Decolourization of Textile Effluents to Maintain Water Quality by Using Activated Carbon

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Abstract

Textile industries use large amount of dyes for finishing process. These dyes are chemical compounds which become a serious environmental problem if they are discharged as wastewater without any treatment. Three activated carbons were prepared from bio-waste material which is characterized by SEM, FTIR and TPD finally their adsorption efficiency in removal toxic materials from textile effluents was tested. During sequential operation of these carbons, textile effluents were decolorized with better results like 7, 5 and 3 m⁻¹ absorbance at wavelengths of 436, 525 and 620 nm respectively. 2 g of each adsorbent and at optimum contact time of 40 min removed 93% of colour form collected textile effluents.

Keywords

Textile effluents, Removal, Dye, SEM, FTIR

I. Introduction

Water is the most precious, limited natural resource on this biosphere which is essential to the survival of all living beings. Discharge of effluents from industrial processes adds hazardous chemicals to surface and ground water. Textile industries consume large volumes of water, dyes and auxiliary chemicals for processing of textiles. Due to incomplete exhaustion and washing operations, 10-20% of dyes were discharged into effluents [1]. Many of these dyes were toxic and carcinogenic thus affecting the aquatic biota and human health [2]. The world population was expected to be increased by 35% by 2050 [3]. This population growth will increase the production of clothes, which in turn, increases fresh water use. So conserving water and reducing water pollution will become a challenging and essential task for textile industries. Numerous methods are available for the removal of colour from waters and wastewaters such as membrane separation, aerobic and anaerobic degradation using various microorganisms, chemical oxidation, coagulation and flocculation, and reverse osmosis. Some of these techniques have been shown to be effective however they have some limitations such as excess amount of chemical usage, accumulation of concentrated sludge that has serious disposal problems and lack of effective colour reduction. The adsorption technique, which is based on the transfer of pollutants from the solution to the solid phase, is known as one of the efficient and general wastewater treatment method [4]. The method is superior to other dye removal techniques in terms of initial cost, simplicity of design, ease of operation, and non-toxicity of the utilized adsorbents compared to other conventional wastewater treatment methods [5]. The cost effectiveness, availability and adsorptive properties are the main criteria in selection of an adsorbent to remove organic compounds from wastewaters [6], also application of adsorption procedure especially based on non-toxic and green adsorbent with high surface area and reactive surface atom is a great demand [7].

In industrial processes activated carbons are widely used as adsorbents which are composed of a micro porous, homogenous structure with high surface area and shows radiation stability [8]. The process for producing high-efficiency activated carbon is not completely investigated in developing countries. Furthermore, there are many problems with the regeneration of used activated carbon. Nowadays, there is a great interest in finding inexpensive and effective alternatives to the existing commercial activated carbon [9]. Exploring effective and low-cost activated carbon may contribute to environmental sustainability and offer benefits for future commercial applications. The costs of activated carbon prepared from biomaterials are very low compared to the cost of commercial activated carbon. Waste materials that have been successfully used to manufacture activated carbon in the recent past include waste wood [10], bagasse [11] orange peel [12], coffee husk [13], pine cone [14], coconut tree [15], sunflower seed hull [16], and Coconut husk [17].

The main objective of the present study is to prepare activated carbons from Sugarcane (SC) waste then the prepared activated carbons were characterized by TPD, FTIR and XPS. The effluents are collected from three different places of Mangalagiri town of Guntur district, Andhrapradesh, India. The efficiency of prepared carbons in decolorizing these textile effluents was studied.

II. Materials and Methods

A. Preparation of Activated Carbons

The raw material Sugar Cane (SC) waste was collected from local juice stall Vijayawada, A.P., India. The waste of sugarcane was used as precursor for preparation of activated carbon. The waste was washed with hot distilled water dehydrated at 110oC. This dried waste was then cut into small pieces. It was mixed with K₂CO₂ solution in 1:1 ratio and was carbonized in uniform nitrogen flow at 500°C. The heating was provided at rate of 10°C min⁻¹. At high temperature more than 500°C, some micropores might have combined to give large number of measopores and macropores. As a result the surface area of prepared activated carbons might be decreased at this temperature. The prepared activated carbon were cooled to room temperature and washed with hot distilled water to remove remaining chemicals and filtered. The washing and filtration steps were repeated until the filtrate showed neutral pH and finally dried. The carbon is named as SC₅₀₀. In order to introduce different functional groups it was divided into two parts. One part was subjected to liquid phase oxidation with 0.1N HNO₂ and the other part was soaked in 0.1N KOH for 3 hr and evaporated at 110°C. Both carbons were washed with distilled water until filtrate showed neutral pH. These carbons were dehydrated in an oven overnight at 105°C and named as 'SC_{HNO3}' and 'SC_{KOH}', indicating the chemical activating agent.

