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Use of ferric chloride contained in the rejects from the steel industry as a coagulant for the fuel washing wastewater treatment

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ABSTRACT

The aim of the present study firstly the treatment of the fuel washing wastewater by a coagulation–flocculation process, using ferric chloride ($FeCl_3$), and secondly the comparison between a commercial $FeCl_3$ (40%) and an industrial effluent rich in $FeCl_3$ (30%) coagulant, in terms of the coast and the pollution reduction efficiency. For these purposes, the fuel washing wastewater was subjected to a coagulation–flocculation treatment using different doses of both coagulants. The obtained results showed that the commercial coagulant exhibited better pollution removal efficiency compared to the one contained in the industrial effluent. Indeed, the commercial coagulant $FeCl_3$ (40%) was capable to remove 92%, 96% and 88% of chemical oxygen demand (COD), nitrate and phenol, respectively, using 120 mg/L of $FeCl_3$ (40%). Moreover, 98% and 61% of turbidity and ammonium ions, respectively, using 160 mg/L of $FeCl_3$ (40%). While, the $FeCl_3$ (30%) contained in the industrial effluent allowed the elimination of 98%, 67%, 56%, 91% and 82% of turbidity, COD, ammonium, nitrate and phenol, respectively. These high removal efficiencies were attained by using 90 mg/L of $FeCl_3$ (30%). The overall data showed that the industrial effluent rich in $FeCl_3$ can be reused due to its significant elimination efficiency and low cost for the treatment of fuel-washing wastewater.

Keywords: Coagulation; Ferric chloride; Fuel washing wastewater; Treatment; Valorization

1. Introduction

Water is extremely used in domestic, agricultural and industrial processes. Such uses result in large amounts of produced wastewater which can be toxic [1,2]. Consequently, the release of the wastewater without any pre-treatment in aquatic environments can result in adverse effects and lead to health and environmental problems [3]. Such wastewater release can also aggravate the water problems in some areas, especially in countries that are considered in a water stress situation [4,5]. In this context, the Moroccan legislation is becoming more rigorous in making mandatory wastewater treatment before its release into the environment.

In addition, the release into the environment of large amounts of polluted wastewater creates a need for improvement of existing treatment technologies, which are less efficient and expensive to remove hazardous contaminants present in discharged industrial effluents.

In 2011, the global production of fuel washing wastewater was about 28 million m^3/y [6]. In addition, the fuel washing wastewater is recognized to induce a harmful effect on the environment due to the presence of a certain

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amount of oil, sediment, and traces of other toxic compounds. It should be noted that this released wastewater cannot be treated only by simple centrifugation, so additional treatment is required for better effluent purification. The thermal power station uses demineralized water to wash the fuel oil to remove mineral salts. In such a plant, the fuel washing water was found to be loaded with pollutants. Consequently, this wastewater must not be discharged into the aquatic environment before any specific treatment. In the present work, we have used the coagulation–flocculation process which is widely used in the treatment of fuel oil washing wastewater. Among the advantages are the investment cost, the operating cost, the ease of use and in general its good processing efficiency [7]. In addition, coagulation–flocculation is a practical energy process that is simple to implement, ecological and efficient [8,9]. For the treatment of industrial wastewater often use physicochemical techniques such as coagulation–flocculation followed by sedimentation as a solution for the treatment of industrial wastewater [10]. The level of efficiency of the process studied depends mainly on the nature of the coagulant agent, the dosage of the coagulant, the pH, the pollutant load and the nature of the organic compounds in the industrial wastewater to be treated [11]. Several coagulants are currently used in conventional wastewater treatment processes, namely aluminum sulfate, ferrous sulfate and ferric chloride [12]. Although $FeCl_3$ is one of the largest coagulants commonly used in wastewater treatment, not only because of its effectiveness in treating a wide variety of wastewater types but also because of its relatively low cost.

The main objective of this work is to enhance and compare the efficiency of commercial $FeCl_3$ and an industrial discharge rich in $FeCl_3$ in reducing the pollution of wastewater from fuel washing generated by the thermal power station. To this end, the coagulation–flocculation process was implemented to treat the wastewater from fuel washing produced by a thermal power station located in Mohammedia (Morocco). The performances of the two coagulants were compared regarding the efficiency of pollution elimination based on several indicators (COD, turbidity, nitrate, ammonium and phenol).

