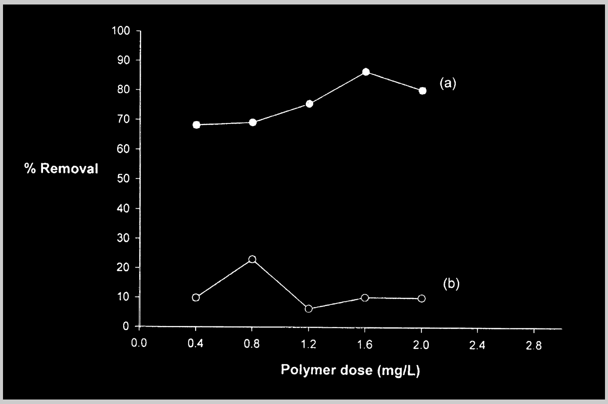
**Full Paper:** The ability of a low cost flocculating material, *Plantago psyllium* mucilage, for removal of solid waste from textile effluent has been successfully investigated. Standard jar test method has been used in this study. The effects of polymer dose, contact time and pH on the %-removal of solid waste are reported. An increase in polymer dose up to a certain level increases solid waste removal. The maximum suspended solid removal, ≈90%, was seen after 5 h at pH 4.0, while the maximum removal of dissolved solids, ≈68%, takes 3 h at pH 7.0. The optimal dosage was found to be 1.6 mg/L. X-ray diffractograms of pure polymer and solid waste from effluent before and after treatment were obtained to suggest the interaction of the waste material with the mucilage.




Effect of varying Psy dosage on percent removal of suspended (●) and dissolved solids (○), temperature = 26 ± 2 °C.

# Flocculation of Textile Wastewater by *Plantago psyllium* Mucilage

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## Introduction

The textile industry is not a single entity but encompasses a range of industrial units, which use a wide variety of natural and synthetic fibers to produce various fabrics. In India, cotton has retained its dominant position not only because of its easy domestic availability but also due to the climatic conditions in the country, which dictate the need for cotton-based wear. Textile processing plants utilize a wide variety of chemicals such as acids, bases, salts, detergents, wetting agents, sizes oxidants, mercerizing, dyes and finishing chemicals. Many of these are not retained in the final product and are discharged in the effluent. The best practicable control technologies for effluent treatment currently available for India are identified as primary treatment and as primary and secondary treatment. Primary treatment comprises screening, oil and grease removal, equalization, coagulant dosing and clariflocculation. Primary and secondary treatment follow either an extended aeration system or an extended aeration and aerated lagoon system.

Organic polymeric flocculants have been used in water purification for several decades as coagulant aids or floc builders after addition of inorganic coagulants like alum, iron salts and lime. Polymers often have been used in the chemically assisted sedimentation of sewage solids to enhance the removal of suspended matter. The concept is applicable as well to the primary coagulation in industrial wastewaters where the separation may be based on flotation, as in examples from the leather, steel, wool scouring, cosmetic, detergent, plastics, dye house, paper, food processing and brewing industries.[1] Recently natural polymer based flocculants have started gaining importance for their harmless, more efficient and environment-friendly nature. Natural polymers such as starch and its chemically modified forms,[2–5] sodium alginate, amylopectin, guargum, xanthan gum,[6,7] seaweed kelp,[8] sargassam,[9] kendu mucilage[10] and chitosan.[11–13] We have recently reported the use of some food grade polysaccharides like okra,[14,15] sodium alginate,[16] and *Plantago psyllium* mucilage grafted with polyacrylamide[17] for the treatment of domestic and industrial wastewater.

*Plantago psyllium* mucilage (Psy) is known as Isabgol husk in India and widely used as laxative. It is easily available and its use is very economical. In the present

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study, we report the use of this natural polysaccharide as a flocculant for the very first time for treating textile effluent. The effectiveness of Psy as a flocculant, on the removal of solid waste, was examined at different pH values and at different duration of treatment using its various dosages. X-ray analysis of the waste before and after treatment was used to suggest the flocculation mechanism.

