



## Direct application of chemically enhanced primary treatment in a municipal wastewater treatment plant: A case study

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### ABSTRACT

A feasible and efficient implementation of an Fe-based chemically enhanced primary treatment (CEPT) in a large conventional wastewater treatment plant (WWTP) was thoroughly evaluated. The influence of the velocity gradient and hydraulic retention time in the coagulation and flocculation stages, as well as the coagulant (FeCl<sub>3</sub>) dosage, were evaluated at lab-scale. Standard Jar Tests were performed to simulate CEPT, and the results were compared with those obtained by primary settling without coagulant. The increase in FeCl<sub>3</sub> dosage and flocculation hydraulic retention time (HRT<sub>F</sub>) increased the primary settling COD removal capacity between 6.6% and 18.1%, highlighting its effect on the easily biodegradable COD fraction. The COD removal capacity increased up to 16.8% when working with an HRT<sub>F</sub> of less than 4 min, facilitating CEPT direct application in conventional WWTPs, avoiding the need to incorporate flocculation chamber, with a corresponding decrease in the associated implementation cost. The presence of residual Fe in the primary settling effluent and an increase in the primary sludge volume were the main drawbacks of the process. The coagulation and flocculation velocity gradients or the coagulation hydraulic retention time did not significantly influence the process.

### 1. Introduction

Wastewater treatment plants (WWTPs) face many challenges in meeting the demands of rapid urbanization. Population growth and the corresponding growth in the contaminant load to be treated, as well as increasingly severe constraints on the quality of effluent discharges, have induced parallel increases in energy consumption (He et al., 2019). Therefore, the search for effective energy-saving strategies has attracted increasing attention in WWTPs, management, and ongoing investigations seek to optimize the relationship between energy and effluent quality (Maktabifard et al., 2018; Checa Fernández et al., 2021).

Electricity is the main energy source required in WWTPs, accounting for around 25–50% of the operating costs of conventional activated sludge installations, being the aeration of activated sludge in biological reactors (>50%), primary and secondary sludge pumping, and sludge dewatering the most impacting elements in the energy consumption (He et al., 2019).

Currently, WWTPs should be designed considering resource maximization and energy recovery, drifting towards zero energy

consumption or even towards energy production. However, even though novel technological solutions are already available for zero energy consumption, their application to upgrading existing installations can be far more challenging than building a new, more efficient one. Therefore, different strategies have been developed to optimize the energy consumption of existing facilities (Maktabifard et al., 2018). These actions must be carried out by improving the energy balance and optimizing the relationship between the energy consumption and effluent quality at affordable costs. In this context, several methods for reducing energy consumption and increasing energy recovery have recently been developed (Maktabifard et al., 2018).

Chemically enhanced primary treatment (CEPT) is a promising alternative intended to recover part of the wastewater energy potential (Muszyński-Huhajlo et al., 2018; Ansari and Farzadkia, 2022). This process employs chemicals to enhance coagulation and flocculation, resulting in a greater removal of phosphorus suspended and colloidal materials from wastewater. Consequently, their load in the primary sludge is increased, thereby avoiding their potential removal during aerobic biological treatment (Wang et al., 2023). Thus, air requirements

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are reduced, significantly decreasing global energy consumption in WWTPs. Furthermore, the anaerobic digestion process will produce more biogas, increasing energy generation (Taboada-Santos et al., 2020).

The degree of coagulation completion and subsequent flocculation for effective CEPT treatment is influenced by several operating factors. Some of the most relevant are i) the type of coagulant and its dosage, ii) the velocity gradient ( $G$ ) and hydraulic retention time (HRT) of the coagulation and flocculation stages, iii) wastewater pH, iv) temperature, and raw wastewater characteristics (Bratby, 2016).

Chemicals, such as iron or aluminum salts, are widely used in CEPT with greater acceptance for iron coagulants such as  $\text{FeCl}_3$  (Muszyński-Huhajlo et al., 2018; Ansari and Farzadkia, 2022). These coagulants are relatively inexpensive and easy-to use because they are already present in most WWTPs for phosphorous precipitation or sulfide control (Fu et al., 2021; Salehin et al., 2020). Struk-Sokolowska (2020) performed a cost analysis to accurately identify the most cost-effective option between the application of  $\text{AlCl}_3$  and  $\text{FeCl}_3$  as coagulants, achieving a positive balance for  $\text{FeCl}_3$ . Sludge obtained using an aluminum-based coagulant is characterized by low biodegradability due to the agglomeration of solid matter by the coagulant. Additionally, aluminum inhibits specific methanogenic activity and, decreases biogas generation during anaerobic digestion (Lin et al., 2018).

A wide range of Fe doses have been applied during CEPT, depending on the substance to be removed, its nature, the yields to be achieved or the characteristics of the wastewater to be treated (Muszyński-Huhajlo et al., 2018; Ansari and Farzadkia, 2022; Wang et al., 2023; Taboada-Santos et al., 2020). Taboada-Santos et al. (2020) tested doses between 25 and 300  $\text{mgFeCl}_3/\text{L}$  in order to pre-concentrate organic matter and remove viruses and micropollutants, while Wang et al. (2023), considered 25  $\text{mgFeCl}_3/\text{L}$  to be optimum for CEPT. Higher coagulant doses usually result in higher removal efficiencies (Ansari and Farzadkia, 2022; Taboada-Santos et al., 2020). However, to achieve a WWTP energy-economic balance without compromising CEPT efficiency, small doses of  $\text{FeCl}_3$  should be added (Muszyński-Huhajlo et al., 2018). Struk-Sokolowska et al. (2020), checked the differences resulting from the use of different  $\text{FeCl}_3$  doses ranging from 10 to 100  $\text{mg}/\text{L}$  and found that only doses from 10 to 40  $\text{mg}/\text{L}$  are cost-effective.

In addition to the cost associated with reagent dose, high iron dosages during CEPT can result in problems both in the subsequent aerobic biological treatment and in the sludge treatment. The aggregated and dense structure of the coagulated sludge can limit the access of bacteria and enzymes to organic matter inside the flocs (Lin et al., 2017) and residual iron concentrations caused alterations in the microbial activity of the activated sludge process, conditioning the sludge sedimentability (Lees et al., 2001).

Previous CEPT studies have mainly focused on the selection of optimal chemical reagents and their concentrations (Muszyński-Huhajlo et al., 2018; Ansari and Farzadkia, 2022; Lin et al., 2018). However, the influence of the parameters corresponding to the rapid mixing and flocculation stages has rarely been evaluated. The coagulation stage (rapid mixing) requires turbulent mixing to form a homogeneous solution, whereas the flocculation stage is intended to increase particle entrapment and floc growth (Bratby, 2016). Therefore, the selection of the mixing conditions (velocity gradient and hydraulic retention time) in the coagulation and flocculation stages is an important factor that will determine the need for new infrastructures in the WWTP, with the consequent associated economic cost. Despite its importance, contradictory recommendations have been provided in the literature for the rapid mixing and flocculation stage parameters. Usually, the flocculation stage parameters are not considered critical, whereas rapid mix parameters are appraised as the most important parameters for the coagulation-flocculation process optimization (Rossini et al., 1999).