2. Material and methods

2.1. Sampling procedures

To assess the effluent quality obtained after the fuel washing, and to conform to the standard methods for the examination of wastewater, samples were taken before their treatment from the Mohammedia City Plant in Morocco. All the wastewater samples collected during the different stages of the process were mixed and placed in plastic collection tanks, then they were sent to the laboratory, and they were stored at 4°C.

2.2. Coagulation–flocculation test

In the present work, industrial effluent rich in $FeCl_3$ (30%) from a steel industry has been adopted as a second coagulant. Hydrochloric acid is used by the industry in several cleaning processes. And since iron oxide and hydrochloric acid (ferrous and ferric) are soluble in water, the rinsing waste is enriched with both hydrochloric acid and ferric chloride. The characteristics of this material and the commercial coagulant $FeCl_3$ (40%) are presented in Table 1.

A laboratory-scale evaluation of chemical coagulation–flocculation was studied employing ten place jar test apparatus. Ten beakers of equal volume (1 L) were used to examine the different doses of coagulant. The sample vials were carefully shaken to reconstitute the settled solids then 500 mL of sample were transferred to each jar test beaker. The experimental process goes through three successive steps: first initial rapid mixing at 160 rpm for 10 min, followed by a slow mixing for 30 min at 30 rpm, and then a final settling step for 1 h. After performing the treatment, the supernatant is withdrawn for analysis. To judge the efficacy of ferric chloride on wastewater treatment, the following characteristics are exanimated: the turbidity, the chemical oxygen demand (COD), ammonium and nitrate ions and phenolic compounds.

2.3. Physicochemical analyses

All samples were analyzed for physicochemical variables according to the procedure established in standard methods for the examination of water and wastewater.

The pH was measured as reported by the NF T 90-008 February 2001 (T 90-008) using (OHAUS Starter 3100). The turbidity was determined according to standard NF EN ISO 7027 March 2000 (T 90-033) by HACH 2100N Turbidimeter. Determination of the COD was evaluated using open reflux, titrimetric method (5220-B) [13]. 10 mL of sample was taken, then 5 mL of potassium dichromate and 15 mL of the silver sulfate solution (10 g/L) were added, then the samples were digested at 150°C for 2 h. The excess dichromate was titrated with ferrous sulfate ammonium in the presence of the ferroin as indicator, to determine the amount of $K_2Cr_2O_7$ consumed. The determination of the nitrates was performed by the spectrophotometric method with sodium salicylate according to standard STN ISO 7890-3 (75 7455). Thus, 5 mL of sample is introduced into a 60 mL capsule and then 0.5 mL of the sodium azide solution and 0.2 mL of acetic acid are added successively. 5 min later, the solution is evaporated to dryness in a water bath, then 1 mL of sodium salicylate solution is added, after evaporating and cooling, the residue is taken up in 1 mL of sulfuric acid. After 10 min 10 mL of deionized water and 10 mL of sodium

Table 1
Characteristics of the coagulants: (commercial, $FeCl_3$ 40%) and (industrial, $FeCl_3$, 30% contained in the rejects from the steel industry)

Parameter	$FeCl_3$ (40%)	$FeCl_3$ (30%)
pH	<1	<1
Conductivity (μS/cm)	18	20
Turbidity (NTU)	9.80	95.3
Fe^{3+} (g/L)	139	101.3
$FeCl_3$ (g/L)	400	295

hydroxide solution develop the yellow color. The value read on the spectrometer at the wavelength 415 nm.

The determination of the ammonium was performed by the spectrophotometric method indophenol blue following the AFNOR standards NF T90-015. 10 mL of the diluted sample are introduced, then 0.5 mL of the phenol and nitroprusside solution and 0.5 mL of the alkaline solution are added. The mixture is shacked and placed in the dark for at least 6 h. The readings are made by the spectrophotometer at 630 nm. Phenolic compounds were evaluated by the colorimetric method using the Folin–Ciocalteu (FC) based on the method described by Singleton and Rossi [14]. 0.5 mL of FC reagent is added to 10 mL of the diluted sample, then after 5 min, sodium carbonate is added. The mixture is homogenized and allowed to stand for 1 h. The absorbance measurement is carried out by a spectrophotometer at 725 nm.

