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Evaluation of a natural coagulant in the polishing treatment of swine slaughterhouse wastewater

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ABSTRACT

The use of natural coagulants represents an important step for sustainable environmental technologies. Consequently, substantial research has been conducted to replace chemical coagulants with biodegradable and non-toxic coagulants based on natural compounds. In this study, the efficiency of a natural tannin-based coagulant (Tanfloc SG®) was tested as a replacer of ferric chloride, to remove organic matter from the polishing treatment step of swine slaughterhouse wastewater. Experimental designs defining the optimal treatment conditions of the wastewater were used to minimise the measured responses. The apparent colour and turbidity values were satisfactorily reduced (78% and 96%, respectively) by Tanfloc SG®, with a chemical oxygen demand reduction of 50%. The ferric chloride presented a lower performance than Tanfloc SG® in almost all analyses. The results showed that Tanfloc SG® performed excellently in the role of the primary coagulant, favourably replacing ferric chloride in the treatment of coagulation, flocculation and sedimentation of the study effluent.

Keywords: Coagulation; Ferric chloride; Sustainability; Tannin

1. Introduction

Many industrial activities involve the use of water, which results in the generation of large amounts of liquid waste. Such wastewater consists of many different components and pollutants that vary according to the activity from which it was produced [1].

Organic matter is considered as the main pollutant of water bodies because it demands consumption of dissolved oxygen for its oxidation [2]. On this basis, the treatment of wastewater is necessary for its subsequent disposal in water bodies, with due monitoring of the content of the pollutants, according to local legislation. A critical step in wastewater treatment is the coagulation process, to remove the organic matter present in the effluent. Coagulants promote the removal of light particles by sedimentation through simple gravity action. Inorganic salts are agents commonly used for this task. However, they result in the drawback of residual metal traces in the sludge, generated during the treatment, as well as in the treated water [3]. Many studies have highlighted the risk of aluminium and other metals to human health, as well as their adverse effects on plants and animals [4,5].

Tannins are organic polymers obtained from natural sources, whose applications have been tested for wastewater treatment in the elimination of various impurities, including organic matter. Tannins are widely distributed in higher plants and are typically found in maximal concentrations in tree bark [6]. Tannin molecules do not inherently possess a residual electrical charge, so they are often chemically treated with the intention of adding electrically charged sites. Significant research has proved the efficiency of modified tannins as coagulating agents, both in water bodies [7] and in water for human consumption [8,9].

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This study investigated the efficacy of a tannin-based organic polymer Tanfloc SG®, to replace ferric chloride in the polishing treatment of the coagulation process of swine slaughterhouse wastewater. Experimental planning was implemented to define the best values for the treatment parameters (coagulant concentration, rapid mixing speed, rapid mixing time, slow mixing speed, slow mixing time, pH and settling time), promoting the maximum reduction in the chemical oxygen demand (COD), turbidity and apparent colour responses.

2. Materials and methods

2.1. Swine slaughterhouse wastewater

The effluent used in the experiments was provided by a swine slaughterhouse located in Paraná State, Brazil, whose activities include the slaughter and processing of swine. The unit slaughters about 6,000 swine daily, and the average water consumption is 750 L/swine head. The generated liquid effluent is sent to primary treatment units, consisting of static screens and decanters. The effluent then proceeds to the secondary or biological treatment, which is composed of stabilisation ponds in series, being two anaerobic lagoons, an aerated lagoon of the complete mixture and a settling pond. Finally, a physico-chemical post-treatment step (coagulation/flocculation/flotation) is performed, with the addition of a coagulant agent to remove the remaining organic load.

The wastewater was sampled in the final stage of the biological treatment plant and was light brown with a light putrefied smell. It was characterised as having a turbidity of 38–63 nephelometric turbidity units (NTU), an apparent colour of 212–311 colour units (CU), an initial pH of 7.9 ± 0.2 and a COD of 173–189 mg L^{-1}. In this paper, the wastewater collected for the experiments was called study effluent. Wastewater with a COD < 500 mg L^{-1} can be classified as low COD [1], as is the case of this wastewater.

