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Comparative studies on adsorption of dyeing industry effluents by activated carbon and polymer coated sawdust prepared from the fruit of *Cordia sebestena*

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ABSTRACT

Activated carbon (*Cordia sebestena* activated carbon [CSAC]) and polymer coated sawdust (polypyrrole coated sawdust [PPy/SD]) prepared from the precursor, fruit of the gardening plant material *Cordia sebestena* were used as adsorbents to treat the dyeing industry effluent samples which were collected immediately after the completion of dyeing process (at the outlet of dyeing machine). Five samples of industrial effluents were taken and their physicochemical characteristics like pH, hardness, total dissolved solid, dissolved oxygen, biochemical oxygen demand and chemical oxygen demand were analysed. Thus, among the two adsorbents, CSAC is a potential adsorbent for most of the dye effluents studied and have moderate colour removal capacity for the sample 3. But polymer coated sawdust (PPy/SD) is an excellent adsorbent for the colour removal of almost all the dye effluents studied. The amount of adsorbents required for the treatment and the cost of the adsorbents were also compared with the commercial charcoal. Hence from the comparison studies, it was found that the treatment of effluents with CSAC is more economical than commercial charcoal and PPy/SD. It could be concluded that even though the price of PPy/SD is higher than others, the amount required for treatment of the effluent samples is very less compared with CSAC and commercial charcoal. Thus, PPy/SD proves that it is an excellent adsorbent and it has high potentiality for the removal of colour from industry effluent as compared with others.

Keywords: Activated carbon; Polymer coated sawdust; Adsorption; *Cordia sebestena* fruit; Dyeing industry effluent

1. Introduction

The textile industry is one of the most complicated industries among manufacturing industry. Wastewater treatment is one of the major problems faced by textile manufacturers. Removing colour from wastewater can be done via several methods namely chemical, biological and physical methods. Physical methods often applied are membrane filtration and adsorption techniques [1]. Among these methods, adsorption using activated carbon as adsorbent has been found to be superior due to low-cost, simplicity of design, flexibility, ease of operation and insensitivity to toxic pollutants [2]. Thus, adsorption has been used extensively in industrial process for separation and purification. The removal of coloured and colourless organic pollutants from industrial wastewater is considered as an important application of adsorption processes [3].

A successful adsorption process not only depends on dye adsorption performance of the adsorbents, but also on the constant supply of the materials for the process. Hence, it is preferable to use low-cost adsorbents, such as an industrial waste, natural ores and agricultural by products. Recently, numerous approaches have been studied for the development of cheaper and effective adsorbents [4]. At present, there is a growing interest in using low-cost, commercially available materials for the adsorption of dyes. A wide variety of materials such as peat [5], various silicas [6], activated clay [7], banana pith [8],

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natural manganese mineral [9], goat hair [10], alum sludge [11] and mixtures of fly ash and soil [12] have been investigated as low-cost alternatives to activated carbon [13].

But new, low-cost, locally available and highly effective alternative dye adsorbent materials were still needed to search. It is therefore of interest to investigate the possibilities of the use of fruit of *Cordia sebestena*, a gardening plant material having no economical importance and considered its fruit as waste material. Thus in this study, the fruit of the gardening plant material *Cordia sebestena* was used as a precursor for the preparation of two adsorbents namely activated carbon (CSAC) and polypyrrole coated sawdust (PPy/SD). Hence, the detailed study of treatment of dyeing industrial effluent samples with the two adsorbents namely CSAC and PPy/SD were analysed for its colour removal. Cost analyses of these two adsorbents with commercial charcoal were also compared in order to find out the efficiency of adsorbent.

2. Experimental

2.1. Preparation of activated carbon

The fruit of *Cordia sebestena* which is considered as waste was dried in sunlight for 10 d. Material was soaked in a boiling solution of 10% H_3PO_4 for 2 h and kept at room temperature for 24 h. At the end of 24 h, the excess solution was decanted off, the material was air dried and carbonised in muffle furnace at 400°C for 30 min. The carbon was powdered and activated in a muffle furnace at 800°C for a period of 10 min. Then, the carbon was washed with plenty of water to remove residual acid, dried and powdered with a particle size of 180–300 μm.

