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Minimization of organic chemical load in direct dyes effluent using low cost adsorbents

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ABSTRACT

The work is carried out to minimize the organic chemical load (unexhausted dye contents) in direct dyes effluent using low cost adsorbents. The studies are made with different direct dyes, i.e. Direct Red 28, Direct Yellow 12, Direct Orange 26 and Direct Blue 1 with various adsorbents. Three different bio/natural materials have been selected as adsorbents. These includes, Sugarcane bagasse pith (SB), Saw dust (SD)—the plant origin products, and Brick powder (BP)—a silica based material obtained from earth's crust on thermal heating. These substances are almost discarded waste products with the possibility of use as adsorbents. Experimental work for the dye removal from the effluent by activated charcoal (AC) has also been carried out and the results are compared with other adsorbents. The amount of unexhausted organic dye present in the effluent is measured as chemical oxygen demand (COD) before and after the treatment. Adsorbent Sugarcane bagasse pith shows good performance as compared to Saw dust and Brick powder. For understanding the behaviour of adsorbents Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and scanning electron microscopy (SEM) has also been carried out.

1. Introduction

Water of good quality is among one of the basic needs for survival of human beings. At present, in most of the cases natural water bodies are being fed with drained liquor, thus making the water unsafe for utilization, as such. This factor poses a threat to the ecological balance. Common water pollutants [1] are the organic and inorganic substances, suspended solids and pathogenic organisms. Directly or indirectly, these pollutants show adverse impact on common characteristics of water like color, odour, dissolved oxygen, chemical oxygen demand (COD), biological oxygen demand, pH, dissolved solids, etc. Among the various industrial organizations, textile mills are the key consumers of water and, consequently, have been pin pointed as one of the major water polluting industries [2]. Because here, in addition to the common industrial usage, the textile mills also use water in different wet processing operations, viz. desizing, scouring, bleaching, mercerizing, dyeing. These wetting processes also use chemicals of organic and inorganic origins as per the needs. Dyeing is necessary to improve the aesthetic and functional values of the textile material by providing it with fascinating colors. Wastewater from these textile wet processing units [3,4] is enriched with polluting chemicals particularly residual organic dyes.

Here direct dyes [5,6] have been used, which possess good affinity with cellulosic fibers. Low cost, excellent color range, good lightfastness, in addition to ease of application to the material are some of the key features of the direct dyes. Direct dyes constitute a small group of anionic colorants. Chemically, most of these are sulfonated organic azo compounds. These dyes are generally soluble in water, being the sodium salts of sulfonic acids. The effluent released from textile dyeing process house is complex in nature with high alkalinity, electrolyte concentration and deeply colored due to unexhausted dye contents. This effluent is unfit for direct release to a water body or to land surface. In case of direct dyes the effluent have around 5–30% [7] of unexhausted dye along with sizable amount of electrolyte and alkali. Dye effluent provides color to the water, a visible pollutant [8], which will persist in the receiving stream. The color acquired by the water body will inhibit the growth of aquatic plants by reducing the penetration of sunlight, and thus, affecting the normal photosynthetic activity of the plants [9]. Also, due to high absorption capacity of solar energy in the visible range of spectrum, the colored water limits the aquatic biota to a narrow depth, consequently reducing the self-purification capacity of stream. Some of these dyes have been found to possess toxic behaviour [10,11] including carcinogenicity. Appreciable stability of the direct dye molecules and their poor biodegradability is the key reason for long-term toxic effects. Further, in some cases, intermediates or dye degradation products have also been found to possess potential health hazards. Dyeing effluent when discharged to land surface negatively affects the soil fertile

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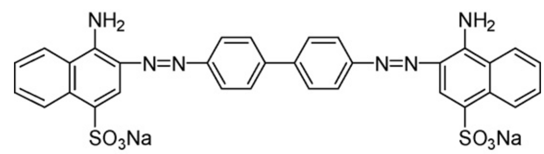
capacity [12], agricultural production [13] and ground water quality [14].