2.2. Materials

The reagents used as coagulants were commercial ferric chloride for analysis (Alphatec, Paraná, Brazil) and Tanfloc SG® in powder form, kindly provided by Tanac SA (Brazil). Tanfloc SG® is a commercial tannin-based product. It is obtained from the bark of black wattle (*Acacia mearnsii*) [3]. According to the manufacturer's technical file, Tanfloc SG® is a low molecular weight, organic and cationic polymer of essentially vegetable origin that can actuate as a coagulant or flocculant, and it is effective in a pH range of 4.5–8.0 [10].

The necessary pH adjustments were made using HCl and NaOH solutions of 1 M. All other materials used for the COD analyses were analytical grade, including mercury II sulphate (Synth, São Paulo, Brazil), silver sulphate (Qhemis, São Paulo, Brazil), potassium dichromate (Dinâmica, São Paulo, Brazil), sulphuric acid (Dinâmica, São Paulo, Brazil) and potassium biphthalate (Vetec, Rio de Janeiro, Brazil).

2.3. Coagulation process

Coagulation assays were conducted using a conventional jar test apparatus (Ethik/218-6 LDB). For each trial, 500 mL samples were poured into 1 L beakers. The pH was adjusted to the desired value using 1 M HCl or NaOH. The coagulant was added, and jar tests began with rapid mixing, followed by slow agitation. The flocs formed were then allowed to settle. At the end of sedimentation, approximately 150 mL samples were withdrawn with a pipette from near 2 cm below the liquid–air interface for chemical analysis. All experiments were carried out at ambient temperature (25°C–30°C).

2.4. Analyses

The analyses were performed according to the Standard Methods for Examination of Water and Wastewater [11] and included COD (5220D method), turbidity (method 2130B), apparent and real colour (method 2120B) and biochemical oxygen demand (BOD) (method 5210D) measurements. The total organic carbon (TOC) and Fe^{2+} were quantified by HACH 10173 (direct method) and 8146 (1,10-phenanthroline method), respectively [12].

2.5. Experimental planning

The experimental plan aimed to define the best value for each independent variable investigated in the treatment of effluent coagulation and thereby minimise the analysed responses. In all experiments, the COD, turbidity and apparent colour responses were examined. Initially, for each coagulant tested, a $2^{(7-3)}$ fractional factorial design, comprising five central points and a total of 21 trials was done, with the ranges used in these steps based on literature data. The optimal values were defined according to the *p*-values (α equal to 10%), for the effects of the independent variables on the responses.

For ferric chloride, to define the ideal coagulant concentration, additional tests were implemented over a wide concentration range (20–260 mg L^{-1}) due to the significant influence this variable showed in the fractional factorial design. For Tanfloc SG®, a second experimental plan, namely, a central compound rotational design (CCRD 2^2), for the final definition of the coagulant concentration and pH values was necessary (Table 1).

The fractional planning and CCRD 2^2 trials were randomised, as suggested by Rodrigues and Iemma [13], and the results were treated with the program Statistica 7.0®. This program was also used to generate the mathematical models for Tanfloc SG® from the CCRD results. The adjustment of the mathematical models was evaluated through analysis of variance (ANOVA), and the equations were applied in the desirability function, with the aid of the software, to define the best experimental condition to minimise all responses simultaneously.

Table 1
Real and coded levels of the variables tested in the central compound rotational design (2^2) for Tanfloc SG®

Independent variable	Code	Level				
		–1.41	–1	0	+1	+1.41
Coagulant concentration (mg L^{-1})	x_1	40	50	75	100	110
pH	x_2	4.7	5.3	6.5	7.8	8.3

The wastewater treated in the two optimal experimental conditions was characterised by turbidity, apparent and real colour, and COD, BOD and TOC analyses. For the wastewater treated with ferric chloride, Fe^{2+} analysis was also performed.