In this context, this work analyzes the influence of the velocity gradient and the hydraulic retention time of both coagulation and flocculation stages ( $G_c$ ,  $\text{HRT}_c$ ,  $G_f$ , and  $\text{HRT}_f$  respectively) as well as the

dosage of low  $\text{FeCl}_3$  concentrations on the CEPT efficiency. Lab-scale experiments were carried out to select the operating conditions to implement an low concentration Fe-based CEPT at a conventional activated sludge WWTP in the most feasible and efficient manner. For this purpose, the removal performance and sludge generation were systematically evaluated, providing useful information for future real implementation. The study is complemented with a cost analysis.

## 2. Materials and methods

### 2.1. Wastewater of study

Real urban wastewater from the Granada-Sur WWTP (Granada-Spain. EMASAGRA) was used in this study. The Granada-Sur WWTP is a conventional activated sludge facility with anaerobic sludge stabilization, designed to treat 3125  $\text{m}^3/\text{h}$ , with a capacity of 662,500 population equivalent. Wastewater samples were collected from the primary settling process influent, post pre-treatment stage (without rubbish, sand, or oil). During the experimental time,  $\text{FeCl}_3$  was not used in the Granada-Sur WWTP.

### 2.2. Experimental procedure

Standard Jar Tests were accomplished to simulate the CEPT process with ferric chloride ( $\text{FeCl}_3$ , 40% w/w) as the coagulant. Tests were performed using six-gang stirrers and placing 1 L of wastewater sample in acid-prewashed beakers. The calibration curve obtained by Bhole (Bhole, 1970) was used to define velocity gradient values.

Owing to the temporal variability of real raw wastewater characteristics, to evaluate the performance of the process, the results from a simple settling treatment (without coagulant application) were compared with the results obtained from the CEPT. Five repetitions were performed for each test, using different raw wastewater samples.

Firstly, the effect of the application of low doses of the selected coagulant was studied. For this purpose,  $\text{FeCl}_3$  was dosed in 1  $\text{mg}/\text{L}$  increments between 1 and 24  $\text{mg}/\text{L}$ . For each test, a pre-determined amount of  $\text{FeCl}_3$  solution was added to 1 L of raw urban wastewater without pH adjustment at a constant temperature of approximately 18 °C. Coagulant addition was performed for less than 30 s of rapid mixing ( $G_c = 261 \text{ s}^{-1}$ ). Rapid mixing was followed by 15 min of slow mixing ( $G_f = 25.1 \text{ s}^{-1}$ ) to encourage flocculation, followed by 20 min of sedimentation.

Additionally, the effect of the coagulation and flocculation mixing conditions was analysed with a second set of experiments, modifying the  $\text{HRT}_c$ ,  $\text{HRT}_f$ ,  $G_c$ , and  $G_f$  while maintaining constant the previously selected coagulant doses. The values of the variables analysed were selected according to previous results reported in the literature (Bratby, 2016; Yan et al., 2009) and considering the characteristics of the WWTP so that CEPT can be applied without the need for civil work.

After the preliminary treatment of the Granada-Sur WWTP, the pretreated wastewater is sent to three primary sedimentation lines through separate pipes. Therefore, the possibility of carrying out hydraulic flocculation in the pipes between the pre-treatment and primary settling was studied by dosing the coagulant at the end of the pretreatment. The length of the pipes that goes to each of the sedimentation lines is different, and the water flow varies hourly. Taking this into account, as well as the dimensions of the conduction and water temperature,  $G_f$  and  $\text{HRT}_f$  were calculated according to the method described by Camp et al (Camp, 1995), for hydraulic flocculation.

The sedimentation stage was modified to evaluate the effect of coagulation-flocculation on the primary sludge volume generated. After coagulation and flocculation, the samples were transferred to an Imhoff cone for 180 min. This settling time was selected according to the HRT in the primary settling tank at the WWTP Granada-Sur.

characteristics of the wastewater used in this investigation.

**Table 1**

Characteristics of the influent wastewater from the Granada-Sur WWTP and effluents obtained from the lab-scale experiment of primary sedimentation without coagulant.

Parameter	Units	Raw Wastewater				Lab-scale Primary Sedimentation Effluent			
		Mean	S.D.	Max.	Min.	Mean	S.D.	Max.	Min.
COD	mg O <sub>2</sub> /L	818.2	175.7	1110.5	441.2	432.9	114.6	816.2	270.7
X	mg O <sub>2</sub> /L	581.3	132.1	856.3	266.3	191.9	37.4	286.0	121.7
S	mg O <sub>2</sub> /L	236.9	78.9	521.8	145.3	241.0	81.4	531.8	148.1
SS	mg/L	425.8	105.4	675.0	242.0	126.2	20.9	186.0	82.0
VSS	mg/L	358.3	88.2	562.5	190.0	109.7	18.4	174.0	74.0
NVSS	mg/L	67.5	24.1	155.0	34.0	16.5	5.2	28.0	0.0
Fe	mg/L	1.5	0.3	2.2	1.0	0.9	0.2	1.5	0.6

### 2.3. Physical and chemical determinations

To determine the optimal dose of FeCl<sub>3</sub>, total and filterable chemical oxygen demand (COD), turbidity, and pH were analysed in the supernatants obtained after the Jar Tests. The wastewater samples used in the study were analysed for total and filterable COD, total suspended solid (TSS), volatile suspended solid (VSS), and residual Fe<sup>3+</sup> concentrations, before and after sedimentation analysis. The same parameters were analysed in the corresponding supernatants obtained after the G and HRT tests. Furthermore, the volume and solid concentration (TSS) of the sludge generated during the sedimentation tests were determined.

TSS were analysed by vacuum filtration, drying at 105 °C, and gravimetric determination by using 0.45 µm filters (Millipore), whilst VSS were analysed by incineration at 550 °C. The VSS values were obtained from the difference between TSS and non-volatile suspended solids (NVSS) obtained from the assay. COD was determined by acid oxidation with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> using the closed reflux micro method. The absorbance of the digestate was measured colorimetrically at λ=600 nm and compared with that of a standard solution of potassium hydrogen phthalate. The filterable fraction of COD (S) was determined on samples filtered through 0.45 µm filters (Millipore), whereas the particulate COD fraction (X) was the difference between the total COD and the S fraction. The pH values were measured using a portable pH meter (WTW pH/Oxi 340i). All methods were performed according to the Standard Methods for the Examination of Water and Wastewater (APHA, 2017).

The residual iron concentration was measured using a Spectroquant analytical Kit N°: 1.14761.0001, being 0.05–5.00 mg Fe/L the range in which the method was applied. Turbidity determination was carried out following the spectrophotometric method according to UNE-EN ISO 7027–1:2016 by using filtered samples (0.45 µm) at λ=650 nm. The values obtained were compared with a standard solution of formazin, using Nephelometric Units (UNT) as reference units.

### 2.4. Economic feasibility analysis

The economic feasibility analysis consisted of an analysis of costs associated with the dosage of FeCl<sub>3</sub>, the decrease in the aeration needs of the activated sludge process, the increase in methane generation and the costs associated with the management of the final dehydrated sludge (20% dry). The costs associated with sludge pumping and sludge dehydration have been neglected.

Energy for aeration needs, energy obtained from the methane generation and final dehydrated sludge were determined by simulation of the Granada-Sur WWTP by using the simulation software WEST® (DHI). Model calibration was carried out following the Water Environment Research Foundation protocol until a suitable fitting of most of the selected outputs was reached (Melcer et al., 2003). Operational parameters have not been modified to check the different scenarios, considering the same temperature, purge flow rate, recirculation flow rate, etc. The parameters included in the mathematical model used to simulate the settling properties in primary clarifiers were changed in each simulation in order to replicate the results of the

coagulation/flocculation lab tests. Activated Sludge Model No. 1 (ASM1) (Henze et al., 1987) and Anaerobic Digestion Model No. 1 (ADM1) (Batstone et al., 2002) were selected for the simulations.

