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Adsorption Isotherm and Kinetic Models for Removal Of Methyl Orange and Remazol Brilliant Blue R by Coconut Shell Activated Carbon

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ABSTRACT: Adsorption technology is one of the efficient and facile method used for wastewater treatment. In this research, coconut shell, an agricultural solid waste was converted into activated carbon via furnace induced and zinc chloride chemical activation techniques. The activated carbon was prepared at activation temperature of 600°C. Anionic dyes, Methyl Orange (MO) and Remazol Brilliant Blue R (RBBR) have been selected due to their harmful effect to the environmental and human. Various effect of parameter such as initial dye concentration, initial pH, adsorbent dosage and agitation speed in batch system were investigated to obtain the optimum condition for both dye adsorption on activated carbon. The optimum dye removal efficiency was around 99% when 5g/L of activated carbon was used. Pseudo-second-order model was the best fitted model with highest correlation (R^2 >0.99) compared to other kinetic models. The adsorption behaviour of MO was perfectly presented by the Freundlich model while RBBR was well described by Langmuir model. The maximum adsorption capacity for MO was 59.17mg/g and RBBR was 35.09mg/g. Fourier-transform infrared spectroscopy (FTIR) was utilised to analyse the chemical characteristics of activated carbon before and after adsorption.

KEYWORDS: Methyl Orange; Remazol Brilliant Blue R; Pseudo-second-order; Freundlich model; Langmuir model.

1. Introduction

Dyes are chemical compounds that have stickiness ability to fabric and surface to impart colour. Synthetic dyes are commonly used in cosmetics, paper, rubber, leather, pharmaceutical, textiles, food industries and more. These industrial and factory are using the dyes to colour their final products and then dispose wastewater containing organics with a strong colour. Those wastewaters disposed without proper treatment caused pollution problems. Dyes pollution can affect the aesthetic merit, prevent sunlight penetration and decreases photosynthesis activities. Dye effluents are usually store as industrial waste by those industries using dye after done colouring the products [1]. The concentrated dye effluent with high temperature can disturb the self-purification process and oxygen transfer mechanism of water. Those polluted water produced a foul odour and creating an eye sore to people [2].

Methyl Orange (MO) is one of the well-known water soluble anionic or acidic dyes, and it is mostly used in printing, food, textile, paper and pharmaceutical industries. MO was metabolized by intestinal microorganisms into aromatic amines if accidently ingest it. Besides that, MO also known as azo dyes which have high toxicity that can cause serious health problem when the aromatic structures of them degrade in wastewater [3]. Remazol Brilliant Blue R is one of the popular reactive dye that often used in textile industry. The class of this dye is toxic, and it is a recalcitrant organic pollutant. This dye is frequently used as starting material to produce the polymeric colorants

There are several conventional methods used to remove dyes from wastewater such as: coagulation and flocculation, ozonation or oxidation, adsorption, and membrane separation [1]. Adsorption of dye by using the agricultural waste is becoming a probable alternative adsorbent to replace the activated carbon because it is economic, largely available, environmental-friendly and efficient for dye removal. These material from agricultural by-product have different adsorption capacity depend on their physic-chemical characteristics [4]. The activated carbon obtained from agricultural by-product has similar or even better adsorption efficiency. Pyrolysis process had been applied to transform the agricultural waste into activated carbon with or without chemical activating agents under controlled condition [5]. The application of agriculture by-product to produce activated carbon was an alternative which is economic, eco-friendly, largely available and renewable. Besides that, any lignocellulosic or any inexpensive material that has low inorganic content and high carbon can be utilised for activated carbon purpose [6,7]. This research aims to study the adsorption removal efficiency of dyes from aqueous solution by using agricultural based activated carbon.

2. Materials and Methods

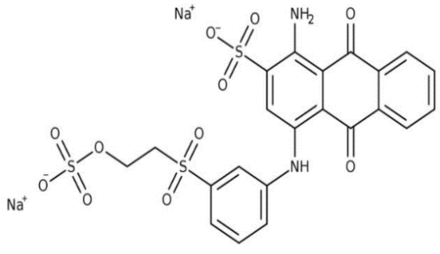
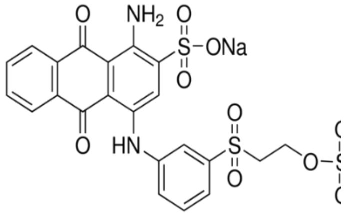
2.1. Chemicals and materials.

