

REMOVAL OF DYE FROM WATER BY COAGULATION USING ALUMINIUM SULFATE AND LIME

Kifle Yeheyes, Feleke Zewge* and B.S. Chandravanshi

Department of Chemistry, Faculty of Science, Addis Ababa University, PO Box 1176
Addis Ababa, Ethiopia. E-mail: zewge@chem.aau.edu.et

ABSTRACT: Wastewaters from textile industries represent a source of water contamination, and should be treated in some way so as to reduce the concentration of the polluting dyes, prior to discharge of the wastewater to the receiving environment. In this study the dye removal capacity of locally produced alum was investigated. The efficiency of the process was investigated in series of batch experiments using simulated textile dye solutions to assess the effects of various process parameters: contact time, alum-lime weight ratio, coagulant dosage, dye's initial concentration, pH and reaction kinetics of coagulation. The result of the study showed that an increase in both coagulant dosage and dye initial concentration increases the percentage of dye removal. In all cases the percentage of dye removal at optimized conditions is nearly or above 90%. Most of the reactions are completed in less than 20 minutes. The highest percentage of the dye removal was observed at pH around neutrality for all the dyes indicating that addition of lime is indispensable to maintain efficient floc formation. The reaction kinetics of Indantrene Brown and Imperon Orange was observed to obey pseudo first order kinetics, with rate constants $k_1 = 0.055 \text{ min}^{-1}$ and $k_2 = 0.114 \text{ min}^{-1}$ for Indantrene Brown and Imperon Orange, respectively. It was found that alum combined with lime is suitable material for removing dyes from wastewaters.

Key words/phrases: Alum, coagulation, dye removal, lime, textile wastewater treatment

INTRODUCTION

Textile industries, particularly those involved in dyeing and finishing processes are major water consumers and are source of considerable pollution (Walsh *et al.*, 2003). Because of a chemical intensive process, textile finishing industry generates effluents containing significant amounts of a wide range of residues and because of inefficiency of the existing dyeing technologies it is estimated that 15–20% of the dye and pigment is lost to the effluent during the dyeing process (Peters and Freeman, 1995).

Due to persistent aromatic structure and recalcitrant nature of various dyes, the disposal of coloured wastewater creates both aesthetic and other serious environmental problems. These wastewaters impede light penetration in the treatment plants, thus upsetting the biological treatment processes within the treatment plant. Residual colour is also known to increase the BOD that causes depletion of dissolved oxygen in water (Pola *et al.*, 2003). Many dyes are known to be toxic

to some microorganisms, and may cause direct inhibition of their activities. Dyes may also be problematic as toxic amines are often produced due to incomplete degradation by microorganisms, where lethal dose may be reached affecting aquatic systems and the associated fauna and flora (Stumm and Morgan, 1996). Moreover, there are considerable evidences that these metabolites of dyes are the actual carcinogenic agents (Kalliala and Talvenmaa, 2000).

In wastewater treatment technology, various techniques have been employed in the past. In general, two major techniques are available for the removal of dyes (Eckenfekder, 1966). They are biological decompositions and physico-chemical processes. Biological decomposition can be used to remove dissolved organics from wastewaters, but since microorganisms themselves rely on organic matter for energy and growth, removal of low level of contaminants may not be significant. Moreover the organic contaminant may not be biodegradable, a property of most dyes too (Peters and Freeman, 1995).

* Author to whom correspondence should be addressed.

Various chemical processes have been used to remove color from textile dyeing wastewater. Among the physico-chemical processes in use are adsorption by activated carbon, biosorbents, and naturally occurring minerals (Robinson *et al.*, 2002; Al-Ghouti *et al.*, 2003; Walker and Weatherley, 2003; Abera Gizaw, 2004; Netpradit *et al.*, 2004); advanced oxidation processes using ozone, UV, hydrogen peroxide, Fenton's reagent, UV/H₂O₂, and ozone/UV (Chu and Ma, 1997; Ince and Gonenc, 1997; Kuo, 1992); chemical coagulation by alum, magnesium carbonate, ferrous sulfate/lime, and vermiculite (Judkins and Hornsby, 1978; Mehrota *et al.*, 1995; Choi and Cho, 1996; Chu, 2001; Georgiou *et al.*, 2002).

