also mitigate the sludge disposal complication. However, the optimum conditions of TSAC preparation and its applicability in the fixed bed adsorption process has yet to be examined. The purpose of this study is to investigate the potential of utilizing TSAC and its pellet (prepared using phosphoric acid impregnated sawdust as a binder) as adsorbents for malachite green (MG) removal in batch and continuous adsorption system. The impregnation ratio was varied (0-2) and the impregnated samples were activated at optimum temperature and time. The physicochemical properties of the TSACs were characterized using Fourier transform infrared spectroscopy, Brunauer-Emmett-Teller (BET) surface area, Scanning Electron Microscope and CHNOS elemental analyzer. The BET surface area and microporosity of TSACs were in the range of 333-1037 m²/g and 27-64%, respectively. The TSACs were capable of removing 77-498 mg/g of MG. The Langmuir model gave the best conformity, suggesting a possible monolayer adsorption. The kinetics experimental data were best fitted to the pseudo-second-order model. The intraparticle diffusion model shows that intraparticle diffusion is not the sole rate-limiting step, while the Boyd model reveals that film diffusion could be the rate controlling step. The adsorption of MG onto TSAC is endothermic and spontaneous by nature. Furthermore, H₃PO₄-impregnated sawdust waste, a potential low-cost binder, yielded TSAC pellets with microporosity, surface area and compressive strength of 16-23%, 668-979 m²/g and 0.4-1.5 MPa, respectively. In addition, the fixed bed adsorption of MG showed that the breakthrough time increased with increasing bed height, and decreasing flow rate and influent dye concentration. The highest bed capacity of 565 mg/g was achieved at influent concentration of 50 mg/L, flow rate of 20 mL/min and bed height of 6 cm (0.954 g). Yoon-Nelson model was the most suitable in describing the dynamic adsorption behavior of the column. This study demonstrated the potential of H₃PO₄-impregnated sawdust waste as a low-cost binder and the applicability of TSAC in MG adsorption for batch and column operations.

ABSTRAK

Industri tekstil Malaysia yang semakin berkembang telah mengakibatkan penjanaan enapcemar tekstil yang banyak dan masalah pelupusannya menjadi isu alam sekitar yang kritikal. Karbon teraktif berasaskan enapcemar tekstil (TSAC) melalui pengaktifan kimia kalium hidroksida dan kalium iodida merupakan laluan berpotensi untuk penyingkiran pencelup dan ia juga boleh mengurangkan kesukaran pelupusan enapcemar. Bagaimanapun, keadaan optimum penyediaan TSAC dan penggunaannya dalam proses penjerapan lapisan tetap masih belum dikaji. Kajian ini bertujuan menyiasat potensi penggunaan TSAC dan peletnya (yang disediakan menggunakan habuk gergaji diisitepu asid fosforik sebagai pengikat) sebagai penjerap untuk penyingkiran malacit hijau (MG) dalam sistem penjerapan berkelompok dan berterusan. Nisbah pengisitepuan dipelbagaikan (0-2) dan sampel diisitepu diaktifkan pada suhu dan masa yang optimum. Sifat-sifat fizik-kimia TSAC dicirikan menggunakan spektroskopi inframerah jelmaan Fourier, luas permukaan Brunauer-Emmett-Teller (BET), mikroskop elektron imbasan dan analisis unsur CHNOS. Luas permukaan BET dan kandungan liang mikro TSACs masing-masing adalah berada dalam lingkungan 333-1037 m²/g dan 27-64 %. TSAC mampu menjerap 77-498 mg/g MG. Model isoterma Langmuir memberikan pematuhan terbaik, menunjukkan bahawa penjerapan bersifat satu lapisan. Data ujikaji kinetik memberikan penyesuaian terbaik dengan model pseudo-tertib-kedua. Model resapan antara zarah menunjukkan bahawa resapan antara zarah bukan langkah kadar-penentu tunggal, manakala model Boyd mendedahkan bahawa resapan filem boleh menjadi langkah kawalan kadar. Penjerapan MG ke atas TSAC bersifat endotermik dan spontan secara semula jadi. Selain itu, sisa habuk gergaji diisitepu H₃PO₄ merupakan pengikat berkost rendah, menghasilkan pelet TSAC dengan liang mikro, luas permukaan dan kekuatan mampatan masing-masing adalah sebanyak 16-23 %, 668-979 m²/g dan 0.4-1.5 MPa. Di samping itu, penjerapan turus tetap MG menunjukkan bahawa masa pembolosan meningkat dengan peningkatan ketinggian turus, dan penurunan kadar aliran dan kepekatan aliran pencelup. Kapasiti turus tertinggi sebanyak 565 mg/g dicapai pada kepekatan aliran 50 mg/L, kadar aliran 20 mL/min dan ketinggian turus 6 cm (0.954 g). Model Yoon-Nelson paling sesuai untuk menggambarkan kelakuan penjerapan dinamik turus. Kajian ini membuktikan potensi buangan habuk gergaji diisitepu H₃PO₄ sebagai pengikat kos rendah dan kebolegunaan TSAC dalam penjerapan MG untuk operasi berkelompok dan turus.

