ACTIVATED CARBON FROM TAMARIND SEED AND JACKFRUIT SEED PREPARED VIA MICROWAVE IRRADIATED FOR DYE REMOVAL

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by

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LIST OF ABBREVIATIONS

AC	Activated carbon
ANOVA	Analysis of variance
BET	Brunauer-Emmett-Teller
CCD	Central composite design
EA	Elemental analysis
GAC	Granular activated carbon
IR	Impregnation ratio
IUPAC	International Union of Pure and Applied Chemistry
JS	Jackfruit seed
JASAC	Jackfrui seed activated carbon
PAC	Powder activated carbon
RSM	Response surface methodology
SEM	Scanning electron microscopy
TS	Tamarind seed
TASAC	Tamarind seed activated carbon

LIST OF SYMBOLS

A	Heat of adsorption constant	L/mg
B	Constant for Temkin isotherm	mg/g h
C_e	Concentration of adsorbate at equilibrium	mg/L
C_t	Concentration of adsorbate at time, t	mg/L
C_o	Initial/inlet adsorbate concentration	mg/L
ka	Langmuir adsorption constant	L/mg
kt	Equilibrium binding constant	L/mg
K_F	Adsorption or distribution coefficient for Freundlich isotherm	mg/g $(L/mg)^{1/n}$
K_L	Rate of adsorption for Langmuir isotherm	L/mg
k_1	Adsorption rate constant for pseudo-first-order	1/h
k_2	Adsorption rate constant for pseudo-second-order	g/mg h
M_1	Initial concentration	mg/L
M_2	Final concentration	mg/L
N	Total number of experiments required/data point	-
n	Constant for Freundlich isotherm	-
Q_o	Adsorption capacity for Langmuir isotherm	mg/g
q_e	Amount of adsorbate adsorbed per unit mass of adsorbent at equilibrium	mg/g
q_t	Amount of adsorbate adsorbed per unit mass of adsorbent at time, t	mg/g
q_t, cal	Calculated adsorption uptake at time, t	mg/g
q_t, exp	Experimental adsorption uptake at time, t	mg/g
R_L	Separation factor	-

R^2	Determination coefficient	-
R	Ideal gas	J/mol.K
S_{BET}	BET surface area	m^2/g
V_{meso}	Mesopore volume	cm^3/g
V_T	Total pore volume	cm^3/g
V_1	Initial volume	m^3
V_2	Final volume	m^3
W_c	Dry weight of prepared activated carbon	g
W_0	Dry weight of precursor	g
W_{KOH}	Dry weight of potassium hydroxide	g
W_{char}	Dry weight of char	g

PENYEDIAAN KARBON TERAKTIF DARIPADA BIJI ASAM JAWA DAN BIJI NANGKA MELALUI KAEDAH PENYINARAN GELOMBANG MIKRO UNTUK PENYINGKIRAN PENCELUP

ABSTRAK

Sumber air seperti sungai, tasik, air bumi dan laut yang berhampiran dengan kawasan industri tekstil lebih mudah mengalami pencemaran air akibat buangan terus air sisa tekstil ke persekitaran. Bahan penjerap seperti karbon teraktif berasaskan sisa pertanian didapati berpotensi untuk digunakan bagi tujuan rawatan air sisa ini. Dalam kajian ini, karbon teraktif daripada biji asam jawa (KTBAJ) serta biji nangka (KTBN) disediakan melalui pengaktifan fizikima secara pemanasan gelombang mikro. Kaedah ini merangkumi impregnasi kalium hidroksida, gasifikasi karbon dioksida dan penyinaran gelombang mikro. Kajian penjerapan dijalankan untuk mengenal pasti prestasi KTBAJ dan KTBN dalam penyingkiran pencelup metilena biru serta remazol biru terang R. Rekabentuk komposit pusat digunakan untuk mengenal pasti keadaan penyediaan optimum bagi masa penyinaran, kekuatan penyinaran serta nisbah impregnasi untuk biji asam jawa serta biji nangka. Keadaan optimum bagi penyediaan KTBAJ adalah 440 W, 2.0 minit dan nisbah impregnasi 0.50 manakala KTBN adalah 440 W, 2.0 minit and nisbah impregnasi 0.60 yang memberikan keputusan penyingkiran metilena biru sebanyak masing-masing 70.4% dan 83.2%; penyingkiran remazol biru terang R iaitu masing-masing 62.7% dan 75.6% serta hasil masing-masing sebanyak 21.8% dan 22.5 %. Karbon teraktif biji asam jawa dan biji nangka mempunyai luas permukaan yang tinggi (>869 m^2/g), isipadu liang (> 0.40 cm^3/g) dan kandungan karbon tetap (>76%). Kedua-dua bahan ini mengandungi liang pori yang bersifat tidak seragam dalam julat meso liang. Kesan kepekatan awal pencelup (25-300 mg/L), masa tindakbalas (0-24 jam) dan suhu larutan (30-60^{o}C) juga turut dikenal pasti. Kadar penjerapan meningkat apabila kepekatan awal serta masa tindakbalas meningkat. Untuk kedua-dua pencelup bagi KTBAJ serta KTBN sesuai digandingkan dalam model Freundlich untuk menepati hubungan penjerapan keseimbangan. Model pseudo-tertib-pertama sesuai mewakili data kinetik bagi kedua-dua bahan penyerap tersebut.

