

## ADSORPTION OF CU(II) FROM AQUEOUS SOLUTION USING ACTIVATED CARBON DERIVED FROM MANGOSTEEN PEEL

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Effects of the mixing method of  $K_2CO_3$  with the source biomass and thermal history on the properties of the mangosteen peel activated carbons (MSACs) were investigated. The one-step impregnation activation process was found to be remarkably effective in increasing the mesoporosity of the activated carbon (L-MSAC) as well as BET surface area ( $S_{BET}$ ) and total pore volume, compared to the solid-solid dry mixing method in a two-stage process. The better fit of Langmuir isotherm indicates a maximum adsorption capacity of Cu(II) to be  $21.74 \text{ mg}\cdot\text{g}^{-1}$  for L-MSAC, which makes it a promising adsorbent for the removal of copper ion from aqueous solutions.

*Keywords:* Mangosteen peel; Activated carbon; Pore structure; Adsorption

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### INTRODUCTION

Activated carbon, containing graphene layers of carbon atoms having  $sp^2$  hybridization, is well known as an excellent adsorbent with well-developed porosity (López *et al.* 2011). The type of source material used, a variety of activation profiles and agents, and post-treatments have been found to be influential with respect to the porous morphology and surface characteristics of activated carbons (ACs), which are important for their expanding application properties (Armandi *et al.* 2010; Daud and Houshamnd 2010). A diverse set of forests and agricultural byproducts have now been increasingly used as alternative precursors for production of high-grade ACs rather than fossil fuels, due to their promising characteristics such as being renewable, cost-effective, and environment-friendly (Mourão *et al.* 2011; Nuithitikul *et al.* 2010).

The mangosteen (*Garcinia mangostana* L.) (GML) tree is widely found in several Asian countries, especially Thailand and Indonesia. It has been considered the “queen of fruits” due to its pleasant taste apart from many functions for disease treatment (Zein *et al.* 2010). For every 10 kg of mangosteen harvested, more than 6 kg of mangosteen peel is generated. The increasing popular consumption of mangosteen fruit has given rise to abundant abandoned mangosteen peel residue, which may be accessible in large quantities from the plants engaged in extraction of pulp juice or bioactive components from the fruit. Mangosteen peel wastes can therefore be explored as a new potential lignocellulosic precursor for activated carbons to derive more economic value (Chen *et al.* 2011; Devi *et al.* 2009).

Herein, the influence of mixing method of chemical activator with precursor, together with pre-carbonization process, on the development of pore characteristics in the

resulting activated carbons was evaluated. The aim of this work was to establish a more efficient way to prepare activated carbons from mangosteen peels (MSAC) with superior performances by  $K_2CO_3$  activation. Moreover, the activated carbons derived from mangosteen peel have been recently reported to be a promising adsorbent for removal of dyes from wastewater (Ahmad and Alrozi 2010; Foo and Hameed 2012). However, there is no information concerning the behaviors of MSAC in renovating metal-contaminated wastewater. Thus, the adsorption capacity for Cu(II) from aqueous solution onto the MSAC prepared in this study was probed based on the equilibrium data fitted to the Langmuir and Freundlich isotherms. The results provide a useful guide for industrial production and application of mangosteen peel activated carbon.

## EXPERIMENTAL

### Materials

Mangosteen peels were collected from the local SAM's Club store in Fuzhou, China. The mangosteen peels were thoroughly washed with distilled water to eliminate any residue and impurities and dried at  $105^\circ C$  for 24 h to reduce moisture content. Dried mangosteen peels were crushed and then milled until they became powder of 40- to 80-mesh. The powder was stored in a desiccator for subsequent use.

All reagents used were of analytical grade purchased from Shanghai National Chemical Reagent Co., Ltd. (China).

