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THE REMOVAL OF METHYLENE BLUE USING BANANA STEM/SUGARCANE BAGASSE HYBRID ADSORBENT AND ITS KINETIC STUDY

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Abstract

Environmental pollution from dye-used industry not only in the form of dust contamination but in the form of water pollution as well. The wastewater from spillage and washing water where dyeing of starch solution and staining process causes the damage to the river ecosystem. This occurrence of water pollution will disrupt the aquatic life, decrease the qualities of the water, as well as causing the biological, physical and chemical consequences. Thus, environmentally-safe water treatment is significantly required to treat the textile wastewater before discharging the effluent. This study was to evaluate the potential of hybrid adsorbent made from banana stem and sugarcane bagasse to remove methylene blue dye in aqueous solution. The dried materials were treated with Potassium hydroxide, KOH for 24 hours to remove its lignin as well as to activate its cellulose part before weighted them evenly to produce hybrid adsorbent. The batch experiments were conducted by mixing two different adsorbent dosages (0.2 g and 0.6 g) into three different initial concentrations of methylene blue; 40 ppm, 70 ppm and 100 ppm each in a conical flask with total contact time of 90 minutes for each sample. The concentrations of methylene blue solution were evaluated for every 15 consecutive minutes using ultraviolet-visible spectrophotometry (UV-VIS). Two kinetic models which pseudo first-order and pseudo second-order model were applied to understand the mechanism and behaviour of these adsorption processes. Overall, methylene blue adsorptivity by both hybrid adsorbent dosages were seen to directly proportional to contact time and more likely to have desorption until the equilibrium condition had achieved. Meanwhile, 0.6 g of hybrid adsorbent had shown higher efficiency of adsorption. The best adsorption activity was at the initial dye concentration which was 40 ppm using 0.6 g of hybrid adsorbent which resulted a maximum adsorption of 97.3% within 90 minutes. The adsorption process in this study followed the pseudo second-order model with R^2 is 0.999. This research has proved the capability of hybrid adsorbent to remove methylene blue in aqueous solution and is recommended to be used in the wastewater treatment.

Keywords: Adsorbent, methylene blue, hybrid absorbent, kinetic study

1.0 INTRODUCTION

A dye is an organic compound that can be used as permanent colour to fabric. Most industries such as textile industry, plastic industry, cosmetic industry and pharmaceutical industry use dye in their daily process. The discharged of dyes from industries are toxic to aquatic life and lead to introduce the potential danger of bioaccumulation that can effect human through food chain. The dyes presence decrease light penetration in water bodies and subsequently prevents the photosynthesis of marine flora. It also

leads to few minor health effect; allergy, dermatitis, skin inflammation, which can cause risk cases such as cancer and cell mutation in humans.

A type of dye such as methylene blue which is appeared as a solid at room temperature, odourless, and dark green powder will form blue solution when dissolve in water. Its molecular formula is $C_{16}H_{18}ClN_3S$ and the molecular weight is 319.85 g/mol. Another name of methylene blue is methylthioninium chloride [1]. Methylene blue has many uses whether in industry of textile, leather, paper, plastics, etc. These industries use dyes in order to colour their products and also consume substantial volumes of water. As a result, they generate a considerable amount of coloured wastewater [2].

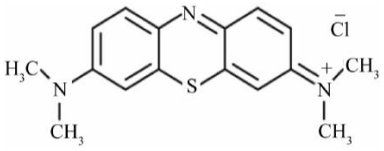

Fig 1. Methylene Blue Structure

Even there are lots of uses of methylene blue in many fields, methylene blue also has its own side effect which are include abnormal urine or stool colour, mild bladder irritation, dizziness, headache, increased sweating, nausea, vomiting, causes eye burns which prime to eternal injury to the eyes of organisms [3]. In addition, the dye has a highly toxicity which give a bad effect on the aquatic lives. It will reduce the oxygen content of water and this subsequently prevents the photosynthesis of marine life then hinders the gases solubility in water [4].

Despite, colour containing dyes is essential to be removed due to the amount of dyes (below 2.0 ppm) [5]. In previous practice, methylene blue effluent was treated by biological process as well as chemical method. The chemical methods such as coagulation, oxidation, reverse osmosis, ion exchange, sorption, membrane separation electrochemical method, flocculation and ultra-filtration [6] which known as an expensive method. Nowadays, adsorption using low cost and high cellulosic contained adsorbent is amongst the most effective technique to remove dyes [7, 8].

