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Improvement of paint effluents coagulation using natural and synthetic coagulant aids

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Abstract

The coagulant iron chloride and the flocculants Polysep 3000 (PO), Superfloc A-1820 (SU) and Praestol 2515 TR (PR) have been used in this study to show the efficiency of coagulation flocculation process in the chemical precipitation method for the removal of organic and colouring matters from the paint industry wastewater. This study also includes the amount of produced sludge.

The results indicate that $FeCl_3$ is efficient at pH range 8–9 and at optimal dose of $650\ mg\ l^{-1}$. Iron chloride allows the removal of 82% of chemical oxygen demand (COD) and 94% of colour. However, sequential addition of coagulant and polymeric additives enhance clearly pollutant removal and produces less decanted sludge compared to the results obtained when the coagulant is used alone. The removal efficiency of COD reaches 91% and that of colour 99%. Coagulation–biflocculation process is more effective than the coagulation–monoflocculation one. The sequential addition of iron chloride, Polysep 3000 (cationic flocculant) and Praestol 2515 TR (anionic flocculant) seems to be the most suitable combination for the treatment of the paint industry wastewaters.

Keywords: Iron chloride; Coagulation–flocculation; Paint wastewater; Polyelectrolytes; Colour

1. Introduction

Latex paints generally consist of organic and inorganic pigments and dyestuffs, extenders, cellulosic and noncellulosic thickeners, latexes, emulsifying agents, anti-foaming agents, preservatives, solvents and coalescing agents. In manufacturing paint and allied products, all the constituents entering mixers or reactors come out as products and, as such, there is no major stream of wastewater associated with the production. Wastewater is mainly generated by the cleaning operations of mixers, reactors, blenders, packing machines and floors. Because of the varying degree of chemicals used, the wastewater contains considerable concentrations of biological oxygen demand (BOD), chemical oxygen demand (COD), suspended solids, toxic compounds and colour [1]. The discharge of such coloured wastewater into the environment is not only aesthetically displeasing, but impedes light penetration, damages the quality of the receiving streams and may be toxic to treatment processes, to food chain organisms and to aquatic life [2]. For these reasons, the effluent treatment is necessary before their discharge into the environment.

Physical and chemical decolourization treatment operations include adsorption [3–5], oxidation [6,7] and chemical precipitation [8,9]. Each of them has its own merits and limitations in application. In recent years, with the technological advancements in polymer chemistry and the increasing interest in nutrient control in addition to the rising construction and energy costs, substantial interest has developed in chemically enhanced wastewater treatment methods [10]. Coagulation–flocculation has always attracted considerable attention for yielding high removal efficiency in wastewater treatment; this process can be directly applied to wastewaters to remove organics together with suspended solids, without being affected by the toxicity in the wastewater. In addition, the main advantage of the conventional processes, like coagulation and flocculation, is the decolourization of the waste stream due to the removal of dye molecules from the effluents, and not due to a partial decomposition of dyes, which can lead to an even more potentially harmful and toxic

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aromatic compound [11]. However, the coagulation process is not always perfect as it may result in small flocs when coagulation takes place at a very low temperature or fragile flocs which break up when subjected to physical forces [12]. It is not only necessary to overcome these problems but also to improve the coagulation and flocculation processes to obtain a good quality effluent and rapid sedimentation of the flocs formed. For this, several products, denominated coagulant aids, can be used to bring together and agglutinate the flocs formed by the coagulant in order to increase floc density and, hence, to improve sedimentation [13,14]. Polymeric additives can act either by polymer bridging or charge neutralisation [15].

The purpose of this study is to improve the coagulation–flocculation process applied to a paint wastewater by adding coagulant aids. The flocculants used are Polysep 3000 (PO) (natural cationic flocculant), Superfloc A-1820 (SU) (synthetic anionic polymer) and Praestol 2515 TR (PR) (synthetic anionic polyelectrolyte). The polymers are used in combination with ferric chloride. As terminology for the use of one polymer in addition to coagulant we suggest “coagulation–monoflocculation”, and for the use of sequential addition of coagulant and two polymers there is “coagulation–biflocculation”. These data provide new information about the role of polymeric flocculants in the industrial wastewater treatment operations, especially coloured effluents.

