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Optimizing the removal of humic acid with polyaluminum chloride and polyaluminum ferric chloride as green coagulants using response surface methodology

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ABSTRACT

Humic acid is a major precursor of chlorinated byproducts that need to be removed from water, via treatment, given their adverse effects on human health. The current study aimed at identifying the optimal conditions to remove humic acid from surface water, using coagulants such as polyaluminum chloride (PACl) and polyaluminum ferric chloride (PAFCl). The effects of independent variables such as total organic carbon (TOC) concentration (1.6–7 mg/L), pH level (5–9), and coagulant dosage (10–50 mg/L) on humic acid removal were studied using response surface methodology and central composite design. A coagulant dose of 15.72 mg/L (for PACl and PAFCl), TOC concentration of 2.66 mg/L, and pH of 7.84 comprised the optimal conditions, and the removal efficiencies of 97.55% and 98.18% were obtained with PACl and PAFCl, respectively. Zeta potential analysis showed that the leading mechanism for coagulation, with both coagulants, was charge neutralization. PACl and PAFCl had adequate potential to remove humic acid from surface water, but PAFCl had a better performance regarding the treatment of aluminum residuals.

Keywords: Coagulation; Humic acid; Polyaluminum chloride; Polyaluminum ferric chloride

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1. Introduction

Natural organic matters are comprised of various compounds in water, especially surface water, where interactions between the hydrological cycle and biological activity produce humic substances [1,2]. The properties of these substances are different from those of other organic compounds, depending on environmental biogeochemical cycles [3–6]. Their presence can increase the levels of dissolved organic carbon (DOC), turbidity, chemical oxygen demand (COD), and chlorinated byproducts (CBPs) in water. Therefore, removing humic substances from water requires advanced treatment processes and facilities [3,4,7].

In many countries, the chlorine used to disinfect drinking water can react with humic substances and generate carcinogenic compounds such as haloacetic acids and trihalomethanes (THMs) [8,9]. According to the United States

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Environmental Protection Agency (USEPA), the highest permissible levels for HAAs and THMs in drinking water are 60 and 80 µg/L, respectively. Therefore, the removal of humic substances, especially humic acid, from drinking water is very important [10].

Some conventional techniques including membrane separation, chemical coagulation-flocculation, advanced oxidation, and hybrid processes are used to remove humic acid [8,11,12]. However, the cost-effectiveness and simplicity of treatment methods should also be considered [13,14]. The USEPA classified coagulation-flocculation as the best method to eliminate total organic carbon (TOC) from water. The coagulation-flocculation process was suggested in previous studies as a method to control disinfection byproduct (DBP) precursors [15–17].

The Al and Fe salts are the main coagulants consumed in water treatment industry. In the other studies alum, polyaluminum ferric chloride (PAFCl), Al_{13} polymer, and polyaluminium chloride (PACl), ferric chloride, and ferric sulfate were wildly used to remove humic acid and turbidity [18–23].

The current study aimed at investigating the removal of humic acid from surface water using the coagulation-flocculation method with polyaluminium ferric chloride (PAFCl) and PACl. To identify the best coagulant, the response surface methodology (RSM) was employed, with a focus on the zeta potential and coagulant dosages for humic acid removal. Finally, after the removal of humic acid in the optimum condition, trihalomethanes formation potential (THMFP) were determined.

2. Materials and methods

2.1. Chemicals

Humic acid (99%) was purchased from Sigma–Aldrich Co. PACl, methanol, Milli-Q water, NaOH, and HCl were provided by Merck Co. (Germany). $AlCl_3 \cdot 6H_2O$, $FeCl_3 \cdot 6H_2O$, and Na_2CO_3 were used to prepare PAFCl, and also $Na_2HPO_4 \cdot 12H_2O$ was added to the solution as a stabilizer, according to Cao et al. [24]. To prepare the stock humic acid solution (1000 mg/L), 1000 mg of humic acid was dissolved in distilled water (1 L) and then, diluted to 10 mg/L. Moreover, hydrochloric acid (0.1 N) and sodium hydroxide (0.1 N) were used to regulate pH. Methanol and Milli-Q water (0.05 µs/cm, Millipore) were used for the high-purity liquid chromatography (HPLC) and DOC experiments.