A production of 1.8 kWh per m<sup>3</sup> of methane has been considered. For sludge production calculations, a fixed increase in sludge related to the contribution of Fe has been considered, assuming its precipitation as Fe(OH)<sub>3</sub>. 220 €/Tn FeCl<sub>3</sub>, 0.15 €/ kWh and 40.4 €/Tn for disposal of dehydrated sludge were considered for the study (Current costs in Spain).

### 2.5. Statistical analysis

For statistical analyses, the data compiled were analysed using the SPSS software package (IBM-SPSS v22). An ANOVA test assessed the homogeneity of variance with a significance level of 5% (P value <0.05), and the Student-Newman-Keuls test assessed the statistical behaviour of the different wastewater characterization parameters with respect to FeCl<sub>3</sub> dosages. Pearson's correlation coefficients (PCC) were calculated between the main coagulation-flocculation variables and the increase in removal efficiency with respect to the primary sedimentation of the analyzed parameters.

Multivariate statistical analysis was performed using the CANOCO software (v4.5 for Windows, ScientiaPro, Budapest). Redundancy analysis (RDA) was carried out to assess the relationship between the main coagulation-flocculation variables and the increase in removal efficiency with respect to the primary sedimentation of the analysed parameters. The Monte Carlo permutation test, with 499 permutations, was used to assess the statistical significance of each variable or parameter in the canonical axes. CanoDraw was used for the graphical presentation of the results.

## 3. Results and discussion

### 3.1. Influent characterization and primary sedimentation performance

Before each experiment, the wastewater that reached the primary settling of the Granada-Sur WWTP was characterized (Table 1). According to the average concentrations, the wastewater can be catalogued as medium contamination degree wastewater (Chen et al., 2020), whose maximum values do not reach high contamination levels. The X fraction represents 71% of the total COD, the VSS concentration was around 85% of TSS, which is usual for urban wastewater (Struk-Sokolowska et al., 2020), and the pH (8.0 ± 0.4) and conductivity (1080.5 ± 157.4 µS/cm) values were within the normal range.

Efficient total COD and X fraction removal of 47.1% and 67.0% respectively, were achieved after lab primary settling without the addition of coagulant (Table 1). Similarly, high suspended solids removal performance was achieved (TSS=70.4%, VSS=69.4%, NVSS=75.5%), whereas the S fraction remained practically unchanged. This fact highlights that the primary settling process removes mainly particulate matter and suspended solids but is not able to remove filterable matter composed of colloidal and dissolved organic matter

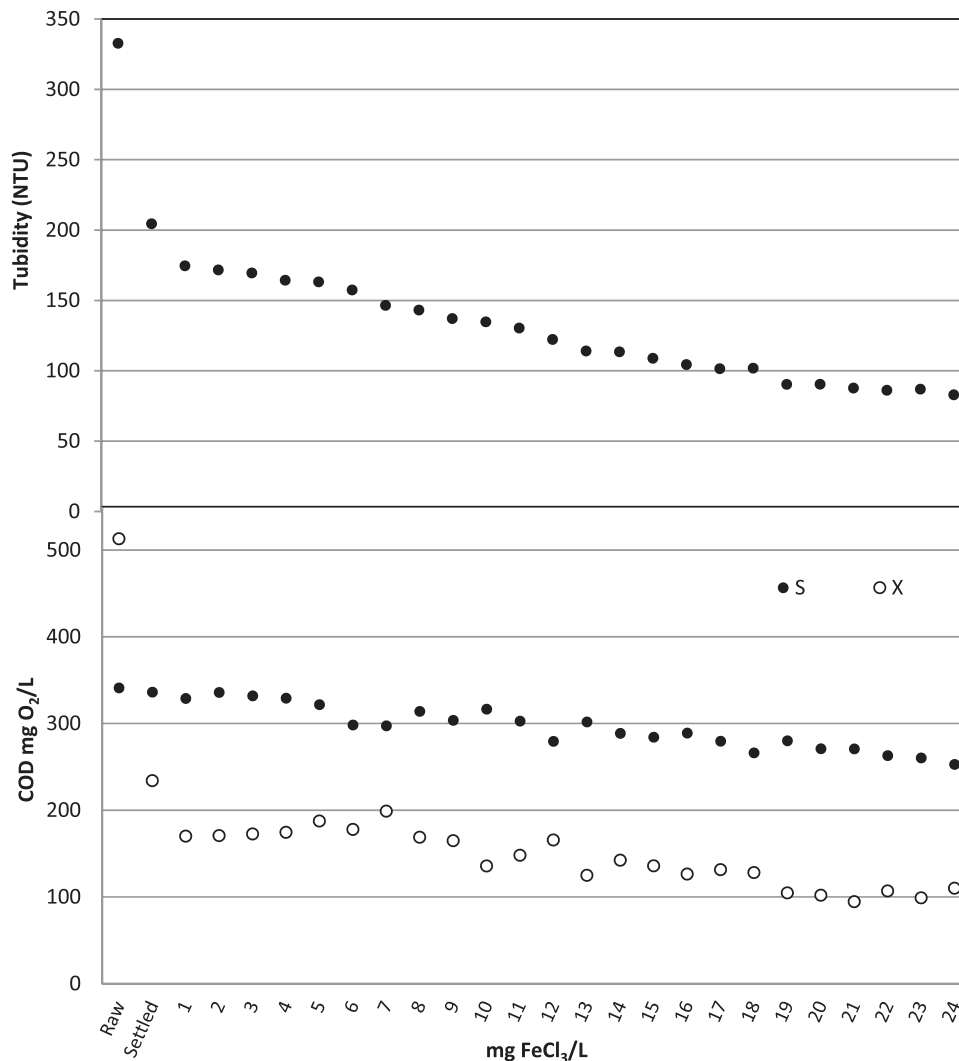


Fig. 1. Values and removal efficiency of (a) turbidity and (b) fractions of COD from urban wastewater by CEPT process using different dosages of FeCl<sub>3</sub>.

(Ruiz et al., 2014). Notably, the influent had an iron concentration of 1.5 mg/L, which decreased significantly after primary settling, reducing its average value by 39.7%.

Muszyński-Huhajło et al. (2018), determined the pollutants removal of raw urban wastewater after 2 hours of sedimentation in an Imhoff cone and the results obtained were typical for this process efficiency in a real WWTP, despite working with discontinuous tests with a perfect laminar regime. However our results revealed a more efficient removal for both TSS and COD, mainly the X fraction. The settling experiment increased the COD/TSS ratio from 1.92 to 3.43 gCOD/gTSS. This increase was higher than those reported in the literature of 1.88–2.13 gCOD/gTSS (Puig et al., 2010), corresponding to settling efficiencies of 60 and 65% for X and TSS, respectively. A higher average settling efficiency was obtained in the current study for TSS, considering the usual performance for primary settling (Struk-Sokolowska et al., 2020; Metcalf, 2013), which justify the higher ratio COD/TSS in the effluent of sedimentation. A large part of the settleable TSS is organic, so the removal efficiency of X fraction was also higher, which justifies the higher COD removal efficiency with respect to the usual performance (Struk-Sokolowska et al., 2020; Metcalf, 2013). These results reveal the presence of a higher percentage of sedimentable particulate matter, both organic and inorganic, in the raw wastewater. So, the higher performance rates were also expected for the coagulation-flocculation tests; consequently for comparative analysis, the increases in performance for each parameter between the sedimentation and

coagulation-flocculation tests were used.

