Supremacy of Magnesium Chloride for Decolourisation of Textile Wastewater: A Comparative Study on the Use of Different Coagulants

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Abstract—**In this study, treatment efficiency of magnesium chloride (MgCl2.6H2O) was compared with respect to ferrous sulphate (FeSO4.7H2O), polyaluminium chloride (PACl), and aluminium chlorohydrate (ACH) for the treatment of textile wastewater. Treatment efficiency was assessed in terms of decolourisation and chemical oxygen demand (COD) reduction of synthetic textile wastewater containing reactive, direct and disperse dyes, along with the other chemical constituents that are normally released from different textile processing units. MgCl2.6H2O/Lime produced colour removal efficiency of 99.68% at a dosage 1200mg/L for the wastewater containing all the three dyes together. MgCl2.6H2O/Lime was also found to be the most effective coagulant system for treatment of textile wastewater containing only reactive dye, which produced 99.73% colour removal at a dosage of 1100 mg/L. For both the direct and disperse dyes, ACH was found to be superior over MgCl2.6H2O/Lime, FeSO4.7H2O/Lime, and PACl. Industrial grade ACH, which is normally used as polyelectrolyte, for the first time was used as coagulant in this study and was also appeared to be significant for decolourisation of textile wastewater containing all the three dyes together. From this study, MgCl2.6H2O/Lime was recommended as the best coagulant for the decolourisation of textile wastewater having very high original pH.**

Index Terms—**Aluminium chlorohydrate (ACH), coagulation, colour removal, magnesium chloride ($MgCl_2.6H_2O$), polyaluminium chloride (PACl), textile wastewater.**

I. Introduction

Textile dyeing and finishing industries are one of the biggest users of potable water as well as the chemical additives during various steps of textile processing. Wastewater discharge from these industries is a major source of environmental pollution. Potential harmful direct and indirect effects of textile wastewater have also been discussed by Verma et al. [1]. Effluents from these industries generally possess very complex characteristics such as deep colour due to the presence of residual dyes, high pH, high temperature, large amount of suspended solids (SS), high chemical oxygen demand (COD) and toxic chemicals. Direct discharge of this effluent into the water bodies or open land is undesirable not only because of its colour, but also due to the production of carcinogenic compound from the decomposition of complex dyes. Some of these dyes are recalcitrant compounds that persist in the environment over a long period of time due their extended half-life [2]. Accumulation of colour due to residual dyes in the wastewater significantly diminishes aesthetic quality, hinders sunlight penetration, affects photosynthetic activity and disturbs the ecosystem of receiving water [3], [4]. Therefore, textile wastewater should effectively be treated to comply the legal as well as the aesthetic standards before discharging it into the environment or municipal wastewater treatment plant.

The available literature shows a large number of well established conventional decolourisation methods involving physico-chemical, chemical and biological processes, as well as some of new emerging techniques like sonochemical or advanced oxidation processes. However, there is no single economically and technically viable method to solve this problem and usually two or three methods have to be combined in order to achieve adequate level of colour removal [5], [6]. Additionally, biological processes are generally cheap, simple and environmental friendly, which can be used effectively to remove the biodegradable organics but to a very lesser extent for removal of colour due to less biodegradable nature of the textile dyes. Almost all advanced oxidation processes are associated with high cost of operation and may produce the toxic by-products. Till date, chemical coagulation/ flocculation has been widely used as one of the most practised decolourisation technology for the treatment of textile wastewater. The main advantage of conventional physico-chemical processes like chemical coagulation and flocculation is the decolourisation of wastewater takes place by removal of dye molecule from the textile wastewater, and not by the partial decomposition of dyes, which can lead to an even more potentially harmful and toxic aromatic compound [7] .