3. Results and discussion

3.1. Fuel washing wastewater characterization

Fuel washing wastewater is a mixture of organic and inorganic pollutants. The reduction of the pollutant load by the physicochemical treatment is influenced by several factors, among them, the characteristics of the organic matter, the nature and the concentration of the other inorganic components, in addition to the design and the exploitation of the treatment. Therefore, the efficiency of the process can vary considerably. The characteristics of the raw wastewater samples are summarized in Table 2.

The analyses revealed that the fuel washing wastewater is characterized by 159 NTU of turbidity. Research has reported that petroleum refinery effluent presents 83 NTU of turbidity and selected the Fenton process as a treatment which is an effective method but it characterizes by its high cost and risks related to handling [15]. Concerning COD, an amount of 576 mg/L was detected in the fuel washing wastewater. While Dermentzis et al. [16] obtained 456 mg/L of COD in wastewater resulted from petroleum tanker truck cleaning. An electrochemical process was adopted in this study and demonstrated that electrocoagulation cannot reduce COD efficiently from petroleum wastewater without the addition of surfactants. On the other hand, the concentrations of ammonium and nitrate ions reached 9.25 and 5 mg/L respectively. The characterization of a similar effluent [17] revealed high concentrations of ammonium and nitrates of 44.16 and 7.41 mg/L, respectively. The fuel washing wastewater also presents a concentration of 14.61 mg/L of phenol. Generally, the presence of phenols in petroleum refinery wastewater is mainly a result of crude oil fractionation and thermal or catalytic cracking [18].

Table 2
Characteristics of the raw fuel washing wastewater

Parameter	Value
pH	8.72
Conductivity (μS/cm)	519.00
Turbidity (NTU)	159.00
Phenol (mg/L)	14.61
Ammonium ions (mg/L)	9.25
Nitrate ions (mg/L)	5.00
COD (mg/L)	576.00
Pt (mg/L)	37.00
Pb (mg/L)	0.35
Zn (mg/L)	3.96
Si (mg/L)	8.20
Al^{3+} (mg/L)	2.90
Fe^{3+} (mg/L)	20.90
Cu (mg/L)	0.50
TKN (mg/L)	20.50
O_2 (mg/L)	0.80

In general, the characteristics of industrial wastewater vary from day to day, which reflects the variation in the concentration of phenol found in the 5 different samples (Fig. 1). This considerable fluctuation can be attributed to the nature of the refined oil and the operating conditions during the refining process.

The variation in phenol concentrations from sample to sample can probably be due to process configuration and plant size. The samples from companies 2 and 3 showed higher concentrations because they were taken at a time when the thermal power plant was leaking fuel oil, while the concentrations found for the other samples are close to the concentration found by EL-Naas et al. [19] who confirmed that the initial concentration of phenol present in a petroleum refinery wastewater is 11.14 mg/L, its removal was carried through adsorption on date-pit activated carbon. Although other studies have found concentrations of 10 and 13 mg/L [20,21].

The concentrations of phenolic compounds differ according to the samples analyzed. It is probably due to the hydrophilic-hydrophobic properties of polyphenols. In general, high molecular weight polyphenolic compounds are labeled as hydrophobic (fat-soluble) based on their polyaromatic nature, while certain simple phenolic structures including phenolic alcohols are hydrophilic, which means that they have an affinity for water [22]. Most of the natural phenols that are separated in the aqueous phase during olive processing are polyphenols of the hydrophilic type [23].

Despite this considerable difference. Phenol concentration found in the studied effluent presents a risk whether for humans or aquatic life because phenol toxicity levels generally range from 9 to 25 mg/L [24]. This analysis leads us to conclude that these wastewaters are toxic and lead to physical, chemical, or biological degradation of the natural qualities of the water. This toxicity disrupts the living conditions and balance of the aquatic environment and compromises the use of water.

3.2. Effect of dose

3.2.1. Turbidity removal assessment

High concentrations of particulate matter can affect light penetration. It also provides fixation sites for other pollutants including metals and bacteria, and that can affect ecological productivity, and habitat quality [25]. For this

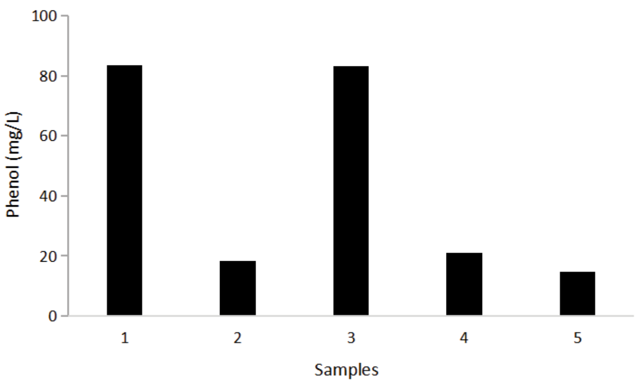

Fig. 1. Phenol concentration in different samples.

