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Comparing treatment methods that remove color from the effluent of an Organized Industrial District (OID)

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ABSTRACT

This study investigates the treatability, in terms of color removal, of an effluent taken from an Organized Industrial District wastewater treatment plant located in Bursa, western Turkey. To remove the color, chemical coagulation, Fenton and Fenton-like oxidation, ozonation, adsorption, and membrane processes were employed, and the results were compared. In coagulation experiments, maximum color removal (87.4%) was obtained at pH 12.2 with a dose of $240 \text{ mg/L} \text{ MgCl}_2 + \text{Ca}(\text{OH})_2$. Fenton and Fenton-like oxidation processes showed high color removal efficiencies (93.1 and 80%, respectively) from the evaluated effluents. The best color removal (93.6%) in this study was achieved by an ozonation process at a dose of 8 g/L-min ozone with a pH of 12 and a contact time of 3 min. In addition to the color removal efficiencies for the applied processes, their associated operating costs were also calculated.

Keywords: Adsorption; Chemical coagulation; Color removal; Fenton and Fenton-like oxidation; Membrane; Organized Industrial District wastewater; Ozonation

1. Introduction

Organized Industrial Districts (OIDs), which are planned and located in appropriate areas of industry, have been applied in Turkey to prevent environmental problems and to use the available resources economically [1]. According to the results obtained from 134 OIDs in Turkey, 161 million m³ of wastewater was treated in 2010 at OIDs [2]. An OID includes different companies, establishes connections between the companies, and gathers different types of wastewater in a single wastewater treatment system [3].

Today in Turkey, 261 OIDs have become a legal entity. Whereas there were only 70 completed OID

projects before 2002, 77 OID projects have been completed over the last 10 years, increasing the total number of existent OID projects to 147. Work on the infrastructure and treatment plants for 65 OIDs has continued under a 2012 investment program. Fifteen of these OID projects were planned to be completed by the end of 2012 [4].

Wastewater treatment plants serving an OID generally reduce emissions to water, and the operation of these systems has its own environmental effects. Appropriate wastewater treatment plants should be established to implement suitable environmental strategies, improve water quality, and allow water reuse. Wastewater originating from the operation of OIDs must be treated in accordance with the legal standards for the discharge area [5].

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Color occurs in wastewater for numerous reasons, such as the degradation of organic matter, the presence of resistant chemicals, the presence of metallic ions, the discharge of industrial wastewater to surface waters, the ability of persistent organic compounds to survive wastewater treatment plants, and the implementation of unsuitable methods in dyeing processes.

According to current standards, color, which causes esthetic problems and limits water reuse, is a limiting parameter for the discharge of wastewater. Dyes, the source of the color, interfere with biological process and are resistant to photo, thermal, and oxidative degradation. Color poses problems for wastewater treatment because of the potential toxicity and its inability to be degraded by traditional biological treatment methods.

Color, in addition to other parameters found in wastewater, should be treated. To achieve this purpose, appropriate color removal processes should be determined. Both color and toxicity should be removed. Numerous color removal methods exist, such as physical, chemical, and biological processes. The most commonly used methods are chemical coagulation/flocculation [6] and aerobic/anaerobic biological treatment [7,8]. Additionally, new methods have been employed, such as adsorption [9], ion exchange [10], electrochemical methods [11], advanced oxidation processes [12–15], and redox mediators [16]. However, single-stage treatment methods removing color are not sufficient to achieve environmental discharge criteria. In the literature, numerous combined processes have been examined to remove color, such as chemical and physicochemical processes [17], aerobic and anaerobic biological treatment [18], activated sludge treatment with nanofiltration [19], electrochemical and advanced oxidation processes [20], nanofiltration and reverse osmosis [21,22], ozonation-coagulation processes [23], and advanced oxidation and biological treatment [24].

In studies investigating the removal of color from wastewater by alternative methods, higher color removal efficiencies have been obtained. Gao et al. [25] employed different coagulants, such as $MgCl_2$, $Al_2(SO_4)_3$, PAC, and FeSO₄, to remove the color from a dye-containing synthetic wastewater. MgCl₂ effectively removed the color compared to other coagulants. Kang et al. [26] investigated the effectiveness of Fenton, Fenton-like oxidation, and coagulation processes on the color removal from a synthetic textile wastewater and found that the Fenton process is an appropriate method for this type of wastewater. In another study conducted by Lopes et al. [27], nanofiltration membranes in a pilot-scale study removed approximately 99% of the color from a textile wastewater. Sarayu et al. [28] obtained 95-99% color removal by ozonation in a semi-batch reactor, whereas

90% of the color was removed by GAC adsorption [29].