Effectiveness of various metallic coagulants has been well established for treatment of textile wastewater containing a single dye or mixture of dyes of the same class with only distilled water for the preparation of synthetic textile wastewater. However, very limited studies have been reported on chemical treatment of synthetic textile wastewater containing majority of the chemical additives that are used in textile industry during different steps of textile processing. To the best of our knowledge, no study has been observed in the literature on the treatment of textile wastewater containing diverse toxic chemicals that are released from textile industries along with the mixture of different classes of dyes. Also, very limited data are

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available to assess the quantity and nature of sludge production [8]. Therefore, the present study was focused to investigate the effectiveness of Ferrous sulphate ($FeSO_4.7H_2O$), Magnesium chloride ($MgCl_2.6H_2O$), Polyaluminium chloride (PACl) as well as Aluminium chlorohydrate (ACH) as coagulants and lime as coagulant aid for the decolourisation and COD reduction of synthetic textile wastewater containing different classes of new generation dyes along with the various other chemicals. The study was focused at evaluating comparative effect of pH and coagulant dosage on colour removal efficiency and amount of sludge production for each of the combinations.

II. Materials and Methods

A. Chemicals

Extra pure magnesium chloride ($MgCl_2.6H_2O$) and ferrous sulphate ($FeSO_4.7H_2O$), industrial grade (with purity 30% w/w) Ployaluminium chloride (PACl) and Aluminium chlorohydrate (ACH) were used as coagulants and lime was used as coagulant aid. 1.0 M H_2SO_4 and lime were used to adjust the desired pH. The other chemical additives used in the preparation of the synthetic wastewater were of analytical grade.

B. Synthetic Textile Wastewaters

Synthetic textile wastewater was prepared as per the reported chemical constituents of real textile wastewater [9], [10], with a total dye concentration of 200 mg/L. The total dye concentration was prepared either with a single dye, or mixing three different 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 during textile processing for various purposes. Wastewaters were prepared using three commercial dyes namely, Reactive Black 5 (RB5), Congo Red (CR) and Disperse Blue 3 (DB3) in the tap water. Dyes were procured from Sigma-Aldrich, Germany.

The characteristics wavelength for each simulated dye wastewater was determined by running a scan of the dye solution on a UV-VIS spectrophotometer. The maximum absorbance wavelength (λ_{max}) for the wastewater was found as 591, 502 and 638 nm respectively for RB5, CR and DB3, which was used to measure the absorbance of respective treated wastewater. Colour content of the wastewater containing mixture of dyes was determined by taking sum of the absorbencies measured at 591, 502 and 638 nm [11]. The characteristics of synthetic textile wastewater were: COD = 1944 to 2007 mg/L, pH = 10.4 to 10.6, Abs(591) = 3.1154 for the wastewater containing RB5; Abs(502) = 0.9264 for the wastewater containing CR; Abs(638) = 0.3540 for the wastewater containing DB3; Abs(1731) = Abs(591) + Abs(502) + Abs(638) = 2.3992 for wastewater containing RB5, CR, and DB3. Percentage colour removal was determined by comparing the absorbance values for the wastewater after treatment to the absorbance value of the wastewater before treatment. Tap water served as a reference.

C. Coagulation and Flocculation Test Procedures

The optimum pH value and coagulant dosage required for efficient colour removal were determined by a jar test procedure. 1 L beakers, containing 500 mL of wastewater were used for the coagulation experiments. Lime or 1.0 M H_2SO_4 was added to each beaker for pH adjustment. Chemical coagulant was added and mixed for 3 min under rapid mixing condition at 80 rpm. The solution was mixed at slow flocculation for 15 min at 30 rpm. After sedimentation for 20 min, supernatants from the top of the beaker were taken for the analysis.

D. Analyses

Colour measurement was carried out after filtration of supernatant through Whatman No. 5 filter paper. Then, the pH of liquid was adjusted to about neutral. The absorbance of liquid was measured. COD was analysed as per closed reflux calorimetric method after digestion of the samples in COD reactor (Model DRB 200, HACH, USA) and then absorbance measurement was carried out by COD spectrophotometer at 600 nm (Model DR 2800, HACH, USA). COD standard curve was developed based on the absorbance. Sludge production (in terms of settled sludge volume) was also measured at optimised conditions for all the combinations. All the methods used for the analysis of wastewater characteristics were as per Standard Methods [12], and performed at room temperature (25±5°C).

