

Full paper

Isotherm Studies of Methylene Blue Adsorption onto Potassium Salts-Modified Textile Sludge

Tang Shu Hui^{a,b}, Muhammad Abbas Ahmad Zaini^{a,b*}

^aCentre of Lipids Engineering & Applied Research (CLEAR), Ibnu Sina ISIR, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Malaysia ^bFaculty of Chemical Engineering, Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Malaysia

*Corresponding author: abbas@cheme.utm.my

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Abstract

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Graphical abstract



In the present work, the adsorption of methylene blue from aqueous solution by textile sludge activated with potassium salts and a composite-KI, KCl, K_2SO_4 and KOH+KCl-was investigated. The impregnation ratio for all activating agents was set at 1:1 and the impregnated textile sludge samples were activated at 500°C for 1h in a furnace. The specific surface area of the activated carbon (AC) was evaluated using BET analysis. The BET surface area varied from 78.8 to $321m^2/g$, where it decreased in the order of K_2SO_4 -AC > KCl-AC > KI-AC > KOH+KCl-AC. Under room temperature condition and a shaking time of 72h, the batch adsorption results show that the minimum and maximum adsorption capacities were recorded as 39.1 and 85.0mg/g for textile sludge activated with KCl and KI, respectively. Langmuir isotherm model gave the best fit to adsorption equilibrium compared to The Langmuir mechanism yielded a maximum monolayer adsorption capacity of 90.9mg/g. Among the four prepared activated carbons, textile sludge activated with potassium iodide (KI-AC) gave the best performance on the adsorption of methylene blue.

Keywords: Activated carbon; adsorption; isotherm; methylene blue; potassium salt; textile sludge

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1.0 INTRODUCTION

Each year, it is estimated that over 7×10^5 tonnes of dyestuff are manufactured with more than 100,000 commercial dyes available globally [1]. Up to 30% of the industrial dyes are wasted in the textile wastewater discharged from various factories including textile, leather, paper, cosmetic plastics and others. Methylene blue (MB) is a common textile dye which is extensively employed in imparting colour onto cotton, wool and silk. Although MB is not considered critically hazardous, it triggers eye burn which in turn may result in irreversible damage to the eyes. Furthermore, it can cause short period of rapid or difficult breathing if accidentally inhaled, whereas ingestion via the mouth gives a burning sensation and may lead to vomiting, profuse sweating, nausea and mental confusion [2]. On the other hand, exposure to large quantity of MB causes the victim to experience acute headache, abdominal and chest pain, mental confusion, profuse sweating, painful micturition and methemoglobinemia-like syndromes [3]. Thus, the removal of methylene blue from water bodies is extremely crucial in maintaining the well-being of life forms.

In the recent times, the rate of sewage sludge generation is escalating in proportion to the advancing development, manufacturing and population growth. The sewage sludge and waste had to be properly handled in order to comply with the stringent environmental regulation. Although these wastes can be disposed through land-filling, incineration and composting for farmland usage [4], these techniques are only a short-term solution as the decreasing landfill area causes fierce rivalry. Besides, the cost related to waste disposal (dewatering and incineration) also elevates and the environmental regulations are gradually becoming stricter and stern [5]. The incineration of sewage sludge is a complicated method as the pollutants produced must be strictly monitored control and it has often received public and political opposition.

One of the economical and effective solutions is to convert these sludge wastes into fresh activated carbons or adsorbents. Monsalvo *et al.* (2011) [5] proved that a significantly high BET surface area of 1832 m^2/g is elicited from the activation of sewage sludge with 1:1 KOH at 750°C. Generally, activated carbon can be prepared either by chemical activation, physical activation or combination of both. Chemical activation prevails over physical activation as it is a one-step reaction (simultaneous carbonization and activation) and has lower activation temperature and higher product yield [6]. It was discovered that these porous sludge derived adsorbents were comparable to other highly porous activated carbons in numerous applications.