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LIST OF ABBREVIATIONS

AC	-	Activated Carbon
ACF	-	Activated Carbon Fiber
ACP	-	Activated Carbon Pellets
BDST	-	Bed Depth Service Time
BET	-	Brunauer-Emmett-Teller
BOD	-	Biochemical Oxygen Demand
C	-	Carbon
CI	-	Color Index
Cl ₂	-	Chlorine
CMC	-	Carboxymethyl Cellulose
CO	-	Carbon Monoxide
CO ₂	-	Carbon Dioxide
COD	-	Chemical Oxygen Demand
DO	-	Dissolved Oxygen
ETAD	-	Ecological and Toxicological Association of Dyes and Organic Pigments Manufacturers
FTIR	-	Fourier Transform Infrared Spectroscopy
GAC	-	Granular Activated Carbon
H ₂	-	Hydrogen
H ₂ O	-	Water
H ₂ O ₂	-	Hydrogen Peroxide
H ₃ PO ₄	-	Phosphoric Acid
IR	-	Impregnation ratio
K ₂ CO ₃	-	Potassium Carbonate
K ₂ SO ₄	-	Potassium Sulfate
KBr	-	Potassium Bromide
KCl	-	Potassium Chloride
KCOOH	-	Potassium Acetate
KOH	-	Potassium Hydroxide

LD50	-	Lethal Dose at 50% survival
MBBR	-	Moving Bed Biofilm Reactor
MG	-	Malachite green
MTZ	-	Mass Transfer Zone
N ₂	-	Nitrogen
NaCl		Sodium Chloride
NaOH	-	Sodium Hydroxide
O ₂	-	Oxygen
PAC	-	Powdered Activated Carbon
PAIS	-	Phosphoric Acid (H ₃ PO ₄) Impregnated Sawdust
PAN	-	Polyacrylonitrile
RO	-	Reactive Orange
RSM	-	Response Surface Methodology
SEM	-	Scanning Electron Microscope
T&C	-	Textile and Clothing
TS		Textile Sludge
TSAC	-	Textile Sludge based Activated Carbon
UV-Vis	-	Ultraviolet-Visible Spectroscopy Diffuse
ZnCl ₂	-	Zinc Chloride

LIST OF SYMBOLS

%	-	Percent
atm	-	Atmospheric Pressure
cm ²	-	Centimeter Square
cm ³	-	Centimeter Cube
g	-	Gram
GHz	-	Gigahertz
h	-	Hour
J	-	Joules
K	-	Kelvin
kg	-	Kilogram
L	-	Liter
m ²	-	Meter Square
m ³	-	Meter Cube
mg	-	Miligram
min	-	Minute
mL	-	MiliLiter
mmol	-	Milimoles
nm	-	Nanometer
°C	-	Degree Celcius
ppm	-	Parts Per Million
s	-	Second
S _{BET}	-	BET Surface Area
µm	-	Micrometer
W	-	Watt
λ _{max}	-	Maximum Wavelength

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Over the years, water pollution control has been a subject of great interest. Manufacturing industries such as textile, paper and leather are major productions that employ dyes and other chemical agents in their process. It is necessary to separate these contaminants from the wastewater prior to its release. The removal of dyes from effluents can be strenuous because most of them are stable to light, heat and oxidizing agents and biologically non-degradable (Abrahart, 1977). Nevertheless, the accelerating growth of Malaysia's textiles and apparel industry (Idris *et al.*, 2007) has led to a higher demand of dyes and improper handling of dye effluent can result in a serious threat to receiving water bodies.