reason, turbidity can be used as a major indicator of water pollution. The most difficult suspended particles to remove in water are those which are too small (colloidal particles causing turbidity) and those which are dissolved (organic matter causing stains) [26]. To find out the optimum dose of coagulants, different doses ranging from 30 to 300 mg/L for $FeCl_3$ (30%) and from 40 to 400 mg/L for $FeCl_3$ (40%) were tested. The effect of the coagulant dose on turbidity reduction is presented in Fig. 2.

Available data indicated that optimal doses of $FeCl_3$ (40%) and $FeCl_3$ (30%) is about 160 and 90 mg/L respectively. Those values allowed reaching the maximal yield of turbidity elimination of 98%. The results of this study can be used as baseline data for petroleum wastewater treatment that uses ferric chloride as a powerful coagulant for turbidity reduction.

Altaher et al. [27] confirmed that the use of organic and inorganic coagulants as ferric chloride, in the treatment of wastewater is generally for the reason of its ability to reduce a huge amount of impurities inclusive of turbidity. In this context, a recently published article on biodiesel wastewater treatment by the coagulation–flocculation process mentioned that turbidity removal can reach between 82% and 99% [28]. Another study on the optimizing of coagulant process exhibited promising effectiveness in turbidity removal which reached 92.9%–99.4% using ferric chloride [29].

3.2.2. COD degradation

Higher COD levels mean more oxidizable organic matter in the sample, which will reduce dissolved oxygen (DO) levels [30]. A reduction in DO can lead to anaerobic conditions, which is harmful to aquatic life forms [31]. The COD test is an important parameter of water quality as it provides an index for assessing the effect of the wastewater released on the receiving environment.

In the present study, different doses of $FeCl_3$ (30%) and $FeCl_3$ (40%) were used in order to determine the optimum coagulant dosage for the COD reduction (Fig. 3).

Regarding the $FeCl_3$ (30%), it has been noted that the highest decrease in COD concentration was obtained by adding a coagulant dose of 90 mg/L. This optimal dose allowed eliminating 67% of COD. A similar investigation was carried out on biodiesel wastewater and demonstrated that the coagulation process resulted in a COD reduction of 63% at an optimum ferric chloride dose of 350 mg/L [32]. While another study on car wash wastewater demonstrated that the coagulation process resulted in a COD reduction of 65.25% using 500 mg/L of polyaluminium chloride coagulant [33]. On the other side, using 120 mg/L of $FeCl_3$ (40%) allowed to reach the highest COD removal of 92%. The result actually comes down to the fact of adsorption of the organic matter by the flocks and the neutralization of the charge [34].

In our case, the fuel washing wastewater quality has been improved by reducing the organic matter, which has reached a value (Fig. 3) below the standard required (COD: 300 mg/L) by state environmental legislation [35].

3.2.3. Reduction of ammonium ions

The ammonium ions are the main ingredient in most fertilizers and detergents. These chemicals are often found in water, either by surface drainage or by direct industrial discharge [36].