III. Results and Discussion

A. Determination of Optimum pH for Chemical Coagulation of Synthetic Textile Wastewater

In wastewater treatment using metallic coagulants, pH plays a very important role in determining coagulation efficiency. Therefore, experiments were designed to determine the optimum pH for all the combinations of synthetic textile wastewater that allowed for maximum decolourisation and COD reduction. The effect of pH on the treatment efficiency was examined using fixed amount of coagulant at various pH conditions (4, 6, 8, 9, 10, 11, and 12) for all the coagulants. Lime and H_2SO_4 was used to adjust the desire pH. It is well known that pH affects the molecular structure of the dyes, which changes the absorbance of the solution. Therefore pH of the untreated wastewater as well as treated wastewater was adjusted to neutral before measuring the absorbencies for evaluating the percentage of colour removal. Table I summarises the optimum pH for selected coagulants against various combination of dyes containing synthetic textile wastewater. Percentage colour removal increases with the increase in pH from 4.0 to 11.0 or 4.0 to 12.0 when $FeSO_4.7H_2O$ and $MgCl_2.6H_2O$, respectively was used as coagulants. For the combination of textile wastewater containing RB5, CR, and DB3 together, the optimal pH was found to be 12.0, whereas for the rest of the two combinations it was 11.0.

TABLE I: TREATMENT EFFICIENCY AT OPTIMISED pH FOR DIFFERENT COMBINATION OF SYNTHETIC TEXTILE WASTEWATER

Coagulants	Targeted parameter	RB5	CR	DB3	RB5+CR+DB3
$FeSO_4.7H_2O$	Dosage (mg/L)	1000	1000	1000	1000
	Optimum pH	12	11	11	12
	Colour removal and COD reduction (%)	96.14 (53.31)	76.98 (51.34)	78.65 (36.09)	91.36 (39.34)
$MgCl_2.6H_2O$	Dosage (mg/L)	1000	1000	1000	1000
	Optimum pH	12	11	11	12
	Colour removal and COD reduction (%)	98.16 (49.24)	92.26 (44.09)	97.63 (37.36)	93.15 (59.01)
PACl	Dosage (mg/L)	2000	200	200	1500
	Optimum pH	10	11	6	10
	Colour removal and COD reduction (%)	64.64 (55.91)	86.00 (41.86)	96.07 (56.58)	65.60 (68.53)
ACH	Dosage (mg/L)	600	200	50	500
	Optimum pH	9	10	4	9
	Colour removal and COD reduction (%)	45.94 (37.29)	98.38 (40.06)	94.78 (41.32)	70.79 (39.18)

% COD reduction is given within the parenthesis ( )

For all the combinations, percentage of colour removal was found to be decreasing at pH greater than the optimum pH. Distinctive variation in the optimum pH of coagulation was observed for all the selected combinations of dye containing wastewater using pre-hydrolysed aluminium based coagulants such as PACl and ACH. This confirms that the optimum pH of coagulation not only depends on the coagulant types but also on the nature of dyes. Unlike DB3, RB5 and CR are the dyes containing sulphate group, which enhances the precipitation of aluminium hydroxide and forms abundant amount of $Al(OH)_4^-$ at alkaline pH. However at lower pH, aluminium based coagulant forms mostly $Al(OH)_3$, $Al(OH)_2^+$ and $Al(OH)^{2+}$ when hydrolysed [15]. At optimum pH, excellent reduction in colour and considerable reduction in COD was observed for the wastewater containing individual dye as well as the mixture of different dyes.

B. Determination of Optimum Coagulant Dosage for Chemical Coagulation of Synthetic Textile Wastewater

The optimum dosage of coagulants for the selected dye containing textile wastewater was determined by varying the coagulant dosage and maintaining the optimum pH. It has already been established that lime can be used to increase the pH as well as it can work as a coagulant/coagulant aid due to its capability to give a certain level of colour removal [16]. Colour removal as a function of coagulant dosage is shown in Fig. 1.