B. Characterization of Prepared Activated Carbons

Thermal stability of carbons was analyzed by temperature programmed desorption (TPD) studies. Point of zero charge was determined for SC_{500} , SC_{HNO3} and SCKOH. The nature of functional groups present on surface of activated carbons was

analyzed by FTIR technique.

C. Determination of Point of Zero Charge (PZC)

Point of zero charge is the pH at which the external surface charge is zero [18]. 50 ml of 0.01N NaCl solution was placed in a closed Erlenmeyer flask. The pH was adjusted to a value between 2 and 12 by adding 0.1 N HCl or 0.1 N NaOH solutions. Then 0.15 g of carbon sample was added and the final pH measured after 48 hr under agitation at room temperature. The PZC is the point where the curve $pH_{\text{(final)}}$ vs. $pH_{\text{(initial)}}$ crosses the line $pH_{\text{(final)}} = pH_{\text{(initial)}}$ and was taken as the PZC of the given carbon [19].

D. SEM Analysis

A scanning electron microscope SEM Hitachi- S520 (OXFORD LINK-ISIS) was used to study the texture of the surface of activated carbons.

E. FTIR Analysis

The surface chemistry characterization of each activated carbon was performed by Fourier Transform Infrared Spectroscopy (FTIR) to identify its surface functional groups. FTIR spectra were recorded on Thermo Nicolet Nexus 670 spectrometer in the wave number range 400-4000 cm⁻¹. To prepare pellets, samples were first ground to powder in an agate mortar and then mixed with KBr at a mass ratio of 1:500. A hydraulic press was used to press the resulting mixtures to discs of 10 mm in diameter at 10 MPa for 5 min.

F. TPD Analysis

TPD analysis was performed on MICROMERITICS CHEMISORB 2750 instrument. Activated carbon samples were kept in high pure Helium flow for 10 min. Gas changed to 10% NH₃ in Helium at a rate of 30 ml min⁻¹ flow. NH3 adsorbed for 30 min. Gas changed to pure Helium (30 ml min⁻¹) and physisorbed ammonia was removed for stable baseline status.

Table 1: Point of Zero Charge Values of Selected Activated Carbons

Activated carbon	PZC Value						
SC ₅₀₀	6.7						
SC _{HNO3}	4						
SC _{KOH}	10						

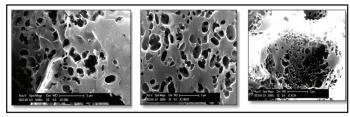


Fig. 1: SEM Images of (a) SC_{500} , (b) SC_{HNO3} and (c) SC_{KOH} at 5 k X magnification

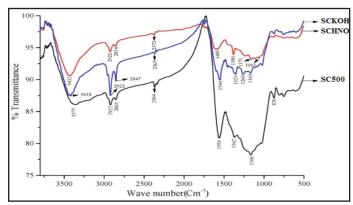


Fig. 2: FTIR Analysis of SC_{500} , SC_{HNO3} and SC_{KOH}

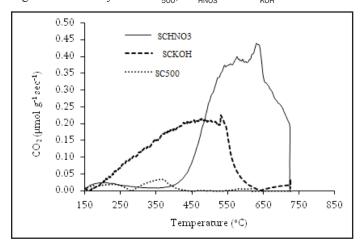


Fig. 3(a): TPD Analysis of CO₂ evolution profiles of SC₅₀₀, SC_{HNO3} and SC_{KOH}

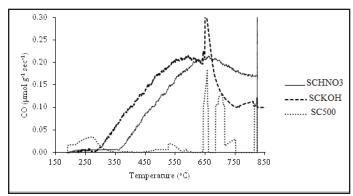


Fig. 3(b): TPD Analysis of CO Evolution Profiles of SC₅₀₀, SC_{HNO3} and SC_{KOH}

III. Results and Discussions

A. SEM Analysis

Fig. 1 represents SEM images of SC_{HNO3} , SC_{KOH} and SC_{600} activated carbons. All these SEM images were taken at 5k X magnification. Significant surface morphological changes were observed on each carbon adsorbent.

