Effectiveness of Aluminum Chlorohydrate (ACH) for Decolorization of Silk Dyebath Effluents

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Effectiveness of Aluminum Chlorohydrate (ACH) for Decolorization of Silk Dyebath Effluents

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ABSTRACT: The present study focuses on the decolorization of real silk dyebath effluents using analytical-grade magnesium chloride (MCl) with and without guar gum (GG) as a coagulant aid and industrial-grade polyaluminium chloride (PACl) and aluminum chlorohydrate (ACH) as coagulants. A higher decolorization efficiency of 97% at a very high dosage of 1800 mg L^{-1} was observed for MCl. However, considering the purity, dosage, decolorization efficiency, and quantity and quality of sludge production, ACH was found to be the best coagulant, giving 91% decolorization efficiency at a dose of just 100 mg L^{-1}. To achieve this high degree of decolorization efficiency, the dosage for PACl was found to be approximately 50% higher than that of ACH. Effective decolorization (>90%) at a very low dosage (100 mg L^{-1}) and minimal quantity of sludge production by ACH demonstrate that ACH is an effective coagulant for decolorization of silk dyebath effluents.

1. INTRODUCTION

Wastewaters from the dyeing industries are one the major sources of surface and groundwater pollution. The textile industry is also one of the largest consumers of potable water and chemical additives. The unused chemical additives are discharged from various steps of textile processing in the form of wastewater. The dyeing and rinsing stages are significant sources of wastewater with complex characteristics such as deep color due to the presence of residual dyes, high turbidity, high pH, large amounts of suspended solids (SS), and high chemical oxygen demand (COD). The presence of textile dyes at very low concentrations is highly visible because of their active chromospheres, and hence, the discharge of these effluents not only renders the recipient water bodies aesthetically objectionable, but also hampers the photosynthetic process, disturbs the ecosystem, and can introduce carcinogenic and detrimental materials into the environment.[1,2] Therefore, it is of utmost importance to treat these types of wastewaters up to the legal and aesthetic standards before they are discharged into the environment.

Despite large numbers of well-established conventional textile wastewater treatment technologies involving biological, physicochemical and/or advanced oxidation processes, there is no single and economically viable option, and generally, two or more methods have to be combined to produce the desired degree of treatment. Biological processes are generally inexpensive, simple, and environmentally friendly; they can be used effectively to remove biodegradable organics but are less effective for the removal of color because of the less biodegradable nature of textile dyes. Almost all advanced oxidation processes are associated with high costs of operation and the possible production of toxic byproducts. The main advantage of chemical coagulation and flocculation is that the decolorization of wastewater takes place by the physical removal of the dye molecules from the textile wastewaters and not by the partial decomposition of the dyes. The partial decomposition of dyes can lead to an even more potentially harmful and carcinogenic aromatic compounds,[3] that are resistant to degradation even under aerobic conditions.[4,5] Sludge production is a major limitation for chemical treatments. However, the selection of a suitable coagulant and the feasibility of sludge disposal make coagulation and flocculation one of the most appropriate technologies for the treatment of textile effluents.

A number of studies have been carried out for the treatment of synthetic dye wastewater using magnesium- and aluminum-based hydrated and prehydrolyzed coagulants. In addition to these metallic coagulants, the effectiveness of natural coagulant such as guar gum (GG) for the treatment of dye solutions has also been investigated in the recent years.[6−8] However, very limited studies have been performed on the effectiveness of these coagulants for the treatment of real dyebath effluents and the quantitative analysis of sludge production. Aluminum chlorohydrate (ACH) is generally used in deodorants and antiperspirants, and belongs to the same group of prehydrolyzed polyaluminium coagulants as polyaluminium chloride (PACl). However, ACH is more hydrated and has a greater percentage alumina content than PACl. To the best of our knowledge, no study has been reported to evaluate the effectiveness of prehydrolyzed aluminum-based coagulants such as ACH for the treatment of real silk dyebath wastewater. Therefore, the present study was focused on investigating , for the first time, the effectiveness of ACH in comparison to PACl, magnesium chloride (MCl) alone, and also MCl in combination with GG for the decolorization and COD reduction of real silk dyebath effluents. The study was focused on evaluating the comparative effects of pH and coagulant dosage on color removal efficiency, along with the quantity of sludge production at optimum conditions.

