Eng Hock Pua, Azrul Nurfaiz Mohd Faizal, Sirajo Lawal and Muhammad Abbas Ahmad Zaini*

Two-stage adsorber design for malachite green and methylene blue removal using adsorbents derived from banana peel

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Abstract: This work was aimed at evaluating the adsorptive characteristics and two-stage adsorber design of banana peel adsorbents for malachite green and methylene blue removal. The adsorbents were characterized for specific surface, functional groups, and morphology. Activated carbon and hydrochar activated carbon exhibit similar textural and surface properties, but different capacities of malachite green and methylene blue. The latter with surface area of 877 m^2/g endows a greater malachite green removal at 582 mg/g, while the former with surface area of 897 m^2/g displays a higher methylene blue capacity of 503 mg/g. The Langmuir model was employed in a two-stage adsorber design. The second stage of adsorber is necessary to accomplish the adsorption process with high performance and minimum dosage of activated carbon.

Keywords: activated carbon; adsorption; banana peel; malachite green; methylene blue; two-stage adsorber.

1 Introduction

The discharge of dyes from the textile, leather, and cosmetics industries is a source of water pollution and becomes a potential threat to aquatic creatures and public health (Amin, Alazba, and Shafiq 2019). Dyes are essentially toxic; they impede light penetration for photosynthetic activity, so reducing dissolved oxygen for respiration and plant growth (Astuti et al. 2019; Torres-Perez et al. 2020). The demand for textile-related products has resulted in the manufacturing of dyes on large scale. Methylene blue and malachite green are among the most commonly used dyes in the textile industry. They are toxic, recalcitrant to degradation, and tend to bioaccumulate along the food chains. Therefore, it is imperative for dye-bearing effluent to be treated before it enters the water bodies. Several treatment methods such as coagulation/flocculation, precipitation, membrane filtration, reverse osmosis, ozonation, advanced oxidation process, and ion exchange are available to remove dyes from wastewater (Shu-Hui and Zaini 2016; Spagnoli, Giannakoudakis, and Bashkova 2017). However, these techniques have several limitations, revolving around the use of chemicals, high maintenance costs, and often resulting in secondary pollution (Ma et al. 2015).

Among others, adsorption has emerged as a preferred treatment for dye wastewater. The process is simple and straightforward, inexpensive, consumes low energy, and produces minimum sludge. Activated carbon has been widely used as an adsorbent in adsorption. It can be produced from carbonaceous feedstocks (Ming-Twang et al. 2015), such as coal, coconut shell, wood (Zubir and Zaini 2020), lignite, rice husk (Van and Thu 2019), oil palm ash, glycerine pitch distillate (Hock and Zaini 2022), cashew nutshell, sawdust, banana peel, etc (Ukanwa et al. 2019). Activators such as phosphoric acid (Zubir and Zaini 2020), zinc chloride (Hock and Zaini 2022), sodium hydroxide and potassium hydroxide (Van and Thu 2019) have been widely used to convert agricultural residues into activated carbon. Compared to other activators, phosphoric acid is less toxic to the environment (Zubir and Zaini 2020). In a recent report, Ravindran et al. (2022) reported the use of *Ulva prolifera* seaweed char for reactive yellow 81 decolorization. Similarly, Sujatha and co-workers (2021) showed the removal of remazol brilliant orange 3R by char derived from *Ulva reticulata* seaweed. For most developing countries, these biomass materials are abundantly available and usually regarded as waste of no commercial value. Nonetheless, the resultant char and activated carbon often possess high porosity, thus providing numerous functional sites for water pollutants entrapment.