It was observed in almost all the cases that percentage colour removal increases with the increase in coagulant dosage.

Above 99% colour removal efficiency was observed at 1000 mg/L of $MgCl_2.6H_2O$/Lime for the wastewater containing RB5. $FeSO_4.7H_2O$/Lime was also found to be effective which gave more than 96% colour removal efficiency at a coagulant dosage of 1200 mg/L. However, considerable decrease in colour removal efficiency was observed at even higher dosage of ACH, which still showed better efficiency as compared to PACl (Fig. 1a).

ACH and PACl in case of textile wastewater containing CR had been observed to produce complete colour removal at a coagulant dosage of 400 and 500 mg/L respectively. Excellent colour removal of more than 98% had also been observed at the higher dosage of 1300 mg/L using $MgCl_2.6H_2O$/Lime and $FeSO_4.7H_2O$/Lime (Fig. 1b).

Again, ACH in case of textile wastewater containing DB3 had been observed to give almost complete colour removal at a reduced coagulant dosage of 300 mg/L. Excellent colour removal over 99% was observed at a dosage of 400 mg/L when PACl was used a coagulant. However, $MgCl_2.6H_2O$/Lime and $FeSO_4.7H_2O$/Lime was also found to be produce approximately similar degree at a colour removal at a dosage of 1000 mg/L and 1300 mg/L respectively (Fig. 1c).

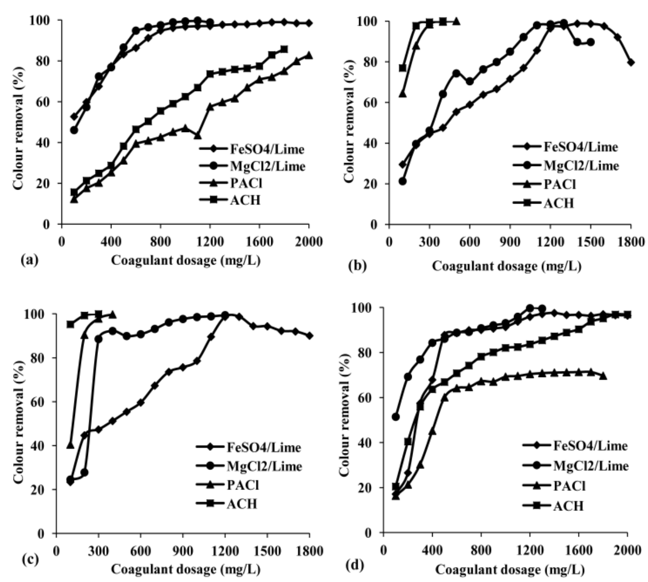

Fig. 1. Effect of coagulant dosage on the colour removal for synthetic textile wastewater containing (a) RB5 (b) CR (c) DB3 (d) RB5+CR+DB3

Colour removal efficiency of 99.68% and 97.11% had been achieved at 1300 mg/L of $MgCl_2.6H_2O$/Lime and $FeSO_4.7H_2O$/Lime respectively, for the wastewater containing RB5, CR and DB3 together. Approximately, 97% colour removal had also been observed at the ACH dosage of 2000 mg/L. However, PACl had been observed to give very low colour removal even at extreme dosage of 1800 mg/L (Fig. 1d). It was also observed during analysis that presence reactive dye in the mixture of dye wastewater affects the treatment efficiency of almost all the coagulants.

This can be attributed by the well established fact that reactive dyes are more difficult to decolourise.