3. Results and discussion

3.1. Ideal treatment condition with Tanfloc SG®

Table 2 shows the effects of the independent variables on the responses measured after treatment of the effluent with Tanfloc SG®, according to the $2^{(7-3)}$ fractional factorial design.

A significant influence of the treatment was only observed for the pH variable. The increase in pH promoted a reduction in COD and turbidity but increased the value of the apparent colour. We then chose to readjust the pH range to 4.7–8.3, so that one of the levels of the CCRD 2^2 planning coincided with the original pH of the wastewater (level +1). This pH range is convenient because the manufacturer attests the effectiveness of the coagulant within the pH range 4.5–8.0.

The variation in the coagulant concentration of 20–120 mg L^{-1} did not promote a significant change in the value of the three responses. However, Tanfloc SG® is an organic compound, and its addition to the wastewater in excess amounts may result in an apparent non-removal of the organic matter from the wastewater, as the organic matter corresponding to the coagulant would be added to the organic matter of the wastewater. Consequently, it was decided to consider the coagulant concentration variable in the CCRD 2^2 in a narrower range, so it was readjusted to 40–110 mg L^{-1}.

Except for pH and coagulant concentration, the other variables were set at the lowest values tested, because their respective *p*-values were >10%, indicating that the variation in responses was not significant. The variables were then set at 100 rpm and 1 min for the coagulation step (rapid step), 15 rpm and 10 min for the flocculation step (slow step) and the settling time was set at 10 min for application in the CCRD 2^2.

From the experimental data obtained from CCRD 2^2 planning, the coefficients of the mathematical models proposed for Tanfloc SG® were generated from the program Statistica 7.0, and the respective *p*-values are presented in Table 3. Unlike

Table 2
Variables and ranges studied in $2^{(7-3)}$ fractional planning and their effects on COD, turbidity and apparent colour responses for Tanfloc SG®

Independent variable	Level			COD		Turbidity		Apparent colour	
	–1	0	+1	Effect (mg L^{-1})	*p*-value	Effect (NTU)	*p*-value	Effect (CU)	*p*-value
Coagulant concentration (mg L^{-1})	20	110	120	6.15	0.4638	–0.25	0.8231	5.25	0.5563
Rapid mixing speed (rpm)	100	250	400	4.35	0.6022	–1.25	0.2755	–9.25	0.3072
Rapid mixing time (min)	1	3	5	2.82	0.7341	–0.25	0.8231	–5.00	0.5750
Slow mixing speed (rpm)	15	45	75	–3.85	0.6442	1.75	0.1357	2.50	0.7781
Slow mixing time (min)	10	20	30	–10.32	0.2280	–0.25	0.8231	–1.50	0.8656
pH	5.0	7.5	9.0	–16.68	0.0626	–3.25	0.0117	28.75	0.0062
Settling time (min)	10	15	20	4.18	0.6167	–0.25	0.8231	0.25	0.9775

Level of significance α equal to 10%. CU, colour units; NTU: nephelometric turbidity units, COD, chemical oxygen demand.

Table 3
Regression coefficients for chemical oxygen demand (COD), turbidity and apparent colour according to the central compound rotation design (2^2) for Tanfloc SG®

	COD		Turbidity		Apparent colour	
	Coefficient	*p*-value	Coefficient	*p*-value	Coefficient	*p*-value
Mean	6.04	0.0034	2.67	0.0018	69.00	0.0000
x_1 (L)	6.04	0.5438	–2.72	0.0002	–27.85	0.0002
x_1^2 (Q)	14.75	0.2390	1.42	0.0070	17.00	0.0038
x_2 (L)	–14.35	0.1824	4.50	0.0000	73.75	0.0000
x_2^2 (Q)	13.00	0.2919	3.17	0.0002	51.00	0.0000
$x_1 \cdot x_2$	–11.75	0.4113	–1.75	0.0059	–25.50	0.0014

x_1 = coagulant concentration; x_2 = pH. Significance level α equal to 5%.

