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## Study on the preparation of polymer-grafted coir fibre based adsorbent and its application to remove a reactive dye from aqueous solution

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### Abstract

Holocellulose was separated from Coir fibre by Sodium chlorite bleaching procedure. The adsorbent was synthesized by graft-copolymerization of acrylamide onto separated cellulose using potassium persulfate as a free radical initiator. Grafting percentage and grafting efficiency were measured and found to be 74.5 and 58% respectively. Prepared adsorbent was characterized by applying Fourier Transform-Infrared Spectrophotometer, Thermogravimetric/Differential Thermal Analyzer and Scanning Electron Microscope. Adsorbent prepared from holocellulose was successfully used at different adsorbent dosage, contact time, initial dye concentration and temperature to remove "Reactive Orange-16", a reactive dye from aqueous solution. Ultraviolet-Visible Spectrophotometric measurement of the dye in untreated and treated solutions showed that 0.2 g Holocellulose and Polyacrylamide-grafted holocellulose can remove 42% and 72% of Reactive Orange-16 dye from its 10 ppm aqueous solution within 1 hrs respectively. It may be proposed that Polyacrylamide-grafted holocellulose can be used in a traditional Textile Effluent Treatment Plant (ETP) as a cost effective adsorbent/alternative for decreasing the concentration of the reactive dye in the effluent.

**Keywords:** Coir fibre; Holocellulose; Polyacrylamide grafted holocellulose; Graft copolymerization; Homopolymer; Extraction; Reactive Orange-16; Adsorption

### Introduction

Population explosion, haphazard rapid urbanization, industrial and technological expansion and thus waste generation from domestic and industrial sources have caused severe problem to human and other living resources (Amuda *et al.*, 2006). The textile industry is one of the industries that generate a high volume of waste water. Many textile industries discharge their effluent directly to nearby river or canal. Pollution of surface water due to heavy metals and non-biodegradable reactive dye containing textile effluent is a topic of major concern of the scientists today. Dyes though present in very small amounts e.g., ppm levels are highly detectable and thereby are capable of causing serious problems to surface water and environment. In fact, most of the commercially used dyes are resistant to biodegradation, photo-degradation and even oxidizing agents. Without proper treatment, these dyes can significantly affect photosynthetic activity in aquatic life due to reduced light penetration (Sharma, *et al.*, 2009; Ramakrishna, *et al.*, 2000).

Brightly colored and water soluble reactive dyes are extensively used in the textile dyeing and printing industries.

Those have good fastness properties owing to the bonding that occurs during dyeing. Reactive dyes are most commonly used in dyeing of cellulose like cotton or flax, but wool and nylon are also dyeable with reactive dyes. Reactive dyeing is now the most important method for the coloration of cellulosic fibre and at the same times it produces huge amount of waste water. Waste water containing reactive dyes is one of the most difficult to treat because they tend to pass through conventional treatment systems unaffected. These dyes, usually, have various synthetic origin and they contain complex aromatic molecular structures. These structural properties make them more stable and more difficult to biodegrade (Sung, *et al.*, 2006; Willmott, *et al.*, 1998; McKay, *et al.*, 1979; Poots, *et al.*, 1976).

Scientists recommend that dyes should be removed completely from effluents prior to their final discharge. Due to the low biodegradation of dyes, a conventional biological treatment process is not a very effective one in treating dyes waste water (Reza, *et al.*, 2012; Husseiny, *et al.*, 2008; Moran, *et al.*, 1997). Adsorption, coagulation, flocculation,

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oxidation, precipitation, filtration, electrochemical processes, etc are the common techniques reported for the removal of dyes from

effluents (Mozumder, *et al.*, 2010; Robinson, *et al.*, 2001). Chemical methods of dye removal accumulate sludge that can create disposal problems. Amongst the techniques, adsorption seems to be one of the most effective methods because of simple operation and easy handling. Different adsorbents such as zeolite, perlite, bentonite, kaolite, rice husk, maize cob, coir, bagass pith, waste sludges, etc. have been employed for removal of dyes from effluents (Sharma, *et al.*, 2009; Sung, *et al.*, 2006; Alkan, *et al.*, 2005; Wang, *et al.*, 2004; Gurses, *et al.*, 2004; Espantaleon, *et al.*, 2003).

The chemical modification of cellulose by graft copolymerization with some vinyl monomers (e.g. acrylamide) has generated interest to the scientists. Specially, grafting by acrylic monomers change a number of characteristics of holocellulose significantly. Polyacrylamide based hydrogels which swell and contract in response to external conditions like salt, pH, temperature and electric stimulus have been studied (Mahdavinia GR, *et al.*, 2004; Hennink WE, *et al.*, 2002). Thus, new areas of the application might be opened for the modified cellulose-type materials (Éva Borbély, *et al.*, 2004). Graft copolymerization of acrylamide monomer onto cellulose using ceric ammonium nitrate as initiator was investigated and hydrolyzed polyacrylamide grafted cellulose was used to absorb some harmful heavy metals from aqueous solution (Abd-Alla, *et al.*, 2007). Coconut husk modified through the use of N-(3-chloro-2-hydroxypropyl)-trimethylammonium chloride was evaluated for its ability to remove Reactive Blue 2, Reactive Yellow 2, Reactive Orange 16 and Reactive Blue 4 from aqueous solution. Unmodified Coconut husk showed very little uptake of these reactive dyes but removal efficiency was significantly increased using of its modified form (Low *et al.*, 1998).

