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Applicability of a new pre-hydrated industrial grade polyaluminium salt for the decolourisation of textile wastewater

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ABSTRACT

This study was conducted to assess the decolourisation and chemical oxygen demand (COD) reduction efficiency of a novel pre-hydrated aluminium salt as aluminium chlorohydrate (ACH) for the treatment of textile wastewater using coagulation/flocculation technology. Though, ACH belongs to the same group of polyaluminium salts, it is significantly different from polyaluminium chloride (PACl) in terms of the degree of hydration and alumina content and was used for the first time as a coagulant for the decolourisation of synthetic as well as real textile wastewater. The experimental results revealed that ACH is highly effective for the treatment of synthetic textile wastewater producing more than 99% of colour removal and 45% of COD reduction efficiency at a very low dosage of 200 $mg\,L^{-1}$. The decolourisation of real textile wastewater was found to be very much in line with the results obtained for the treatment of synthetic textile wastewater. Superior decolourisation efficiency at a very low dosage as compared to that of the pre-established studies for real textile wastewater and least volume of easily dewatered sludge production makes ACH a novel and very promising coagulant for the treatment of industrial textile wastewater.

Keywords: Aluminium chlorohydrate; Coagulation; Decolourisation; Textile wastewater

1. Introduction

Textile industries are one of the most chemically intensive industries on the earth and one of the major concerns for a green environment. It generates a large volume of wastewaters at different steps of textile processing. The major characteristics of textile wastewater from dyeing and finishing units are high pH, strong colour, high organic matter, high turbidity, large amount of suspended solids (SS), and significant amounts of salts. Various azo and anthraquinone dyes are increasingly being used in the textile industries for the dyeing of different types of fabrics. The residual dyes having very complex structures, are responsible for the colour of wastewater and can be extremely detrimental if it is discharged into the environment without adequate treatments. The presence of residual dyes also disturbs the aquatic life by obstructing the penetration of light and oxygen transfer [1]. The direct and indirect effects of such type of wastewaters to the environment have already been described by Verma et al. [2].

Treatment of textile wastewaters in terms of colour removal mainly involves physicochemical, chemical and biological processes, as well as some of the new

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emerging techniques like sonochemical and/or advanced oxidation processes. Each of these technologies has some advantages as well as some limitations and drawbacks in their application. Moreover, these wastewaters are very difficult to decolourise by conventional biological processes due to the recalcitrant nature of most of the textile dyes. Among the currently used physicochemical treatment processes, coagulation/flocculation has received a considerable attention because of their high colour removal efficiency and ease of operation. However, coagulation/flocculation alone cannot be proved to be effective in removing dissolved organics as per the standard environmental discharge limits set by the environmental pollution authorities of different countries. Therefore, coagulation/flocculation can be used as a primary treatment for the removal of colour prior to biological treatment [3] for the removal of dissolved organics. 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. The main advantage of chemical coagulation and flocculation is that the decolourisation 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 [4,5] that are resistant to degradation even under aerobic conditions.

During the recent years, various aluminium, iron and magnesium based hydrated as well pre-hydrolysed metallic salts have been used to decolourise the textile effluents. In addition to these metallic coagulants, the effectiveness of natural coagulants for the treatment of dye wastewaters has also been investigated in the recent years. However, very limited studies have been performed on the effectiveness of these coagulants for the treatment of real textile wastewaters and the quantitative analysis of sludge production.

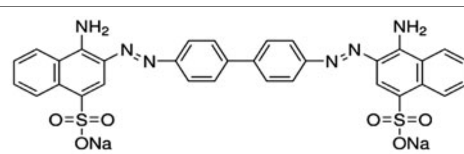
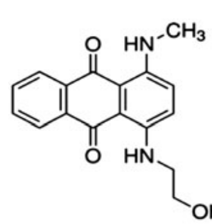
In this study, an attempt has been made to investigate the effectiveness of a novel aluminium- based industrial grade aluminium chlorohydrate (ACH) as a coagulant for the treatment of textile wastewater containing diazo and anthraquinone dyes along with the other chemical constituents that are normally present in textile effluents. ACH is an industrial grade chemical that is generally used by the industries in deodorants and antiperspirants production and belongs to the same group of pre-hydrated polyaluminium salts such as polyaluminium chloride (PACl). The effectiveness of ACH has also been verified for decolourising real silk dyeing effluent. The study has been focused on evaluating the comparative effects of pH and coagulant dosage for the colour and COD reduction efficiency along with the amount of sludge production.