The highest available grade of dye and solvent were obtained from Sigma-Aldrich (Milwaukee, USA). The stock dye solution was prepared by adding 1 gram of dyes powder of each MO and RBBR with 1000ml of distilled water. The prepared stock dye solution is then kept at room temperature for further procedures. Table 1 shows the properties of phenol red and cresol red. A set of stock dye solution with different concentration was prepared in advance before proceeding to conduct the experiment. The coconut shell activated carbon (CSAC) as an adsorbent was prepared by cleaning the raw materials and removing the moisture using oven for 24 hours. The dried adsorbent was carbonized in a horizontal furnace, under nitrogen gas at 500 ^{o}C for 1 h. the N_2 flow rate was 2.5 L/min. The activated carbon was ground in the blender to form fine powder.

2.2. Adsorption experiment.

Adsorption experiment was conducted by preparing 1g of adsorbent with 100 ml dye into the 250-ml conical flask. The initial pH of the dye solution can be adjusted by adding 0.1M of NaOH or 0.1M of HCl until the desired pH was obtained. Activated carbon was then added into the dyes' solution prepared and shake at ambient temperature (30°C) by using labnet shaking incubator until the equilibrium was reached. After adsorption, the samples were collected, and the suspensions was separated by Sartorius filter paper. All conical flasks were put on to the rotary shaker with agitated speed of 100 rpm at room temperature. After filtration, the dye removal was analyzed by UV-Vis spectrophotometer (Perkin Elmer Lambda Bio+) at λ_{max} 464nm (Methyl Orange) and λ_{max} 538nm (RBBR).

Table 1. Physical and chemical characteristics of MO and RBBR.

Properties	**Physical-chemical**	
Name	Methyl Orange (MO)	Remazol Brilliant Blue R (RBBR)
Structure		
Molecular formula	$C_{14}H_{14}N_3NaO_3S$	$C_{22}H_{16}N_2Na_2O_{11}S_3$
Appearance	Orange Powder	Blue Powder
Molecular weight	327.334 g/mole	626.533g/mole
CAS Number	547-58-0	2580-78-1
Absorption, λ_{max}	464 nm	538 nm

The formulas for both adsorption capacity and the percentage rate on removal for each sample are shown below:

$$\text{Removal rate (\%)} = \frac{Co-Ct}{Co} \times 100$$

$$\text{Adsorption capacity, } q\ \left(\frac{mg}{g}\right) = \frac{Co-Ct}{M} \times V$$

where M is the adorbent mass, V is the volume of dye solution, C_o is the initial concentration of the dye solution and C_t is the dye concentration after adsorption process. Summarizes for all experiments of batch adsorption were summarized in Table 2.

Table 2. Summarizes for all experiments of batch adsorption.

Parameters	**Initial pH**	**Dosage (g/L)**	**Agitation (rpm)**	**Initial Concentration (mg/L)**
Effect of Initial pH	3 – 12	1	100	10
Effect of Dosage	6.5	1 - 10	100	10
Effect of Contact Time	6.5	1	50 - 300	10
Effect of Initial Concentration	6.5	1	100	10-60

2.3. Isotherm and kinetic studies.

The kinetics of adsorption Pseudo-first-order model and Pseudo-second-order model are used to differentiate kinetic equations according to adsorption capacity affected by initial dye concentration. Elovich model is used to assume the surfaces of adsorbent are heterogeneous and it is a multilayer adsorption process The kinetic models used in this experiment is as follows:

$$\text{Pseudo-first-order : } \ln(q_e - q_t) = \ln q_e - K_1 t$$

$$\text{Pseudo-second-order : } \frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$$

$$\text{Elovic : } q_t = (\frac{1}{b}) ln(ab) + \frac{1}{b} ln\ t$$

Where q_e is the amount of the adsorbate at equilibrium (mg/g), q_t is maximun uptake of adsorbate from plot ln q_e versus C_e, K_1 is the pseudo-first-order rate constant, k_2 is the pseudo-second-order rate constant, t is the contact time with adsorbent (min), a is initial sorption rate (mg/gh), and b is extent of surface coverage and activation energy for chemisorption (g/mg).