Dyes are chemical compounds that can associate themselves to surfaces or fabrics to impart colour. Malachite green (MG) is a blueish green cationic dye which is widely used in global textile and clothing industry and aquaculture manufacturing as a biocide, despite of being carcinogenic and genotoxic. This colorant is very soluble in water but is resistant to exposure of light, causing difficult separation from industrial effluents via conventional chemical and biological treatment. By exploiting its high solubility in water, MG can be easily eliminated from wastewater through adsorption process using solid adsorbents or activated carbon (Gouranchat, 2000; Baek *et al.*, 2010). Thus, more research and studies should be focused on the removal of MG from water bodies to maintain the water quality and health of living beings.

There are many physical and chemical treatment technologies that can be applied for dye removal wastewater, for instance ion exchange, flocculation and coagulation, membrane filtration, oxidation and electrolysis. Adsorption process an effective and inexpensive method as it utilizes low cost adsorbents and no sludges are

produced (Hena, 2010). In other words, adsorption is an excellent choice for dye removal in terms of initial cost, simplicity of design, ease of operation and insensitivity to toxic substances.

Textile sludge (TS) is a potential adsorbent material for the production of activated carbon in dye removal owing to its high carbon content, inert towards toxic substance and low initial cost (Kacan, 2016; Tang and Zaini, 2017). In practice, the preparation of activated carbon can be carried out in two methods which are chemical activation and physical activation. In chemical activation, the raw material is initially subjected to impregnation with activating agents like KOH, NaOH or K_2CO_3 before thermal activation. Chemical activation prevails over physical activation due to its higher surface area and pore development, short activation time, low activation temperature and higher yield (Katesa *et al.*, 2010; Patil and Kulkarni, 2012). The textile sludge based activated carbon (TSAC) prepared via KI+KOH activation has high surface area and MG adsorption capacity of $481 \text{ m}^2/\text{g}$ and 167 mg/g , compared to KI activated TSAC ($123 \text{ m}^2/\text{g}$ and 37 mg/g) and KOH activated TSAC ($301 \text{ m}^2/\text{g}$ and 108 mg/g) (Tang and Zaini, 2017). Composite KI+KOH activating agent has shown promising outcome and further study should be done.

The conversion of powdered activated carbon (PAC) into different shapes (cylindrical, pellets, spherical, ring, etc.) is crucial in many industrial applications like adsorption and catalyst support as they possess good compressive strength and high volumetric adsorption capacity and ease transportation problem. Activated carbon pellet (ACP) can be fabricated by extruding PAC together with a binder, in order to hold the carbon particles in a stable compressed state. The binder ought to (a) produce ACP with good compression strength with lowest binder/AC ratio to maximize the quantity of AC, (b) retain or enhance the specific surface area of AC (does not trigger pore blocking). H_3PO_4 -impregnated waste sawdust (PAIS) is a promising and cost-effective binder as the PAC produced has good surface area and mechanical strength (Li *et al.*, 2014a; Li *et al.*, 2014b).