2. Materials and methods

2.1. Materials

Industrial-grade ACH [$Al_2Cl(OH)_5$, purity 30% w/w], was used as coagulant. ACH is pre-hydrated aluminium salt belongs to the group of polyaluminium coagulants, more hydrated and having more alumina content than PACl. 1.0 M H_2SO_4 and 1.0 N NaOH were used to adjust the desired pH. The other chemical additives used in the preparation of the synthetic wastewater were of analytical-grade. The dyes were

Table 1
Dye characteristics

Name	Type	Molecular structure	Maximum absorption wavelength
Congo red (CR)	Diazo		502 nm
Disperse blue 3 (DB3)	Anthra-quinone		638 nm

procured from Sigma Chemicals. The name, type, molecular structure and the wavelength of maximum absorbance the dyes selected for this study are shown in Table 1.

2.2. Textile wastewaters

Synthetic textile wastewater was prepared as per the reported chemical constituents of real textile wastewater [6,7], with a total dye concentration of 200 mg L^{-1}. The total dye concentration was prepared using either a single dye, or mixing two dyes in equal ratio along with the various chemical additives such as starch, acetic acid, sucrose, sodium carbonate, sodium hydroxide, sulphuric acid, detergent, and sodium chloride, which are used at different steps of textile processing. Wastewaters were prepared using two commercial dyes namely, congo red (CR) and disperse blue 3 (DB3) in the tap water. The characteristic wavelength for each of the simulated textile wastewaters was determined by running a scan of the dye solution on a UV–vis spectrophotometer. The maximum absorbance wavelength (λ_{max}) for the synthetic wastewater has been found as 502 and 638 nm respectively for CR, and DB3, and was used to measure the absorbance of respective treated wastewater. The colour content of the wastewater containing mixture of dyes was determined by taking the sum of the absorbances measured at 502 and 638 nm [8].

The real silk dyeing wastewater was obtained from the dyeing unit of a local hand mill situated in Khurda district of Odisha, India. Two distinctive peaks at 425 and 475 nm were obtained during the scan of this dyeing wastewater. Therefore, the net absorbance in this case was also determined by taking the sum of the absorbances at 425 and 475 nm.

Major characteristics of synthetic textile wastewater and real silk dyeing wastewater are shown in Table 2.

The percentage colour 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 absorbances of the solution before treatment and after treatment of silk dye bath effluents, respectively. Tap water served as a reference.

2.3. Coagulation and flocculation test procedures

The optimum pH and coagulant dosage required for efficient colour removal were determined by a jar test procedure. One-litre beakers containing 500 mL of wastewater were used for the coagulation experiments. Chemical coagulant was added and mixed for 3 min under rapid mixing condition 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 taken for the analysis. All the experiments were performed in duplicates, and the averages are reported as the observed values.

2.4. Analyses

The colour measurement was carried out after filtration of supernatant through Whatman No. 42 filter paper. The pH of filtrate was adjusted to about neutral before the absorbance was measured. The COD was analysed 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 measurement was carried out by COD spectrophotometer at 600 nm (model DR 2800, HACH Company, Loveland, CO).

