



**A Study on Rice Husk Ash-Based Activated Carbon in
Different Forms Through Adsorption Isotherm**

by

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LIST OF ABBREVIATIONS

AC	Activated carbon
ACP	Activated carbon pellet
ACS	Activated carbon sphere
Ag	Silver
ARHA	Activated rice husk ash
ATR	Attenuated total reflectance
BET	Brannur-Emmet Teller
BJH	Barret-Joyner Halenda
bRHA	Black rice husk ash
CAA	Calcium alginate aerogel
CaCl ₂	Calcium chloride
CaCO ₃	Calcium carbonate
CBRHA	Controlled burn rice husk ash
Cd	Cadmium
CO ₂	Carbon dioxide
DBP	Disinfection by product
DOC	Dissolved organic carbon
DTG	Differential thermogravimetric
EDX	Energy dispersive X-ray
EPS	Expanded polystyrene
FONP-PAC	Ferroferric oxide nanoparticles-powdered activated carbon
FTIR	Fourier Transform Infrared
GAC	Granulated activated carbon
H ₂ SO ₄	Sulfuric acid
H ₃ PO ₄	Phosphoric acid
HF	Hydrofluoric acid
HMT	Hexamethylenetetramine
KOH	Potassium hydroxide
MAFI	Ministry of agriculture and food industries
MB	Methylene blue
MHCC	Magnetic hollow carbon composite
MP	Mangosteen peel
MPAC	Mangosteen peel-activated carbon

Na ₂ SiO ₃	Sodium silicate
NaHCO ₃	Sodium bicarbonate
NaOH	Sodium hydroxide
OBRHA	Open-burn rice husk ash
PAC	Porous activated carbon
PAC	Powdered activated carbon
PAIS	Phosphoric acid impregnated sawdust
Pb	Lead
PSAC	Palm shell activated carbon
PVA	Polyvinyl acetate
RH	Rice husk
RHA	Rice husk ash
RR	Remazol red
RSSL	Rice self-sufficiency level
SA	Sodium Alginate
SEM	Scanning electron microscope
SiO ₂	Silicone dioxide
STP	Standard temperature and pressure
TGA	Thermogravimetric analysis
TOC	Total organic carbon
TSS	Total suspended solid
VOC	Volatile organic compound
wRHA	White rice husk ash
XRD	X-ray Diffraction
ZnCl ₂	Zinc chloride

LIST OF SYMBOLS

μm	micrometer
\AA	Armstrong
c	Armstrong
c	BET constant
c_e	Concentration at equilibrium
c_t	Concentration at time
g/mol	Gram per mol
K_F	Freundlich constant
K_L (L/mg)	Langmuir constant
mm	millimeter
n	Freundlich exponent
nm	Nanometer
$^{\circ}\text{C}$	Degree Celsius
P	Equilibrium pressure of adsorbate
pH	Percent of hydrogen
P_o	Saturation pressure of adsorbate
q_{max} (mg/g)	adsorbent's maximum adsorption capacity
R^2	coefficient of determination
R_L	shape of the isotherm
rpm	Rotation per minute
S_{BET}	BET surface area
wt/wt	Weight percentage
λ	Wavelength
v	Adsorb gas quantity

Kajian Abu Sekam Padi sebagai Karbon Teraktif dalam Bentuk Berbeza Melalui Sifat Penjerapan Isoterma