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2. EXPERIMENTAL SECTION

2.1. Reagents and Materials. Analytical-grade MCl [$MgCl_2 \cdot 6H_2O$, purity 99.99% w/w], industrial-grade PACl [$Al_2(OH)_3Cl_3$, purity 30% w/w] and industrial-grade ACH [$Al_2(OH)_5Cl$, purity 30% w/w] were used as coagulants. Although both PACl and ACH are prehydrated aluminum salts belonging to the class of polyaluminium coagulants, in practice, ACH is more hydrated and has a higher alumina content than PACl. Analytical-grade GG was used as a coagulant aid, and 1.0 M H_2SO_4 and NaOH were used to adjust the pH.

2.2. Wastewater. Real silk dyebath effluents were obtained from a local textile hand mill situated in Khurda district of Odisha, India. Major characteristics of dyebath effluents are listed in Table 1.

Table 1. Characteristics of Silk Dyebath Effluents

parameter	value	parameter	value
color	deep brown	EC (mS cm^{-1})	20.22 ± 2
pH	12.10–12.51	turbidity (NTU)	60 ± 10
COD (mg L^{-1})	847 ± 20	absorbance at 425 nm	2.2822
TSS (mg L^{-1})	372 ± 50	absorbance at 475 nm	2.0155
TDS (mg L^{-1})	9780 ± 100		

Absorbance measurements of effluents before and after treatment were carried out after filtration of the solution through Whatman No. 5 filter paper and subsequent neutralization to pH 7.0. The characteristic wavelengths of the dyebath wastewater were determined by running a scan of the neutralized (pH 7.0) effluents on a UV–vis spectrophotometer. During this scan, two distinct peaks at 425 and 475 nm were observed. Therefore, the maximum absorbance wavelengths (λ_{max}) for this wastewater were considered to be 425 and 475 nm. The color content of the wastewater was determined by taking the sum of the absorbencies measured at 425 and 475 nm. The percentage color removal was determined by comparing the absorbance values for the wastewater after treatment to the absorbance values of the wastewater before treatment. Tap water served as a reference.

2.3. Coagulation and Flocculation Test Procedures. The optimum pH value and coagulant dosage required for efficient color removal were determined by performing a jar test. One-liter beakers containing 500 mL of wastewater were used for the coagulation experiments. H_2SO_4 (1.0 M) was added to each beaker for pH adjustment. Chemical coagulant was added and mixed for 3 min under rapid stirring at 80 rpm. The solution was then mixed at slow flocculation for 15 min at 30 rpm. After sedimentation for 30 min, supernatants from the top of the beaker were removed for the analysis. All runs were performed in duplicate, and the results are presented as the averages of two values each.

2.4. Analyses. Color measurement was carried out after filtration of the supernatant through Whatman No. 5 filter paper. The pH of the filtrate was adjusted to about neutral before the absorbance was measured. COD was analyzed according to the closed reflux colorimetric method after digestion of the samples in a COD reactor (model DRB 200, Hach Company, Loveland, CO), and then absorbance measurements were carried out on a COD spectrophotometer at 600 nm (model DR 2800, Hach Company, Loveland, CO). The percentage color reduction efficiency was determined using the equation

$$\text{reduction efficiency (\%)} = [(A_b - A_t)/A_b] \times 100\% \qquad (1)$$

where A_b and A_t are the absorbencies of the solution before treatment and after treatment of silk dye bath effluents, respectively.

Sludge production (in terms of settled sludge volume) was also measured at optimized conditions for all coagulants. All of the methods used for the analysis of wastewater characteristics were according to the Standard Methods[9] and were performed at room temperature (25 ± 5 °C).

3. RESULTS AND DISCUSSION

3.1. Effect of pH on Chemical Coagulation of Dyebath Effluents. Coagulation/flocculation is a pH-dependent process, as pH plays a very important role in the formation of perceptible hydroxide and coagulation occurs within a specific pH range for each coagulant. Therefore, experiments were designed to determine the optimum pH for the coagulation that resulted in maximum decolorization and COD reduction. The effect of pH on the treatment efficiency was examined at a constant coagulant dose and varying pH (4, 6, 8, 10, 11, 12, and 12.5) for all coagulants. As pH affects the molecular structure of dyes, the absorbance of the solutions changed at varying pH conditions. Therefore, the pH values of the untreated and treated wastewaters were adjusted to neutral before the absorbance was measured to evaluate the percentage color removal.

