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Improvement of alum and PACl coagulation by polyacrylamides (PAMs) for the treatment of pulp and paper mill wastewater

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Abstract

A study using coagulation–flocculation method for the treatment of pulp and paper mill wastewater has been carried out. The efficiency of alum and polyaluminum chloride (PACl) when used alone and in coupled with cationic polyacrylamide (C-PAM) and anionic polyacrylamide (A-PAM) on the treatment of pulp and paper mill wastewater were studied. The reduction efficiency of turbidity and chemical oxygen demand (COD), removal efficiency of total suspended solids (TSS), sludge volume index (SVI) and settling time are the main evaluating parameters. In coagulation–flocculation process using single coagulant, coagulant dosage and pH play an important role in determining the coagulation efficiency. At the optimum alum dosage of 1000 mg/L and optimum pH of 6.0, turbidity reduction is found to be 99.8%, TSS removal is 99.4% and COD reduction is 91%. The optimum dosage and pH for PACl are 500 mg/L and 6.0, respectively, at which it gives 99.9% reduction of turbidity, 99.5% of TSS removal and 91.3% of COD reduction. A combination of inorganic coagulant and flocculant or polymer is applied in which alum and PACl are used coupled with the C-PAM (Organopol 5415) and A-PAM (Chemfloc 430A). Overall, alum coupled with Organopol 5415 is the best system among all systems studied. It gives 99.7% reduction of turbidity, 99.5% removal of TSS and 95.6% reduction of COD, and at the same time with low SVI (38 mL/g) and low settling time (12 s).

Keywords: Coagulation; Flocculation; Polyelectrolyte; Sludge volume index

1. Introduction

The pulp and paper mill is a major industrial sector utilizing a huge amount of lignocellulosic materials and water during the manufacturing process. It can consume as high as 60 m^3 of freshwater per tonne of paper produced [1]. According to Thompson et al. [1], currently the concept of "zero liquid effluent" has been suggested and is applicable in area with extremely limited water sources. However, the recovery and reuse of the water can increase the concentration of organic and inorganic species, which in turn can affect paper formation, bacteria loading or lead to corrosion and odours. Common pollutants include suspended solids (SS), colour compound, heavy metals, organic and inorganic substances, phenols, chloroorganics, cyanide, sulphides and other soluble substances [2]. The wastewater can cause considerable damage to the receiving waters if discharged untreated [3–5].

Unlike freshwater, pulp and paper mills wastewater contains fibre and can cause unique solid/liquid separation challenges. Most solid/liquid separation systems have difficulty operating when the requirements are to produce high quality water, to remove fine particles, to operate continuously and remove high quantities of fibre. Chemical coagulation followed by sedimentation is a probed technique for the treatment of high suspended solids wastewater especially those formed by colloidal matters. Research and practical applications have shown that coagulation will lower the pollution load and could generate an adequate water recovery [6–10]. As a result of the smaller load, the wastewater treatment plant might be designed more energy efficiently at a smaller footprint and might be built at lower investment costs [11].

Coagulation is mainly induced by inorganic metal salts, e.g. aluminum and ferric sulphates and chlorides. Polyelectrolytes of various structures, e.g. polyacrylamides, chitosan, polysaccharides, polyvinyl and many more are usually used as coagulant aids to enhance the formation of larger floc in order to improve the rate of sedimentation. According to Aguilar et al. [12], anionic polyacrylamide when added with ferric sulphate or

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polyaluminum chloride led to a significant increase in the settling speed. In the earlier work done by Stephenson and Duff [13], they found that the removal of total carbon, colour and turbidity of up to 88, 90 and 98%, respectively, were observed in the treatment of mechanical pulping effluent using ferric chloride, ferrous sulphate, aluminum chloride and aluminum sulphate.

Aluminum and iron salts are widely used as coagulants in water and wastewater treatment and in some other applications. Their mode of action is generally explained in terms of two distinct mechanisms: charge neutralization of negatively charged colloids by cationic hydrolysis products and incorporation of impurities in an amorphous hydroxide precipitate so-called sweep flocculation [14]. The relative importance of these mechanisms depends on factors such as pH and coagulant dosage.

Recently, the use of synthetic polyelectrolytes as flocculants for suspended solids removal in wastewater treatment has grown rapidly [15,16]. Acrylamide is a crystalline and relatively stable monomer which is soluble in water and many organic solvents. Acrylamide is a polyfunctional molecule that contains a vinylic carbon–carbon double bond and an amide group. Girma et al. [17] reported that the electron deficient double bond of acrylamide is susceptible to a wide range of chemical reactions including nucleophilic additions, Diels-Alder, and free radical reactions. Flocculations of suspended particles occur via charged amide or carboxylic groups. Polyacrylamide (PAM) is a commonly used polymeric flocculant because it is possible to synthesize polyacrylamides (PAMs) with various functionalities (positive, neutral, or negative charge) which can be used to produce a good settling performance at relatively low cost.