2. Materials and methods

Wastewater samples are collected from the discharges of a paint factory. The wastewater is mainly characterized by its colour variation and high load of COD, generated effluent is discharged into municipal sewer. The characteristics of the raw wastewater (RW) are as shown in Table 1.

2.1. Coagulant and flocculants used

2.1.1. Iron chloride

Two principal inorganic coagulants used in water treatment are salts of aluminium and ferric ions. However, ferric ions are often the coagulants chosen to destabilize the colloidal and suspended solids [16]. In this study, iron chloride ($FeCl_3$) for reagent grade is used as coagulant.

2.1.2. Coagulant aids

The coagulant aids used in this study are for commercial grade.

- *Polysep 3000*: P3000 is a natural organic polymer supplied by AQUAREX-ARCIE, Italy. It is a stable product prepared with acidified vegetable tannic substances. The flocculant is a brown liquid cationic flocculant of a viscosity between 10 and 50 cps and a pH value close to 1.5 and density about 1.1 g cm^{-3} at 20 °C [9].
- *Superfloc A-1820*: Superfloc A-1820 is an anionic polyacrylamide. The flocculant of high molecular weight is supplied by Cytec Industries. It is an opaque liquid of a viscosity of 600 cps (at 0.5%), specific gravity from 0.99–1.03 (at 25 °C) and the freezing point is −18 °C.
- *Praestol 2515 TR*: Praestol 2515 TR is a modified polyacrylamide, poly(acrylamide-*co*-sodium acrylate). Anionic polyelectrolyte with very high molecular weight, provided as powder by Stockhausen, Krefeld, Germany.

2.2. Procedure in the jar tests

Jar test experiments are conducted under controlled laboratory conditions using a standard jar test apparatus. Four equal-volume polyethylene beakers are used to examine the four different dosages of coagulant/flocculant in each run. The sample bottles are thoroughly shaken for resuspension of possibly settling solids and then the appropriate volume of sample is transferred to the corresponding jar test beakers. The optimum coagulant and flocculants doses are determined on the basis of COD and colour removal and the amount of sludge produced. For each test, 1000 ml of paint wastewaters was taken in a 1000 ml worked volume beaker and, after addition of coagulant, mixed for 5 min at 150 rpm to insure complete dispersion. In coagulation–monoflocculation tests, after coagulant addition, a known amount of polymer solution is added while rapid stirring continues for another 1 min more. In coagulation–biflocculation tests, after coagulant addition, a desired amount of polymer solution is added first into the beaker. After the mixing time of 30 s, the second flocculant is added and stirred for 30 s at 150 rpm. Sequential addition of flocculants is a better mode than co-addition. In this mode, a maximum amount of the second

Table 1
Characteristics of paint wastewaters

Parameters	Concentrations			Moroccan guide level (rejection into sewer)
	Minimum	Maximum	Average	
pH	6.70	7.80	7.35	6.5–8.5
Conductivity (ms cm^{-1})	2.05	2.68	2.33	2.7
Turbidity (NTU)	1.09	56.00	26.83	–
SM (mg l^{-1})	4735.29	13,350.00	9532.43	600
COD (mg l^{-1})	4438.19	25105.69	16342.32	1000
BOD (mg l^{-1})	960.00	1968.00	1465.20	500
TKN (mg l^{-1})	50.03	490.30	199.73	–
Total phosphorus (mg l^{-1})	1.42	16.08	7.46	10
Chloride (mg l^{-1})	177.65	355.00	266.33	–
Sulphate (mg l^{-1})	55.35	5768.87	2389.01	400

Number of samples = 6.

polymer is available for complexation coupled with bridging [17]. After rapid mixing the slow mixing stage takes place for 15 min at 30 rpm, while the final settling step lasts 1 h.

2.3. Performance evaluation

To assess the efficiency of coagulant and flocculants on paint industry wastewater treatment, the following are considered: turbidity, chemical oxygen demand, colour and the amount of sludge.

Turbidity: The turbidity is determined by turbidity meter (HI 93703 Microprocessor turbidity meter).

Chemical oxygen demand: COD and other physico-chemical parameters for wastewater characterisation measurement were performed according to standardised methods [18].