the coefficients in COD prediction after treatment with the coagulant ($p \geq 0.05$), all coefficients referring to the models for turbidity and apparent colour were significant ($p \leq 0.05$). The proposed mathematical models, containing all the respective coefficients shown in Table 3, are given in Eqs. (1)–(3):

$$\text{COD} = 79 + 6.04x_1 + 14.75x_1^2 - 14.35x_2 + 13x_2^2 - 11.75x_1x_2 \quad (1)$$

$$\text{Turbidity} = 2.67 + 2.72x_1 + 1.42x_1^2 + 4.50x_2 + 3.17x_2^2 - 1.75x_1x_2 \quad (2)$$

$$\text{Apparent colour} = 69 - 27.85x_1 + 17x_1^2 + 73.75x_2 + 51x_2^2 - 25.50x_1x_2 \quad (3)$$

The adjustment of the three mathematical models to the experimental data was verified through ANOVA (Table 4). The R^2 values were high for turbidity and apparent colour, indicating that 99% of the variations observed in the responses could be explained by both models.

The predictive model of COD, in turn, was able to explain only 55% of the observed variations (Table 4). It is a low value, which makes the model inefficient in representing the experimental data. The $F_{\text{calculated}}$ value of 1.2 for COD was inferior to $F_{\text{tabulated}}$ ($F_{(0.05;5;5)} = 5.05$). Thus, the criterion $F_{\text{calculated}} \geq F_{\text{tabulated}}$ was not met, and the model should not be used to predict COD. The values of $F_{\text{calculated}}$ for turbidity and apparent colour were quite high and much higher than $F_{\text{tabulated}}$, which confirmed the excellent fit of both models to the experimental data.

The response surfaces generated by the mathematical models of turbidity and apparent colour are shown in Fig. 1. The centres contained in the contour curves confirm that the studied ranges cover the optimum region, in which the lowest response value is obtained, that is, the greater efficiency of the treatment.

The adjusted Eqs. (2) and (3) were applied to the desirability function to combine the two optimal conditions (one for each equation) into a single one, which can minimise all responses simultaneously. The results are plotted in Fig. 2.

According to the plots obtained using the desirability function (Fig. 2), the optimum coagulant concentration and pH for Tanfloc SG® were 75 mg L^{-1} and 6.5, respectively. Under these conditions, the greatest possible reduction in the measured responses was expected. The condition defined as optimal was 100 rpm and 60 s for the fast step, 15 rpm and 10 min for the slow step, a settling time of 10 min, pH 6.5 and a coagulant concentration of 75 mg L^{-1}.