The main objectives of the present study are to investigate the coagulation–flocculation efficiencies of alum and PACl when used alone and in coupled with cationic and anionic PAMs in the treatment of pulp and paper mill wastewaters and to select the most appropriate coagulant–flocculant scheme with the technical analysis criteria. The effects of coagulant dosage, flocculant dosage and pH are studied. The turbidity, TSS and chemical oxygen demand (COD) concentrations and SVI are used as evaluating parameters.

2. Material and methods

2.1. Materials

The wastewater was collected from the wastewater treatment plant equalization tank of a paper mill in Penang, Malaysia. Tissue papers are the main product of the mill with a monthly capacity of 3000 metric tonnes. The wastewater produced by the plant was 96 m^3 per tonne of paper produced. Wastewater samples were characterized and the analyses are given in Table 1. These parameters were measured based on the Standard Methods for the Examination of Water and Wastewater [18].

Alum and PACl were used as coagulants and very high molecular weight cationic polyacrylamide (C-PAM), Organpol 5415, with low charge density and high molecular weight anionic polyacrylamide (A-PAM), Chemfloc 430A, with high charge density were used as flocculants. Organopol 5415 was supplied by Ciba Speciality Chemicals and Chemfloc 430A was supplied by Chemkimia. Distilled water was used to prepare all the PAM feedstock solutions of 0.1%.

Table 1
Chemical characteristics of the wastewater used

Parameters	Value[a]
TCOD[b] (mg/L)	3087
SCOD[c] (mg/L)	318
Suspended solids (mg/L)	5240
Turbidity (NTU)	4770
pH	7.3–8.3

[a] Values show the average values of 20 samples.
[b] Total chemical oxygen demand.
[c] Soluble chemical oxygen demand.

2.2. Experimental procedure

Jar test procedures were performed with the conventional jar apparatus (Stuart Science Flocculator model, SWI) using 500 ml wastewater samples. Different combinations of pH (5, 6, 7, 8, 9, 10), alum dosage (200, 500, 800, 1000, 1500, 2000 mg/L), PACl dosage (50, 100, 200, 500, 1000, 1500 mg/L) and PAMs dosage (1, 2, 3, 4, 5, 6 mg/L) were tested. The selected coagulant dosage was added to 500 mL of wastewater and it was stirred for a period of 2 min at 200 rpm. It was followed by a further slow mixing of 15 min at 40 rpm after the selected PAM dosage was added to the same solution. The pH of the solution was adjusted accordingly. The flocs formed were allowed to settle for 30 min. After settling, the turbidity, TSS, and COD of the supernatant were determined. The remaining portion of the treated wastewater samples was used to determine the sludge volume index (SVI). All parameters were determined according to the APHA method.

3. Results and discussion

3.1. Coagulation with alum

In coagulation–flocculation processes using inorganic coagulant, coagulant dosage and pH play an important role in determining the coagulation efficiency. In wastewater treatment using inorganic coagulants, an optimum pH range in which metal hydroxide precipitates occur, should be determined. The addition of metal coagulants depresses the wastewater pH to a lower value. The jar test experiments with alum, using pulp and paper mill wastewater with pre-adjusted pH of 5.0, 6.0, 7.0, 8.0, 9.0 and 10.0, for each pH value with the alum dosages of 200, 500, 800, 1000, 1500 and 2000 mg/L, were run. The effects of pH adjustment and coagulant dosage by alum on turbidity, TSS and COD are illustrated in Fig. 1(a–c), respectively. In general, decreasing the pH from the alkaline levels to near neutral levels has a strong positive effect on reduction/removal of turbidity, TSS and COD.

Fig. 1 clearly shows that turbidity reduction, TSS removal and COD reduction efficiencies increase with increase in coagulant dosage and pH adjustment till it reaches its highest value, optimum pH, after which the reduction and removal efficien-