Dye effluent is highly colored due to unfixed organic dyes, alkaline in nature and possesses high chemical oxygen demand values beyond the accepted values [15]. When discharged without adequate treatment, it adversely affects various segments of the environment. Removal of color (organic chemical load) [16] from the effluent is more challenging than any other waste due to complex structural nature of dye molecules and their close affinity with water. The situation becomes more complicated with wider varieties of dye used. Some common methods [17–19] used for minimization of chemical load in the effluent are—Physical Processes, Chemical Processes and Physico-Chemical Processes. Physical processes like equalization, neutralization, sedimentation, filtration, etc., are almost ineffective in the treatment of colored effluent and thereby possess limited scope. Chemical processes are based on oxidation, reduction, etc., and are already in practice for treating textile effluent. The limitations of these methods are the replacement of dye by some other chemical moieties. Various physico-chemical processes [20] like coagulation–flocculation, adsorption, ion exchanges, membrane separation techniques, etc., have been explored for the textile effluent treatment.

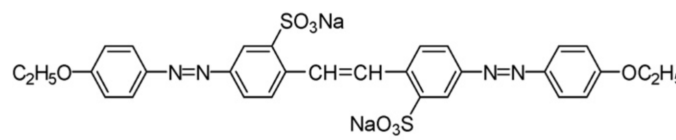
Adsorption [21–23], one of the key physico-chemical processes, finds extensive use in wastewater treatment for the removal of odour, color, disinfectant, etc. It is primarily a surface phenomenon, which includes utilization of surface forces, leading to concentration of materials on the surface of the solid bodies, and has been commonly used as an effective method for lowering the concentration of dissolved organic dyes from the effluent. Common environmental applications of the adsorptive processes are preferably embodied in the carbon adsorption system [24,25]. Particularly, activated carbon adsorption proved its practical utility in the minimization of chemical load of the dyeing effluent. Several researchers [17] like Herger and Relly (1970), Dejohn (1976), Mackey et al. (1980), Gupta et al. (1986), Lin (1993), Venkata Mohan (1997), etc., have also highlighted the activated carbon potential in decolorisation of dyes effluent including direct dyes, over a period of time. However, carbon derived from agriculture waste/byproducts have also been explored by various workers [26,27] for textile dyes effluent.

Though activated carbon is reasonably effective in the decolorisation of the dyeing effluent, needs either regeneration or disposal, once it is fully loaded. Further, high cost of activated carbon coupled with the problems associated with regeneration is the major limiting factor in its frequent use. These factors lead to the development of suitable alternate adsorbents. Many researchers [28–30] focussed their attention towards this and came forward with various adsorbents of different origins like Silica, Coal dust, Brick powder, Wood ash, Tea leaves, Rice husk, Sugarcane bagasse pith, Saw dust, Maize cob, Wheat straw, etc., that can be used for treatment of the textile dye effluent.

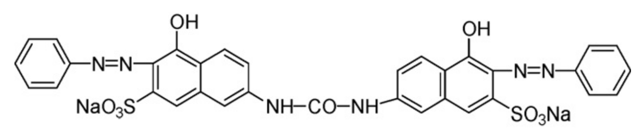
Mostly the above low cost adsorbents are having plant origin or derived from earth's crust. The natural adsorbents prepared from plants have cellulose as their major component, but also possess hemicellulose and lignin in the significant amounts. These compounds show an appreciable effect on the overall adsorption properties of materials. The present work is concentrated at the measurement of unexhausted dye contents present in the effluent, characterization of adsorbents and to minimize the chemical load of the effluent using low cost adsorbents before its discharge to the surroundings. The studies are made and compared with different dyes with different adsorbents. For systemic studies, effluent is generated on laboratory scale by dyeing of cotton fabric with direct dyes. The effluent so obtained was deeply colored in nature due to the presence of residual dyes. The amount of unexhausted organic dye present in the effluent is measured as chemical oxygen demand before and after the adsorbent treatment. Selection of different direct dyes have been made to provide appealing colors like red, yellow, orange and blue, to the fabric. Owing to this, four direct dyes of different color and structures, i.e. Direct Red 28, Direct Yellow 12, Direct Orange 26 and Direct Blue 1 have been chosen for studies. The structures and nomenclature of dye molecules are given below



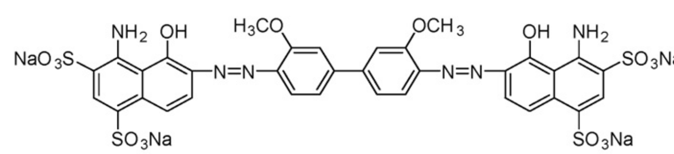
Direct Red 28
C I No. 22120



Direct Yellow 12
C I No. 24895



Direct Orange 26
C I No. 29150



Direct Blue 1
C I No. 24410

Minimization of organic dye of the effluent has been carried out using adsorption method, as this leads to the actual removal of dye contents from effluent and not merely the replacement of dye by any other chemical. For adsorption studies, three different bio/natural materials have been selected. Sugarcane bagasse pith (SB), Saw dust (SD)—the plant origin products with cellulose as main constituent along with hemicellulose, lignin, and with small quantities of pectines, coloring matter, waxes and oils. Cellulose is a known adsorbent material with excellent surface properties.