The number of studies investigating the removal of color from OID effluents is limited. Üstün and Akal Solmaz [30] investigated the effectiveness of the Fenton process in removing color from OID wastewater, and 96% of the color was removed at a pH of 4 with a 1/5 M ratio of Fe²⁺/H₂O₂. Ozonation and Fenton processes have been used to remove the color from an OID effluent by Kestioğlu et al. [31], and these processes removed more than 90% of the color. Therefore, one general aim of this study is to increase the number of published studies investigating color removal from OID effluents. The main objective of this study is to investigate color removal from an OID effluent by chemical coagulation, Fenton and Fenton-like oxidation, ozonation, adsorption, and membrane processes. In addition to this purpose, the applied processes, except membrane and ozonation, were evaluated in terms of operating costs.

2. Material and methods

2.1. Wastewater samples

Wastewater samples investigated in this study were obtained from the effluent of an OID located in Bursa (western Turkey). The samples were analyzed in accordance to Standard Methods [32].

Wastewater samples generated from different industries are characterized as mixed industrial wastewaters, and these mixed wastewaters are discharged after physical, chemical, and biological treatments. The characteristics of wastewater vary over time because of fluctuating plant operating parameters and climatic conditions.

2.2. Experimental procedure

To remove color from the wastewater in this study, chemical coagulation, Fenton and Fenton-like oxidation, and adsorption processes were applied at a laboratory scale. Additionally, ozonation and membrane processes were applied at a pilot scale. For all these methods, color was measured at 456 nm using a spectrophotometer (DR 2800, HACH LANGE Spectrophotometer, Bad Neuenahr, Germany) after filtering the supernatant through 0.45-µm filters.

2.2.1. Chemical coagulation experiments

In chemical coagulation experiments, employing a Jar Test apparatus (Velp-Scientifica C6F model, Italy), $Ca(OH)_2$, NaOH, $MgCl_2 + Ca(OH)_2$, and $MgCl_2 + NaOH$

Table 1 Characterization of wastewater samples

Parameter	Unit	Value
pН	_	7.5–8.1
Color	Pt-Co	350-600
TSS	mg/L	12-232
Conductivity	mŠ/cm	2.81-3.37

reagents were used at varying doses between 60 and 240 mg/L MgCl₂, and Al₂(SO₄)₃18H₂O, FeCl₃, and FeSO₄ (supplied from Merck, Germany) reagents were used at varying doses between 50 and 500 mg/L. The optimal pH and reagent doses to achieve the best color removal were determined for each chemical at room temperature (20 °C). Five-hundred milliliters of the wastewater sample (described in Table 1) were separately dosed with each chemical at varying concentrations. The samples were then flash mixed (3 min at 30 rpm), coagulated (30 min at 30 rpm), and allowed to settle (45 min). After sedimentation, the supernatant was analyzed in accordance with Standard Methods [32]. To adjust the pH, 1 M H₂SO₄, 1 M NaOH, and 10% Ca(OH)₂ were added in appropriate amounts.

2.2.2. Fenton and Fenton-like experiments

Fenton and Fenton-like experiments were performed at room temperature using varying dose of FeSO₄·7H₂O-H₂O₂ (for Fenton experiments) and FeCl₃·6H₂O-H₂O₂ (for Fenton-like experiments) at varying pHs to determine the optimal dose that provided the best color removal. The pH was manually adjusted to a desired range (pH 2-6) using 1 M sulfuric acid and/or sodium hydroxide before starting the experiments. During the determination of the optimal pH, the concentrations of FeCl₃·6H₂O, FeSO₄·7H₂O (supplied from Merck), and H₂O₂ (supplied from Merck, 35%, w/w) were fixed at 200, 100, and 100 mg/L, respectively. During the optimization of the chemical additives, H_2O_2 , FeSO₄·7H₂O, and FeCl₃·6H₂O doses varied between 50 and 300 mg/L.