### 3.2. Coagulant dosage effect

The effect of FeCl<sub>3</sub> doses between 1 and 24 mg FeCl<sub>3</sub>/L was analysed, not considering higher concentrations because they might not be cost-effective (Muszyński-Huhajło et al., 2018; Struk-Sokolowska et al., 2020). The addition of FeCl<sub>3</sub> produced a decrease in turbidity proportional to the dosage of coagulant, reaching values of 82.5 UNT and maximum removal efficiency of 75% with respect to raw wastewater for the highest doses tested, whereas an 57.5% for COD (82.7% for X fraction and 25.0% for S fraction) with respect to raw wastewater were observed (Fig. 1). This COD performance was similar to that obtained by Struk-Sokolowska (2020) for an analogous dose in the Bialystok WWTP but lower than that achieved in the same study for the Wrocław WWTP or higher than those achieved by Ansari and Farzadkia (2022) despite not applied coagulant aid. Regardless of the applied dose, another crucial factor to take into account in the effectiveness of the process is the raw wastewater, both in terms of its characteristics and the physico-chemical properties of its compounds. According to this, the results obtained are appropriate for the analysed wastewater. So coagulant dose must be adjusted based on the objectives to be achieved with the CEPT and the type of wastewater to be treated.

The S fraction concentration decreased linearly with the addition of coagulant ( $R^2 = 0.915$ ), whereas this clear linear fit was not observed for



**Table 2**

Student-Newman-Keuls test for the different wastewater characterization parameters ( $p$ -value < 0.05) at different dosages of FeCl<sub>3</sub>.

FeCl <sub>3</sub> dosage (mg FeCl <sub>3</sub> /L)	X	S	Turbidity
1	a	ab	a
2	a	a	a
3	a	ab	a
4	a	ab	ab
5	a	ab	ab
6	abcd	ab	abc
7	ab	ab	abcd
8	abc	ab	abcde
9	abcd	ab	abcdef
10	abcde	ab	abcdef
11	abcde	ab	abcdef
12	abcde	ab	abcdef
13	bcdefg	ab	bcdef
14	bcdef	ab	bcdef
15	cdefg	ab	cdef
16	defg	ab	cdef
17	defg	ab	def
18	efg	ab	def
19	efg	ab	ef
20	fg	ab	ef
21	fg	ab	ef
22	fg	ab	ef
23	g	ab	ef
24	g	b	f

a,b,c... Similar letter show a similar statistical behavior

the X fraction or turbidity. The Student-Newman-Keuls test (Table 2) showed a different statistical behaviour for the S fraction with respect to the X fraction and turbidity, with the latter showing similar statistical behaviour. The S fraction concentrations showed significant statistical differences at the extremes of the coagulant doses tested, but not for the rest, whereas a clear inflection points in the statistical behaviour were observed for the X fraction concentrations and turbidity for doses between 12 and 13 mg FeCl<sub>3</sub>/L and between 19 and 20 mg FeCl<sub>3</sub>/L. For lower doses, the statistical differences were less significant, with some variation in the statistical behaviour for doses between 5 and 6 mg FeCl<sub>3</sub>/L.

The addition of small doses of the coagulant (< 6 mg FeCl<sub>3</sub>/L) did not significantly affect the S fraction concentration. However, for the same dose range, the X fraction concentration was reduced by 24% over the removal performance of sedimentation without coagulant. However, the removal increase was observed to the same extent for a dose of 1 mg FeCl<sub>3</sub>/L as for 5 mg FeCl<sub>3</sub>/L, not appreciating an increase in removal performance with increasing the coagulant dose in this range. The same behaviour was observed for turbidity, with an increase in removal performance of approximately 11% over that obtained with sedimentation without coagulant, which was justified based on the contribution of the X fraction to turbidity.

According to the results obtained, the X fraction removed after the addition of low doses of coagulant is non-settleable particulate organic matter because it has not been removed by sedimentation without coagulant. For this fraction, the addition of the coagulant facilitated their sedimentation, without the need to dosage high coagulant concentrations; thus, with just the addition of 1 mgFeCl<sub>3</sub>/L the removal performance of COD increased by 12% with respect to the capacity of

**Table 3**

Characteristics of the different operational variables assayed.

Operational variables	Units	Values
Dose	mg FeCl <sub>3</sub> /L	6.0, 12.0, 20.0
HRT <sub>C</sub>	s	0.0, 10.0, 20.0, 30.0
HRT <sub>F</sub>	min	0.0, 1.0, 2.0, 4.0, 5.0, 15.0, 30.0
G <sub>C</sub>	s <sup>-1</sup>	0.0, 120.0, 188.0, 261.0
G <sub>F</sub>	s <sup>-1</sup>	0.0, 25.1, 59.8, 150.0, 200.0, 261.0

sedimentation without coagulant.

A downward trend was observed in both the concentrations of the S and X fractions and turbidity after coagulant addition at concentrations between 6 and 12 mg FeCl<sub>3</sub>/L. However, erratic behaviour not clearly linear was observed. An increase in the coagulant dose up to 12 mg FeCl<sub>3</sub>/L caused an increase in the removal performance of the S fraction that ranged between 8.0% and 16.0%; therefore, it can be considered that an effective destabilization of certain colloids (components of the S fraction) occurs, which facilitates their subsequent aggregation and sedimentation (Bhole, 1970). Aggregation can occur between destabilized colloids or with non-settleable particles of the X fraction, thereby increasing the removal of this fraction.

As shown in Fig. 1, there is a clear linearity between the decrease in the S fraction concentration and the reagent dose when coagulant concentrations greater than 12 mg FeCl<sub>3</sub>/L are used. Nevertheless, the behaviour of the X fraction continues to be erratic, although it follows a downward trend until a dose of approximately 20 mg FeCl<sub>3</sub>/L, from which the removal of this fraction seems to stagnate. For this range of concentrations, turbidity maintains a downward trend, although its slope gradually slows down as the dose is increased.

Based on these results, three inflection points can be described as a function of FeCl<sub>3</sub> dosage (6.0, 12.0, and 20.0 mg FeCl<sub>3</sub>/L), the origin of which may be due to the different nature of the particulate material in the wastewater analysed. Increases in the dose could lead to an increase in the ionic density in the aqueous solution around the colloid, causing a decrease in the "Z" potential. This decrease depends on the ionic density at the surface of the colloid, which in turn depends on the nature of the colloids (Bhole, 1970). Accordingly, colloids of lower ionic density destabilize sooner, aggregate in the system, and disappear. This aggregation is also possible with non-settleable suspended organic solids, which in turn can be dragged by flocs that settle.