The most widely used and effective physico-chemical method available for removing color-producing substances from wastewater is adsorption on activated carbon; however, the running cost is very high (Robinson *et al.*, 2002). Furthermore regeneration solutions produce a small additional effluent, while regeneration by refractory technique results in a 10-15% loss of the sorbent and its uptake capacity. Currently, there is a growing interest in using low cost easily available materials for this purpose (Walker and Weatherley, 2003) however; the technologies are not well established.

Advanced oxidation processes are very attractive with regard to the destruction of dyes to simple substances. For example, decoloration of sixteen commercial dyes in the aqueous phase was investigated by UV (Chu and Ma, 1997), where the removal rates of dyes ranged from 8 (anthraquinone dyes) to 98% (polyazo dyes) depending on the dye structures. A similar alternative is the use of UV/H₂O₂ to oxidize dye wastewater (Ince and Gonenc, 1997). Fenton's reagent was also used for dye decoloring; about 88% of dye being removed at pH 3.5 (Kuo, 1992). However, these technologies are very expensive and the oxidation reaction may not be complete.

Chemical coagulation by the use of inorganic coagulants such as alum, magnesium carbonate, ferrous sulfate, and vermiculite is another feasible way of removing color from dye wastewater. The treatment with ferrous sulfate regulating the pH in the range 9.0±0.5 using lime was effective, but it proved to be very costly mainly due to the massive production of solids that precipitated (Georgiou *et al.*, 2002). The treatment of textile dye waste with magnesium carbonate was investigated by

(Judkins and Hornsby, 1978; Mehrota *et al.*, 1995). The results indicated that the removal of color was good with vat and sulfur dyes, but less effective on dispersed dyes. It was reported that 1000 mg/L magnesium carbonate removed 92% of color from vat dye and sulfur dye effluent at pH 10, and 38% of color from basic dye effluent at pH 11. A lower color removal of 75% was observed in vat dye effluent where 14 000 mg/L of ferrous sulfate was used as the coagulant at an optimum pH of 10 (Judkins and Hornsby, 1978).

The performance of vermiculite extract solution as a coagulant for extracting colour from dye wastewater was also tested (Choi and Cho, 1996). This process generated less sludge at lower coagulant doses than processes using conventional inorganic coagulants, and its performance increased with increasing temperature and agitation intensities. The removal of dyes from textile dyeing wastewater by recycled alum sludge (RAS) generated by the coagulation process itself was studied (Chu, 2001). It was found that RAS is a good way of removing hydrophobic dye in wastewater, while simultaneously using fresh alum. The chemical coagulation systems discussed above are operating at high pH and the removal efficiency depends on the nature of the individual dye under consideration. Although chemical coagulation has been widely used, in water treatment, for control over colloidal and suspended particles, the available information regarding the removal of dyes from textile effluents is very limited. Relatively fewer studies have been conducted on the application of methods based on chemical coagulation.

In all textile industries in Ethiopia, the process liquor containing unfixed dyestuffs and auxiliaries is directly discharged to the environment without any proper treatment. Thus, they can be considered as major sources of pollutants. The Provisional Environmental Standard for Ethiopia proposed by the Environmental Protection Authority (EPA) sets emission limits of 5 mg/L of adsorbable organic compounds (EPA, 2003). Conventional treatment processes and discharge into municipal systems will no longer be tolerable at various levels. This fact calls for a combination of biological and physico-chemical processes along with a complementary treatment option to remove the dyes and, if possible, the residual COD (Marmangne, 1996). The availability of locally produced alum at an industrial scale is an

attractive feature to apply coagulation-flocculation process for dye removal. Thus the objective of the study was to test the feasibility of dye removal from textile effluent by using locally produced aluminium sulfate and lime.