Adsorption process is a phenomenon in which solid and liquid molecule get adhered on the surface of porous solid. Adsorbent can absorb matters due to its natural porosity with high surface area. Adsorbent are used normally with diameter size of 0.5 mm to 10 mm in the form of rods, moulding or spherical pellets. The characteristics of good adsorbent are having high thermal stability and small pores. The small pores provide high surface area hence increase the adsorption capacity [9].

Heavy metal can be treated by any materials that contains high cellulose content. For example, bagasse that containing 36% of cellulose was proved as a great bio-sorbent [10]. Banana stem is also a bio-material that containing cellulose as much as 39.12%, 72.71% of holocellulose, 8.88% of lignin and 1.90% of acid soluble lignin [11]. Carboxyl and hydroxyl functional group in cellulose provide an active site for the metal [12]. A research on colour removal showed that, red colour and acid brilliant blue in aqueous solution have effectively being removed by the banana pith through adsorption mechanism [13].

Banana stem such as *Musa paradisiaca* and sugarcane bagasse known as *Saccharum officinarum* are waste product that are produced from agriculture. Banana stem will be left abundantly in plantation and sometimes being burned or being used as fertilizer [14]. Meanwhile, sugarcane bagasse will be thrown away because of its unpleasant odour. However, both plants-waste have been widely used as a single form of natural dye adsorbent and proven to be a good adsorbent as per studied by few researchers [6,7,8,9,13,14]. Hence, the combination of banana stem and sugarcane bagasse as one adsorbent is possible to be produced and is expected to enhance the effectiveness in dye adsorption process. This conversion of plant-waste to an added-value product such as a hybrid adsorbent will benefit in the wastewater management.

The focus of this research was (1) to prepare the hybrid adsorbent from banana stem and sugarcane bagasse; (2) to evaluate the adsorption potential of hybrid adsorbent in the removal of methylene blue in aqueous solution; (3) to study evaluate the effect of adsorbent dosages (g), initial methylene blue concentration (ppm) and contact time (min) on adsorption process; (4) to identify the best kinetic model fitted to the optimum adsorbent parameters.

2.0 EXPERIMENTAL

2.1 Adsorbent preparation

Banana stem and sugarcane bagasse were collected from nearby village. The materials were cut into small sizes, washed with tap water and boiled for about two hours to remove its sap and sugar. After being boiled, the samples were washed with distilled water to remove impurities before dried it under the sunlight. It was further dried in the oven with temperature of 55°C for a day to remove excess water. For treatment, the dried samples were soaked in Potassium hydroxide, KOH for 24 hours for a day to remove its lignin and to activate its cellulose content. The treated samples were washed with distilled water and dried again in the oven. The samples were weighted until the equilibrium moisture content was obtained. After the process, the treated samples were ground and sieved to 250 micron size (Fritsch, model: Analysette 3) [15] before the sorbent was formulated. Table 1 shows the sorbent formulation.

Table 1. Hybrid adsorbent formulation

Dosage (gram)		
Treated banana stem	Treated sugarcane bagasse	Total hybrid adsorbent
0.1	0.1	0.2
0.3	0.3	0.6

2.2 Methylene blue solution preparation

Methylene blue was supplied by R&M Chemical, Malaysia. 1 gram of methylene blue powder was dissolved in 1000 mL of distilled water to prepare a stock of 1000 ppm of methylene blue solution. Different concentration of methylene blue solutions; 40 ppm, 70 ppm and 100 ppm were formulated using Eq. 1 [16] and the calibration standard was presented in Table 2. The pH was adjusted to pH 3 by slowly titrate hydrochloric acid to the solution.

$$(V_{stock} \times C_{stock}) = (V_{calibration} \times C_{calibration}) \quad (1)$$

where V_{stock}, C_{stock}, $V_{calibration}$ and $C_{calibration}$ is volume of the stock standard being used, concentration of the stock standard being used, desired volume of the calibration standard and desired concentration of the calibration standard, respectively.