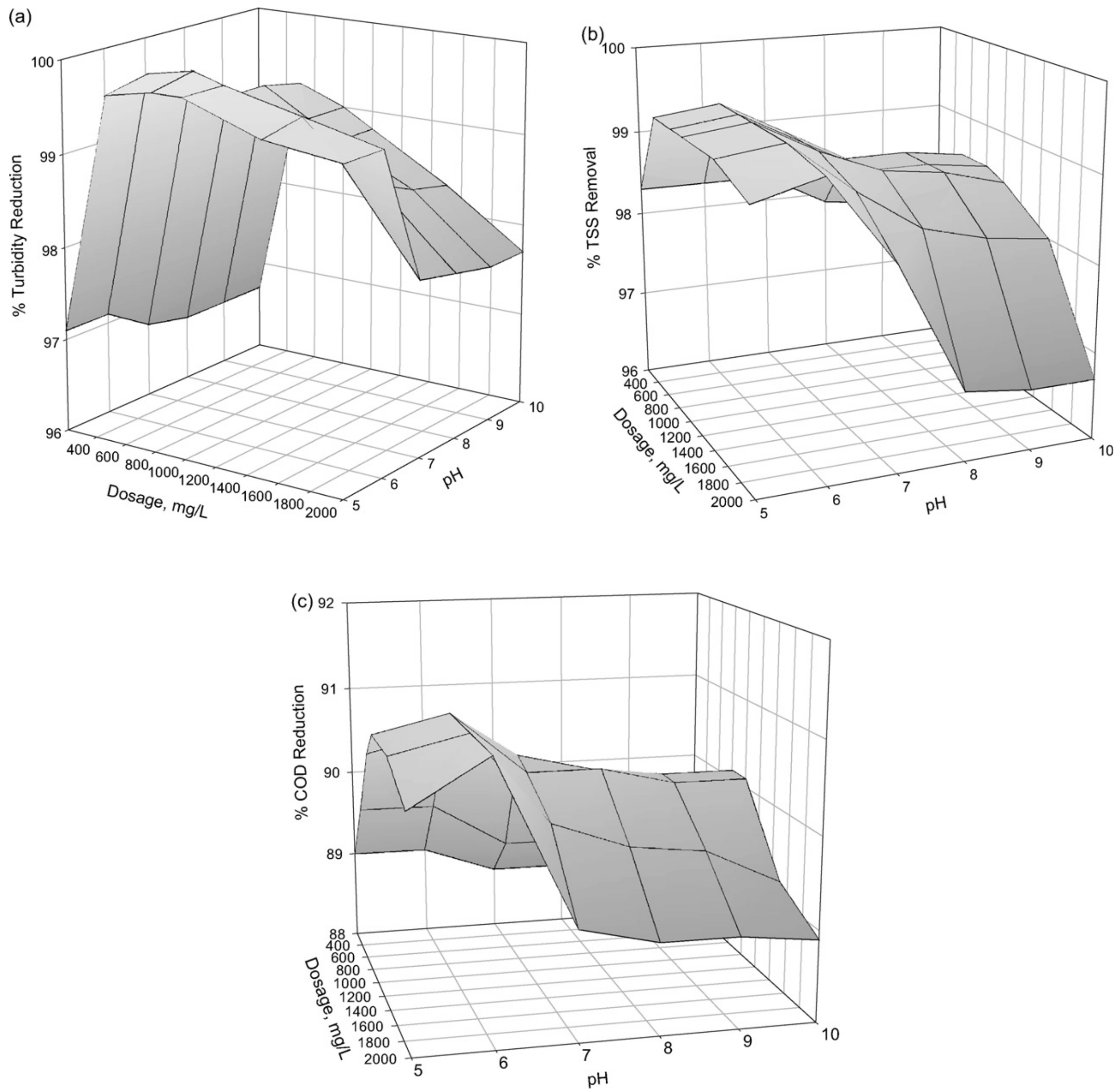


Fig. 1. Effects of alum dosage and pH on (a) turbidity reduction, (b) TSS removal and (c) COD reduction.

cies start to decrease. It can be seen that an optimum range of pH exists between 5.0 and 6.5 beyond which effluent quality deteriorates. The optimum dosage is approximately 1000 mg/L and the optimum pH is approximately 6.0. At the alum optimum dosage of 1000 mg/L and optimum pH of 6.0, turbidity reduction is 99.8%, TSS removal is 99.4% and COD reduction is 91%.

3.2. *Coagulation with PACl*

To study the effects of the PACl dosage and pH on the turbidity reduction, TSS removal and COD reduction, jar tests were conducted with the PACl dosages of 50, 100, 200, 500, 1000 and 1500 mg/L and pH adjusted from 5.0 to 10.0. The results obtained are shown in Fig. 2. The effects of coagulant dosage and pH with PACl are parallel to the effects observed with alum. The turbidity reduction, TSS removal and COD reduction efficiencies increase with increase in coagulant dosage and pH till it reaches its highest value, optimum pH, after which the reduction and removal efficiencies start to decrease. The effects of coagulant dosage and pH on turbidity reduction are not significant within the dosage range and pH studied. However, the turbidity reduction efficiency starts to drop at pH 10.0. The highest turbidity reduction is 99.9% and the lowest turbidity reduction achieved by PACl is 99.3%.