The PZC values of SC $_{\rm 500}$ (at IR1), SC $_{\rm HNO3}$ and SC $_{\rm KOH}$ were tabulated in Table 1. The PZC values of SC $_{\rm HNO3}$ and SC $_{\rm KOH}$ were changed from 6.7 to 4 and 10 respectively. It might be due to the change in their surface functional groups generated by liquid phase oxidation.

B. FTIR Analysis

FTIR spectra of SC_{500} , SC_{HNO3} and SC_{KOH} were shown in fig. 2. A distinct peak was observed at $1558\,\mathrm{cm^{\text{-}1}}$ for SC_{500} only. This might

be due to polyaromatic C=C stretching vibrations in sp2-hybridized carbons [20, 21]. This indicated the non-polar (hydrophobic) nature of SC500. However, C=O stretching vibrations of quinones might also be included at this peak. These might have produced quinone type functional groups on surface of SC $_{500}$. Bands in the range 1350-1380 cm $^{-1}$ were attributed to C-N bond stretching vibrations of aryl amines which might appear because sugarcane contains amino acids. A series of small peaks in the region 870 cm $^{-1}$ were attributed to aromatic C-Cl bonds.

In comparison with other FTIR graphs (of SC_{HNO3} & SC_{500}), SC_{KOH} is having very sharp peaks at 2922 and 2841 cm⁻¹. This might be attributed due to the presence of 2H-chromene as reported elsewhere [22, 23]. The V-shaped band at 3424 cm⁻¹ might be attributed to the O-H stretching of alcohols. SCKOH activated carbon was prepared by treating SC_{600} with potassium hydroxide and evaporated at 110°C and then washed with distilled water. It was observed that SC₅₀₀ was having quinone type functional groups. Quinones were sensitive to the action of aqueous alkalis, in the presence of which, depending on the conditions, they decompose to organic acids [24], form hydroxy-derivatives of quinones [25] or polymerize to products of the type of the huminic acids [26]. The presence of 2H-chromene might be the result of these reactions. Similar type of surface modifications were observed when char of Jatropha curcas L waste was mixed with KOH solution and evaporated at 80°C [27]. The bonds present at 870 cm $^{-1}$ were disappeared in SC_{KOH} activated carbon. The C-Cl bond might have been converted to C-OH bond during the treatment of SC₅₀₀ with KOH.

B. TPD Analysis

Fig. 3 (a) and 3 (b) shows TPD analysis profile for CO₂ & CO desorption from SC_{500} (at IR1), SC_{HNO3} and SC_{KOH} . Surface oxygen groups present on these activated carbons decomposed upon heating. They release CO₂ and CO at different temperatures. CO₂ peaks result from the decomposition of carboxylic acids [28] at low temperature or lactones at high temperature and anhydrides give both CO and CO₂. Groups like phenols, carbonyls, ethers and quinones originate CO peaks. When compared with SC_{HNO3} (shown in Fig. 3(a)), SC_{KOH} had less number of oxygen functional groups, but much higher than SC_{500} . In both CO_2 and CO desorption profiles, SC_{500} showed less oxygen content. However, comparatively, SC₅₀₀ showed more oxygen content in CO profile than that of CO₂ desorption studies. It may be assumed that during preparation of activated carbon, heat treatments under inert atmosphere at high temperature effectively removed oxygen. However active sites present on activated carbon might have readsorbed oxygen to less extent. Hence SC_{500} had very less oxygen

content which in other words provide more hydrophobic surface. Activated carbon with more hydrophobic surface was one of the required properties for adsorption of non-ionic organic pollutants from aqueous solution. In desorption studies of CO profile, SC_{KOH} showed release of CO at around 750°C. It evidenced that soaking the activated carbon in potassium hydroxide solution changed the surface chemistry of carbon by introducing different oxygen functional groups.

IV. Application

The prepared carbons were used to remove toxic and carcinogenic dyes from textile effluents which are collected from a place called Mangalagiri of Guntur district, Andhrapradesh. Mangalagiri is world famous for the handmade pure cotton sarees. It is a major hub for cotton trade with end to end cotton related industries situated in the region supporting a major portion of population. Weavers in and around Mangalagiri tend to work from home with all members of family giving in their bits at different levels of the weaving process. Textile effluents are collected from fabric dyeing place in Mangalagiri. Weaving of series on looms is the livelihood for many people in Mangalagiri. These weavers are using different dyes in dye bath for the requirement of colour. These commercial dyes contain mixture of dyes and does not provide any scientific information regarding structure, chemical composition etc.