MATERIALS AND METHODS

Materials

Seven different dyes were collected from Akaki Textile SC. They were selected randomly from the most frequently used group of dyes known as reactive-, vat-, and azo-dyes. The aluminium sulfate used in this study was purchased from Awash Melkassa Aluminium Sulfate and Sulfuric Acid Factory (Ethiopia). The alum was 17% Al_2O_3 about 54% aluminium sulfate). Lime, CaO (99%, BDH, UK) was used at a 0.5 to 0.165-alum/lime-weight ratio to adjust the pH for effective coagulation. Keeping the ratio fixed a series of alum/lime concentrations were prepared by dissolving it in 500 mL distilled and/or de-ionized water. Concentrated HCl was used to adjust the pH Analytical grade reagents were used unless stated otherwise. pH was measured using pH meter (Model 205, Beckman, USA). All experiments were conducted using distilled and/or de-ionized water. The concentration of dyes was measured using Beckman UV-Vis spectrophotometer (Model Du-65, USA).

Analytical methods

It was necessary to choose a sound and expeditious method to determine the dye concentration in solution before and after the coagulation. As the dye concentration may be controlled through colour, the chosen method was spectrophotometry in the visible region. The absorption spectrum (plot of absorbance versus wavelength) allowed us to determine the most suitable wavelength (λ_{max}) for absorbances measurement. Different concentrations of dye solutions from each type were prepared to determine their respective λ_{max} , depending on the characteristic intensities of the dyes' colours. The calibration curves (absorbance versus concentration) were constructed by dissolving 5, 10, 20, 40, 50, 100, 150, 200, up to 500 mg of each dye in a 500 mL volumetric flask using distilled water and measuring their absorbance at their respective λ_{max} . The calibration curve was linear for

a given dye within the concentration range tested (Table 1). The concentration of dye in solution was calculated from the absorbance measurements using the calibration curve.

Wavelength of maximum absorbance

The wavelengths of maximum absorbance (λ_{max}) and effective concentration ranges determined for the seven dyes considered in this study are given in Table 1. Where SO: Solfasol Orange, CR: Cibacron Red, IO: Imperon Orange, SY: Solar Yellow BG, IG: Indanthrene Brown, HB-R-S: Hydron Blue R-Stabilol and RTB: Remazol Turquoise Blue G. From the table it is clearly seen that the effective concentration ranges for different dyes are different as it depends on the intensities of the colors. The absorbances of different standard solutions were used to construct the calibration curves. The correlation coefficients of all the studied dyes show strong linear ($R=0.9988-0.9999$) relationship between the concentration of the standard solutions and the absorbance at the dye's wavelength of maximum absorbance.

Table 1. The wavelengths of maximum absorbance and effective concentration ranges of the dyes.

Dye type	λ_{max} of the dye (nm)	Effective conc. range (mg/L)
SO	407	$40 \leq X \leq 400$
SYBG	399	$20 \leq X \leq 100$
RTBG	625	$20 \leq X \leq 150$
CR	540	$20 \leq X \leq 80$
IO	502	$20 \leq X \leq 100$
HBRS	600	$40 \leq X \leq 200$
IB	322	$20 \leq X \leq 125$

Batch mode coagulation studies

In order to carry out the coagulation tests it was necessary to know the optimum alum/lime weight ratio to attain the pH value at which a maximum coagulation of the alum will be formed. The optimum weight ratio of alum to lime was determined by preparing 100 mL of 5 g/L solution of $\text{Al}_2(\text{SO}_4)_3$ and varying the dosage of lime.

The reaction time required for the removal of each dye was determined by mixing a single component dye solution with 500 mg/L of coagulant at constant temperature. The dye-coagulant solution was prepared in 500 mL beaker and was rapidly mixed (100 rpm) for 2 minutes

followed by slow mixing (40 rpm) for 30 minutes and allowed to settle for 180 minutes. 5 mL of the supernatant was taken to measure its absorbance at different time intervals to determine the concentration change.