Fig. 2(b) and (c) shows that the TSS removal and COD reduction trends are similar to each other. This may be due to the high organic contents of the suspended solid particles. The effect of increasing the PACl dosage only reveals minor impacts on the reduction/removal efficiencies of TSS and COD. Nevertheless, pH shows significant effects on the TSS removal and COD reduction in the alkaline pH range between 8.0 and 10.0. The TSS removal efficiency decreases from more than 99% to below

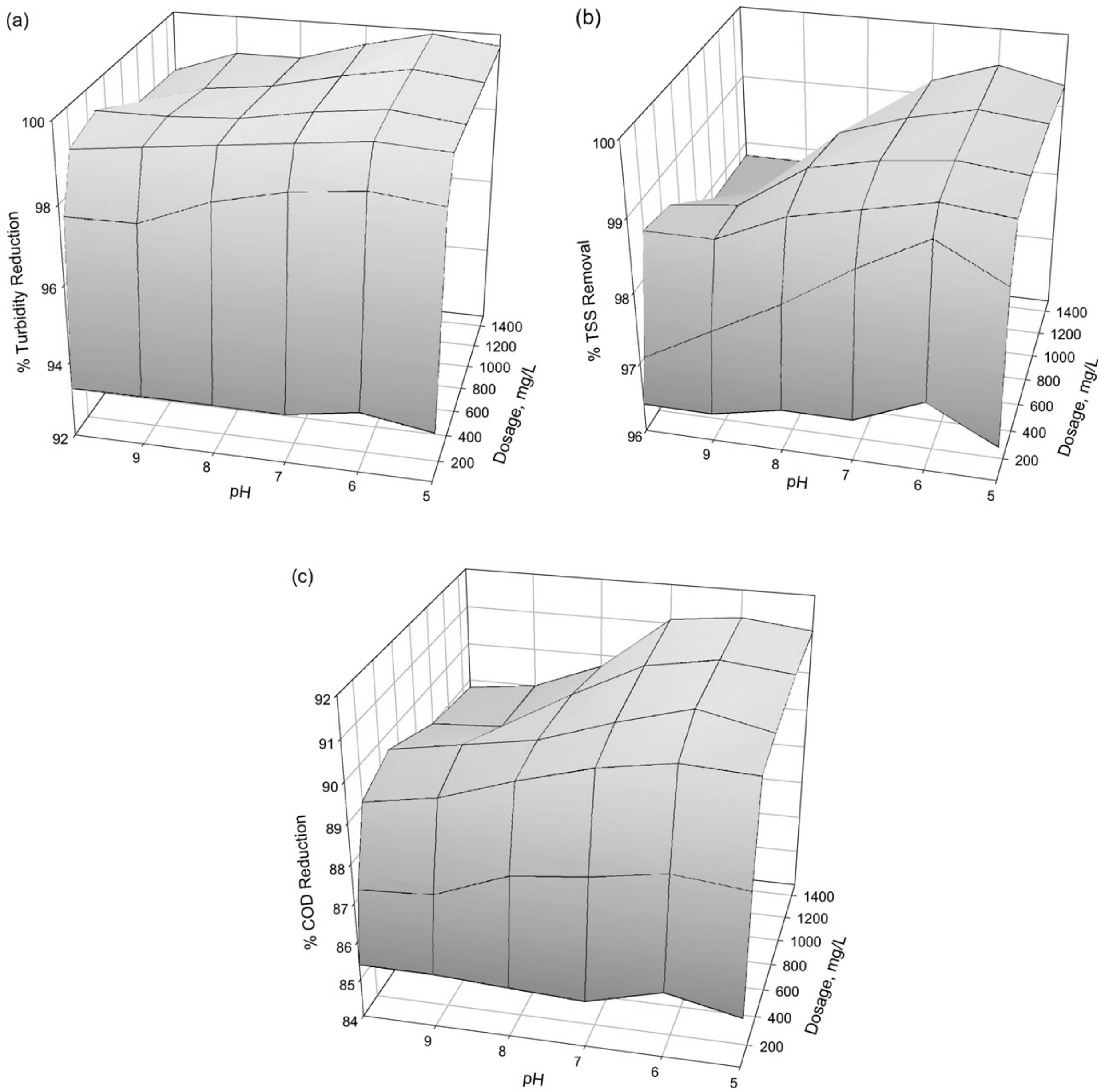


Fig. 2. Effects of PACl dosage and pH on (a) turbidity reduction, (b) TSS removal and (c) COD reduction.

98%. The COD reduction efficiency decreases from more than 91% to below 86%.

The optimum PACl dosage and pH are 500 mg/L and 6.0, respectively. This result reveals that the optimum coagulant dosage for PACl is less than that of alum but the optimum pH for both coagulants remains the same at pH 6.0. According to Huang and Pan [19], at lower pH and lower coagulant dosage, the only mechanism for destabilization of particles is charge neutralization. At low pH, because the aggregates are small in size, the mechanism of colloidal destabilization is mainly charge neutralization. At lower dosage, PACl behaves like the alum salt; therefore, charge neutralization is the principal mechanism for destabilization.