## Experimental Part

*Plantago psyllium* mucilage was obtained from its husk (Sidhpur Sat-Isabgol Factory, Gujrat, India) and was used after purification. It was purified by precipitation from aqueous solution with alcohol and finally washed with acetone. The FTIR spectrum of purified *Plantago psyllium* mucilage was recorded on a Bruker Vector-22 spectrophotometer. The viscosity of the polymer solution was measured by the method given in our previous publications.[14, 15]

Textile wastewater samples were obtained from Saroj Textile Mills, Panki Industrial Area, Kanpur, India. The samples were collected from the main stream of the wastewater coming out from the processing unit at fixed time on three consecutive days. The samples were characterized within twenty-four hours and were stored under refrigeration. Flocculation studies were conducted within the next twenty-four hours. The pH of the wastewater samples and polymer solution was measured by a Microprocessor pH-meter CP 931. The buffer solutions, prepared by using ready-made buffer tablets (E-Merck chemicals), were used for maintaining the pH of the wastewater samples. The conductivity of the wastewater samples was measured by a Century Microprocessor conductivity meter CC 631 and COD was measured by usual standard methods.

Flocculation studies were conducted by a standard jar test as described in the literature.[18, 19] Beakers of 1 000 mL capacity each equipped with a variable speed (0–100 rpm) agitator were used. In each beaker 500 mL of wastewater was taken and the requisite amount of polymer was added into it by means of a syringe. The agitator was first adjusted to 100 rpm for 1 min and then continued for the total period of 10 min at 50 rpm. The agitator was subsequently stopped and the wastewater was allowed to settle for 1 h. A measured volume of 20 mL of sample was taken to determine the solid content of the effluent before and after treatment with the polysaccharide. The solid contents were calculated by the equation given in ref.[20] To determine the total dissolved solids, known volume of filtered samples were evaporated and solids thus obtained were dried and weighed. For total solids, unfiltered samples were taken. Suspended solids in wastewater were determined by subtracting the total dissolved solids from the total solids.

Flocculation studies were carried out at three pH values 4.0, 7.0 and 9.2. A large amount of buffer (450 mL buffer in 50 mL wastewater) was used to control the pH. X-ray diffraction patterns of powder samples of grafted copolymer, solid waste and flocs were obtained at ambient conditions on an Iso-Debyflux-2002 X-ray diffractometer (Rich and Seifert) with a Cu $K_a$ radiation source.

## Results and Discussion

### *Characterization*

The FTIR spectrum of the prepared *Plantago psyllium* mucilage gives characteristics peaks of –OH between 3 609 and 3 288 $cm^{-1}$, —COOH between 1 635 and 1 617 $cm^{-1}$ and ether linkage at 1 419 $cm^{-1}$. The intrinsic viscosity of the polymer was found to be 1.29 dL/g. The pH values, conductivity values and COD of the wastewater samples ranged between 4.0 and 4.2, between 5.6 and 6.0 mS and between 815 and 910 mg/L, respectively. Total solid contents and suspended solids ranged between 5 590 and 5 625 mg/L and 95 and 120 mg/L, respectively, whereas the turbidity ranged between 18.0 and 18.28 NTU. The pH values of 100 mL of aqueous solutions having different concentrations of Psy were almost neutral (between 7.62 and 7.84). The pH of the textile effluent after addition of different doses of Psy was found to be varying between 5 and 6.

