



UNIVERSITI PUTRA MALAYSIA

**ADSORPTION OF BASIC AND ACID DYES USING PALM KERNEL
SHELL-BASED ACTIVATED CARBON**

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**ADSORPTION OF BASIC AND ACID DYES USING PALM KERNEL
SHELL – BASED ACTIVATED CARBON**

By

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ADSORPTION OF BASIC AND ACID DYES USING PALM KERNEL SHELL – BASED ACTIVATED CARBON

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A series of batch laboratory studies were conducted in order to investigate the practicability of palm kernel shell (PKS)-based activated carbon for removal of basic dye, Basic Blue 9 (BB 9) and acid dye, Acid Orange 52 (AO 52) from their aqueous solutions. Three different particle sizes of PKS were used and categorized as PKS – S, PKS – M and PKS – L. The adsorption capacities of the PKS-based activated carbons were compared with those of commercial grade coal-based activated carbons at the same conditions. All batch experiments were carried out at a constant temperature of 28°C ($\pm 2^\circ\text{C}$) using incubator shaker that operated at 150 rpm.

Batch equilibrium study shows that Adsorption of BB 9 and AO 52 were highly pH dependent. Removal of BB 9 increased with pH with maximum removal observed at pH 7.0, and decreased thereafter with further increase in the initial pH. However, pH effect on AO 52 removal shows that AO 52 removal decreased with an increase in initial pH with the optimum initial was observed at 3.5.



Batch equilibrium data also had good agreement with the Langmuir, Freundlich and Redlich-Peterson isotherm models with correlation coefficients > 0.9 . Overall, the Redlich-Peterson isotherm showed the best fit for all adsorbents under investigation in terms of correlation coefficient as well as error analysis of the results. For all the systems in this study, the analysis of isotherm shape factor showed that adsorption was favorable.

For the adsorbents under investigation, PKS-S has the highest adsorption capacity followed by PKS-M. PKS-L and commercial coal based pellet form have almost equally-balanced adsorption capacity. Of all the adsorbents, commercial coal-based in powder form exhibits the lowest adsorption capacity. Obtained results revealed that PKS based activated carbon is a highly potential alternative adsorbent for treatment of dye-containing wastewater. The maximum capacity of the adsorbents for BB 9 were 333.33 mg/g, 322.58 mg/g and 212.77 mg/g for PKS – S, PKS – M and PKS – L, respectively, while for powder and pellet commercial grade coal based, the capacities were 204.08 mg/g and 217.39 mg/g, respectively. On the other hand, the maximum capacities of the same adsorbents for AO 52 were 344.83 mg/g, 333.33 mg/g, 263.16 mg/g, 238.09 mg/g and 322.58 mg/g, respectively. Adsorption capacities of the same adsorbents were found to be higher for adsorption of AO 52 compared to those of BB 9 due to the smaller molecular size of the former.

Batch kinetic studies were also performed to investigate the rate limiting of the adsorption process. Results obtained revealed that the adsorption of both BB 9 and AO 52 was rapid at the beginning, but approached equilibrium slowly.



Experimental data can be modeled using pseudo-second-order kinetic model as first order kinetic model does not represent the whole range of adsorption process. Other than that, intraparticle diffusion was found to be prominent at a certain stage of adsorption but it would not be the only limiting step that controlled the adsorption dynamic.

Kinetic data also showed that the adsorption rates were a function of initial adsorbate concentration, adsorbent particle size and adsorbent mass. For all systems under consideration, the values of k_2 increased significantly as adsorbent dose increases. Nevertheless, the values of k_2 were inversely proportional to the initial adsorbate concentration and adsorbent particle size. On the other hand, values of k_p were found to be directly proportional to the initial adsorbate concentration but decreased gradually as adsorbent particle size and adsorbent dose increases.