Present study is a preliminary attempt for removal of a reactive dye from aqueous solution using polyacrylamide grafted holocellulose. Ultimate aim of the research work is to develop a cost effective Textile Effluent Treatment Plant (ETP) constructed by combining biological (aerobic treatment) and physical (adsorption) processes.

### Materials and methods

Coir fibre was collected from local source (Jessore, Bangladesh). Technical grade Sodium chlorite (Assay-80%,

BDH Ltd, England), Acetic acid (Assay-100%, Merck, Germany) and Sodium acetate (Assay-99.5%, S.D.Fine-chem Ltd, Mumbai) were used as bleaching agents. Reagent grade acrylamide monomer (Assay-98.5%, Merck, Germany) was used in this study without further purification. Potassium persulfate (Assay-98.5%, Merck, Germany) was used as free radical initiator. Reactive Orange-16 (Dye content, >70%; Sigma-Aldrich Co. LLC) was collected from a local textile dyeing factory. The FT-IR spectra of holocellulose and polyacrylamide-grafted holocellulose were taken in the range 400 - 4000 $\text{cm}^{-1}$  on a SHIMADZU FT-IR (Model PRESTIGE 21 Japan). The SEM micrograph was taken by SEM (Hitachi-2600SN, Japan). TG/DTA 6300 (Model-EXSTAR 6000, SII, Seiko Instrument Inc.) was used to evaluate the thermal behaviors of the sample. The analyses were carried out under nitrogen atmosphere at a heating rate of 20°C/min. The temperature difference between an inert (Alumina) reference and the sample was recorded and mass loss was calculated from the TG-Thermograms. Dye concentrations were determined by measuring absorbance at 495 nm before and after treatment, with CINTRA UV-Visible Spectrometer (Serial-V3678).

### Preparation of Adsorbent

The coir fibre was washed several times with distilled water to remove dirt and water soluble extractives and dried at 105  $\pm$  2°C for 6 hrs and then holocellulose was separated by the Sodium chlorite bleaching procedure:

Sodium chlorite (1L 2.5%) solution and dried Coir fibre (20 g) was taken in a three-necked flask. pH of the solution was maintained at 3-4 by adding acetic acid-sodium acetate buffer solution and boiled for 4 hrs. Mixture obtained was filtered and residue (holocellulose) was washed several times with hot water. Then the holocellulose was dried at 105  $\pm$  2°C for 6 hrs. The yield of holocellulose was 32%.

Extracted holocellulose (5 g) was immersed in water (100 mL) containing potassium peroxydisulphate (0.5 g). Acrylamide (Am) (6.5 g) was added to it and the mixture was stirred vigorously at 70°C in a water bath until a solid mass was obtained. The whole sample was extracted with water in a soxhlet for 6 hours in order to remove residual acrylamide and the homopolymer, polyacrylamide (PAm) formed during the grafting reaction, because the water can dissolve acrylamide and polyacrylamide easily. The polyacrylamide grafted holocellulose (PAm-g-holocellulose) obtained was washed with water and dried at 80°C. The con-

control experiment was carried out in the same way as the graft copolymerization but in the absence of holocellulose.

The grafting parameters including the grafting percentage (GP) and grafting efficiency (GE) were determined as follows:

$$GP (\%) = (W_p - W_o) \times 100 / W_o$$

$$GE (\%) = (W_p - W_o) \times 100 / W_m$$

Where  $W_o$ ,  $W_p$  and  $W_m$  were the gravimetrically determined weight of holocellulose, purified polyacrylamide grafted holocellulose and acrylamide monomer respectively.

#### Preparation of Dye solution

Calculated amount of collected "Reactive Orange-16" was dissolved in distilled water to prepare stock solution (100 ppm). Experimental solutions of desired concentrations (2-10ppm) were obtained by successive dilution.

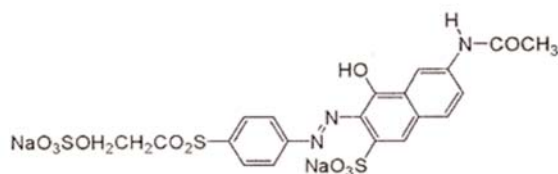


Fig. 1. Structure of Reactive Orange-16

#### Adsorption Experiment

In each adsorption experiment, 10 ml of dye solutions of known concentrations were taken. The mixture, Polyacrylamide grafted holocellulose (PAM-g-holocellulose) and dye solution, was taken at room temperature ( $25 \pm 1^\circ\text{C}$ ) and was stirred on a rotary orbital shaker at 200 rpm. The sample was withdrawn from the shaker at the pre-determined time intervals and adsorbents were separated by

filtration from the solution. This process was carried out with holocellulose also.