Decolourization of industrial dye wastewater by traditional means like biological and coagulation-flocculation treatment had basically displayed unsatisfactory removal performance. Adsorption technique is one of the most successful and advantageous treatment methods in dye removal as it does not only possess a simple operation and design. Activated carbon predominates among other adsorbents in the adsorption process due to its big porous surface area, high surface reactivity and good adsorption efficiency. Nevertheless, the present commercial activated carbon derived mostly from charcoal is expensive. Thus, the current investigations are concentrated on the conversion of divergent biomass waste for instance wheat shell [7], orange peel, banana peel [8], corncob [9] and peanut hull [10] into affordable activated carbon. These activated carbons were employed for various applications such as removal of metal ions, adsorptive desulfurization and decolourization of dye effluents. The objective of this work is to evaluate the feasibility of textile sludge based activated carbon for the removal of methylene blue (MB). The effect of different potassium salts on the adsorption of MB were studied. Langmuir and Freundlich isotherms were used to fit the experimental data of methylene blue adsorption.

2.0 MATERIALS AND METHODS

2.1 Materials

Dried granular textile sludge was obtained from American & Efird (Malaysia) Sdn. Bhd., located in Kulai, Johor state of Malaysia. Both granular potassium iodide (KI) and potassium sulphate (K₂SO₄) were purchased from QREC (Asia) Sdn. Bhd., whereas the potassium hydroxide (KOH) pellets and granular potassium chloride (KCl) were obtained from R&M Chemicals, Malaysia and EMD Milipore Corporation, respectively. All of the chemicals used in this study are of analytical reagent (AR) grade and were used as received without further treatment or purification. Distilled water and hydrochloric acid were used as washing agents.

A cationic dye, methylene blue (MB), also known as basic blue 9 was chosen as an adsorbate and it possesses empirical formula of $C_{16}H_{18}N_3SCl$, molecular weight of 319.9g/mol and colour index (CI) of 52015. The chemical structure of methylene blue is portrayed in Figure 1 [11]. The dye was diluted to the desired concentration prior to adsorption experiment. Preparation of the working solutions of desired concentrations was performed by properly diluting the stock solution.



Figure 1 Molecular structure of methylene blue

2.2 Preparation of Activated Carbon

The granular textile sludge was dried at 110° C for 48h, followed by separated impregnation with KI, KCl, K₂SO₄, and a mixture of KOH and KCl at impregnation ratio of 1:1. The solid in liquid mixtures were heated on hot plate at 90°C for 1.5 h. The samples were left to dry in an oven for 24 h to complete the impregnation before undergoing activation in a box furnace at 500°C for 1 h. The resultant activated carbons were gradually cooled to room temperature before they were washed sequentially with HCl solution and distilled water. An acid wash is used to eliminate amine, residual organic and mineral matters, while washing with distilled water eliminates non-organic compounds including ash. The samples were rinsed and washed several times with distilled water until the pH is constant, indicating the absence of residual anions. After that, they were dried at 110°C for 24 h prior to use.

2.3 Characterization of Activated Carbon

Moisture content and ash content of raw textile sludge were calculated using Equations 1 and 2, respectively. The material was put in a beaker and heated in the oven at 110°C for 24 h. The beaker was taken out and weighed to get the final dry weight. The moisture content was calculated as,

% moisture content =
$$\frac{m_w - m_d}{m_d} \times 100\%$$
 (1)

where m_w is the weight of wet sample (g) and m_d is the weight of dry sample (g). Dry textile sludge was put into a crucible and gasified in a furnace at 900°C for 2h. The crucible was taken out and weighed to get the final weight of ash. The ash content was calculated as,

% ash content =
$$\frac{m_a}{m_d} \times 100\%$$
 (2)

where m_a is the weight of ash (g).

The yield of textile sludge-activated carbon was calculated as.

$$yield = \frac{m}{m_0} \times 100\% \qquad (3)$$

where m is the mass of activated carbon (g) and m_0 is the mass of raw textile sludge (g).

The activated carbons were characterized using Brunauer-Emmett-Teller (BET) method at 77 K, where the nitrogen adsorption-desorption isotherms were discovered using a surface and pore size analyzer (Micrometrics PulseChemiSorb 2705, USA). The activated carbon was first degassed at 300°C in an inert condition for 24 h prior to nitrogen adsorption. N₂ adsorption isotherm was acquired by employing a relative pressure range of 10^{-5} to 0.995 of nitrogen gas.

2.4 Batch Equilibrium Studies

Experimental solutions with various dye concentrations were prepared by diluting the methylene blue (MB) dye stock solution. The batch adsorption experiments were carried out for all four activated carbons. A set of 50mL MB solution with five different initial concentrations (10, 25, 50, 100, 150 mg/L) were prepared and placed in 150 mL conical flasks. About 0.05 g of activated carbon was then added into each conical flask.