As previously reported (Struk-Sokolowska et al., 2020; Lin et al., 2018), a higher coagulant dose resulted in greater COD removal capacity during CEPT, which lowered the energy costs of the aerobic biological treatment while improving the quality of the final effluent (Taboada-Santos et al., 2020). However, the increase in the coagulant dose entails a greater sludge production and higher cost associated with the reagent consumption (Muszyński-Huhajlo et al., 2018); therefore, for high coagulant doses, the benefits do not compensate (Muszyński-Huhajlo et al., 2018; Struk-Sokolowska et al., 2020). Furthermore, the use of very high doses of FeCl<sub>3</sub> does not improve the COD removal performance. Hu et al. (2023). tested different FeCl<sub>3</sub> doses (29–290 mgFeCl<sub>3</sub>/L), reaching for the lowest dose tested a COD removal performance around of 61%, similar to our results, while the yields achieved for higher doses only slightly improved these results. In the same study, it was observed that with the lowest dose of coagulant a slight removal performance for the S fraction was achieved, not improving it for higher doses. Similar results were observed by Taboada-Santos (2020), reaching a complete X fraction removal for doses of 150 mgFeCl<sub>3</sub>/L, while a large part of S fraction remains in the wastewater despite adding 300 mg FeCl<sub>3</sub>/L. Considering this, the different behaviours of the X and S fractions with respect to the coagulant concentration obtained opens the possibility of achieving significant reductions in energy consumption due to the efficient removal of X fraction by using low coagulant concentrations.

### 3.3. Influence of dosage and mixing conditions

Based on the results previously obtained when studying the effect of the FeCl<sub>3</sub> dosage and the corresponding statistical analysis performed, three dosages (6.0, 12.0, and 20.0 mg FeCl<sub>3</sub>/L) were selected to analyze the influence of mixing conditions on the CEPT efficiency.

Firstly, the characteristics of the WWTP were analysed to select mixing conditions that avoided including new elements in the facility. In this context, the possibility of carrying out a hydraulic flocculation stage in the connecting pipe between pre-treatment and primary settling was investigated (instead of building a flocculation chamber). The HRT<sub>F</sub>

Table 4

Pearson correlation coefficient between operational variables and wastewater quality parameters after CEPT.

	COD	X	S	SS	VSS	NVSS	Fe <sup>3+</sup>
Dose	0.734	0.402	0.587	0.662	0.629	0.244	-0.313
HRT <sub>C</sub>	0.041	0.070	-0.011	-0.092	-0.112	0.034	0.010
HRT <sub>F</sub>	0.134	0.031	0.118	0.092	0.039	0.146	0.234
G <sub>C</sub>	0.092	0.042	0.064	-0.001	-0.036	0.078	0.110
G <sub>F</sub>	-0.080	-0.021	-0.062	0.026	0.066	-0.081	-0.141

■ Significant correlation with p-value < 0.01    ■ Significant correlation with p-value < 0.05

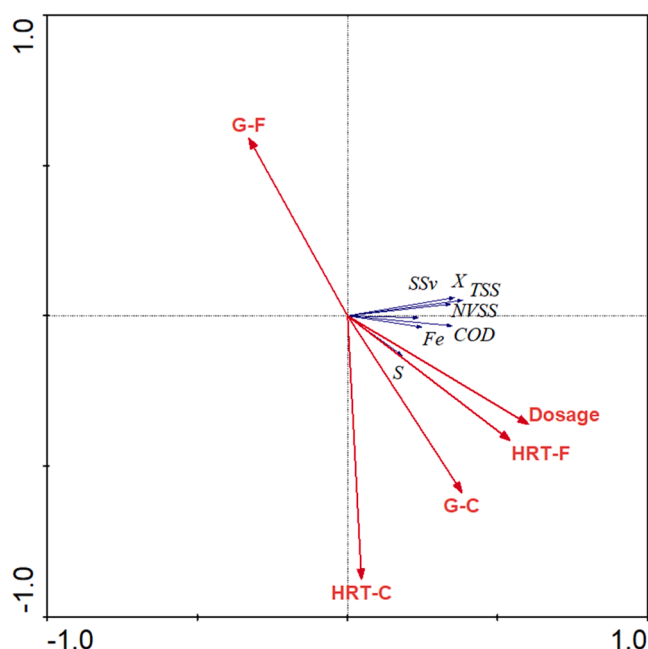


Fig. 2. Redundancy analysis (RDA) ordination diagrams (diplots) showing the relation between the main coagulation-flocculation variables and the increase of removal performance with respect to primary settling.

values reached in the connection pipe ranged between 1 and 4 min (depending on the time of year and whether the WWTP worked at minimum, average, or peak flows). Concerning the  $G_F$  values achieved, these will also be affected by the water flow rate. The calculations made resulted in minimum and maximum values of  $183 \text{ s}^{-1}$  and  $280 \text{ s}^{-1}$ , respectively. For the calculations, turbulence conditions have been taken into account, so as not to cause the flocs to break. These results have been considered when studying the influence of mixing conditions. Thus, the tested coagulation and flocculation mixing conditions (HRT<sub>F</sub>, HRT<sub>C</sub>,  $G_F$ , and  $G_C$ ) were selected (Table 3) according to the values reached for in situ hydraulic flocculation and the most common ones employed in the literature (Bratby, 2016; Yan et al., 2009).

Table 4 summarizes the PCC obtained between the analysed operational variables of CEPT and the increase in the removal performance of the wastewater quality parameters concerning primary settling without coagulant. The results showed that, for the analysed ranges, the most influential variables on the process performance were the coagulant dose and HRT<sub>F</sub>. For the coagulant dose, a high positive correlation (p-value < 0.01) was observed for all the parameters analysed, except for the residual Fe, for which a negative correlation was found. Thus, the

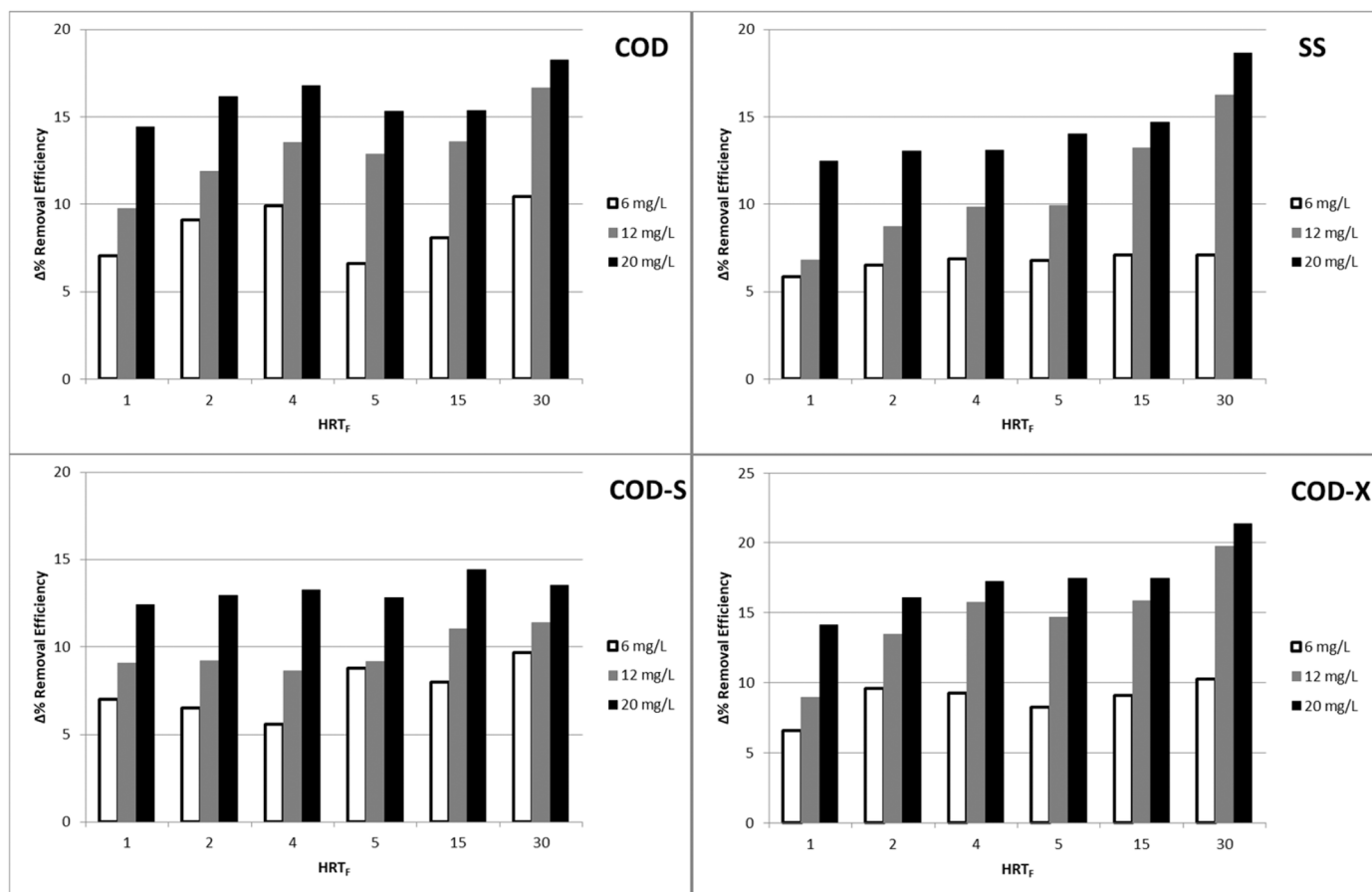
concentration of the main quality parameters of the wastewater decreased after CEPT as the coagulant dose increased, whereas the residual Fe concentration further increased. For doses between 1 and 24 mg FeCl<sub>3</sub>/L, the results obtained were in agreement with those previously reported in the literature, increasing the CEPT performance with higher coagulant doses (Muszyński-Huhajlo et al., 2018; Struk-Sokolowska et al., 2020; Hu et al., 2023). However, the negative effects of high residual Fe concentrations on aerobic biological treatment (Rossini et al., 1999) should be considered when using the highest coagulant doses.