2.2. Sampling

The samples were taken from the Shaharchi River in Urmia city, Iran [25]. One hour after sampling, raw water samples were sent to the lab and maintained in the dark at 4°C to prevent biological activities before use. Also, the required diluted humic acid was prepared by adding different dosages of the stock humic acid solution into the raw water taken from Shaharchi River to produce TOC concentrations from 1.6 to 7 mg/L.

2.3. Experimental design

The Design-Expert (version 10) software was used to assess the impact of independent variables on humic acid removal (responses) with PACl (R1) and PAFCl (R2) (Table 1), using central composite design (CCD) and response surface methodology (RSM). RSM was used to study the effect of independent variables (Table 1) on the response (TOC removal efficiency) and optimize the variables. In this method, significant variables are firstly determined in a factorial design. Then, experiments are designed according to the Montgomery method in a central composite rotatable design for independent variables [26].

As described above, the humic acid solutions containing TOC (1.6–7 mg/L) were provided by adding TOC into the raw water (taken from Shaharchi River).

The variables were selected based on previous studies. The following empirical equations (second-order polynomial regression models) were specified based on the experimental results:

$$Y = \beta_0 + \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_{12}X_1X_2 + \beta_{13}X_1X_3 + \beta_{23}X_2X_3 \\ + \beta_{11}X^2{}_1 + \beta_{22}X_{22} + \beta_{33}X^2{}_3)$$

The predicted response (Y) was correlated with the set of regression coefficients (β): intercept (β0), interaction (β12, β13, and β23), linear (β1, β2, and β3), and quadratic (β11, β22, and β33). ANOVA was used to determine the goodness of fit and analyze interactions between responses and variables. The coefficient of determination (R^2) was measured to describe the quality of the polynomial model. A lack of fit (LOF) test was also performed to confirm the statistical significance of the results. An insignificant LOF was preferred ($P > 0.05$), which showed the validity of the model. The model terms were assessed at $P < 0.05$ with a 95% confidence interval (Dehghani et al., 2017).

2.4. Experimental procedure and analytical methods

The experiments were performed using jar tests (Hach Co. Ltd., USA), utilizing six 1000 mL beakers in which samples were flash mixed (coagulated) at 120 rpm for 120 s and slow-mixed (flocculated) at 40 rpm for 10 min, followed by sedimentation for 20 min [27]. The DOC levels of the treated water were measured using combustion techniques (method 5310B) in a TOC-VCSH analyzer (Shimadzu, Japan). An ultraviolet-visible spectrophotometer (DR-5000, Hach-Lange) was applied to measure UV absorption at a wavelength of 254 nm (method 5910B) [28].

After rapid mixing, 5.0 mL of the suspension was sampled for zeta potential using a zeta meter (Malvern, United Kingdom). Residual aluminum (Al), Fe, humic acid, and THM concentrations were determined using

Table 1
Independent variables range

Factors	Range
TOC (mg/L) (A)	1.6–7
Solution pH (B)	5–9
Coagulant dosage (mg/L) (C)	10–50

HPLC, inductively coupled plasma-mass spectrometry, and gas chromatography/mass spectrometry (GC/MS), respectively. To present the images and the elemental content of coagulants, energy-dispersive spectroscopy (EDX) along with scanning electron microscopy (PHENOM Pro X SEM, WEGA/TESCAN; Czech Republic) was employed.

2.5. Determination of trihalomethane formation potential (THMFM)

After determining the optimal conditions for humic acid removal, the THM formation potential was evaluated using the standard methods to examine water and wastewater [28]. In this test, chlorine was added to water treated with a coagulant to produce residual free chlorine concentrations above 3 mg/L [28]. After 7 days, the total THM level was measured according to USEPA method 524.2 (USEPA, 1995) using GC/MS (6890 N, Agilent, USA) combined with a purge and trap system [29].