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

3. Results and discussion

3.1. Determination of optimum pH for chemical coagulation of textile wastewaters

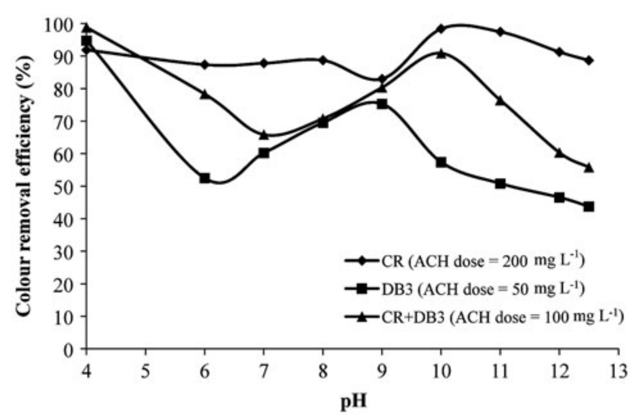
The subsequent experiments were designed to determine the optimum pH for the coagulation that allowed for maximum decolourisation. The effect of pH on the treatment efficiency was examined at a constant

Table 2
Major characteristics of textile wastewater

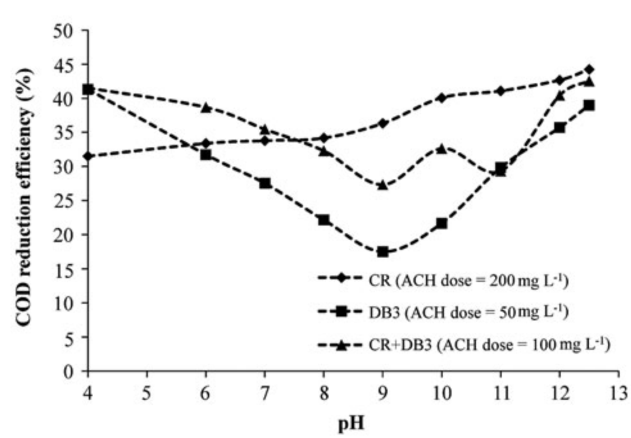
Type of wastewater	COD (mg L^{-1})	pH	Absorbance at their λ_{max}	Turbidity (NTU)
Synthetic wastewater containing CR	1965 ± 40	10.4 ± 0.2	0.6313	86 ± 5
Synthetic wastewater containing DB3	1965 ± 40	10.4 ± 0.2	0.7945	115 ± 8
Synthetic wastewater containing CR + DB3	1965 ± 40	10.4 ± 0.2	1.4252	104 ± 8
Real silk dyeing wastewater	850 ± 20	12.3 ± 0.2	4.2977	60 ± 10

coagulant dose and varying pH (4, 6, 7, 8, 9, 10, 11, 12 and 12.5) for all the combinations. As pH affects the molecular structure of dyes, the absorbance of the solution changes 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 colour removal.

No distinctive and continuous trends in the treatment efficiency were observed during variation of pH when ACH was used as the coagulant for different types of wastewaters (Fig. 1(a) and (b)). For the wastewater containing CR, maximum decolourisation and COD removal efficiency of 98 and 40%, respectively, were obtained at pH 10 using ACH dosage of 200 mg L^{-1}. For the wastewater containing DB3, maximum decolourisation and COD removal efficiency of 95 and 41%, respectively, were obtained at pH 4 using ACH dosage of 50 mg L^{-1}. A maximum of 98% colour removal and 41% COD reduction were achieved at pH 4 using ACH dosage of 100 mg L^{-1} for the treatment of synthetic textile wastewater containing CR and DB3 together.



(a)



(b)

Fig. 1. Effects of pH on the treatment efficiency for different types of synthetic textile wastewater containing CR, DB3, and CR + DB3 (a) decolourisation efficiency and (b) COD reduction efficiency.

The treatment efficiency of a coagulant is strongly related to the type of hydroxides formed and their solubility. ACH generally forms $Al(OH)_3$, $Al(OH)_2^+$ and $Al(OH)^{2+}$, including the dominant polymeric species as Al_{13}^{7+}, during hydrolysis at lower pH [10], which remove dyes through adsorption and charge-neutralisation mechanisms. Beyond pH 7 with increasing alkaline conditions, ACH starts forming $Al(OH)_4^-$ with a growing concentration up to pH 10, as a result, the destabilisation of colloidal particles was not observed and decolourisation efficiency decreased slightly [11,12]. Therefore, at alkaline pH values of 7–10, the application of ACH effectively removes impurities by enmeshing them in the growing amorphous particles [13,5]. The optimum pH conditions for ACH can be attributed to these mechanisms. Therefore, it can be said that the principal removal mechanism of ACH for the wastewater containing DB3 is adsorption and charge neutralisation, whereas for wastewater containing CR, it is sweep flocculation. These findings clearly revealed that optimum pH not only depends upon the type of coagulant but also on the dye type.