As stated by Huang and Pan [19], PACl is a prehydrolyzed inorganic coagulant. It is prepared by reacting NaOH or Na_2CO_3 with concentrated $AlCl_3$ solution under controlled reaction rate and mild temperature. The extent of prehydrolysis of pure PACl is described in terms of the ligand ratio r, which is the molar ratio of hydroxide ($[OH^-]$) to the total Al present in solution ($[Al_{total}]$). Theoretically, r is in the range between 0 and 3, or basicity between 0 and 100%. Basicity is the percentage of Al^{3+} acidity satisfied by prehydrolysis or base addition. The major dissolved Al species from PACl available for coagulation is controlled by r. For $r < 1$ (basicity <33%), monomeric Al species such as Al^{3+} and $AlOH^{2+}$ dominate. Some dimeric Al such as $Al_2(OH)_2{}^{4+}$ may be present. The basicity of the PACl used in the present study is 30%. Evidently, this indicates that the main Al species present for the destabilization of colloidal particles are monomeric and dimeric Al which functions only through charge neutralization.

3.3. Effect of flocculant dosage

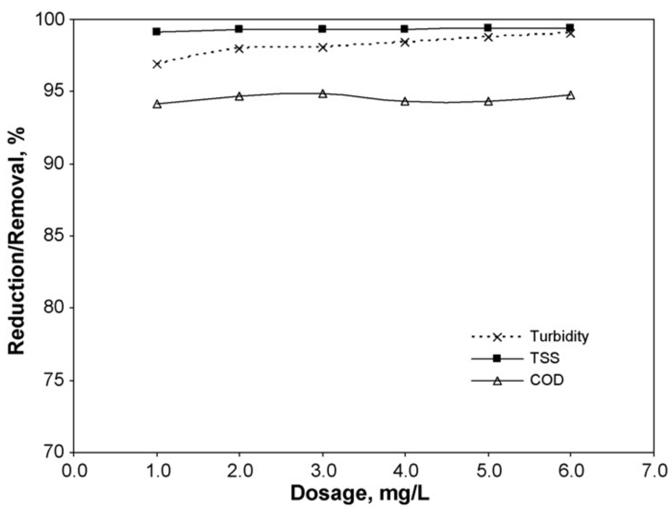
Alum and PACl are used in coupled with C-PAM (Organopol 54515) and A-PAM (Chemfloc 430A). The effect of cationic and anionic flocculant dosages on the reduction of turbidity and COD and the removal of TSS was investigated. Flocculant dosage was increased from 1.0 to 6.0 mg/L with a fixed amount of alum (500 mg/L) or PACl (200 mg/L). The initial pH of wastewater was adjusted to pH 6.0. The reduction or removal of turbidity, TSS and COD efficiencies were calculated from the turbidity, TSS and COD initial concentration in the raw wastewater and final concentration in the supernatant.

The results obtained for alum + Organopol 5415, alum + Chemfloc 430A, PACl + Organopol 5415 and PACl + Chemfloc 430A treatments are depicted in Figs. 3 and 4. The reduction and removal efficiencies of turbidity, TSS and COD did not change significantly or were imperceptible when different concentrations of flocculant were added. The reduction and removal efficiencies of turbidity, TSS and COD distributed evenly throughout all the dosage range studied as exhibited in Figs. 3 and 4. It can be seen that increasing flocculant dosage does not always improve the reduction or removal rates. Overall, the reduction efficiencies of turbidity and COD and the removal efficiency of TSS are more than 90% even at low alum (500 mg/L) and PACl (200 mg/L) dosages.

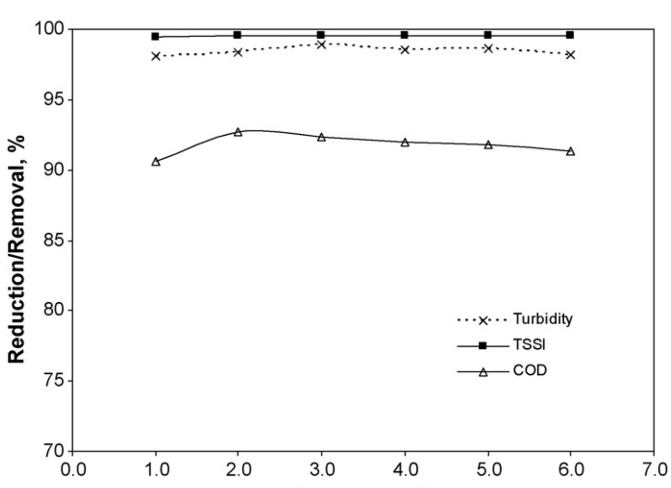
3.4. Effect of flocculant addition

3.4.1. Turbidity reduction, TSS removal and COD reduction

The effect of flocculant (C-PAM and A-PAM) addition on the reduction and removal of turbidity, TSS and COD, settling time and SVI was investigated. Coagulant dosage was increased from 50 to 2000 mg/L with a fixed dosage of flocculant (1 mg/L). The initial pH of wastewater was adjusted to pH 6.0. Fig. 5(a), which plots the effect of flocculant addition on the turbidity reduction, points to the substantially increased performance for both coagulants as the coagulant dosage increases. The addition of flocculant leads to a significant increase in efficiency. Alum