Continuous fixed bed column operations are vastly implemented in various industrial and pollution control processes like treatment of dye wastewater via a fixed

bed of carbon adsorbent, gas or liquid catalytic reaction by structured catalyst bed and separation of ions by a packed ion-exchange resin bed. Column adsorption study is crucial as involves the investigation of other operational complications including irregular flow rate in column, underperformance of adsorbents in continuous system, recycling and regeneration of spent adsorbent (Gopal *et al.*, 2016). Unlike batch adsorption, fixed bed column adsorption may not run under equilibrium state due to insufficient time to achieve equilibrium adsorption (Gopal *et al.*, 2016, Yagub *et al.*, 2015). Precise modelling and simulation of the dynamic pattern of fixed bed systems is indispensable in designing an optimal column sorption process. Over the years, a few models were formulated based on fundamental mass transport mechanism (surface, pore and film diffusion) for the prediction of different adsorption systems. In order to save time and cost, the pilot plant testing can be done by applying shortcut models - bed service time (BDST) model (Hutchins, 1973) and mass transfer zone (MTZ) model (Michaels, 1952) which provide straightforward method and fast estimation of adsorber performance (Markovska *et al.*, 2001). The bed service time (BDST) model has been effectively employed to report and anticipate the column adsorption of dye using various adsorbents (Markovska *et al.*, 2001, Gopal *et al.*, 2016, Yagub *et al.*, 2015). In this work, four mathematical models - Bohart-Adams, Thomas, Yoon Nelson and Clark models are used to construct the column breakthrough curves of adsorbent and explain the mechanism of fixed bed column adsorption.

Response surface methodology (RSM) is a group of mathematical and statistical approaches which is extensively used in the industrial field to develop, improve and optimize processes (Myers *et al.*, 2016, Xue *et al.*, 2016). The empirical model building involves the measurement of output response (output variable) or performance measure affected by several potential independent variables (input variables) on a continuous scale. The researcher or engineer can control and manipulate the input variables of an experiment or test. More than one response variables are usually incorporated in the majority of industrial RSM applications (Myers *et al.*, 2016). A series of tests or runs are carried out using different sets of input variables to determine the output response and identify the rationale behind its changes (Raissi and Farsani, 2009).

In short, the growing menace of water pollution has always been the centre of concern. With the discovery of TSAC, the environmental pollution can be cut down twice the scale as this will not only assist greatly in water treatment technology, but also reduce the need of landfill and incineration and thereby averting both water and land pollution. Moreover, H₃PO₄-impregnated sawdust waste is a potential low-cost binder, which also contributes to the conversion of waste into reusable material. This study is expected to contribute further understanding on dye wastewater treatment using TSAC in industrial scale.

1.2 Problem Statement

Synthetic textile fiber is a popular industry in Malaysia. There are 662 licensed and 1000 small scale textile and apparel factories in 2012 (Malaysian Investment Development Authority (MIDA), 2012). Johor (28.6 %) was found to be the highest contributing source of the textile industrial wastewater pollution, followed by Penang (28.2 %) and Selangor (15.6 %) (Muyibi *et al.*, 2008). Malaysian textile industries treat its own wastewater and the treated effluent should comply with the discharge limit before emission. This would in turn generate large amount of sludge which requires further handling and disposal (Pang and Abdullah, 2013). The Malaysia Department of Environment classifies sludge produced from wastewater treatment plants as scheduled waste and should be disposed of under a prescribed premise only (Hanum *et al.*, 2019). The total quantity of scheduled waste generated by textile industry surged from 744 tons (2007) to 1559 tons (2009) (Pang and Abdullah, 2013) and the estimated total cost of managing sewage sludge in Malaysia is RM1.38 billion per year (Kadir and Mohd, 1998). The generation of large amount of sludge and its disposal problem has become a critical environmental issue. Thus, the conversion of textile sludge into activated carbon can reduce the sludge disposal problem and at the same time preserve the environment. The carbon content of textile sludge is in the range of 23-42.3 % (Kacan, 2016; Sohaimi and Ngadi, 2016; Tang and Zaini, 2017), indicating it can be used as a precursor for the preparation of AC.