A. Treatment of Industrial Textile Wastewater

All the experiments were carried out in 250 ml conical flasks with 100 ml textile effluent at room temperature (25±2°C). Textile effluents were directly taken without prior filtration. The flasks, along with test solution and 1g of the adsorbent were shaken in horizontal shaker at 120 rpm for 60 min and filtered. For this 100 ml of textile effluent was first treated with SC_{500} and the solution was filtered, the filtrate was treated with SC_{HNO3} adsorbent for 1 hr. It was then filtered and finally similar process was also followed with SC_{KOH} activated carbon under similar experimental conditions. Applying German standard method DIN 38 404 [29], measurement of colour absorbance (in m-1) was done at the standard wavelengths of 436, 525 and 620 nm at each stage [30].

The colour intensity of the sample was expressed as absorption coefficient calculated from the equation

$$Color (absorbance, m^{-1}) = \frac{absorbance \ X \ dilution \ factor}{path \ length \ (m)} \quad (1)$$

COD and BOD analysis was performed using American Public Health Association method [31].

Table 2. Textile effluents characteristics before treating with carbons												
es				lity	Conductivity (mS cm ⁻¹)	nded solid	d grease	Colour absorbance (absorbance m ⁻¹)				
Samples	Hd	COD (mg l ⁻¹)	BOD (mg l ⁻¹)	Turbidity (NTU)	Condu	Suspended (mg l ⁻¹)	Oil and (mg l ⁻¹)	436 nm	525 nm	620 nm		
Sample 1	7	358	50	8.2	3.1	10	2.1	85	125	110		
Sample 2	8	260	80	9.6	2.5	21	1.9	45	80	55		
Sample 3	6	430	30	12.1	1.9	17	1.4	115	85	65		

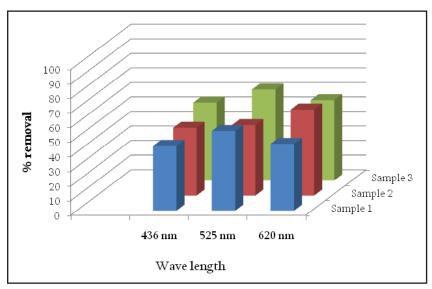


Fig. 4: Testing the efficiency of prepared adsorbents in colour removal from textile effluents

B. Characteristics of textile effluents after treating with carbons

After treating with SC_{500} , SC_{HNO3} and SC_{KOH} activated carbons the changes in characteristics of textile effluents were compiled in Table 2 and the corresponding graph was shown in Fig. 4. After treating with activated carbons, great reduction in COD and BOD values was also observed.

Table 3. Testing the efficiency of prepared adsorbents in colour removal from textile effluents

	рН	COD (mg l ⁻¹)	BOD (mg l ⁻¹)			Colour absorbance (absorbance m ⁻¹)													
Samples		SC_{500}	${ m SC}_{ m HNO3}$	SC_{KOH}	SC_{500}	${ m SC}_{ m HNO3}$	$\mathrm{SC}_{\mathrm{KOH}}$	SC ₅₀₀	SC ₅₀₀			SC _{HNO3}			SC _{KOH}			% Colour removal		
								436 nm	525 nm	620 nm	436 nm	525 nm	620 nm	436 nm	525 nm	620 nm	436 nm	525 nm	620 nm	
Sample 1	6.8	175	86	37	21	20	20	84	115	98	65	70	79	53	55	62	44.22	54.55	45.62	
Sample 2	8.4	154	78	26	45	35	34	35	63	52	29	58	31	22	44	23	46.35	48.24	58.93	
Sample 3	6.7	211	108	59	15	11	11	98	75	51	62	42	36	49	35	31	53.34	62.37	55.08	

Moderate colour removal was observed for all samples for all three sorbents and was tabulated in Table.3. The removal of colour from the effluents was lower than sorption from a single component system. In order to fix dyes to the fiber strongly, different type of chemicals used as fixing agents, might be a cause of this moderate percent removal and not only that it could be due to the following factors, (i) interaction between the dyes and other components in the effluents, (ii) change of adsorbent surface due to adsorption and (iii) competition of other components of the effluent for active sites on the carbon surface where displacement effects replace the other components from the adsorption sites.