No significant increasing or decreasing trends in treatment efficiency were observed during variation of the pH when PACl (100 mg L^{-1}) or ACH (50 mg L^{-1}) was used as the coagulant. However, a continuous increase in the treatment efficiency was observed as the pH was increased from 4.0 to 12.0 when MCl (800 mg L^{-1}) was used as the coagulant (Figure 1). Beyond pH 12, slight decreases in the decolorization and COD reduction were observed. The optimum pH for MCl was observed to be as high as 12.0 in the alkaline region, whereas for PACl and ACH, the optimum values were 6 and 4, respectively (Figure 1a,b). The results of the present study are in good agreement with the findings reported by Gao et al.,[10] who investigated the effects of magnesium chloride dosage on color removal efficiency of textile wastewater containing various types of dyes. The enhancement in coagulation efficiency with increasing pH can be attributed to the fact that metal ions are easily hydrolyzed under alkaline conditions and form perceptible hydroxides. The structure of perceptible hydroxides provides a large surface area and positive superficial charges, which removes colloidal impurities through adsorption and charge neutralization.[11] However, aluminum-based coagulants generally form $Al(OH)_3$, $Al(OH)_2^{+}$, and $Al(OH)^{2+}$, as well as the dominant polymeric species Al_{13}^{7+}, during hydrolysis at pH in the acidic range (pH < 6), which remove the dyes through adsorption and charge-neutralization mechanisms.[12] At around neutral pH (pH 6–8), aluminum ions have limited solubility because of the precipitation of soluble hydroxides, which leads to the possibility of sweep flocculation. Therefore, at pH values of 6–8, application of aluminum salts effectively removes impurities by enmeshing them in the growing precipitates.[12,13] The low optimum pH conditions for PACl and ACH can be attributed to these mechanisms. Therefore, it can be said that the principal removal mechanism for ACH is adsorption and charge neutralization, whereas for PACl, it is sweep flocculation.

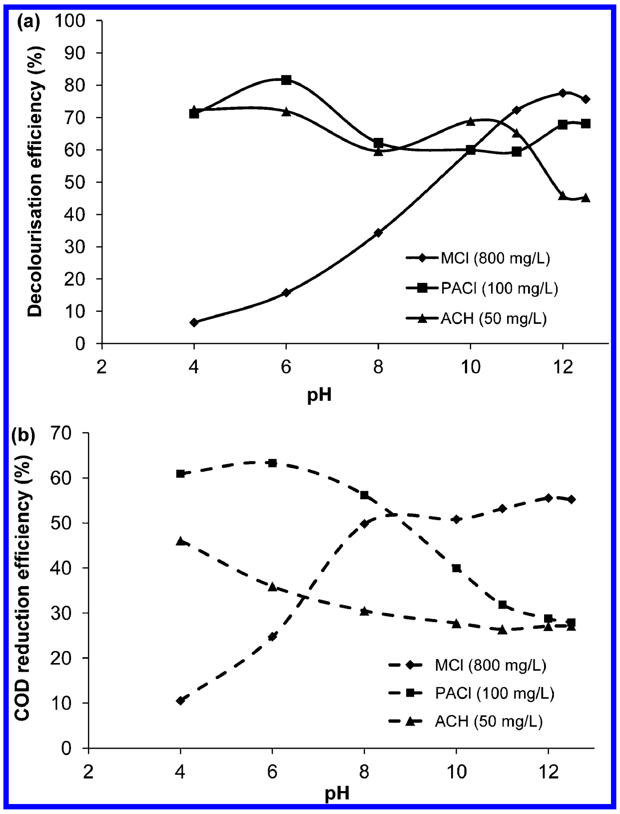

Figure 1. Effect of pH on treatment efficiency of different coagulants for real silk dyebath effluents: (a) decolorization efficiency, (b) COD reduction efficiency.