The experimental results revealed that, comparatively reduced coagulant dosage is required for the decolourisation of the wastewater containing disperse dye as compared to the reactive and direct dye. This can be explained and linked by the fact that disperse dyes are least soluble in nature and hence competitively more efficient to adsorb onto the hydroxide precipitates. Moreover, pre-hydrolysed metallic salts namely ACH and PACl were found to be very promising for the decolourisation of wastewater containing DB3 and CR. This might be due to the better surface adsorption of disperse and direct dyes to the polyneuclear species of aluminium hydroxides. $MgCl_2.6H_2O$/Lime and $FeSO_4.7H_2O$/Lime were capable to give excellent decolourisation of the wastewater containing RB5. Further, more than 99% colour removal efficiency was observed for almost all the combinations of wastewater at only 1200 mg/L of $MgCl_2.6H_2O$/Lime. Based upon the results of this study, $MgCl_2.6H_2O$/Lime can be proposed as the best coagulant combination to decolourise the textile wastewater.

Similar trends of COD reduction were observed with increasing coagulant dosage as obtained in case of colour removal for all the combinations of synthetic textile wastewater using $FeSO_4.7H_2O$/Lime, $MgCl_2.6H_2O$/Lime, PACl and ACH (Fig. 2). A maximum of 62.02% COD reduction was observed at the optimum coagulant dosage of 1200 mg/L $MgCl_2.6H_2O$/Lime for the wastewater containing all three dyes together (Fig. 2d). Highest COD reduction of 70.32% had been observed at extreme PACl dosage of 1800 mg/L. Significant COD reduction was also obtained for remaining combinations of synthetic textile wastewater using $FeSO_4.7H_2O$/Lime and ACH as shown in Fig. 2.

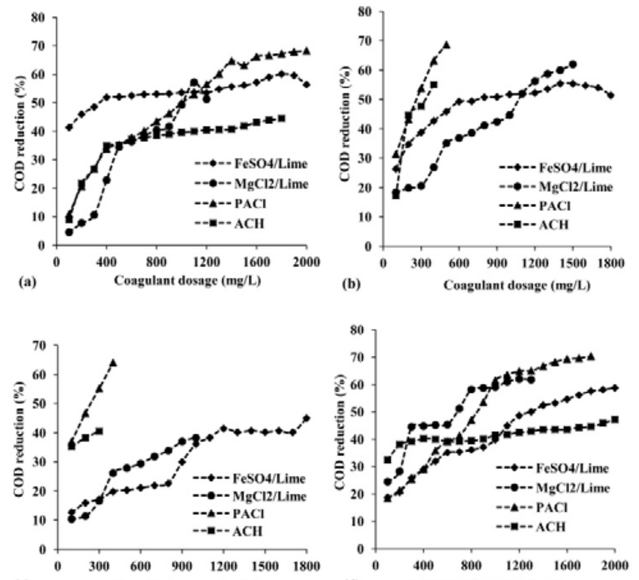

Fig. 2. Effect of coagulant dosage on the COD reduction for synthetic textile wastewater containing (a) RB5 (b) CR (c) DB3 (d) RB5+CR+DB3

Comparatively, PACl was observed as the better coagulant than $MgCl_2.6H_2O$/Lime, $FeSO_4.7H_2O$/Lime and ACH for COD reduction. This can be explained by the fact that the pre-hydrolysed nature of PACl forms relatively stable polyneuclear hydroxide flocs even at moderately alkaline conditions [15]. These flocs are instrumental in leading to sweep coagulation mechanism in the presence of bicarbonate alkalinity and thereby results in better COD reduction than that of other coagulation mechanisms such as charge neutralisation and adsorption. Further, COD reduction capability of $MgCl_2.6H_2O$/Lime was found to be lying in between PACl and $FeSO_4.7H_2O$/Lime for all the combinations of textile wastewater except the synthetic textile wastewater containing all three dyes together. Out of selected coagulants, ACH showed the lowest COD reduction efficiency for all the combinations of wastewater.