2.2. Preparation of polypyrrole coated sawdust

PPy/SD was synthesized on sawdust surface of the fruit of *Cordia sebestena*. In order to prepare PPy/SD, 5.0 g sawdust immersed in 50 mL of 0.2 M freshly distilled pyrrole before polymerisation. The excess of the monomer solution was removed by simple decantation. Then, 50 mL of 0.5 M ferric chloride as an oxidant solution was added into the mixture gradually and the reaction was allowed to continue for 4 h at room temperature. The PPy/SD was filtered, washed with distilled water, dried in an oven at about 60°C and sieved (with a particle size of 180–300 μm) before use [14]. The coating percentage of each polymer onto sawdust determined by weight difference of the dried sawdust before and after coating and it was nearly 5% [15].

2.3. Batch experiments for selected synthetic dye solution

Batch adsorption experiments were conducted by shaking 0.1 g of CSAC, and PPy/SD with 100 mL aqueous solution of various categories of dyes at a concentration of 50 ppm for 3 h time duration at 30°C of temperature with natural pH conditions. The adsorbent was removed by centrifugation and the concentration of dye in the supernatant liquid was determined spectrophotometrically (Elico make BL-198). The same procedure was followed for the dye house effluent with the prepared two adsorbents. The result of the percentage of adsorption for the selected synthetic dyes and for the effluent is given in Table 1.

It was found that the percentage of adsorption for the selected synthetic dyes and for the effluent is increased in the order for the prepared two adsorbents: CSAC << PPy/SD.

Thus, the percentage removal is high for PPy/SD adsorbent.

The detailed kinetic and thermodynamic studies of the selected synthetic dyes for the prepared adsorbents have carried out [16–19].

2.4. Detailed adsorption studies of effluent

Dyeing industrial effluent samples were collected from various dye processing industries in Perundurai SIPCOT (State Industries Promotion Corporation of Tamil Nadu Ltd.) industrial estate. The wastewater samples were collected immediately after the completion of dyeing process (at the outlet of dyeing machine). We have collected the effluent on different days based on their processing of different fabric with different dye usage. But, we have made the analysis of effluent on the day of collection itself. The samples were analysed for the physicochemical characteristics as per the standard procedures [20–22]. Various methods employed for the analysis are given in Table 2. The taken effluent samples (five samples) concentration were analysed using Bio UV–Visible spectrometer (Elico make BL-198) by fixing the λ_{max} as the absorption wavelength.

Then, 200 mL of the effluent was agitated with CSAC and PPy/SD for 4 h. The adsorbent gradually added into the effluent until the maximum decolourisation was achieved. All the effluent samples were agitated with commercial charcoal (with a particle size of 180–300 μm) by increasing the dosage gradually until the maximum possible decolourisation was attained. Then, the supernatant solution was separated using centrifuge apparatus and analysed. By using Bio UV–Visible spectrometer (Elico make BL 198), the percentage of colour removal was determined based on absorbance (OD) at λ_{max} (Table 3). The required dose of adsorbents (CSAC, PPy/SD and commercial charcoal) varies from sample to sample. On the basis of the optimum dose of adsorbent required to treat 100 M^3 of dye house effluent, the approximate cost of effluent was computed and compared with the commercial charcoal.

2.5. Characterisation of adsorbents

Physicochemical characteristics of adsorbents were analysed as per the standard testing methods [20–22].

Table 1
The percentage of adsorption of selected synthetic dyes and effluent

S.No.	Name of the dye	Percentage of adsorption	
		CSAC	PPy/SD
1	Acid Orange 7	66.19	89.23
2	Basic Red 29	68.53	84.33
3	Direct Green 6	60.85	80.73
4	Reactive Orange 16	63.17	82.17
5	Effluent	68.62	88.23

The morphological characteristics of the adsorbents were studied using FEI-Quanta 200 scanning electron microscope (SEM).

The SEM image of CSAC in Fig. 1 reveals that small cavities, pores and smooth surfaces on CSAC indicate the presence of interconnected porous network. It has a structure with cracked morphology with long ridges. The SEM image of PPy/SD in Fig. 2 reveals that the surface of PPy/SD consists of smooth, round glassy particles with irregular shape.

2.6. Fourier transform infrared

Infrared spectra of activated carbon (CSAC), sawdust of the fruit material (SD) and polypyrrole polymer composite (PPy/SD) were measured with a Fourier transform infrared (FTIR) spectrophotometer shown in Figs. 3(a) & (b) to elucidate the functional group presenting in CSAC, SD and PPy/SD and the results are given in Table 3.

