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Preparation of Activated Carbon from *Sindora Siamensis* Seed and *Canarium Sublatum Guillaumin* fruit for Methylene Blue Adsorption

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ABSTRACT

Activated carbons produced from *Sindora Siamensis* (SSAC) seed and *Canarium Sublatum Guillaumin* (CSGAC) fruit were prepared by chemical activation with zinc chloride, their characteristics and their methylene blue (MB) adsorptions were investigated. The effects of zinc chloride concentrations and activation temperatures were examined. The surface chemical characteristics of activated carbons were determined by scanning electron microscope (SEM). Adsorption capacity was demonstrated with iodine numbers. The Langmuir and Freundlich equilibrium isotherm theories were applied to describe MB adsorption. The equilibrium adsorption results were complied with Langmuir isotherm model and its maximum monolayer adsorption capacity for SSAC and CSGAC are 672.6 and 487.6 mg/g for MB adsorption. The value of R_L was found to be below 1.0, indicating that the resultant activated carbon was favorable for MB adsorption. These results indicate that SSAC and CSGAC shells could be utilized as a renewable resource to develop activated carbon which is a potential adsorbent for MB.

1. Introduction

Activated carbon is the most commonly use and most effective adsorbent because of its high adsorptive capacity (Chen *et al.*, 2003; Daifullah *et al.*, 2007). Therefore, it has been widely used as adsorbent (Hung et al., 2005) and in catalysis (Lee *et al.*, 2006) or separation

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processes (Rodriguez-Reinoso et al., 2002) such as purification of drinking water, treatment of exhaust gas and waste water. However, its application fields are restricted due to its high cost. Presently, the use of low cost plants and agricultural wastes is considered promising adsorbents for adsorption applications. In recent years, a lot of research has been reported on activated carbons from plants or agricultural wastes, such as olive mill waste (Abdelkreem, 2013), seaweed (Rathinam *et al*., 2011), cherry stones (Jaramillo *et al*., 2009), fluted pumpkin seed shell (Verla *et al*., 2012), fluted pumpkin stem waste (Ekpete *et al*., 2011), rice straw (Gao *et al*., 2011), cattail (Shi *et al*., 2011), coconut husk (Tan *et al*., 2008), coconut shells (Yang *et al*., 2010), tobacco residues (Kilic *et al*., 2011), sugar cane bagasse, and sunflower seed hull (Liu *et al*., 2010), etc.

The *Sindora Siamensis* (SS) and *Canarium Sublatum Guillaumin* (CSG) are a large evergreen tree found in open semi–deciduous forests in Cambodia, Laos, Malaysia, Thailand, and Vietnam. The diagnostic characters of SS are deciduous or evergreen tree, bark smooth to slightly fissure. Leave compounds rachis swollen at the base. Flower in panicle is yellow–green. Fruit an ovoid–round spiny, flattened pod often with blobs of white resin (Sam *et al*., 2004). The CSG is 20–35 m tall. Branchlets are brown tomentose when young, glabrescent, lenticellate, with conspicuous leaf scars.

The objective of this study is to produce activated carbon from a *Sindora Siamensis* (SSAC) seed and *Canarium Sublatum Guillaumin* (CSGAC) fruit by chemical activation using $ZnCl_2$ and adsorption capacity of activated carbons are also investigated. $ZnCl_2$, as an important chemical activating agent, has been widely used in the process of activated carbon preparation (Horng *et al*., 2010; Rathinam *et al*., 2011; Gao *et al*., 2013). It is an efficient dehydration reagent that promotes the decomposition of carbonaceous material, induces the charging and aromatization of the carbon, restricts the formation of tar and increases the carbon yield. Thus to our best knowledge, however, have not yet been reported the activation of SS seed and CSG fruit with $ZnCl_2$ chemical activation. The major novelty of this work is the production of activated carbons from $ZnCl_2$ impregnated SS seed and CSG fruit samples by chemical activation technique in various SS/$ZnCl_2$ and CSG/$ZnCl_2$ ratios and a temperature range of 500–700°C. The study covers the effect of operating parameters such as temperature of activation and impregnation ratio on the product quality. The product quality is characterized based on the iodine number and MB adsorption isotherm.

