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Discoloration of Synthetic Dyeing Wastewater Using Polyaluminium Chloride

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ABSTRACT

The efficiency of polyaluminium chloride (PAC) in discoloration of synthetic dyeing wastewater composed of reactive or direct dye has been investigated, taking into account the proven advantages of PAC in water purification processes (compared to the conventional coagulants such as Al2(SO4)3, FeSO4, etc.). The efficiency of PAC was determined by UV/VIS spectroscopy, controlling the UV/VIS absorbance changes of the wastewater samples after their treatment with the appropriate amount of PAC. Coagulant concentration and pH influence on the removal efficiency was followed in order to derive the optimum results. Excellent efficiency of PAC (100%) was found for the treatment of direct dye, Cuprophenil Blau 3 GL, at optimal pH=7.1 and concentration of 15 mg/L Al. The optimal results for reactive dye Bezaktiv Türkisblau V-G, enabling almost 80% dye removal, were obtained at pH=7.1 and PAC concentration of 20 mg/L Al.

Key Words: Poly (aluminium) Chloride, Coagulation, Discoloration, Dyeing Wastewater, Reactive Dye, Direct Dye, UV/VIS Spectroscopy.

1. INTRODUCTION

Wastewater from dyeing processes in the textile industry is a considerable source of environmental contamination, and its treatment for decolorization and removal of dye substances represents a substantial part of the integral processes for industrial wastewater purification. The effluent from the dyeing is characterized by strong color, high pH, high COD, high temperature and low or no biodegradability. There are more than 10,000 dyes incorporated in the Colour Index and available commercially, most of which are difficult to decolorize due to their complex aromatic molecular structure and synthetic origin [1]. One of the major factors determining the release of a dye into the environment is its degree of fixation on the fiber.

During the last decade, among different classes of dyes the use of reactive ones continually increased, mainly because of the increased utilization of cellulose fibers in the textile industry [2]. Generally, reactive dyes contain functional groups like azo, phthalocyanine, anthraquinone, formazane, and oxazine as chromophore. During the dyeing process, under the influence of heat in alkaline conditions, a dye's reactive sites react with the functional groups of the fiber. However a large fraction of the applied reactive dye is wasted because in the process of dyeing reactive dye is hydrolyzed to some extent and some of the reactive dyestuff is inactivated by this competing hydrolysis reaction. Consequently, the degree of dye fixation to cellulose fibers can be relatively low (in the range of 50-90%), and the release of reactive dyes into dyebath effluent is usually high. Compared with other dyes, reactive dyes represent severe pollutants. Direct dyes are widely used and are among the most economical dye classes. Chemically they are salts of complex sulfonic acids, soluble in water and with physical affinity for a wide variety of fibers (cotton, silk, wool, nylon, etc.). Substantive dyeing is normally carried out in a neutral or slightly alkaline dyebath, at or near boiling point, with the addition of either sodium chloride or Glauber's salt. The hazard- setting aside aesthetic considerations- is caused when colored agents interfere with the transmission of light through water and hinder photosynthesis, resulting in ecological imbalance [3]. Hence, removal of dyes from textile dyeing wastewaters is a major environmental problem and complete dye removal is necessary because dyes will be visible even at very low concentrations [4, 5].

Various methods have been investigated for treating dye bearing effluents, based on physical and chemical processes, and their combinations, such as coagulation-

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flocculation, ion-exchange, membrane filtration, electroflotation, electrokinetic coagulation, precipitation, oxidation, ozonation, adsorption and biosorption [1, 6-24]. Various advanced oxidation processes which have attracted a lot of attention in recent years, their chemistry, major application ranges and effects of dye-assisting chemicals, are reported in [25]. The coagulation/flocculation process is in extensive use for pre-, main, and post-treatment. For full-scale treatment, which allows reuse of chemicals and water, processes of ultrafiltration, nanofiltration and reverse osmosis are also used. However, for all these processes pre-treatment is needed for removal of dye and organic materials, consisting of coagulation and sedimentation.