A significant positive correlation (p-value < 0.01) was obtained between the HRT<sub>F</sub> and the removal of residual Fe, whereas a significant correlation (p-value < 0.05) was obtained for the COD and NVSS removal. The HRT<sub>F</sub> increase for up to 30 minutes increased the removal performance of primary treatment, mainly in COD and NVSS, avoiding high residual Fe concentrations in the aerobic biological process. As shown in Table 4, the  $G_F$  increase resulted in an increase in the residual Fe concentration (p-value < 0.05), while  $G_C$  and HRT<sub>C</sub> did not have a statistically significant influence on any of the analysed parameters.

An RDA analysis was performed to evaluate the influence of the main coagulation-flocculation variables on the increase in removal efficiency with respect to primary settling. The model represented 93.5% of the variance on the first axis and 98.0% of the cumulative variance on the second axis. All analysed variables, except  $G_F$ , presented p-values lower than 0.05. The RDAs results are represented on a diplot diagram (Fig. 2).

As shown in Fig. 2, the vectors obtained in the RDA analysis were grouped and oriented in the same direction with a similar length for all quality parameters except for the S fraction. As observed by the PCCs (Table 4), the coagulant dose (p-value = 0.002) was the most influential operational variable on the removal performance increase, mainly affecting the S fraction and COD. However, the statistical behaviour was contrary to that observed through PCC in the case of the residual Fe concentration. The second most influential operational variable was the HRT<sub>F</sub> (p-value = 0.006), with a significant influence on the S fraction. Both  $G_C$  (p-value = 0.044) and HRT<sub>C</sub> (p-value = 0.006) appeared to be less influential variables but were statistically significant. Thus, variations in  $G_C$  between values ranging from 120 to  $261 \text{ s}^{-1}$  and selecting HRT<sub>C</sub> between 10 and 30 s did not significantly affect the performance achieved.  $G_F$  did not show a statistically significant behaviour (p-value = 0.8480).

The coagulant dosage and HRT<sub>F</sub> increase in the applied ranges demonstrated a positive effect in enhancing the COD removal capacity of CEPT, mainly in the X fraction (Fig. 3). The largest increase in COD removal efficiency was observed when the dosage increased from 12 to 20 mg FeCl<sub>3</sub>/L, which was significantly higher than when the dosage increased from 6 to 12 mg FeCl<sub>3</sub>/L. However, changes in operational conditions have also influenced dosage effects; therefore, at lower HRT<sub>F</sub>, the effect of the dose increase was greater. The highest increase in COD



**Fig. 3.** Increase of the removal efficiency (%) of CEPT with respect to primary settling according to FeCl<sub>3</sub> dosage and HRT<sub>F</sub> for COD, TSS, “S” COD fraction and, “X” COD fraction.

removal with respect to the test without coagulant (18.2%) was achieved by applying the highest coagulant dose (20 mg FeCl<sub>3</sub>/L), and 30 min of HRT<sub>F</sub>, resulting in a mean COD effluent concentration below 300 mg O<sub>2</sub>/L. However, when employing a considerably lower HRT<sub>F</sub> (4 min), an increase of up 9% in COD removal was achieved when using a coagulant dosage of 6.0 mg FeCl<sub>3</sub>/L (effluent concentration of 376 mg O<sub>2</sub>/L), increasing to 13.5% if the dosage was increased to 12 mg Fe/L (effluent concentration of 338 mg O<sub>2</sub>/L), and up to 16.8% for the highest dose tested (effluent concentration of 311 mg O<sub>2</sub>/L).

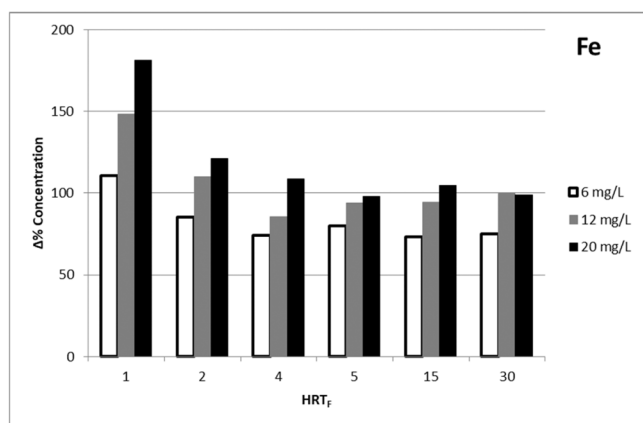
The observed yields facilitate the CEPT application in conventional WWTPs without incorporating a flocculation chamber, simply applying

the coagulant at a point with a high mixing degree and taking advantage of hydraulic agitation in the pipe of water from the pretreatment to the primary settling. For the analysed WWTP, the HRT<sub>F</sub> reached in the pipe of water from the pretreatment to the primary settling ranged between 1 and 4 min. For these flocculation conditions, the minimum increases observed with respect to the removal performance of primary settling ranged between 7.1% and 9.9% for coagulant doses of 6 mg FeCl<sub>3</sub>/L (effluent concentration between 390 mg O<sub>2</sub>/L to 367 mg O<sub>2</sub>/L).