Although almost the same high decolorization efficiencies were observed for ACH at pH values of 4 and 10 (Figure 1a), the flocs formed at pH 10 was appeared to be light and fragile and did not settle by gravity for a substantial period of time of more than 2 h. However, the sludge thus formed could be removed very easily by filtration using filter paper. On the other hand, the sludge formed at pH 4 was bulky and dense and settled very easily by gravity within a 30-min settling time. Therefore, pH 4 was considered to be the optimum pH for ACH.

3.2. Effect of Coagulant Dosage on Chemical Coagulation of Dyebath Effluents. The optimum coagulant dosage for the treatment of dyebath effluents was determined by varying the coagulant dosage and maintaining a constant optimum pH. Treatment efficiency results for various coagulants in terms of color, COD, and turbidity reduction as functions of coagulant dosage are shown in Figure 2.

3.2.1. Treatment Efficiency with MCl. It was observed that the percentage color, COD, and turbidity reductions increased with increasing coagulant dosage. Color, COD, and turbidity reductions of 97.42%, 61.47%, and 98.34%, respectively, were obtained at a very high dosage of 1800 mg L^{-1} MCl. Excellent color removal of close to 97% was also obtained at a slightly lower dosage of 1600 mg L^{-1} MCl (Figure 2a).

3.2.2. Treatment Efficiency with PACl. A maximum of 91% color removal was obtained at a PACl dosage of 200 mg L^{-1}. Excellent COD and turbidity reductions of approximately 82% and 99%, respectively, were obtained at the same dosage of PACl. At 600 mg L^{-1}, PACl produced a maximum 97.45% COD reduction; however, at this dosage, the decolorization efficiency decreased considerably. A decrease in the color

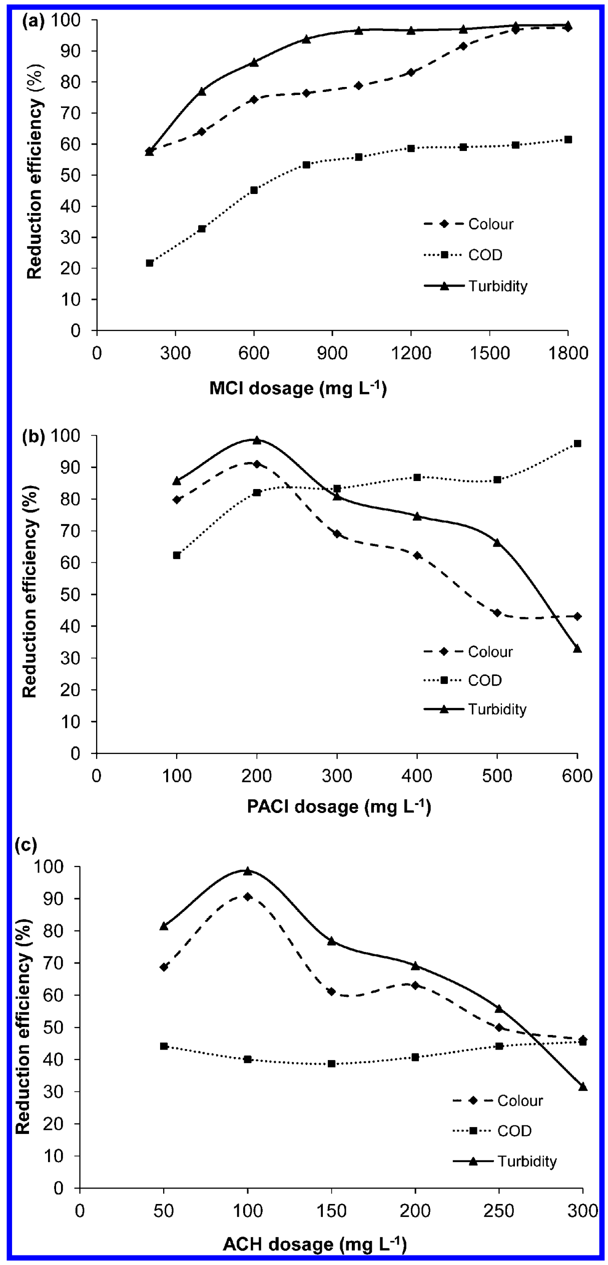

Figure 2. Effect of coagulant dosage on the treatment efficiency of dyebath effluents with (a) MCl, (b) PACl, (c) ACH.

removal efficiency was observed above the optimum dosage of the coagulant (Figure 2b).