Polymeric coagulants based on Al are increasingly used at treatment plants for wastewater, waterpool water, potable water etc. [26, 27], among which polyaluminium chloride (PAC), a polymerized form of alum, is most often used. The main advantage of PAC is its declared ecological appropriateness together with significantly higher efficiency at lower concentrations, lower temperatures and wider pH interval, compared with the conventional coagulants based on Al- or Fe(III)-chlorides and sulfates [28-30]. PAC has proved itself as an efficient coagulant especially for removal of organic materials present in the water, independent from their origin and complex composition [31]. Examinations concerning the application of PAC for treatment of wastewater containing different organic pollutants have shown that the efficiency of PAC depends foremost on the conditions used during aggregation/coagulation, coagulant dosage and pH. Optimal pH for achieving the most efficient removal of organic materials depends on the type of compounds to be removed and the functional groups present. It was shown that for colored organic compounds most efficient removal proceeds in acidic conditions, at pH≤6.5 [32, 33].

The purpose of this study was to investigate the applicability of PAC as a coagulant/flocculant for the removal of textile dyes from dyeing wastewater, and to determine the optimal coagulation conditions (coagulant dosage, pH) for wastewater pre-treatment. For that reason reactive dye aqueous solutions, direct dye aqueous solutions and synthetic dyeing wastewaters were prepared and treated. After the treatment with polymeric coagulant, the changes in dye concentration were followed using UV/VIS spectrophotometer, by measuring the absorbance at the max (responsible for dye color).

2. EXPERIMENTAL

2.1. Preparation of dye solutions for preliminary examination

For the preliminary examination of PAC coagulation/flocculation effectiveness, aqueous solutions of two types of dyes were used: Bezaktiv Türkisblau V-G (reactive dye, product of Bezema; λ_{max} =266, 330, 619 and 664 nm) and Cuprophenil Blau 3 GL (direct dye, product of Ciba; λ_{max} =305, 651 nm). The reactive dyes general structure is represented as X-B-R, where X is water-soluble group (-SO3Na or -OSO3Na), B is phthalocyanine (Bezaktiv Türkisblau V-G) and R is the structure reactive part (-SO2-CH=CH2) responsible for the chemical reaction with water. Direct dye Cuprophenil Blau 3 GL, with linear structure of molecules and containing azo- and sulphonic groups, was used for the PAC effectiveness analysis. Solutions were prepared in a 2 L beaker, with 0.01% dye concentration in distilled water and pH=6-6.5.

2.2. Preparation of synthetic dyeing wastewater

The characteristics of wastewater released from the dyeing process vary by the wastewater plant, sampling time and process conditions. Therefore it was required to prepare synthetic wastewaters with average concentrations of additives in for the testing samples.

2.2.1. Preparation of reactive dye solutions

Synthetic reactive dye wastewaters were prepared by mimicking operating conditions of the dyeing process, according to the procedure described in [34]. The reactive dye (Bezaktiv Türkisblau V-G) was first dissolved in water, containing NaCl (AD Alkaloid, Skopje), Na2CO3 (Merck, Darmstadt) and NaOH (AD Alkaloid, Skopje) to obtain an alkaline solution with 0.0001% dye concentration. This concentration reflects the average dye amount (influenced by the process conditions) released into the wastewater during the dyeing process [35]. Then, synthetic dye wastewaters were prepared by 1 h solution hydrolysis at 50 oC. Before PAC addition, the pH of the solutions was adjusted (HI 9321 Microprocessor pH meter) to the required value using standard solutions: 1 N and 2 N NaOH, 1 N and 2.25 N H2SO4 (Merck, Darmstadt).

2.2.2. Preparation of direct dye solutions

Corresponding preparation was also used with direct dye (Cuprophenil Blau 3 GL) having low dye exhaustion efficiency, applying the same procedure as described for reactive dye (2.2.1). Preparation of synthetic direct dye wastewaters excluded hydrolysis pace, since there is no hydrolysis of direct dyes.

2.3. Characteristics of PAC solution

PAC is a product of Phosphoric Fertilizers Industry C.A, Thessalonica, with characteristics presented in Table 1.

Table 1. Declared characteristics of PAC solution.

Characteristics	*Value*
Al_2O_3 (%)	14 ± 0.5
Chlorides (%)	21 ± 2.5
Basicity (%)	40 ± 5
Sulphates (%)	< 0.1
pH (1 % solution)	1.2–2.5
Specific Gravity at 20°C	1.31 ± 0.03

Dosing solutions of PAC were prepared by diluting the commercial coagulant several times to obtain predetermined amount of Al.