The solid-solution mixtures were kept in a orbital shaker (SASTEC ST-WSZ100A) and shaken at room temperature of 30°C, and a constant speed of 120 rpm to ensure uniform particle contact. After 72 h, the mixture was filtered to separate out the spent adsorbent as carbon fines may interfere and affect the accuracy of concentration measurement. The filtrate was collected to analyse the final concentration using a UV-vis spectrophotometer (Dynamica Halo VIS-10) at $\lambda_{max} = 680$ nm. The blank samples were also prepared as control. Appropriate dilutions of dye solution were carried out whenever necessary. The equilibrium adsorption capacity (Q_e) was calculated as,

$$Q_e = \frac{(C_0 - C_e)}{m} V \qquad (4)$$

where C_0 is the initial concentration of dye solution (mg/L), C_e is the equilibrium concentration of dye solution (mg/L), *m* is the mass of activated carbon (g) and *V* is the volume of dye solution (L).

2.4.1 Langmuir Isotherm

According to Weber and Chakkravorti, (1974) [12], the Langmuir isotherm hypothesizes monolayer coverage of adsorbate over a surface, and holding a limited number of adsorption sites of uniform adsorption energy with no transmigration of adsorbate in the plane of surface. At equilibrium, no further adsorption can take place indicating a saturation point is achieved and this gives rise to the formation of a plateau graphically. Besides, it is also assumed that all sites are identical and energetically equivalent and the adsorbent is structurally homogeneous. The linearized form of Langmuir isotherm is expressed as [13],

$$\frac{C_e}{Q_e} = \frac{1}{Q_m b} + \frac{C_e}{Q_m} \tag{5}$$

where *b* is the Langmuir isotherm constant (L/mg), Q_m (mg/g) is the maximum monolayer coverage (mg/g), C_e is the equilibrium concentration of the adsorbate (mg/L) and Q_e is the amount of adsorbate adsorbed per unit mass of adsorbent at equilibrium (mg/g). By plotting C_{e}/q_e against C_e , the values for both the Langmuir isotherm constant, *b* and the maximum monolayer coverage capacities, Q_m can be determined, where the gradient of the linear plot is numerically similar to $1/Q_m$ while the yintercept is equal to $1/bQ_m$ [13].

A dimensionless constant related to the Langmuir isotherm usually known as separation factor (R_L) is utilized to study the practicability of adsorption on adsorbent. It is described as,

$$R_L = \frac{1}{1 + bC_0}$$
(6)

where *b* (L/mg) is the Langmuir constant and C_o is the initial adsorbate concentration (mg/L). The adsorption is more favourable when a lower R_L value is obtained. In short, R_L value depicts the selective nature of adsorption to four categories — unfavourable ($R_L > 1$), linear ($R_L = 1$), favourable ($0 < R_L < 1$) and irreversible ($R_L = 0$) [12].

2.4.2 Freundlich Isotherm

On the other hand, Freundlich isotherm presumes multilayer adsorption process on a heterogeneous surface. Basically, Freundlich model involves an exponential term of equilibrium concentration, and thus suggesting that the adsorbate concentration on the adsorbent surface increases along with the adsorbate concentration in solution [14]. The linear form of Freundlich equation is given as,

$$logQ_e = logK_F + \frac{1}{n}logC_e \qquad (7)$$

where K_F is the Freundlich isotherm constant related to adsorption capacity and *n* is the adsorption intensity. A straight line can be elicited by plotting log Q_e against log C_e , whereby the slope is equal to 1/n. The surface heterogeneity or adsorption strength is computed using the gradient regions between 0 and 1, whereby if the its value is near to zero, the adsorption is more heterogeneous. The value less than 1 suggests chemisorption process in which 1/n more than one implies cooperative adsorption [15].

3.0 RESULTS AND DISCUSSION

3.1 Characteristics of Activated Carbon

The moisture content and ash content of the textile sludge are 11.3 and 16.0%, respectively. The yields for textile sludge activated with KI, KCl, K₂SO₄ and a composite of KOH+KCl are 31.9, 31.1, 29.9 and 20.8%, respectively. In general, the yields are around 30% with KCl-AC possessing the highest yield, while the lowest yield was recorded for KOH+KCl-AC. This is due to the fact that KOH is a strong base or alkaline hydroxide which is capable of disintegrating the starting material into powder form owing to the formation of pores as a result of gasification reactions [16]. The potassium salts act as dehydrating agents to limit the formation of tar that obstructs the pores on the activated carbon. During activation of the impregnated samples, the metallic potassium ions intercalate into the carbon structure resulting in the low yield and the development of pores.