Table 2. Methylene blue calibration standard

V_{stock} (ml)	C_{stock} (ppm)	$V_{calibration}$ (ml)	$C_{calibration}$ (ppm)
28	1000	700	40
49	1000	700	70
70	1000	700	100

2.3 Adsorption experiments

In kinetic study, a batch of conical flask contains 50 mL of different concentrations of methylene blue solution; 40 ppm, 70 ppm, and 100 ppm, were added each with 0.2 g and 0.6 g of hybrid adsorbent. The conical flasks were shaken using an orbital shaker (Wisd; Model: SHO-2D) for 90 minutes of contact time at 120 rpm. The concentration of the methylene blue solution was determined for every 15 minutes [17]. The experimental conditions were at 25°C and 1 atm.

2.4 Adsorption equilibrium study

For every 15 minutes of contact time, the samples were filtered to minimize interference of the sorbent using centrifuge at 150 rpm at room temperature. The residual solutions were analysed using UV-Vis spectrophotometer (Varian; Model: Cary 50, Agilent Technologies) with a wavelength of 664.9 nm. The adsorption capacity at equilibrium was calculated using Eq. 2 [18].

$$q_e = \frac{(C_o - C_e)V}{m} \quad (2)$$

Where C_0 and C_e (mg/L) are the liquid-phase concentrations of dye at initial and equilibrium, respectively. V is the volume of the solution (L), and m is the mass of dry adsorbent used (g).

Calibration equation was presented as Eq.3.

$$Abs = 0.13984*conc + 0.25316 \quad (3)$$

2.5 Adsorption kinetics study

The adsorption kinetics study is very beneficial in order to understand the mechanism involved during the adsorption process such as chemical reaction and mass transfer. It also important in any designing of large-scale adsorption facilities. Amongst kinetic models used to fit the kinetic adsorption experiments, the most popular are the pseudo-first order equation and pseudo-second order equation.

The procedures of kinetic experiments were similarly as the procedure of adsorption equilibrium study. It started by finding the value of adsorption capacity at 15 minutes of contact time intervals, and the concentrations of dye were measured by UV-Vis. The adsorption capacity at time t was calculated using Eq. 4. [19].

$$q_t = \frac{(C_o - C_t)V}{m} \quad (4)$$

where C_0 and C_t (mg/L) are the liquid-phase concentrations of dye at initial and any time, t respectively. V is the volume of the solution (L), and m is the mass of dry adsorbent used (g). The rate constant of adsorption was determined from the pseudo first-order equation given by Langergren and Svenska [20] as shown in Eq. 5.

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (5)$$

where q_e and q_t are the amounts of methylene blue adsorbed (mg/g) at equilibrium and at time t (min), respectively, and k_1 is the rate constant adsorption (1/min). Value of k_1 was obtained from the plots of $\ln(q_e - q_t)$ versus t for different initial concentrations of methylene blue.

On the other hand, a pseudo second-order equation based on equilibrium adsorption [21] was stated as Eq. 6.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (6)$$

where k_2 (g.min/mg) is the rate constant of second-order adsorption. It can obtained from the interceot value from the plot of t/q_t versus t, that showing a

linear relationship. The value of q_e also can be determined from the slope of every linear equation produced. This method is apply to predict the behaviour of the adsorption process.

Despite the value of R^2, the sum of squared error (SSE, %) is also used to verify the validity of both kinetic models using Eq. 7 [21].

$$SSE\ (\%) = \sqrt{\frac{\sum(q_{e,exp} - q_{e,cal})^2}{N}} \qquad (7)$$

Where *N* is the number of data points.

3.0 RESULTS AND DISCUSSION

3.1 Effect of contact time

Contact time is among the important factors in adsorption process. The experimental outcome can be seen in Fig. 2 and Fig. 3 which showed that the adsorption occurred and achieve equilibrium within 90 minutes. At initial contact time, the amount of the methylene blue onto hybrid adsorbent increases with time. However, at sometimes, the measurement showed a constant or fluctuate value which either no more was removed from solution or desorption occurred. This may be due to the strong attractive forces between the methylene blue molecules and the adsorbent.

Factors such as high solute concentration gradient and all adsorbent sites were still vacant at initial times, could be the causes towards changing in the methylene blue removal rate [21]. In addition, there was no significant change in methylene blue starting from minute 15 to minute 90, reflected on dynamic equilibrium had occurred during the adsorption-desorption process. It was also reflected that the highest adsorption capacity of the absorbent has been achieved under all operating conditions.

