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Combined treatment of textile wastewater by coagulation–flocculation and advanced oxidation processes

Cristina López-López[a,b], Jaime Martín-Pascual[a,b], Juan Carlos Leyva-Díaz[a,b], María V. Martínez-Toledo[b], María M. Muñío[c], José M. Poyatos[a,b,*]

[a]*Department of Civil Engineering, University of Granada, E.T.S.I.C.C.P., Campus de Fuentenueva s/n, 18071 Granada, Spain, Tel. +34 958 246154; Fax: +34 958 246138; email: jpoyatos@ugr.es (J.M. Poyatos)*
[b]*Institute of Water Research, University of Granada, 18071 Granada, Spain*
[c]*Department of Chemical Engineering, University of Granada, 18071 Granada, Spain*

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ABSTRACT

Textile wastewater is one of the main environmental pollutants which exist in our society. Textile effluents cause great concern due to the alteration of properties of water bodies such as differences in temperature, organic load, pH, colour and turbidity. Turbidity is one of the most important parameters that should be removed from industrial wastewater because the penetration of ultraviolet (UV) light into the water body can be affected. As a consequence, the main aim of this research was to study the improvements of the efficiency of advanced oxidation processes (AOPs) with the introduction of a coagulation–flocculation (CF) as a pre-treatment to remove the turbidity of textile wastewater. The experiments were carried out with five industrial coagulants under different concentrations. The turbidity was removed to a level of almost 99% with one of the coagulants (FLOCUSOL-PA/18). The total organic carbon (TOC) and colour removals were studied for each AOP after the CF process in this research. The colour removal was almost 100% for all AOPs. The higher values of TOC and turbidity removals were 94.2 and 6.9%, respectively, with the heterogeneous photocatalysis process. The data show that the use of CF as a pre-treatment of the influent with turbidity improves the efficiency of the AOP.

Keywords: Turbidity; Coagulation–flocculation; H_2O_2/UV; Photo-Fenton; Heterogeneous photocatalysis; Textile wastewater

1. Introduction

The textile industry is known to be one of the most extreme water and energy consuming industries, causing intense pollution [1]. Moreover, it is one of the largest users of water and complex chemicals which are discharged as wastewater, causing a global concern for water recuperation and reuse [2]. The direct discharge of this wastewater into water bodies pollutes the water and affects the flora and fauna [2]. Therefore, determination of the quality criteria, focusing on conventional pollution parameters, such as organic matter content, pH, suspended solids (SS), heavy metals, microbiological load, temperature, colour, turbidity and toxic chemicals, has become a main objective of this kind of industry [3–5]. Textile wastewater has particles with a wide variety of

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*Corresponding author.

shapes, sizes and densities [6]. The removal of particles from wastewater is of great interest because many of the chemical and microbiological pollutants present in the water are absorbed by particles [7–9].

A complex and synergistic treatment of textile wastewater is the advanced oxidation process (AOP) which involves the conversion of organic pollutants to less complex species and even to their complete mineralisation through the generation of highly reactive free radicals [10,11]. AOPs have been widely studied and considered a promising technology, especially dealing with highly toxic and low biodegradable wastes [11,12]. AOPs are based on the use of UV radiation and oxidants such as hydrogen peroxide (H_2O_2), that are often combined with metallic or semiconductor catalysts and UV radiation [13]. Photoassisted systems such as photo-Fenton and heterogeneous photocatalysis ($TiO_2/H_2O_2/UV$) appear to be the most attractive AOPs for water treatment applications [14]. Firstly, the photo-Fenton process has proven to be a good alternative to treat a wide variety of pollutants in a more efficient way so that the biodegradability of the effluents is improved [15]. This process appears to have the capacity to decolourise and mineralise the textile industry dyes completely in a short reaction time [16]. On the other hand, the $TiO_2/H_2O_2/UV$ process is an emerging technology which can also easily decolourise and considerably reduce the organic load of dyehouse and related effluents [17].

However, given the origin of this wastewater, one of the most important parameters to consider is the presence of turbidity [18]. The penetration of UV light into the water source and, thus, process efficiency can be adversely affected by turbidity [19]. An excessive level of turbidity can reduce both the photomineralisation and photodisinfection efficiencies of the pollutants present in water due to the shielding effects which attenuate light penetration [20–22]. Turbidity leads to the weakened penetration of direct light and decrease the light photon absorption [23]. For these reasons, a pre-treatment is recommended to remove turbidity for the success of the process [12].