The experiment was done by varying the amounts of adsorbents, contact time, initial dye concentration and temperature. The amount of dye adsorbed onto holocellulose and polyacrylamide grafted holocellulose was calculated as follows:

$$\% \text{ Removal} = (\text{Initial absorbance of the dye solution} - \text{After treatment absorbance of the dye solution}) / \text{Initial absorbance of the dye solution} \times 100$$

To ensure the accuracy, reliability and reproducibility of the experimental data, all the batch experiments were performed in triplicate and the mean values were used.

## Result and discussion

### Graft Copolymerization of Acrylamide onto Holocellulose of Coconut Fibre

The remarkable gain in weight of residue, over the control reaction, gives a strong evidence for grafting of acrylamide onto extracted holocellulose. The values of GP and GE of holocellulose were found to be 74.5% and 58% respectively as shown in Table I.

The grafting reaction initiated by potassium peroxydisulfate was characterized by first producing free radicals on the cellulose backbone and then adding acrylamide molecules to the cellulose macro-radical formed ( $\text{Cell}^*$ ). In the presence of acrylamide, cellulose was added to the double bond of acrylamide, resulting in a covalent bond between acrylamide and holocellulose, with reaction of a free radical on the monomer ( $\text{Cell-Am}^*$ ) i.e., a chain was initiated. Thus, subsequent addition of acrylamide molecules to the initiated chain propagates the grafting reaction on the holocellulose

Table I. Graft copolymerization of acrylamide onto holocellulose

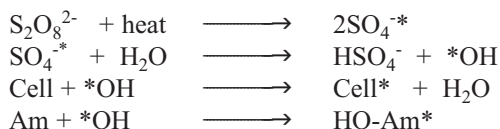
Wt. of holocellulose (g)	Wt. of acrylamide (g)	Wt. of grafted polymer (g)	Wt. of purified PAM grafted- holocellulose (g)	GP (%)	Average GP (%)	GE (%)	Average GE (%)
5.06	6.54	10.21	8.95	76.87		59.48	
5.15	6.48	9.83	8.74	69.70		55.40	
5.11	6.57	9.96	8.82	72.60	74.52	56.46	58.07
5.09	6.55	10.31	9.05	77.79		60.45	
5.05	6.52	9.93	8.87	75.64		58.58	

(Jindal *et al.*, 2010). Most probable mechanism of the grafting polymerization as follows:

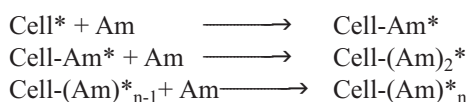
*Ionization of water soluble potassium persulphate*



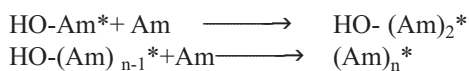
*Initiation*



*Propagation*

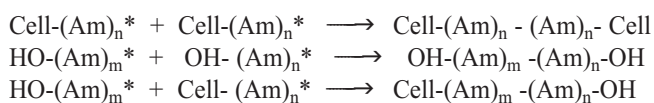


*Main side reaction*



*Termination*

Finally, the termination of the growing grafting chain may occur by coupling or combination or other mechanism. Example of probable coupling reactions is given below:



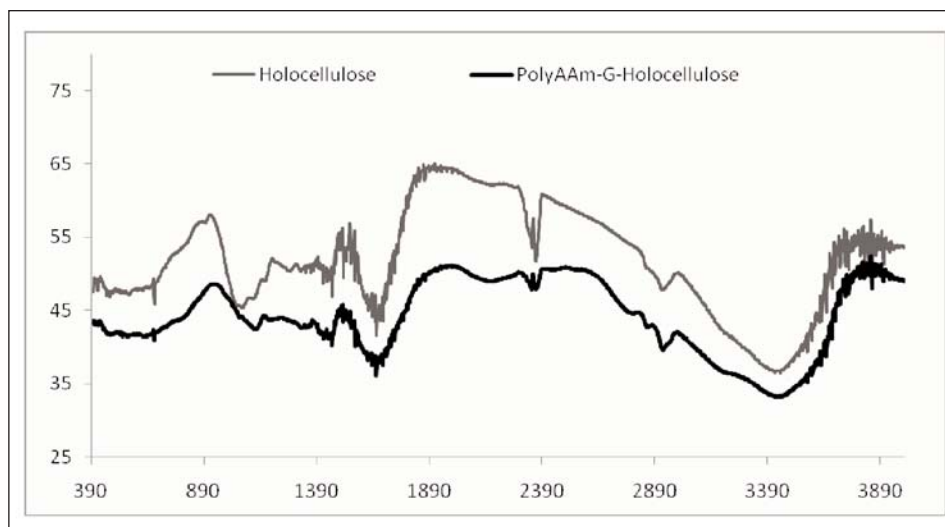
**Fig. 2. Mechanism of Graft copolymerization of acrylamide onto holocellulose**