Nitrogen is the most extensively employed adsorbate in determining the BET surface area of carbon blacks compare to other noble gases such as argon, krypton and xenon. The BET surface area was ascertained using a single point plot mainly owing to its rapid operation and lower testing cost. The BET single point method encounters no theoretical errors and provides accurate values over a larger pressure region. The BET surface area were recorded as 191, 213, 321 and 78.8 m²/g for KI-AC, KCI-AC, K₂SO₄-AC and KOH+KCI-AC, respectively. In short, the BET surface area descended in a manner of K₂SO₄-AC > KCI-AC > KI-AC > KOH+KCI-AC.

Porous surface area were developed when the potassium metal intercalated into the carbon network which was further removed through acid washing and thus resulting in different pore sizes. The formation of potassium metal is attributed to the intermediate reaction of potassium salts (emerging from the hydrolysis of potassium metal) with the carbon in the precursor [16]. It was found that the textile sludge activated by potassium sulfate (K₂SO₄) gave an impressive BET surface area of 321m²/g. This phenomenon could be associated with the ability of sulfate to increase the boiling point and consequently hinders the excessive carbon burnt off and gasification which causes the well-formed pores to fuse and collapse. In 1889, this nonflammable and water soluble white crystalline salt was used to accelerate sulphuric acid digestion by raising the boiling point [17], but nowadays K_2SO_4 is widely used as fertilizers. Nevertheless, to our knowledge, there is no work conducted using potassium sulfate (K₂SO₄) and potassium iodide (KI) for the preparation of activated carbon.

3.2 Effect of Initial Dye Concentration

Methylene blue (MB) is a basic or cationic colorant possessing approximate dimensions of 1.43 nm×0.61 nm×0.4 nm [18]. There are four steps involved in the adsorption mechanism. These include liquid phase mass transfer where dye molecules shift from the bulk of solution to the outer surface of activated carbon; diffusion of dye molecules through the interface surrounding the activated carbon (external diffusion step); adhesion of dye molecules onto the active sites located on the outer layer of activated carbon (adsorption step); and intraparticular diffusion of dye molecules through the pores of activated carbon (internal mass transfer). In the adsorptionrelated wastewater treatment, mass transfer step is regarded as the presiding process since the dye molecules adsorb onto active sites in a rapid manner [19].

Figure 2 illustrates the equilibrium adsorption of MB by the four prepared activated carbons. A sharp increase can be seen at low concentrations signifying high affinity of adsorption towards the MB molecules. When the concentration rises further, the amount of solute adsorbed increases at a more gradual rate until almost a horizontal plateau is shown implying that the saturation is attained. At this point, nearly no more increase in adsorption would be observed. The MB adsorption capacity descended in an order of KI-AC > K₂SO₄-AC > KOH+KCI-AC > KCI-AC, where KI-AC has the best adsorption performance of 85.0mg/g while KCI-AC has the lowest removal of 39.1 mg/g.



Figure 2 Methylene blue adsorption onto activated carbons

The BET surface area is a factor determining the efficiency of adsorption process, apart from dispersion and electrostatic (functional groups) interaction. Better adsorption performance of both KI-AC (85.0 mg/g) and K₂SO₄-AC (63.3 mg/g) are attributed to their high BET surface area of 191 and $321m^2/g$, respectively. The smaller adsorption capacity of K₂SO₄-AC may reflect that it consists of mainly micropores (high BET surface area), leading to the incompatible adhesion of adsorbate molecules (MB, molecular size of 1.43nm) onto the activated carbon. On the other hand, KOH+KCl-AC demonstrated a 62.0 mg/g MB adsorption even though its BET surface area is only 78.8 m²/g. Regardless of the BET surface area, the good adsorption performance of both KI-AC and KOH+KCl-AC could be related to the presence of acidic functional group at the carbon surface and possible contribution of the mesoporous structure.