2.6. Identification of optimal conditions

Optimization was accomplished based on the following criteria: TOC range 1.6–7 mg/L; pH 5–9; coagulant dosage 10–50 mg/L; and a maximum response for both R1 and R2.

3. Result

Fig. 1 shows SEM–EDX images of the coagulants (magnification 10,000X and 13,000X) to illustrate the surface morphology of the samples and the percentage weight of the elements. The humic acid removal efficiency of the coagulation-flocculation process was assessed using RMS as a function of independent variables including TOC (A), solution pH (B), and coagulant dosage (C). The removal efficiency was measured by summing the constant, three first-order effects, three second-order effects, and three interaction effects according to Eq. (1). To develop mathematical equations, the experiments were conducted in line with CCD, as presented in Table 2.

To determine the goodness of fit, ANOVA was applied. Based on the CCD and input variables, a second-order model was used for the experimental results. The final equations based on the coded factors are given in Table 3. The significance of the model was determined using the P values. According to Table 3, at a 5% confidence level, all the models were significant ($P < 0.05$).

The response surface plots for humic acid removal with PACl and PAFCl are shown in Figs. 3 and 4, respectively. To determine the effect of variables on removal efficiency, one variable was kept constant, while the others varied within the experimental ranges. Based on the coefficients in the regression equations (Table 3), coagulant dosage and pH value were the most significant variables and had the greatest effects on removal efficiency.

The optimal conditions for each variable are given in Fig. 5. Overall, optimal conditions produce maximum efficiency relative to the range of all variables.

The variations in zeta potential of the raw water and different dosages of PACl and PAFCl are shown in Fig. 6.

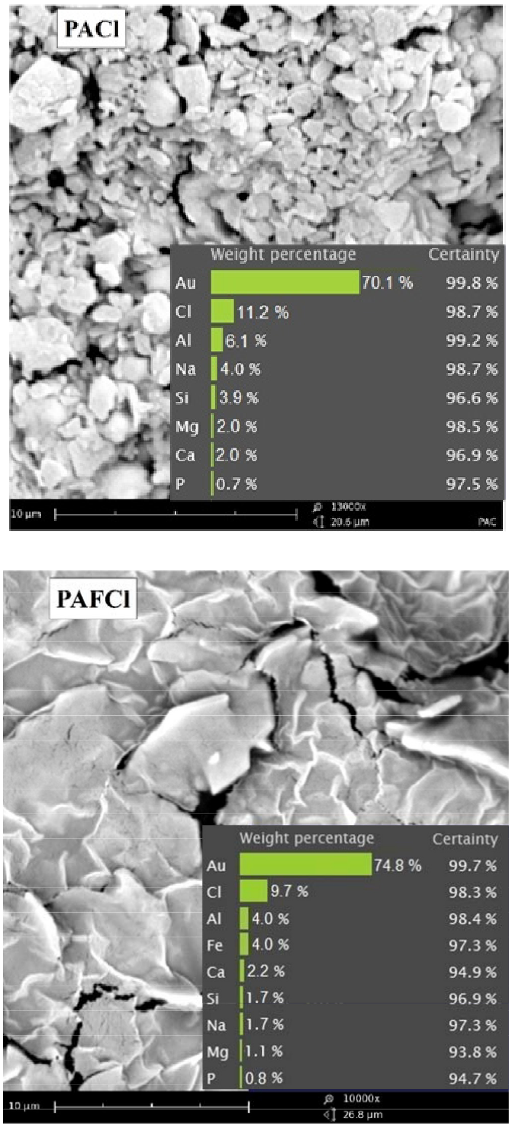

Fig. 1. SEM-EDX images of the coagulants.

4. Discussion

4.1. Characteristics of the coagulants

To determine and compare the coagulant morphology, Fig. 1 indicates that PAFCl had a higher percentage of the components than PACl, and Al levels in PACl exceeded those of the PAFCl. Zhang et al., [30] reported that PAFCl had larger branched chains and a better morphology than PACl; this result was similar to that of the current study. Therefore, the flocs produced by PAFCl were more homogeneous, and more structured sheets, compared with those of PACl.