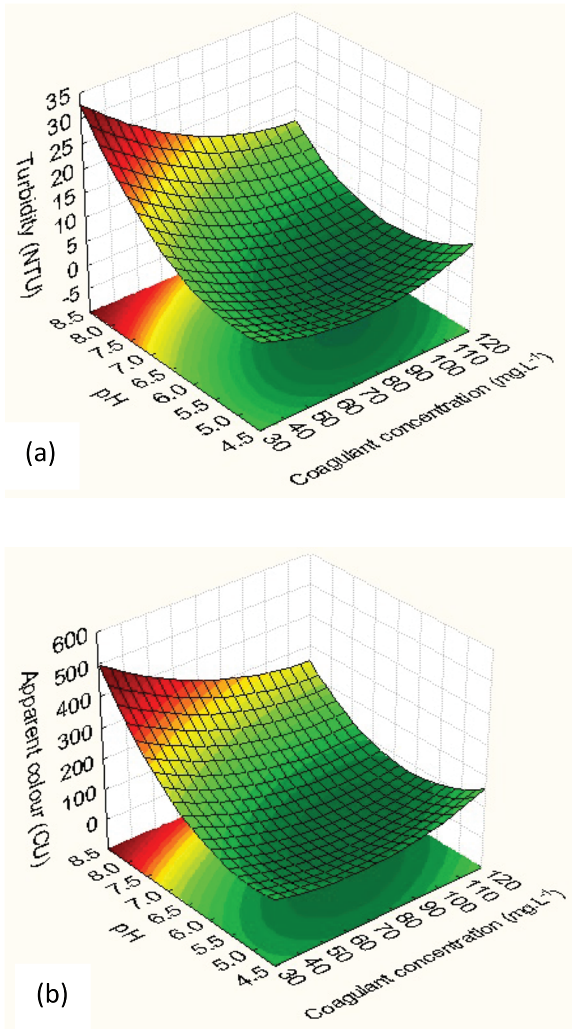

Fig. 1. Response surfaces for (a) turbidity and (b) apparent colour of effluent treated with Tanfloc SG® under optimum conditions.

Table 4
Analysis of variance of the mathematical models generated by the central compound rotational design (2^2) for Tanfloc SG®

Variation	COD			Turbidity			Apparent colour		
	Sum of squares	$F_{\text{calculated}}$	p-value	Sum of squares	$F_{\text{calculated}}$	p-value	Sum of squares	$F_{\text{calculated}}$	p-value
Regression	4,183.6	1.2	0.4176	291.1	99.7	0.0000	67,033.0	212.3	0.0000
Residual	3,440.1			2.9			315.8		
Lack of fit	1,854.1	0.78	0.6043	2.2	2.2	0.3222	147.8	0.6	0.6799
Pure error	1,586.0			0.7			168.0		
Total	7,623.6			294.0			67,348.7		
	$R^2 = 54.88\%$			$R^2 = 99.01\%$			$R^2 = 99.53\%$		

$F_{\text{tabulated}}$ for COD, turbidity and apparent colour: $F_{(0.05;5;5)} = 5.05$. Significance level α equal to 5%. COD, chemical oxygen demand.