An alternative for solving the problems related to the turbidity in the influent of AOP could be coagulation–flocculation (CF). This process provides the removal of colour and turbidity in industrial wastewater [24], being a preferred option for removing turbidity and colour from wastewater [25]. Moreover, CF is one of the most commonly used water effluent treatments [11], being a versatile method which has been proven to have high removal efficiency in the chemical oxygen demand (COD) and SS too [26].

One alternative for wastewater treatment is to apply a physical–chemical procedure such as CF to eliminate most of the organic materials firstly, followed by an AOP as the second treatment [11]. The CF process usually consists of the rapid dispersal of a coagulant into the wastewater followed by an intense agitation commonly defined as rapid mixing [27]. This can achieve higher particle removal in the effluent and deliver improved the filtered water turbidity compared with conventional treatment processes [28].

This combination of processes has been studied by other authors [2,11,29] using coagulants such as polyaluminium chloride (PACl), polyaluminium ferric chloride (PAFCl), polyferrous sulphate (PFS) and polyferric chloride (PFCl) [2]. Marañón et al. [29] with a coagulation–flocculation process as a pre-treatment with ferric chloride, aluminium sulphate and aluminium polychloride (PAX) coagulants, obtained 98% turbidity removal, 91% colour removal and 26% COD removal. Rodrigues et al. [11] applied $FeCl_3$ as coagulant agent followed by AOP in the combined process.

The aim of this research was to study the effects of the introduction of a CF as a pre-treatment on an AOP for treating textile wastewater to remove the turbidity with CF and improve the efficiency of the AOP. Five coagulants with different concentrations were studied in order to determine the effect of turbidity removal which may prevent the penetration of UV light through wastewater samples during the AOP process. The three AOPs were carried out with 5 g/L of H_2O_2 and the total organic carbon (TOC), turbidity and colour removal over time were studied. A comparative study of different coagulants was carried out in the CF to evaluate its efficiency in the removal of turbidity.

2. Material and methods

2.1. *Experimental procedure*

Fig. 1 shows a schematic diagram of the pilot plant used in the present research. The pilot plant consisted of a pre-treatment of coagulation–flocculation prior to three different AOPs. The coagulation–flocculation process was performed in a batch reactor with a 1 L of operative volume. The coagulation was carried out for 1 min of hydraulic retention time (HRT) using five coagulants under a homogeneous high agitation of 120 rpm. The flocculation was conducted over 15 min of HRT under low agitation (30 rpm). The coagulants used were SICOAG C-21, FLOCUSOL-PA/18, FLOCUSOL-CM/1, SIFLOC C 40 L PLUS and SIFLOC C-30 with doses of 5, 5, 0.1, 0.1 and 1 g/L, respectively.

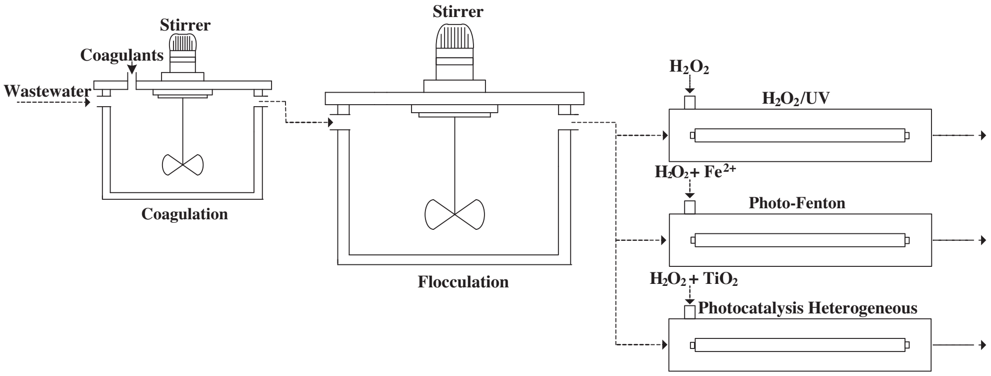

Fig. 1. Diagram of the pilot plant.