Table 2
Method of analysis and units of various characteristics of effluents

S.No.	Parameter	Method of estimation
1	pH	Using calibrated pH meter
2	Conductivity, μS/cm	Using calibrated conductivity meter
3	TDS, mg/L	Gravimetric
4	Total hardness, mg/L in $CaCO_3$ equivalent	Volumetric (EDTA titration method)
5	Total alkalinity, mg/L in $CaCO_3$ equivalent	Volumetric (acid neutralisation titration method)
6	Chloride, mg/L	Volumetric
7	Sulphate, mg/L	Gravimetric
8	BOD, mg/L	Volumetric method with $K_2Cr_2O_7$
9	COD, mg/L	Volumetric

When comparing FTIR spectra of CSAC, SD and PPy/SD, some of the peaks in SD have disappeared when sawdust is coated with the polymer polypyrrole. This confirms the formation of PPy/SD over SD. The peak of 2,923.56 cm^{-1} in SD which corresponds to C–H stretching is not found in PPy/SD. Similarly, the peak of 1,419.35 cm^{-1} that corresponds to C–O stretching and the peak of 619.038 cm^{-1} corresponds to out of plane C–H bending mode are not found in PPy/SD. This indicates that the polymer pyrrole has been coated over the sawdust of *Cordia sebestena*. The percentage of dye removal is high in PPy/SD and this is due to the surface modification of the sawdust by the polymer polypyrrole. Hence, the presence of these functional groups may be attributed for the enhancement of the percentage of dye removal in PPy/SD. Similarly, there are no peaks found in SD and PPy/SD in the region of 532.257 and 404.978 cm^{-1} which correspond to inplane aromatic ring deformation vibration in CSAC. This confirms the formation of activated carbon (CSAC).

3. Results and discussion

The characteristics of effluents before and after treatment are given in Table 4. The following observations could be concluded from Table 5 with respect to the physical and chemical properties of the effluent samples after adsorption process using CSAC and PPy/SD prepared from the precursor *Cordia sebestena* fruit.

3.1. Colour and appearance

The colours of all the five effluent samples were bright before treatment. After adsorption process, the offensive odour from the effluents was completely removed and most of the samples were decolourised except sample 3 by CSAC. Even though the sample 3 was not completely decolourised by

Table 3
Peak assignments of functional groups of CSAC, SD and PPy/SD

Peak positions/samples			Possible assignments	References
CSAC	SD	PPy/SD		
3,422.06	3,344.93	3,370	O–H stretching	[24]
2,922.59	2,923.56	–	C–H stretching	[25,28]
–	1,726.94	1,549.52	C=O stretching of carbonyl group	[26,27]
–	1,625.7	–		
–	1,419.35	–	C–O stretching and OH bending of alcohol and carboxylic acids	[23,27]
–	1,319.07	1,309.43		
–	1,265.07	1,168.65		
1,045.23	1,033.66	1,041.37		
–	–	900.694	–CH deformation	–
–	619.038	–	C–C stretching	[29]
532.257	–	–	Out of plane C–H bending mode	[30]
404.978	–	–	Inplane aromatic ring deformation vibration	[31]

Table 4
Characteristics of effluent samples before and after adsorption

S.No	Properties	Sample 1			Sample 2			Sample 3			Sample 4			Sample 5		
		BA	AA		BA	AA		BA	AA		BA	AA		BA	AA	
			CSAC	PPy/SD		CSAC	PPy/SD		CSAC	PPy/SD		CSAC	PPy/SD		CSAC	PPy/SD
1	Colour and Appearance	Dark brown	Nil	Nil	Dark pink	Nil	Nil	Black	Light green	Nil	Wine red	Nil	Nil	Dark blue	Nil	Nil
2	pH	7.08	6.84	6.36	10.49	9.28	6.18	10.44	10.26	7.71	10.57	10.25	7.52	10.17	8.98	6.85
3	Conductivity µS/ cm	7.78	7.62	5.28	22.21	6.03	4.55	69.09	53.63	31.57	74.97	36.32	32.94	11.11	8.15	7.84
4	TDS,mg/L	5,450	3,320	2,980	4,100	2,500	2,100	4,836	3,754	3,157	5,248	3,632	3,294	8,150	7,300	6,800
5	Alkalinity mg/L	1,200	120	90	3,100	500	300	9,000	430	250	8,200	510	230	1,500	130	120
6	Total Hardness mg/L	1,125	320	240	900	400	240	1,200	140	120	1,400	160	120	700	320	200
7	Chloride mg/L	1,700	290	210	650	300	250	1,200	540	240	1,400	310	230	750	323	250
8	Sulphate mg/L	660	280	260	711	180	178	2,496	1,966	1,645	2,676	1,871	1,717	379	296	250
9	BOD, mg/L	480	12	8	270	10	9	950	60	20	240	11	10	390	50	30
10	COD, mg/L	2,080	114	102	1,880	112	98	1,840	109	82	1,610	106	94	1,140	98	43