2.4. Coagulation and flocculation experiments

Experiments were conducted in a 2 L beaker containing 1 L synthetic dyeing wastewater. After pH adjustment, the samples were treated using predetermined concentration of polymeric coagulant. Initially, the sample in the beaker was rapidly mixed for 2 min (200 rpm). Then PAC solution was added and rapid mixing continued for one more minute, followed by slow mixing for 5 min at 50 rpm. Subsequently, flocculation/precipitation allows the agglomerates to settle out of suspension. After definite precipitation time, the clear solution located at the top of the beaker (1-2 millimetres below the solution surface) was taken for further analyses. The changes in dye concentration with time were determined by measuring the absorbance at λ_{max} of the dye, before treatment with PAC and after 30 and 60 min precipitation time. To determine the PAC induced changes of in pH at the beginning of coagulation, pH measurements were also performed immediately after adding PAC solution.

For reactive dye solutions these experiments were performed at pH = 4.4, 7.1 and 9.8, and PAC concentration of 5, 15 and 20 mg/L Al. For direct dye solutions, our research comprised the following conditions: pH = 5.1, 7.1, 8.1 and 10.3, and PAC concentration of 10, 15, 20 and 25 mg/L Al.

2.5. UV/VIS spectrophotometry

The concentration of dye remaining in the solution after the treatment with PAC is measured colorimetrically, using a Hewlett Packard 8452A UV/VIS spectrophotometer. The efficiency of PAC was calculated according to the expression:

$$E = \frac{(A_0 - A_t)}{A_0} \times 100 \quad (\%),$$

where A_0 is the UV/VIS absorbance at λ_{max} before the treatment with PAC, and At is the absorbance after t minutes of treatment with PAC. Since more than one absorbance peak is characteristic for UV/VIS spectra of the investigated dyes, λ_{max} in the visible region responsible for dye color is taken for the calculation of PAC efficiency. The absorbancies in the visible region were taken as more relevant in determining efficiency of PAC, since at wavelengths lower than 210 nm the observed absorbancies are characteristic for hydrogen bonds of water ($n \rightarrow \sigma^*$ transitions). For example, for reactive dye Bezaktiv Türkisblau V-G, the absorbance at λ_{max} = 664 nm (related to phtalocyanine unit) is used for the calculations of PAC efficiency. For direct dye Cuprophenil Blau 3 GL, the absorbance at λ_{max} = 651 nm is used for the PAC efficiency calculations.

3. RESULTS AND DISCUSSION

One of the most important parameters for coagulation processes, when using inorganic coagulants such as PAC, is pH of wastewater [30], especially for efficient removal of total organic carbon (TOC). Investigations of the alum efficiency in water treatment processes suggested that optimal pH for elimination of TOC and suspended particles is around pH=6. However, unlike Al2(SO4)3, PAC has high efficiency not only at pH<7, but also under alkaline conditions [29], which is of special interest for pre-treatment of wastewaters from the dyeing process with reactive or direct dyes, since their effluent exhibits high pH.

3.1. Preliminary examination of PAC efficiency

Our previous investigations revealed that optimal PAC concentration for removal of different organic pollutants is in the range of 2.5-20 mg/L Al [35]. Therefore, preliminary tests with direct dye (Cuprophenil Blau 3 GL) aqueous solutions, using PAC as a coagulant, were performed at concentrations in the range of 2.5-15 mg/L Al. At concentrations between 2.5-10 mg/L Al, PAC did not show significant coagulating activity when used on direct dye Cuprophenil Blau 3 GL at pH<7. Adjustment of the solution's pH was not done in these preliminary investigations, since it was of interest to evaluate the efficiency of PAC at “natural” pH value of dye solution (starting pH=6.6 was reduced to pH=6.24 after adding PAC). High efficiency was observed for concentrations of 10 mg/L Al and above, with immediate decoloration of dye solution and momentum flocculation. The efficiency of PAC expressed as a decrease of absorbance maximum at λ=651 nm, is 85-95% for a coagulation time of 5-30 min. However, PAC has shown insufficient efficiency in treatment of reactive dye Bezaktiv Türkisblau V-G aqueous solution at its “natural” pH, irrespective of the coagulant concentration.