3.2. *Effect of ACH dosage on treatment efficiency*

The effect of coagulant dosage on treatment efficiency was investigated by varying the coagulant dosage and maintaining the optimum pH constant. Treatment efficiency in terms of colour, COD and turbidity reduction for the synthetic textile wastewater at different combinations containing CR, DB3 and CR + DB3 are shown in Fig. 2.

ACH in case of synthetic textile wastewater containing CR at the dosage of 250 mg L^{-1} had been observed to produce excellent colour and turbidity removal of above 98 and 97%, respectively. A considerable COD reduction of more than 44% had also been observed at the same dosage of coagulant (Fig. 2a). In case of synthetic textile wastewater containing DB3, above 99% colour and turbidity removal along with 45% COD reduction were obtained using ACH at a dosage of just 175 mg L^{-1} (Fig. 2(b)). The decolourisation and COD reduction efficiency of close to 99 and 45%, respectively, has been achieved at only 200 mg L^{-1} of ACH in case of synthetic textile wastewater containing CR and DB3 together at equal ratio (Fig. 2(c)). The solubility of chemicals is an important factor behind their removal efficiency by means of coagulation/flocculation technology. The removal of less soluble chemical compounds is more than that of the highly soluble chemical compounds by

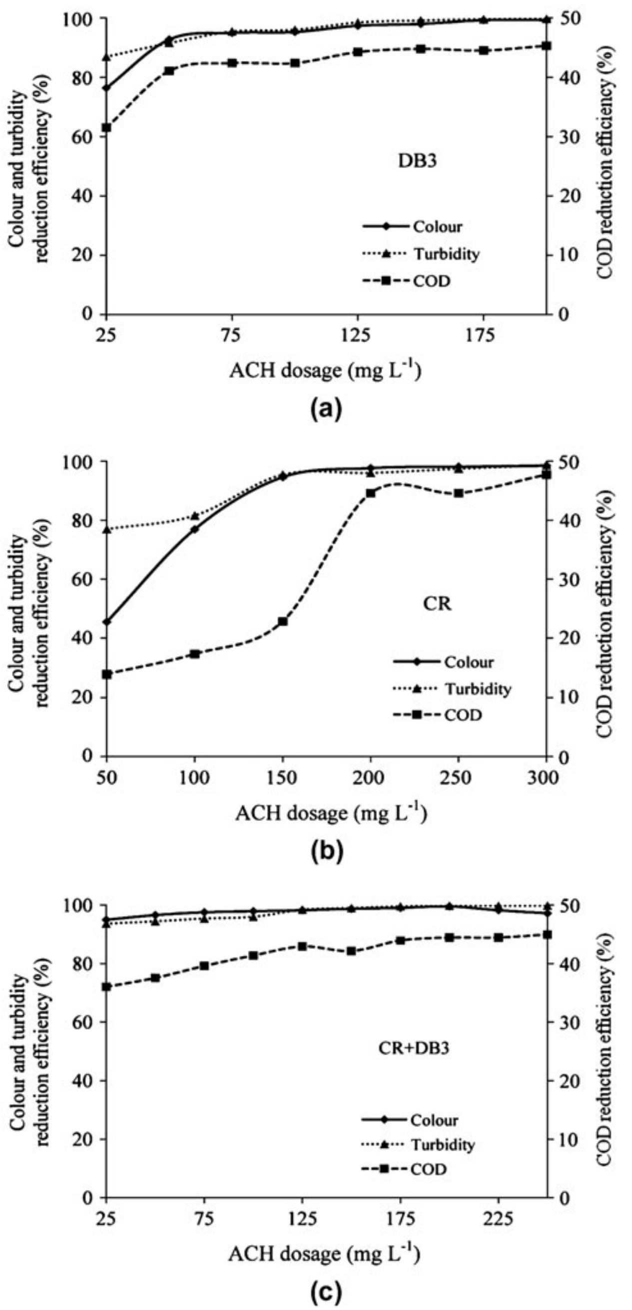


Fig. 2. Effects of coagulant (ACH) dosage on the treatment efficiency for synthetic textile wastewater containing (a) CR, (b) DB3 and (c) CR + DB3.