The AOPs were carried out in a batch reactor at a constant temperature of 25 ± 0.5°C and a volume of 800 mL with continuous mixing using a magnetic stirrer. The reactor consisted of a cylindrical quartz glass with a 150-W medium pressure mercury lamp and a quartz glass inside it. The reactor was covered with an opaque material to avoid interference from other external radiation.

The AOPs used were H_2O_2/UV, photo-Fenton and H_2O_2 at 0.25, 0.5, 1, 2 g/L [30] and 5 g/L to study the behaviour with an excess of oxidant for the three processes [31]. The amounts of catalysts used were 40 mg/L of Fe^{2+} and 200 mg/L of TiO_2 [32] for photo-Fenton and heterogeneous photocatalysis, respectively. The samples were collected from the photoreactor every 15 min [32–34]. The samples from the heterogeneous photocatalysis process were filtered through a 0.45-μm Millipore filter to remove TiO_2 particles [35].

The pilot plant was fed from a mixture of urban wastewater of the wastewater treatment plant Los Vados in Granada (Spain) and 500 mg/L of a commercial dye (IBERIA tinte 12 granate). The influent presented an average value of 621.5 ± 35.8 mg/L of TOC, pH of 7.4 ± 1.2 and a temperature about 20.1 ± 3.9°C.

2.2. Physical and chemical determinations

The turbidity measurements were taken at 860 nm according to UNE-EN ISO 7027:1999 and the absorbance for colour measurements were taken at 436, 525 and 620 nm according to UNE-EN ISO 7887:1994, both using a Heλios γ spectrophotometer (ThermoSpectronic), while the colour removal measurements for the results were taken at 436 nm. The pH was determined using a pH meter (Crison pH 25$^{®}$). TOC analysis was used to follow the degree of mineralisation during the different wastewater treatments. The TOC was determined using a Formarcs^{HT} TOC/TN analyser by oxidative combustion at 950°C.

2.3. Kinetic analysis

The TOC removal through a pseudo-first-order model was checked in this research. The kinetic model described by Calero et al. [31] allows for adjustment of the process under different operation conditions. Data were fitted to the different models, minimising the sum of squares error between empirical and modelling data.

3. Results and discussion

Some novel pre-hydrolysed coagulants such as PACl, PAFCl, PFS and PFCl have been found to be more effective and suggested for the decolourisation of textile wastewater [2]. Five coagulants of this type were tested in order to analyse different concentrations of turbidity removal in the effluent of AOP. Table 1 shows the efficiencies obtained with the coagulants used. The colour removal was higher than 84.5% in all cases, although this removal was almost 96.0% with the SICOAG C-21. Several studies have shown that coagulation–flocculation allows high efficiencies to be obtained, similar to those reported in the present study [29,36,37].

However, the coagulant used showed differences in turbidity and the TOC removals. The efficiency of TOC removal was lower than the performance of colour removal, which changed from 52.9 to 82.2% with SICOAG C-21 and FLOCUSOL-CM/1, respectively. These data of organic matter consumption were in accordance with those obtained by other authors such as Rodrigues et al. [11] or Stephenson and Duff [36].