Brick powder (BP)—a silica based material obtained from earth's crust on thermal heating. Chemically it possesses oxides of silicon and aluminium as main constituents along with calcium aluminosilicate and other mineral oxides.

These substances are low cost discarded waste products with the possibility of use as adsorbents for minimization of chemical load from the effluent. Experimental work for the dye removal from the effluent by activated charcoal (AC) has also been carried out and the results are compared with other adsorbents.

2. Materials and methods

Chemicals/dyes used were either of commercial or laboratory grade. Cotton fabric and raw materials for preparation of adsorbents were arranged from local sources. Experiments were conducted in triplicate and the average values have been reported. Effluent for experimental work was obtained by dyeing of cotton fabric with each of the four different direct dyes, i.e. Direct Red 28, Direct Yellow 12, Direct Orange 26 and Direct Blue 1, respectively, using the common procedure, i.e. exhaustion method. A stock solution (100 ml) of dye was prepared in water by dissolving 1.0 g dye and 0.5 g of sodium carbonate. For 2% shade, 20 ml of this solution was added to the dye bath while stirring, containing 230 ml of water and the temperature was raised to 40 °C. Then 10 g of cotton fabric was added in the dye bath and was dyed for 10 min. The temperature of bath was then raised to 50 °C in 15 min, then 3.0 g of sodium chloride was added in two lots, in a gap of 15 min with continuous stirring. Finally, the temperature of the dye bath was raised to 95 ± 2 °C in 25–30 min. Fabric was kept in the dye bath at this temperature for 45–50 min. After the completion of dyeing, the material was taken out from dye bath and was dried in air. The effluent was finally made up to 250 ml for studies. Concentrations of unexhausted dye contents in each of the effluent were determined (average values given), i.e. Direct Red 28 (6.6%), Direct Yellow 12 (22.5%), Direct Orange 26 (9%) and Direct Blue 1 (21%), using spectrophotometric method.

Method based on the principle of adsorption has been used for the removal of unused dye present in the effluent. The removal of unexhausted dye from the effluent has been calculated in the form of percentage reduction in chemical oxygen demand by comparing the values, before and after particular treatment. For the reference, the values obtained from the effluent samples without any treatment are taken as 100%. Four adsorbents of different origin have been used, three were prepared in the laboratory, while the remaining one, i.e. activated charcoal was obtained from the local market.

2.1. Preparation of adsorbents

Natural discarded materials, i.e. Sugarcane bagasse pith, Saw dust and Brick powder were used for the preparation of three different adsorbents. These adsorbents were crushed; sun dried and sieved to 40–60 mesh (ASTM) size, for studies. Whereas activated charcoal of same particle size was used for comparative study.

Sugarcane bagasse pith/Saw dust (obtained from the wood of plant—Acacia nilotica) were arranged from the local market, dried well in bright sunlight for 5 days. Then chopped into small pieces and finely grounded. Washed with distilled water and left it overnight in 1% formaldehyde solution [31,32] to leach the coloring matter. The treatment with formaldehyde solution was repeated for one more time. Finally, washed thoroughly with distilled water, dried and sieved to the desired size.

Brick powder—brick pieces were mechanically grounded, washed with distilled water, dried and sieved to desired mesh size.

2.2. Experimentation

Studies for minimization of organic chemical load were carried out in batch mode with 50 ml of each type of dye effluent using adsorbent doses, with a gradient of 100 mg till optimum value (optimized dose of adsorbent beyond which COD value of treated effluent is almost same) is obtained for the particular adsorbent under study. Adsorption process was performed at 28 ± 2 °C with a constant agitation of test sample for 1 h by magnetic stirrer. Treated effluent was filtered through Whatman 41 filter paper [33] and the filtrate was used for measuring chemical oxygen demand. The percentage reduction in COD was obtained by comparing the values of treated effluent sample with original values. The standard methods [34] were used to measure chemical oxygen demand in the dye effluent samples before and after a particular treatment.