*Identification of Grafting from Infrared Spectra*

The FT-IR spectra of holocellulose and polyacrylamide grafted holocellulose (PAm-g-holocellulose) were shown in the Fig. 3. Broad and strong absorption of OH bonds of holocellulose at  $3420\text{cm}^{-1}$  was increased and broader in the prepared polyacrylamide grafted holocellulose. This characteristic is due to the incorporation of NH group from amide part of polyacrylamide.  $\text{sp}^3\text{-CH}$  peak of holocellulose at  $2930\text{cm}^{-1}$  was increased and sharper in polyacrylamide grafted holocellulose. This is due to the conversion of  $\text{sp}^2\text{-CH}$  bonds of acrylamide monomer to  $\text{sp}^3\text{-CH}$  bonds of polyacrylamide grafted holocellulose. Usually, hydrolyzed cellulose shows an absorption peak near  $1700\text{-}1750\text{ cm}^{-1}$ . It could be assigned to the absorption of C=O group formed due to chemical treatment of Coir fiber. The absorption of C=O group was relatively increased and broader in case of polyacrylamide grafted holocellulose.

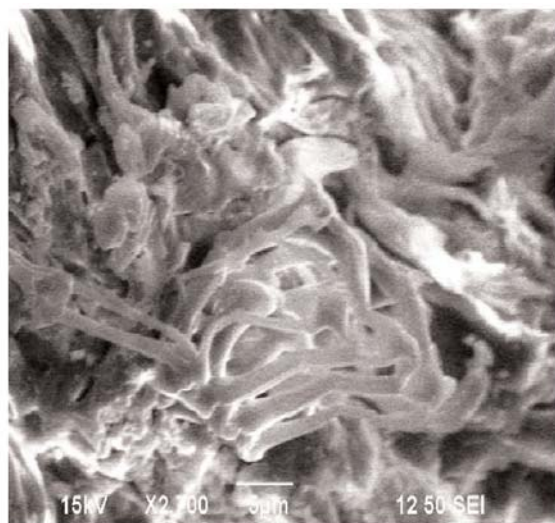
*Scanning Electron Micrograph*

The SEM images (represented in Figs. 4 and 5) were obtained to characterize the microstructure of the holocellulose and polyacrylamide grafted holocellulose. The SEM image of holocellulose showed unit cells that have fibrous structure containing intercellular voids. The SEM micrograph of polyacrylamide grafted holocellulose showed a different orientation of fibres on the surface, which resulted due to the grafting of acrylamide onto holocellulose fibres. Grafted cellulose showed morphology of rough surface textures which provide new adsorption sites to participate in binding of dye molecules.