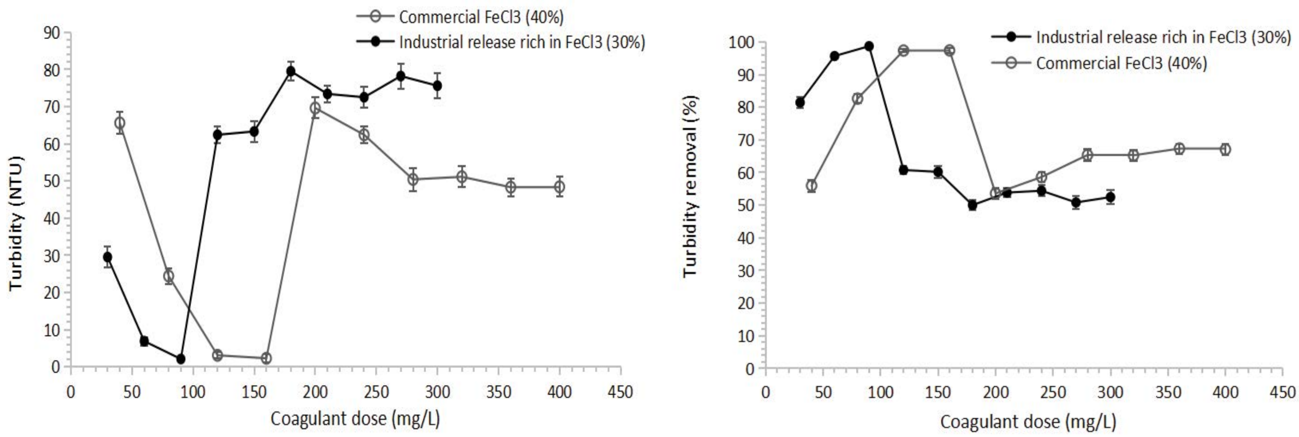

Fig. 2. Effect of the industrial release rich in $FeCl_3$ (30%) and commercial $FeCl_3$ (40%) on the reduction of turbidity, in terms of concentration and efficiency.

The results showing the effect of the dosage of the two coagulants on the elimination of ammonium are presented in Fig. 4.

Concerning $FeCl_3$ (30%), the optimal dose used for reducing 56% of ammonium ions is 90 mg/L. However, for $FeCl_3$ (40%) the ammonium concentration decreased by 61% using 160 mg/L as an optimal coagulant dose.

It is noted that the NH_4^+ reduction in the coagulation process is not too high. It is recognized that ammonium in industrial wastewater is generally eliminated by nitrification, thus, it is achieved by the complete oxidation of ammonium to nitrate [37]. This method requires the use of aerobic autotrophic nitrifiers and anaerobic heterotrophic denitrifiers for the conversion of nitrogen compounds to gaseous nitrogen (N_2) [38]. Unfortunately, autotroph microorganisms are sensitive to high loads of organic matter and ammonium. In addition, the nitrification process is prone to take time and occupies large spaces for the installation of separate aerobic and anaerobic reservoirs [39]. In the current case, ammonium was adsorbed on flocks obtained from the realization of the coagulation–flocculation process.

In aquatic systems, ammonium effects are less marked [40]. Whereas, the real problem is not the exposure of ammonium in water but rather the transformation of NH_4^+ into an aqueous form of NH_3 [41] according to Eq. (1).

$$NH_4^+ + OH^- \leftrightarrow NH_3 + H_2O \qquad pKa = 9.2 \qquad (1)$$

The presence of ammonia in a balance between the two chemical species: unionized ammonia (NH_3) and ammonium ions (NH_4^+) do not pose a risk to aquatic life. The neutral and unionized form spreads easily through the cell membranes at the gills of the fish. This makes NH_3 highly toxic to aquatic organisms [42]. At a pH greater than pKa (9.2) the NH_3 form of $NH_{4(aq)}^+/NH_{3(aq)}$ predominates.

Under the optimal conditions of the coagulation process using both coagulants, the pH decreases from 8.72 to a value less than 4. Therefore, the presence of ammonium no longer presents a risk.

3.2.4. Removal of nitrate ions

Nitrates (NO_3^-) are naturally present in the environment and are an important nutrient for plants [43]. Withal,