4.2. Central Composite Design analysis

According to Table 3, a LOF value greater than 0.05 (ranging 0.11–0.16), R^2 value close to 1, and reasonable agreement with the adjusted R^2 indicated an acceptable fit between the experimental data points and values predicted

Table 2
Central composite design and observed responses (R_1: Humic acid removal using PACl, R_2: Humic acid removal using PAFCl)

Run	Actual factors			Humic acid removal (%)	
	A: TOC (mg/l)	B: pH	C: Coagulant dose (mg/l)	PACl	PAFCl
1	5.91	5.81	41.89	69	74
2	2.69	8.19	18.11	80	85
3	4.30	7.00	30.00	88	93
4	5.91	8.19	18.11	71	76
5	2.69	5.81	18.11	58	63
6	2.69	8.19	41.89	43	51
7	4.30	7.00	30.00	77	81
8	2.69	5.81	41.89	27	45
9	4.30	7.00	30.00	86	91
10	5.91	5.81	18.11	60	65
11	4.30	7.00	30.00	85	90
12	5.91	8.19	41.89	56	61
13	4.30	7.00	30.00	88	93
14	7.00	7.00	30.00	92	97
15	4.30	7.00	10.00	95	93
16	4.30	5.00	30.00	30	35
17	4.30	7.00	30.00	89	94
18	4.30	7.00	50.00	66	71
19	4.30	9.00	30.00	70	75
20	1.60	7.00	30.00	82	87

by the quadratic model. Contrasts in the associated error of the predicted response can be measured as adequate precision (signal-to-noise ratio). The optimum signal-to-noise ratio is 4 or above; larger ratios (e g, 13.31 and 11.48) indicate an adequate signal, as shown in Table 3 [31,32].

The standard error-to-mean response ratio is described as the coefficient of variance (CV), which is an index of reproducibility. A CV less than 10% indicates that the model is reproducible [31]. In Table 3, both models are shown as reproducible, since they have CVs less than 10% (9.40 and 8.44). Fig. 2 presents the predicted diagnostic plots versus actual removal efficiency. These plots indicate the acceptable agreement of experimental data with the predicted values in the quadratic model.

4.3. Process optimization and effects of various variables

Optimal removal was reported when the TOC level was 2.66 mg/L, pH was 7.84, and coagulant dosage was 15.72 mg/L, with removal efficiencies of 97.55% and 98.18% for PACl and PAFCl, respectively; these are the peaks in Figs. 3 and 4. Removal efficiencies reduced with deviations from the most favorable conditions; in other words, a rise or fall in any variable can decrease the responses.

In a study by Nourmoradi et al. [8] maximum humic acid adsorption was reported in a solution of nanozeolite and nanocarbon with a pH of 5 [8]. Edzwald and Benschoten [33] showed that humic acid adsorption by metal hydroxide precipitates depends on a solution with a high pH and coagulant dosage. The main mechanism at lower pH and coagulant dosages was the precipitation of humic

Table 3
The ANOVA results for response parameters (R_1: Humic acid removal using PACl, R_2: Humic acid removal using PAFCl)

Response	Final equation in terms of coded factors	p	PLOF	R^2	Adj. R^2	AP	S.D	CV	PRESS
R_1	$87.37 + 4.75A + 7.5B - 8.99C - 5.00AB + 7.75AC - 3.75BC - 2.54A^2 - 15.62B^2 - 4.83C^2$	0.0001	0.1147	0.9439	0.8877	13.310	6.64	9.40	3106.21
R_2	$91.55 + 3.53A + 6.83B - 6.95C - 3.75AB + 5.67AC - 5.00BC - 1.77A^2 - 14.86B^2 - 5.25C^2$	0.0002	0.1657	0.9365	0.8729	11.479	6.39	8.44	2967.26

P: probability of error; PLOF: probability of lack of fit; AP: adequate precision; S.D.: standard deviation; CV: coefficient of variance; PRESS: predicted residual error sum of squares.