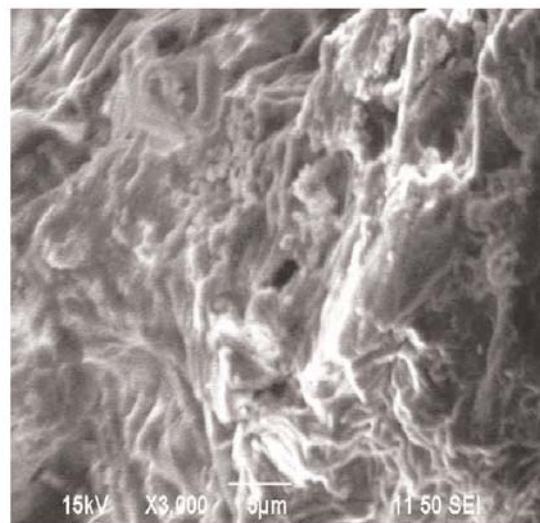


**Fig. 3. FT-IR spectra of holocellulose (up) and Polyacrylamide grafted holocellulose (bellow)**





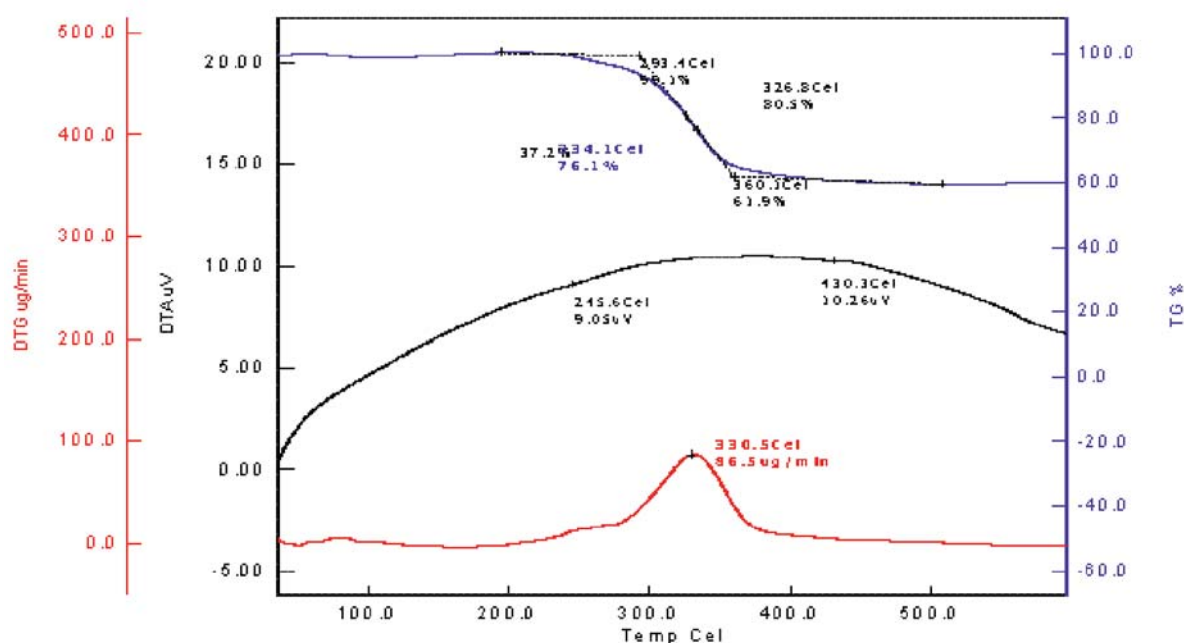
**Fig. 4. SEM Image of holocellulose**



**Fig. 5. SEM Image of Pam-g-holocellulose**

Fig. 6 and 7 showed the TG, DTA and DTG curves of holocellulose and polyacrylamide grafted holocellulose respectively. In Fig. 6, the first stage continuing up to about 120°C, was related to the loss of the absorbed and bound water. The mass loss of this stage was about 0.9%. Following a thermal stability zone between 120-200°C, the second stage was probably associated with structural degradation on the main chain. In this stage, a more significant weight loss (76.1%) occurs above 200°C. It was found that a major degradation point of cellulose at 330.5°C, where maximum

degradation occurred. DTA curve of holocellulose showed the exothermic peak at 245.6°C. DTG curve of holocellulose depict one predominant peak at 330.5°C where the maximum degradation rate was 86.5µg/min. In Fig. 8 the TG curve also showed the two stage degradation. The first stage, continuing up to about 120°C, was related to the loss of the absorbed and bound water with other volatile impurities. The mass loss of this stage was about 4.7%. Following a thermal stability zone between 120-200°C, the second stage was probably associated with structural degradation on



**Fig. 6. TG/DTA thermogram of holocellulose**

the main chain. In this stage, a more significant weight loss (64.4%) occurred above 200°C. A major degradation point of polyacrylamide grafted holocellulose was found at 370°C, where maximum degradation occurred. DTA curve of grafted holocellulose show endothermic peaks at 300°C and 392.8°C. DTG curve of polyacrylamide grafted holocellulose gave peak at 370°C where the maximum degradation rate was 0.193mg/min.

In comparison of Figs. 6 and 7, the TG, DTA, DTG curves of holocellulose and polyacrylamide grafted holocellulose (PAM-g-holocellulose) showed that the degradation occurred due to thermal decomposition by breaking the polymer linkages. A minor degradation occurred for the presence of low boiling volatile components and moisture. From these TG-thermograms, it was clearly revealed that melting temperature of polyacrylamide grafted holocellulose increased significantly compared to holocellulose (330.5°C to 370°C). DTA also confirmed the stability of polyacrylamide grafted holocellulose than raw holocellulose. From the analyses of both thermograms, it was clear that polyacrylamide grafted holocellulose was more thermally stable than holocellulose.

#### Adsorption Measurement at different conditions: Batch Method

##### The Effect of absorbance dosage

It was found that the percentage of dye removal was increased with the increment of adsorbent dosage (Fig. 8).

Particle sizes of the adsorbents were 50-60 mesh. In case of holocellulose, the percentage of dye removal was increased from 36.8% to 43.8%, but in case of polyacrylamide grafted holocellulose the percentage increased from 66.6% to 77.4%, as the adsorbent dosage increased from 0.2 to 1.0 g. This was due to the increased in adsorbent dosage attributed to increase in surface area and thus availability of adsorption site.