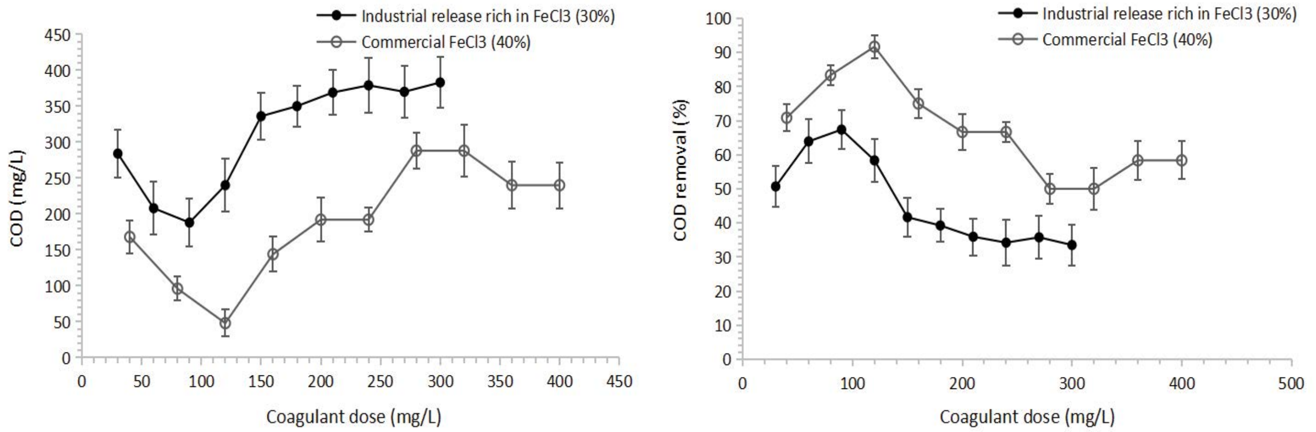

Fig. 3. Effect of the industrial release rich in $FeCl_3$ (30%) and commercial $FeCl_3$ (40%) on the reduction of COD in terms of concentration and efficiency.

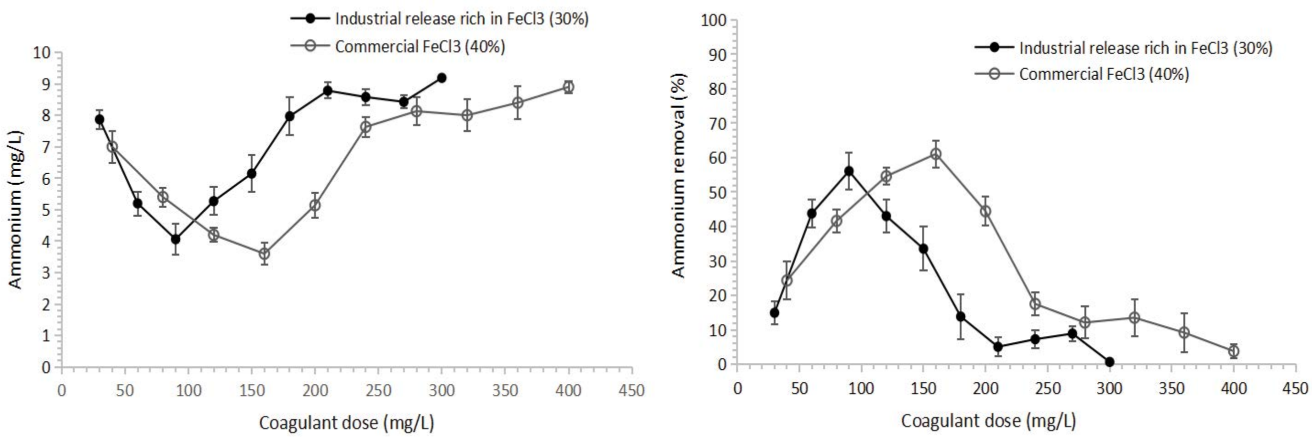

Fig. 4. Effect of the industrial release rich in $FeCl_3$ (30%) and commercial $FeCl_3$ (40%) on the reduction of ammonium in terms of concentration and efficiency.

contamination of water sources by nitrates is an international concern in recent years [44]. Many studies have shown that nitrate is toxic to aquatic animals because nitrate reacts with hemoglobin causing oxygen deficiency in their bodies (methemoglobin) [45].

Fig. 5 shows the nitrate removal results obtained by testing different doses of the two coagulants $FeCl_3$ (30%) and $FeCl_3$ (40%).

In the present study, the optimal $FeCl_3$ (30%) dose remarked was 90 mg/L of coagulant, which has been able to remove 91% of nitrate ions. On the other side, 96% of nitrate ions were eliminated applying $FeCl_3$ 40% with 120 mg/L as an optimal dose.

As already reported, a chemical reaction to occur between iron and nitrate, pH values must be higher than 9 [46]. Since the initial pH value was 8.72, the removal of nitrates could not be achieved by chemical reaction with iron from the coagulant. Consequently, the removal of nitrates by ferric chloride was affected by adsorption and precipitation. A comparison between the two technologies coagulation and electrocoagulation according to their efficiency of water nitrates was made [47]. The results obtained from this study demonstrated that electrocoagulation is a more efficient process for the appearance of nitrates that comes down to the preference of the adsorption of nitrate anions on the surfaces of more metal hydroxide precipitates.