### Production of Activated Carbons

Mangosteen peel activated carbons (MSACs) using  $K_2CO_3$  as chemical activator were prepared by two different activation strategies. The ideal preparation conditions in a two-stage activation approach by the solid-solid mixing method were selected according to our preliminary single-factor experiments. First, pre-carbonization of the as-dried mangosteen peel powder was carried out in a muffle furnace at  $450^\circ C$  for 2 h without the flow of inert gas to yield mangosteen peel-based char. The as-prepared char was then physically mixed with  $K_2CO_3$  by sufficiently dry milling in an agate mortar at a chemical to char (K/C) ratio of 1.0 (w/w). Afterward, the mixture was placed into a porcelain crucible with a cover and heated to  $800^\circ C$  for 120 min in a furnace under a self-generated atmosphere at a heating rate of  $10^\circ C /min$  for activation.

On the other hand, the one-step impregnation activation process was conducted as follows: as-dried mangosteen peels (40- to 80-mesh size) were immersed into 30 wt%  $K_2CO_3$  solution for 12 h at room temperature with the same chemical to mangosteen peels ratio of 1.0 based on mass. Thereafter, the obtained slurry was then dried overnight at  $110^\circ C$ . The resulting mixture was eventually subjected to pyrolysis according to the same experimental details as mentioned above during the two-stage activation process.

After chemical activation, the final products were cooled down to room temperature and sequentially boiled with a 0.5 M HCl solution. After that, the activated carbons were washed several times with distilled water until their filtrate nearly reached neutral pH. The washed samples were dried overnight at  $105^\circ C$  before further use. The

resultant activated carbons prepared using the solid-solid mixing method and wet impregnation method were denoted as S-MSAC and L-MSAC, respectively.

### Batch Adsorption Experiments

A stock solution of Cu(II) at 1000 mg/L concentration was prepared by dissolving a required quantity of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  in deionized water. Prior to the adsorption experiments, the solutions were further diluted to the desired concentrations. In batch adsorption experiments, 25.0 mg of L-MSAC was added to 50 mL aqueous solutions having the known copper ion concentrations of 5, 10, 20, 50, 100, and 200 ppm with initial pH  $6.7 \pm 0.3$ . The stoppered conical flasks were shaken at 120 rpm using an electric shaker and maintained at  $30 \pm 2^\circ\text{C}$  for 8 h to attain equilibriums. The supernatant of the suspensions after centrifugation was then filtered through a  $0.45 \mu\text{m}$  PVDF (polyvinylidene fluoride) membrane syringe filter, and the residual concentrations of Cu(II) ion in the filtrate were analyzed by atomic absorption spectrophotometry (AAS) after being diluted to suitable levels. The amount of copper ion uptake at equilibrium,  $q_e$  (mg/g), was calculated by Eq. (1):

$$q_e = V(C_0 - C_e) / m \quad (1)$$

where  $C_0$  (mg/L) is the initial concentration of adsorbate,  $C_e$  (mg/L) is the equilibrium concentration in the liquid phase,  $V$  is the volume of liquid phase (L), and  $m$  is the mass of the adsorbent (g).

### Regeneration Experiments

Firstly, adsorption equilibrium was carried out according to the same procedures as described in aforementioned adsorption experiments. Herein, 0.2 g of L-MSAC was added to 50 mL of 50 ppm Cu(II) aqueous solution. Once the adsorption equilibrium was reached, the saturated L-MSAC adsorbent was separated from solution and fully washed with deionized water. The washed carbon was placed in 50 mL of 0.1 M  $\text{H}_2\text{SO}_4$  for the desorption test (Stirk and Staden 2002; Wang *et al.* 2011). The mixture was then kept in a water bath shaker at 120 rpm for 4 h at  $30 \pm 2^\circ\text{C}$ . After desorption, the final concentration of Cu(II) in solution was measured to evaluate the recovery efficiency. The regenerated adsorbent was thereafter separated and thoroughly rinsed with distilled water and again loaded with Cu(II) solution of concentration of 50 ppm as previously described. The regeneration studies were conducted for three successive adsorption–desorption cycles with the same operating conditions. The regeneration efficiency ( $RE$ ) was calculated according to the following equation (Gupta and Sharma 2002),

$$RE(\%) = \frac{q_r}{q_0} \times 100 \quad (2)$$

where  $q_r$  (mg/g) is the adsorption capacity of the regenerated adsorbent and  $q_0$  (mg/g) is the original metal uptake of the virgin L-MSAC.

