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Sorption Kinetic Studies of Medical Grade Activated Carbon Prepared from Papaya Seeds

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Abstract

The aim of this investigation was to determine the adsorption behavior and kinetics of methylene blue in aqueous solution on activated carbons prepared from papaya seeds by way of the two stage activation method in self-generated atmosphere using a muffle furnace. The optimised activated carbon, AK7, had a sorption kinetic that complied with the pseudo-second order kinetics and was fitted well to Langmuir isotherm model. The highest adsorption capacity of 97.09 mg g⁻¹was obtained when the samples (AK7) were subjected to activation temperature of 500°C for 60 minutes with the percentage of methylene blue removal efficiency of 99.5%.

Keywords: Papaya seeds, Activated carbon, Sorption studies

1. Introduction

Activated carbons are versatile adsorbents with a wide range of applications that concerns principally with the removal of impurity species by adsorption from the liquid or gas phase. They also find use as catalyst support, in waste water treatment, in sugar syrup purification, in air pollution control as well as in the pharmaceutical and chemical industries. Many researchers have prepared activated carbon from various carbonaceous precursors (Gergova 1994) such as various grain sorghum (Diao 2002), fruits stones, rice and peanut husk (Ahmedna 2000), wood (Anuar 2002, Anuar 2001), wheat straw (Ahmedna 2000), coconut and oil palm shell (Collin 2005, Collin 2006), etc. Activated carbon is also the most commonly used support in first aid process of detoxification, owing mainly to its highly porous structure and high surface area. In medical application, adsorption kinetic is an important consideration, as fast acting adsorbents can prevent poison from further being taken in by the gastrointestinal track. Adsorption kinetics depends on the surface area and porosity of the charcoal prepared. Activated carbons are highly effective in removing both natural and synthetic toxins in the gastrointestinal tract. It is also use to treat animals for drug overdose and helps prevent the absorption of most poisons or drugs by the stomach and intestines (Boyd 1999). When toxic substances enter the human or animal digesting system, activated carbon can attached the toxic molecule onto its surface. Because activated carbon is not digested, it stays inside the intestine tract and eliminates the toxin when the person or animal has a bowel movement (Vale 1993). Laxative medicine that stimulates bowel movement is sometimes given together with the activated carbon to shorten the amount of time taken for the carbon to move through the colon system and reduce the possibility of constipation and reabsorption of the toxin thorough the wall of intestine. The selection method of preparation and precursor was explained in our previous paper (Collin : CARBON 2006). In order to prepare activated carbon for medical use, the precursors must come from vegetable source and be low in ash content (Jankowska 1991). There is no international standard for medicinal activated carbon. Different pharmacopoeias specify the use of different compound (Methylene blue, Iodine, phenol and others) to determine acceptable adsorptive capacities of activated carbon. Literature survey showed little or no interest of these authors in preparing and characterizing medical grade activated carbon on a laboratory scale. Most efforts were towards the preparation for commercial and general use activated carbons. This led to a few queries. First, is it possible to prepare medical grade activated carbon under normal

activated carbon producing scale from papaya seeds? Second, how effective is the prepared activated carbon in removing arsenic from an aqueous system? Papaya seeds are agriculture by-product that currently has no economic value; can offer an inexpensive and economic gain-able source activated carbon. In spite of its abundant availability and ease of gain, this precursor has received little attention from researchers to exploit its potential as to date. A number of activated carbon significantly influences its adsorptive, electrochemical, catalytic, and other properties. However, few papers reported on the effects on chemical preparation conditions and preparation methods on the carbon. Generally, activation methods can be classified as single-stage or two-stage activation process carried out either in an inert medium or self-generated atmosphere (Collin : CARBON 2006). Since medical grade activated carbon are mostly employed in liquid phase of livestock's body fluid, it is essential to test its adsorption capacity in the same phase. This work describes the evaluation of activated carbons prepared from papaya seeds, by comparing the methylene blue adsorption kinetics of H_3PO_4 impregnated activated carbons in aqueous systems.