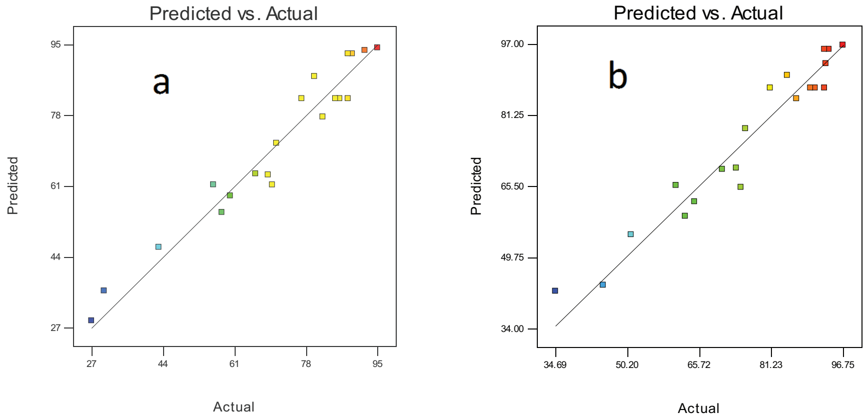

Fig. 2. Design-expert plot; predicted vs. actual values plot for humic acid removal (a using PACL, b using PAFCl).

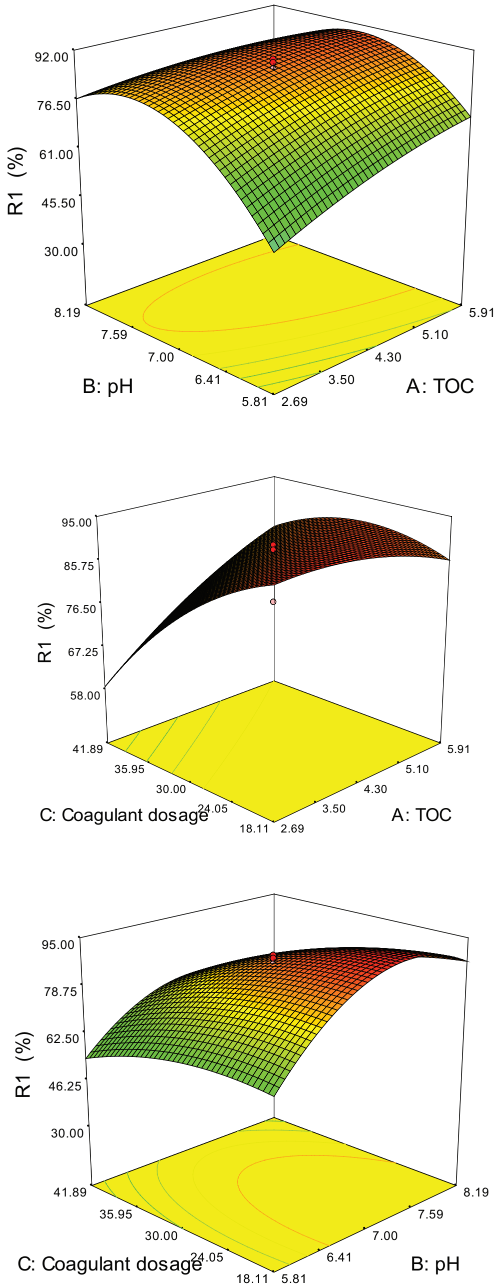

Fig. 3. Design-expert plot; response surface plot for humic acid removal using PACl.

acid-metal complexes. In the current study, removal efficiencies at the optimal points in the experimental settings were 92% and 95% for PACl and PAFCl, respectively. The efficiencies measured in the experiments and estimated by the model had close agreement.