## Characterization

The iodine number and methylene blue (MB) adsorption tests were conducted according to GB/T 12496.8-1999 (testing standard of activated carbon in China) and GB/T 12496.10-1999. Iodine number is commonly a probe molecule of adsorption capacity, reflecting the amount of micropores with diameters larger than 1 nm. The MB molecule is used as a measure of the pores greater than 1.5 nm in diameter (Deng *et al.* 2009; Lim *et al.* 2010). The iodine number and MB adsorption properties are expressed as milligrams of adsorbate adsorbed by one gram of carbon. The yield of the activated carbons is defined as the weight ratio on a dry basis of the resulting activated carbon to the mangosteen peels.

The pore structure characteristics of activated carbons were determined by nitrogen adsorption at 77 K using an automatic Micromeritics ASAP-2020 volumetric sorption analyzer. The  $S_{\text{BET}}$  was calculated by the BET equation, whereas the micropore volume, micropore surface area, and external surface area were obtained using the t-plot method, and the mesopore volume was obtained by subtracting the micropore volume from the total pore volume.

A Shimadzu AA-6300C atomic absorption spectrometer was used for the determination of copper. A deuterium lamp was used for background corrections. The hollow cathode lamp was operated at 3.0 mA, and the wavelength was set at 324.7 nm. The flame type was air-acetylene.

## RESULTS AND DISCUSSION

### Textural Characteristics of Mangosteen Peel-Derived Activated Carbons

The effect of activation profiles on the yield, iodine, and methylene blue (MB) adsorption and porosity parameters of the produced activated carbons of S-MSAC and L-MSAC is shown in Table 1. The L-MSAC showed a much higher adsorption capacity of iodine and MB molecules; this is a result of much greater BET surface area and total pore volume for L-MSAC than those of S-MSAC. This is probably favored by the better infiltration of  $\text{K}_2\text{CO}_3$  into the precursor with the soaking process, thus providing a higher activation degree of C- $\text{K}_2\text{CO}_3$  reactions. This in turn results in a better development of the porosity (Bagheri and Abedi 2009).

When using the direct physical dry mixing method, the activator could hardly enter into the interior of the char, so the development of porosity in S-MSAC was relatively poor. On the other hand, the representative yield of the activated carbons decreased from about 23.1% for S-MSAC to 20.3% with respect to L-MSAC. This result suggests that the total burn-off ratio was less in the two-stage activated carbons. It could be inferred that production of S-MSAC involved with pre-carbonization process may provide a carbonaceous intermediate of well-organized and dense aromatic structure with more stability, giving rise to much gently gasification of the carbons with existing oxidizing gases released or self-generated during the activation process (Pastor *et al.* 1999; Yun *et al.* 2001).

**Table 1.** Selected Properties of Mangosteen Peel-Based Activated Carbons Prepared in Present Work and in Previous Studies

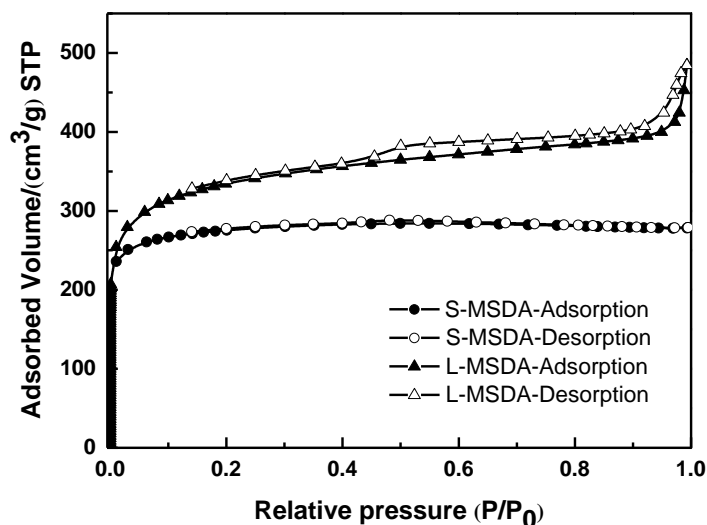
	Types of Activated Carbons			
	S-MSAC	L-MSAC	MPAC-2010 <sup>a</sup>	MPAC-2012 <sup>b</sup>
Iodine number (mg·g <sup>-1</sup> )	927	1189	-	-
MB adsorption (mg·g <sup>-1</sup> )	98	180	-	-
Yield (%)	23.1	20.3	20.8	-
Typical properties				
Specific surface area (m <sup>2</sup> ·g <sup>-1</sup> )	908	1106	994	1099
Total pore volume (cm <sup>3</sup> ·g <sup>-1</sup> )	0.43	0.75	0.57	0.61
Micropore area (m <sup>2</sup> ·g <sup>-1</sup> )	702	659	-	-
External Surface Area (m <sup>2</sup> ·g <sup>-1</sup> )	187	447	-	-
Micropore volume (cm <sup>3</sup> ·g <sup>-1</sup> )	0.34	0.32	-	0.33
Average pore diameter (nm)	1.82	2.71	2.61	2.22