Table 1
FTIR characterization of adsorbents.

Adsorbents	FTIR characterization
Activated charcoal	2885 cm^{-1} (*w*), aliphatic C—H stretching
Sugarcane bagasse pith	3338 cm^{-1} (*bs*), bonded O—H stretching 2917 cm^{-1} (*m*), aliphatic C—H stretching 1730 cm^{-1} (*m*), C=O stretching 1605 cm^{-1} (*w*), aromatic ring stretching 1506 cm^{-1} (*m*), ... do . . . . 1460 cm^{-1} (*w*), ... do . . . . 1252 cm^{-1} (*m*), C–O stretching
Saw dust	3461 cm^{-1} (*bs*), bonded O–H stretching 2885 cm^{-1} (*m*), aliphatic C—H stretching 1734 cm^{-1} (*m*), C=O stretching 1508 cm^{-1} (*m*), aromatic ring stretching 1456 cm^{-1} (*w*), ... do . . . . 1260 cm^{-1} (*m*), C—O stretching
Brick powder	3608 cm^{-1} (*m*), O—H stretching 1039 cm^{-1} (*s*), Si—O stretching

3. Results and discussion

3.1. Characterization of adsorbents

For better understanding the surface properties, characterizations of all the adsorbents were carried out using Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques.

3.1.1. Fourier transform infrared spectroscopy

Frequencies, at which the sample absorbs the IR radiations, reflects the chemical identification of the sample. FTIR spectra of the adsorbents have been carried out in KBr on Shimadzu 8201 PC spectrophotometer and the values of main peaks are shown in Table 1. Symbols—*b*, *s*, *m* and *w* represents, *broad*, *strong*, *medium* and *weak* nature of peaks, respectively, in FTIR studies.

3.1.2. X-ray diffraction

Here, Philips Powder Diffractometer PW 1710 instrument with Cu Kα radiations is used for understanding the nature (amorphous/crystalline) of adsorbents (Figs. 1–4) and the observations has been summarized in Table 2.

3.1.3. Scanning electron microscopy

In this case, Philips EL-30, ESEM, scanning electron microscope at 1000× under environmental mode, has been used to study

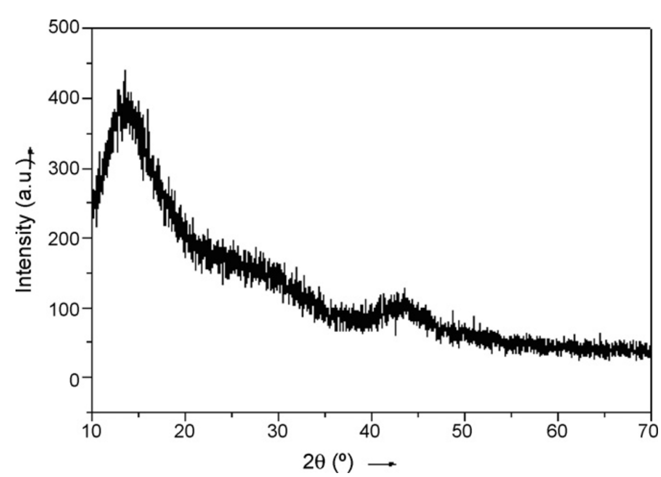


Fig. 1. XRD pattern of activated charcoal.

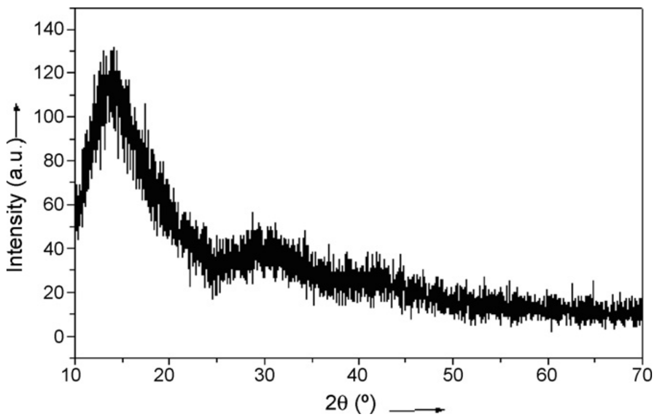


Fig. 2. XRD pattern of Sugarcane bagasse pith.