chemical coagulation/flocculation [14]. The observed COD removal efficiency was considerably less than that of the decolourisation efficiency for all the experiments. This is due to the fact that the solubility of the organics responsible for dissolved COD is much higher than that of the dyes in water.

From Fig. 2 and it has been observed that at optimum pHs, colour and COD removal efficiency increases with the increase in dosage of coagulant. However, there has been no further improvement in the removal efficiency after attaining a maximum at a certain coagulant dose, termed as optimum coagulant dosage. The experimental results revealed that, ACH is highly effective for the removal of both diazo and anthraquinone dyes at a very low dosage. However, the much lesser dosage is required for almost complete decolourisation of wastewater containing anthraquinone (disperse) dye. This can be related to the less soluble nature of disperse dyes. The result of this study is in good agreement of the finding reported by Gao et al. [15], who investigated the effect of magnesium chloride dosage on colour removal efficiency of textile wastewater containing various types of dyes. However, for achieving the same degree of decolourisation, the required dosage of magnesium chloride (99% pure) is almost 2–3 times higher than that of the ACH (30% pure) dosage, as observed in this study. Similar studies with PACl as coagulant has also been reported to consume very high dosage of the order 600 mg L^{-1}[13], which is almost 2–3 times higher than that of the required ACH dosage. Therefore, from this study, it can be said that the use of industrial grade ACH is an efficient coagulant for the decolourisation of textile wastewaters.

3.3. *Efficacy of ACH for the treatment of real silk dyeing wastewater*

To verify the effectiveness of ACH as a coagulant, a similar test procedure as of the synthetic textile wastewaters has been carried out for the treatment of real silk dyeing wastewater. The optimum pH for efficient decolourisation in this case has been observed at pH 4.0, giving a maximum of 72% decolourisation and 46% COD reduction efficiency at a dose of 50 mg L^{-1}. Further, the variation of treatment efficiency with ACH dosage had also been investigated by keeping the optimum pH constant. A significantly different trend in the treatment efficiency has been observed for real silk dye wastewater (Fig. 3) compared with synthetic textile wastewater combinations. Colour removal efficiency of maximum 90.63% was obtained at only 100 mg L^{-1} of ACH, along with an excellent turbidity reduction of 98.66% and COD reduction of 40%.

From Fig. 3, it can be observed that the colour removal efficiency decreases sharply after an optimum coagulant dosage of 100 mg L^{-1} ACH. Turbidity reduction has also been observed to follow the similar trend. The decrease in the colour and turbidity removal at higher dosage may be attributed to the fact that addition of ACH in excess of optimum dosage provides positive superficial charges to the

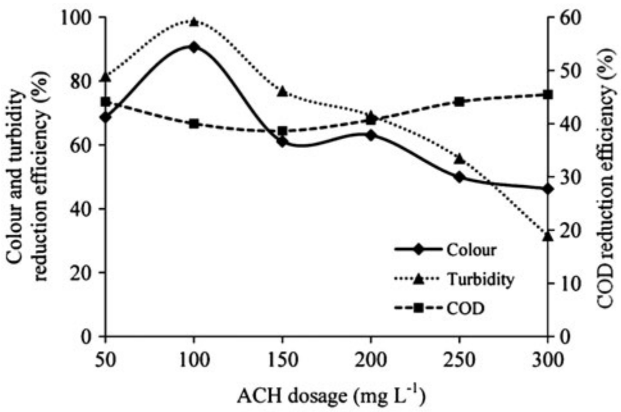


Fig. 3. Effects of ACH dosage on the treatment efficiency for real silk dyeing wastewater.