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**PENJERAPAN PEWARNA JENIS BES DAN ASID PADA KARBON
TERAKTIF DARIPADA TEMPURUNG KELAPA SAWIT**

Oleh

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Satu siri ujikaji telah dijalankan di makmal untuk mengkaji kesesuaian penggunaan karbon teraktif daripada tempurung kelapa sawit (PKS) untuk menjerap bahan pewarna jenis bes, Basic Blue 9 (BB 9) dan jenis asid, Acid Orange 52 (AO 52) daripada larutan berair. PKS yang digunakan terdapat dalam tiga saiz dan dikategorikan sebagai PKS – S, PKS – M dan PKS – L. Dua jenis karbon teraktif komersial berasaskan kayu telah dikaji melalui eksperimen yang sama pada kondisi yang serupa bagi menaksir kapasiti penjerapan setiap PKS berbanding karbon teraktif komersial. Semua ujikaji dijalankan pada suhu tetap iaitu 28°C ($\pm 2^\circ\text{C}$) menggunakan penggoncang inkubator yang beroperasi pada kelajuan 150 rpm.

Penjerapan BB 9 didapati sangat dipengaruhi oleh pH larutan di mana penjerapan meningkat bila pH meningkat sehingga pH mencapai tahap optimum 7.0. selepas pH optimum, penjerapan menurun bila pH semakin meningkat. AO 52 pula memerlukan

keadaan larutan yang berasid untuk mencapai tahap penjerapan yang tinggi di mana penjerapan maksimum dicatatkan pada pH 3.5.

Data keseimbangan didapati sesuai dengan model Langmuir, Freundlich dan Redlich-Peterson dengan nilai $R^2 > 0.9$. Secara keseluruhannya, model isoterma Redlich-Peterson adalah paling sesuai untuk semua system yang dikaji berdasarkan nilai R^2 yang tinggi serta nilai sisihan yang rendah. Antara semua sistem yang dikaji, didapati PKS – S mempunyai kapasiti penjerapan yang paling tinggi untuk kedua-dua pewarna diikuti oleh PKS – M. PKS – L mencatatkan kapasiti penjerapan yang hampir sama dengan karbon teraktif komersial jenis pellet manakala karbon teraktif komersial jenis debu mempunyai kapasiti penjerapan yang paling rendah. Kapasiti maksimum bagi setiap karbon teraktif dalam penjerapan BB 9 ialah masing-masing 333.33 mg/g, 322.58 mg/g dan 212.77 mg/g untuk PKS – S, PKS – M dan PKS – L manakala karbon teraktif komersial jenis debu dan pellet masing-masing mencatatkan nilai 204.08 mg/g dan 217.39 mg/g. Sementara itu, kapasiti penjerapan maksimum terhadap AO 52 bagi setiap penjerap yang dinyatakan mengikut urutan di atas ialah 344.83 mg/g, 333.33 mg/g, 263.16 mg/g, 238.09 mg/g dan 322.58 mg/g.

Kajian kinetik juga dijalankan untuk mengkaji kadar penjerapan dan meneliti faktor-faktor yang menentukan kadar tersebut. Tiga jenis model telah diuji iaitu kinetik order pertama, kedua dan intrapartikel. Data kinetik didapati sesuai dengan model kedua dan model intrapartikel. Sementara itu, faktor kepekatan awal pewarna, saiz serta dos karbon teraktif yang digunakan didapati memberi kesan yang signifikan ke atas kadar penjerapan.

Data kinetik juga menunjukkan bahawa nilai k_2 bagi semua sistem yang dikaji meningkat bila dos karbon teraktif meningkat tetapi berkadar songsang dengan kepekatan awal pewarna serta saiz karbon teraktif. Sementara itu, nilai k_p didapati berkadaran secara terus dengan kepekatan awal pewarna tetapi peningkatan pada saiz dan dos karbon teraktif menyebabkan nilai k_p berkurangan.

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LIST OF NOTATIONS / SYMBOLS

Notations / symbols

\AA	Length, = 10^{-10} m	
α_L	Energy of adsorption	$\text{dm}^3 \cdot \text{mg}^{-1}$
b_j	Constant in Equation 2.7	
β	Heterogeneity factor	
C_o	Initial concentration	$\text{mg} \cdot \text{L}^{-1}$
C_e	Bulk phase concentration	$\text{mg} \cdot \text{L}^{-1}$
C_t	Concentration of the solution at time t	$\text{mg} \cdot \text{L}^{-1}$
D_{meso}	Average mesopore diameter	\AA
D_{micro}	Average micropore diameter	\AA
K_F	Freundlich adsorption capacity	mg/g
K_j	Redlich-Peterson constant	$\text{L} \cdot \text{g}^{-1}$
K_L	Langmuir constant	$\text{L} \cdot \text{g}^{-1}$
m	Adsorbent mass	g
n	Surface heterogeneity	
R_L	Separation factor	
t	time	minute
q_e	Solid phase concentration at equilibrium	mg/g
q_t	Solid phase concentration at time t	mg/g
S_{BET}	BET specific surface of activated carbon	m^2/g
V	Volume of solution	mL
V_{meso}	Mesopore volume	cm^3/g
V_{micro}	Micropore volume	cm^3/g
V_{total}	Total pore volume	cm^3/g