***Corresponding author: Muhammad Abbas Ahmad Zaini**, Centre of Lipids Engineering & Applied Research (CLEAR), Ibnu-Sina Institute for Scientific & Industrial Research (ISI-SIR), Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia, E-mail: abbas@cheme.utm.my. https://orcid.org/0000-0003-2674-2670
Eng Hock Pua and Azrul Nurfaiz Mohd Faizal, Department of Chemical Engineering, Faculty of Chemical & Energy Engineering, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia
Sirajo Lawal, Centre of Lipids Engineering & Applied Research (CLEAR), Ibnu-Sina Institute for Scientific & Industrial Research (ISI-SIR), Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia

Banana is planted almost everywhere around the globe, especially in tropical countries (ca. 106.7 million tons of production in 2013). It is a tall and sturdy herbaceous plant, with a succulent and very juicy tubular stem, composed of leaf-petiole sheaths consisting of long and strongly overlapping fibers (Tock et al. 2010). The fruit is a main source of income from the making of banana chips. Consequently, the abundant peel leftover which has no added value could be a source of pollution if it is not managed and disposed of appropriately (Pathak, Mandavgane, and Kulkarni 2016). It has been reported that banana peel accounts for more than 41.3 million tons per year, serving as a potential biomass feedstock (Cutz et al. 2016; Fernandes et al. 2013). Thus, it can be potentially exploited into char and activated carbon because of its rich carbon content for dye wastewater treatment. In this work, banana peel was activated using phosphoric acid.

To date, there is still limited literature available to unlock the true potential of banana peel adsorbents – char and activated carbon in two-stage adsorber design for evaluating their performance at minimum dosage. Thus, this work is embarked to assess the removal efficiency and optimization of adsorbents for malachite green and methylene blue removal. The adsorptive profiles were generated using Langmuir constants to enrich the body of present knowledge and to shed insight into industrial dye wastewater treatment.

2 Materials and methods

2.1 Materials

The banana peel was obtained from the local market in the Johor state of Malaysia. Phosphoric acid for activation, and malachite green and methylene blue for adsorption were supplied by QReC (Malaysia). All chemicals are of analytical grade and were used as received.

2.2 Banana peel adsorbents

The banana peel was oven-dried at 100 °C for 24 h. Char (labeled as BP600) was produced by subjecting banana peel in a furnace under anoxic environment at 600 °C for 2 h. Activated carbon (labeled as R2.5) was obtained with H_3PO_4 activation at mass ratio of 2.5:1 (activator: banana peel) using the same condition as char. Hydrochar-activated carbon (labelled as R2.5A) was prepared from hydrochar of banana peel using the same procedures as R2.5. Banana peel hydrochar was formerly prepared in water using Teflon autoclave at 190 °C for 16 h.

2.3 Characterization of materials

The thermal degradation of banana peel was obtained using a Perkin-Elmer thermogravimetric analyzer (TGA 7) under nitrogen flow of 10 mL/min and heating rate of 15 °C/min. The specific surface of adsorbents was determined using a Micromeritics surface area analyzer (ASAP 2020) at liquid nitrogen temperature of 77 K. The surface chemistry of banana peel and its derived adsorbents was qualitatively assessed using a Perkin Elmer FTIR spectrometer (Spectrum One). The morphology of adsorbents was characterized using a Hitachi scanning electron microscope (TM3000).

The adsorption capacities of adsorbents were determined at different malachite green and methylene blue concentrations using a bottle-point technique. The residual concentration was measured using a UV–vis spectrophotometer (Spectrumlab752 Pro) at wavelengths of 650 nm (malachite green) and 590 nm (methylene blue). The adsorption capacity was calculated from mass balance,

$$q_e = \frac{V}{m}(C_o - C_e) \tag{1}$$

where q_e (mg/g) is the equilibrium adsorption, V (L) is the volume of dye solution, m (g) is the mass of activated carbon, C_o (mg/L) is the initial dye concentration and C_e (mg/L) is the equilibrium dye concentration. The equilibrium data were fitted into the Langmuir model, as follows,

$$q_e = \frac{q_m b C_e}{1 + bC_e} \tag{2}$$

Langmuir isotherms to explain how the interactions of the adsorbate molecules with the adsorbent occur. It is the most used model to describe the adsorption data. In fact, it also predicts how the adsorbate molecules interact with adsorbent and attains equilibrium (Hock and Zaini 2022).