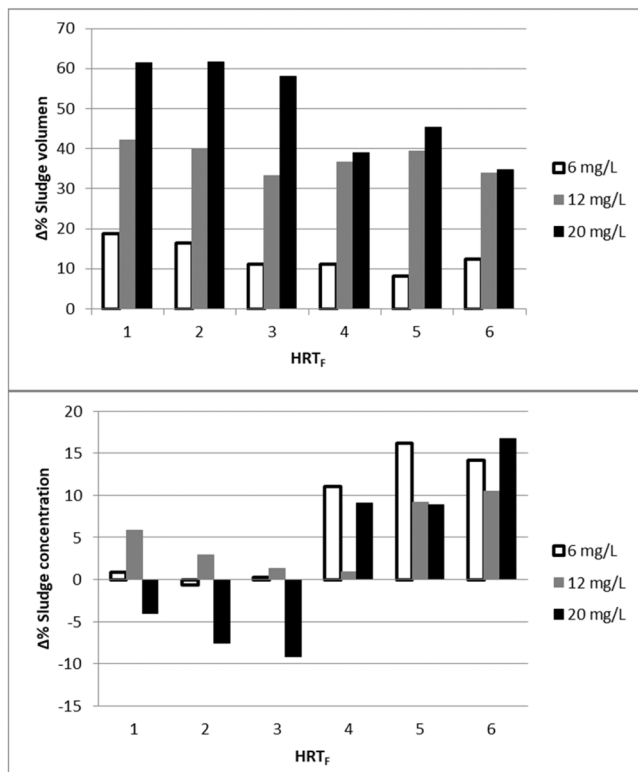
The removal performance of the S fraction increased from 5.6% to 14.4% under the most unfavourable and favourable conditions, respectively (Fig. 3). The increase in S removal performance was most remarkable when changing the coagulant dose from 6 to 12 mg FeCl<sub>3</sub>/L. Moreover, it is important to note that with optimal coagulation conditions the S fraction removal was considerably improved compared to that obtained in primary settling without coagulant, which remained practically unchanged (Table 1).

Based on the increase in the removal performance of the S fraction, the coagulant dose was the most influential variable. Therefore, the negative influence of applying a low HRT<sub>F</sub>, such as that obtained with hydraulic agitation in the pipe of water from the pretreatment to the primary settling, can easily be alleviated by increasing the coagulant dose. This is of great interest because an increase in the removal performance of the S fraction results in a lower concentration of easily biodegradable organic matter loaded into the aerobic biological reactor. For a given oxygen transfer capacity, depending on the installation analysed, this results in a decrease in the energy requirements of aeration in the biological reactor (Chen et al., 2020). Thus, the cost of the activated sludge process could be significantly reduced.

In the case of TSS, an increase in the yield achieved regarding



**Fig. 4.** Increase of the percentages of residual Fe concentration of CEPT with respect to primary settling according to FeCl<sub>3</sub> dosage and HRT<sub>F</sub>.



**Fig. 5.** Increase of the percentages of sludge volume and sludge concentration of CEPT with respect to primary sedimentation according to FeCl<sub>3</sub> dose and HRT<sub>F</sub>.

primary settling was observed in all cases under study. As shown in Fig. 3, the minimum and maximum TSS removal performances increased by 5.9% and 18.7%, respectively (effluent concentration between 101 mg/L to 47 mg/L), which were similar to those observed for COD increase. As previously mentioned, the most influential variable was the coagulant dose. Changes in the operational mixing conditions also improve the process performance, showing a greater influence on the HRT<sub>F</sub> increase from 1 to 2 min than from 2 to 4 min.

The residual Fe concentration in the CEPT effluent was always higher than that observed in the influent. Thus, FeCl<sub>3</sub> addition caused an increase in the residual Fe concentration, with the applied dose being the

**Table 5**  
Results of simulation for the different operational conditions assayed.

Dose	HRT <sub>F</sub>	G <sub>F</sub>		Aeration	Methane	Sludge	
mgFeCl <sub>3</sub> /L	min	s <sup>-1</sup>	Kg Fe/d	-Δ kWh/d	Δ kWh/d	-Δ Tn/d	Δ Tn Fe/d
6	1	261	1.1	335.9	935.3	1.6	1.5
6	2	200	1.1	622.7	1214.0	3.0	1.5
6	4	150	1.1	670.2	1565.7	3.2	1.5
6	5	25.1	1.1	340.0	910.0	5.4	1.5
6	15	25.1	1.1	564.9	1100.0	7.5	1.5
6	20	25.1	1.1	850.0	1632.0	8.2	1.5
12	1	261	2.3	916.4	1708.1	4.3	3.0
12	2	200	2.3	968.9	2630.7	5.5	3.0
12	4	150	2.3	1240.1	3001.2	5.8	3.0
12	5	25.1	2.3	1113.0	2979.1	6.5	3.0
12	15	25.1	2.3	1720.0	3155.2	8.5	3.0
12	20	25.1	2.3	2301.9	3488.9	9.6	3.0
20	1	261	3.8	3140.0	3552.0	6.1	4.9
20	2	200	3.8	3270.0	3790.0	7.2	4.9
20	4	150	3.8	3302.5	3812.3	8.1	4.9
20	5	25.1	3.8	3491.6	3920.0	7.2	4.9
20	15	25.1	3.8	3482.9	4031.2	8.6	4.9
20	20	25.1	3.8	3668.6	4239.3	10.0	4.9

most influential variable and reaching an increase of up to 180% with respect to the initial concentration values. However, increases in the HRT<sub>F</sub> reduced the residual Fe concentrations in the effluent (Fig. 4). Lees et al. (2001) observed that low residual coagulant concentrations (1–2 mg Fe/L) caused a reduction in microbial activity of the activated sludge process, changed species diversity and the floc structure, conditioning the sludge volume produced and their sedimentability. During the current study, residual Fe concentrations ranged from 1.64 to 3.78 mg Fe/L. Therefore, the evaluation of this parameter is of great importance because it can limit the selection of the most suitable operational conditions for CEPT. According to these results, residual Fe concentration is one of the main drawbacks of CEPT application without the incorporation of new elements for flocculation because under these conditions, a low HRT<sub>F</sub> should be applied. Thus, the negative effect on removal performance must be offset by increasing the coagulant dose, favouring the presence of residual Fe.

### 3.4. Sludge volume

When implementing CEPT in a WWTP, it is important to consider the potential increase in sludge generation. Without coagulant addition, the sludge volume obtained in the Imhoff cone presented a mean value of  $15.1 \pm 3.7$  mL/L. The FeCl<sub>3</sub> addition considerably increased the sludge volume in all cases under study, with increments ranging from 8.1% ( $16.3 \pm 4.0$  mL/L) to 61.8%, ( $24.4 \pm 6.9$  mL/L) as shown in Fig. 5. The largest increase was observed with the application of a higher coagulant dosage. Conversely, an increase in the HRT<sub>F</sub> slightly decreased the sludge generated. As depicted in Fig. 5, the most significant increase in sludge volume was observed with an increase in FeCl<sub>3</sub> dosage from 6 mg FeCl<sub>3</sub>/L to 12 mg FeCl<sub>3</sub>/L for all HRT<sub>F</sub> assayed, whereas the increase was less significant when the dosage was increased from 12 mg FeCl<sub>3</sub>/L to 20 mg FeCl<sub>3</sub>/L.