AC	Activated carbon
AO 52	Acid Orange 52
BET	Brunnauer-Emmet-Teller
BB 9	Basic Blue 9
IUPAC	International Union of Pure and Applied Chemistry
PKS	Palm Kernel Shell



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CHAPTER 1

INTRODUCTION

1.1 Background

Around the world, chemical industries and manufacturing plants grow at an alarming rate in almost all countries, which released a broad range of harmful industrial wastes to the environment. Textile industries are among the areas that expanded rapidly to fulfil the increasing demand of world population. This industry consumes large volumes of water and chemicals for wet processing and due to inefficiencies in dyeing techniques, some of the dyes maybe discharged into waterways and if reactive dyes are used the dye loss can be up to 50% (Mc Mullan et al., 2001). Considering both volumes discharged and effluent composition, the wastewater generated by the textile industry is rated as the most polluting among all the industrial sectors (Reid and Green, 1996). Wastewater from the textile industry had also been declared as one of the major sources of wastewater in ASEAN countries in the year 2000 (Setamanit et al., 2002).

The effluent discharged from the textile industry mainly contains dyes (organic and inorganic), wax, grease, heavy metals, surfactant and suspended solids (Ahmad et al., 2002). Dye-containing wastewater has been reported to be difficult to treat using conventional wastewater treatment due to its recalcitrant nature. Due to their chemical structure, most dyes are resistant to fading on exposure to light, water and many chemicals (Robinson et al., 2001) and the structural complexity as well as synthetic



origin make it very difficult to be decolourised and decomposed biologically.

Wastewater containing dyes must be properly treated before being discharged to the environment because even when released in small concentrations, it will impart colour that will consequently reduce the aesthetical value to the receiving water. It will also impede light penetration to the water body. These dyes were also found to have a potential chronic health hazard to human beings and other adverse impacts such as toxicity to aquatic life.

1.2 Environmental Regulations For Wastewater Generated From Textile Industry

The Malaysian government gazetted The Environmental Quality Act 1974 and Environmental Quality (Prescribed Premises) (Scheduled Wastes Treatment and Disposal Facilities) Regulations 1979 to inhibit indiscriminate discharge of effluent from textiles industries into the watercourse. Other than that, the effluent from textile industries has to be treated to meet regulatory standards as specified in Environmental Quality (Sewage and Industrial Effluents) Regulations 1979 whilst all factories from textile industries have also to comply with Environmental Quality (Scheduled Wastes) Regulations 1989.



1.3 Treatment Method

Due to more stringent legislation, several studies have been performed to find an effective and economical way for treatment of dye-containing wastewater. Those studies can be categorised into three; physical, chemical and biological methods as shown in **Table 1.1.**

Table 1.1 Advantages and disadvantages of the current methods of dye removal from industrial effluents (Compiled and modified from Robinson et al., 2001)

Treatment methods	Advantages	Disadvantages
Fentons reagent	Effective decolourisation of both soluble and insoluble dyes	Sludge generation
Ozonation	Applied in gaseous state: no alteration of volume	Short half-life (20 minutes)
Photochemical	No sludge production	Formation of by-product
NaOCl	Initiates and accelerates azo-bond cleavage	Release of aromatic amines
Cucurbituril	Good sorption capacity for various dyes	High cost
Electrochemical destruction	Breakdown compounds are non-hazardous	High cost of electricity
Activated carbon	Good removal of wide variety of dyes	Very expensive
Peat	Good adsorbent due to cellular structure	Specific surface areas for adsorption are lower than activated carbon
Wood chips	Good sorption capacity for acid dyes	Requires long retention times
Silica gel	Effective for basic dye removal	Side reactions prevent commercial application