3.2.5. Removal of phenol

Among the pollutants that may be present in industrial wastewater. Phenol is one of the most hazardous compounds in industrial wastewater due to its high toxicity and low biodegradability [48]. It is present in several industries effluents including petrochemicals, oil refining [49]. The U.S. Environmental Protection Agency (EPA) [40] has considered phenol as a priority pollutant since the existence of phenol in natural waters during disinfection and oxidation processes may be a major cause of the formation of other toxic compounds [50]. Moreover, this compound is easily soluble in water, oils, carbon disulfide and many organic solvents [51]. The phenol removal efficiencies obtained by varying the doses of the two coagulants are shown in Fig. 6.

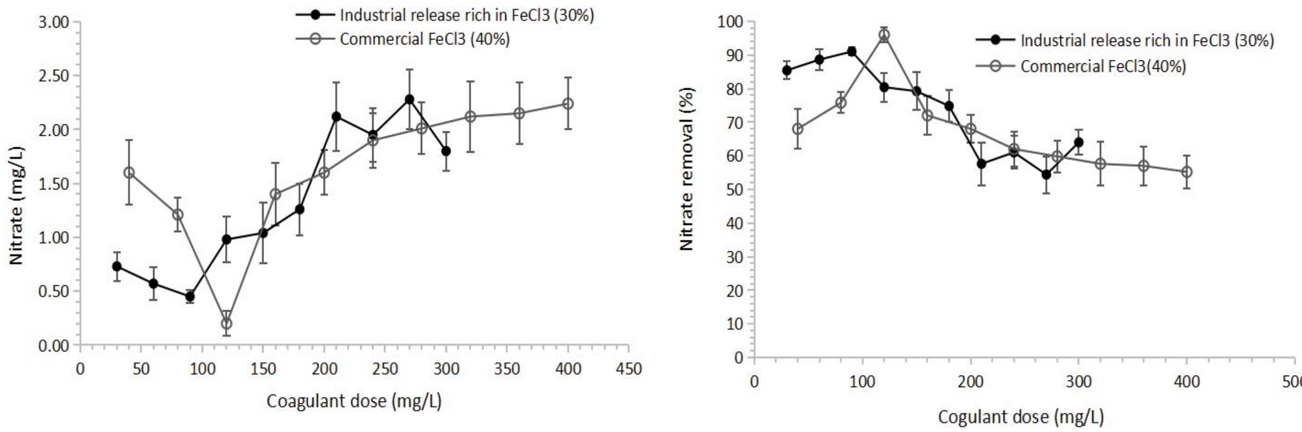

Fig. 5. Effect of the industrial release rich in $FeCl_3$ (30%) and commercial $FeCl_3$ (40%) on the reduction of nitrate ions in terms of concentration and efficiency.

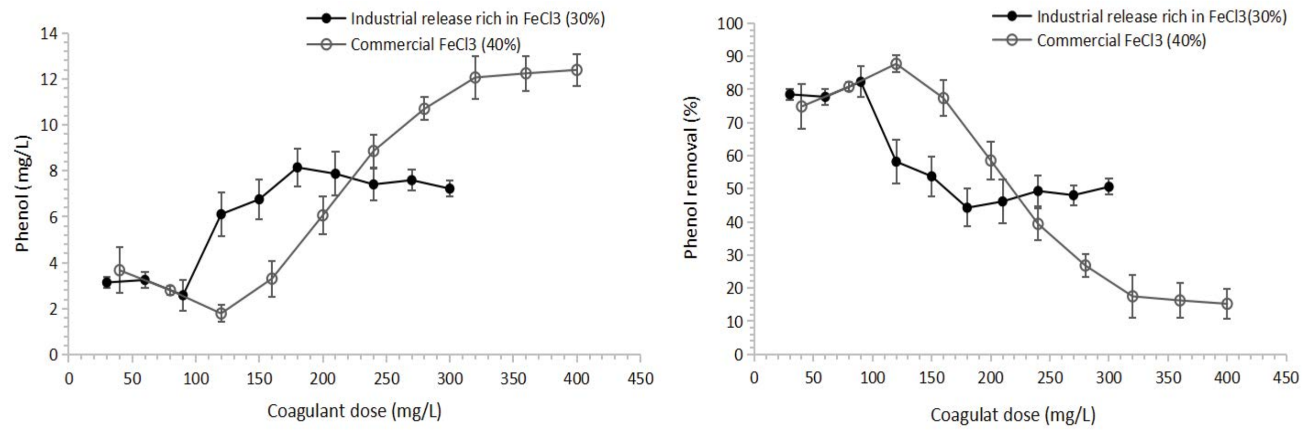

Fig. 6. Effect of the industrial release rich in $FeCl_3$ (30%) and commercial $FeCl_3$ (40%) on the reduction of phenol in terms of concentration and efficiency.