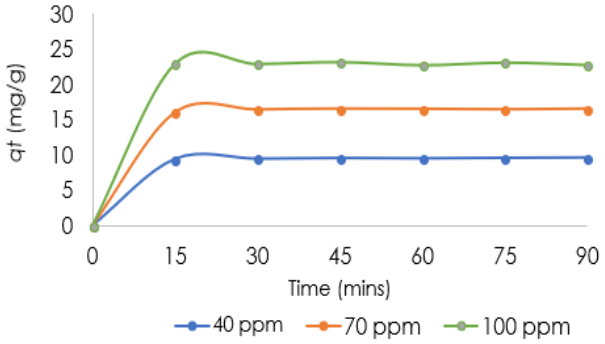

Fig. 2 The variation of adsorption capacity of 0.2 g sorbent with time (pH3, 30°C)

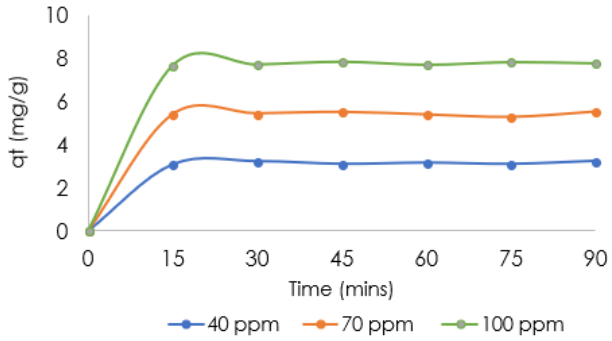

Fig. 3 The variation of adsorption capacity of 0.6 g sorbent with time (pH3, 30°C)

However, desorption occurred at contact time of 60 minutes and 90 minutes using 0.2 g at 100 ppm. Similar desorption behaviour was observed at contact time of 75 minutes using 0.6 g over 70 ppm of methylene blue. The decrease adsorption rate, predominantly indicates the possible monolayer formation of methylene blue on the adsorbent surface [22]. Lack of accessible active sites required for further uptake after attaining the equilibrium may be also a cause for desorption [23].

The effect of contact time was also investigated by Nharingo [26] using initial dye concentration of 60 ppm with single sugarcane bagasse sorbent dosage of 0.2 g at 25°C temperature and agitation rate of 150 rpm over a period of 180 minutes. The finding stated that the adsorption was rapid at the initial stages and gradually decreased with time until it became constant in 80 minutes after contacted the sugarcane absorbent and the methylene blue. This showed that a single form of sugarcane bagasse adsorbent has lower adsorptivity effect compared to the hybrid one thus taking more time to complete the adsorption/desorption activity.

3.2 Effect of adsorbent dosage

The effect of adsorbent dosage on removal percentage of methylene blue was investigated. Fig. 4 shows the removal percentage of methylene blue by two different adsorbent dosages which were 0.2 g and 0.6 g. As can be seen in the figure, the adsorption efficiency of the hybrid adsorbent for the removal of methylene blue increases with increasing adsorbent dosage at all initial methylene blue concentration. The highest removal percentage was obtained using 0.6 g sorbent over 40 ppm methylene blue concentration as much as 97.3% within 90 minutes.

This result contradicts with the previous study by Nurul *et. al* [15] whereas the highest percentage removal of Methylene Blue obtained was by 0.6 g of a

single banana stem adsorbent as much as 91.47% within 90 minutes. Therefore, the hybrid adsorbent has obviously empowered the removal of methylene blue compared to the banana stem adsorbent which existed alone.

Besides, the minimum removal percentage of methylene blue was recorded as much as 90.9% in 100 ppm methylene blue concentration using 0.2 g adsorbent within 90 minutes. Indeed, the total number of active sites increased with increasing adsorbent dosage. This result was also supported by other researchers who reported that the higher adsorbent dosages provided to the greater adsorption of methylene blue. The availability of more adsorption sites due to larger adsorbent surface area was contributed from the larger amount of dosages [24].