2.4 Two-stage adsorber design

The two-stage adsorber design as shown in Figure 1 is aimed to optimize the adsorbent used in adsorption.

The expression for adsorbent mass required for adsorption was obtained by combining the adsorption capacity in Equation (1) with the Langmuir model in Equation (2).

$$m = \frac{V}{q_m b C_e}(C_o - C_e)(1 + bC_e) \tag{3}$$

For the two-stage process, the total mass is,

$$m_1 + m_2 = \frac{V}{q_m b C_1}(C_o - C_1)(1 + bC_1) + \frac{V}{q_m b C_2}(C_1 - C_2)(1 + bC_2) \tag{4}$$

where C_1 is the concentration leaving stage 1 and becomes the feed for stage 2, and $C_2 = C_e$. The optimum mass can be calculated by differentiating Equation (4) with respect to C_1.

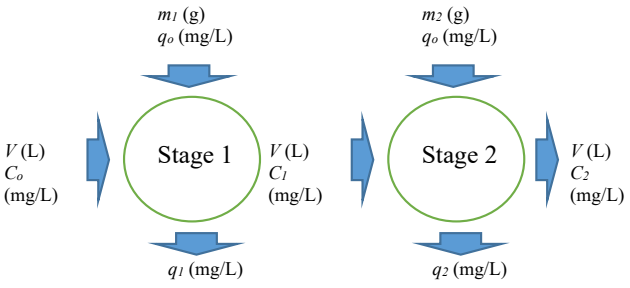

Figure 1: Schematic of two-stage adsorber design.

$$\frac{d(m_1 + m_2)}{dC_1} = 0 \tag{5}$$

Solving,

$$C_1 = (C_o C_2)^{0.5} \tag{6}$$

The performance of two-stage adsorber can be calculated as,

$$\text{Removal (\%)} = \frac{C_o - C_2}{C_o} \times 100 \tag{7}$$

Different values of removal efficiency were set to allow the comparison of the amount of activated carbon needed in a single stage adsorber with that of a two-stage adsorber.

2.5 Statistical analysis

The most significant parameter and confidence level were analyzed by response surface methodology (RSM) of Minitab 17 Statistical software. The regression analysis was carried out using Box-Behnken design matrix. The fit statistics of the quadratic model equation to optimize the two-stage adsorber in achieving a cost-competitive methylene blue and malachite green removal were reported.

3 Results and discussion

3.1 Characteristics of materials

Thermogravimetric analysis was conducted to examine the weight loss pattern of banana peel as suitable adsorbent feedstock. Figure 2 shows the thermogravimetric profiles of banana peel. The material is thermally unstable; it easily disintegrates with increasing temperature. Banana peel contains nearly 22% moisture and light volatiles. The liberation of heavier organic fractions occurs at 550 °C. At 900 °C, the remaining residue is 42%, which is composed of fixed carbon and minerals (Hamzat, Nasri, and Zaini 2018; Kumar and Chandrashekar 2014).

Table 1 summarizes the FTIR assignment of banana peel and its adsorbent derivatives. The broad absorption band at around 3400 cm^{-1} is generally assigned to O–H stretching vibration of alcohols and phenols, while that at 2400 cm^{-1} indicates the –NH related component. The peak at 1640 cm^{-1} is the characteristic of C=C stretching vibration of alkenes. The peak around 1400 cm^{-1} corresponds to –OH bending vibration of phenols or tertiary alcohols. The band at 1200 cm^{-1} signifies the aromatic –C–H in plane bending vibration of aromatic ring, while the band at 1025 cm^{-1} is associated with the stretching vibrations of –P–O–C in aliphatic phosphates. Only banana peel exhibits a broad absorption band at 2930 cm^{-1} that is generally assigned to methylene C–H asymmetric/symmetric stretching vibration.