4.4. Surface charge and coagulation mechanism of PACl and PAFCl

Adding coagulants to raw water increased its zeta potential. The zeta potential of water containing the optimal doses of PACl and PAFCl (15.72 mg/L) were –2 and –5 mV, respectively. These values indicated the high surface charge of the coagulants as well as their strong ability to neutralize charges. In similar studies, zeta potential values to remove humic acid and turbidity using alum, ferric sulfate, ferric chloride, Al_{13} polymer, and PACl were reported near zero in optimum conditions [7,19,20,34]. These result were in agreement with the current study observations. However, in some researches zeta potential values were

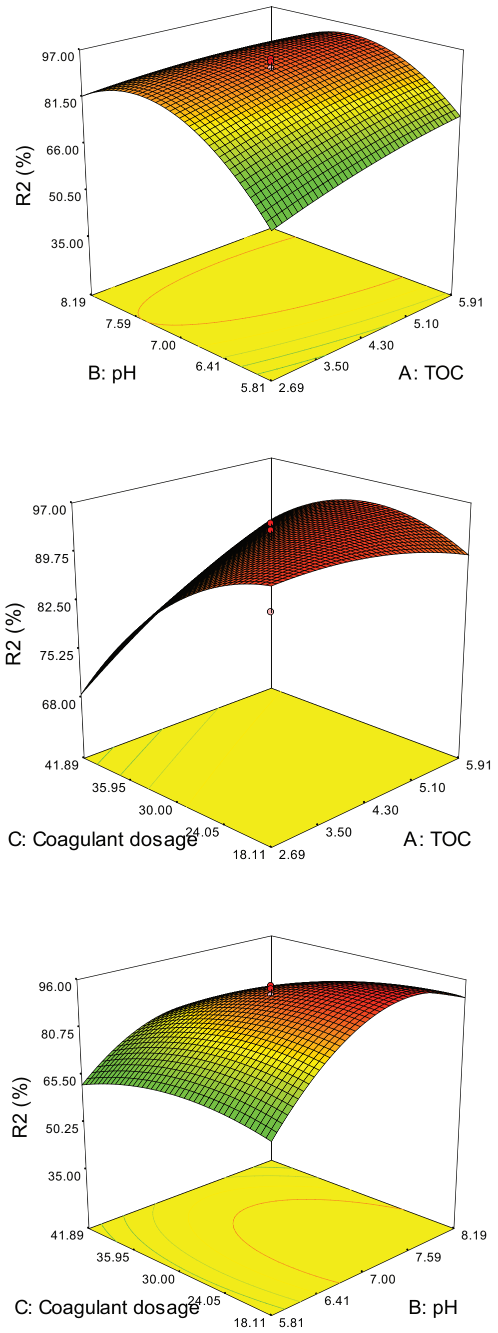

Fig. 4. Design-expert plot; response surface plot for humic acid removal using PAFCl.

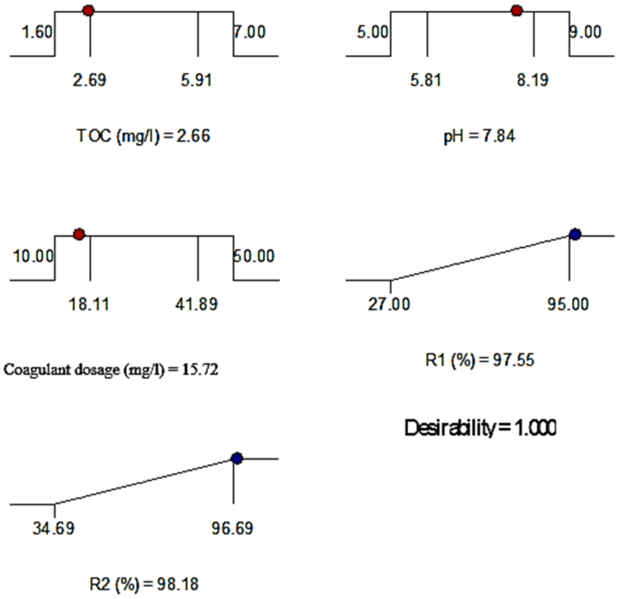

Fig. 5. Design-expert plot; optimization plot (R_1: humic acid removal using PACL, R_2: humic acid removal using PAFCl.