##### Effect of Contact time

Experiments were performed to determine the time required for the dye removal process. The effect of contact time can be summarized from in Fig. 9. The curve showed two distinct phases in both cases (holocellulose and PAM-g-holocellulose). The first phase involved rapid dye uptake in the beginning and 78.5% (by PAM-g-holocellulose) and 50.6% (by holocellulose) adsorption was completed within one hour, followed by the subsequent removal of the dye, which continued for a longer period and attain saturation. The first phase may be attributed to the chemisorptions type of interaction where as second phase indicates the utilization of all the active sites over the adsorbent surface and attainment of the saturation phase.

##### Effect of Initial dye concentration

For performing this experiment, 0.2 g adsorbents of grafted holocellulose and holocellulose were treated with 20 ml of

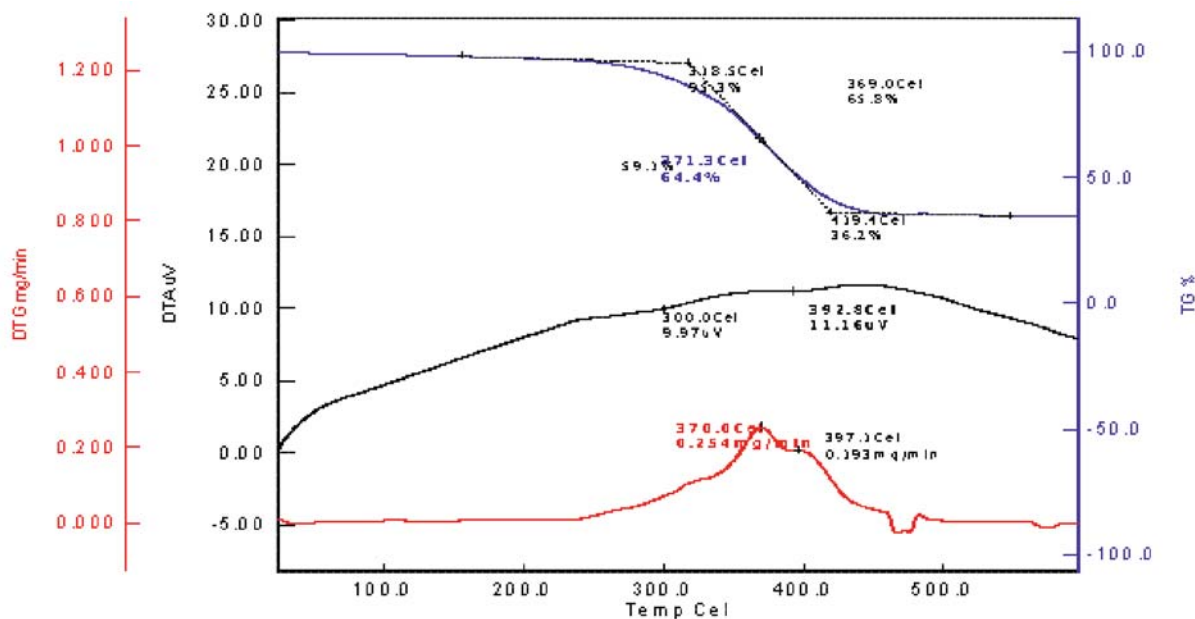


Fig. 7. TG/DTA thermogram of polyacrylamide grafted holocellulose

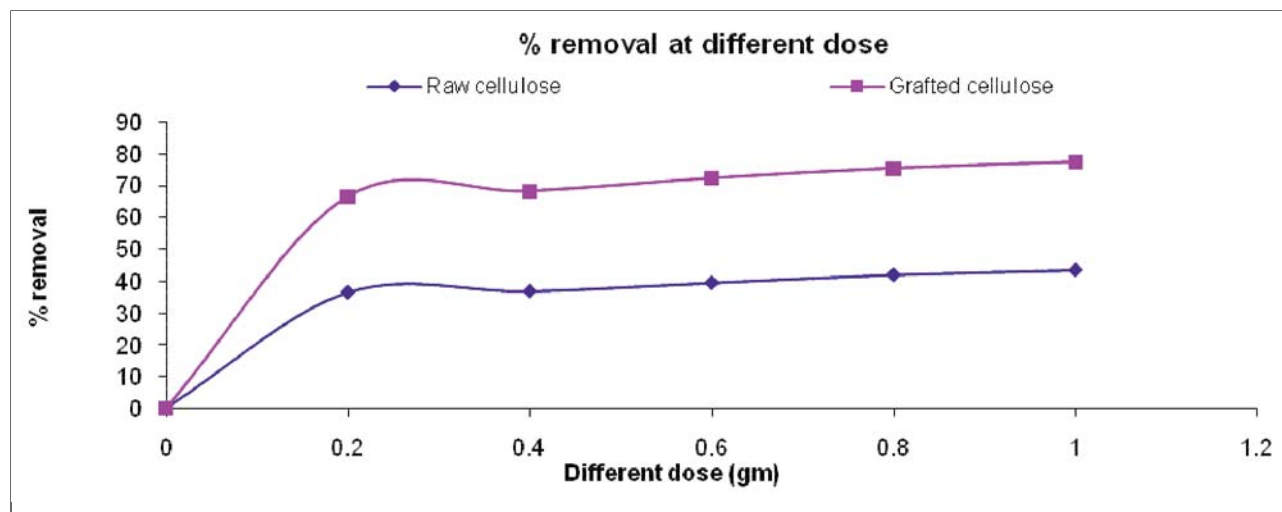


Fig. 8. Effect of absorbent dosage of holocellulose and Pam-g-holocellulose

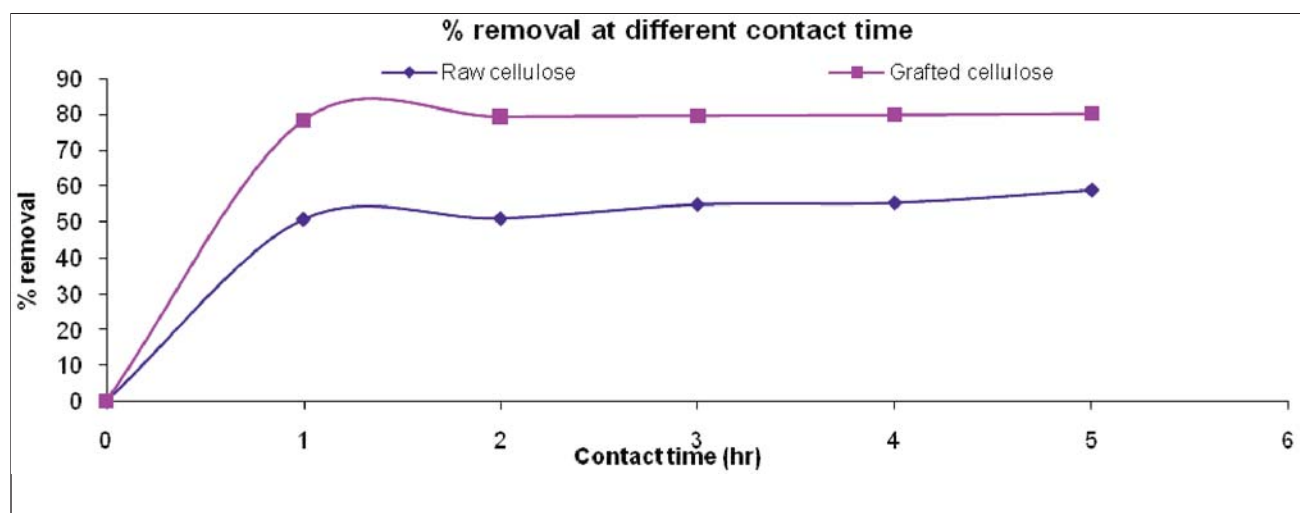


Fig. 9. Effect of contact time of holocellulose and PAM-g-holocellulose

reactive dye solutions of concentrations 2-10 ppm for 1 hour accompanied by mild shaking at room temperature. The result obtained is summarized in Fig. 10.

Although the percent removal of dye slightly decreased but the reactive dye adsorption capacities of grafted holocellulose and raw holocellulose were increased with increasing of the initial reactive dye solutions. Initial concentration provides an important driving force to overcome all mass transfer resistance of the reactive dye between the aqueous and solid phases. Hence a higher initial concentration of dye will enhance the adsorption process using the same amount of adsorbent.

#### *Effect of Temperature*

The data of dye adsorption onto raw holocellulose and PAM-g-holocellulose at different temperature indicates a minor change in the dye removal efficiency. Percent removal of dye for the two adsorbents (raw holocellulose and PAM-g-holocellulose) were investigated and found that results favored with increased temperature (Fig. 11). By increasing the experimental temperature from 25 to 65°C, the dye removal efficiency of PAM-g-holocellulose increased from 78 to 84%, while for holocellulose it was increased from 45 to 53%. So, it was clear that adsorption equilibrium was slightly a thermo-dependent process.