Figure 3 displays the morphology of BP600, R2.5 and R2.5A. The surface of adsorbents is irregular and rough with splits. The effect of H_3PO_4 activation is visible on the surface of R2.5 and R2.5A as opposed to BP600. The development of pores occurs when the reaction between the carbon atoms and the dehydrating agent is promoted in the extended interlayers of carbon matrix (Tang and Zaini 2015).

Table 2 summarizes the textural characteristics and adsorption capacities of adsorbents. R2.5 and R2.5A exhibit high specific surfaces of 897 m^2/g and 877 m^2/g, respectively. The capacity of malachite green is higher for R2.5A which is 582 mg/g, while R2.5 exhibits a greater methylene blue of 503 mg/g. For both activated carbons, the predicted Langmuir expressions are $q_e = 69.7C_e/(1 + 0.1C_e)$ and $q_e = 3.30C_e/(1 + 0.006C_e)$, respectively. Despite the comparable attributes of both activated carbons, the varying performance demonstrated in dye adsorption could be resulted due to intrinsic characteristics of dyes such as molecular size, steric hindrance, charge density, etc.

3.2 Two-stage adsorber design

A two-stage adsorber design was proposed for R2.5A and R2.5 using Langmuir model to minimize the amount of activated

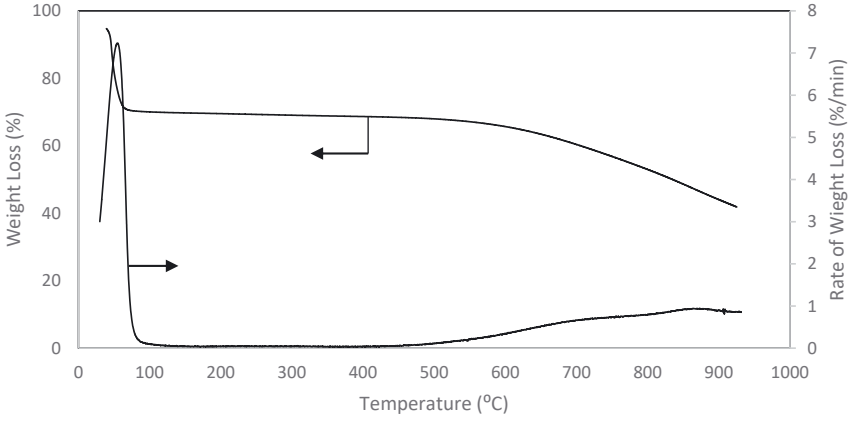

Figure 2: Thermal degradation profiles of banana peel.

Table 1: FTIR assignment of banana peel and its adsorbents.

Peak (cm^{-1})	Assignment	Functional group
3400	–OH stretching	Alcohols and phenols
2347	Amino-related component	Aminos
1640	C=C stretching	Alkenes
1407	–OH bending	Phenol or tertiary alcohols
1202	C–H in plane bending	Aromatic ring
1025	–P–O–C stretching	Aliphatic phosphates
519	–C–I stretching	Aliphatic iodo compounds

Table 2: Textural properties and dye capacities of adsorbents.

Adsorbent	BET area (m^2/g)	Average pore width (nm)	Malachite green capacity (mg/g)	Methylene blue capacity (mg/g)
BP600	5.0	21.0	222	70.1
R2.5	897	1.68	386	503
R2.5A	877	1.56	582	337

