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Original Research Paper

Methylene Blue Adsorption onto Low Cost Powdered Activated Carbon from Agricultural Waste - *Morus* Plant

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ABSTRACT

Activated carbon is one of the cheapest materials widely used in removal of pollutants from water and industrial effluents. Various waste materials, obtained from agriculture like rice husk, sugarcane bagasse, oil palm shell, coconut shell, coconut husk, bamboo cane, pongam seed coat, palm tree, cashew nut sheath, rubber seed coat have been reported as the raw materials for preparation of activated carbon. In this study carbon was prepared from *Morus* plant by chemical activation method. These samples were then activated at 800°C in a muffle furnace. The characterization studies have been carried out to assess their suitability as carbon adsorbents in treatment of dye house effluents. Batch adsorption studies were conducted to evaluate the effect of contact time and adsorbent dose on the removal of methylene blue dye at a temperature of 30°C. The results revealed that they are good adsorbents for the removal of the dye.

INTRODUCTION

Dyes are important class of water pollutants emanated from dye manufacturing and textile industries. Disposal of dye house effluent from textile industries has become a major problem in textile industry. The waste chemicals and dye-house effluents must be treated properly. Many physical and chemical treatment methods including adsorption, coagulation, precipitation, filtration, electrodialysis, membrane separation and oxidation have been used for the treatment of dye containing effluents. The adsorption process is one of the most efficient methods of removing pollutants from wastewater. Further, the adsorption process provides an attractive alternative treatment, especially, if the adsorbent is inexpensive and readily available (Ozacar & Sengil 2003). Activated carbon is the most widely used adsorbent for the removal of colour from textile effluents because it has a high capacity for organic matter (Jusoh et al. 2004). Early uses of carbon were reported for water filtration and for sugar solution purification. The ability of activated carbon to remove a large variety of compounds from contaminated waters has led to its increased use in the last thirty years. Adsorption capacity of activated carbon and its efficiency relies on the surface area, pore size distribution, adsorbate molecule size, temperature, pressure, moisture content, and concentration of adsorbate. Through the activation process, the carbon attains a very high surface area to mass ratio. Active carbon is an amorphous form of carbon, which is specially treated to produce a very large surface area ranging from 300 to 2000 m^2/g (Usmani 2001). Activated carbon has been frequently used as an adsorbent. Despite its extensive use in the water and wastewater treatment in industries, activated carbon remains an expensive material. In recent years, the need for safe and economical methods for elimination of pollutants from contaminated waters has necessitated research interest towards the production of low

cost alternatives to commercially available activated carbon (Nasim et al. 2004). Therefore, there is an urgent need that all possible sources of agro-based inexpensive adsorbents should be explored and their feasibility for the removal of pollutants should be studied in detail. Agricultural waste, which is easily available and inexpensive, may be a better option for adsorbent production. Moreover, it can be a way to avoid pollution caused by dumping or burning of the waste materials. Carbonization could be one of the options of disposing of waste materials. Carbonization products from plant and animal waste material may be utilized as a substitute for wood charcoal (Yoshiyuki & Yutaka 2003). Agricultural waste materials and byproducts used for the production of activated carbons include saw dust (Kadirvelu et al. 2000), turmeric waste (Karthikeyan et al. 2008), corn cob (Diaudeen et al. 2008), rice husk (Seiji et al. 2009), barley straw (Husseien et al. 2007), palm kernel shell (Jumasiah et al. 2006), cow dung (Rengasamy 2003), bagasse (Liew Abdullah et al. 2005), chestnut shell and grape seed (Diden et al. 2009), pine fruit (Tariq et al. 2009), *Phoenix sylvestris* leaves (Arivoli & Thenkuzhali 2008), sugar beet pulp (Ozer et al. 1998), and okra waste stem (Mohsen 2007). All these activated carbons have been successfully used for the adsorption processes.

India is basically an agricultural country with plenty of agricultural wastes. At present there is an urgent need to develop new and cheaper indigenously prepared activated carbons from the abundant agricultural wastes (Liew Abdullah et al. 2005). Agricultural by-products and some industrial wastes contain high carbon content and hence they can be used as starting/raw materials for the preparation of indigenously prepared activated carbons (Pattabiraman 1993). Although some attempts have already been made to economize the activated carbon (AC), the scope for minimizing its cost and development of alternative adsorbent materials to commercial AC by preparing ACs from agricultural wastes is still open. India produces more than 400 million tones of agricultural waste annually (Kannan & Veemaraj 2009).