Colour measurement: Prior to colour measurement, the sample is filtered through a membrane filter (0.45 μm) to prevent turbidity. Colour measurements are carried out with a spectrophotometer. Since the wastewater contains different kinds of pigments (depending on the production), the traditional method of applying the maximum absorbance is not used. Colour content is determined using a UV–vis (Model 7800 UV–vis spectrophotometer) by measuring the absorbance at three wavelengths (436, 540 and 660 nm) and taking the sum of these absorbencies [19,20].

Amount of sludge: The volume of decanted sludge is estimated by the volumetric method using the Imhoff cones [21]. Once the experiment has been performed in the jar test, the beaker contents are transferred to special graduated conical containers (Imhoff cones). After 1 h of settling, the sludge production is determined by direct reading as ml of sludge/l of wastewater treated.

3. Results and discussion

The paint wastewater are characterised by including substantial organic matter, high salinity, sulphate rich and high suspended solid. In principle, the wastewater does not meet the proposed effluent standards (Table 1). In order to meet these trade-related demands and to respect acceptable environmental standards, it is necessary to subject the effluent to an appropriate treatment. BOD/COD index [22,23] indicates that a biological treatment seems to be difficult, and then a physicochemical process is required. The coagulation flocculation process using iron chloride in combination with coagulant aids is used in the treatment of this effluent. Treatment efficiency is evaluated in term of pollutant removals (turbidity, COD and colour) as well as in terms of sludge production.

3.1. Determination of the optimal operating conditions

The pH solution is an important factor in the coagulation process [19]. The use of coagulant at its optimum pH displays maximum pollutant removal. With such optimum pH conditions, the soluble residual iron amount in the wastewater will be lower than 2 mg l^{-1} [24]. To optimise the pH of the coagulation process, a known volume of prepared ferric chloride solution is added to a jar containing 1 l of wastewater at different pH values adjusted with concentrated H_2SO_4 and NaOH. The effect of coagulation pH (pH of the wastewaters during coagulation flocculation process) on turbidity removal from jar tests for coagulation of paint wastewater using 500 mg l^{-1} of ferric chloride is shown in Fig. 1. It can be noticed that turbidity removal is most effective at a pH range between 8 and 9.

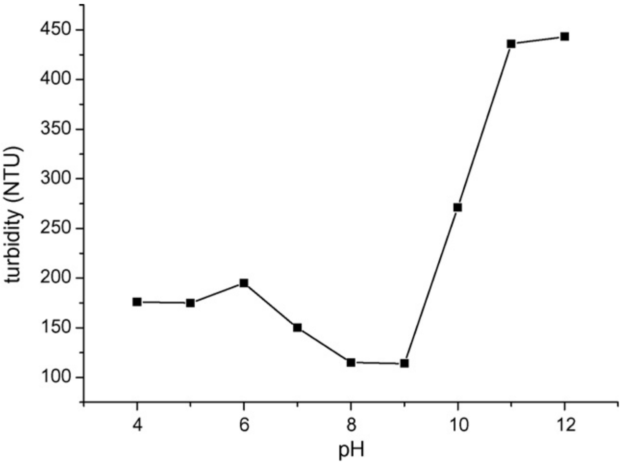

Fig. 1. Turbidity vs. coagulation pH using 500 mg l^{-1} of iron chloride.

The study of the effect of the iron chloride dosage on the COD and colour removal has been undertaken by varying the amount of coagulant in the wastewater, while keeping other conditions constant (pH 8.5) (Fig. 2). It is evident that for the quantitative removal of 82% of COD and 94% of colouring matter, under experimental conditions of the coagulation essay, a minimum dosage of 650 mg l^{-1} of $FeCl_3$ is required. Further increase in the coagulant dosage does not produce better removal rate. The coagulant dosage should be proportional to the quantity of colloids present. A further increase in coagulant dose causes restabilization of the particles as the charge reversal on the colloids occurs [25].