Adsorption isotherm is an invaluable curve that illustrate the phenomenon governing the mobility or retention of a substance from the aquatic environments or aqueous porous media to a solid-phase with a constant pH and temperature. The isotherm models (two parameter) that widely used are Langmuir, Freundlich, Temkin and Dubinin-Radushkevich.

Langmuir isotherm is is derived by assuming the adsorption is a monolayer surface that contain a limited number of adsorption sites of uniform strategies of adsorption with no transmigration of adsorbate in the plane of surface. The Langmuir equation is written in linear form:

$$\frac{C_e}{q_e} = \frac{1}{k_L q_m} + \frac{C_e}{q_m}$$

where C_e is the concentration of adsorbate at equilibrium (mg/g), q_e is the amount of the adsorbate at equilibrium, q_m is maximum adsorption capacity, and K_L is Langmuir rate constant (mg/g).

Temkin isotherm is applied to explain observed results and find out the nature of adsorption. The Temkin isotherm is written in linear form:

$$q_e = \frac{RT}{b_T} lnA_T + \frac{RT}{b_T} lnC_e$$

where q_e is the amount of adsorbate at equilibrium (mg/g), R is Universal gas constant (8.314J/mol K), T = Temperature (K), b_T is the Temkin constant, A_T Temkin isotherm equilibrium binding constant (L/g), and C_e is the adsorbate concentration at equilibrium (mg/g)

Freundlich isotherm is based on the assumption that a heterogeneous surface with non-uniform distribution of adsorption heat over the surface through a multilayer adsorption process. The equation is written below:

$$log\ q_e = log\ K_F + \frac{1}{n}\ log\ C_e$$

where K_F is the adsorption capacity (L/mg), q_e is the amount of adsorbate at equilibrium (mg/g), C_e is the adsorbate concentration at equilibrium (mg/g), and 1/n is the adsorption intensity.

Dubinin-Radushkevich isotherm is used to explain the porous structure effect of adsorbent on the adsorption process. The linear form of Dubinin-Radushkevich model is expressed as:

$$lnq_e = \ln(q_{DR}) - K_{DR}\varepsilon^2$$

Where qDR is The capacity of theoretical isotherm saturation (mg/g), KDR is Dubinin-Radushkevich isotherm constant (mol^2/kJ^2), ε is Dubinin-Radushkevich isotherm constant.

2.4. FTIR

Fourier transform infrared spectroscopy (FTIR) is a vibrational spectroscopic technique that normally utilised to determinate the chemical characteristics functional groups of adsorbents that possibly involved during the adsorption of dyes. The molecular structure and energy level transition of a sample can be obtained by absorb, transmit, reflect, scatter or photoluminescence the electromagnetic radiation that interact with the samples. The functional group of the raw adsorbents and adsorbents after decolorization was analyzed by using Fourier Transform Infrared Spectroscopy (FTIR) (Spectrum one, Perkin Elmer, USA) recorded in the spectral range of 400-4000 cm^{-1}.

3. Results and Discussion

3.1. Effect of Initial Dye Concentration

The effect of initial concentration of dyes were carried out by using different initial dye concentration such as 10, 20 ,30, 40 ,50 and 60 mg/L respectively. The experiment variables such as 1 g/L dosage, natural pH, 50ml of solution, 100 rpm agitation speed, 360 min contact time and 30±3 °C temperature was set in constant for both dyes. All the solution with 10 mg/L above initial concentration was filtered and then diluted after 6 hours adsorption process. Table 3 shows the effect of initial concentration on adsorption of MO and RBBR by CSAC. The results showed that the highest adsorption capacity for MO and RBBR was 47.52 mg/g and 33.58 mg/g respectively. For MO, the highest removal efficiency was 86.99% while RBBR was 95.06%. The adsorption capacity increases due to high driving force for mass transfer at higher initial dye concentration. On the other hand, the increase in initial dye concentration caused a decrease in removal efficiency and this may be due to the saturation of adsorption site on the adsorbent surface [8,9].