### *Flocculation Studies*

#### Determination of Optimum Dosage of Psy

The effect of Psy dosage on solid removal from textile effluent is presented in Figure 1. It shows the plots of %-removal of (a) suspended solids (SS) and (b) dissolved solids (TDS) vs. Psy dosage. It is apparent from the plots that a Psy dosage of 0.8 and 1.6 mg/L produced maximum%-removal of TDS and SS, respectively. With increase in Psy dosage from 0.4 mg/L to the optimal, %-removal of SS and TDS also increased but a further increase in dosage caused a decreasing trend in solid removal. This behavior could be explained by the fact that the optimal dose of flocculant in suspension causes a larger amount of suspended solid to aggregate and settle. However, Chan and Chiang[21] suggest that an over-optimal amount of flocculant in suspension would cause the aggregated particle to redisperse in the suspension and would also reduce particle settling.

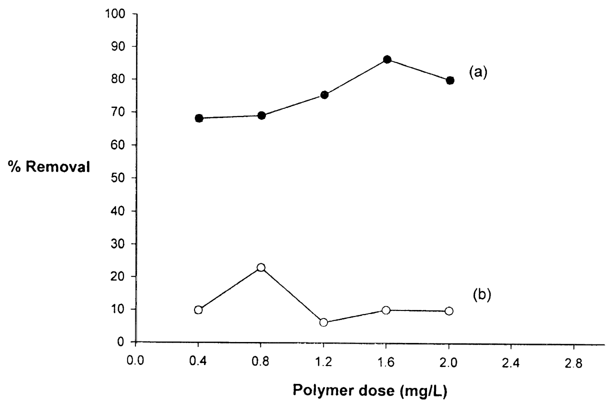


Figure 1. Effect of varying Psy dosage on percent removal of suspended (●) and dissolved solids (○), temperature = 26 ± 2 °C.

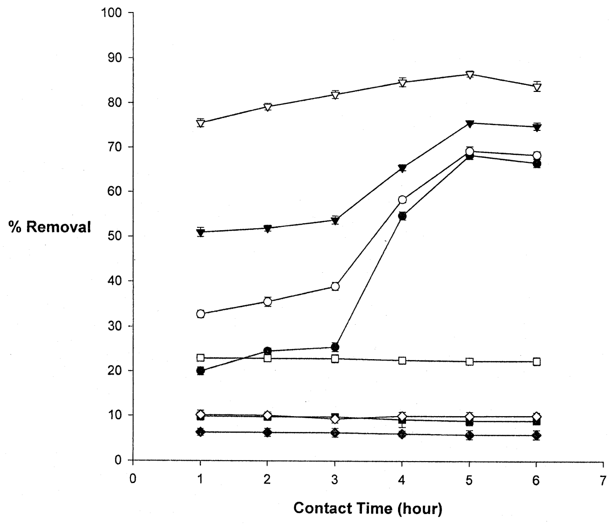


Figure 2. Percent removal of suspended solids versus contact time, Psy dosage = (●) 0.4 mg/L, (○) 0.8 mg/L, (▼) 1.2 mg/L, (▽) 1.6 mg/L. Percent removal of total dissolved solids versus contact time, Psy dosage = (■) 0.4 mg/L, (■) 0.8 g/L, (◆) 1.2 mg/L, (◇) 1.6 g/L.

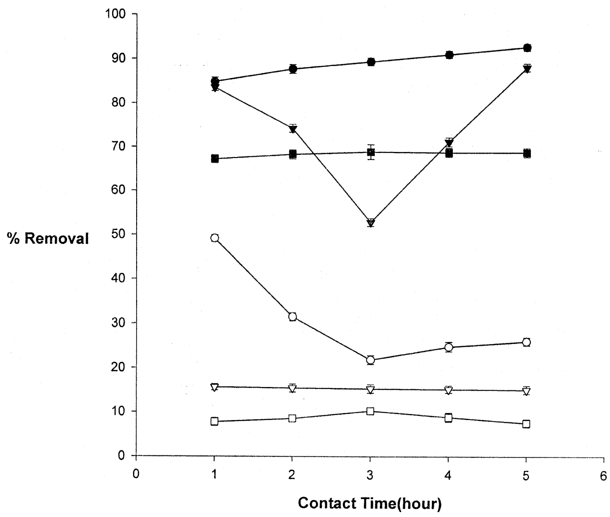


Figure 3. Percent removal of suspended solids versus contact time with varying pH. Psy dosage = 1.6 mg/L, pH = (●) 4.0, (○) 7.0, (▼) 9.2. Percent removal of total dissolved solids versus contact time with varying pH. Psy dosage = 1.6 mg/L, pH = (▽) 4.0, (■) 7.0, (□) 9.2.