C. Sludge Production

The amount and characteristics of the sludge produced during coagulation/ flocculation depends upon the type of coagulant used and the operating conditions [17]. Therefore, sludge production was measured at optimised pH and at optimum coagulant dosage for all the combinations. It was measured based upon the volume occupied by the flocs in 500 mL of sample volume after settling for 1h in the Imhoff cone. A maximum of 120, 88, 85 and 25 mL settled sludge/500 mL of sample was observed in case of $FeSO_4.7H_2O$/Lime, ACH, PACl and $MgCl_2.6H_2O$/Lime respectively, for the wastewater containing CR. PACl and ACH was observed to produce similar amount of sludge which was still lower as compared to $FeSO_4.7H_2O$/Lime. Increased amount of sludge production in case of PACl and ACH as compared to $MgCl_2.6H_2O$/Lime can also be linked to the purity of these compounds.

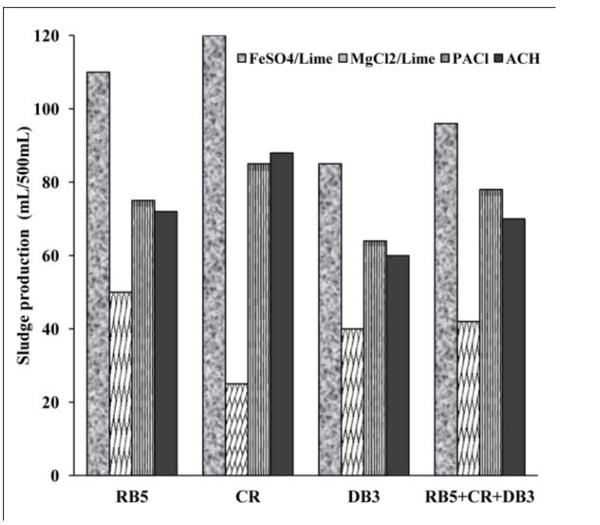

Fig. 3. Sludge production at optimised conditions for various combinations of synthetic textile wastewater

For all the combinations, volume of sludge produced was less when $MgCl_2.6H_2O$/Lime was used as coagulant (Fig. 3). Maximum 50 mL settled sludge/500 mL of sample was observed for the wastewater containing RB5. Significantly reduced sludge production using $MgCl_2.6H_2O$ may be explained by the fact that it shows very high adsorption for the dyes, particularly for direct dyes, and other chemical additives and thereby produces more compact sludge as compare to the $FeSO_4.7H_2O$/Lime, PACl and ACH. The results of the present study are in good agreement with the findings reported by Bidhendi et al. [8].

D. Analysis of Spectrogram and Colour Removal Mechanism of the Coagulants

Spectral analysis and investigation of colour removal mechanisms were performed for wastewater containing RB5, CR and DB3 dyes. All the three combinations showed almost the same trends for all the four coagulant systems ($FeSO_4.7H_2O$/Lime, $MgCl_2.6H_2O$/Lime, PACl, and ACH). Therefore, wastewater containing RB5 against $FeSO_4.7H_2O$/Lime and $MgCl_2.6H_2O$/Lime were selected to explain the spectrogram and colour removal mechanism. The spectral analysis was examined on untreated and treated wastewater at optimum pH and coagulant dose (shown in the Fig. 1a). The results are shown as the curve "a" for untreated wastewater and "c" for treated wastewater, respectively in Fig. 4(i) and Fig. 4(ii). It was found that the wastewater after treatment did not show any distinctive dye peaks in the visible wavelength range from 400 to 700 nm in both the cases. This indicates that the dye from wastewater was transferred into the hydroxide precipitate obtained by coagulation-flocculation. Further, precipitates were filtered and acidified to convert into the solution. The filtrate is then neutralised and analysed by spectrophotometer, and the results are shown as the curve "b" in Fig. 4(i) and Fig. 4(ii). It can be seen that the shapes of curve "a" and "b" appear to be almost similar and also the peaks have been observed to form at the same wavelength for both the cases. The color of neutralised solution was same as the untreated wastewater but absorbance value was less. This might be due to the fact that the complete conversion of the precipitate into the solution by acidification is almost impossible. Hence, it can be said that the removal of colour by coagulation was merely a physical phenomenon. There was no chemical change of dye molecules before, and after the coagulation as both the peaks have been found at the same wavelength for "a" and "b". The analysis is in good agreement with the observation reported by Gao et al. [16].