3.2.3. Treatment Efficiency with ACH. A similar trend as in the case of the PACl was observed for ACH as well. However, the ACH dosage was found to be almost 50% less than that of the PACl (Figure 2c). A color removal efficiency of maximum 91% was obtained at only 100 mg L^{-1} ACH. Also, an excellent turbidity reduction of 99% and a significant COD reduction of 40% were observed at the same dosage of coagulant.

It was observed that the percentage color, COD, and turbidity reductions increased with increasing MCl dosage. No significant improvement in the treatment efficiency was observed even at very high dosage of MCl. The experimental results revealed that MCl effectively removes color from the dyebath effluents at a high dosage and also at very high optimum pH. It was also observed that both the PACl and ACH produce virtually colorless solutions at their optimum dosages. However, the dosage requirement for achieving the same degree of color removal was about 50% less for ACH. The decrease in ACH dosage compared to PACl can be related to the fact that ACH is more hydrated and contains almost 30–50% more alumina than PACl. A continuous decrease in the color removal was observed at higher dosages of PACl and ACH beyond the optimum dosages. Unlike ACH and MCl, PACl was found to give a promising COD reduction efficiency even at lower dosages. A similar finding of excellent COD reduction efficiency by PACl was also reported by Wong et al.[13] for the treatment of synthetic textile wastewater containing reactive and disperse dyes. For PACl, the dominant color removal mechanism is sweep flocculation, and for ACH, it is adsorption and charge neutralization. The difference in COD removal efficiency between PACl and ACH is due to the difference in removal mechanisms. Also, the dosage of PACl was almost twice that of the ACH at a particular decolorization efficiency; therefore, more flocs formed for PACl than for ACH (Figure 4). The higher quantity of formed flocs leads to more entrapment of organics within their higher volume of precipitated hydroxides, resulting in a higher COD removal efficiency than for ACH. The decrease in the color removal at higher dosage can be attributed to the fact that the addition of excess coagulant provides positive charge to the neutralized flocs and simultaneously reactivates the dye molecules, which repel each other and thereby produce a more turbid solution. Similar findings were also observed by Tun et al.[14] and Tan et al.,[15] who reported 65.4% and 88.4% color removals for textile wastewater using PACl and MCl dosages of 800 and 1500 mg L^{-1}, respectively, at corresponding optimum pH values of 7.5 and 11. The high dosage requirement for MCl might be related to the facts that (i) the dosage of MCl is a function of hydroxyl ion concentration, the solubility of magnesium ions in equilibrium with solid $Mg(OH)_2$, and the minimum amount of $Mg(OH)_2$ that must be precipitated;[16] (ii) the charge of magnesium ions is less positive than that of aluminum ions, and (iii) the relative adsorption capacities of magnesium hydroxide flocs and aluminum hydroxide flocs are different. Further, the coagulant dosage requirement also depends on the complexity (such as dye content and types) of wastewater being treated. Generally, for all types of textile wastewater, MCl produces considerable decolorization at higher dosages and at very alkaline pH values.

3.2.4. Treatment Efficiency of MCl in Combination with GG. Analytical-grade MCl is much more expensive than industrial-grade PACl and ACH. As it was observed that a very high dosage of MCl was required to obtain the desired degree of treatment efficiency, the incorporation of a coagulant aid was planned to reduce the MCl dosage. The flocculating properties of plant-based natural polysaccharides such as GG, seed extracts, tannin, and cactus have been investigated by various researchers.[6,17−19] However, recent studies have revealed that application of GG as a coagulant or coagulant aid is highly effective for the treatment of textile wastewaters.[7,20] Therefore, it was decided to use GG as a natural coagulant aid in combination with MCl. As the required optimum dosages of PACl and ACH alone were much lower, use of GG as a coagulant aid with PACl and ACH was not performed in this study. The incorporation of GG with MCl was aimed at evaluating the effect of GG on reducing the high MCl dosage. The application of GG was not planned to evaluate its comparative effects as a coagulant aid with all of the coagulants used in this study.