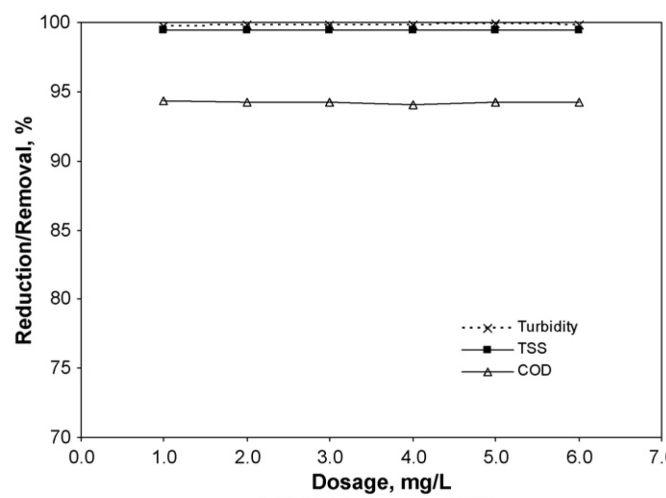


(a) Alum + Organopol 5415

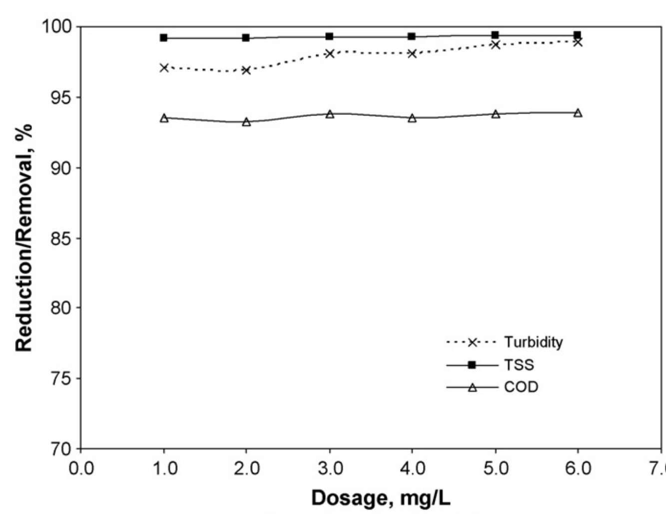


(b) Alum + Chemfloc 430A

Fig. 3. Effect of flocculant dosages on the turbidity, TSS and COD reduction/removal in flocculant/alum treatment. Alum dosage = 500 mg/L and pH 6.0.



(a) PACl + Organopol 5415



(b) PACl + Chemfloc 430A

Fig. 4. Effect of flocculant dosages on the turbidity, TSS and COD reduction/removal in flocculant/PACl treatment. PACl dosage = 200 mg/L and pH 6.