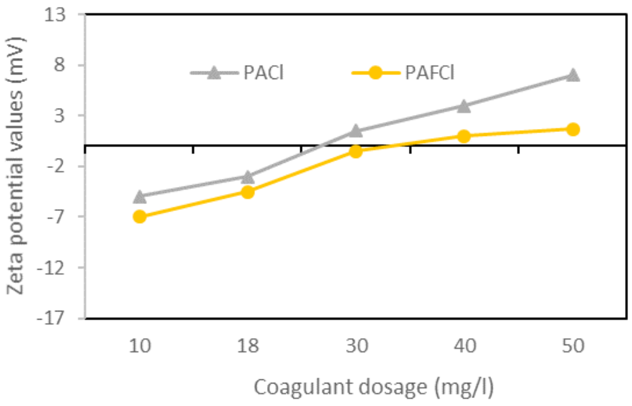

Fig. 6. Zeta potential values in raw water and different dosages of coagulant.

exactly reported zero in the highest removal efficiency of humic acid [34,35], which it is comparable to the current study findings. This difference can be attributed to the range of coagulant consumption dosages.

Based on the zeta potential (near zero for PAFC) of the optimal coagulant dosage, charge neutralization was the major mechanism of humic acid removal by PACl and PAFCl. In the studies of colloidal particle, turbidity, and organic matter removal with PACl and PAFCl, the major mechanisms of coagulation were charge neutralization, bridge connection, and sweep flocculation [24,30,36,37].

4.5. Effects of aluminum coagulant type on water quality

Al residuals and control of CBPs precursors are among the major challenges of using coagulants to treat drinking water [12]. Some characteristics of raw and treated water at optimal dosages of PACl and PAFCl are shown in Table 4. After coagulation at these doses, Al residuals and THM formation potential were lower than the USEPA and World Health Organization recommendations [38,39].

Table 4
Some characteristics of water before and after coagulation in optimum condition

Parameter	Before coagulation	After coagulation	
		PACl	PAFCl
DOC (mg/l)	1–3	0.86 (±0.032)	0.7 (±0.013)
Humic acid (mg/l)	0.2–0.3	<0.1	<0.1
Color (Pt. Co.)	<1	<1	<1
EC (µS/cm)	220– 250	270 (±1.3)	310 (±1.21)
Turbidity (NTU)	1–5	<1	<1
Alkalinity (mg/l as $CaCO_3$)	100–140	120	110
SUVA* (l/mg·m)	2–5	<2	<2
pH	7–8	7–8	7–8
Al (mg/l)	<0.05	0.01 (±0.05)	0.009 (±0.002)
Fe (mg/l)	<0.2	0.1 (±0.01)	0.07 (±0.003)
THMFP** (µg/l)	>100	42	35

*Specific UV absorbance (UV254/DOC). ** Trihalomethanes formation potential.

Specific UV absorbance (SUVA; UV_{254}/DOC) is a good index to describe the potential for CBPs formation in water. Water samples with SUVAs higher than 4 L/mg/min generally had a greater molecular weight and were of more hydrophobic nature than humic substances, with a greater potential to cause CBPs production [40]. As shown in Table 4, both PACl and PAFCl had relatively good potential to decrease SUVA and THMs formation potential, whereas the use of PAFCl resulted in a lower concentration of Al residuals in treated water compared with the use of PACl. This finding was similar to those of the studies on turbidity and color removal with PAFCl and PACl [12,41].

5. Conclusion

The optimal conditions were TOC level of 2.66 mg/L, pH 7.84, and coagulant dosage of 15.72 mg/L, with removal efficiencies of 97.55% and 98.18% for PACl and PAFCl, respectively. Based on the zeta potential in the optimal conditions, charge neutralization was the main coagulation mechanism for humic acid removal with PACl and PAFCl. These coagulants also showed good potential to reduce the SUVA index, although PAFCl had an advantage in terms of removing Al residuals.

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