<sup>a</sup> Ahmad and Alrozi, 2010.

<sup>b</sup> Foo and Hameed, 2012.

As depicted in Fig. 1, the nitrogen adsorption isotherm of S-MSAC presented a typical type I feature according to the IUPAC classification, indicative of the essentially microporous character with a narrow pore size distribution (Sun and Webley 2011). While for L-MSAC, a much rounder knee with a gently increasing slope of the isotherm plateau and a hysteresis loop of type H4 were observed to yield a type IV isotherm, which is characteristic of slit-shaped pores as described in many activated carbons associated with a combination of microporous and mesoporous structures (Foo and Hameed 2011; Lu *et al.* 2010; Oliveira *et al.* 2009). Although the micropore volumes for S-MSAC and L-MSAC were almost comparable, as presented in Table 1, it should be noted that L-MSAC showed a vast majority contribution of mesopores to the high surface area compared to S-MSAC, as the mesoporosity was about 20.9% and 57.3% for S-MSAC and L-MSAC, respectively. Meanwhile, L-MSAC was detected with an average pore diameter of 2.71 nm, which is obviously larger than the corresponding value in the case of S-MSAC (1.82 nm).

A more aggressive pore-widening activation process that occurred by soaking treatment compared with the physically dry mixing method was thereby proposed. The lower micropore surface, together with appreciably greater mesoporosity, in the case of L-MSAC is also likely relevant to the less well-organized carbon structure in the raw substrate, which is relatively labile to gasification reaction during pyrolysis, leading to further pore enlargement. In contrast, for S-MSAC, a comparatively stable biochar is formed with the inclusion of pre-carbonization process before activation, which is more resistant to a further pore-widening decomposition during activation, and hence gives rise to a dominant microporous structure (Yun *et al.* 2001).



**Fig. 1.** N<sub>2</sub> adsorption (full symbols)-desorption (open symbols) isotherms at 77 K for S-MSAC and L-MSAC

Based on the results mentioned above, it was found that L-MSAC displays a larger specific surface area, higher total pore volume, and much more developed mesopores comprised of considerable micropores, which is propitious for enhancing its adsorption capacities (Hartono *et al.* 2005; Sevilla *et al.* 2011). Moreover, L-MSAC prepared in this study exhibited better porosity development than those ever reported for the mangosteen peel derived ACs as listed in Table 1, despite the higher chemical impregnation ratio and two-step activation process being used (Ahmad and Alrozi 2010; Foo and Hameed 2012). Besides, the BET specific surface area of L-MSAC ( $1189 \text{ m}^2 \cdot \text{g}^{-1}$ ) produced by  $\text{K}_2\text{CO}_3$  was also much higher than that ( $756 \text{ m}^2 \cdot \text{g}^{-1}$ ) derived from 50% (v/v)  $\text{H}_3\text{PO}_4$  activation (Devi *et al.* 2009). Consequently, the one-step  $\text{K}_2\text{CO}_3$  activation process with impregnation mixing method can be recommended as a more favorable way for production of mangosteen peel derived activated carbons with more developed porosity.