ABSTRAK

Abu sekam padi (RHA) adalah hasil sampingan kilang padi semasa proses pengeringan beras, dah dihasilkan setiap tahun di Malaysia dengan banyak. Tanpa pengurusan yang betul akan menyebabkan isu alam sekitar yang teruk termasuklah pencemaran air dan udara. Oleh itu, proses pengaktifan kimia digunakan untuk menukar RHA kepada karbon teraktifkan berliang (PAC) yang berpotensi untuk digunakan dalam rawatan sisa air. PAC dihasilkan melalui pengaktifan kimia menggunakan pelbagai jenis dan kepekatan (0.5 M, 1.0 M, 1.5 M dan 2.0 M) agen pengaktifan (natrium hidroksida (NaOH), kalium hidroksida (KOH), zink klorida ($ZnCl_2$), dan asid sitrik) pada suhu pemprosesan yang lebih rendah iaitu 80 °C selama 2 jam. Liang karbon teraktif adalah penting dalam menentukan sifat penjerapan pewarna merah remazol (RR). Keputusan menunjukkan 1.0 M Na-PAC, yang merupakan RHA dirawat dengan 1.0 M NaOH mempunyai prestasi tinggi dalam kedua-dua sifat fizikal dan penjerapan. Luas permukaan BET telah ditambah baik daripada 54.14 m²/g kepada 305.27 m²/g dan saiz liang dari 47 nm hingga 5 nm. Isoterma penjerapan Na-PAC sesuai dengan Jenis IV, yang dikelaskan sebagai mesoporos dan disokong oleh imej SEM yang menunjukkan kehadiran struktur berliang. Keputusan dari XRD menunjukkan kehadiran struktur amorfus dan kristal yang dipengaruhi oleh struktur silika seperti yang diasaskan dalam keputusan FTIR. Berdasarkan ini, tiga bentuk Na-PAC yang berbeza telah dikaji: serbuk, manik makro dan belon makro. Manik makro disediakan melalui pengelan natrium alginat, manakala belon makro menggunakan kaedah penggulingan, menggunakan EPS sebagai templat untuk menghasilkan struktur sfera berongga. Kajian penjerapan menggunakan spektrofotometer UV menunjukkan bahawa Na-PAC dalam bentuk serbuk mempunyai kecekapan penyingkiran pewarna RR yang lebih tinggi diikuti oleh manik makro dan belon makro dengan penyingkiran pewarna 100 % pada minit 20, 80 dan 120. Data keseimbangan penjerapan belon makro dan manik makro paling sesuai dengan padanan isoterma penjerapan Langmuir berbanding isoterma penjerapan Freundlich. Ia menunjukkan bahawa proses penjerapan berlaku dalam monolayer di permukaan PAC. Bentuk serbuk dan manik makro menunjukkan kecekapan penyingkiran pewarna RR yang lebih baik, tetapi kedua-duanya sukar diurus dan dipisahkan pada akhir proses dalam aplikasi sebenar. Oleh itu, sifat terapung sendiri dan keupayaan penjerapan belon makro memberikan kelebihan sebagai sampel yang dioptimumkan untuk mengkaji kesan pelbagai jenis pengikat. Empat jenis pengikat telah digunakan (epoksi resin, natrium alginat, silikon resin dan grout) untuk menghasilkan belon makro. Keputusan menunjukkan bahawa natrium alginat belon makro mempunyai penjerapan yang lebih baik dengan lebih 80 % penyingkiran pewarna dalam 120 minit berbanding tiga pengikat yang lain. Hal ini kerana natrium alginat yang digunakan tidak menghalang tapak penjerapan di atas permukaan Na-PAC, manakala Na-PAC di dalam grout tersebar di dalam pewarna, Na-PAC dalam silikon dan epoksi menghalang proses penjerapan. Kesimpulannya, 1.0 Na-PAC telah menghasilkan liang karbon teraktif yang mampu untuk mengeluarkan pewarna RR dalam masa yang ditetapkan dalam tiga bentuk untuk proses penjerapan. Jenis pengikat juga

mempengaruhi kebolehan penyerapan, menjadikan natrium alginat pengikat yang sesuai tanpa menghalang proses penyerapan.

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A Study on Rice Husk Ash-Based Activated Carbon in Different Forms Through Adsorption Isotherm

ABSTRACT

Rice husk ash (RHA) is a by-product generated by rice mills during the rice drying process, and it is abundantly produced in Malaysia every year. Without proper management can result in severe environmental issues, including water and air pollution. Therefore, in this study, chemical activation process was utilised to convert RHA into porous activated carbon (PAC) which has potential to be used in wastewater treatment. This PAC was produced by chemical activation using different types and concentration (0.5 M, 1.0 M, 1.5 M, and 2.0 M) of activating agents (sodium hydroxide (NaOH), potassium hydroxide (KOH), zinc chloride (ZnCl₂) and citric acid) at a lower processing temperature of 80 °C for 2 hours. The porosity of the activated carbon is crucial in determining the remazol red (RR) dye adsorption properties. Results indicate that 1.0 M Na-PAC, which is RHA treated with 1.0 M NaOH, demonstrates high performance in both physical and adsorption properties. The BET surface area improved from 54.14 m²/g to 305.27 m²/g, and the pore size from 47 nm to 5 nm. The adsorption isotherm of Na-PAC is fit to Type IV, which is classified as mesoporous were also supported by the SEM image showing presence of the porous structure. Results from the XRD shows the presence of amorphous and crystalline structure which were affected by the silica structure as founded in the FTIR results. Based on this, three different forms of Na-PAC were studied: powder, macrobeads and macroballoons. The macrobeads were prepared by gelation of sodium alginate, while macroballoons used the rolling method, using EPS as a template to produce hollow sphere structure. The adsorption study using UV-spectrophotometer reveals that Na-PAC in powder form possesses a high efficiency of RR dye removal followed by macrobeads and macroballoons with 100 % dye removal at 20, 80 and 120 minutes, respectively. The adsorption equilibrium data of macroballoons and macrobeads were best fitted with the Langmuir adsorption isotherm compared to the Freundlich adsorption isotherm. It shows that the adsorption process occurs in monolayer at the surface of the Na-PAC. Powder and macrobeads forms show good removal efficiency of RR dye, however, both are difficult to manage and separate at the end of the process in the actual application. Therefore, the self-floating properties and adsorption capability of macroballoons give an advantage as the optimised sample to study the effect of different types of binder. Four types of binders were employed (epoxy resin, sodium alginate, silicone resin and grout) to produce macroballoons. Results demonstrated that sodium alginate macroballoon possesses better adsorption with more 80 % of dye removal within 120 minutes as compared to the remaining three binders. This is due to the sodium alginate used did not block the adsorption site on the surface of Na-PAC, meanwhile Na-PAC in grout dispersedly in dye solution, Na-PAC in silicone and epoxy hindered from adsorption process. In conclusion, 1.0 Na-PAC produced high porosity activated carbon that was able to remove the RR dye within the allotted time when applied in three distinct forms for the adsorption process. The types of binder also affect the adsorption ability, giving sodium alginate the most suitable binder to produce macroballoons without hindering the adsorption process.