In the same way, the optimum coagulant aids doses in coagulation–monoflocculation process are determined by vary-

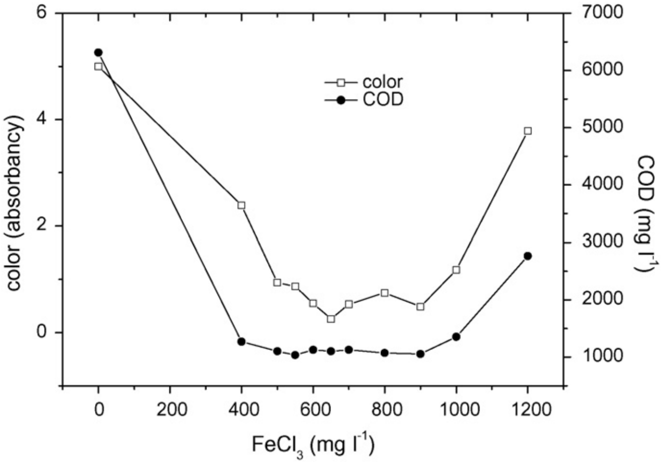

Fig. 2. Effect of iron chloride coagulation on COD and colour removals.

Table 2
Coagulant aids doses used in coagulation flocculation process

Coagulant aids	Coagulation–monoflocculation	Coagulation–biflocculation
Polysep 3000 (mg l^{-1})	160	80
Superfloc A-1820 (mg l^{-1})	2.4	1.2
Praestol 2515 TR (mg l^{-1})	5	2.5

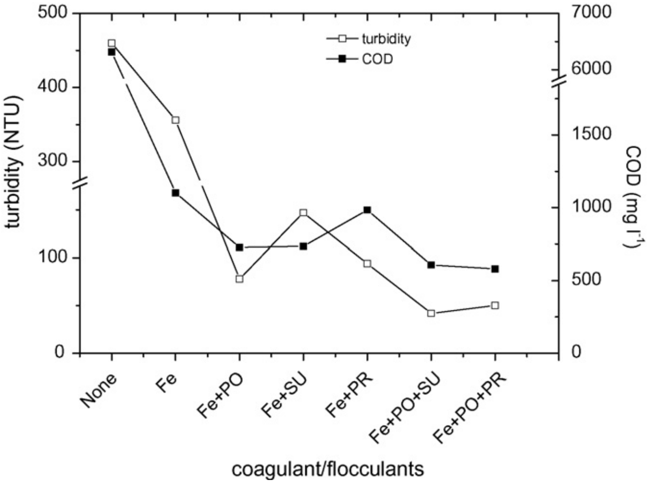

Fig. 3. Effect of coagulant aids addition on turbidity and COD removals.

ing the amount of coagulant aid in the wastewater, and by keeping constant the coagulant dose and pH (650 mg l^{-1} of $FeCl_3$ and pH 8.5). The results are given in Table 2. For the coagulation–biflocculation process, the polyelectrolytes doses are obtained through dividing the optimal dose used in the case of coagulation–monoflocculation by two.

3.2. Pollutant removals

The study of the effect of sequential addition of coagulant ($FeCl_3$) and flocculants on the turbidity and COD removals (Fig. 3) indicates that polymeric additives enhance clearly the pollution removal more than when the coagulant is used alone. Furthermore, sequential addition of coagulant and two polymers (coagulation–biflocculation) enhance the turbidity and COD removal efficiencies more than sequential addition of coagulant and one polymer (coagulation–monoflocculation). The residual COD in treated wastewaters using coagulant alone, coagulation–monoflocculation and coagulation–biflocculation are 1200, 730 and 580 mg l^{-1}, respectively. Thus, treated wastewaters using sequential addition of coagulant and polymers, respect the Moroccan acceptable environmental standards in terms of COD (1000 mg l^{-1}). Table 3 indicates that COD removal efficiency reaches 91% using coagulation–biflocculation process and 88% in the case of coagulation–monflocculation. However, the sequential addition of coagulant and Praestol 2515 TR is less effective than other combinations as the COD is removed off 84% only.