Morus or mulberry is a genus of 10-16 species of deciduous trees native to warm, temperate, and subtropical regions of Asia, Africa, and the Americas, with the majority of the species native to Asia. The *Morus* plant of mulberry family is most important in silkworm industry. Silkworms feed on the leaves of the white mulberry. Mulberry fruits are tender and juicy and resemble blackberries. The fruits of *M. rubra* are made into wine and leaves are considered as a valuable agricultural and wildlife feed. Except leaves and fruits, the remaining parts of the mulberry plant become agricultural waste material. The present deals with the conversion of this waste into activated carbon for removal of methylene dye from coloured wastewaters.

MATERIALS AND METHODS

Raw material: The waste mulberry plant stem and roots were collected from local sericulture farms in and around Pudukkaraipudur village of Tamilnadu and cut into small pieces. The cut material was washed several times with water to remove soil and other impurities. The plant residue was dried in sunlight until all the moisture was evaporated. The dried material was cooled and powdered in a pulverizer. The *Morus* plant powder was stored in plastic containers to avoid moisture, ready for carbonization process.

Preparation of carbon: The dried material was treated with excess of sulphuric acid. Charring of the *Morus* powder occurred immediately, accompanied by evolution of heat and fumes. When the reaction subsided, the mixture was left in an air oven maintained at 120-130°C for a period of 24 hours. At the end of this period, the product was washed with large volume of water to remove free acid and dried at 110°C. The carbon obtained was subjected to thermal activation in minimum amount of

oxygen in a muffle furnace. The sample was packed into a stainless steel reactor and sealed to exclude as much air as possible. A tube hole was placed in the top of it in order to vent off gases from combustion of the sample during heating. The temperature was controlled manually during the heating operation. The sample was heated with an average heating rate of 3°C/min until a final temperature of 800°C was obtained, which was maintained for 30 minutes. The carbon obtained was pulverized in a blender and sieved to 100 mesh.

Methylene blue solution: Methylene blue (MB) (3, 9-bis dimethyl-aminophenazo thionium chloride) a cationic dye (Raghuvanshi et al. 2004), a G.R. product supplied from E. Merk chemicals Pvt. Ltd Company, was used without further purification. All the methylene blue solutions were prepared with distilled water. The stock solution of 1000 mg/L was prepared by dissolving 1g of methylene blue in 1000 mL distilled water. The experimental solutions with different concentrations were prepared by diluting the stock solution with distilled water (Sarioglu & Atay 2006). The concentration of methylene blue in the supernatant solution after and before adsorption was determined using a double beam UV/VIS spectrophotometer at a wavelength of 661 nm.

Characterization of activated carbon: Characterization of activated carbon was done by the procedure laid by ISI (1989). Conductivity and pH were analysed by conductivity meter and pH meter, moisture content (%) by mass, ash % (on dry basis) by mass, and volatile matter, bulk density, specific gravity, porosity, matter soluble in water by standard procedures.

Batch equilibrium studies: To evaluate the efficiency of adsorbents, laboratory batch mode studies were conducted. 0.05 g to 1 g of adsorbent, taken separately, was shaken in 100mL aqueous solution of dye of varying concentrations for different periods of time at natural pH. At the tend of predetermined time intervals, adsorbent was removed by centrifugation at 2000 rpm for 10 minutes and supernatant was analysed for the residual concentration of methylene blue, spectrophotometrically. The effects of time of contact and amount of adsorbent on the adsorption process were studied.

RESULTS AND DISCUSSION

The average values of moisture and ash content of the activated carbon obtained from *Morus* plant (ACM) were 1.34% and 10.54% respectively (Table 1). The carbon yield was 24.4%, and it was greater than those reported earlier using coconut shell as raw material and fruits of *Khaya senegalensis.* The present results also show that the lower the moisture and ash content the greater the % yield of activated carbon (Casmir 2009). As water competes with organic molecules for activated sites on carbon, the lower the moisture content the higher the adsorptive capacity. One gram of activated carbon can have a surface area in excess of 500 m^2. The surface area of activated carbon obtained from *Morus* plant was 885.38 m^2/g (Table 2). The activated carbon produced from mulberry plant has high surface area and highly developed micropore structure than the commercial activated carbon. The mulberry activated carbon has a pH of 9.56. This is in agreement with earlier work (Cheremisinoff & Ellerbusch 1978), which showed that alkaline surfaces are characteristic of carbons having vegetable origins. The amount of MB adsorbed is expected to be more favourable as the carbon surfaces become more alkaline (Abram 1973). The relatively high bulk density of 0.996 g. mL^{-1} indicates longer period of filtration cycle (Abram 1973).