CHAPTER 1 : INTRODUCTION

1.1 Research Background

Malaysia produced roughly 1.68 million metric tonnes of rice in 2021, an increase of around 180 thousand metric tonnes over the previous year's output volume (Makhtar et al., 2022). In 2019 alone, rice production was approximately 1.88 million metric tonnes, generating around 0.53 million rice husks (RH) (Sarena et al., 2019) and expected to increase up to 2.5 million in 2023. Its production is estimated to increase as targeted by the Ministry of Agriculture and Food Industries (MAFI) to get a 100 % rice self-sufficiency level (RSSL) in the next two years. To ensure adequate rice is available and to limit rice imports from neighbouring nations, MAFI has boosted Malaysia's RSSL target to 25 % by 2025. (PMO, 2021). The consistent amount of rice production has led to the supply of RH, as well as rice husk ash (RHA), as the rice mill's waste product in Malaysia is available throughout the year.

RHA is a by-product of a rice mill factory in which RH is burned to an average of $\sim 700\text{ }^{\circ}\text{C}$ - $800\text{ }^{\circ}\text{C}$ for the rice drying process, leaving the abundant mass of RHA. Most of the rice husk ash usually ends up either being dumped or burned in open spaces, thus causing damage to the land and water pollution (Teixeira et al., 2019), which is inherently linked to environmental problems and climate change. Commercial suppliers such as Zhengzhou Zhulin Activated Carbon Co., Ltd tend to use RH as the precursor material in producing AC as they can control the carbonisation parameter for uniform production of RHA or biochar. This is due to the different burning methods can produce two types of RHA; controlled burn RHA (CBRHA) with set carbonisation settings and

open burn RHA (OBRHA) with carbonisation in the furnace without controlled parameters (Selvaranjan et al., 2021).

The commercial process also involves high processing temperatures during the carbonisation and activation process and specific environments that need inert gases such as nitrogen to assist the process. Similar findings were reported by Reza et al. (2020) that show the average carbonisation temperature is in the range of 400 °C to 1000 °C, with the activation process temperature set between 500 °C to 900 °C; some inert gases used in activation include nitrogen and carbon dioxide. As summarized, the process of converting RHA into activated carbon (AC) takes a long processing time as the pyrolysis process has a limited increment in the temperature per minute according to the furnace setup and also requires a high processing temperature. Furthermore, RHA supplied by Dibuk Sdn. Bhd. already has been exposed to carbonisation at 800 °C in the cyclone furnace. Singh (2018) reported that RH was burned at a high temperature, 700 °C has shown increasing carbon content. Previously reported that the abundant RHA left from the process is commonly used directly as the soil media for agriculture fields as fertiliser or soil stabiliser (Adhikary & Jana, 2016; Pushpakumara & Mendis, 2022; Raja et al., 2022), filler in cement or called pozzolanic materials (Liu et al., 2022; Maryoto & Heri Sudiby, 2019; Sunarjono et al., 2023), or being treated with chemical activating agent into AC for adsorption process (Liu et al., 2012; Mortada et al., 2023; S. Wang et al., 2022) for water and air pollution.

The conversion of RHA into AC involves two main processes: carbonisation and activation (MacDermid-Watts et al., 2021; Saad et al., 2019). The activation process can be divided into two types: physical and chemical. The physical activation