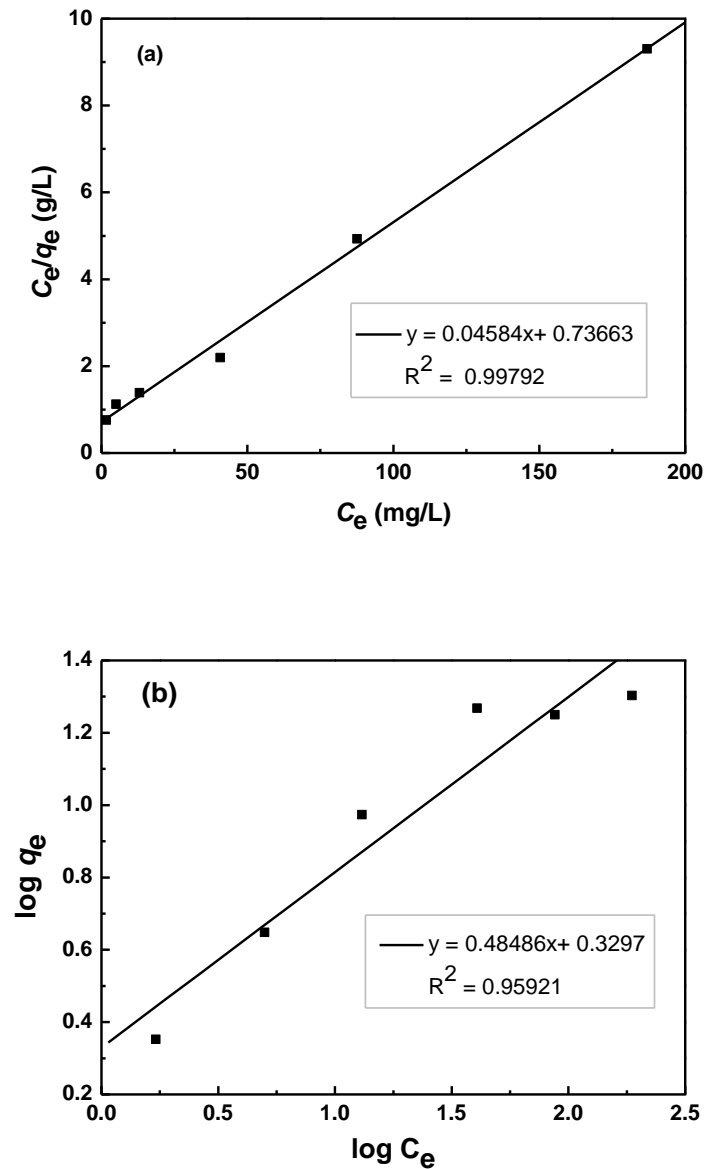
### Adsorption Isotherm of Copper (II) by L-MSAC from Aqueous Solution

The adsorption isotherm is fundamental to determine the adsorption behavior of an adsorbent, which can be clarified by some constants to predict the surface properties and affinity of the adsorbent for certain adsorbates. To quantify the adsorption capacity of L-MSAC for the removal of Cu(II) from aqueous solution, the linear forms of the Langmuir (Eq. (3)) and Freundlich (Eq. (4)) equations were applied to the equilibrium data. The adsorption of copper ion adjusted to the linearized equations of Langmuir and Freundlich models are shown in Fig. 2,

$$\frac{C_e}{q_e} = \frac{1}{q_{\max} K_L} + \frac{C_e}{q_{\max}} \quad (3)$$

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (4)$$

where  $C_e$  (mg/L) is the equilibrium concentration of the copper (II) solution,  $q_e$  (mg/g) is the adsorption at equilibrium,  $K_L$  (L/mg) is a Langmuir adsorption constant related to the affinity of the binding sites, and  $q_{\max}$  (mg/g) is the maximum adsorption capacity.  $K_F$  is a Freundlich constant indicating the relative adsorption capacity of the carbon ( $\text{mg/g} \cdot (\text{L/mg})^{1/n}$ ), and  $1/n$  is the adsorption intensity. The magnitude of  $n$  predicts of the favorability of the adsorption process (Özcimen and Ersoy-Mericboyu 2009).