Regarding the TSS concentration in the sludge, the mean concentration without coagulant addition was  $13.9 \pm 2.1$  gSST/L, with concentrations ranging from  $-9.2\%$  ( $12.6 \pm 1.9$  gSST/L) to  $16.8\%$ , ( $16.2 \pm 2.4$  gSST/L). An upward trend was observed as the HRT<sub>F</sub> increased for all coagulant concentrations tested, although for low HRT<sub>F</sub>, the trend decreased as the sludge volume increased (Fig. 5).

The increase in sludge volume generated when CEPT is applied requires an increase in the primary sludge pumping capacity, which makes it difficult to adapt the facilities of a conventional WWTP to CEPT. This circumstance makes it necessary to review not only the primary sludge pumping capacity but also the entire sludge treatment line of the WWTP, because the operational conditions of the sludge thickening equipment, the hydraulic retention time of the anaerobic stabilization of sludge, and the operational conditions of the sludge dewatering equipment can be affected (Metcalf, 2013).

The application of CEPT without the incorporation of new elements for flocculation, that is, a simple dosage of coagulant, leads to a low HRT<sub>F</sub>, a circumstance in which the highest primary sludge volumes are produced with the lowest TSS concentration. Therefore, it is significantly important to review the capacity of the WWTP sludge line under these circumstances before practical implementation.

### 3.5. Economic feasibility analysis

Coagulant doses of 6.0, 12.0, and 20.0 mg FeCl<sub>3</sub>/L, were considered for the economic feasibility analysis, working with different HRT<sub>F</sub> and G<sub>F</sub> (Table 3). Simulations without coagulant addition were considered as a reference to obtain the differences in aeration needs, methane generation and final dehydrated sludge (20% dry).

The most significant advantages over the operational cost of a large WWTP when CEPT is applied are the redirection of part of the biodegradable organic matter to anaerobic digestion which decrease energy needs in the activated sludge process (Muszyński-Huhajlo et al., 2018), while methane generation increases (Ansari and Farzadkia, 2022). Our



**Table 6**

Cost analysis for the different operational conditions assayed (€/d).

Dose mgFeCl <sub>3</sub> /L	HRT <sub>F</sub> min	G <sub>F</sub> s <sup>-1</sup>	Coagulant	Aeration	Methane	Sludge	Total
6	1	261.0	-247.5	50.4	140.3	4.9	-51.9
6	2	200.0	-247.5	93.4	182.1	60.2	88.2
6	4	150.0	-247.5	100.5	234.9	69.4	157.2
6	5	25.1	-247.5	51.0	136.5	157.4	97.4
6	15	25.1	-247.5	84.7	165.0	243.8	246.1
6	20	25.1	-247.5	127.5	244.8	272.5	397.3
12	1	261.0	-495.0	137.5	256.2	54.0	-47.3
12	2	200.0	-495.0	145.3	394.6	104.5	149.4
12	4	150.0	-495.0	186.0	450.2	115.4	256.6
12	5	25.1	-495.0	167.0	446.9	142.0	260.8
12	15	25.1	-495.0	258.0	473.3	223.6	459.8
12	20	25.1	-495.0	345.3	523.3	270.0	643.6
20	1	261.0	-825.0	471.0	532.8	46.9	225.7
20	2	200.0	-825.0	490.5	568.5	91.2	325.2
20	4	150.0	-825.0	495.4	571.8	126.6	368.8
20	5	25.1	-825.0	523.7	588.0	92.1	378.9
20	15	25.1	-825.0	522.4	604.7	148.8	450.9
20	20	25.1	-825.0	550.3	635.9	205.3	566.5

study reveals energy savings in the activated sludge system between 335.9 and 3668.6 kWh/d for a conventional WWTP with capacity to treat 3125 m<sup>3</sup>/h, while increases in methane production range between 910.0 and 4239.3 kWh/d (Table 6).

According to ASM1 model (Henze et al., 1987) and ADM1 model (Batstone et al., 2002) the redirection of part of biodegradable organic matter by CEPT avoids its transformation in unbiodegradable organic matter during de aerobic process thus allowing greater methane generation. As a consequence, lower generation of sludge can be expected. The biodegradable organic matter transformed into unbiodegradable biomass during the aerobic process will not be biotransformed during the anaerobic process, accumulating as sludge, while if it is redirected to the anaerobic process, a large part will be transformed into methane not generating biomass.

However, the cost associated with the coagulant dosage ranged between 247.5 and 825.0 €/d for 6 mg FeCl<sub>3</sub>/L and 20 mg FeCl<sub>3</sub>/L respectively (Table 6). Likewise, the addition of Fe meant a direct increase in sludge production, which has been considered fixed for each dose, ranging between 1.5 and 4.9 Tn/d (Table 5).

Despite the direct relationship between the coagulant dose and the effectiveness of CEPT, the most economically viable conditions have been obtained for an intermediate dose, due to the significant production of methane and mainly to the lower cost associated with coagulant consumption (Table 6), reaching a saving of 0.86€/m<sup>3</sup> of treated wastewater. However, the greatest economic savings have been achieved for mixing conditions in which it is necessary to implement a flocculation vessel to achieve the necessary HRT<sub>F</sub> and G<sub>F</sub>. This implies the implementation of new elements in the WWTP, whose economical amortization limits the economic benefits.

If the results obtained working with mixing conditions for which the implementation of new elements in the WWTP is not necessary (HRT<sub>F</sub> between 1 and 4 min), variable circumstances can be observed, with an increase of the economic cost to significant savings, mainly working with the higher dose assayed (Table 6). These mixing conditions are variables as mentioned in Section 3.3 but they are not penalised by the economical amortization of new elements and their application is direct, reaching average savings of up to 0.4€/m<sup>3</sup>.

#### 4. Conclusions

CEPT with FeCl<sub>3</sub> concentrations lower than 24 mgFeCl<sub>3</sub>/L was evaluated at lab-scale to implement it in a large conventional activated sludge WWTP in the most feasible and efficient manner. For this purpose, removal performance and sludge generation were assessed. The following conclusions can be drawn from the main results of this study.

- The FeCl<sub>3</sub> dosage and HRT<sub>F</sub> are the most influential variables in the increase of COD and TSS removal capacity, with the FeCl<sub>3</sub> dosage being the most relevant.
- HRT<sub>C</sub> between 0.0 and 30.0 seconds, G<sub>C</sub> between 0.0 and 261 s<sup>-1</sup>, and G<sub>F</sub> between 0.0 and 261 s<sup>-1</sup> were variables with little significant influence in the process.
- Increases in the COD removal capacity obtained by primary settling when CEPT is applied between 6.6% and 18.1% and mainly the increase in the S fraction removal capacity, make the process energetically attractive.
- Increases in COD removal capacity of up to 16.8% working with HRT<sub>F</sub> of less than 4 min, achieved in the wastewater pipe between the pretreatment and primary settler, facilitates the incorporation of CEPT in conventional WWTPs without incorporating new elements for flocculation.
- The main drawbacks of the incorporation of CEPT in conventional WWTPs are the presence of residual Fe in the primary settler effluent and an increase in the primary sludge volume.
- A positive economical balance can be obtained by the implementation of a CEPT working with doses minor to 20 mgFeCl<sub>3</sub>/L, with an economic saving in operational cost of up to 0.86€/m<sup>3</sup> of treated water, which can go down to 0.4€/m<sup>3</sup> without the incorporation of new elements in the WWTP.

In line with the above, operational conditions should be chosen considering a balance between enhancing the removal performance and obtaining the lowest possible residual effluent Fe concentration and affordable sludge volume. In this context, the present work provides valuable information for future practical implementation of the process.

#### Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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