2. Experiment

2.1 Materials and Preparation of Activated Carbon

Sindora Siamensis seed and *Canarium Sublatum Guillaumin* fruit were collected from Maha Sarakham province of Thailand. The precursor was crushed and sieved in order to get a standardized particle dimension. Five to forty grams of $ZnCl_2$ were dissolved in 200 mL of distilled water, and then 20 g of raw ash was mixed with the $ZnCl_2$ solution and stirred at approximately 85°C for 2 hr. The mixtures were dehydrated in an oven at 110°C for about 24 hr. The resulting activated carbons were then chemically activated at 500, 600 and 700°C for 3 hr in atmosphere. The activated samples were washed in 3 M hydrochloric solution by heating at around 90°C for 30 min to remove the zinc compounds. Then, they were washed several times with warm distilled water, and finally with cold distilled water. The washed samples were dried at 110°C for 24 hr to prepare the activated carbons. Each sample was stored in sealed bottle vial.

2.2 Sample Characterization

The iodine number is a technique employed to determine the adsorption capacity of activated carbons. The iodine number indicates the porosity of the activated carbon and it is defined as the amount of iodine adsorbed by 1 g of carbon at the mg level. Iodine number can be used as an approximation for surface area and microporosity of active carbons with good precision. The iodine adsorption was determined using the sodium thiosulfate volumetric method (ASTM, 2006). It is a measure of activity level (higher number indicates higher degree of activation), often reported in mg/g. It is a measure of the micropore content of the activated carbon by adsorption of iodine from solution (Elliott *et al*., 1989). The procedure of the iodine number determination is as follows: activated carbons were weighed out into conical flasks (sample weight ranged between 100 and 400 mg). Ten millilitres of 5% (in weight) hydrochloric acid solution were added to each flask and then mixed until the carbon became wet. The mixtures were then boiled for 30 s and finally cooled. One hundred millilitres of 0.05 M standard iodine solution were added to each flask. The contents were vigorously shaken for 30 s and then immediately filtered. A 50 ml aliquot of each filtrate was titrated by a standardized 0.1 M sodium thiosulfate solution. The morphologies of SSAC and CSGAC were examined by a LEO/1450 scanning electron microscope (SEM). The samples were dried at 105°C for 2 hr and coated with a thin gold film to give electrical conduction on the carbon external surface.

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2.3 Adsorption Isotherm

Methylene blue kinetic and isotherm adsorption experiments were carried out to evaluate the adsorption performance. The equilibrium adsorption research was completed by adding a fixed amount of activated carbon into 25 ml different initial concentrations of MB. The kinetic adsorption studies were performed by adding 0.2 g activated carbon into 200 ml different initial concentrations of MB. The aqueous samples were taken at pre–set time intervals and their concentrations were determined. Concentration determination of all the samples was filtered before they were measured by UV spectrophotometer (Perkin Elmer/lamda 12) at the maximum absorption wavelength of 664 nm.

The Langmuir and Freundlich equilibrium isotherm theories were applied to describe the MB adsorption. Langmuir equation can be represented by the following Eq. (1) (Singh *et al.*, 2008; Fierro *et a*l., 2008):

$$\frac{C_e}{q_e} = \frac{1}{K_L Q_{\max}} + \frac{C_e}{Q_{\max}} \tag{1}$$

where C_e (mg/L) is the equilibrium concentration of MB in solution, q_e (mg/g) is the amount of MB adsorbed at the equilibrium time, Q_{max} (mg/g) is the maximum capacity, and K_L (L/mg) is the Langmuir constant related to the free energy or net enthalpy of adsorption.

Freundlich model is an empirical equation assuming heterogeneous adsorptive energies on the adsorbent surface, which can be written as Eq. (2) (Valente *et al.*, 2011):

$$\log q_e = \log K_F + \frac{1}{n}\log C_e \tag{2}$$

where K_F is the Freundlich constant and taken as an indicator of adsorption capacity, in (L/mg) and $1/n$ is an empirical constant related to the magnitude of adsorption driving force (Fan *et al.*, 2008).