2. Experimental

The papaya seeds were collected daily from a local fruit stall in Kota Kinabalu, Sabah, Malaysia. After cleaning and processing it into activated charcoals using a muffle furnace, liquid phase adsorption studies were conducted. The chamber of muffle furnace consists of small vapor column with a small opening to the atmosphere, where decomposition of the precursors occurs in a mixture of initial existing atmosphere and gaseous decomposition produced during the process. Each of the samples was pretreated with phosphoric acid to precursor weight ratio of 1.0, 1.5, and 2.0 respectively (Collin : CARBON 2006) as shown in table 1. In both semi carbonization and activation processes, the samples were exposed to heat straightaway after the furnace temperature reached desire temperature. Since temperature progression was not the focus of the study, heating rate can be neglected. Desiccators served as the storing means while waiting for the furnace to attain the desired temperature. Leaching processes were crucial steps to remove the soluble ash formed during the activation process. Ash consisted mainly of oxides and some smaller portion of sulphates, carbonates, iron, aluminium, calcium, sodium and other metal and their compounds (Jankowska 1991). In the preparation of medical grade activated carbon, the content of heavy metal should be minimized; therefore it is advisable to remove the ash beforehand. Ash is commonly removed by leaching the activated carbon with acids (Jankowska 1991). As the samples were pretreated with phosphoric acid, refluxing the samples with distilled water was sufficient to remove the ash. The end product was characterized for percentage of yield, pH value, moisture and ash content, which was presented in our previous paper (Collin : CARBON 2006). In this paper, we present the sorption kinetics and sorption isotherm of the prepared carbon.

2.1 Methylene blue adsorption isotherm

The adsorption isotherms of methylene blue onto activated carbon were studied by applying the linear Langmuir equation 1

$$\frac{C_e}{Q_e} = \frac{1}{Q_m} C_e + \frac{1}{Q_m \cdot K_L} \tag{1}$$

and the Freundlich equation 2

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

Where C_e is the amount of adsorbate in the solution at equilibrium, Q_e is amount of methylene blue adsorbed by activated carbon, Q_m is the amount of adsorbate adsorbed to form monolayer coverage, and K_L , n and K_F are the equation constants (Rahman 2003).

2.2 Removal efficiency of methylene blue

The adsorption behavior of the activated carbon were studied by evaluating the removal efficiency, R_E , of methylene blue, calculated as equation 3

$$R_E = \frac{\left(C_o - C_e\right)}{C_o} \times 100 \tag{3}$$

Where C_o is the initial concentration of aqueous solution of methylene blue and C_e is the amount of adsorbate in the solution at equilibrium, R_E is expressed in term of percentage (Rahman 2003).

2.3 Iodine adsorption kinetics

0.1g of sample was added to a 0.25 L Erlenmeyer flask containing 10mgdm⁻³ aqueous solution of iodine. The flask was kept in a thermostat shaker at 30°C for 45 minutes. The content was gravity filtered and the concentration of the iodine determined. The procedure was extended to 60, 90, and 120 minutes. The kinetics and dynamics of adsorption of iodine on activated carbon was studied by applying the Lagergren first order and pseudo-second order rate equations that have

been most widely used for the adsorption of an adsorbate from an aqueous solution, which are expressed by the equations 4 and 5 respectively

$$\log(q_{e} - q_{t}) = \log q_{e} - \frac{k_{ad}}{2.303}t$$
(4)

$$\frac{t}{q_{t}} = \frac{1}{k_{ad}} \frac{1}{q_{e}^{2}} + \frac{1}{q_{e}}t$$
(5)

where q_e and q_t are the amount of dye adsorbed per unit mass of the adsorbent (in mgg⁻¹) at equilibrium time and time t, respectively, and k_{ad} is the rate constant.

2.4 Nitrogen adsorption by using BET method

BET isotherms were determined from the adsorption and desorption capacity of the activated carbons placed in inert atmosphere of nitrogen gas. It is assumed that the nitrogen molecules will form a single layer filling the pores on the surface of the activated carbon, which is known as the adsorption process. When the pressure in the chamber is gradually reduce, the single layer of molecule will leave the surface of the activated carbon, which is known as the desorption process.

3. Results and discussion

Methylene blue is a relatively large molecule and is often employed as an adsorbate in the liquid phase adsorption. It is a type of basic dye which is cationic (positively charged) and will therefore react with material that is negatively charged. Thus, it serves as a model compound for adsorption of medium size organic compounds from aqueous solutions (Stavropoulos 2005). It also indicates the specific surface area of the carbon which results from the existence of pores of dimension greater than 1.5nm (Jankowska 1991). Figure 1 shows the chemical structure of methylene blue molecule.