2.2.3. Ozonation experiments

The ozonation process employed an ozone generator (TOGC8X, Triogen Ozone Generator (8 g/h) Triogen, UK) on a pilot-scale test. To determine the optimal pH, the initial pH of the sample was adjusted to a desired pH value (3–12) by adding 1 M NaOH and 1 M H_2SO_4 , and ozone was applied for 2 min to each wastewater sample. To determine the optimal contact time and dose of ozone, wastewater samples were exposed to ozone for 0.5, 1, 2, and 3 min.

2.2.4. Adsorption experiments

The adsorption experiments employed powderedactivated carbon (PAC) and granular-activated carbon (GAC). For this purpose, 1 L jars were used, and the pH of the wastewater was adjusted to be in the range of 4–10 in the Jar Test apparatus. Separately, 1 g/L PAC and 1 g/L GAC were added to the vessels and mixed for 24 h to determine the optimal pH.

After determining the optimal pH, the pH of the wastewater samples was adjusted to the desired value and 0.2-2 g/L PAC (adsorption experiments with PAC), and 1-2 g/L GAC (adsorption experiments with GAC) as added to the vessels to determine the optimal dose to achieve the best color removal efficiencies. At the end of the 24 h reaction, the color in each vessel was measured after filtration.

2.2.5. Membrane experiments

The pilot-scale membrane processes consisted of three different membrane units, and color removal was achieved with three different combinations of membranes. The first process employed a pressure sand filter (77/28 F 2175 MM/AC, Aquamatch pressure sand filter (5 m³/h), Aquamatch, Turkey), the second combination employed a pressure sand filter + microfiltration (Fokuswater bag filter microfiltration (5 μ m pore size), Fokuswater, Turkey), and the third combination employed a pressure sand filter + microfiltration (MINI UF 4 Aquamatch ultrafiltration (5.1–6.4 m³/h), Aquamatch, Turkey).

3. Results and discussion

3.1. Physicochemical treatability studies

3.1.1. Determination of the optimal pH

In the chemical coagulation experiments, $Ca(OH)_2$, NaOH, MgCl₂ + Ca(OH)₂, and MgCl₂ + NaOH reagents were used, and the optimal pH was determined to be 12.2 for each of these coagulants (Fig. 1(a)). Sedimentation at pH 10.2 did not occur because of turbidity, and the results obtained were worse than the initial color values. In a study conducted by Gao et al. [25], the color removal efficiency of MgCl₂/Ca(OH)₂ was compared with that of Al₂(SO₄)₃, polyaluminum chloride (PAC), and FeSO₄/Ca(OH)₂.

According to the experimental results, the color removal efficiency of MgCl₂ was related to the type of dye, depended on the pH of the waste, and responded to the dose of the coagulants. Treatment of wastes containing reactive or dispersed dyes with MgCl₂ yielded an optimal color removal ratio when the pH



Fig. 1. Effect of pH on color removal during coagulation experiments.

of the solution was equal to or above 12. For both the reactive and dispersed dye wastes, $MgCl_2/Ca(OH)_2$ was shown to be superior to $MgCl_2/NaOH$, $Al_2(SO_4)_3$, PAC, and $FeSO_4/Ca(OH)_2$ in its ability to remove color. According to Fig. 1(b), the optimal pH values for the experiments adding $FeCl_3$, $FeSO_4$, or $Al_2(SO_4)_3.18H_2O$ were determined as 4.5, 10.5, or 6, respectively.

3.1.2. Determining the optimal coagulant dose

From the coagulation experiments, the color removal efficiencies at a pH of 12.2 for various coagulants at an optimal dose were found to be as follows: 78% for MgCl₂+NaOH and 87.4% for MgCl₂+Ca(OH)₂ (Fig. 2). The best color removal efficiencies were 67.4, 47, and 82.6% for FeCl₃ (300 mg/L), FeSO₄ (400 mg/L), and alum (400 mg/L), respectively. These removal efficiencies are compatible with those reported in the literature. Selcuk [33] evaluated and compared the performance of ferrous and aluminum sulfate coagulants on a textile wastewater; approximately 50-60% of the color was removed at 1,000 and 1,500 mg/L ferrous and aluminum sulfate, respectively. Tan et al. [34] used MgCl₂ as a coagulant to investigate the effectiveness of chemical precipitating coloring agents. The results showed that MgCl₂ is capable of removing more than 90% of the coloring material at a pH of 11 with a dose of 4 g MgCl₂ per L of dye solution. In another study, color removal experiments in a pilot plant were performed by Georgiou et al. [35], and treatment with lime alone proved to be effective in removing the color (70–90%) from textile wastewater. Additionally, treatment with ferrous sulfate with a pH maintained in the range 9 ± 0.5 using lime was equally effective.