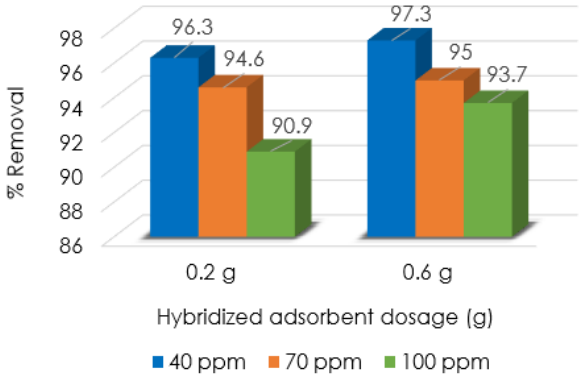

Fig. 4 Removal percentage of methylene blue using different sorbent dosage (g)

3.3 Effect of Initial Concentration

Initial concentration of methylene blue plays a significant role in the adsorptivity process. The experiment were carried out with three initial methylene blue concentrations (40 ppm, 70 ppm and 100 ppm) at constant temperature (25°C) and pH3. As can be seen in Fig.5, the removal percentage of methylene blue was found to decrease with increasing in initial dye concentration. It can be observed from the experimental result, the removal percentage of methylene blue using 0.2 g of sorbent decreased from 96.3 % to 94.6% and then to 90.9% when 40 ppm, 70 ppm and 100 ppm were used as the initial concentration, respectively. The same behaviour was obtained when using 0.6 g as adsorbent.

At lower methylene blue concentration, the ratio of the available adsorption sites of adsorbent to the initial number of methylene blue molecule is large and consequently the fractional adsorption becomes independent of initial concentration. However at higher concentration, the available site of adsorption process become saturated, hence methylene blue removal percentage which depends upon the initial concentration, decreases [25].

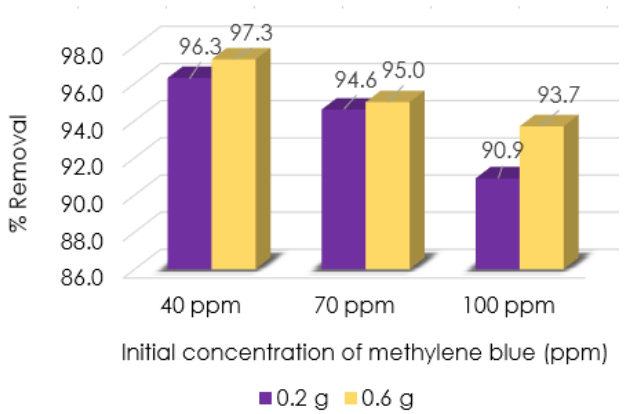

Fig. 5 Removal percentage of methylene blue using different initial concentration

3.4 *Adsorption kinetics*

As 0.6 g was identified to be the best adsorbent dosage in this study, hence the data from its kinetic experiments adsorbent were further analysed and fitted to kinetic model equations. The best fit model was selected based on coefficients correlation, R^2 whereby the higher correlation (R^2>0.99) gives the best fit model to the kinetic data. Two kinetic models were applied to model the rate of methylene blue adsorption hybrid adsorbent.

The pseudo first-order kinetic equation describes that adsorption rate is based on adsorption capacities, where it assumes that one adsorbate molecule is adsorbed onto one active site [4]. The kinetic data were as well analysed using Ho's pseudo second-order kinetics model. This model is based on the assumption the sorption follows second-order chemisorption [21]. The pseudo second-order equation was also used to describe adsorption rate based on adsorption capacities [5]. In this equation, one adsorbate molecule is assumed to be held onto two active sites.

For the pseudo first-order model, it can be seen in Table 3 and Fig. 6, the correlation coefficient value, R^2 at all initial methylene blue concentrations were found to be laid in the range of 0.5 to 1.0 which determined a strong linear relationship. However, the variance between all experimental $q_{e,exp}$ values and calculated $q_{e,cal}$ values were very high for about 40% to 80% higher than the experimental $q_{e,exp}$ value. These results explain a poor fit between the experimental and calculated data. Therefore, adsorption of methylene blue onto hybrid adsorbent is not following the pseudo first-order reaction.