3.1 Methylene blue adsorption isotherm

The adsorption capacity of methylene blue using different impregnation ratio of phosphoric acid was shown in figure 2. In the impregnation ratio of 1.0, 1.5, 2.0, the loadings were very close, but still the trend suggested a continuous increase with the increasing of dye concentration. From figure 3, it is observed that the adsorption capacity of AK5 was higher than AK1. This indicates that the increase in activation temperature is proportionate to the adsorption capacity of methylene blue. These trends were consistent with the study done by Lafi (Lafi 2001). Figure 4 illustrates the relationship between the activation time and the adsorption capacity of methylene blue. Increasing the activation time could lead to pore damaged, severely affecting the carbons ability to adsorb the methylene blue as the activation time was increased from 30 to 90 minutes. When the adsorption data of AK1 to AK9 were tested using equation 1 and 2, they demonstrated compliance to the Langmuir model as indicated by its relatively higher linear correlation coefficients. The values of R² were all between 0.96 and 0.99 which indicating a good mathematical fit as shown in Table 2. The Langmuir adsorption model is a typical model for monolayer adsorption and was adapted to model isotherms and calculate the methylene blue (MB) adsorption capacities of the prepared activated carbons (Stavropoulos 2005). Table 2 summarizes the parameters of the methylene blue adsorption for Langmuir model and Freundlich model from AK1 to AK9. The highest adsorption capacity was AK 7 which was 97.09 mg g⁻¹, while the lowest was AK3 which was 52.91 mg g⁻¹. This may be due to the porosity of the activated carbon where AK 7 yielded the highest percentage of micropore; 86.87% as shown in Table 4, proving that adsorption capacity is proportionate to the surface area of the activated carbon (Lafi 2001). A ratio of surface area accessible to MB compared to BET surface area (S_{MB}/S_{BET}) is also given in the Table 2. Taking the surface area of methylene blue (S°_{MB} to be 1.93 m²mg⁻¹), the surface area of the activated carbons accessible to methylene blue (S_{MB}) was calculated according to equation 6.

$$S_{\rm MB} = S^{\circ}_{\rm MB} X q_{\rm m} \tag{6}$$

Where q_m is the amount adsorbed per unit mass of adsorbent corresponding to complete coverage of the adsorption site, calculated from the slope of linear Langmuir isotherm (Stavropoulos 2005).

Pore sizes create limited accessibility of methylene blue molecule in activated carbon. From table 2, the surface area, estimated by methylene blue adsorption, increased with activation temperature by comparing AK2 and AK6 which was 94.61 m²g⁻¹ and 155.65 m²g⁻¹ respectively. Higher temperature impelled the opening and enlargement of the pores, which increased the uptake of methylene blue. Besides that, the surface area estimated by the N₂ adsorption isotherms showed higher values than the surface area estimated by Langmuir isotherms. This was due to the size of N₂ molecule, which has a much smaller molecular cross-section than the methylene blue molecule. Therefore, N₂ adsorption can enter not only the mesopores, but also the micropores. The ratio of S_{MB}/S_{BET} indicates the fraction of porosity accessible to methylene blue molecule. AK 4 showed a larger S_{MB} compared to S_{BET} , therefore, might indicated that the adsorption mechanism was not limited to the porous nature. The S_{MB}/S_{BET} ratio for AK 5 and AK 9 showed that the methylene blue

only accessed about 17% and 18% of the total porosity of the carbon. This low coverage of internal surface area confirmed once again the microporosity character of the activated carbon since methylene blue adsorbs in mesopores (Stavropoulos 2005).

3.2 Removal efficiency of methylene blue

Figure 5 shows the removal efficiency of methylene blue versus initial concentration of methylene blue. The percentage of removal efficiency for all activated carbons was within the range of 68% to 99%. Figure 5 indicates that AK2, AK3 and AK4 had a low efficiency to remove the methylene blue. This trend indicates that longer activation time of the precursors which was prepared at 400°C with 1.5 impregnation ratio, may damaged the pore and affect its ability to remove methylene blue. AK7 had the highest removal efficiency of methylene blue, which was 99.5%.

3.3 Iodine adsorption kinetics

The iodine adsorption kinetics for the activated carbons studied in this work were presented in table 3. It was observed that the adsorption capacity followed the pseudo-second order model as indicated by its near relative high linear correlation. Researchers (Molino-Sabio 2004) indicated that this adsorption capability was due to the influence of the phosphoric acid to the porosity development in the carbon, which led to a more heterogeneous pore distribution. This is important for the fast uptake of adsorbates or toxic substances that enter the body as heterogeneous pore distribution enhances the surface area and serves as a pathway to the inner pores of the carbon. Phosphoric acid impregnation also helps to clean up the carbon surface groups that will hamper the electrostatic binding forces between the surfaces of the carbon and the adsorbates, thus, improving adsorption rate. Acid impregnated carbons are negatively charged (sometimes referred to as L-type carbons), therefore are most suitable to adsorb positively charged adsorbates. However some researchers (Ibezim 1999) have determined that the performance of some types of activated carbons can be severely hampered by acidic conditions in the gastrointestinal contents.