neutralised flocs and thereby simultaneously reactivates the dye molecules that finally repel each other to produce more turbid solution by restabilisation mechanism [16]. This restabilisation phenomenon was not appeared in case of synthetic textile wastewater, which can be linked to the difference in nature and complexity between the real silk dye effluent and synthetic textile wastewaters. Based on the optimum pH value of 4.0, that is, in the acidic regime, adsorption and charge neutralisation could be thought of as the principal colour removal mechanism of ACH for the treatment of real silk dyeing wastewater.

From this study, it can be said that the ACH is not only an efficient coagulant for the decolourisation of synthetic textile wastewaters but also for real textile effluents. The lesser dosage requirement for decolourising real textile wastewater compared with synthetic textile wastewaters can be linked to the difference in residual dyes concentration as well as the type of dyes being used. Although the nature and concentration of residual dyes in the real textile wastewaters were unknown, the use of 200 $mg\,L^{-1}$ dyes in the preparation of synthetic textile wastewaters lies in the upper limits of the residual dyes concentration normally been reported in the literature. The less dosage of ACH for the treatment of real textile wastewater was probably due to the less residual dyes concentration than that of the synthetic textile wastewaters used in this study. The observed optimum ACH dosage of just 100 $mg\,L^{-1}$ was considerably lower than that of the dosage required for other metallic as well as pre-hydrated metallic coagulants reported by the several other researchers for the treatment of real textile wastewaters [13,15]. Thus, ACH can be considered as one of the most efficient industrial grade coagulant for the decolourisation of textile wastewaters.

3.4. Sludge production

The quantity and quality of the sludge produced during coagulation/flocculation depend upon the type of coagulant used and the operating conditions [17]. Therefore sludge production, in terms of settled sludge volume and SS, was measured at optimised pHs and at an optimum coagulant dosage for all the types of wastewaters. The settled sludge volume was measured based upon the volume occupied by the flocs in 500 ml of sample volume after settling for 1 h in the Imhoff cone.

It can be observed from Fig. 4 that ACH produced a maximum of 55, 40 and 47 mL settled sludge per 500 mL of sample with 440, 360 and 404 $mg\,L^{-1}$ SS for the treatment textile wastewater containing CR, DB3 and CR + DB3, respectively, at the optimised conditions. Only 25 mL settled sludge per 500 mL of sample and 301 $mg\,L^{-1}$ SS were observed for the real silk dyeing wastewater using ACH (Fig. 4).

A comparatively higher volume of sludge production in the case of synthetic textile wastewater containing CR might be due to the poor adsorption of dyes onto the polymeric aluminium hydroxide flocs. The lesser quantity of sludge production in the case of real textile wastewater was related to the lesser optimum coagulant dosage requirement in comparison with synthetic textile wastewaters. Also, the sludge produced during coagulation of real silk dyeing wastewater was observed to be more compact in nature, which can easily be dewatered. Significantly higher volume of sludge production has been reported by Bidhendi et al. [18], who investigated the sludge production during treatment of industrial dyeing wastewater using alum, $FeSO_4$, $FeCl_3$ and $MgCl_2$ at their optimised pH.

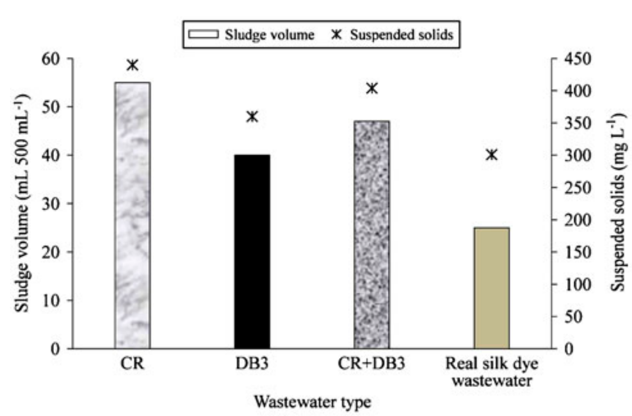


Fig. 4. Sludge production at optimised conditions for different types of wastewater using ACH.