Table 3. Effect of initial concentration on adsorption of MO and RBBR by CSAC.

Initial Concentration (mg/L)	Methyl Orange		Remazol Brilliant Blue R	
	***q* (mg/g)**	**Removal (%)**	***q* (mg/g)**	**Removal (%)**
10	10.10	86.99	9.51	95.06
20	20.13	87.21	15.80	88.89
30	25.37	78.10	23.21	71.21
40	35.03	75.00	30.62	74.70
50	38.12	68.27	33.58	65.38
60	47.52	68.60	33.58	52.31

3.2. Effect of Dosage

The effect of AC dosage on MO and RBBR were evaluated by varying the mass of AC with 0.05g, 0.1g, 0.15g, 0.2g, 0.25g and 0.5g respectively. The constant variables such as initial concentration of 10mg/L, natural pH, 50ml volume of solution, 100 rpm agitation speed, 360 min contact time and 30±3 °C temperature was fixed correspondingly for both dyes Table 4 shows the effect of dosage on adsorption of MO and RBBR by CSAC. The highest adsorption capacity for MO and RBBR can be reached was 11.24 mg/g and 9.01 mg/g when using 1g/L AC dosage. The highest removal efficiency for MO and RBBR was achieved at 99.71% and 98.77% respectively with 5g/L AC dosage used. From these data, the saturation point for both dyes are around 5g/L and further increasing the dosage does not increases the removal efficiency. Based on the results obtained below, the AC dosage used greatly affect the adsorption capacity and removal efficiency due to the quantity of sorption site on the adsorbent surface increased when the adsorbent dosage increase [10].

Table 4. Effect of dosage on adsorption of MO and RBBR by CSAC.

Dosage (g/L)	Methyl Orange		Remazol Brilliant Blue R	
	q (mg/g)	Removal (%)	*q* (mg/g)	Removal (%)
1	11.24	96.82	9.01	90.12
2	5.64	97.11	4.75	95.06
3	3.77	97.40	3.21	96.30
4	2.88	99.13	2.44	97.53
5	2.32	99.71	1.98	98.77
10	1.09	93.93	0.89	88.89

3.3. Effect of pH

The effect of pH on adsorption of MO and RBBR was studied by preparing different apart of initial pH of dye solution such as 2, 4, natural, 10, and 12. Table 5 shows the effect of pH on adsorption of MO and RBBR by CSAC. The highest adsorption capacity and removal efficiency for MO was 11.14 mg/g and 95.95%. Furthermore, the highest adsorption and removal efficiency for RBBR was 8.89 mg/g and 88.89%.

Table 5. Effect of pH on adsorption of MO and RBBR by CSAC.

pH	Methyl Orange		Remazol Brilliant Blue R	
	q (mg/g)	Removal (%)	*q* (mg/g)	Removal (%)
2	11.04	95.09	8.89	88.89
4	11.14	95.95	8.27	82.72
Natural (6.5±0.3)	10.91	93.93	8.89	88.89
10	8.36	71.97	3.09	30.86
12	9.50	81.79	6.79	67.90

It obviously shows that the adsorption capacity and removal efficiency of MO and RBBR at acidic pH is higher than at alkaline pH. This indicated that the force of interaction between the AC and dyes that only H+ ions could influence the adsorption capacity and removal efficiency. Thus, the interaction is stronger when the solution is acidic due to competence of H^+ ion with dye cation for the sorption sites [11]. This can also be due to the pKa value of both dye is higher at that particular pH [12]. In addition, the adsorption of both dyes was not favourable at higher pH due to competence of OH^- with dye anions and also electrostatic repulsion [13].