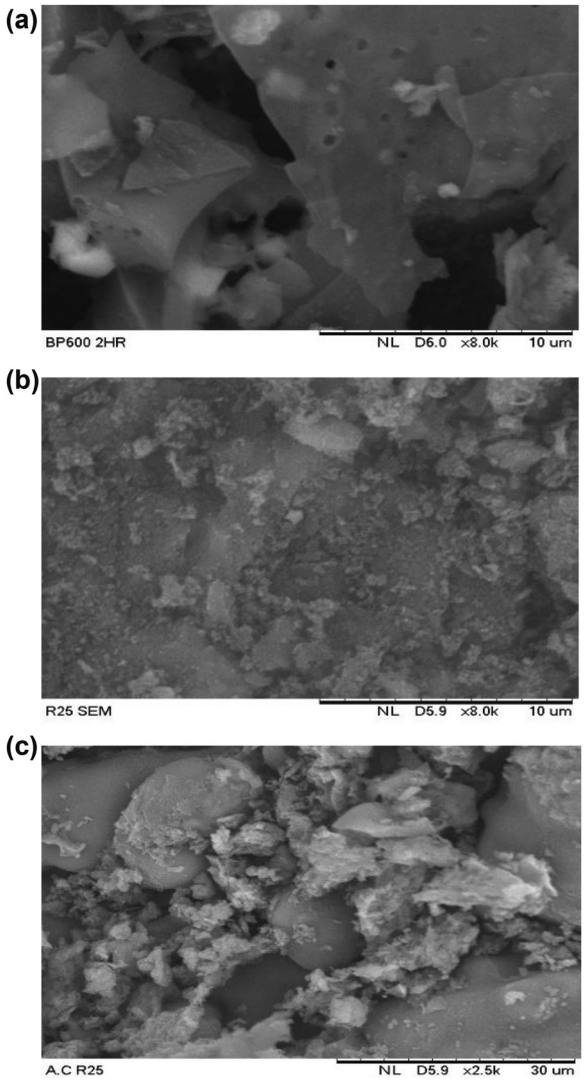

Figure 3: SEM images of (a) BP600, (b) R2.5, and (c) R2.5A.

carbon required for malachite green and methylene blue adsorption, respectively. The required amounts of R2.5A and R2.5 to reduce the effluent concentrations to 0.5–50 mg/g, and to achieve the performance of 80–99% are visualized in Figures 4 and 5, respectively.

In Figure 4(a–b), C_1 is lower for greater performance because of low equilibrium to compensate for the remaining removal in the second stage. In other words, the amount of R2.5A used in the first stage is higher for higher target performance. As the malachite green removal increased, the amount of R2.5A also increased with C_o. This pattern is also true for R2.5 profiles against methylene blue removal. From Figure 4(c), to achieve a 99.9% malachite green removal, the minimum dosages of R2.5A required for a two-stage adsorber are 46.3 g and 126 g for C_o = 100 mg/L and 2000 mg/L, respectively. Likewise, the dosages required for methylene blue removal by R2.5 at the same concentrations are greater at 169 g and 274 g, respectively. R2.5A requires a lesser dosage than R2.5 to achieve the desired removal performance of dyes, which is also true for any target effluent concentrations. It implies the affinity (favourability) of malachite green adsorption onto hydrochar activated carbon.

In Figures 4(d) and 5(d), the efficiency for the second stage is always lower than that of the first stage, as the intermediate concentration, C_1 is lower. Likewise, the efficiency for the first stage is significant for high removal performance using high C_o (Hijab et al. 2020). At high C_2, the efficiency of the second stage is minimal. However, the gap of efficiency in the second stage is broadened when the target performance is high, and the influent concentration is low. Therefore, adding a second stage even for low C_2 is essential to achieve the desired removal efficiency with minimum dosage of R2.5A and R2.5.

3.3 Statistical indicators

Tables 3 and 4 summarize the fit statistics and analysis of variance (ANOVA) of RSM quadratic model for methylene blue and malachite green adsorption in two-stage adsorber design. To determine the maximum removal and minimize the amount of adsorbent, three adsorption parameters, namely initial concentration (C_o, 100–2000 mg/L), adsorbent