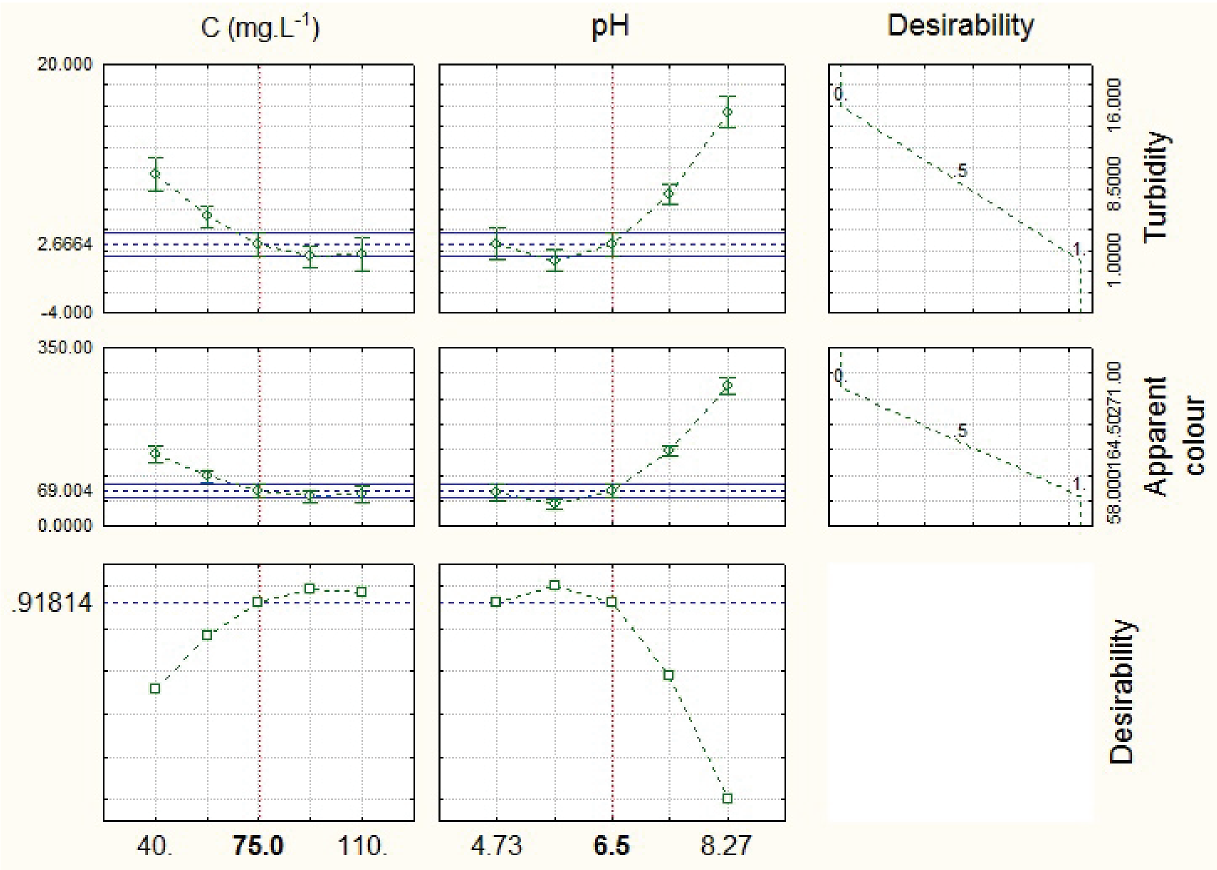

Fig. 2. Optimum global condition obtained by the use of the desirability function applied to predictive models of turbidity and apparent colour for Tanfloc SG®. *C*: coagulant concentration.

3.2. Ideal treatment condition with ferric chloride

According to the data presented in Table 5, the concentration of ferric chloride showed a significant effect as a coagulant within the range tested, promoting a reduction of the three responses.

The pH variable did not change the responses. Hence, the pH of the wastewater was maintained at its original value (pH 7.8) for the following tests, to define the ideal concentration of the coagulant. The values of 100 rpm and 10 s for the fast step were fixed from the fractional factorial design, as well as the 10 min and 15 rpm for the slow step, a settling time of 5 min and the initial wastewater pH. The speed and time of the fast and the time of the slow mixtures had their values set at the lowest values tested, as they showed no significant influence on the treatment, as with the settling time. The slow mixing speed promoted a significant reduction of 9.15 mg L^{-1} in COD, when its value increased from 15 to 75 rpm, not advocating a significant effect on the other responses. The effect of 9.15 mg L^{-1} was considered low by the authors. Therefore, it was decided to fix the parameter at the highest value of 75 rpm, because some experimental responses measured for COD had an estimated standard deviation of 20 mg L^{-1}. For this reason, it was considered valid to maintain the slow mixing speed at the lowest value tested, namely, 15 rpm. Further tests to define the coagulant concentration were required, due to the significant influence of this variable on the three responses.

The ferric chloride in the coagulation treatment was tested over a wide concentration range of 20–260 mg L^{-1}. The behaviour of the responses as a function of coagulant concentration is illustrated in Fig. 3.

Fig. 3 illustrates that increasing the coagulant concentration in the treatment resulted in a reduction in the three responses. At 50 and 140 mg L^{-1}, the responses showed a more pronounced decreasing trend. Thus, the tests were repeated at these concentrations and 120 mg L^{-1}. The results (Table 6) revealed that there was no significant difference between the lowest and the highest concentration of ferric chloride in the treatment. Therefore, the lowest concentration (50 mg L^{-1}) was defined as the optimal value for the following tests. Accordingly, the optimal treatment condition with ferric chloride was 100 rpm and 10 s for the fast step, 15 rpm and 10 min for the slow step, 5 min for the settling time, pH 7.8 (original effluent pH) and a coagulant concentration of 50 mg L^{-1}.