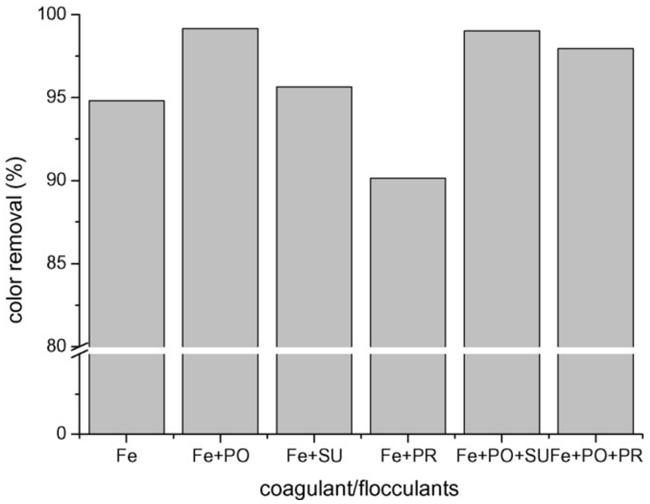

Fig. 4. Effect of coagulant aids addition on colour removal.

Fig. 4 shows the percentage of colour removal versus coagulant and flocculants. Compared to the result obtained when the coagulant is used alone, it appears that coagulation–biflocculation process and coagulation–monoflocculation using PO or SU addition enhance the colour removal efficiency. However, the sequential addition of coagulant and PR disadvantage the colour removal. Table 3 indicates that colour removal rates either using sequential addition of iron chloride and PO or a coagulation–biflocculation process are higher and appreciably equal. This may be explained by the fact that these combinations include Polysep 3000, which is an efficient flocculant for the removal of colour from wastewaters [9]. Colour removal is mainly attributed to flocculated decolourization process. Through energetic and hydrophobic interactions, the dye molecules intensively bind with the flocculant [26,27] leading finally to colour removal.

The addition of Praestol to wastewaters that are previously treated with coagulant or coagulant and Polyep 3000 induces a

Table 3
Percent colour and COD removals, sludges production and chemical cost for coagulation flocculation process using coagulant or coagulant in combination with flocculants

Combinations	Doses (mg l^{-1})	COD removal (%)	Colour removal (%)	Sludges (ml l^{-1})	Cost (€ m^{-3})
Fe	650	82.55	94.82	198	0.453
Fe–PO	650–160	88.46	99.18	186	0.880
Fe–SU	650–2.4	88.37	95.66	152	0.461
Fe–PR	650–5	84.42	90.14	90	1.703
Fe–PO–SU	650–80–1.2	90.35	99.04	168	0.670
Fe–PO–PR	650–80–2.5	90.80	97.96	128	1.291

little improvement of COD reduction and disadvantages colour removal. The colour removal, 94% when ferric chloride is added and 99% when using coagulant and PO, diminishes up to 90 and 98%, respectively, when the polyelectrolyte PR is added (Table 3). This may be explained by the fact that Praestol disturbs the interactions between dye molecules and primary flocs formed either by coagulant or by coagulant and Polysep3000.

The difference of percentage removal between colour and COD indicates that there exist different mechanisms for the removal of colour and COD from paint manufacturing wastewaters using these coagulant and coagulant aids. The maximum COD reduction is 90%. This may be due to the solubility of a part of COD; consequently it cannot be removed by decantation.

Combination of coagulant and coagulant aids improves the coagulation flocculation process. However, the pollutants removal efficiency is related to the nature of polyelectrolytes used [28]. Coagulation–biflocculation is more efficient than coagulation–monoflocculation in the treatment of paint wastewaters. It is the polymer–polymer interaction which results in excellent interparticle bridging, thus enhanced flocculation is obtained with double flocculants [29].

3.3. Sludges production

In addition to pollutants removal, sludge production is considered in this work, as it may affect the economic feasibility of the proposed method. In the solid–liquid separation, sludge dewatering has been pointed out as one of the most expensive processes [30].

As is shown in Table 3, the addition of coagulant aids produces a lower volume of decanted sludges compared to the result obtained when the coagulant is used alone. The amount of the produced sludge,198 ml l^{-1} when ferric chloride is added, diminishes up to 90 ml l^{-1} when this coagulant acts together with PR polyelectrolyte. However, the sludge produced using sequential addition of coagulant and PO is voluminous, resulting in an important sludge layer compared to the results obtained with other combinations of coagulant and flocculants. The amount of the sludge produced during the coagulation–flocculation process is highly dependent on the specific coagulants and flocculants used [13].