3.5. Spectral analysis and colour removal mechanism

All the different combinations of textile wastewater used in this study were undertaken to investigate the spectral analysis and colour removal mechanism of ACH. The spectral analysis was examined on untreated and treated wastewater at optimum pH and coagulant dosage for the wastewater containing CR, DB3, CR + DB3 together and real silk dye wastewater. The results are shown as spectra C_1 for untreated wastewater and C_3 for treated wastewater (Fig. 5).

It can be observed from Fig. 5 that the wastewaters after treatment did not show any distinctive peak in the visible region. This revealed that the dye from the wastewater was transferred into the hydroxide precipitate obtained by coagulation-flocculation. Further, precipitates (sludge) were filtered and acidified using 1.0 M H_2SO_4 to convert into the solution. The filtrate is then neutralised and analysed by spectrophotometer, and the results are shown as spectra C_2 in Fig. 5. The shapes of the spectrums C_1 and C_2 are almost similar and peaks have been observed to form almost at the same wavelength for all the types of wastewaters. The colour of neutralised solution was almost similar but the absorbance value was less. This was due to the fact that the complete conversion of the dye precipitates from the sludge into the solution by acidification is not possible. As there were no changes in the peaks absorbances, it can be said that removal of colour by ACH was merely a physical phenomenon. There was no chemical change of dye molecules before and after coagulation as both peaks have been found to form at the same wavelength for C_1 and C_2 spectrums. Based on the optimum pH values, adsorption-charge neutralisation and sweep-flocculation can be considered as the dominant colour removal mechanisms for ACH.

3.6. Comparison on the effectiveness of ACH with the pre-established studies

The promising and superior treatment efficiency of ACH as a coagulant can also be compared with the pre-established studies using various coagulants for the treatment of real textile wastewaters.