**Fig. 2.** Linear plots of adsorption isotherms of Cu(II) onto the L-MSAC fitted to the Langmuir (a) and Freundlich (b) models

**Table 2.** Isotherm Parameters for Copper (II) onto the L-MSAC Obtained by Linear Equations at 303 K

Langmuir	Freundlich
$q_{max} = 21.74$	$K_F = 2.136$
$K_L = 0.0624$	$1/n = 0.485$
$R^2 = 0.998$	$R^2 = 0.959$
$R_L = 0.762$	

According to the  $R^2$  values of correlation coefficients given in Table 2, the experimental data of copper ion adsorption fit the Langmuir isotherm better than the Freundlich model. A dimensionless equilibrium parameter  $R_L$  between 0 and 1 deduced from the Langmuir isotherm was obtained, indicative of a favorable homogenous adsorption process of Cu(II) onto the L-MSAC (Danish *et al.* 2011; Hameed and Rahman 2008). The monolayer maximum adsorption capacity ( $q_{max}$ ) of L-MSAC for the uptake of Cu(II), derived from the application of Langmuir equation was 21.74 mg/g in this work. The adsorption capacity of L-MSAC for Cu(II) in aqueous solution was higher than many of the reported values from various adsorbents, as summarized in Table 3. Although not being best of all, the L-MSAC can yet be regarded as a good adsorbent candidate in decontamination of wastewater containing copper ion.

**Table 3.** Adsorption Capacities of Selected Adsorbents for the Uptake of Copper (II) from Aqueous Solution

Adsorbents	$q_{max}$ (mg·g <sup>-1</sup> )	Reference
Activated carbon of rice hulls	3.92	(Teker <i>et al.</i> 1999)
Activated carbon of rubber wood sawdust	5.73	(Kalavathy <i>et al.</i> 2005)
Activated carbon of apricot stone	24.21	(Kobyas <i>et al.</i> 2005)
Activated carbon of hazelnut husk	6.64	(Imamoglu and Tekir, 2008)
Activated carbon of palm peel	1.58	(Onundi <i>et al.</i> 2010)
Activated carbon of polyacrylonitrile fiber	12.16	(Zainia <i>et al.</i> 2010)
Activated carbon of sewage sludge	10.56	(Wang <i>et al.</i> 2011)
Activated carbon of date stone	37.3	(Danish <i>et al.</i> 2011)
Activated carbon of potato peel	62	(Moreno-Pirajána and Giraldo, 2011)
Activated carbon of mangosteen peel	21.74	This work

### Reusability of the regenerated activated carbon

The performance of the spent L-MSAC for subsequent reuse was investigated by three repeated adsorption-desorption cycles. After exposure to 0.1 M H<sub>2</sub>SO<sub>4</sub> for each successive adsorption cycle, it was found that over 80% recovery of the copper ions could be readily achieved. The percentage removal of the copper ion was 99.6% in the beginning, followed by a gradual decrease to 84.1% after the next two cycles. The regeneration efficiencies (*RE*) were calculated as 93.1% and 83.4% for cycles 2 and 3,



respectively. On the basis of the results, the spent activated carbon regenerated using 0.1 M H<sub>2</sub>SO<sub>4</sub> could be reused at least for three adsorption–desorption cycles with good re-adsorption capabilities.

## CONCLUSIONS

1. The one-step impregnation activation process is proposed to be a more effective method for production of activated carbon with significantly improved adsorption capacities (on the basis of iodine and methylene blue adsorption data), BET surface area, total pore volume, and highly developed mesopores, in comparison with the two-stage activation approach with solid-solid mixing method.
2. L-MSAC made by one-step impregnation activation exhibits a maximum adsorption capacity of 21.74 mg·g<sup>-1</sup> for copper (II) in aqueous solution. The spent L-MSAC treated with H<sub>2</sub>SO<sub>4</sub> showed relatively high recovery and regeneration efficiencies in three adsorption-desorption cycles. Thus, mangosteen peel-based activated carbon can be regarded as a promising adsorbent for the removal of harmful heavy metal ions from wastewaters.

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