3. Results and Discussion

3.1 Production Yield

The production yields (%yield) of SSAC and CSGAC are listed in Table 1, the yield values of SSAC were 85.26, 75.26, and 72.86% for activated at 500, 600, and 700°C, respectively. The yield values of CSGAC were 84.94, 77.46, and 73.02% for activated at 500, 600, and 700°C, respectively. It is obviously seen that the yield decreases with increasing temperature. As generally recognized, O and H atoms could be evolved into CO, CO_2, H_2O, CH_4, aldehydes or tar in the carbonization process of lignocellulosic materials. Moreover, the yield is slightly difference in various $ZnCl_2$/SS or $ZnCl_2$/CSG ratios. Hence, the yield depends on the amount of carbon removed by combining with O and H atoms. However, $ZnCl_2$ would selectively stripe H and O away from SSAC and CSGAC as H_2O and H_2 rather than CO, CO_2 or hydrocarbons (Caturla *et al*., 1991).

Table 1: Yield (%) and capacity of the prepared carbons in the adsorption of iodine numbers.

Temperature (°C)/Types of activated carbon	Yield (%)		Iodine number (mg/g)	
	SSAC	CSGAC	SSAC	CSGAC
500	85.26	84.94	148.23	146.33
600	75.26	77.50	147.40	142.09
700	72.86	73.02	132.24	118.58

3.2 Iodine Number

The capacities of the prepared carbons in the adsorption of iodine with SSAC and CSGAC are list in Table 1. It is found that the iodine numbers for iodine adsorbed on SSAC were 148.23, 147.40, and 132.24 mg/g for activated at 500, 600, and 700°C, respectively and iodine numbers for iodine adsorbed on CSGAC are 146.33, 142.09, and 118.58 mg/g for activated at 500, 600, and 700°C, respectively. This data indicate that the activated carbon prepared from SS is more suitable for iodine adsorption than CSGAC, which the preparation of activated carbon at 500°C is the highest porosity in both SSAC and CSGAC. The tested activated carbons adsorb significant amounts of iodine which are strongly related to the degree of activation.

3.3 Textural Characterization by SEM

The morphology in terms of amorphous, pore size and pore distribution of SSAC and CSGAC was investigated. The SEM photographs of raw ash, SSAC and CSGAC for activation at 500–700°C are shown in Figures 1 and 2. The result showed that the highly amorphous structure could be prepared from the SS seed and CSG fruit. The fibrillary structures of all SSAC and CSGAC reveal that the pores are not cross–linked. Figure 1a–1d

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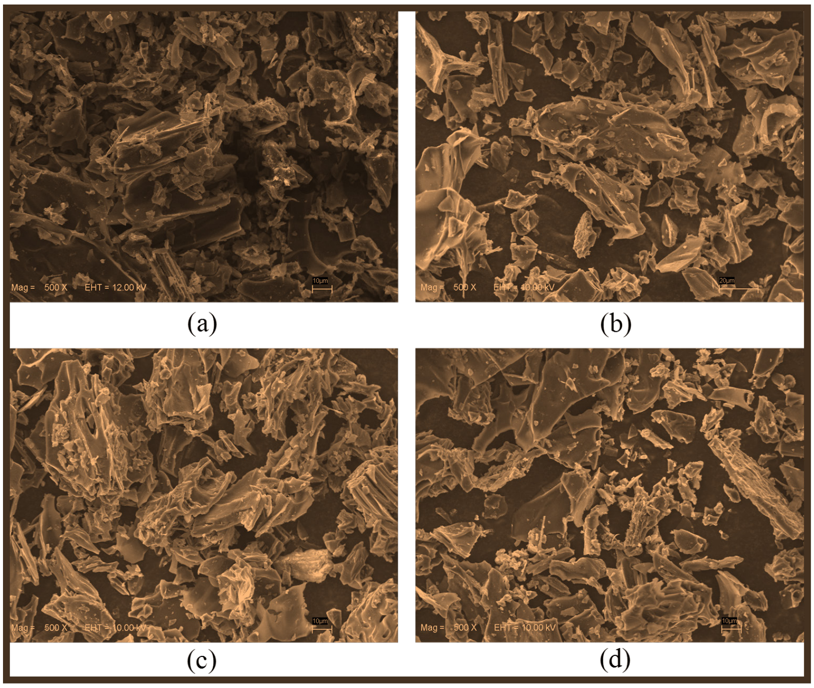