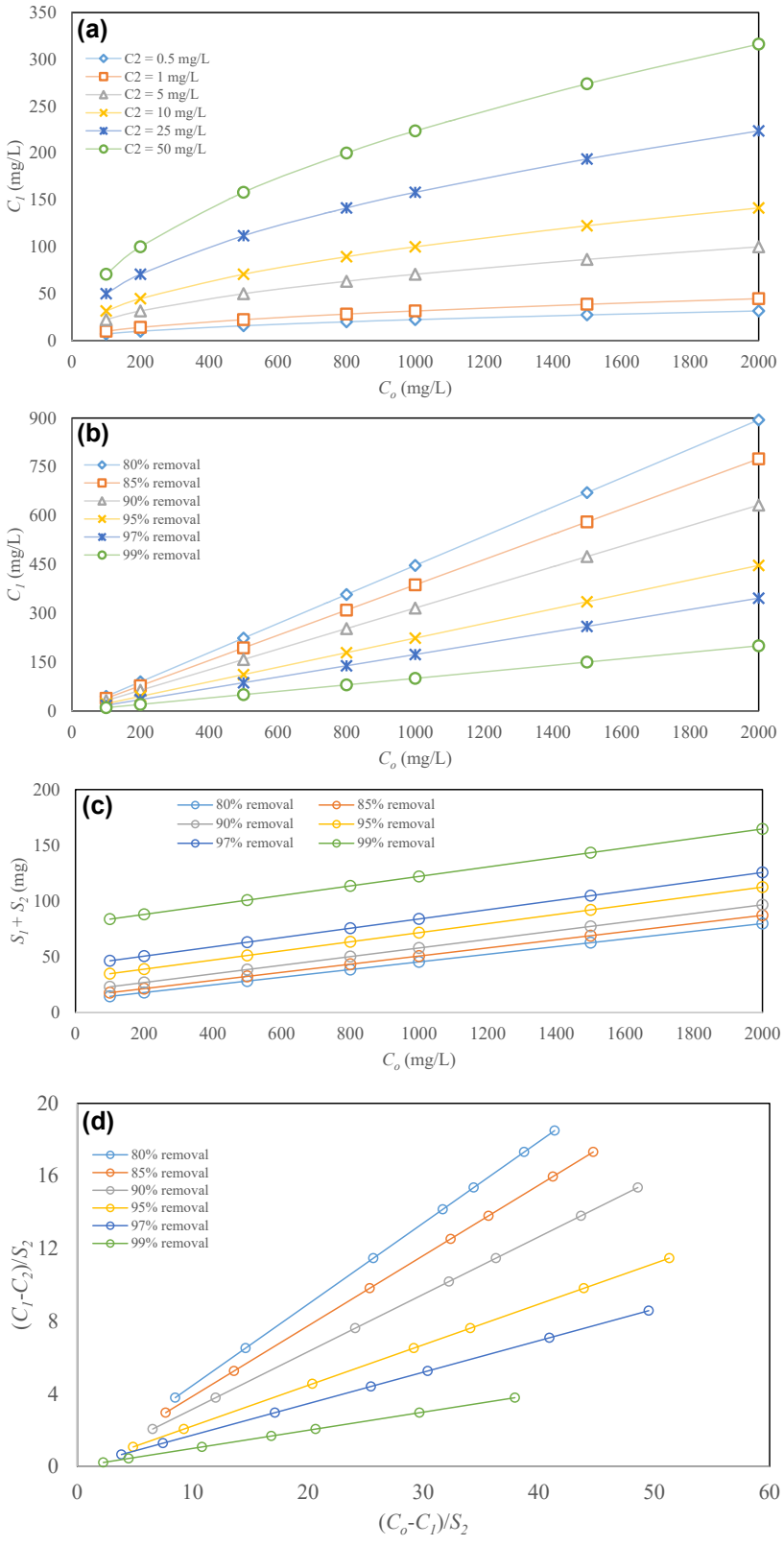

Figure 4: Two-stage adsorber design for malachite green removal by R2.5A. (a) Intermediate concentration, C_1 against influent concentration, C_o for target effluent concentrations; (b) intermediate concentration, C_1 against influent concentration, C_o at different removal performance; (c) combined mass of R2.5A for various target effluent concentrations; (d) malachite green removal in each stage for various removal performance.