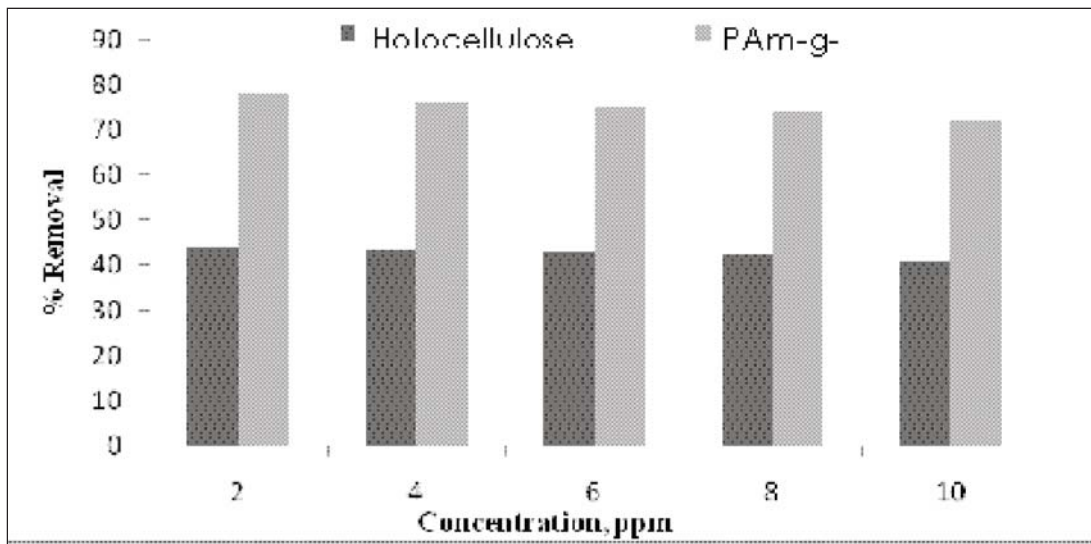


Fig. 10. Effect of initial dye concentration of holocellulose and PAm-g-holocellulose

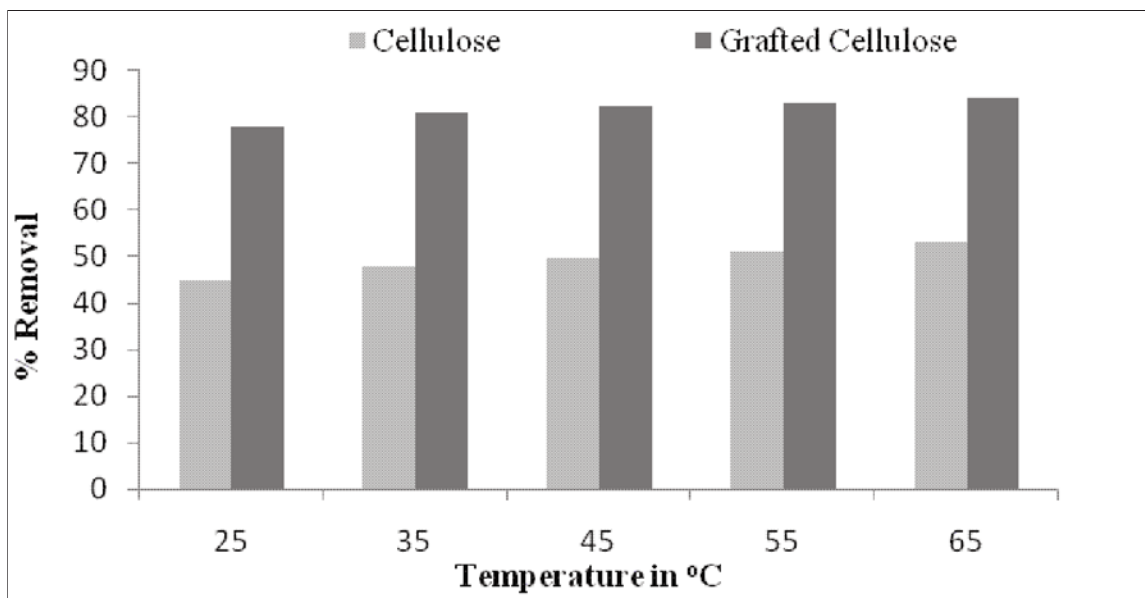


Fig. 11. Effect of temperature on the adsorption by holocellulose and PAm-g-holocellulose

The temperature had two major effects on the adsorption process. Increasing the temperature was known to increase the rate of diffusion of the adsorbate molecules across the external boundary layer and in the internal pores of the adsorbent particle, owing to the decrease in the viscosity of the solution.

### Conclusion

Holocellulose materials were separated (32%) from coir fibre by sodium chlorite bleaching procedure for 4 hrs.

Grafting percentage (GP) and Grafting efficiency (GE) of acrylamide onto separated holocellulose were found to be 74.5% and 58% respectively. Grafting reaction was found to be effective at 70°C for 3-4 hours and grafting reaction was confirmed by the analyses of FT-IR spectrum, SEM images and TG/DTA thermograms. Polyacrylamide grafted holocellulose showed a good performance on adsorption of Reactive Orange-16 than holocellulose. Increasing the temperature from 25 to 65°C, adsorption of the dye slightly increased in



both cases (from 45 to 53% for holocellulose and 78 to 84% for PAm-g-holocellulose). Maximum rate of adsorption occurred at first one hour contact time and then slightly increased and reached at equilibrium after 1.5 hrs. The effect of initial dye concentration in the adsorption process showed that 0.2 g holocellulose and polyacrylamide grafted holocellulose can remove 42% and 72 % of Reactive Orange-16 dye from its 10 ppm aqueous solution within 1 hrs respectively.

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