## Effect of Contact Time

The flocculation efficiency of the mucilage with varying contact time is shown in Figure 2. It shows the plots between %-removal of the solids (suspended and dissolved) and contact time using different polymer doses. The maximum dissolved (22.98%) and suspended (86.36%) solid removal occurred after 1 and 5 h, respectively, using the optimal dosage. In the absence of Psy, the%-removal of SS and TDS was found to be less than 1% even after 24 h.

The maximum solid removal occurred only after a particular duration, i.e., the optimal treatment time. After this duration, a reverse trend was shown. The suspended solids removal always improves with time but the results obtained in the present study did not follow this statement. Therefore, the most plausible reasons for the reverse trend may be due to (i) the destabilization of the aggregated particles after a long duration,[14,15] (ii) the change in surface chemistry of the edge face with time that changes the adsorption of polymer with time,[22] (iii) the degradation of Psy in solution with time. Out of these three reasons, only (i) and (ii) seem to play a role. No degradation was seen in the flocculant even after six hours, so reason (iii) was ruled out. Biodegradation of the flocculant was measured by measuring its viscosity at different time intervals. There was no change observed in the viscosity of flocculant solution with time.

## Effect of pH

The effect of pH on the flocculation capacity of mucilage was found by conducting the experiments at three different pH values, 4.0, 7.0 and 9.2. Figure 3 shows the plots between%-removal of solids (suspended and dissolved) and varying contact time at different pH values using the flocculant concentration of 1.6 mg/L. From the plots it is seen that Psy produced 92.40%-removal of suspended solid at pH 4.0, 87.84% at pH 9.2 and 25.88% at pH 7.0, and the time required was 5 h. In case of dissolved solids, the maximum removal was 15.22, 68.76 and 10.22% at pH 4.0, 7.0 and 9.2, respectively, and the time taken was 3 h. The maximum efficiency for SS removal was observed at acidic pH and for TDS removal neutral pH was suitable. Thus, for achieving best flocculation results, the pH of the treating solution should be initially acidic to remove SS and then it should be kept neutral to remove the maximum % TDS.

The maximum removal of SS and TDS at two different pH values suggests that the wastewater processing would require a 2-step procedure. In the present case, the pH of the wastewater after addition of Psy ranged between 5.0 and 6.0. By a slight lowering in the pH value, the maximum SS removal may be achieved at step 1 and then in the second step, by an increase in pH value up to neutral, the maximum TDS removal may be achieved.

Usually a change in pH does not affect the efficiency of natural polymers.[6] Here changes in %-removal of solids with varying pH values may be due to its effect on the constituents of the textile wastewater.[23] Metals like Mn, Ni, Pb, Fe, Al, Zn, Cu and Cd are usually present in the textile effluent. In the present study, the wastewater seems to have Pb, Ni, Mn and Cd as confirmed by XRD. It may be suggested that lowering of pH results in oxidation of these metals, thus aggregating these particles.[24] The increase in total dissolved solids (TDS) removal, at neutral pH, may be due to the presence of such sub-