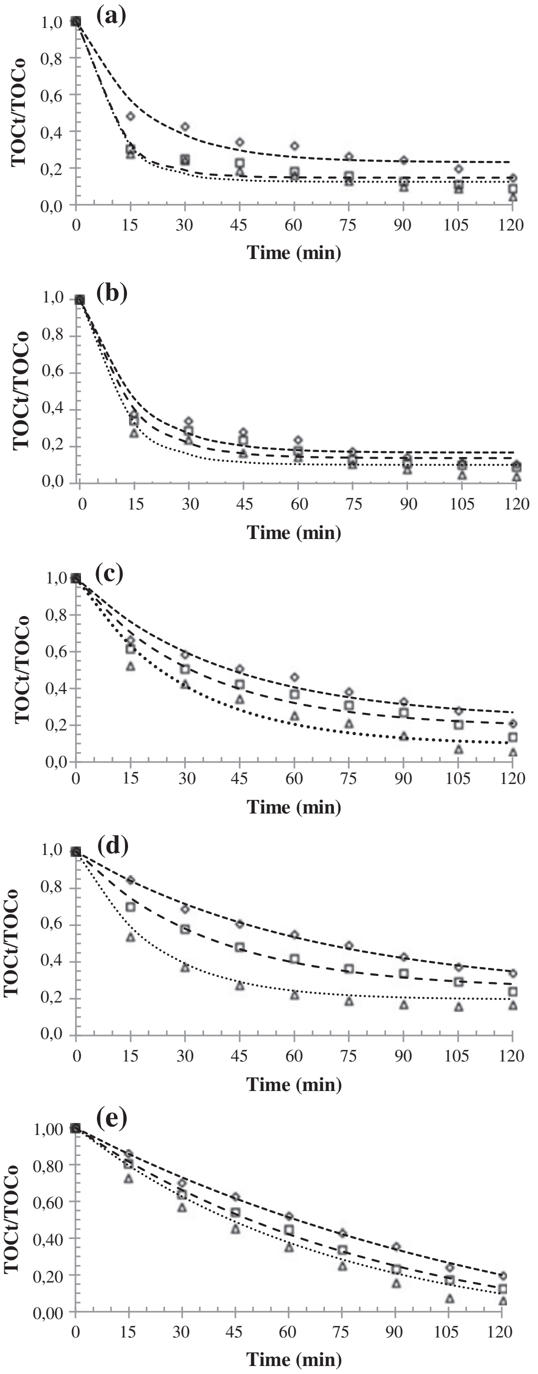

Fig. 2. Relation to TOC_t/TOC_0 for each AOP: H_2O_2/UV (◇), photo-Fenton (□), and photocatalysis heterogeneous (Δ) with their model data (- - -), (— —) and (···), respectively, of the coagulants: SICOAG C-21 (a), FLOCUSOL-CM/1 (b), FLOCUSOL-PA/18 (c), SIFLOC C 40 L PLUS (d), and SIFLOC C-30 (e).

A high range of values was obtained by varying the efficiencies of turbidity removal from 9.5 to 98.2%. Yonar et al. [12] obtained removal percentages of turbidity and COD of 84.0 and 39.0%, respectively, for treating urban wastewater in a similar research which used a conventional coagulation–flocculation with a dose of aluminium sulphate of 60 mg/L as a coagulant at pH of 7. Trinh and Kang [38] reported similar values of turbidity (91.4 and 86.3%) and COD (31.2 and 34.3%) using two coagulants in drinking water treatment. However, other authors have obtained higher efficiencies regarding organic matter removal, such as Martín et al. [39], who obtained a removal rate of turbidity of 72.0% and a COD efficiency of 82.0% in the wastewater treatment derived from sauce manufacturing. Therefore, coagulation–flocculation is considered a reliable technology for removing colour in a dyed wastewater. In relation to the removal of TOC, it depends mainly on the characterisation of the wastewater, especially on the presence of non-biodegradable substances [40].

The different turbidity removals obtained can facilitate an analysis of the effect of turbidity on the behaviour of the AOP due to the fact that turbidity removal would play a crucial role in the practice as the penetration of UV light through the wastewater is important for the success of the process in the long term [12].

Table 2 shows the removal efficiencies of colour, turbidity and TOC obtained after the AOP processes. The efficiencies of colour removal were higher than 94.0%, whereas the colour removal was lower than 7.0%. However, important differences were detected in the TOC removal, which could be caused by the effect of effluent turbidity. TOC removal increased from 61.6 to 94.2%. Independent of the coagulant, the heterogeneous photocatalysis was the most efficient process, while the H_2O_2/UV process presented the lowest removal rate. Some differences were obtained by comparing the different coagulants for TOC removal. In general, the highest efficiencies were obtained with the wastewater pre-treated with the FLOCUSOL-CM/1 coagulant and the lowest performances with the SICOAG C-21 coagulant.

Moreover, some differences were obtained in the evolution of TOC removal over time. A kinetic analysis was carried out to study these differences. Fig. 2 shows the empirical data obtained and the representation of the pseudo-first-order model.