3.3. Performance comparison between coagulants Tanfloc SG® and ferric chloride

Triplicate assays for each of the two coagulants under the ideal conditions defined in sections 3.1 and 3.2, were undertaken. The analyses and results obtained are shown in Table 7.

Table 5
Variables and ranges studied in $2^{(7-3)}$ fractionated planning and their effect on COD, turbidity and apparent colour for ferric chloride

Independent variable	Level			COD		Turbidity		Apparent colour	
	–1	0	+1	Effect (mg L^{-1})	*p*-value	Effect (NTU)	*p*-value	Effect (CU)	*p*-value
Coagulant concentration (mg L^{-1})	10	30	50	–34.32	0.0000	–27.88	0.0000	–85.00	0.0000
Rapid mixing speed (rpm)	100	200	300	–7.85	0.1255	5.12	0.2717	10.75	0.4548
Rapid mixing time (seconds)	10	20	30	2.50	0.6095	–1.12	0.8046	–14.50	0.3180
Slow mixing speed (rpm)	15	45	75	–9.15	0.0790	–3.38	0.4627	–14.00	0.3343
Slow mixing time (minutes)	10	20	30	3.97	0.4206	–0.12	0.9780	–7.75	0.5879
pH	5.0	6.5	8.0	–11.80	0.3187	–5.50	0.4076	43.25	0.1886
Settling time (minutes)	5.0	12.5	20.0	2.67	0.5850	–5.62	0.2300	–11.00	0.4446

Level of significance α equal to 10%. CU, colour units; NTU, nephelometric turbidity units; COD, chemical oxygen demand.

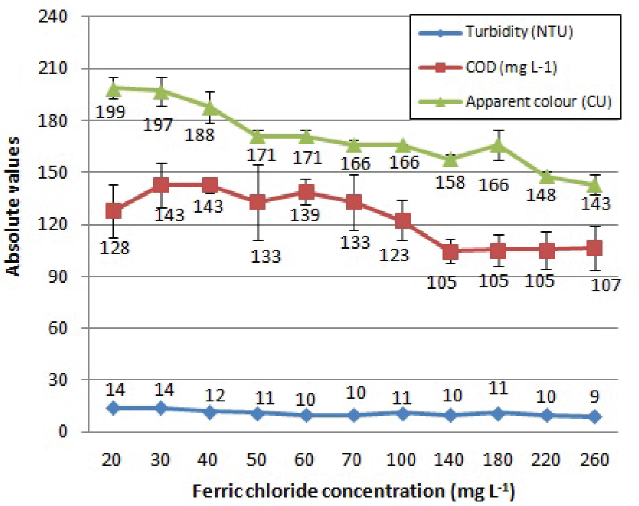

Fig. 3. Chemical oxygen demand (COD) values, turbidity and apparent colour as a function of ferric chloride dosage in coagulation assays (triplicate assays).

Table 6
COD, turbidity and apparent colour depending on the concentration of ferric chloride

Coagulant concentration (mg L^{-1})	50	120	140
COD (mg L^{-1})	115.2[a]	132.9[a]	120.2[a]
Turbidity (NTU)	8[a]	7[a]	8[a]
Apparent colour (CU)	134 [a]	131 [a]	131 [a]

Different letters in the same line represent different means (Tukey's test, $p \leq 0.05$).
CU: colour units. NTU: nephelometric turbidity units, COD, chemical oxygen demand.

Treatment with Tanfloc SG® reduced the wastewater turbidity by 96%, resulting in a residual value of only 2 NTU, and the apparent colour was decreased by 78%. Treatment with ferric chloride reduced the apparent colour by only 48% and also resulted in low turbidity wastewater but with a value almost five times greater than the wastewater treated with the organic coagulant.