Different ratios of GG and MCl were used to maintain a constant optimized coagulant mixture dosage of 1600 mg L^{-1}. The results are shown in Figure 3. It can be observed that the

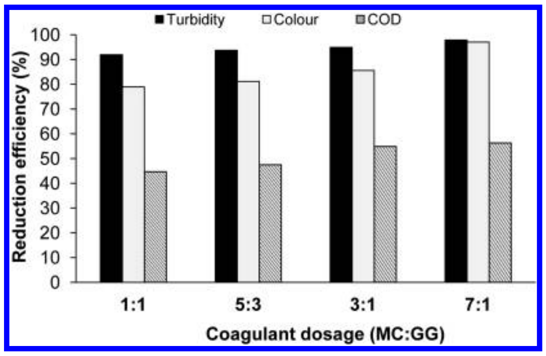

Figure 3. Treatment efficiency of dyebath effluents using MCl aided with GG at different ratios.

color removal efficiency decreased with increasing concentration of GG, or equivalently, the color removal efficiency increased at increasing concentration of MCl. The increase in decolorization efficiency with increasing dosage of metallic coagulant might be related to its coagulation mechanism. MCl is prehydrolyzed and substantially charged, which removes dye molecules predominantly by charge neutralization and adsorption,[21] whereas GG is partially charged and removes dye molecules mainly by adsorption. A marginal improvement in color removal efficiency was observed at the MCl/GG ratio of 7:1, as compared to the case when MCl alone at 1600 mg L^{-1} was used as the coagulant. The experimental results revealed that, for the same degree of treatment efficiency, use of a natural coagulant aid reduced the metallic MCl dosage only marginally.

Excellent decolorization efficiency was observed in the case of MCl as a coagulant at very high dosage. Further, addition of GG with MCl was observed to produce only marginal improvements in decolorization efficiency. From all of the results, it can be concluded that ACH is more effective than MCl, MCl with GG, and PACl for decolorization at very low dosage. However, PACl is most effective for COD reduction among all of the investigated coagulants.

3.4. Sludge Production. The quantity and quality of sludge production during coagulation/flocculation depend on the type of coagulant used and the operating conditions.[22] Therefore, sludge production was measured at the optimized pH and coagulant dosage for all coagulants. It was measured based on the volume occupied by the flocs in 500 mL of sample volume after the solutions had been allowed to settle for 1 h in an Imhoff cone. A minimum of 25 mL of settled sludge per 500 mL of sample was produced for the treatment of real silk dyebath effluents at optimized conditions using ACH, which was significantly lower than the amounts of sludge produced by MCl, PACl, and MCl/GG (7:1), for which 40, 35, and 50 mL, respectively, of settled sludge were observed per 500 mL of sample (Figure 4). The higher amount of sludge produced in the case of MCl as the coagulant can be related to the formation of fragile hydroxide flocs and their poor settling behavior. Addition of GG with MCl considerably increased the sludge production and, hence, would not be recommended as a better coagulant combination. The significant reduction in sludge production using ACH as the coagulant can be explained by the fact that ACH shows very high adsorption for the used

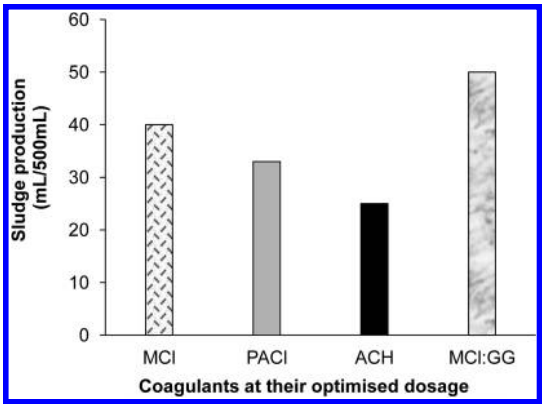

Figure 4. Sludge production at optimized conditions for various coagulants.

dyes and other chemical additives and produces more compact sludge than PACl, MCl, and MCl/GG (7:1). As industrial-grade coagulants, PACl and ACH both produced heavy and bulky sludge that could easily be dewatered.