In this study, the best color removal efficiency was obtained at a pH > 12 with a $MgCl_2 + Ca(OH)_2$ dose of 240 mg/L MgCl₂, the optimum dose from the coagulation experiments. The differences in the color between raw and treated wastewater are shown in Fig. 3.

3.2. Fenton and Fenton-like processes

Fenton and Fenton-like processes have been used to treat wastewater because of the ease of operation, simple system, cost efficiency, and ability to operate under a wide range of temperatures [36]. These methods employ ferrous or ferric salts and hydrogen peroxide under acidic conditions in which Fe^{2+} as oxidized to Fe^{3+} is given in Eq. (1) (for Fenton process) and Fe^{3+} is reduced to Fe^{2+} as given in Eq. (2) (for Fenton-like process). This process produces a strong oxidizing agent (hydroxyl radical, \cdot^{OH}) [4,37].

$$H_2O_2 + Fe^{2+} \to Fe^{3+} + HO^- + HO$$
 (1)

$$H_2O_2 + Fe^{3+} \rightarrow Fe^{2+} + HO_2 + HO$$
 (2)

The operational parameters that directly affect the efficiency of these processes are the concentrations of ferrous or ferric salt, concentration of hydrogen peroxide, ratio between the concentrations of the ferrous or ferric salt and hydrogen peroxide, pH, temperature, and reaction time [38]. The pH is the important parameter affecting the processes, as notably acidic pH values (near 3) are usually optimal for Fenton oxidation reactions [39]. Whereas higher pH values hinder Fenton reactions, a pH below the optimal pH can inhibit oxidation [4].



Fig. 2. Effect of coagulant dose on color removal during coagulation experiments.



Fig. 3. Appearance of raw and treated wastewaters followed by coagulation experiments.

The optimal pH was determined at the beginning of this study. The pH was adjusted to between 2 and 6, and the color removal efficiencies were observed. The maximum color removal efficiency was achieved at a pH of 3 (for Fenton process) and 4 (for Fenton-like process) (Fig. 4(a)). From Fig. 4(a), at these pH values (200 mg/L FeSO_4 ·7H₂O and 100 mg/L H_2O_2 for Fenton-like process), the color removal efficiencies from the effluent of the OID was calculated to be 85.3% for the Fenton process and 51.6% for the Fenton-like process, respectively.

At a fixed pH (pH 3 for Fenton and pH 4 for Fenton-like experiments) and a constant H_2O_2 concentration of 100 mg/L, varied doses of FeSO₄·7H₂O and FeCl₃·6H₂O between 50 and 300 mg/L were used to determine the optimal dose for Fenton and Fenton-like processes. The color removal efficiency for the constant concentration of hydrogen peroxide and varied doses of FeSO₄·7H₂O and FeCl₃·6H₂O is illustrated in Fig. 4(b). Optimal FeSO₄·7H₂O and FeCl₃·6H₂O doses were determined as follows: CFeSO₄ = 250 mg/L for Fenton process and CFeCl₃ = 300 mg/L for Fenton-like process in this study.

The other significant step was to determine the optimal H_2O_2 dose for Fenton and Fenton-like processes. Optimal H_2O_2 dose determination experiments were performed at constant FeSO₄·7H₂O doses and pH values. Varying doses of H_2O_2 (between 50 and 300 mg/L) were applied. Color removal efficiencies at varied doses of H_2O_2 and constant concentrations of FeSO₄ and FeCl₃ are shown in Fig. 4(c). From Fig. 4(c), optimal conditions for Fenton and Fenton-like processes were determined to occur at a pH of 3, CH₂O₂ = 250 mg/L and CFeSO₄ = 200 mg/L (93.1% color removal), and at a pH of 4, CH₂O₂ = 300 mg/L and CFeCl₃ = 250 mg/L (80% color removal), respectively.