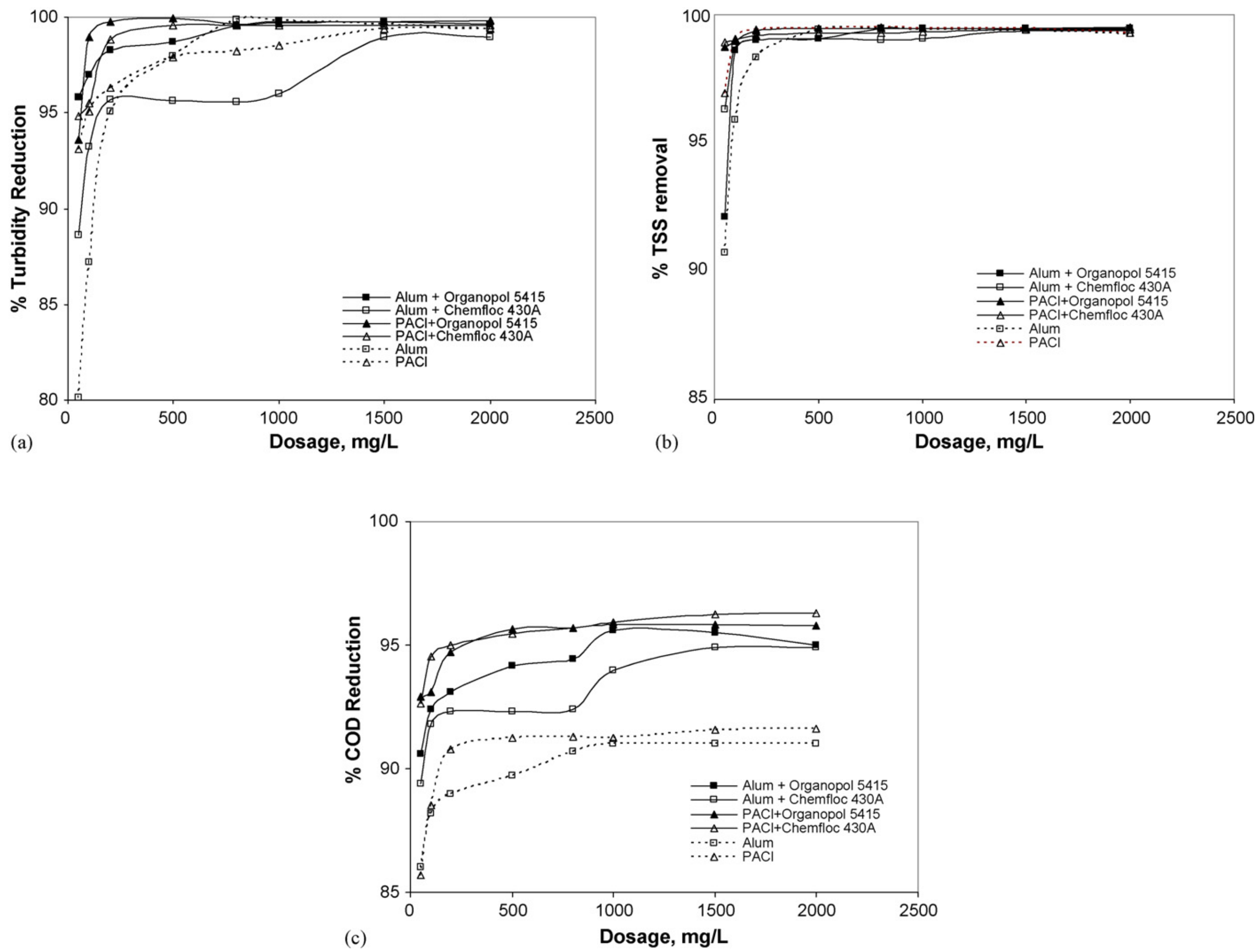


Fig. 5. Effect of flocculant addition on (a) turbidity reduction, (b) TSS removal and (c) COD reduction. Flocculant dosage = 1 mg/L.

and PACl provide good performances (more than 88%) even at the lowest dosage assayed.

In the case of alum, the positive effect of using C-PAM (Organopol 5415) could be clearly observed for low coagulant dosages of 50, 100 and 200 mg/L since a much greater reduction efficiency is achieved when only the alum is used. The reduction efficiency of turbidity is improves from 80 to 96% for the alum dosage of 50 mg/L. This means that lower quantities of alum are needed to obtain an acceptable reduction in turbidity. The other coagulant, PACl, also shows improved performances with dosages of 50, 100 and 200 mg/L, although the improvement is not as great as in the case of alum. The performance of PACl + Organopol 5415 in terms of turbidity reduction is the best combination coagulant + flocculant system among all combinations investigated.

The effect of flocculant addition on TSS removal is depicted in Fig. 5(b), which shows a similar tendency as in Fig. 5(a). The efficiency of the TSS removal increases with coagulant dosage, although the improvement is not as great as in the case of the turbidity reduction. All systems studied achieve more than 99% removal of TSS when the coagulant dosage approaches optimum dosage of each system.

Fig. 5(c) shows the effect of flocculant addition on the reduction efficiency of COD. The results show that the COD reduction is gradually improved with increasing coagulant dosage. The addition of flocculant, both C-PAM and A-PAM, initiates a significant increase in COD reduction efficiency. Alum and PACl exhibit very high COD reduction efficiency (>89%) even at the lowest dosage applied (50 mg/L). This indicates that the use of flocculant lowers the coagulant dosage needed to obtain a satisfactory reduction in COD. Obviously, the PACl performs much better than alum in the reduction efficiency of COD.

The optimum coagulant dosages for alum + Organopol 5415, alum + Chemfloc 430A, PACl + Organopol 5415 and PACl + Chemfloc 430A are 1000, 2000, 1000 and 1500 mg/L, respectively, in terms of turbidity, TSS and COD reduction/removal efficiencies. These results suggested that the used of Organopol 5415 will reduce the usage of coagulants.