3.5. Analysis of Spectra and Color Removal Mechanisms of the Coagulants.

Spectral analysis and investigation of color removal mechanisms were performed for the real silk dyebath effluents. Spectral analyses were performed on untreated and treated real dyebath effluents at the optimized pH and coagulant dosage for all of the combinations. However, as no significant differences were observed in the spectra of dyebath effluents treated with all of the coagulants, only the spectra of silk dyebath effluents treated with ACH are presented here as a representative to discuss the color removal mechanisms (Figure 2c). The results are shown as spectrum a for untreated wastewater and spectrum c for treated wastewater in Figure 5. From Figure 5, it can be noticed that there are no distinctive peaks in the spectrum of the treated wastewater (spectrum c) in the visible wavelength range from 400 to 700 nm. This indicates that the dyes from the wastewater were transferred into the hydroxide precipitates during coagulation–flocculation.

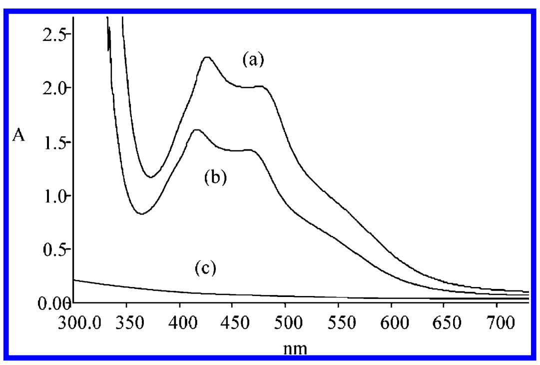

Figure 5. Spectra of silk dyebath effluents when treated at optimized conditions using ACH.

Further, these precipitates were filtered and acidified to neutral pH to dissolve them in solution. The filtrate was then analyzed by spectrophotometer, and the results are shown as spectrum b in Figure 5. It can be seen that the shapes of spectra a and b are very similar and the peaks occur at the same wavelengths in the two cases. Hence, it can be said that the removal of color by coagulation was merely a physical phenomenon. The color of the neutralized solution was the same as that of the untreated wastewater but the absorbance was lower. This is because the complete dissolution of the precipitates into the solution by acidification to neutral pH is almost impossible. There was no chemical change in the dye molecules before and after the coagulation, as both the peaks were found at the same wavelengths for spectra a and b.

On the basis of the findings of this study, the color removal mechanism of MCl can be described as follows: (i) The metal ions from the coagulants are converted into their insoluble metal hydroxides at pH values greater than 11.0. (ii) These complex and insoluble structures of hydroxides provide a large adsorptive surface area and positive superficial charge, leading to adsorption and charge neutralization. Therefore, it can be said that the removal of color using MCl as the coagulant takes place predominantly by adsorption and charge neutralization. Also, based on the optimum pH values discussed previously, the principal color removal mechanisms can be considered as sweep flocculation and adsorption/charge neutralization for PACl and ACH, respectively.

4. CONCLUSIONS

Treatability studies of real silk dyebath effluents were successfully carried out using the selected coagulants. The treatment efficiencies of the coagulants depend significantly on the pH of the wastewater. Aluminum salts such as ACH and PACl were found to be very effective in the decolorization of dyebath effluents at a very low coagulant dosages. Although the decolorization efficiency was observed to be greater for MCl, considering both the dosage and the decolorization efficiency, ACH was found to be superior to both MCl and PACl. Also, the increase in decolorization efficiency (~6%) obtained by using 99.99% pure MCl was marginal compared to that obtained with 30% pure industrial-grade ACH or PACl. A maximum 91% decolorization efficiency was observed for both ACH and PACl. However, the ACH dosage was considerably reduced by 50% compared to the PACl dosage to achieve the same degree of decolorization. Also, the lowest production of easily dewaterable sludge and the considerable color removal at a very low dosage make ACH an attractive coagulant for the decolorization of silk dyebath effluents. Hence, ACH can be recommended as an efficient coagulant for the decolorization of dyeing wastewaters. However, secondary treatment is required to take care of the rest of the dissolved organic matters to meet the safe discharge standards set by the environmental pollution authorities of different countries.

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Notes

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■ ABBREVIATIONS

ACH = aluminum chlorohydrate
COD = chemical oxygen demand
EC = electrical conductivity
GG = guar gum
MCl = magnesium chloride
PACl = polyaluminium chloride
SS = suspended solids
TDS = total dissolved solids
TSS = total suspended solids

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