In a study done performed by Meriç et al. [40], the removal of Reactive Black 5 (RB5) by a Fenton oxidation process was investigated and an optimal pH and temperature for 100 mg/L of RB5 were found to be 3 and 40 °C, respectively. With 100 mg/L of FeSO₄ and 400 mg/L of H₂O₂, 71% of COD and 99% of the color was removed. Üstün and Akal Solmaz [30] investigated the Fenton process's efficiency on color and COD removal from the effluent of an OID and achieved lower concentrations than the color limits given by EN ISO 7887, and approximately 50% COD removal with a 80 mg/L Fe²⁺ and 50 mg/L H₂O₂ at pH 4.

 $FeSO_4$ ·7H₂O is used in both chemical coagulation and Fenton oxidation processes. The color removal in chemical coagulation process is only 47% instead in Fenton oxidation process is 93%. Fenton process employs ferrous and hydrogen peroxide under acidic conditions in which Fe^{2+} is oxidized to Fe^{3+} . This process produces a strong oxidizing agent, the hydroxyl (hydroxyl radical, ^{oOH}) is a powerful, non-selective



Fig. 4. (a) Effect of pH on color removal during Fenton and Fenton-like processes (CFeSO₄ = 200 mg/L, CFeCl₃ = 100 mg/L and CH₂O₂ = 100 mg/L). (b) The effect of FeSO₄ and FeCl₃ concentrations on color removal efficiencies during Fenton and Fenton-like processes (pH 3 and CH₂O₂ = 100 mg/L for Fenton process, pH 4 and CH₂O₂ = 100 mg/L for Fenton-like process). (c) The effect of the concentration of H₂O₂ on color removal efficiencies during Fenton and Fenton-like process (pH 3 and CFeSO₄ = 200 mg/L) (93.1% color removal) Fenton-like process (pH 4 and CFeCl₃ = 250 mg/L) (80% color removal)).

oxidant, so it is effective in organic and industrial wastewater components.

3.3. Ozonation process

Ozonation is an effective AOP to remove refractory and/or toxic chemicals from water and wastewater [41]. Ozone itself is a strong oxidizing agent and has a high color removal efficiency, which is effective at high pH values [12]. Alkaline solutions accelerate ozone decomposition [42].

In this study, the ozonation process resulted in a color removal efficiency of 93.6% at pH 12 with an ozone dose of 0.534 g/L during 2 min of contact time. For determining the optimal contact time at pH 12, ozone was applied for 3 min, and the color removal efficiency was found to be 93.6%, which is similar with the respective findings of other studies (Fig. 5). Significant decolorization occurred during ozonation, resulting in more than a 95% reduction in color [43]. Sarayu et al. [28] studied decolorization of various azo dyes by ozonation in a semi-batch reactor and obtained 95-99% color removal at pH 10 with an ozone dose of 4.33 mg/L and a contact time of 30 min. In another study, an ozonation process was used to treat textile wastewater and over 98% of the color was removed after 20 min of ozone contact time [33].

3.4. Adsorption processes

Adsorption experiments were performed with two different activated carbons: PAC and GAC. The optimal pH was determined to be 4 and 3 for a 60 min contact time for PAC and GAC, respectively. The color removal efficiency increased with increasing doses of PAC and GAC. The best color removal efficiency achieved was 85.2% using 2 g/L PAC and 63.7% using 2 g/L GAC (Fig. 6). Hassani et al. [29] investigated the effectiveness of the GAC adsorption process for dye removal from textile wastewater. The doses of GAC that are used for 5 mg/L of dye vary between 0.2 and 0.7 g. The results showed that GAC cannot remove dispersed red dye. Acidic red, direct red, and reactive red at a concentration of 5 mg/L were removed by GAC at an efficiency up to 90, 88, and 43% in 30, 60, and 120 min, respectively. In another study conducted by Guendy [44], the removal of dye from textile wastewater by adsorption increased from 75 to 90% and from 80 to 95% for 2.5 and 5 g GAC, respectively, by decreasing the dye's initial concentration from 50 to 10 ppm at a pH of 8 and with 120 min of contact time at room temperature.

3.5. Membrane processes

As shown in Fig. 7, the best color removal efficiency of 61% was achieved with the BKF+MF+UF



Fig. 5. Results of the ozonation process (a) 0.534 g ozone/L, (b) pH 12.