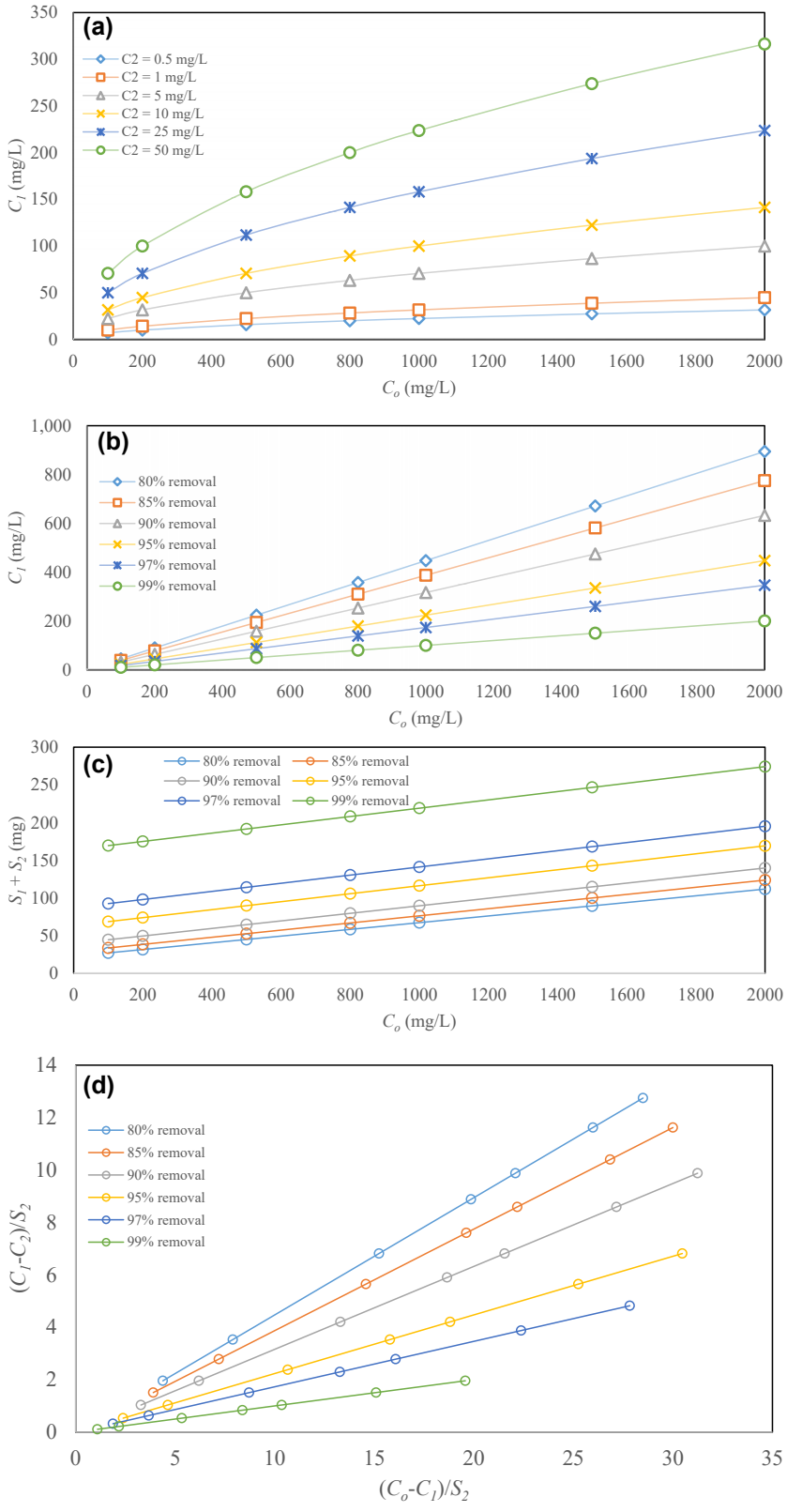

Figure 5: Two-stage adsorber design for methylene blue removal by R2.5. (a) Intermediate concentration, C_1 against influent concentration, C_o for target effluent concentrations; (b) intermediate concentration, C_1 against influent concentration, C_o at different removal performance; (c) combined mass of R2.5 for various target effluent concentrations; (d) methylene blue removal in each stage for various removal performance.