C. Decolorization of textile effluents with increasing adsorbent dosage:

To achieve better results like 7, 5 and 3 m⁻¹ absorbance at wavelengths of 436, 525 and 620 nm respectively, the dose of adsorbents increased for treatment of three effluents at same contact time of 60 min and the corresponding graphs were shown

in Fig.5 for three samples. In all cases, it was observed that as the adsorbent dosage increased, the percent removal of colour of the effluents also increased.

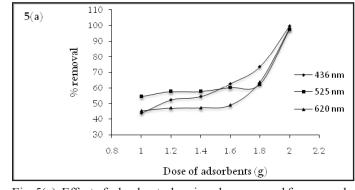


Fig. 5(a). Effect of adsorbents dose in colour removal from sample

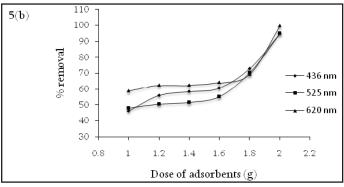


Fig. 5(b). Effect of adsorbents dose in colour removal from sample

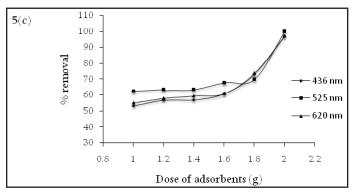


Fig. 5(c). Effect of adsorbents dose in colour removal from sample

In sequential operation with SC_{500} , SC_{HNO3} and SC_{KOH} of sample 1, increase in percent removal from 44.22 to 100 % at 436 nm, 54.55 to 97.53% at 525nm and 45.62 to 98.25 % at 620 nm was observed when the dose increased from 1 to 2g.

In case of sample 2, the percent removal increased from 46.35 to 95.13 % at 436 nm, 48.24 to 95.30 at 525 nm and 58.93 to 100 % at 620 nm.

In case of sample 3, the percent removal increased from 53.34 to 97.15 % at 436 nm, 62.37 to 100 at 525 nm and 55.08 to 97.11 % at 620 nm observed.

Good response was identified by increasing the adsorbent dosage in decolorization of textile effluents.

D. Decolorization of textile effluents with increasing contact time

To find the optimum contact time of adsorbents for removal of colour from textile effluents to their permissible value, the same experiment was done by increasing the agitation time with fixed adsorbent dose (2 g). The corresponding graphs were shown in Fig. 6 for three samples.

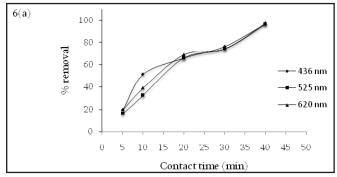


Fig. 6(a). Effect of contact time of adsorbents in color removal from sample 1

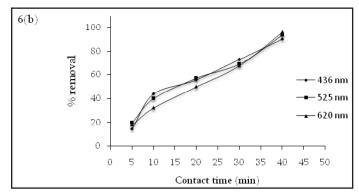


Fig. 6(b). Effect of contact time of adsorbents in color removal from sample 2

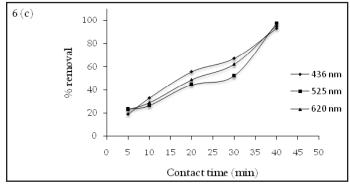


Fig. 6(c). Effect of contact time of adsorbents in color removal from sample 3

It was noticed that in all adsorption systems the dye removal was increased with increase in agitation time and reached equilibrium within 40 min. The observed percent removal of sample 1, 2, and 3 were 97.37, 96.43 and 95.66% respectively.

V. Conclusion

The presence of both micro and mesopores on this carbon may facilitate the adsorption of different types of pollutants with varying sizes. TPD analysis revealed that SC_{HNO3} and SC_{KOH} were having more oxygen content than SC_{500} . All the three activated carbons were thermally stable up to 300°C. Hence these can be used for adsorption of pollutants from aqueous solutions at elevated temperatures also. FTIR analysis pointed out that surface chemistry was peculiar and specific for each activated carbon. All the collected effluents were highly coloured and prior treatment was always necessary before discharging. The prepared activated

carbons successfully removed the colour of the effluents. It was observed that besides removal of colour, there was concurrent reduction of COD and BOD of the effluents. The effluents treated until the acceptable limit of 7, 5 and 3 m⁻¹ absorbance reached at the standard wavelengths of 436, 525 and 620 nm respectively within 40 min and by using 2g of each adsorbent.

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