Table 7
Characterisation analyses performed on the effluent treated with ferric chloride and with Tanfloc SG® under optimum conditions and at various pH values

Analysis	Tanfloc SG®	Ferric chloride
COD (mg L^{-1})	78[a]	80[a]
Turbidity (NTU)	2[b]	9[a]
Apparent colour (CU)	51[b]	160[a]
Real colour (CU)	46[b]	126[a]
BOD (mg L^{-1})	54[a]	61[a]
TOC (mg L^{-1})	24[a]	28[a]
Ferrous iron (mg L^{-1})	–	0.65

Different letters on the same line indicate significantly different means (Tukey's test, $p \leq 0.05$). BOD, biochemical oxygen demand; COD, chemical oxygen demand; CU, colour units; NTU, nephelometric turbidity units; TOC, total organic content.

Regarding the real colour, it presented lower values than those of the apparent colour. This decrease was expected because the centrifugation removes the liquid turbidity, an interferer that contributes to the increase in the apparent compared with real colour. The treatment with ferric chloride was the least efficient in colour reduction, with a real colour reduction of only 17%. For the Tanfloc SG® treatment, the real colour reduction was 63%, a much higher value than that presented by wastewater after treatment with the inorganic coagulant.

The COD reduction was satisfactory with both coagulants. The values measured for the two treatments did not statistically differ, with an average 50% decrease in COD relative to the study effluent.

The BOD values were statistically equal after treatment with both coagulants (Table 7). However, when analysing the BOD/COD ratios presented in Table 8, it is observed that the biodegradable fraction of the wastewater increased from 0.45 to 0.69 after treatment with Tanfloc SG®, indicating that

Table 8
BOD/COD and TOC/COD ratios for the effluent treated with Tanfloc SG® and also with ferric chloride, treatment variables under optimal conditions

	Tanfloc SG®	Study effluent	Ferric chloride	Study effluent
BOD/COD	0.69	0.45	0.76	0.47
TOC/COD	0.31	0.25	0.38	0.27

BOD, biochemical oxygen demand; COD, chemical oxygen demand; TOC, total organic content.

the biodegradability of the wastewater increased, which is desirable. For the treatment with ferric chloride, the biodegradable fraction increased from 0.47 to 0.76, resulting in a slighter larger fraction than that presented by the wastewater treated with the organic coagulant.

The TOC analysis is considered a direct parameter in determining the organic carbon content of the sample, which, unlike the COD analysis, does not account for the inorganic compounds present. Compared with the COD, the TOC/COD relationship provides a more accurate evaluation regarding the removal of organic matter from the treatment system. Specifically, the ratio expresses the efficiency of the treatment in the elimination of organic matter and, typically, a low TOC/COD ratio indicates a low carbon content [14]. The TOC/COD relationships for the effluent treated with Tanfloc SG® and ferric chloride, respectively, are shown in Table 8. It is observed that Tanfloc SG® presented a lower TOC/COD ratio than ferric chloride, which demonstrates a greater efficacy by the former coagulant to remove organic matter from the effluent. The TOC/COD ratio for ferric chloride, conversely, was relatively high in relation to the study effluent.

4. Conclusion

The organic coagulant Tanfloc SG® has been shown to be significantly better than ferric chloride in its optimal treatment conditions because it provided a more significant decrease in the turbidity and the apparent and real wastewater colours after coagulation treatment. The residual turbidity achieved with Tanfloc SG® was only 2 NTU, representing a reduction of 96% of the response, and the apparent colour was reduced by 78%, with excellent reduction values. Tanfloc SG® also presented a lower TOC/COD ratio than ferric chloride, indicating that the effluent treated with the polymer promoted a greater reduction in organic matter. Therefore, Tanfloc SG® performed very well in the role of the primary coagulant in the polishing process, favourably replacing ferric chloride in the treatment of coagulation, flocculation and sedimentation of the study effluent, with the advantage of being an environmentally clean compound obtained from renewable sources.

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