Adsorption using activated carbon is an effective physical treatment for dye wastewater. Studies have reported the generation of TSACs through different methods, but their surface area were only 195-481 m²/g. In one of the studies, the composite KI+KOH activating agent had successfully converted TS into TSAC. The TSACs activated at 0.7:0.3:1 (KI:KOH:TS), 500 °C and 1 h possessed surface area, microporosity and MG adsorption capacity of up to 481 m²/g, 47 % and 167 mg/g, respectively (Tang and Zaini, 2017). Besides that, Kacan (2016) also obtained TSAC with a surface area of 336 m²/g at activation condition of 0.5:1 (KOH:TS), 700 °C and 0.75 h, whereas Sohaimi and co-workers (2017) produced TS biochar with a surface area of only 195 m²/g at carbonization condition of 700 °C and 1 h (without activating agent). Wong and colleagues (2018) chemically activated the TS using H₂SO₄ at condition of 1:1 (H₂SO₄:TS), 650 °C and 0.5 h. The H₂SO₄-TSAC has a surface area and microporosity of 222 m²/g and 63 %. However, these findings are only satisfactory as compared to commercial AC with surface area 820 m²/g and MG adsorption capacity of 222 mg/g (Malik *et al.*, 2007). Moreover, the TSACs also exist in a fluffy powdery form due to its porous nature, resulting in prolonged settling time and cumbersome adsorbent handling. These problems may complicate the scaling up of the adsorption process using TSAC for industrial applications. Up to date, no studies were performed using TSAC in fixed bed column adsorption of MG, most likely due to its powdery form and low surface area.

In the previous study, KI+KOH activation was carried out at fixed temperature and time of 500 °C and 1 h, with only three impregnation ratios of 1:1:1, 0.7:0.3:1 and 0.5:0.5:1 (Tang and Zaini, 2017). Therefore, in this study, the TSAC preparation conditions via KI+KOH activation including activation temperature, time and impregnation ratio (KI:KOH:TS) was evaluated to produce the highest achievable surface area of TSAC. Furthermore, the TSAC was also converted into pellet form using the PAIS binder. Although PAIS binder has been used to convert commercial AC powder and peanut shell char into pellets (Li *et al.*, 2014; Li *et al.*, 2014 b), no studies have attempted to use TSAC powder. Since the base materials used were different, the pellets produced also have different physicochemical properties. The newly improved TSACs and its derivatives were also characterized for their physicochemical properties and adsorption performance. In addition, the fixed bed

adsorption column for the removal of MG was also performed and the operating parameters such as bed height, flow rate and influent dye concentration were investigated.

1.3 Objective of the Study

The objectives of this research are:

- i. To evaluate the effects of activation temperature, time and impregnation ratio for the production of textile sludge based activated carbon (TSAC) via KI+KOH activation and to characterize the resultant products.
- ii. To establish the adsorption isotherms, kinetics and thermodynamics of malachite green (MG) adsorption onto TSAC.
- iii. To evaluate the adsorptive and mechanical properties of phosphoric acid (H_3PO_4)-impregnated sawdust as a binder for TSAC pellets.
- iv. To investigate the bed height, influent flow rate and initial dye concentration for fixed-bed column adsorption of MG

1.4 Scope of the Study

The scope of this study is divided into four parts. The first scope of study covers the preparation and characterization of textile sludge based activated carbon (TSAC) via KI+KOH activation. The one-factor-at-a-time method was employed to study the effect of activation temperature, time and impregnation ratio on the TSAC surface area. Initially, the activation temperature (400-800 °C) and time (0.5-2 h) were varied and the adsorption capacity of methylene blue (MB), also known as MB number was used as a rapid assessment of the TSAC surface area and overall adsorptive capacity because the specific adsorbate has yet to be selected at the beginning. As the MB adsorption capacity increases, the surface area also increases because it correlates with surface area (Joshi, 2015). This method has been widely used for the

characterization of activated carbon (Abdullah *et al.*, 2001; Nunes and Guerreiro, 2011; Raposo *et al.*, 2009; Joshi, 2015; Adibfar *et al.*, 2014). Response surface methodology (RSM) was performed additionally to compare with the optimum experimental activation temperature and time. The effect of impregnation ratios (0-2) were later evaluated at the optimum activation temperature and time. The physicochemical properties of synthesized TSACs were determined using BET, FTIR, SEM, Boehm titration, pH_{pzc} and CHNOS analysis. The RSM was not performed concurrently for all three variables because the MB number was used as the overall screening indicator for activation temperature and time only. Moreover, the previous study showed that the best impregnation ratio was 0.7:0.3:1 (KI:KOH:TS), but the activation temperature and time were not investigated (Tang and Zaini *et al.*, 2017).