Table 3: Fit statistics of RSM model by Box-Behnken design matrix for methylene blue and malachite green removal in two-stage adsorber design.

	Methylene blue removal	Malachite green removal
Standard deviation	3.07	4.50
Mean	91.9	92.5
C.V. %	3.34	4.86
R^2	0.921	0.808
Adjusted R^2	0.778	0.561
Predicted R^2	−0.220	−1.90
Adequate precision	7.89	6.38

Table 4: Simplified ANOVA for RSM quadratic model.

	Methylene blue removal		Malachite green removal	
	***F*-value**	***p*-value**	***F*-value**	***p*-value**
Model	6.46	0.0268	3.27	0.0663
A (C_o)	21.3	0.0058	12.0	0.0106
B (S_1)	1.33	0.301	0.0556	0.820
C (S_2)	4.31	0.0926	1.04	0.341
AB	2.66	0.164	0.606	0.462
AC	3.83	0.108	2.78	0.139
BC	10.6	0.0224	1.24	0.303
A^2	0.221	0.658	0.0255	0.878
B^2	8.86	0.0309	9.07	0.0196
C^2	5.52	0.0655	2.00	0.200
Lack of fit	15.0	0.0631	20.1	0.0071

dosage in stage 1 (S_1, 14–182 g) and adsorbent dosage at stage 2 (S_2, 13–92 g) were varied, while the other adsorption parameters were kept constant.

The negative predicted R^2 implies that the overall mean shows a better prediction of response than the current model. In some cases, a higher-order model may offer a better prediction. Adequate precision is a measure of signal-to-noise ratio, in which a ratio greater than 4 is desirable. For both methylene blue and malachite green removal, the models can be used to navigate the design space because of the adequate signal.

From Table 4, the model *F*-value of 6.46 for methylene blue removal implies the model is significant. There is only a 2.68% chance that an *F*-value is large due to noise. The *p*-value less than 0.05 (95% confidence level) indicates the model terms are significant. In this case, A, BC, B^2 are significant model terms for methylene blue removal, while A and B^2 for malachite green removal. Values greater than 0.1 signifies that the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve the reliability of the model. The lack of fit *F*-value of 15.0 for methylene blue removal suggests that there is a 6.31% chance of noise to elevate a lack of fit. For malachite green removal, a value of 20.1 indicates that the lack of fit is significant, as there is only a 0.71% chance that a lack of fit F-value this large could occur due to noise. Generally, a relatively low probability (<10%) for lack of fit is undesirable.

4 Conclusions

Activated carbons were prepared from banana peel to assess two-stage adsorber design for malachite green and methylene blue. Despite a comparable specific surface of about 890 m^2/g, the activated carbons demonstrate distinct capacities of malachite green and methylene blue. The maximum capacity of malachite green was recorded at 582 mg/g by hydrochar activated carbon (R2.5A), while banana peel activated carbon (R2.5) exhibits a maximum methylene blue capacity of 503 mg/g. In two-stage adsorber design, the minimum dosage of R2.5A is smaller compared to that of R2.5 for the removal of dye at any target effluent concentrations, signifying the favorability of malachite green adsorption. The efficiency of the first stage should be sufficient enough to allow the removal of high dye concentration, while the second stage is necessary to accomplish the removal process with minimum dosage of activated carbon. Banana peel is a promising activated carbon feedstock for dye wastewater treatment. Notwithstanding that, the effects of various competing constituents on dye adsorption characteristics in a real wastewater need to be further investigated for practical applications.

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