Taking into account the relatively low degree of fixation of reactive dyes to the fibre, it was of interest to determine the optimal pH range and PAC concentration, using dye solutions with additives present in the real dyeing processes and conditions close to the real ones. Therefore for further experiments with reactive dye Bezaktiv Türkisblau V-G, synthetic wastewaters were prepared by mimicking real processes effluents, as described in the Experimental part (2.2.1). Comparative analysis was also performed with direct dye Cuprophenil Blau 3 GL having low dye exhaustion efficiency, using synthetic wastewaters prepared by procedure 2.2.2.

3.2. Efficiency of PAC in coagulation of synthetic reactive dye wastewaters

Experiments with reactive dye Bezaktiv Türkisblau V-G were carried out in a pH range of 4 to 10. Results from the examination of PAC efficiency in coagulation of synthetic reactive dye wastewaters are presented in Table 2.

Table 2. Changes in PAC efficiency derived from UV/VIS spectra of synthetic reactive dye wastewaters, with different pH and PAC concentration range between 5-20 mg/L Al.

pH	Concentration of PAC in synthetic reactive dye wastewater					
	5 mg/L Al		15 mg/L Al		20 mg/L Al	
	Time (min)	E (%)	Time (min)	E (%)	Time (min)	E (%)
4.4	30	3.8	30	28.5	30	28.0
	60	9.0	60	36.0	60	33.0
7.1	30	39.0	30	64.0	30	69.0
	60	47.7	60	67.0	60	76.3
9.8	30	2.3	30	5.4	30	2.0
	60	9.0	60	6.0	60	13.0

Table 2 represents the PAC efficiency dependence on pH, for concentration range between 5-20 mg/L Al. It can be seen that the best results were obtained at pH=7.1, with a higher applied concentration of PAC. The efficiency of PAC at concentrations of 15 and 20 mg/L Al, at pH=7.1 and 30 min of coagulation is 64 and 69% respectively. Changes in absorbance maximum for reactive dye wastewaters, after the addition of PAC, can be seen in Figure 1, for the experiments carried out at pH=7.1.

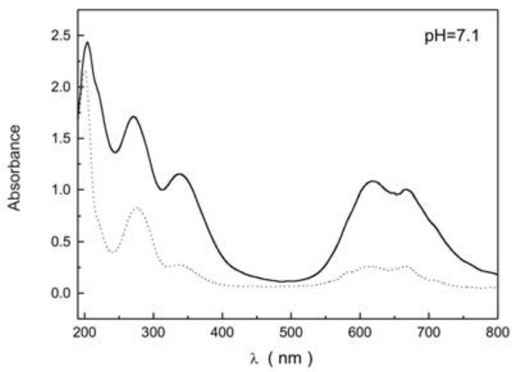

Figure 1. UV/VIS spectrum of synthetic reactive dye wastewater, before treatment (connected line) and after 60 min treatment (dotted line) with 20 mg/L Al, pH=7.1.

At pH=9.8, the efficiency reached only 13% after 60 min of coagulation and PAC concentration of 20 mg/L Al. In acid medium, at pH=4.4, the efficiency of PAC is around 30% after 30 min coagulation, at concentrations of 15 and 20 mg/L Al. For the optimum pH, efficiency of PAC is not significantly changed after 30 min coagulation.

The PAC efficiency difference in preliminary and main experiments, during treatment of reactive dye solutions at optimum pH range, is a result of the hydrolysis step included in the preparation of synthetic reactive dye wastewaters. In the preliminary experiments we have reactive dye dissolved in its non-hydrolyzed form, and in the main experiments we have the hydrolyzed form of Bezaktiv Türkisblau V-G. During discoloration with polymeric coagulant, PAC undergoes a hydrolysis reaction resulting in insoluble aluminium polyhydroxides which precipitate forming high-volume flocs. The flocs remove color and colloidal matter by their adsorption onto/within the formed metal hydroxides. The hydrolyzed form of the reactive dye is more prone toward precipitation with PAC hydrolyses products as a result of established attraction forces.