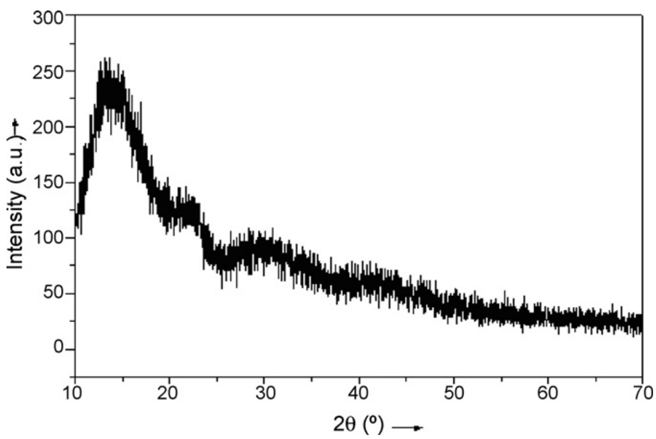


Fig. 3. XRD pattern of Saw dust.

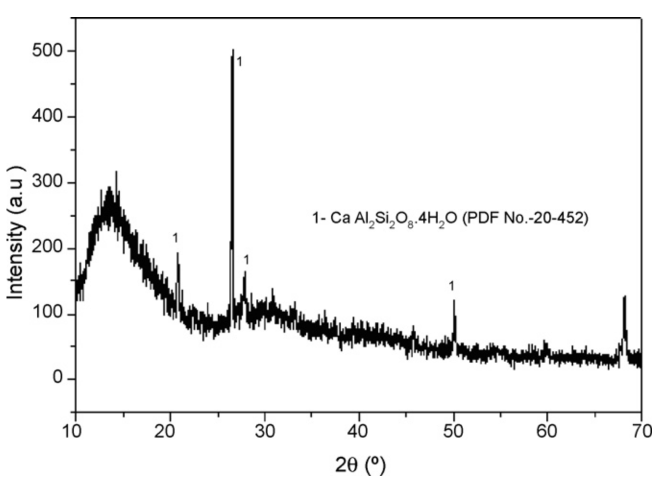


Fig. 4. XRD pattern of Brick powder.

Table 2
XRD observations of adsorbents.

Adsorbents	XRD observations
Activated charcoal	Amorphous
Sugarcane bagasse pith	Amorphous
Saw dust	Amorphous
Brick powder	Heterogeneous in nature containing amorphous and crystalline phases ($CaAl_2Si_2O_8{\cdot}4H_2O$, PDF No. -20-452)

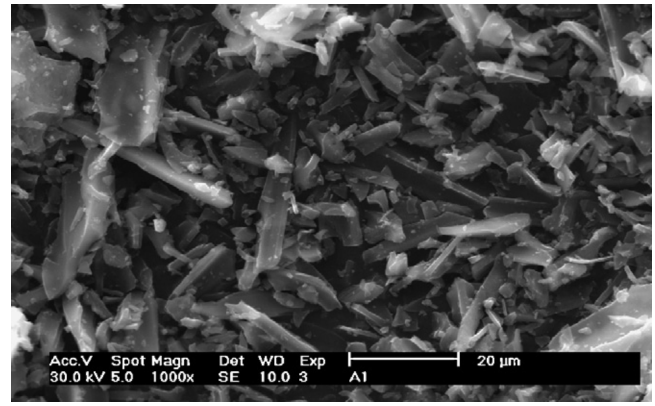


Fig. 5. Scanning electron micrograph of activated charcoal.

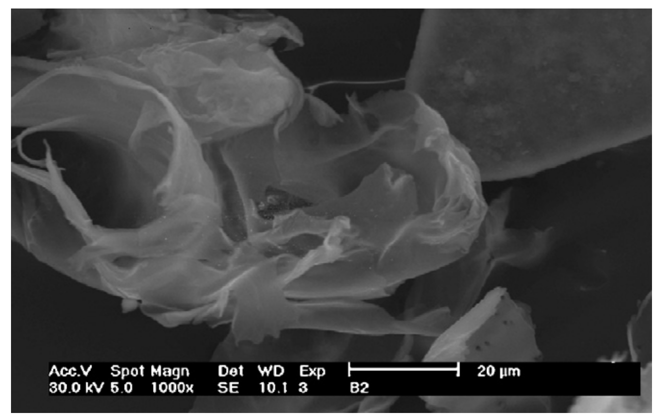


Fig. 6. Scanning electron micrograph of Sugarcane bagasse pith.

surface morphology of the adsorbents. Scanning electronic micrographs of the adsorbents used in the present work have been shown in Figs. 5–8 and the observations are summarized in Table 3.