The effect of coagulant dose on the removal of dye from solution was investigated by varying the amount of alum at fixed initial dye concentration. The reaction and settling times were the same as mentioned above. This experiment allowed us to determine the optimum alum dosage, which was used to determine the effect of other parameter described below.

The effect of initial concentration on the percentage of dye removal was investigated by preparing each experimental dye solutions from the stock solution (2. g/L) and diluted with distilled water to make it 500 mL. Then each of the experimental solution was mixed with defined quantity of coagulant (optimum dose), and stirred until equilibrium. The final dye concentration was obtained from the measurement of the absorbance of the supernatant.

The influence of pH on the removal efficiency of each dye was studied at fixed dye concentration and coagulant dosage. Concentrated HCl and solid CaO were used to adjust the required pH values of the dye solutions.

The kinetics of coagulation for two representative dyes was examined. The experiment was carried out by mixing 100 mg/L of each dye with 400 and 150 mg/L of coagulant, respectively, in 1000 mL solution and measuring the absorbance of 5 mL supernatant at each time interval. The selected dye concentrations were made in the range of calibration curve values so that its absorbance can be accurately measured just after the reaction begins.

For all process parameter investigations batch coagulation experiments were carried out by preparing experimental dye solution along with coagulant in 500 mL conical flasks at $23 \pm 2^\circ\text{C}$. All experiments were conducted in triplicates and controls consisting of the same experimental set up except for the parameter to be determined and/or varied were run simultaneously. The replicated data obtained from the experiment were analyzed by means of descriptive statistic and inference was made using correlation factors.

RESULTS AND DISCUSSION

Alum to lime weight ratio

When aluminium salt is added to water, a series of soluble hydrolysis species are formed (Bersillon *et al.*, 1978). Well documented species described in the literature that can be formed at low pH (<6.0) include monomer ($\text{Al}(\text{OH})_2^{2+}$, $\text{Al}(\text{OH})_2^+$, dimers ($\text{Al}_2(\text{OH})_2^{4+}$, trimers ($\text{Al}_3(\text{OH})_4^{5+}$; as well as the polymer $\text{Al}_{13}\text{O}_4(\text{OH})_{24}^{7+}$). Solid amorphous aluminium hydroxide ($\text{Al}(\text{OH})_3$ (s)) forms when aluminium ion in solution is supersaturated with respect to the solid phase at or near neutral pH. Furthermore, the negatively charged complex $\text{Al}(\text{OH})_4^-$ become predominant at high pH (>9.0). The removal of particular dye may depend on its interaction with the different species present at different pH. In wastewater treatment process, it is desired that the dyes should be attached to the alum floc and separated by settling and/or filtration. In addition, when alum is added to the water the pH decrease to below 4.0 which is unacceptable level both from the floc formation and treated water quality point of view.

The determination of alum-lime weight ratio showed that the maximum coagulation was observed at a ratio of 3, which corresponds to the pH value of about 7.5 (Table 2). Therefore, in the actual dye removal experiment the pH was adjusted to this ratio. In other works related to coagulation, similar results were also reported (Peters and Freeman, 1995). From reaction time study (data not shown), it was observed that the dye removal was rapid at the initial stages of contact time and gradually decreases with lapse of time. The results of the experiment, for most of the dyes showed that up to 90% dye removal was achieved at this pH.

Table 2. Effect of alum lime weight ratio on the final pH of the treated solution

No.	CaO added (g)	Alum/lime weight ratio	Final pH of the solution
1	0.05	10	4.02
2	0.10	5	4.22
3	0.15	3.3	6.10
4	0.165	3	7.50
5	0.20	2.5	8.83
6	0.25	2	9.29

Experimental condition: mass of alum=0.5 g, volume of the solution=100 mL, reaction time=60 min, settling time=180 min, temperature of the reaction=20.8°C).