ACTIVATED CARBON FROM TAMARIND SEED AND JACKFRUIT SEED PREPARED VIA MICROWAVE IRRADIATED FOR DYE REMOVAL

ABSTRACT

Water sources such as rivers, lakes, groundwater and seas that located nearby to textile industries can be easily polluted if direct discharge of dye wastewater to the environment is occurred. Adsorbent such as activated carbon particularly produced from agrowaste has been found to be suitable for this purpose. In this study, tamarind seed based activated carbon (TASAC) and jackfruit seed based activated carbon (JASAC) were prepared by using physiochemical activation. It consisted of potassium hydroxide (KOH) impregnation, CO_2 gasification and microwave irradiation. A study of batch adsorption for methylene blue (MB) and remazol brilliant blue R (RBBR) were carried out to determine the performance of TASAC and JASAC. Central composite design (CCD) was used to evaluate the optimum preparation conditions of radiation time, radiation power and impregnation ratio (IR) for TASAC and JASAC. The optimum conditions for TASAC were 440 W, 2.0 min and IR 0.5 while for JASAC was 440 W, 2.0 min and IR 0.6, which resulted MB removal of 70.4% and 83.2%, respectively; RBBR removal of 62.7% and 75.6%, respectively and yield of 21.8% and 22.5%, respectively. TASAC and JASAC have high surface area (>869 m^2/g), pore volume (> 0.40 cm^3/g) and fixed carbon content (>76%). The TASAC and JASAC have heterogeneous type pore size in mesoporous region. The effect of initial dye concentrations (25-300 mg/L), contact times (0-24 hours) and solution temperatures (30-60^{o}C) were determined. The MB and RBBR adsorption by TASAC and JASAC were increased as the initial dye concentration and contact time increased. The adsorption equilibrium data for both dyes onto TASAC and JASAC were best fitted by *Freundlich* model. Meanwhile, the kinetic data was best represented by the pseudo first-order kinetic model.

CHAPTER ONE

INTRODUCTION

1.1 Textile production: Environmental issue and impact

Water is one of the important living sources that has many purposes such as irrigation for agriculture, industrial sectors and transportation. However, the source of clean water is in critical mode due to the urban modernisation and industrial growth. For the past decades, textile industries have grown tremendously for producing fabrics and clothing to meet the world demand. In Malaysia, textile sector is one of the sectors that contributes to the annual national income. According to the Malaysian Investment Development Authority report, the textile industry currently employs over 68,000 workers across more than 970 registered garment and textile factories in the country, of which over 400 are making ready-made garments, and the rest are operating in major sub-sectors including polymerisation, spinning, weaving, knitting and wet processing, and textile accessories (MIDA, 2015). In 2017, Malaysia's textile industry was one of the top ten largest export earners in the country, with an export value reaching RM 15.3 billion, representing 1.6% of Malaysia's total exports of manufactured goods (MIDA, 2018). For satisfying the customer demands, varieties of latest patterns and coloured fabrics are produced. Coloured fabrics are manufactured by using dye. Dye is produced from synthetic material which contains high amount of chemicals that might affect the environment.

For producing small amount of coloured fabrics, large amount of dye paste is applied. Besides that, huge amount of water is used to dissolve the dye for producing the coloured mixture. Furthermore, water is applied to remove the excess dyes that have been attached to the coloured fabrics. Moreover, dye equipment such as screening is cleaned by using high amount of water (Carmen and Daniela, 2010). Wastewater that was generated from these processes contains large amount of dye and it consumes 16% of total water usage depending on the type of dyes used and this dyeing sector contributes to 15% - 20% of the total waste water flow (Elango et al., 2017). The characteristic of the dye is toxic and carcinogenic.