CSAC, the percentage removal is 76.92%. The reason for poor adsorption may be the nature, concentration of pollutants present in the effluent and also mainly due to the bulkiness of the dye molecule. The percentage of colour removal from dye effluents are presented in Table 5. From Table 5, the decrease in OD value proves that >80% of the colouring matter is removed from the effluents by CSAC and >90% of the colouring matter is removed from the effluents by PPy/SD. The Table 6 depicts the tolerance limits of industrial effluents [32,33].

3.2. pH

The pH values of all the effluent samples were greater than 10 except the sample 1 before treatment. It shows all the effluent samples were basic in nature. An improvement was noticed in the effluent samples after the treatment with both the adsorbents (CSAC and PPy/SD). The pH values of all the effluent samples were reduced to greater extent after treatment with PPy/SD than CSAC. Because, the electrostatic repulsion between the positively charged effluent samples and the surface of adsorbent is lowered and hence pH values decreases and consequently the colour removal efficiency with the adsorbent is increased.

3.3. Conductivity and total dissolved solid

Regarding conductivity of the five effluent samples studied, the prepared adsorbents decreased the conductivity of all samples. Total dissolved solids (TDS) are the important parameter for evaluating the suitability of effluent for irrigation purpose. Since, these solids might clog both the soil pores and components of water distribution system [34,35]. TDS level was high for samples 1, 4 and 5, and the remaining samples 2 and 3 showed good improvement after adsorption treatment.

After removal of the colour and TDS from the effluents, the treated water can be used for house hold or irrigation purposes. Since the TDS value after treatment was found above the tolerance limit. The relationship between TDS and conductivity is a function of the type & nature of the dissolved cations and anions in the water and also by the nature of any suspended materials [36].

3.4. Total alkalinity and total hardness

Most of the effluent samples were alkaline in nature. As far as alkalinity is concerned, the two adsorbents decreased the alkalinity of all five samples studied. It also substantiates the reduction of basicity of effluents. Since the total alkalinity is mainly because of dissolved gases. Hardness is defined as the concentration of multivalent cations in the solution and the hardness varies from sample to sample and place to place [37].

3.5. Chloride and sulphate

Appreciable amount of chlorides and sulphates were removed by CSAC and PPy/SD. The chloride level is within the permissible level for all the samples after treatment for both the adsorbents. The samples 3 and 4 have the sulphate levels are well above the permissible levels. Hence, high chloride contents are harmful for metallic pipes and

Table 5
Percentage of colour removal from dye effluents

S.No.	Sample number	λ_{max} of the effluent (nm)	OD before adsorption	OD after adsorption			
				CSAC	Percentage of colour removal	PPy/SD	Percentage of colour removal
1	S-1	450	0.75	0.14	81.33	0.06	92.00
2	S-2	470	0.92	0.33	82.06	0.15	91.84
3	S-3	510	0.39	0.09	76.92	0.03	92.30
4	S-4	450	0.85	0.16	81.17	0.06	92.94
5	S-5	570	0.58	0.23	80.00	0.09	92.17

Table 6
Tolerance limits of industrial effluents [32,33]

S.No.	Properties	Discharge		
		Inland surface water	Public sewer	On land for irrigation
1	pH	5.5–9	5.5–9	5.5–9
2	TDS, mg/L	2,100	2,100	2,100
3	Chloride, mg/L	1,000	1,000	1,000
4	Sulphate, mg/L	1,000	1,000	1,000
5	BOD, mg/L	30	350	100
6	COD, mg/L	250	–	–

agricultural crops and also kill some microorganisms which are important in some food chains of aquatic life [38].

3.6. BOD and COD

The presence of oxygen in water in dissolved form is necessary to keep it fresh and sparkling. If it is in high level, it makes the water better tasty and at the same time causes corrosion. Biochemical oxygen demand (BOD) is the measure of oxygen required for the biologically oxidisable impurities, but chemical oxygen demand (COD) is the measure of oxygen required for the biologically oxidisable or biologically inert organic matters present in the wastewater. Therefore, COD value is always greater than that of BOD. It was found that both BOD and COD values after treatment was within the tolerance limit. The COD and BOD decreased in accordance with colour removal. An improvement was noticed in the effluent samples after the treatment with respect to pH, COD and BOD.