The second part of the scope of study is establishing the adsorption isotherms, kinetics and thermodynamics of malachite green (MG) onto TSAC. MG dye was used as adsorbates and the batch adsorption was performed to evaluate the performance of activated carbon. The equilibrium isotherm was conducted at different initial concentration (20-1000 mg/L) with contact time of 72 h, whereas three concentrations and at varying time intervals were applied for kinetic studies. Isotherm models consist of Freundlich, Langmuir, Temkin and Redlich-Peterson whereas kinetics models include pseudo-first-order, pseudo-second-order, Boyd and intraparticle diffusion. Thermodynamics study was carried out at temperatures between 30 °C and 60 °C. The thermodynamics model was used to evaluate the parameters of Gibbs free energy (ΔG°), enthalpy change (ΔH°) and entropy change (ΔS°).

The third part of the scope of study includes transforming the powdered TSAC into cylindrical activated carbon pellets by using the H_3PO_4 -impregnated sawdust waste (PAIS) binder. The mass ratio of H_3PO_4 : sawdust: TSAC was fixed at 1.7:1:0.4. The mixture will be manually extruded into a cylindrical mould and pyrolyzed at 560 °C for different time (1 – 3 h) (Li *et al.*, 2014a). The TSAC pellets produced were tested for their adsorptive and mechanical properties such as compressive strength, bulk density and ability to maintain its shape in water. The TSAC pellets were used to perform batch adsorption equilibrium experiments.

In order to achieve the fourth scope of study, the TSAC granules were used for the fixed bed adsorption column study. The original powdered TSAC was not applicable in the fixed bed column as the wool could not hold it in place, whereas the size of the pellets ($D = 8.5$ mm, $H = 6.3$ mm) was too large resulting in a less favourable column packing and incomplete breakthrough curves (BTC). BTC is the plot of normalized concentration (C_t/C_0) as a function of time, where C_t is the effluent concentration and C_0 is the influent concentration (mg/L) (Han *et al.* 2009). A complete sigmoidal or S-shaped BTC can be obtained when the $C_t/C_0 \approx 1$, indicating the exhaustion of the adsorbent bed (Afroze *et al.*, 2016). A complete BTC is crucial in the analysis of the operational and dynamic behaviour of an adsorption fixed-bed column (Han *et al.* 2009). Therefore, the large TSAC pellets were crushed into tiny granules with average particle size of 0.5 mm. This is because some adsorption column studies reported that complete BTCs were attained using pine cone AC (0.5 mm) (Samarghandi *et al.*, 2014) and sludge adsorbent (0.6 mm) (GEPEA Université de Nantes, 2009), whereas larger sized granular zeolite (1.6 mm) (Abu-Lail *et al.*, 2010) and bagasse activated carbon (1.5 mm) (Karunarathne and Amarasinghe, 2013) produced incomplete BTCs. The effect of bed height (2-6 cm), influent flow rate (15-30 mL/min) and initial dye concentration (20-80 mg/L) on the fixed-bed column adsorption of MG using TSAC granules were investigated. The breakthrough curve analysis was also performed. The adsorption column data were fitted into four column models namely Thomas, Yoon-Nelson, Bohart-Adams and Clark model.

1.5 Significance of Study

The growing menace of water pollution has always been the centre of concern. With the discovery of high surface area TSAC, the environmental pollution can be cut down twice the scale as this will not only assist greatly in water treatment technology, but also reduce the use of landfill and sludge incineration and thereby averting both water and land pollution. This study is imperative as it investigates the best conditions to prepare TSAC via KI+KOH activation and explores the improvements of the physical and mechanical properties of TSAC. Besides, H_3PO_4 -impregnated sawdust

waste is a potential binder, which also contributes to the conversion of waste into reusable material. Moreover, the fixed bed adsorption of MG is also being investigated, whereby the bed height, influent flow rate and initial dye concentration for fixed-bed column adsorption of MG using TSAC were evaluated. This study is expected to contribute further understanding on dye wastewater treatment using TSAC.

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