3.4. Effect of Agitation Speed

The effect of agitation speed on adsorption capacity and removal efficiency was investigated by setting various speed such as 50, 100, 200 and 300 rpm. The constant variables such as initial concentration of 10mg/L, natural pH, 50ml volume of solution, 1 g/L dosage, 360 min contact time and 30±3 °C temperature was fixed for both dyes. Table 5 shows the effect of agitation speed on adsorption of MO and RBBR by CSAC. Both adsorption capacity and removal efficiency started to decrease when increased to 300 rpm speed. Thus, the optimum agitation speed for both dyes was 200rpm. The highest adsorption capacity for MO and RBBR was 11.31 mg/g and 9.63 mg/g while the removal efficiency is over 90% for both dyes. It was reported that the adsorption capacity and removal efficiency were affected by agitation speed as the agitation speed increases, both adsorption capacity and removal efficiency increase. This was because the boundary layer resistance to mass transfer around the adsorbent particle decreased and increase the mobility of the system [14].

Table 6. Effect of agitation speed on adsorption of MO and RBBR by CSAC

Agitation (rpm)	Methyl Orange		Remazol Brilliant Blue R	
	q (mg/g)	Removal (%)	q (mg/g)	Removal (%)
50	8.83	76.01	7.28	72.84
100	10.91	93.93	8.89	88.89
200	11.31	97.40	9.63	96.30
300	11.07	95.38	9.26	92.59

3.5. Kinetic Studies

Table 7. Kinetic parameters for MO and RBBR on CSAC

Kinetic Model	Adsorption constant	Methyl Orange	Remazol Brilliant Blue R
Pseudo-first-order	q_e (mg/g)	0.8176	0.864
	K_1 (1/min)	0.0173	0.0199
	R^2	0.8855	0.8306
Pseudo-second-order	q_e (mg/g)	1.0467	0.7784
	K_2 (g/mg min)	0.0578	0.0669
	R^2	0.9967	0.9922
Elovich	b (g/mg)	7.1225	7.6046
	a (mg/g min)	0.5251	0.1489
	R^2	0.9725	0.9414

In this study, three kinetic models: Pseudo first order, Pseudo second order and Elovich were proposed to elucidate the mechanism background of MO and RBBR adsorption. The linearized equation were used to calculate the experimental kinetic data for both dyes. The values of equilibrium adsorption capacity (q_e), correlation coefficients (R^2) and other constant parameters of kinetic models were calculated and tabulated in Table 7. Thus, pseudo-second-order kinetic model was the best fitted to the experimental data of both dyes removal compared to other kinetic models. The fit of this model explained that the valence forces from the sharing of electrons between adsorbent surface and dye molecules were affect the adsorption rate [15].

3.6. Isotherm Studies

In order to optimize the adsorption condition for dye, it is crucial to build the most accurate correlation for the equilibrium curve. In this research, four different isotherm model equations: Langmuir, Freundlich, Temkin and Dubinin-Radushkevich were applied to fit the experimental equilibrium data of adsorption of coconut shell based activated carbon ($ZnCl_2$) on Methyl Orange and Remazol Brilliant Blue R. Table 8 shows the values of maximum adsorption capacity (q_m), equilibrium adsorption capacity (q_e), correlation coefficients (R^2), and other constant parameters for four isotherm model equations for both dyes. The R^2 of the four models for MO ascend in the order of: Dubinin-Radushkevich > Langmuir > Temkin > Freundlich meanwhile for RBBR the R^2 ascend in order of: Dubinin-Radushkevich > Temkin > Freundlich > Langmuir. The best fitted model was concluded for MO, Freundlich and RBBR, Langmuir. The adsorption of MO on activated carbon tended to homogeneous adsorption while RBBR was monolayer adsorption. Besides that, the R_L for RBBR was found to be 0.15 which is less than 0.29 as compared to the result from [16] and this enhanced that the Langmuir isotherm was favorable adsorption of RBBR on activated carbon. The value of q_m calculated by Langmuir for MO (59.17 mg/g) and RBBR (35.09 mg/g) which are much lower than the previous result [13,17].