The kinetic parameters and correlation rates (R^2) are shown in Table 3. All fittings present a correlation rate that is higher than 0.97; therefore, the model can be considered representative of the evolution experimented, as observed by other authors [31]. The overall rate constant (k) (min^{-1}) allows a comparison of the degradation rate, which is directly proportional to k, meaning that the process will require less time to

Table 1
Removals of colour, turbidity and TOC obtained with the five coagulants used

	Colour removal (%)	Turbidity removal (%)	TOC removal (%)
SICOAG C-21	95.6	92.9	82.2
FLOCUSOL-PA/18	93.4	98.2	55.7
FLOCUSOL-CM/1	92.5	26.7	52.9
SIFLOC C 40 L PLUS	84.5	18.7	70.3
SIFLOC C-30	78.3	9.5	55.5

Table 2
Removals of colour, turbidity and TOC obtained in the H_2O_2/UV, photo-Fenton and heterogeneous catalysis using as effluent the wastewater pre-treated with the five coagulants used

Coagulant	AOP	Colour removal (%)	Turbidity removal (%)	TOC removal (%)
SICOAG C-21	H_2O_2/UV	97.4	0.0	61.6
	Photo-Fenton	97.4	0.0	74.0
	Photocatalysis heterogeneous	98.7	0.0	82.7
FLOCUSOL-PA/18	H_2O_2/UV	94.8	0.0	75.7
	Photo-Fenton	96.6	0.0	78.6
	Photocatalysis heterogeneous	99.1	0.0	89.3
FLOCUSOL-CM/1	H_2O_2/UV	95.8	1.2	87.6
	Photo-Fenton	94.9	1.8	92.1
	Photocatalysis heterogeneous	99.1	2.3	94.2
SIFLOC C 40 L PLUS	H_2O_2/UV	95.7	1.3	66.2
	Photo-Fenton	94.3	1.3	76.1
	Photocatalysis heterogeneous	98.2	2.0	83.4
SIFLOC C-30	H_2O_2/UV	96.7	5.6	80.0
	Photo-Fenton	95.8	5.6	87.0
	Photocatalysis heterogeneous	99.3	7.0	93.6

Table 3
Kinetic parameters of pseudo-first-order model for each AOP and coagulant

	Pseudo-first-order model			
Process	Coagulant	C_e (mg/L)	k (min^{-1})	R^2
H_2O_2/UV	SICOAG C-21	0.77	0.054729	0.9764
	FLOCUSOL-PA/18	0.83	0.069339	0.9776
	FLOCUSOL-CM/1	0.77	0.024497	0.9815
	SIFLOC C 40 L PLUS	0.78	0.015223	0.9979
	SIFLOC C-30	1.38	0.007273	0.9984
Photo-Fenton	SICOAG C-21	0.85	0.101004	0.9872
	FLOCUSOL-PA/18	0.86	0.078335	0.9854
	FLOCUSOL-CM/1	0.81	0.030075	0.9836
	SIFLOC C 40 L PLUS	0.75	0.027368	0.9945
	SIFLOC C-30	1.18	0.011301	0.9986
Photocatalysis heterogeneous	SICOAG C-21	0.88	0.099211	0.9855
	FLOCUSOL-PA/18	0.90	0.091434	0.9856
	FLOCUSOL-CM/1	0.91	0.034280	0.9825
	SIFLOC C 40 L PLUS	0.81	0.047107	0.9986
	SIFLOC C-30	1.13	0.013374	0.9974

degrade the organic matter when the value of *k* is higher. The values of *k* varied from 0.0072 to 0.1010 under the different treatments. The average values of *k* were 0.1388, 0.0797, 0.0296, 0.0299 and 0.1064 with SICOAG C-21, FLOCUSOL-PA/18, FLOCUSOL-CM/1, SIFLOC C 40 L PLUS and SIFLOC C-3, respectively, concerning the pre-treatment. Therefore, the AOP had a higher degradation rate after the coagulation–flocculation with SICOAG C-21. The average values of *k* obtained were 0.0342, 0.0496 and 0.0894 with H_2O_2/UV, photo-Fenton and photocatalysis heterogeneous, respectively, regarding the different AOPs used. Thus, the AOP with the highest removal rate was heterogeneous photocatalysis. In relation to each coagulant, the highest *k* value was obtained with TiO_2 as the catalyst and the lowest with the H_2O_2/UV process, showing medium values for the photo-Fenton process. It was observed that the value of *k* increased with turbidity removal in the pre-treatment of coagulation–flocculation, concerning the fastest process (photocatalysis heterogeneous), showing higher values with the coagulants SICOAG C-21 and FLOCUSOL-PA/18, which removed more than 90.0% of turbidity. However, the value of *k* was almost 0.013 with SIFLOC C-30, which only removed 9.5% of turbidity. This trend is similar in all processes, as shown in Table 3, as the effluent with lower turbidity showed a higher degradation rate, and the use of coagulation with SIFLOC C-30 (less turbidity removed) required more time for consumption.