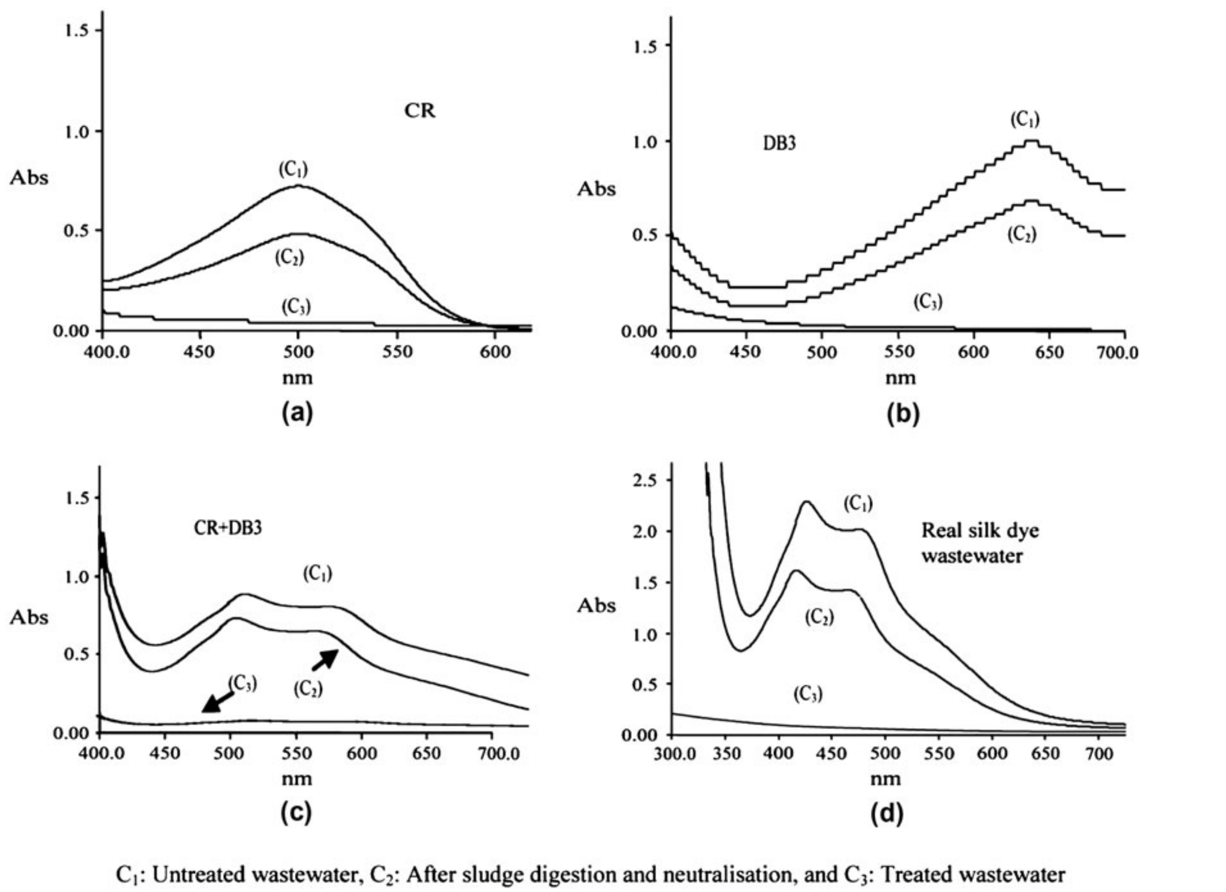


Fig. 5. Spectrogram of different types of wastewater at optimised conditions using ACH (a): CR, (b): DB3, (c): CR + DB3 and (d): real silk dye wastewater.

Table 3
Effectiveness of various coagulants for treatment of real textile wastewaters

Coagulant used	Dosage (mg L^{-1})	Optimum pH	Colour removal (%)	COD reduction (%)	Reference
Alum	300	5.3	83	–	[19]
Alum	5,000	4.0	74	58.6	[20]
Alum	1,500	7.0	60	56	[21]
Magnesium chloride	400	8.7	98	80	[18]
Magnesium chloride	1,500	11.0	97.9	88.4	[22]
Ferrous sulphate	1,000	9.5	50	59	[21]
Ferrous sulphate	70,000	5.7–6.7*	84.9	80.2	[23]
Ferrous sulphate	800**	10.0	48.56	55.72	[24]
Polyaluminium chloride	800**	7.5	75.49	65.4	[24]
Ferric sulphate	70,000	5.7–6.7*	57.9	55.2	[23]
Ferric chloride	200	8.3	97	83	[18]
ACH	100	4.0	90.63	40	*Present study*

*Original pH of wastewater.
**With flocculants.

It can be seen from the Table 3 that coagulants like magnesium chloride and ferric chloride were observed to be more effective for the decolourisation as compared to ACH, but at a very high coagulant dosage. Effectiveness of aluminium-based metallic salt such as PACl was also investigated by Tun et al. [24]. They have found considerable treatment efficiency for real textile wastewater at a dosage of 800 mg L^{-1} along with the use of flocculent as a coagulant aid. The optimum coagulant dose for ACH was found to be only 100 mg L^{-1}. Also at this low ACH dosage, virtually colourless effluent was observed after the treatment, rendering the effectiveness of ACH as a coagulant for the treatment of textile wastewaters.

4. Conclusions

Decolourisation of synthetic as well as real textile wastewater has been successfully carried out in this study using ACH as a coagulant. In view of the results obtained in this study, it can be concluded that ACH was highly capable to decolourise both the synthetic and real silk dyeing wastewater at a very low dosage. The highest decolourisation and COD reduction efficiency of more than 99 and 45%, respectively, were observed for all the types of synthetic textile wastewater investigated in the study. A significant colour removal efficiency of more than 90% was also obtained for the treatment of real silk dyeing wastewater at ACH dosage of just 100 mg L^{-1}. Excellent treatment efficiency and reduced volume of sludge production at a very lesser dosage makes ACH a novel coagulant and may be recommended for industrial application. 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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