3.4.2. Flocs settling time

In the coagulation–flocculation process, the settling speed of the flocs formed is important since this will influence the overall cost and efficiency. In order to evaluate this parameter, the settling time for the flocs formed to reach half of the solution

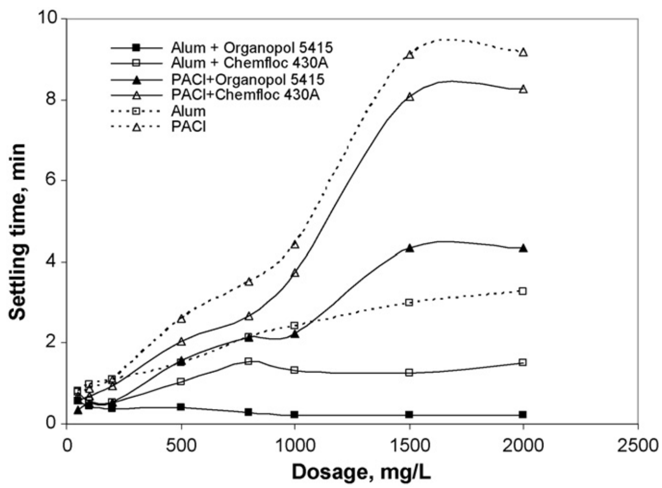


Fig. 6. Effect of flocculant addition on flocs settling time.

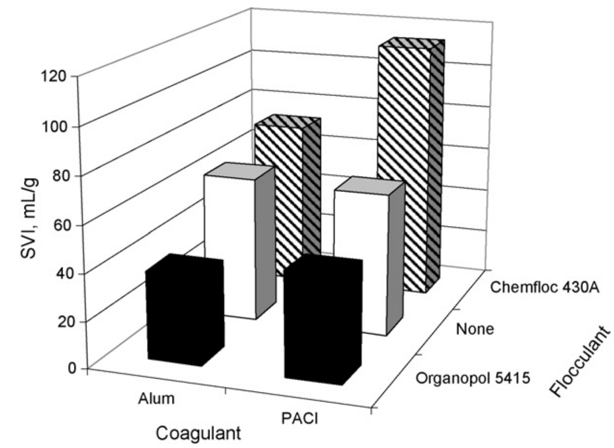


Fig. 7. Effect of flocculant addition on SVI.

height in jar tests was recorded and the results are shown in Fig. 6. The addition of flocculant had a significant effect on the settling time when alum and PACl were used as coagulants. This may be due to the bridging flocculation mechanism of the high molecular weight flocculant by which this flocculant may coil the flocs formed by the coagulants to become more compact, high strength and larger flocs [20,21]. The increase of flocs size favors the flocs settling speed and therefore reduces the settling time of the flocs formed. The flocs generated by alum settle faster than the flocs generated by PACl. Alum + Organopol 5415 produces the flocs that only need less than 1 min settling time for all dosages studied.

3.4.3. Sludge volume index (SVI)

The sludge produced in the physical–chemical treatment is due to the amount of organic matter and total solids in suspension that are removed and the compounds formed from the coagulant used, since practically almost all of the latter will form part of the sludge solids. In general, the amount and characteristics of the sludge produced during the coagulation–flocculation process depend on the coagulants used and on the operating conditions. In order to observe the volume and settling characteristics of the sludge produced, SVI was determined.

The results obtained for the optimum conditions are shown in Fig. 7. When single coagulant is used, the one that produces less SVI is PACl. This is in agreement with one of the advantages attributed by the literature to this coagulant, namely it reduces the quantity of sludge and improves its dewatering [14,22]. The addition of Organopol 5415 reduces the SVI obtained with respect to the SVI value when single coagulant is used. However, the addition of Chemfloc 430A increases the SVI value especially when it is used together with PACl where the highest SVI value is 114 mL/g. The presence of this anionic flocculant may alter the coagulation by hydrolyzing metal ions in terms of replacement of hydroxyl ion or the kinetics of precipitation. According to Duan and Gregory [14], in the neighborhood of the optimum pH of freshly precipitated aluminum hydroxide, coagulation and destabilization of particles is due to the coating of the inherently unstable aluminum hydroxide possibly resulting from ionization of the precipitate surface or adsorption of anionic ions. The presence of anionic ions or polymers in solution can reduce significantly the positive charge of aluminum hydrolysis products. Furthermore, visual inspection of the sludge indicated the formation of small, loose and low density flocs for the mixture of coagulant and Chemfloc 430A.

SVI reduces significantly (40% from the initial value) when cationic Organopol 5415 is used as flocculant together with alum. In Fig. 7, the SVI diminishes from 63 mg/L up to 38 mL/g when alum is acts together with cationic Organopol 5415.

4. Conclusions

The treatment of pulp and paper mill wastewater using alum and PACl coagulant in coupled with PAMs enhances the reduction/removal of turbidity, TSS and COD, improves the flocs settling time and produces lower volume of sludge compared to the results obtained when the coagulants are used alone. However, the increase of PAMs dosage does not have significant effect on alum and PACl coagulation. C-PAM (Organopol 5415) performs better than A-PAM (Chemfloc 430A) when coupled with alum and PACl in the treatment of pulp and paper mill wastewater. Overall, alum coupled with Organopol 5415 is the best system among all systems studied which show the highest efficiency in terms of reduction of turbidity, removal of TSS, reduction of COD, SVI and settling time. The additions of PAM have improved the treatment performances.

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