3.3. Efficiency of PAC in coagulation of synthetic direct dye wastewaters

Experiments with direct dye Cuprophenil Blau 3 GL were carried out in a pH range of 5 to 10 and PAC concentration of 10-25 mg/L Al. Results from the examination of PAC efficiency in coagulation of synthetic direct dye wastewaters are presented in Table 3.

Table 3. Changes in PAC efficiency derived from UV/VIS spectra of synthetic direct dye wastewaters, with different pH and PAC concentration range between 15-25 mg/L Al.

pH	Concentration of PAC in synthetic direct dye wastewater					
	15 mg/L Al		20 mg/L Al		25 mg/L Al	
	Time (min)	E (%)	Time (min)	E (%)	Time (min)	E (%)
5.1	30	44.2	30	40.1	30	45.8
	60	61.9	60	49.4	60	61.5
7.1	30	69.4	30	69.0	30	66.7
	60	100	60	99.8	60	100
8.1	30	90.7	30	86.0	30	81.1
	60	91.5	60	88.1	60	89.1
10.3	30	25.0	30	31.3	30	49.1
	60	50.0	60	47.8	60	73.8

Results revealed that the removal of dye is complete (100%) at pH=7.1, after 60 min coagulation with PAC in concentration of 15-25 mg/L Al; at lower concentration (10 mg/L Al) the efficiency was 95%. Comparing the coagulation efficiency while increasing coagulant dosage from 15-25 mg/L Al, it can be concluded that efficiency is practically independent from the PAC concentration for the period of 30 min. Changes in absorbance maximum after adding 15 mg/L Al, for the experiments carried out at pH=7.1, can be seen in Figure 2.

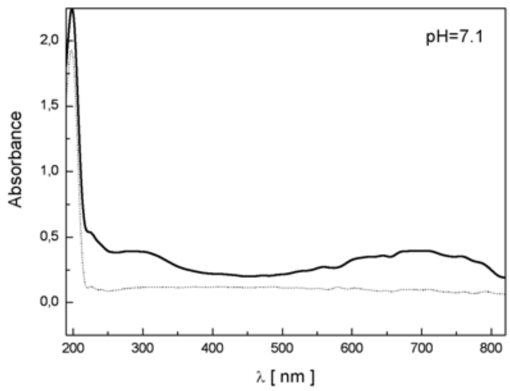

Figure 2. UV/VIS spectrum of synthetic direct dye wastewater, before treatment (connected line) and after 60 min treatment (dotted line) with 15 mg/L Al, pH=7.1.

In mild alkaline medium (at pH=8.1), for PAC concentration of 15 mg/L Al, dye removal reached 81% after only 5 min and almost 91% after 30 min; results after 60 min are similar to the ones obtained for higher PAC concentrations. Results obtained at pH=5.1 confirm the previous observation, conceived at pH=7.1 and pH=8.1, that coagulant efficiency is practically unchanged with the increase of PAC concentration in the range of 15-25 mg/L Al. The efficiency of coagulation is significantly lower at pH=10.3 (25-50% for 30-60 min and 15 mg/L Al), although in this case increase of PAC concentration is reflected in the improvement of coagulation efficiency up to 74% (for 25 mg/L Al and 60 min coagulation). This can be explained by the decreased solution alkalinity in the presence of higher PAC doses, bringing pH closer to the optimum range.

4. CONCLUSION

The obtained results revealed that PAC can be successfully applied for dye wastewater pre-treatments, both for reactive and direct dyes, at pH=7-8 and at low PAC concentrations (15-20 mg/L Al). The experiments were conducted with synthetic dyehouse effluents, mimicking the real dyeing process conditions. Highest efficiency in removal of reactive dye Bezaktiv Türkisblau V-G was realized at pH=7.1 (76% with 20 mg/L Al, after 60 min settling time), with certain improvement in the efficiency found when PAC concentration increased from 5 to 20 mg/L Al. Efficiency of almost 100% in removal of direct dye Cuprophenil Blau 3 GL was achieved at pH=7.1 (PAC concentration of 15 mg/L Al), with little influence of the coagulant concentration in the range of 10-25 mg/L Al.

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