Effect of coagulant dose

Effect of coagulant dosage at a fixed dye concentration is shown in Fig. 1. The percentage of dye removal increased with increasing coagulant dose. The coagulant dosage with higher percentage of dye removal was considered as an optimum coagulant dose for each dye, for the next experiment on effect of dye concentration. However at some points a major drop in the dye removal was observed as the coagulant concentration is increased. From related works it is observed that this result might be due to the restabilization of dye particles (Judkins and Hornsby, 1978). During the restabilization stage, the agglomerated dye

alum particles that are suspended in solution cannot be removed by gravity settling. However, they can still be removed by filtration. On the other hand, the removal capacity of the coagulant decreases as the coagulant dose increases for a fixed initial concentration of the dye (Fig. 2). This indicates that it is important to keep the alum dose minimum to achieve maximum capacity and removal efficiency. In addition, the sludge production is also increased with increasing dose as expected from the following simplified equation (1):

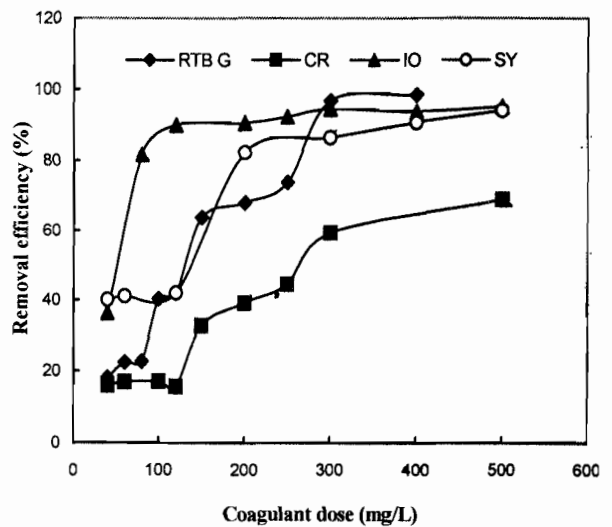
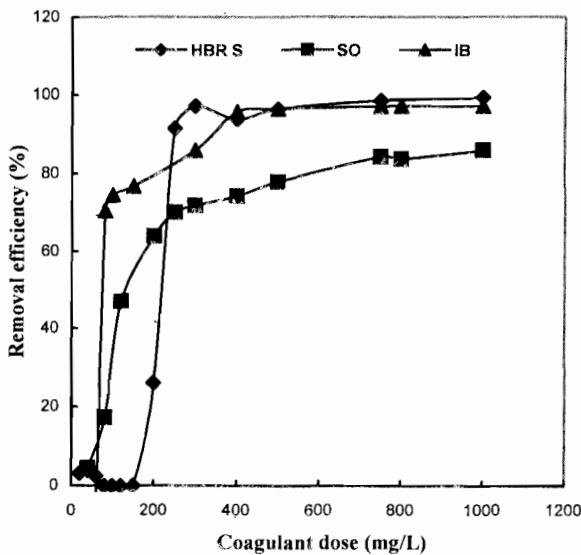
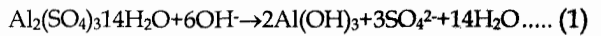


Fig. 1. Effect of coagulant dose on percentage of dye removal at fixed dye concentration.