Table 1: Surface area (m^2/g) of different substances.

1	Activated carbon	500-1500
2	Activated clays	150-225
3	Silica gel	200-600
4	Activated alumina	175
5	Kieselghur	4.2
6	Fused copper	0.23

Effect of adsorbent dose and time: Generally, adsorption of methylene blue has been considered as one of the

Table 2: Properties of activated carbon prepared from *Morus* plant.

pH	9.56
Conductivity	2.53, mho cm^{-1}
Bulk density	0.9663, g/mL
Moisture	1.34, %
Ash	10.54, %
Specific gravity	1.89
Surface area	885.38, m^2/g
Water soluble matter	7.62, %
Volatile content	0.9, %
Porosity	48.87, %

most important means of assessing removal capacity from the aqueous phase. The adsorption of methylene blue dye on activated carbon from *Morus* plant has been studied by changing the quantity of the adsorbent (0.05, 0.1, 0.2, 0.3, 0.4, 0.5 and 1.0 g/100 mL) in the test solution while keeping the initial dye concentration (10 mg/L), temperature (29±1°C) and pH constant. Experiments were carried out at different contact times for 120 minutes. The adsorption by activated carbon from *Morus* plant (ACM) is presented in Table 3. From the data, the amount of dye adsorbed at various intervals of time indicates that the removal of dye initially increases with time but attains equilibrium within 40-50 minutes. The adsorption process was found to be very rapid initially, and a large fraction of the total concentration of dye was removed in the first 30 minutes. It was observed that an increase in the dose of the adsorbent resulted in the increase in adsorption of methylene blue dye in the solution. Increase in the adsorption with adsorbent dose can be attributed to increase in adsorbent surface area and availability of more adsorption sites. These preliminary studies state that adsorbents prepared from unused parts of *Morus* plant can be used effectively for adsorption of dyes.

CONCLUSION

The AMC is quite effective in removing methylene blue from aqueous solution. The percentage of removal increased with the increase of contact time and achieves equilibrium within a short time. About 89 % of methylene blue dye was removed from the test solution. The AMC showed better

Table 3: Effect of adsorbent dosage on MB removal by activated carbon from *Morus* plant (ACM).

Time min	50 ppm		100 ppm		150 ppm		200 ppm		250 ppm	
	abs	%	abs	%	abs	%	abs	%	abs	%
10	0.818	48.8	0.608	61.9	0.452	71.7	0.415	74.0	0.391	75.5
20	0.785	50.8	0.458	71.3	0.444	72.2	0.370	76.8	0.324	79.7
30	0.754	52.8	0.455	71.6	0.422	73.6	0.355	76.9	0.314	80.3
40	0.705	55.8	0.451	71.7	0.418	73.8	0.342	78.6	0.276	82.7
50	0.610	61.8	0.449	71.9	0.408	74.4	0.336	79.0	0.245	84.7
60	0.608	61.9	0.446	72.0	0.378	76.3	0.316	80.2	0.242	84.8
90	0.605	62.0	0.445	72.1	0.362	77.3	0.297	81.4	0.241	84.9
120	0.604	62.1	0.441	72.3	0.350	78.1	0.266	83.3	0.231	85.5
Time min	300 ppm		350 ppm		400 ppm		450 ppm		500 ppm	
	abs	%	abs	%	abs	%	abs	%	abs	%
10	0.334	79.09	0.323	78.5	0.265	83.4	0.222	86.1	0.211	86.8
20	0.306	80.83	0.219	86.28	0.250	84.34	0.219	86.4	0.198	87.6
30	0.204	87.22	0.205	87.16	0.221	86.15	0.196	87.7	0.195	87.8
40	0.203	87.28	0.200	87.47	0.192	87.97	0.192	88.0	0.184	88.5
50	0.184	88.47	0.191	88.03	0.192	87.97	0.188	88.2	0.184	88.5
60	0.183	88.53	0.177	88.91	0.188	88.22	0.181	88.7	0.182	88.6
90	0.178	88.85	0.175	89.04	0.177	88.91	0.175	89.04	0.174	89.1
120	0.177	88.91	0.174	89.1	0.174	89.1	0.174	89.1	0.173	89.2

performance compared to commercially prepared activated carbon. The present study concludes that activated carbons are effective in removing methylene blue. In addition activated carbon can be conveniently and economically prepared from the mulberry plant. The waste mulberry plant is available abundantly at a small price and can be obtained as agricultural byproduct in the country.

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