Conversely, the KCl-AC exhibits the lowest adsorption capacity of only 39.1 mg/g despite having a larger surface area (191 m²/g). This occurrence may indicate the presence of

molecular sieving effect owing to possible microporous structure of KCI-AC and the size of MB molecules. Penetration of dye molecules into the micropores on the activated carbon surface is impractical as the MB molecule is larger in molecular size. As a result, the adsorbate molecules adsorbed onto mesopores and macropores which have bigger molecular diameter than MB. Gokce and Aktas, (2014) [19] proposed that molecular sieving effect reduces the adsorption capacity as MB molecule is unable to penetrate the active sites on the activated carbon due to its larger dimension.

3.3 Isotherm Studies

Adsorption isotherms are crucial in comprehending the concept of adsorptive molecular distribution between the solid phase and the liquid phase during adsorption equilibrium. When the rate of dye adsorption is same as the desorption rate, adsorption equilibrium is achieved. In order to discover the most appropriate model for design purposes, the fitting of experimental data into various isotherm models is an indispensable step to analyze the adsorption behaviour. In this study, two common adsorption isotherm models which are Langmuir and Freundlich were used to evaluate the interaction between the adsorbate and the activated carbon.

3.3.1 Langmuir Isotherm Model

Linear lines were obtained using Langmuir equation for C_{e}/q_{e} against C_{e} as displayed in Figure 3. By letting the gradient of the straight line and the intercept equal to $1/Q_{m}$ and $1/bQ_{m}$, the value of Q_{m} and b were determined. The correlation coefficient, R^{2} value was used to test the applicability of isotherm equation in explaining the adsorption mechanism. The values of R^{2} and the Langmuir constants (*b* and Q_{m}) are tabulated in Table 1.



Figure 3 Langmuir isotherm for MB adsorption onto activated carbons

 Table 1
 Langmuir isotherm model parameters for MB adsorption on prepared activated carbon

Activated carbon	Q_e	Q_m	b	R^2	R_L
KI-AC	85.0	90.9	0.0966	0.926	0.0520
KCl-AC	39.1	39.1	0.738	0.999	0.0071
K ₂ SO ₄ -AC	63.3	64.9	1.10	0.995	0.0048
KOH+KCl-AC	62.0	62.9	0.140	0.998	0.0365

Generally, the correlation coefficient, R^2 of 0.99 or greater reflects that the adsorption data of MB by activated carbons fits well to the Langmuir isotherm. KCl-AC, K₂SO₄-AC and KOH+KCl-AC gave R^2 values more than 0.99, while KI-AC has a slightly lower R^2 of 0.926.

The applicability of Langmuir model suggests that the MB adsorption onto the prepared activated carbons is a monolayer adsorption that occurs over a surface with homogeneous energy site which are fairly accessible for interaction [20]. Moreover, the calculated Q_m values are close to the experimental ones (Q_e). High values of correlation coefficient, R^2 and close difference between Q_m and Q_e revealed the acceptable fitting of Langmuir isotherm model. Among the prepared activated carbons, KI-AC possesses the highest computed monolayer adsorption capacity which is 90.9 mg/g.

The separation factor (R_L), a dimensionless constant associated to the Langmuir isotherm was used to demonstrate the favourability of the adsorption process. From Table 1, the R_L values for the MB adsorption onto potassium salts-activated carbon is in the range of 0.0048-0.0520, which falls in the category of 0 < R_L < 1. This shows that the adsorption of MB onto the textile sludge based activated carbons within the concentration range is a favourable process [20].

The correlation between the initial concentration, C_0 and separation factor, R_L was scrutinized and is shown in Figure 4. As the initial concentration rose, the value of R_L declined and moved towards zero at abscissa. At initial concentration between 12 and 43 mg/L, the R_L value drops considerably, and thereafter gradually decreases at a slower pace. The adsorption mechanism of MB was near to the ideal irreversible process as the R_L moves towards zero instead of having a complete reversible occurrence [21]. Pezoti *et al.* (2014) [20], and Peydayesh and Rahbar-Kelishami (2015) [21] also reported the similar trend of R_L versus C_0 in the MB adsorption by buriti shell based activated carbon and *Platanus orientalis* leaf, respectively.