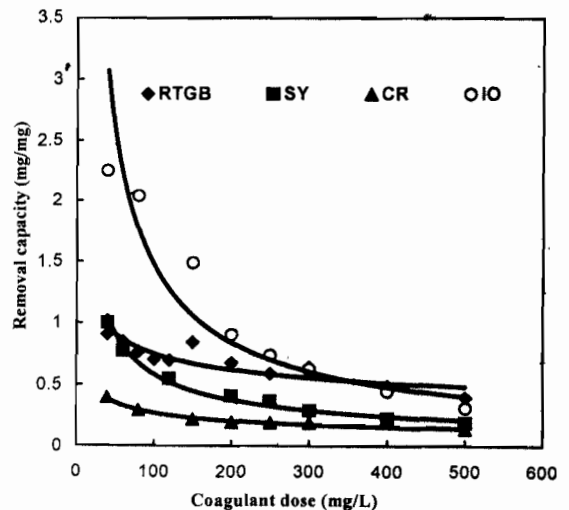
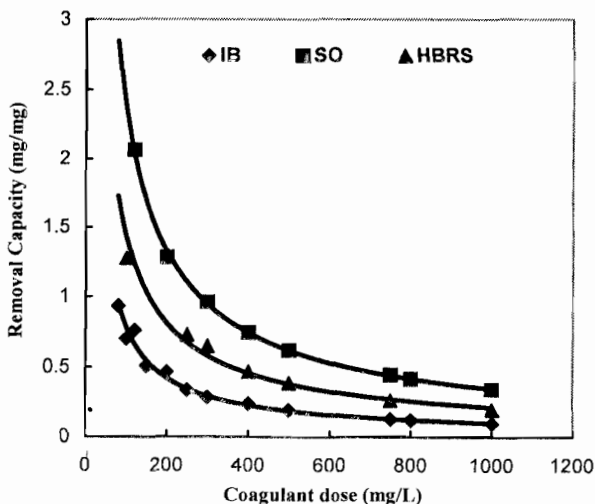


Fig. 2. Effect coagulant dose on removal capacity at fixed dye concentration.

Effect of initial dye concentration

The effect of initial dye concentration at a fixed quantity of the coagulant dosage is shown in Fig. 3. An increase in dye initial concentration was followed by an increase in percentage of dye removal at fixed coagulant dose. This is expected because in surface adsorption reactions the equilibrium concentration of a given solute in the solid phase is proportional to the equilibrium concentration in the liquid phase. Experimental results with mixture of dyes at initial concentration of 500 mg/L (data not shown) indicated that removal efficiency from 70% to 90% can be achieved depending on the type of the dye. This shows the possibility of the application of the proposed method for the treatment of real textile wastewater. From Fig. 4 it is observed that increasing the initial dye concentration increases the removal capacity of the coagulant. The correlation coefficients of the experiments show that there is a strong linear relationship between the initial concentration of the dye solutions and the removal capacity of the coagulant. This result indicates that the method is suitable for the treatment of high-strength textile effluent in terms of dye concentration.

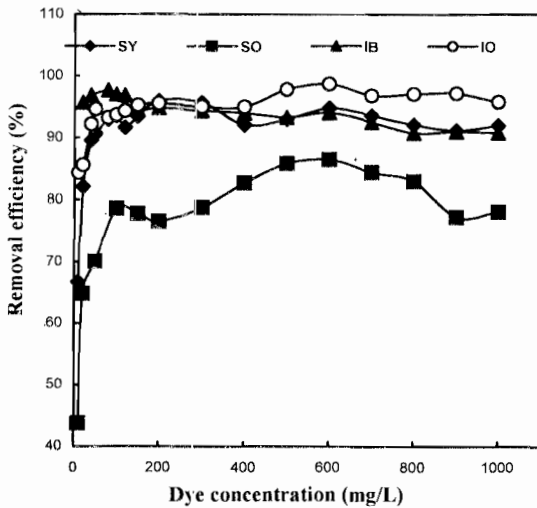


Fig. 3. Effect of initial dye concentration on percentage of dye removal at fixed coagulant dose.