Thus, among the two adsorbents, CSAC is a potential adsorbent for most of the dye effluents studied and have moderate colour removal capacity for the sample 3. But polymer coated sawdust (PPy/SD) is an excellent adsorbent for the colour removal of almost all the dye effluents studied.

3.7. Cost analysis

The adsorption treatment of five different effluent samples and their comparative cost analysis results using CSAC, PPy/SD and commercial activated charcoal were calculated and given in Table 7. The optimum dose of adsorbent required to treat 100 M^3 of dye house effluent varies from sample to sample depending upon the nature and concentration of pollutants present in the effluent. The collection of the fruit of *Cordia sebestena* is very economical and cheap. Since the fruit of this plant material has no value and use. It is available as waste material in the environment. The only thing is that the cost arises for their collection, drying, grinding and sieving process alone. The cost for the preparation of CSAC is around Rs. 65/kg, PPy/SD is around Rs. 230/kg and commercial charcoal is around Rs. 185/kg. Pyrrole is highly expensive chemical, so the cost of preparation of PPy/SD is not

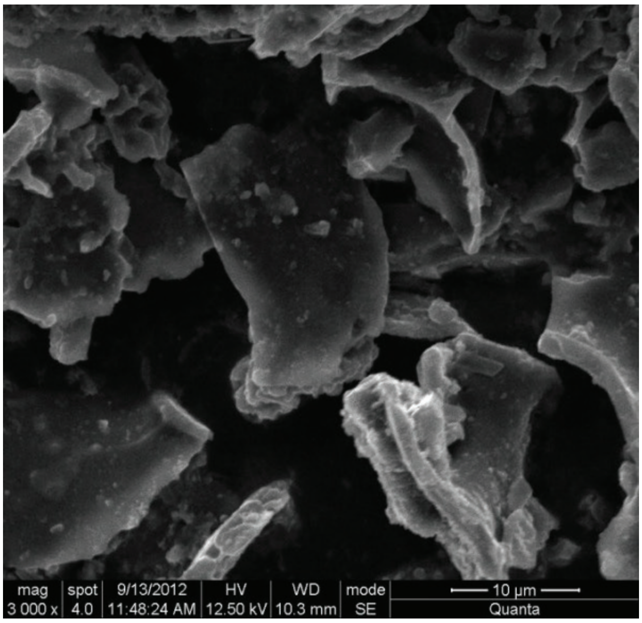

Fig. 1. SEM image of CSAC.

Table 7
Cost analysis for the treatment of 100 M^3 of effluents

Sample number	Amount of adsorbent required (kg)			Cost of treatment (Rs.)		
	CSAC	PPy/SD	Commercial charcoal	CSAC	PPy/SD	Commercial charcoal
S-1	61.7	27.17	50	4,010.50	6,249.10	9,250.00
S-2	121.85	54.44	100	7,920.90	12,521.20	18,500.00
S-3	195.00	97.50	150	12,675.00	22,425.00	27,750.00
S-4	61.59	26.89	50	4,003.35	6,184.70	9,250.00
S-5	62.5	27.12	50	4,062.50	6,237.60	9,250.00

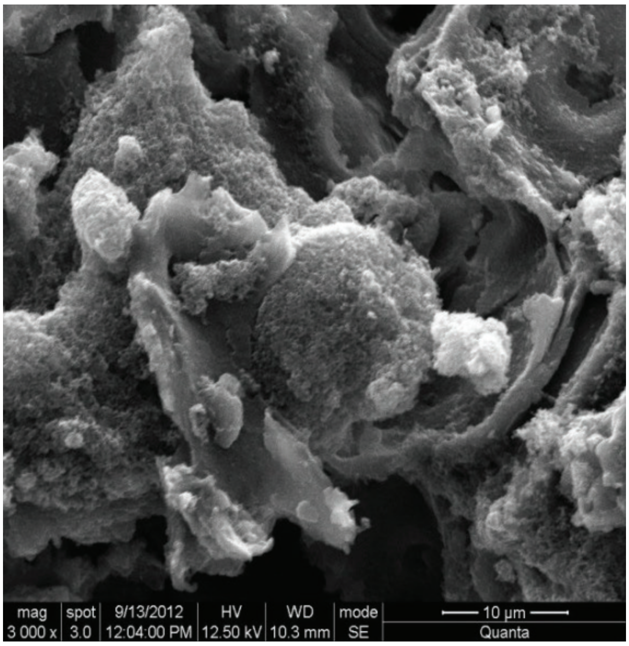

Fig. 2. SEM image of PPy/SD.

economical. But it is an excellent adsorbent and it has high potentiality for the removal of colour from industry effluent as compared with CSAC.