3.2. Minimization of organic chemical load from dye effluent

Direct dyes effluent has been subjected to adsorbents treatment for the minimization of unexhausted dye contents (organic chemical load). Adsorption of color particles from the solution on to the adsorbent surface involves [35]—transport of dye particles from the bulk solution to the exterior surface of adsorbent, movement of these dye particles across the interface to the external site where adsorption occurs, migration of adsorbed dye molecules into the

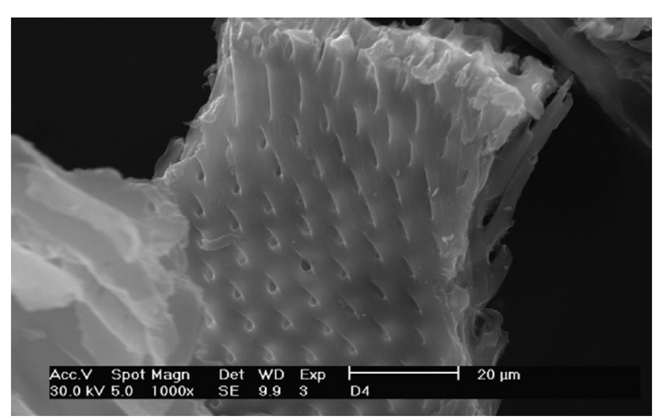


Fig. 7. Scanning electron micrograph of Saw dust.

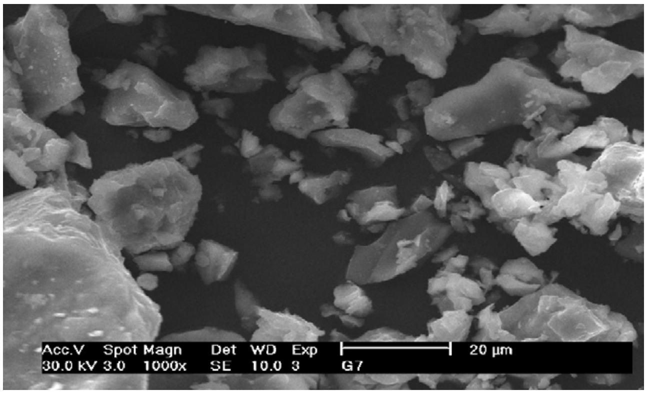


Fig. 8. Scanning electron micrograph of Brick powder.

Table 3
SEM observations of the adsorbent's surface.

Adsorbents	SEM observations
Activated charcoal	Needle like structural features with cave type openings
Sugarcane bagasse pith	Loosely bound fibrous structural features
Saw dust	Regular features of pores, which are evenly distributed
Brick powder	Discrete particles of varying dimensions having smooth and hard surface with sharp edges

pores of adsorbents, interaction of dye molecules with interior surfaces bounding the pores of the adsorbents. One or more of the above steps may be deciding factors in the treatment of dye effluent for adsorption efficiency in a particular adsorbate–adsorbent system.

The experimental work has been carried out with three different adsorbents, i.e. Sugarcane bagasse pith, Saw dust and Brick powder, for the removal of unexhausted dye from the selected dyes effluent. These adsorbents have different physical and chemical nature and are easily available, low cost materials with high adsorption affinities. The results are reported with optimized doses (which shows maximum minimization of organic chemical load) of the above adsorbents and have been compared with activated charcoal, under similar conditions.

Organic pollutants (unexhausted dye contents) of the effluent have been measured in terms of chemical oxygen demand. The COD values of dye effluent, i.e. Direct Red 28, Direct Yellow 12, Direct Orange 26, Direct Blue 1 were also found out (before treatment) and has been shown in Table 4.

Percentage reduction in the COD values of direct dyes effluent samples treated with optimized doses of adsorbents has been shown in Figs. 9–12.

After examining the adsorbents performance (optimized dose as well as percentage reduction in COD values) for the various dyes effluent from Figs. 9 to 12, it is noted that activated charcoal gives the best results for the decrease in COD of dye effluents under study. The best efficiency of activated charcoal is due to its organophillic [33] character and presence of matrix of micro pores in it that yields greater active surface area, enhancing adsorption. Amorphous characteristics of this material as indicated by XRD, also favours its

Table 4
COD in the selected dyes effluent.