3.4 Adsorption isotherm of BET

Figure 6 shows the BET adsorption isotherms of the activated carbons were Langmuirian in shape and being typical type I isotherm with open hysterisis in the BDDT (Brunauer, Deming, Deming, Teller isotherm) classification, which is characteristic of adsorption on a microporous adsorbent. It tends monotonically to the limiting adsorption associated with a complete monolayer (Anuar 2004). These findings were corroborated by other researches (Youssef 2005, Yang 2003, Castro 2000, Guo 1998). The highest adsorption capacity was obtained by AK 9 whereas the lowest adsorption capacity was AK 4. This was due to its physical properties where the AK 4 produced the lowest BET surface area, micropore surface area, micropore volume.

3.5 Porosity of activated carbons

BET and micropore surface area, micropore volume, average pore diameter and percentage of micropore of activated carbon are given in table 4. Although the highest BET surface area of activated carbon was the sample AK9 which had $895.7m^2g^{-1}$, but, AK 5 yielded the highest for micropore surface area and micropore volume which was $687.5m^2g^{-1}$ and 0.276 cc g^{-1} respectively. Contrary, the lowest was AK4 which was $198.5m^2g^{-1}$ and $168m^2g^{-1}$. Moreover, the micropore volume of activated carbon was between $0.07ccg^{-1}$ and $0.27ccg^{-1}$ and $168m^2g^{-1}$. Moreover, the micropore volume of activated carbon of AK6 produced the highest average pore diameter with 21.3 Å while the impregnated activated carbon of AK7 produced the lowest average pore diameter with 18.4Å. The percentage of micropore showed that the H₃PO₄ impregnated activated carbon produced the highest percentage at 86.9%. This showed that the majority of the pores were micropores and a significant amount of pores have been enlarged to either mesopores or macropores. Therefore it was possible to suggest that the chemical activators had an effect in increasing pore sizes.

(a) Effect of Impregnation Ratio of H₃PO₄ on Porosity Development

Increased impregnation ratio was associated with an increased development in BET surface area as shown in table 4. An approximate decreased in micropore surface area, micropore volume and percentage of micropores appeared with respectively increased in impregnation ratio from 1.0, 1.5 to 2.0 (table 1) which was AK8, AK6, AK9 respectively. These results were generally consistent with results reported by Girgis 1999. This was due to the intense chemical attack of the highly concentrated H_3PO_4 , which collapsed the micropore and produced the development of meso and macroporosity (Molino-Sabio 2004).

(b) Effect of Activation Temperature on Porosity Development

Increasing the temperature from 400°C to 500°C progressively increased the BET surface area, micropore surface area and micropore volume. It can be observed by comparing the sample between AK1 and AK5 or AK2 and AK6 which was activated in 30 and 45 minutes respectively with 1.5 impregnation ratios. These results were consistent with the research done by other researchers (Castro 2000, Jagtoyen 1998, Solum 1995, Hayashi 2000). Therefore, a lower activation temperature produced microporous carbons (Diao 2002).

(c) Effect of Activation Time on Porosity Development

However when the activation duration was lengthened from 30 to 45 minutes, the BET surface area, micropore surface area and micropore volume also increased. But increasing the activation duration from 45 to 90 minutes caused the BET surface areas, micropore surface area and micropore volume to decrease. This trend was entirely consistent with the research done by (Diao 2002). This indicates that the pore structure of the carbon developed over a duration of 30 to 45 minutes would caused some of the pores to enlarge or even collapse, thus reducing the BET surface area and micropore s

4. Conclusion

In conclusion, this study showed that the use of dehydrating agent of H_3PO_4 and the two-stage self-generated atmosphere method played a major role in shaping and transforming papaya seeds into activated carbon with well-developed porosity and high adsorption capacity. Due to the activated carbon produced with carbonization and followed by physical activation in an open system (without gas control), only low temperature is needed to complete the decomposition of organic constituents. Generally, lower yield of activated papaya seeds were produced as the pyrolysis temperature and time increased. Activated carbons produced from papaya seed were basic due to the washing process. The activated carbons which were prepared by this method have been proven to be strongly effective in removing methylene blue dye in aqueous solution. It yielded a majority of micropores structure in which the percentage of micropore was between 82% and 87% with the average pore diameter of 19 to 21Å. The kinetic studies indicated that all the prepared activated carbons fitted well in Langmuir isotherms with pseudo-second order model. Besides being an adsorbent for aqueous based impurities, these activated carbons have a potential to be used in pharmaceutical or in medical industries as no toxic chemicals were involved in the activation process. Therefore it is hoped that papaya seeds will provide a clean, cheap and efficient precursor to prepare medical grade activated carbons for the purpose of gastrointestinal decontamination.