Fig. 7 shows the linear plots of t/q versus t, whereby the data were obtained from the pseudo second-order kinetics model. As can be seen, the results showed a close value between the calculated $q_{e,cal}$

values with the experimental $q_{e,exp}$ which tabulated in Table 4. At all initial dye concentrations, straight lines with extremely high correlation coefficients, R^2 (>0.99) were obtained. This indicated the applicability of the pseudo second-order model and its adsorption mechanism of methylene blue on hybrid sorbent. From this study, the best model was selected depends on the highest value of R^2 and its value of SSE %, which generally showing the lower value. For this current research, the adsorption of methylene blue on hybrid adsorbent were best described by the second-order kinetic model.

Table 3. The pseudo first-order adsorption data at different initial dye concentrations

MB conc. (mg/L)	$q_{e,exp}$ (mg/g)	Pseudo first-order rate equation			
		K_1 (min^{-1})	R^2	$q_{e,cal}$ (mg/g)	SSE(%)
40	3.250	0.045	0.691	2.471	0.294
70	5.543	0.055	0.496	2.600	1.112
100	7.890	0.042	0.568	1.92	2.256

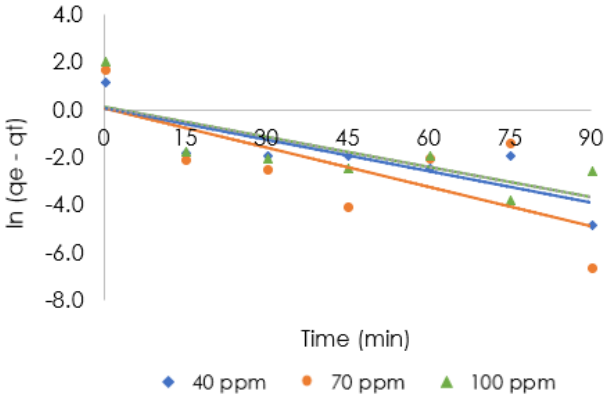

Fig 6. Pseudo first-order kinetics for methylene blue adsorption by 0.6 g

Table 4. The pseudo second-order data for different initial dye concentrations

MB conc. (mg/L)	$q_{e,exp}$ (mg/g)	Pseudo second-order rate equation			
		K_2 (gmin/mg)	R^2	$q_{e,cal}$ (mg/g)	SSE %
40	3.250	0.429	0.999	3.200	0.019
70	5.543	3.273	0.999	5.447	0.036
100	7.890	0.697	0.999	7.831	0.022

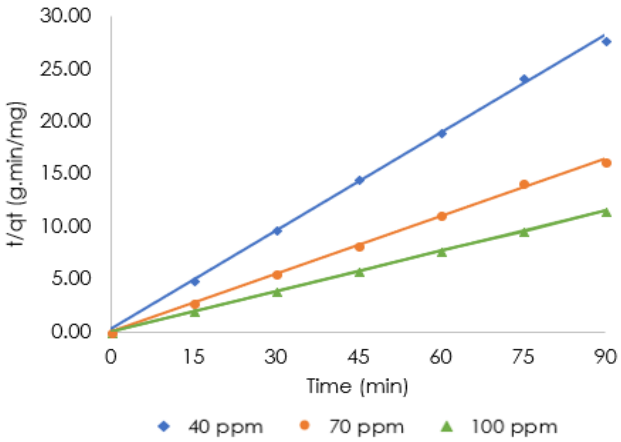

Fig 7. Pseudo second-order kinetics for methylene blue adsorption by 0.6 g

4.0 CONCLUSION

Experimental data indicated that the adsorption capacity was strongly reliant on operating parameters such as contact time, adsorbent dosage, and initial dye concentration. The contact time was found to directly proportional to the removal percentage or adsorption capacity of methylene blue solution. Lower initial concentration of solution was proven suitable for this type of adsorbent because the higher the initial concentration, the higher the tendency of desorption to occur. The best adsorption capacity was at the initial concentration of 40 ppm methylene blue by 0.6 g of adsorbent which resulted as much as 97.3% of removal percentage in 90 minutes. It was found the R^2 values (>0.999) of the pseudo-second-order model was much higher than pseudo-first-order model. The calculated $q_{e,cal}$ values of the pseudo-second-order model were all closed to experimental $q_{e,exp}$ values. Hence, the pseudo-second-order kinetic model was more suitable to describe the adsorption behaviour of methylene blue over the hybrid adsorbent.

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