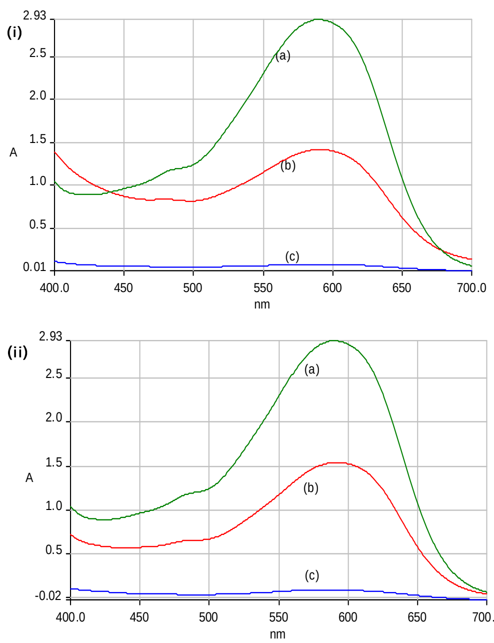

Fig. 4. The spectrogram of RB5 at optimum conditions using (i) MgCl2.6H2O/Lime and (ii) FeSO4.7H2O/Lime ["a": Untreated wastewater, "b": After sludge digestion and neutralisation, "c": Treated wastewater]

On the basis of the findings of this study, colour removal mechanisms of ferrous sulphate and magnesium chloride can be described as: (i) the metal ions from the coagulants first get converted into their insoluble metal hydroxides at pH greater than 11.0, (ii) these complex and insoluble structures of hydroxides provide a large adsorptive surface area and positive superficial surface charge leading to adsorption and charge neutralisation, (iii) finally, due to the presence of bicarbonate ions, lime reacts to form calcium carbonate precipitates, which remove dyes through the sweeping flocs mechanism [18]. Therefore, it can be said that the removal of colour using ferrous sulphate and magnesium chloride as coagulant and lime as coagulant aid takes place predominantly by adsorption and charge neutralization along with the sweep flocculation principle. Colour removal mechanism of PACl and ACH is not well understood and therefore, sweep flocculation is thought to be the principle coagulation mechanism due to the formation of huge amount of flocs at the increasing dosage of coagulant [19].

IV. Conclusions

Decolourisation and COD reduction efficiency of coagulants significantly depends upon the pH of wastewater. Pre-hydrolysed coagulants such as PACl and ACH were found to be effective in decolourising the wastewaters containing direct and disperse dyes. Further, magnesium chloride in combination with lime was found to be the best over the other coagulants for decolourisation and COD reduction of textile wastewater containing all the three dyes together. In the present study, a maximum of more than 99% decolourisation efficiency and 62% COD reduction efficiency was observed for treating synthetic textile wastewater containing a total 200 mg/L of reactive, direct and disperse dyes along with other chemical additives by using $MgCl_2.6H_2O$/Lime as coagulants. Reduced sludge production and excellent color removal makes $MgCl_2.6H_2O$/Lime a novel and attractive coagulant system, especially for the textile wastewater having very high original pH. Hence, $MgCl_2.6H_2O$/Lime may be recommended as an efficient coagulant system for the treatment of textile wastewater, which removes dyes by the dual mechanisms of adsorption and charge neutralization along with the sweep flocculation. Since, the system can reduce a maximum COD of approximately 60%, the remaining COD will be more than that of the safe discharge standards set by the Environmental Pollution Authority of different countries. Therefore, the secondary treatment is required to take care of the rest of the organic matters to meet the safe discharge standards.

V. Future Scope of Research

Future experiments are required to minimize the MgCl2.6H2O dose by combining with some natural coagulants or coagulant aids. ACH was also appeared as a very promising coagulant for decolourisation of the textile wastewater containing direct and disperse dyes, at a very less dosage. The effectiveness and coagulation mechanism of this coagulant is yet to be established. Therefore, it is required to conduct more and more research work on the coagulation mechanism of this coagulant.

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