The cost of CSAC and PPy/SD preparation was calculated as follows: for the preparation of 1 kg of CSAC, it requires 2 kg of precursor. The amount of H_3PO_4 required to process 2 kg of precursor is 400 mL. The rate of commercial H_3PO_4 is Rs. 120/L. Therefore, the expense for the preparation of 1 kg of CSAC is Rs. 65 (Rs. 48 for H_3PO_4 and Rs. 17/kg as overhead charges). For the preparation of 1 kg of PPy/SD, it requires 1 kg of precursor. The amount of pyrrole (0.2 M) required to process 1 kg of precursor is 13.4 mL. The amount of $FeCl_3$ (0.5 M) required to process 1 kg of precursor is 81 g. The rate of pyrrole is Rs. 330/25 mL. The rate of $FeCl_3$ is Rs. 200/500 g. Therefore, the expense for the preparation of 1 kg of PPy/SD is Rs. 230 (Rs. 177 for pyrrole, Rs. 32 for $FeCl_3$ and Rs. 21/kg as overhead charges). The rate of commercial charcoal is Rs. 185/kg.

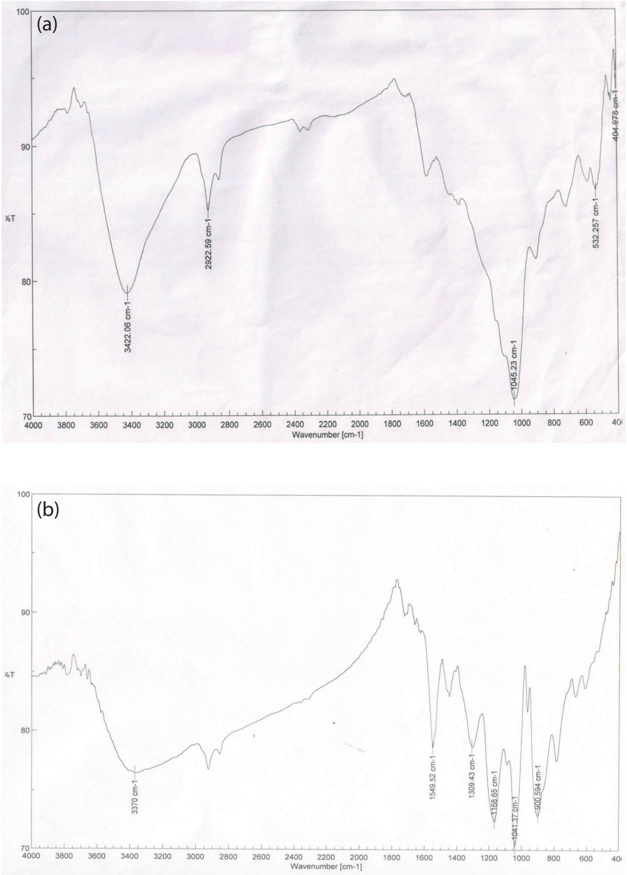

Fig. 3. (a) FTIR spectra of CSAC and (b) FTIR spectra of PPy/SD.

4. Conclusions

The efficiency of adsorbents CSAC and PPy/SD was analysed for the treatment of dyeing industry effluent samples. The colours of all the five effluent samples were bright before treatment. After adsorption process, the offensive odour from the effluents was completely removed and most of the samples were decolourised except sample 3 by CSAC. All other parameters like hardness, alkalinity, chlorides, sulphates, TDS, COD and BOD were also got reduced to a permissible limits (tolerance limits of industrial effluents given by Central Pollution Control Board [CPCB]) with both the adsorbents. Even though the sample 3 was not completely decolourised by CSAC, the percentage removal was 76.92%. Thus, among the two adsorbents, CSAC is a potential adsorbent for most of the dye effluents studied and have moderate colour removal capacity for sample 3. But polymer coated sawdust (PPy/SD) is an excellent adsorbent for the colour removal of almost all the dye effluents studied. The amount of adsorbents required for the treatment and the cost of the adsorbents were compared with the commercial charcoal. Hence from the comparison studies, it could be concluded that even though the price of PPy/SD is higher than others, the amount required for treatment of the effluent samples is very less compared with CSAC and commercial charcoal. Thus, PPy/SD proves that it is an excellent adsorbent and it has high potentiality for the removal of colour from industry effluent as compared with others.

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