Table 8. Isotherm Model for MO and RBBR on CSAC

Isotherm Model	Adsorption constant	Methyl Orange	Remazol Brilliant Blue R
Langmuir	q_m (mg/g)	59.1716	35.0877
	K_L (L/mg)	0.1318	0.5689
	R^2	0.9506	0.9813
	R_L	0.3952	0.1495
Freundlich	n	1.9231	3.0941
	K_f (L/mg)	9.3864	12.3851
	R^2	0.9543	0.9614
Temkin	A (L/g)	1.4363	7.7842
	B (J/mol)	200.6645	398.8824
	R^2	0.952	0.9335
Dubinin-Radushkevich	K	-1E-7	-2E-8
	q_e (mg/g)	93.6065	32.3140
	R^2	0.9286	0.7871

3.7. FTIR

FTIR spectral characteristics of activated carbon before and after adsorption of MO and RBBR were shown in Table 9. Various functional groups were found on the surface of activated carbon before and after adsorption. There are some peaks shifted, disappeared and new peaks were also detected after adsorption of both dyes. As shown in Table 9, new peaks, 3697.52 cm^{-1} (O-H stretch), 3451.51 cm^{-1} (O-H stretch, H-bonded), 2952.05 cm^{-1} (O-H stretch) were appeared after adsorption of MO while new peaks for RBBR as follows: 3690.06 cm^{-1} (O-H stretch), 3451.51 cm^{-1} (O-H stretch, H-bonded), 3145.87 cm^{-1} (O-H stretch). The peak at 3265.15 cm^{-1}, 2676.23 cm^{-1}, 2102.22 cm^{-1}, 1871.12 cm^{-1} had small changes maybe occurred due to the sensitivity of the equipment. The peak at 1535.66 cm^{-1} was disappeared after adsorption of MO and had minor change after adsorption of RBBR. The peak at 1118.20 cm^{-1} and 767.83 cm^{-1} changed obviously after the adsorption of both dyes.

Table 9. FTIR spectral characteristics of activated carbon before and after adsorption of MO and RBBR

Before adsorption (cm^{-1})	After MO adsorption (cm^{-1})	After RBBR adsorption (cm^{-1})	Functional Group	Compound
-	3697.52	3690.06	O-H stretch	alcohol
-	3451.51	3451.51	O-H stretch, H-bonded	alcohols, phenols
3265.15	3265.15	3287.15	O-H stretch, H-bonded, N-H stretch, -C≡C-H, C-H stretch	alcohols, phenols, carboxylic acid, 1°, 2° amines, amides, alkynes (terminal)
-	2952.05	2959.51	O-H stretch	carboxylic acid, alcohol
2676.23	2646.41	2661.32	O-H stretch	carboxylic acid
2102.22	2094.76	2094.76	N=C=S stretch, -C≡C- stretch, C≡N stretch	Isothiocyanate, alkynes, nitriles
1871.12	1856.21	1863.87	C=O stretch, C-H bend	carbonyls (general), carboxylic acids, aromatic compound
1535.66	-	1520.75	N-O asymmetric stretch	nitro compounds
1118.20	1051.11	969.11	=C-H bend, C-O stretch, C-N stretch	alcohols, carboxylic acids, esters, ethers, aliphatic amines, alkenes
767.83	708.19	775.29	=C-H bend, N-H wag, C-H "oop", C-Cl stretch	Alkenes, 1°, 2° amines, aromatics, alkyl halides

4. Conclusions

In this research, the activated carbon which produced by using coconut shell and activated by $ZnCl_2$ was utilised to remove anionic dyes, MO and RBBR from aqueous solution. The highest removal efficiency for MO, 99.71% and RBBR, 98.77% as well as both was achieved when 5g/L activated carbon was used. Pseudo-second-order was the best fitted model for both dyes. The adsorption of MO was well explained by Freundlich isotherm model while RBBR was well described by Langmuir isotherm model. The chemical characteristics of activated carbon before and after adsorption of both dyes were found by using FTIR. Overall, the outcome of this research has concluded that agricultural waste, coconut shell is an effective and efficient activated carbon for adsorption of anionic dye, MO and RBBR from textile industries effluent.

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Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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