Figure 1: SEM micrographs for (a) raw ash, (b) SSAC for activated at 500°C, (c) SSAC for activated at 600°C and (d) SSAC for activated at 700°C

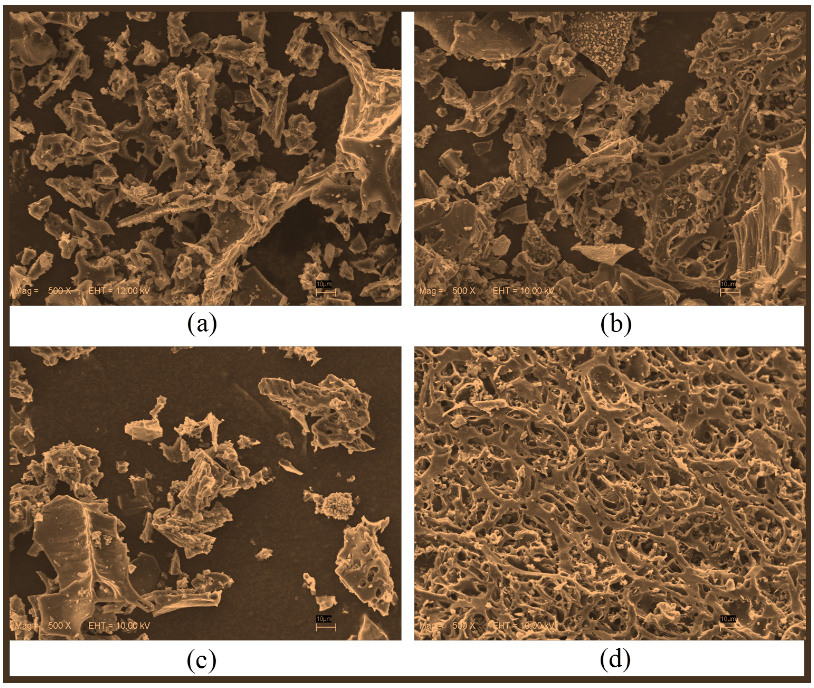

Figure 2: SEM micrographs for (a) raw ash, (b) CSGAC for activated at 500°C, (c) CSGAC for activated at 600°C and (d) CSGAC for activated at 700°C

shows that the surface of SSAC is relatively organized with large pores. Whereas, Figure 2a–2c indicates the external surface of CSGAC is similarly to SSAC but for CSGAC activated at 700°C (see Figure 2d) giving small pore volumes. It has been suggested that the cavities resulted from the evaporation of $ZnCl_2$ during carbonization, leaving the space previously occupied by the $ZnCl_2$ (Hu *et al*., 2001).