Dyes effluent	COD (mg/l)
Direct Red 28	266
Direct Yellow 12	442
Direct Orange 26	202
Direct Blue 1	392

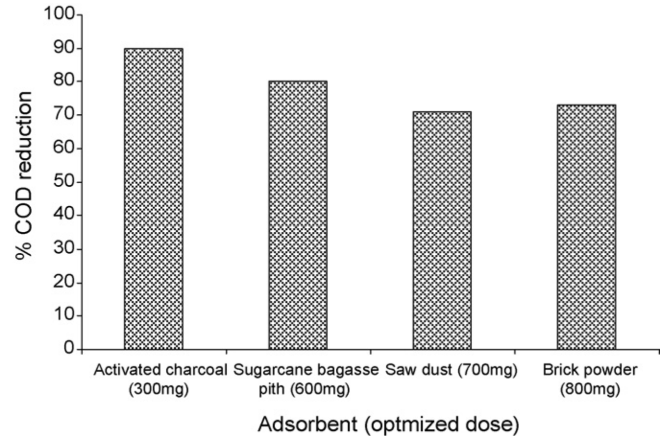


Fig. 9. Reduction in COD of Direct Red 28 effluent with adsorbents.

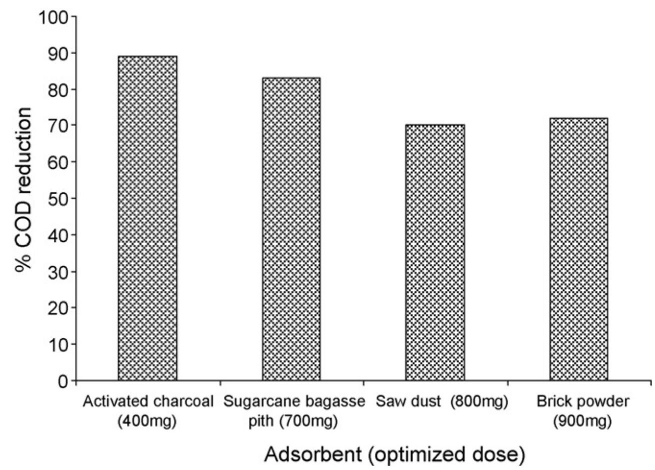


Fig. 10. Reduction in COD of Direct Yellow 12 effluent with adsorbents.

characteristic feature for excellent removal of residual dye contents of the effluent. SEM image of this adsorbent shows caves type openings supporting its performance.

The adsorbent Sugarcane bagasse pith shows good removal of organic dye (COD) from the effluent, whereas Saw dust shows poor performance in most of the cases. Both of these adsorbents possess cellulose, hemicellulose, lignin, waxes, pectines, etc., as their constituents. Out of these cellulose forms main bulk of the above adsorbents. The presence of number of hydroxyl groups in cellulose,

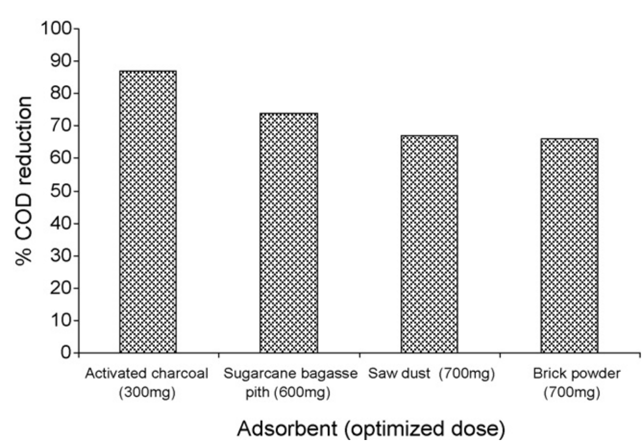


Fig. 11. Reduction in COD of Direct Orange 26 effluent with adsorbents.