Effect of pH

Figure 5 shows the influence of pH on the removal of dye by coagulation over a pH range of 3 to 12. The experimental results have shown that the coagulation of dyes used in this study has some distinct different characteristics compared to the well-established coagulation behaviour of colloidal particles in surface water. As can be seen, the maximum removal efficiency was observed at pH values around neutrality. The appearance of

the maximum in dye removal with in the pH range around neutrality could be due to the reaction of dyes at the surface of the aluminium hydroxide flocs. The effect of pH might be related to the properties of alum in aqueous solution and the functional groups on the individual dye molecules. Even though many dyes are commercially available, due to the unavailability of the chemical structure in the published literatures, it was not possible to undertake detail investigations on the reaction mechanisms of coagulation in relation to pH. Nonetheless, the experimental results indicate that the removal efficiency is high at pH values where maximum coagulation occurs.

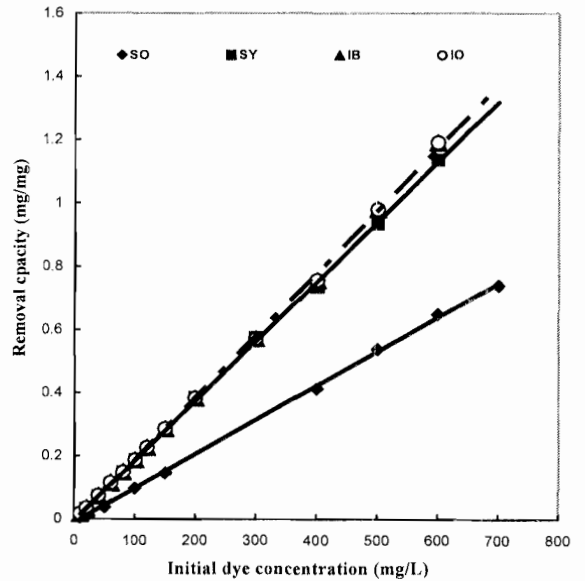


Fig. 4. Effect of initial dye concentration on dye removal capacity at fixed coagulant dose.

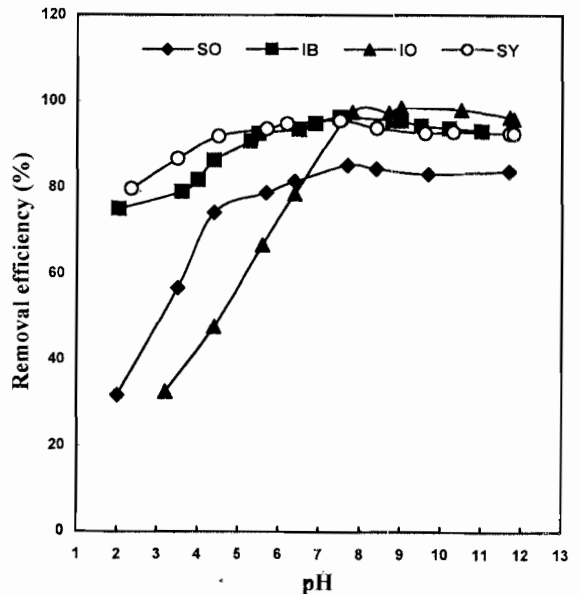


Fig. 5. Effect of pH on percentage dye removal efficiency.

When a metal salt $Al_2(SO_4)_3$ is used as a coagulant in concentrations sufficiently high to cause precipitation of a metal hydroxide (*i.e.*, $Al(OH)_3$ as shown in equation (1)), dye may interact with the amorphous aluminium hydroxide solids and carbonates of calcium and magnesium during the mixing process and settle with the precipitate. Similar phenomena were observed in other studies dealing with coagulation of humic acids and dissolved organic matter by aluminium sulfate (Semmens and Field, 1980; Duan *et al.*, 2002). As can be seen from Fig. 5, the removal efficiency is relatively high at $pH > 9.0$, which is possibly due to the combined effect of adsorption on to aluminium hydroxide and carbonates of calcium and magnesium. Other studies dealing with the removal of fulvic acids by lime addition (Liao and Randtke, 1985) reported that the proposed mechanism was co-precipitation in which the organic species adsorb on to calcium carbonate.