TOC removal was influenced by the turbidity of the effluent of the AOP. It can be observed in Fig. 3, which relates the constant *k* with the turbidity removal in the coagulation–flocculation. The *k* values increase with the removal rate of turbidity of the pre-treatment, independently of the process, with the removal rate of TOC being higher when the influent showed a lower turbidity. Thus, the efficiency of TOC removal in AOP increases when the turbidity of the influent decreases. The use of coagulation–flocculation as pre-treatment of influent with high turbidity can improve the efficiency of the AOP.

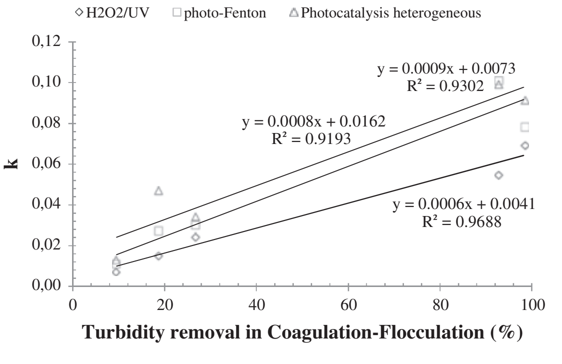

Fig. 3. Effect of turbidity removal on coagulation–flocculation of each AOP.

Bearing in mind the results of organic matter removal obtained by López-López et al. [41] in a conventional AOP with the same pH and concentration of oxidant for treating textile wastewater, the improvement of the pre-treatment can be observed. Reductions of the HRT necessary to obtain the same or higher removal rate, with 120 min of duration of a conventional AOP, were got. The organic matter removal rate was higher after 15 min of AOP with the SICOAG C-21 (92.9% of turbidity removal rate). However, the time required to get a higher removal rate of organic matter was at least 105 min with the SIFLOC C-30 (9.5% of turbidity removal rate). Therefore, the reduction of HRT decreased with the coagulant turbidity removal rate.

Módenes et al. [42] reported a COD removal of around 90.0% for the treatment of a tannery effluent with the photo-Fenton process within 120 min, using a pH value of 3, a H_2O_2 concentration range of 15–30 g/L and a Fe^{2+} concentration range of 0.375–0.50 g/L. The HRT result also reduced with the use of pre-treatment when compared with higher H_2O_2 and Fe^{2+} concentrations. Furthermore, only 15 min was necessary to achieve the same removal rate with SICOAG C-21, whereas the HRT increased to 105 min with the coagulant with a lower turbidity removal rate (SIFLOC C-30). Thus, the use of a pre-treatment such as CF enabled a reduction of the HRT.

4. Conclusions

The following conclusions were drawn from the study of a combined treatment with coagulation–flocculation and AOPs:

(1) The coagulation–flocculation is a reliable technology for removing the turbidity of textile wastewater and efficiencies between 9.5 and 98.2% were obtained, being the highest efficiency obtained with FLOCUSOL-PA/18.
(2) A TOC removal rate between 61.6 and 94.2% were obtained, being the highest rate obtained in the H_2O_2/UV process after SICOAG C-21 and in the heterogeneous photocatalysis after pre-treatment with FLOCUSOL-CM/1, respectively.
(3) Colour was removed almost totally with the different AOPs tested, being the highest efficiency of 99.3% in the heterogeneous photocatalysis after pre-treatment with SIFLOC C-30.

(4) The highest overall rate constant (*k*) was obtained in the photo-Fenton process after the use of SICOAG C-21 as coagulant, although the most efficient process was the heterogeneous photocatalysis.
(5) In relation to the above, the use of coagulation–flocculation as a pre-treatment of the influent with high turbidity can improve the efficiency of the AOP, reducing the HRT.
(6) The reduction of HRT decreased with the coagulant turbidity removal rate. The same removal rate was achieved with SICOAG C-21 at 15 min, so a correct selection of coagulation–flocculation process as a pre-treatment can reduce the operation cost of AOP system.

In general, the use of coagulation–flocculation processes as a pre-treatment of an AOP increases the efficiency of the AOP as a consequence of the decrease of turbidity in the influent.

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