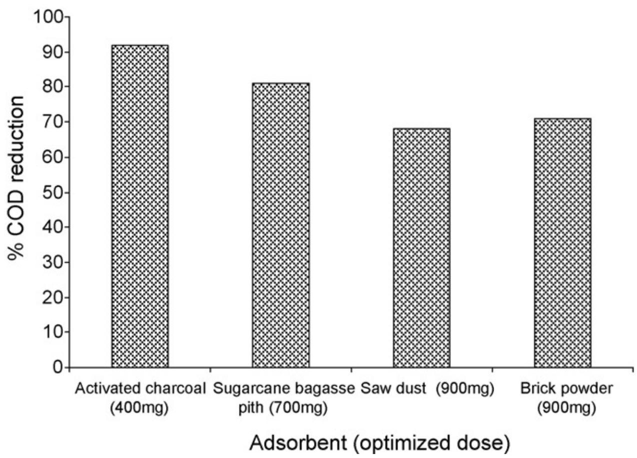


Fig. 12. Reduction in COD of Direct Blue 1 effluent with adsorbents.

hemi cellulose and lignin part of the adsorbents forms hydrogen bonds with the dye molecules, helping in removal of unexhausted dye contents of the effluent and thereby decrease in COD values. A negative charge [36] is partly developed on the surface of these adsorbents in water, due to the presence of cellulose. This negative charge on the adsorbent surface leads to the electrostatic repulsion of the anionic dye molecules in the effluent affecting organic dye uptake and, thus COD value. However, the presence of salts in the effluent reduces [30] the negative charge on the adsorbents surface to some extent.

FTIR studies confirm the presence of polar groups like –OH and –C=O in Sugarcane bagasse pith and Saw dust. These polar groups help in enhancing the binding capacity of adsorbents for the dyes molecules. Also these adsorbents possess good surface area due to their amorphous nature as reflected by XRD. SEM indicates fibrous structure in Sugarcane bagasse pith and pores on surface of Saw dust, which are evenly distributed. These surface morphological features of the adsorbents, provides sites for better adsorption. These adsorbents possess comparatively a poor removal of unexhausted dye contents from the effluent than carbonaceous adsorbents, i.e. activated charcoal, because of the composite effect of the above discussed factors.

Among the two plants origin adsorbents, Saw dust shows the low adsorption efficiency for unexhausted dye contents of the effluent. Firstly, this might be due to incompatibility of size between the dye molecules and the pores of the adsorbent. Secondly, the presence of waxes, pectines and other impurities adds hydrophobic character. Thirdly, waxes and impurities also acquire reasonable surface area of adsorbent causing an overall decrease in the active surface area. All these factors affect the adsorption efficiency of Saw dust and reduces dye uptake, thereby, less COD reduction.

Lastly, the Brick powder is also tried as adsorbent for the reduction of COD in dyes effluent. It possesses silica, alumina, calcium aluminosilicate, etc., as its constituents. Silica forms the major component of this adsorbent. It shows peak for –OH group in the FTIR spectrum, the hydroxyl group helps in the binding of dye molecules to the adsorbent. XRD indicates heterogeneous nature of this material having mixture of amorphous and crystalline phases, showing hydrated calcium aluminosilicate (zeolite) as one of the crystalline phase. The crystalline phase leads to a decrease in the active surface area for adsorption, resulting into poor reduction in COD of dyes effluent by the adsorbent. Further, the negative charge on the aluminosilicate may lead to some electrostatic repulsion between the adsorbent and negatively charged dye molecule present in effluent. SEM image of this material shows smooth and hard surface reflecting poor adsorption efficiency. Collectively, all above factors leads to the overall poor removal of unexhausted dye contents of the effluent by this adsorbent. The silica based adsorbent; Brick powder thereby, shows poor reduction in COD in majority of cases similar to Saw dust.

Above results reflect that adsorption is a complex phenomenon, the extent and rate of adsorption process depends on the physical and chemical nature of the adsorbent and the adsorbate. The surface adsorption of organic dye which is responsible for COD of the effluent depends upon the dye structure, molecular shape and size together with concentration. Diffusion of dye molecules within the adsorbent will also be affected by the pore size on the surface of adsorbent. Results show the decrease in COD values in adsorbents treated samples as compared with as such (before treatment) dye effluent. This is due to the removal of unexhausted organic dye contents from the effluent by the adsorbent treatment.

4. Conclusion

Low cost discarded waste materials (Sugarcane bagasse pith, Saw dust and Brick powder) have been used as adsorbents in the direct dyes effluent on laboratory scale for the minimization of harmful unexhausted organic dye contents, before its discharge to the environment. Behaviour of adsorbents has been correlated with their FTIR, XRD and SEM observations. Results clearly indicate the appreciable decrease in chemical oxygen demand values in adsorbents treated samples.

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