In the case of coagulation–biflocculation, the combinations used lead to appreciably equal removal percentages of colour and COD. However, the use of cationic (PO) and anionic (PR) flocculants has a higher dewaterability of sludges than using PO and SU additions. This indicates that Praestol (anionic flocculant) is effective on the dewaterability of decanted sludges. However, this polyelectrolyte cannot be used in coagulation–monoflocculation process as a sequential addition of iron chloride and PR because it reduces colour removal efficiency compared to the use of coagulant alone (Table 3). Bohm and Kulicke [31] have observed a better dewaterability of dredged when conditioned with both cationic and anionic polyelectrolytes simultaneously.

In order to compare the results obtained with $FeCl_3$ and each of the combination of coagulant and flocculants, we consider the ratios between the volume of sludge produced and the percent of COD and colour removals. These ratios have been estimated by taking into account the performances of the removal of organic and colouring matters (Fig. 5). The results indicate that the ratios obtained using sequential addition of coagulant and flocculants are markedly less than those obtained when the coagulant is used alone. However, the coagulation–biflocculation using sequential addition of coagulant, PO and PR produces the least amount of sludge for a given amount of COD and colour removed in the treatment of this effluent.

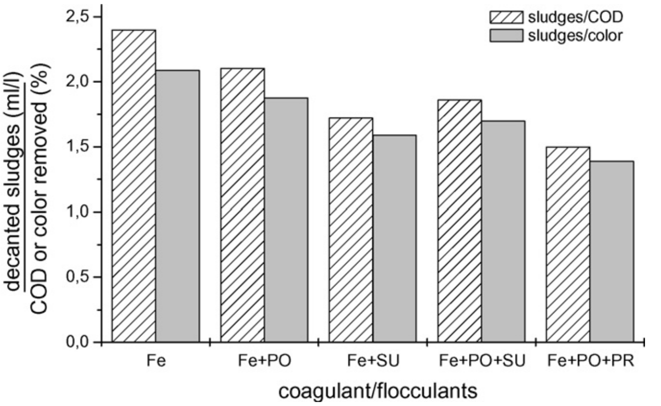

Fig. 5. Ratios between the volume of sludge produced and COD and between sludge and colour for different coagulant and flocculants.

The handling treatment and removal of the sludge generated in the coagulation–flocculation process are important aspects to consider when choosing the products to be used as coagulant and coagulant aids [13]. Considering the results obtained if a small amount of sludge is to be treated, the most suitable combination for the treatment of paint wastewaters would be coagulant ($FeCl_3$), PO (cationic polymer) and PR (anionic polyelectrolyte).

3.4. Effect on cost of coagulation–flocculation process

The proper determination of coagulant and flocculant types and dosages will not only improve the resulting water characteristics, but also decreases the cost of treatment. Table 3 shows the optimal doses for coagulant and each flocculant, and also shows the cost of the different products used. Compared to the results obtained when only the iron chloride is used, sequential addition of coagulant, PO and SU has little effect on the cost and improves the COD and colour removals but it does not reduces noticeably the amount of decanted sludge. The use of coagulant, PO and PR affect the cost but improves pollutant removals and leads to noticeable reduction of decanted sludges volume (Table 3).

4. Conclusion

The treatment of paint wastewaters using sequential addition of coagulant and polymeric additives enhance clearly turbidity, COD and colour removals and produces lower volume of decanted sludges compared to the results obtained when the coagulant is used alone. Coagulation–biflocculation process is more effective than the coagulation–monoflocculation one. The improvement of pollutant removal and the amount of sludges produced depends on the specific coagulant aids used. Therefore, the choice of an appropriate combination of coagulant and flocculants is of importance.

The natural cationic flocculant Polysep 3000 acts together with iron chloride and leads to better COD and colour removals. Nevertheless, anionic polyelectrolyte Praestol 2515 TR is effective for the dewaterability of decanted sludges.

The sequential addition of iron chloride, Polysep 3000 (cationic flocculant) and Praestol 2515 TR (anionic flocculant) seems to be the most suitable combination for the treatment of the paint wastewaters. It gives the best results and produces the least amount of decanted sludges for a given amount of COD and colour removal.

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