Figure 4 Relationship between initial methylene blue concentration and separation factor

3.3.2 Freundlich Isotherm Model

The Freundlich isotherm model assumes that the adsorption is multilayer over a heterogeneous surface [15]. Figure 5 shows the linear plots of Freundlich model, from which the slope and the intercept are equivalent to 1/n and log K_F , respectively. The Freundlich parameters n and K_F are listed in Table 2. The adsorption data were not fitted to the Freundlich model as the R^2 values are lower, i.e., between 0.718 and 0.881. Thus, the model

poorly described the MB adsorption onto the textile sludgeactivated carbons.



Figure 5 Freundlich isotherm for adsorption of MB onto prepared activated carbon

 Table 2
 Freundlich isotherm model parameters for MB adsorption on prepared activated carbon

Activated carbon	K_F	1/n	n	R^2
KI-AC	15.8	0.350	2.86	0.866
KCl-AC	16.9	0.182	5.50	0.773
K ₂ SO ₄ -AC	16.9	0.312	3.21	0.881
KOH+KCl-AC	20.3	0.266	3.76	0.718

Constant *n* provides crucial information on the system regarding the MB adsorption onto activated carbon. It is denoted as the heterogeneous factor which suggests that the adsorption system is linear when n = 1, chemical-type (n < 1) or physical-type (n > 1) [22]. On the other hand, the adsorption intensity or the surface heterogeneity was evaluated using the 1/n, whereby the surface is more heterogeneous when 1/n value approaches zero [15]. In this work, the 1/n values ranging from 0.182 to 0.350 indicating the high surface heterogeneity of the activated carbons. While *n* values between 2.86 and 5.50 suggested that the process is physical adsorption (physisorption). Pezoti *et al.* (2014) [20] and Mahapatra *et al.* (2012) [23] both reported similar characteristics for the MB adsorption by activated carbons derived from buriti shells and food processing industry sludge, respectively.

3.4 Comparison of MB Adsorption by Various Adsorbents

The maximum monolayer adsorption of MB in this study was compared with other various adsorbents formerly employed for the same application. The information are summarized in Table 3. It was found that the potassium iodide (KI)-activated textile sludge portrayed a better maximum monolayer adsorption of MB (90.9 mg/g) than majority of the previously investigated adsorbents. It may be due to a considerably higher BET surface area and mesoporous texture of KI-AC. On the other hand, various adsorbents (Table 3) portrayed dissimilar adsorption capacity, and this could be due to the nature of the starting materials, activation or treatment procedures and the setting conditions for adsorption. This study shows that the conversion of granular textile sludge into activated carbon is a promising and feasible route in minimizing the sludge disposal complications: averting the air pollution (incineration of sludge) and land contamination (sanitary landfill), besides its usefulness for the removal of dyes.

Table 3 Comparison between the maximum monolayer adsorption ofMB onto different adsorbents.

Adsorbent	$Q_m (\mathrm{mg/g})$	Reference
Textile sludge activated carbon	90.9	This work
Raw and sulfuric acid pretreated	7.80	[24]
defatted algal biomass (DAB)		
Biochar from anaerobic digestion	9.50	[25]
residue (BC-R)		
Marble dust sorbent	16.4	[26]
Orange peel	18.6	[8]
Banana peel	20.8	[8]
Wheat shell	21.5	[7]
Sludge from food processing industry	23.6	[23]
Corncob	28.7	[9]
Carbon nanotubes	46.2	[27]
Ficus carica bast activated carbon	47.6	[28]
Waste rubber tires	65.8	[29]
Peanut hull	68.0	[10]

4.0 CONCLUSION

Activated carbons were prepared via chemical activation using different potassium salts at impregnation ratio of 1:1. The BET results show that the surface area increased in the order of $K_2SO_4-AC > KCl-AC > KI-AC > KOH+KCl-AC$, where K₂SO₄-AC had the highest surface area of $321m^2/g$. From the batch adsorption, it was found that 39.1 and 85.0mg/g of MB were removed by KCl-AC and KI-AC, respectively. Langmuir isotherm gave the best fit to the adsorption data compared to Freundlich model, indicating a monolayer coverage and uniform activity distribution on the carbon surface. The maximum monolayer adsorption of Langmuir model is 90.9 mg/g. Among the four prepared activated carbons, KI-AC with BET surface area of 191 m²/g gave the best MB adsorption. It can be concluded that textile sludge is a potential low cost precursor for the preparation of activated carbon that can be effectively employed in the adsorption of MB.

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