3.4 Methylene Blue Adsorption Capacity

The Langmuir and Freundlich equilibrium isotherm theories were applied to describe the MB adsorption. Langmuir equation can be represented by the following Eq. (1). Freundlich model is an empirical equation assuming heterogeneous adsorptive energies on the adsorbent surface, which can be written as Eq. (2). The essential features of Langmuir isotherm can be expressed by the term of separation factor or equilibrium parameter (R_L), which is defined as $R_L = 1/(1 + K_LC_0)$ (where C_0 is the initial concentration of adsorbate and K_L is its Langmuir constant). The R_L value refers to the nature of adsorption as irreversible ($R_L = 0$), favorable ($0 < R_L < 1$), linear ($R_L = 1$) or unfavorable ($R_L > 1$) (Bouhamed *et al*., 2012; Liu *et al*., 2010). The Langmuir and Freundlich calculated parameters are given in Tables 2 and 3. The q_e values of SSAC for activated at 500, 600, and 700°C were 441.6, 672.6, and 462.6 mg/g, respectively. And the q_e values of CSGAC for activated at 500, 600, and 700°C were 429.3, 487.6, and 203.8 mg/g, respectively. This data indicate that the activated carbon prepared from SS is more suitable for iodine adsorption than CSG, which the preparation of activated carbon at 600°C is the highest porosity in both SSAC and CSGAC. The values of R_L are found to be below 1 for MB adsorption, indicating that the resultant activated carbons were favorable for MB adsorption. Average correlation coefficients R^2 for SSAC and CSGAC were 0.95 and 0.89, respectively, indicating that the two models fits to the experimental data. The Freundlich isotherm model shows a better description for MB adsorption data with average R^2 for SSAC and CSGAC in the range of 0.95 and 0.87, respectively. Freundlich parameters K_F and 1/n were obtained from the slope and intercept, respectively, which demonstrate whether the adsorption is favorable or not. A high value of K_F, is indicative of a high adsorption capacity (Salame *et al*., 2003). In short, 1/n is a measure of the surface heterogeneity, ranging between 0 and 1, as its value gets closer to zero, the surface become more heterogeneous (Ahmaruzzaman *et al*., 2005). Since all the value of 1/n is less than 1, it indicates a favorable adsorption (Mohanty *et al*., 2005).

The lower determination SSAC coefficients R^2 of the Langmuir suggest that the isotherm data do not fit the Langmuir model. The higher determination SSAC coefficients R^2 of the Freundlich equation suggest that the Freundlich equation can be used to fit the experimental adsorption data and evaluate the maximum dye adsorption capacities of the three adsorbents. Whereas, the determination CSGAC coefficients R^2 of the Langmuir is equal

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to Freundlich suggesting that the adsorption capacities not difference. The results also indicate that the adsorption of MB by the three adsorbents takes place in a monolayer adsorption manner.

Table 2: Parameters of Langmuir adsorption isotherm models for MB adsorbed by the different temperatures.

Temperature (°C)/Types of activated carbon	SSAC				CSGAC			
	Q_e (mg/g)	K_L (L/mg)	R_L	R^2	Q_e (mg/g)	K_L (L/mg)	R_L	R^2
500	441.6	0.075	0.382	0.9302	429.3	0.043	0.510	0.8942
600	672.6	0.078	0.373	0.9554	487.6	0.120	0.285	0.7909
700	462.6	0.091	0.340	0.9597	203.8	0.009	0.822	0.9997

Table 3: Parameters of Freundlich adsorption isotherm models for MB adsorbed by the different temperatures.

Temperature (°C)/Types of activated carbon	SSAC			CSGAC		
	K_F (L/mg)	1/n	R^2	K_F (L/mg)	1/n	R^2
500	2.077	0.391	0.9798	5.941	0.348	0.8729
600	2.228	0.315	0.9583	5.906	0.409	0.3254
700	2.008	0.409	0.9247	3.862	0.658	0.9315

4. Conclusion

Activated carbons were produced from *Sindora Siamensis* seed and *Canarium Sublatum Guillaumin* fruit by chemical activation with zinc chloride, their characteristics and their methylene blue adsorptions were investigated. The effects of zinc chloride concentrations and activation temperature were examined. The morphology, pore size, and pore distribution characteristics of activated carbons were determined by SEM method. Adsorption capacity was demonstrated with iodine numbers. The Langmuir and Freundlich equilibrium isotherm theories were applied to describe methylene blue adsorptions. The equilibrium adsorption results were complied with Langmuir isotherm model and its maximum monolayer adsorption capacity for SSAC and CSGAC are 672.6.0 and 487.6 mg/g for methylene blue adsorption. The value of R_L was found to be below 1.0, indicating that the resultant activated carbon was favorable for phenol adsorption. These results indicate that SSAC and CSGAC shells could be utilized as a renewable resource to develop activated carbon which is a potential adsorbent for methylene blue.

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