Reaction rates

The kinetics of two representative dyes, IB and IO, was examined. Figure 6 shows the time course change of the concentration of two dyes under consideration for kinetic studies. From the possible alternatives of the kinetics models the result of the experiment show that the reaction of the studied dyes follows pseudo first order kinetics, which is depicted by the following equation (2):

$$C = C_0 e^{-kt} \dots\dots\dots (2)$$

where C_0 is the initial dye concentration, C is the concentration of dye at any time t , k is the pseudo first-order reaction rate constant, and t is the reaction time.

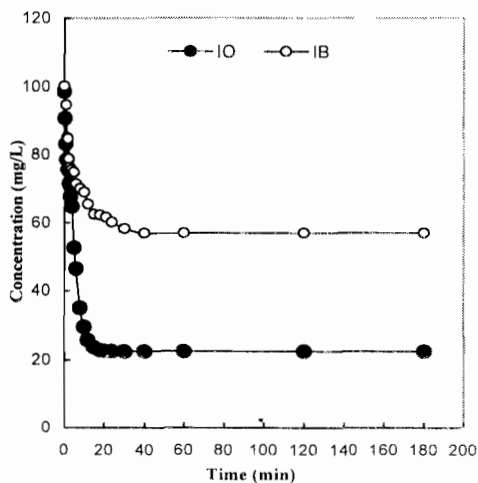


Fig. 6. Change in dye concentration as a function of time.

A plot of $\ln C$ against reaction time should give a straight line with slope k (Fig. 7). From the values of the rate constants $k_1=0.055 \text{ min}^{-1}$ for IB and $k_2=0.114 \text{ min}^{-1}$ for IO it can be observed that the reaction is relatively fast. This indicates the possibilities of applying coagulation for high rate continuous treatment.

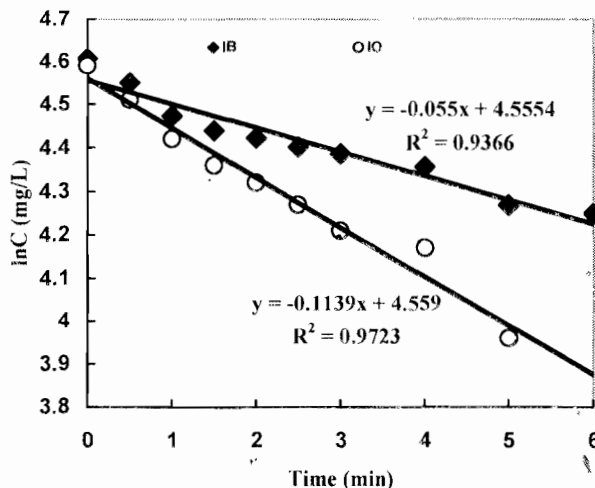


Fig. 7. Kinetics of coagulation and concentration versus time relationship.

CONCLUSION

The use of alum and lime as coagulant allowed a significant reduction in the concentration of different dyes from aqueous solution. Batch experiments using simulated textile dye solutions indicated that operating parameters such as contact time, alum-lime weight ratio, coagulant dosage, dye's initial concentration, and pH influence on the performance of dye removal. Under laboratory conditions, the study showed that less than 1 g/L of alum is quite sufficient to remove nearly or above 90% of the dye. This percentage of dye removal was effective for concentration ranges between 20 and 1000 mg/L of simulated textile wastewater. The highest percentage of the dye removal was observed at pH around neutrality for all the dyes indicating that addition of lime is indispensable to maintain efficient floc formation. Most of the reactions are completed in less than 20 minutes. The reaction kinetics of Indantrene Brown and Imperon Orange was observed to obey pseudo first order kinetics, with rate constants $k_1= 0.055 \text{ min}^{-1}$ and $k_2= 0.114 \text{ min}^{-1}$ for Indantrene Brown and Imperon Orange, respectively. It was found that alum combined with lime is suitable material for removing dye in wastewater.

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