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Table 1. Preparation parameters of activated carbons AK1 to AK9.

Sample	Ratio of	Semi- carbonization		Activation	
	Impregnation	Temperature (°C)	Time (min)	Temperature (°C)	Time
					(min)
AK 1	1.5	200	15	400	30
AK 2	1.5	200	15	400	45
AK 3	1.5	200	15	400	60
AK 4	1.5	200	15	400	90
AK 5	1.5	200	15	500	30
AK 6	1.5	200	15	500	45
AK 7	1.0	200	15	500	60
AK 8	1.0	200	15	500	45
AK 9	2.0	200	15	500	45

Sample	Langmuir Model				Freundlich Model			
	R ²	K _L	q _m , mgg ⁻¹	$S_{MB} m^2 g^{-1}$	S_{MB}/S_{BET}	R ²	K _F	n
AK 1	0.97	0.496	81.30	156.91	0.27	0.83	3.95	1.86
AK 2	0.96	1.943	49.02	94.61	0.15	0.60	4.21	5.21
AK 3	0.99	1.853	52.91	102.11	0.19	0.59	4.29	5.03
AK 4	0.99	1.303	54.05	104.32	0.53	0.53	4.19	4.86
AK 5	0.99	2.288	74.07	142.96	0.17	0.83	4.98	3.21
AK 6	0.99	2.067	80.65	155.65	0.22	0.71	5.24	2.21
AK 7	0.99	4.120	97.09	187.38	0.24	0.94	7.17	1.72
AK 8	0.99	5.218	83.33	160.83	0.23	0.76	6.26	2.25
AK 9	0.99	4.680	85.47	164.96	0.18	0.69	6.20	2.38

Table 2. Methylene blue adsorption kinetics for activated carbons AK1 to AK9.

Table 3. Iodine adsorption kinetics for activated carbons AK1 to AK9

Lagergren kinetics						
	First order	Pseudo second order				
Sample	R ²	R ²	k _{ad}	q _e		
			$(\min g mg^{-1})$	$(mg g^{-1})$		
AK1	0.9040	1.0000	19.68504	0.1193		
AK2	0.8789	0.9998	454.5455	0.0048		
AK3	0.5932	0.9993	416.6667	0.0042		
AK4	0.8789	0.9999	384.6154	0.0078		
AK5	0.8954	0.9997	333.3333	0.0042		
AK6	0.9763	0.9997	294.1176	0.0072		
AK7	0.8954	0.9998	212.7660	0.0018		
AK8	0.9334	0.9994	270.2703	0.0015		
AK9	0.9763	0.9997	208.3333	0.0272		

Table 4. Porosity data of the activated carbons AK1 to AK9.

	BET surface	Micropore	Micropore	Average pore		Iodine
	area	surface area (m ²	volume (cc	diameter	Micropore	Number
Sample	$(m^2 g^{-1})$	g ⁻¹)	g ⁻¹)	(Å)	(%)	$(m^2 g^{-1})$
AK1	585.9055	498.9877	0.199241	19.9508	85.17	150.9
AK2	638.6633	521.4834	0.208899	19.4113	81.65	268.2
AK3	547.6578	456.4519	0.182578	19.7493	83.35	212.3
AK4	198.4713	168.0074	0.066648	19.8076	84.65	106.2
AK5	822.8388	687.4916	0.276198	18.5601	83.55	380.0
AK6	713.7507	588.9832	0.237155	21.3555	82.52	229.1
AK7	775.5432	673.7001	0.269542	18.3874	86.87	363.2
AK8	694.2739	602.0462	0.241316	18.5343	86.72	296.2
AK9	895.6582	553.6766	0.222338	19.6096	61.82	217.9



Figure 1. Chemical structure of methylene blue.



Figure 2. Adsorption isotherms with different impregnation ratio.



Figure 3. Adsorption isotherms with different activation temperature.



Figure 4. Adsorption isotherms with different activation time.



Figure 5. Removal efficiency of methylene blue



Figure 6. BET adsorption isotherm