Fig. 6. Results of adsorption experiments.

combination and 2 min of contact time. In the absence of UF, the color removal efficiency achieved by BKF+MF and BKF were determined to be 11.7 and 8.8%, respectively.

Lopes et al. [27] investigated textile wastewater treated by a nanofiltration-membrane pilot-unit and obtained a color removal efficiency of 99%. The color removal of a biologically treated textile effluent through submerged filtration using a nanofiltration membrane was investigated by Zheng et al. [45]. Under the trans-membrane pressures of 0.8 bars and a volume concentrating factor of 4.0, the submerged nanofiltration system exhibited a steady permeate flux of $5.151/m^2h$ with a color removal efficiency of 99.3%.

Process	Reagents	Basis	Cost (€/kg)	Reagent cost (€/d)	Total chemical cost (€/d)	Average treatment cost (€/m ³)	Color removal (%)
Chemical coagulation	Ca(OH) ₂	kg	0.064	121.6	213.9	0.32	82
	NaOH	kg	0.76	568.9	762.9		63.1
	MgCl ₂ ·6H ₂ O + Ca(OH) ₂	kg	0.33	79.5	289.6		87.4
	Al ₂ (SO ₄) ₃ ·18H ₂ O	kg	0.24	94.4	329.8		82.6
	FeCl ₃ ·6H ₂ O	kg	0.29	84.9	402.1		67.4
	FeSO ₄ ·7H ₂ O	kg	0.16	82.6	315.3		47
Fenton oxidation	FeSO ₄ ·7H ₂ O	kg	0.16	33.04	278.1	0.4	93.1
	H_2O_2	kg	0.55	138.1			
Fenton-like oxidation	FeCl ₃ ·6H ₂ O	kg	0.29	56.64	309.8	0.43	80
	H_2O_2	kg	0.55	165.7			
Ozonation	O_3	_	-	_	786.8 (acid+base)	-	93.6
Adsorption	GAC	kg	1.6	22,240	22347.5	0.51	63.7
	PAC	kg	3.1	6,136	6,378		85.2
Membrane process	PSF+MF+UF	-	-	_	-	-	61

 Table 2

 Operating costs of the studied processes^{a,b,c,d}

^aCost of labor and sludge disposal not included.

^bTreatment costs were calculated for a 1000 m3/d flow rate.

^cTotal chemical costs includes the chemicals used to adjust and neutralize the pH.

^dCosts associated with ozonation and membrane processes could not be determined because of insufficient data.



Fig. 7. Results of membrane processes.

3.6. Cost evaluation

The applied processes, except membrane and ozonation, were evaluated in terms of their operating costs (on the basis of the chemical requirements), and their cost profiles were compared in Table 2. The operating costs of the processes were found to be reasonable. The lowest operating cost, which belongs to the chemical coagulation process, was calculated to be $0.32 \notin /m^3$.

4. Conclusions

In this study, chemical coagulation, Fenton and Fenton-like oxidation, ozonation, adsorption, and membrane processes were used to decolorize the effluent of an OID wastewater treatment plant that lacked a previous color removal process. The following conclusions can be drawn from this study:

- Chemical coagulation using MgCl₂ was effective in removing over 87% of the color from an OID effluent.
- (2) Fenton and Fenton-like oxidation processes have shown satisfactory color removal on the evaluated samples.
- (3) The best color removal was achieved at high pH values (pH > 12) by the ozonation process, resulting in a color removal efficiency of 93.6%.
- (4) The pore diameter of the membranes was insufficient to filter the coloring agents. Therefore, a satisfactory color removal could not be achieved with membranes.
- (5) The operating cost of chemical coagulation with Ca(OH)₂, calculated as 0.32 €/m³, was the lowest for the applied processes.

Consequently, the best color removal in this study was obtained by both ozonation and Fenton oxidation.

However, the applications of these processes are limited because of the high operating costs and difficulty in controlling these processes. Therefore, a satisfactory color removal efficiency was obtained by chemical coagulation with Ca(OH)₂ compared with other processes. In addition, when considered in terms of operating costs, chemical coagulation with Ca(OH)₂ was determined to be the best alternative to effectively remove color from OID wastewaters.

Conflict of interest

The authors have declared no conflict of interest.

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