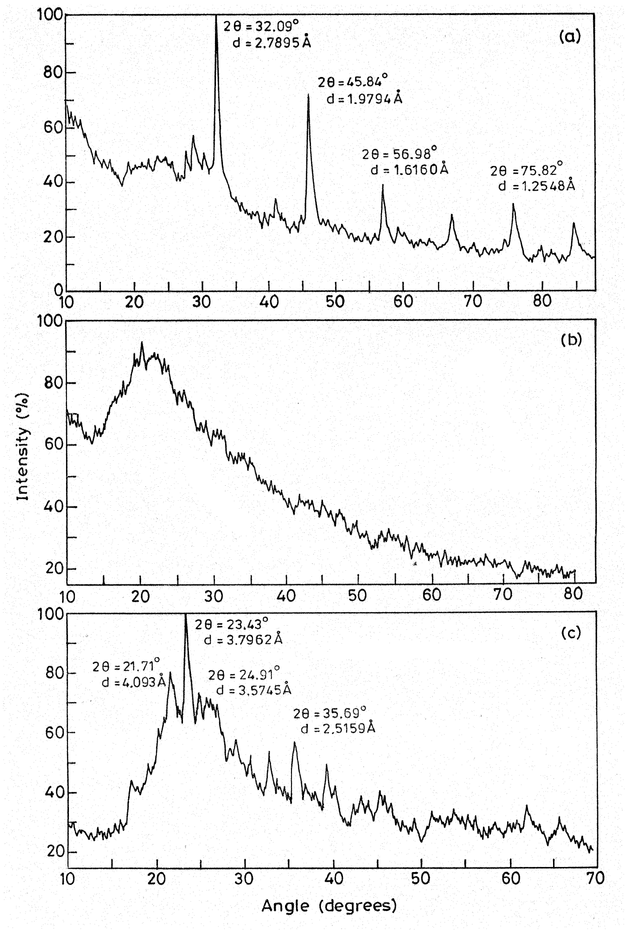


Figure 4. X-ray diffraction patterns of (a) solid waste, (b) Psy and (c) flocs.

stances, which are highly soluble at acidic pH and less soluble in alkaline pH but are precipitated at neutral pH.

Although the XRD patterns do not give any specific evidence for the mechanism of flocculation, they may be used as supportive evidence. Figure 4 presents the comparison of XRD patterns observed for the solid waste, Psy and flocs at room temperature from $2\theta = 10$ to $80^\circ$. The diffraction pattern (a) shows a crystalline nature of solid waste, whereas pattern (b) shows a complete amorphous nature of Psy. The flocs showed a diffraction pattern quite different from the diffractograms of solid waste and Psy. The $2\theta$ (diffraction angle) and the $d$-values (diffracting intensities) observed in (a) are changed altogether in pattern (c). This constitutes primary evidence that different crystal type was formed.[25] The change in the $2\theta$ angle and $d$-values indicates the change in nature of crystalline waste material in wastewater during the flocculation process. This may be due to the interactions between free hydroxyl groups of polysaccharide and contents of the textile waste.[14]

Anionic polymers are known to produce larger flocs by a bridging mechanism.[26] In this experiment, however, the extent of change observed in the patterns (a) and (c) suggests that apart from secondary bonding between flocculant and solid waste, there may also be an involvement of primary bonding like chelation[27] between crystalline matter of the waste and the Psy.

## Conclusions

The natural anionic polysaccharide of *Plantago psyllium* mucilage was found to be a very effective flocculant, capable of removing more than 90 and 68% of suspended and dissolved solids, respectively, from the textile effluent. The efficacy of Psy in flocculating textile wastewater is dependent on the pH of the medium under the test conditions. The most suitable pH values for SS and TDS removal were acidic and neutral, respectively. Therefore the overall treatment of this effluent sample involved two steps. The optimal treatment time was 3–5 h. A very low flocculant concentration of 1.6 mg/L was capable of removing appreciable amount of solids. XRD patterns of the treated and untreated solid waste showed Pb, Ni, Mn and Cd, and these patterns were used as supportive evidence for suggesting the possible mechanism of flocculation. It was concluded from the results that apart from secondary bonding (bridging) between flocculant and solid waste, there might also be an involvement of primary bonding like chelation between crystalline matter of the waste and the Psy.

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