The effluent from textile factories is released to the nearby rivers and lakes which affect the biodiversity and creates water pollution. The effluent contains high level of turbidity due to the high concentration of dye. Dye usually floats onto the surface of the water which blocks the sunlight pathway. This situation will upset the photosynthesis process for aquatic plants (Maria et al., 2013). Another impact towards the ecosystem is fish will undergo mutation process due to the dye toxicity from the effluent. Moreover, humans that consume the mutation fish will have mutagenic cancer cells that might affect human health such as necrosis and blindness (Hassaan and Nemr, 2017). Furthermore, the accumulation of dye effluent in the river or lakes produces bad odour for surrounding that develops discomfort to the humans. Besides that, clean water sources will reduce due to the release of dye effluent into the rivers and lakes which may cause water crisis in the future.

Dye can be produced from two different materials. One is from natural-made and the other one is synthetic-made. With this kind of methods of production, varieties of coloured fabrics are produced. There are many stages for producing good quality of fabrics or textiles. The final stage of the production requires dye to be used for producing desired-coloured fabrics. Dye is an aromatic compound that are being produced from the process of chemical synthesis. The colour from the dye is appearing when there is a structure which is being created known as chromogene-chromophore bonding structure which known to be as electron-acceptor while the dye capacity is produced from the group of structure known as auxochrome (Maria et al., 2013). Auxochrome is an electron donor which undergo oxidation process.

Dye can be divided into two types which are basic dye and reactive dye. Basic dye has its own characteristics. It is synthetic class dye which apparently cheap in price. Besides that, basic dye is one type of salt which contains cationic that responsible for colour production. Moreover, basic dyes are hydro chlorides salts and it is cationic dyes (El Qada et al., 2008). Basic dyes can produce brilliant shades of colours due to its characteristics. Fabrics like cotton has no affinity towards basic dyes but dyeing for cotton can be done by using fixation or dyeing advanced systems. Basic dyes are more affordable in bulk for large dyeing operating systems and it is cost-effective for producing coloured fabrics. Mostly, methylene blue and crystal violet are used in the textile industries.

The other type of dye is reactive dye. Most of the dyeing operation of fibres are done by reactive dyes. Reactive dyes and fibre are fused by using covalent bonding. Reactive dye has its own characteristics. In nature, reactive dyes are anionic. It is water soluble dye and washable. It has light fastness properties. The resistant towards fading intensity to the light is known as light fastness properties (Demirbas, 2009). Strong covalent bond is formed between the reactive dyes and cellulosic fibre. Varieties of colour range can be produced by using reactive dyes. Reactive dyes have more brilliant colour. Relatively it is quite cheap for bulk production of coloured fabrics. The common reactive dye that have been applied in the fabric production are remazol brilliant blue R.

1.2 Tamarind seed and jackfruit seed

Choosing a precursor is very significant for AC production. The best way to choose is to consider a few factors. The precursor must be cheap and it is renewable. Moreover, the amount of fixed carbon content is very crucial in order to covert raw materials into activated carbon. The production of good precursor can be obtained from agriculture waste due to its high carbon content.

Tamarind seed is produced from tamarind pulp which can be found in tropical area. Tamarind tree is originated from Africa and being cultivated in India. It reached South East Asia through human transportation. The scientific name for tamarind is *tamarindus indica* (Ha et al., 2015). Tamarind is edible and usually is used in Asian cuisine as an enhancer. The taste of tamarind is sour and the shape is like a pulp. Cuisine such as curry and stew are using tamarind extract for balancing the spicy-sour taste. Other than food industry, tamarind is also used for medicine. It is used to cure sore-throat traditionally. It highly contains vitamin C and calcium which is good for human health. Each mature is estimated to produce 180-225 kg tamarind per season annually (Rao and Mathew, 2012).

Jackfruit is known as *Artocarpus heterophyllus* in its scientific name. Jackfruit is originated from India. It has been cultivated in South East Asia because it is very suitable to be planted in a tropical area. In Malaysia, 2,975 ha of jackfruit area is planted (Salleh, 2010). Up to 500 seeds can be found in each fruit. Seeds are recalcitrant and can be stored up to a month in cool, humid conditions (Elevitch and Manner, 2006). Normally, jackfruit has aromatic fruity smell that has high attraction. The taste of jackfruit is sweet and it is edible. The flesh of jackfruit is yellow in color. Jackfruit can be eaten directly or can be turned into delicious cuisine such as desserts. In Malaysia, jackfruit is also used in curry to enhance the