As regards $FeCl_3$ (30%), 90 mg/L has reduced phenol concentration by 82% (Fig. 6). Whereas using 120 mg/L of $FeCl_3$ (40%) the phenol removal reached 88%.

A review on phenol removal explained that the key factor for the appropriate treatment process is the level of phenol concentration (low, medium, high) [49]. It is important to emphasize that the coagulation–flocculation process is convenient with the fuel washing wastewater due to his low phenol concentration, so it easily adsorbed by flocks. This physicochemical method was highlighted by Özbelge et al. [52] on the wastewater of a tire-cord manufacturing plant using ferric chloride with a concentration of 50% which allows obtaining the optimal conditions of treatment with an elimination yield up to 94%.

3.3. Comparison for the performance of coagulants

The removal efficiency obtained with $FeCl_3$ (30%) was compared to that achieved with $FeCl_3$ (40%) in similar conditions. Fig. 7 illustrates the comparative study of the removal yields of different parameters by 40% $FeCl_3$ and industrial waste rich in 30% $FeCl_3$.

Experimental results revealed that the dose of 90 mg/L $FeCl_3$ (30%) is the optimal dose for the removal of all parameters studied with yields of 98%, 67%, 56%, 91%, 82% for turbidity, COD, ammonium, nitrate and phenol, respectively. while COD, nitrate ions and phenol are the only pollutants that could achieve the highest removals at the optimal dose of 120 mg/L $FeCl_3$ (40%) with yields of 92%, 96% and 88%, respectively.

The Brownian motion of the colloidal particles suspended in the wastewater causes a repulsion of these particles by forming a stable dispersed suspension, which creates difficulties during subsequent operations [53].

The most important mechanisms to explain the coagulation of inorganic iron salt are adsorption and neutralization. The addition of $FeCl_3$ to the wastewater leads to the formation of positively charged iron and chloride ions which neutralize with the negatively charged colloidal particles. Once the charge is neutralized, the small particles in suspension are able to stick together. Shi et al. reported that the amorphous ferric hydroxide ($Fe(OH)_3$) formed in the ferric chloride coagulation treatment, polymerized to form large particles (~5,000 nm) rapidly [54].

In addition, ferric ions react with hydroxide ions in water to form ferric hydroxide, which in turn can adsorb contaminant particles by sweep flocculation [53].

This clarifies that the difference in concentration of Fe^{3+} and $FeCl_3$ in the two coagulants explains the difference in the optimal yields of the two coagulants, the commercial ferric chloride and the ferric chloride-rich discharge. Thus, the concentration and the coagulant dose are crucial to enhance the effectiveness of the coagulation process and influence the choice of coagulant.

4. Conclusions

To conclude, the present study consists on the evaluation of the reduction in pollution of an industrial release rich in toxic pollutants, using $FeCl_3$ (40%) as a commercial coagulant and an industrial release of steel industry rich in $FeCl_3$ (30%). The results obtained from the coagulation–flocculation led to the conclusion that the use of commercial $FeCl_3$ (40%) eliminated 92%, 96% and 88% of COD, nitrate, and phenol, respectively. While the effluent rich in $FeCl_3$ (30%) eliminated 98%, 67%, 56%, 91%, and 82% respectively of turbidity, COD, ammonium, nitrate, and phenol, provided a comparative study between the two coagulants. These high removal amounts have been attained using an optimal dose of 120 mg/L for the commercial $FeCl_3$ (40%) and 90 mg/L for the industrial release rich $FeCl_3$ (30%). Overall, these findings demonstrate that:

- Coagulation–flocculation with $FeCl_3$ is a more interesting technique to use to effectively reduce pollution from an industrial release rich in toxic pollutants.
- The comparative study of the two coagulants showed that coagulation by industrial discharge rich in $FeCl_3$ leads to a significant reduction in pollution by using a lower concentration compared to commercial $FeCl_3$.
- The valorization of the industrial coagulant leads to a considerable saving for the fight against pollution

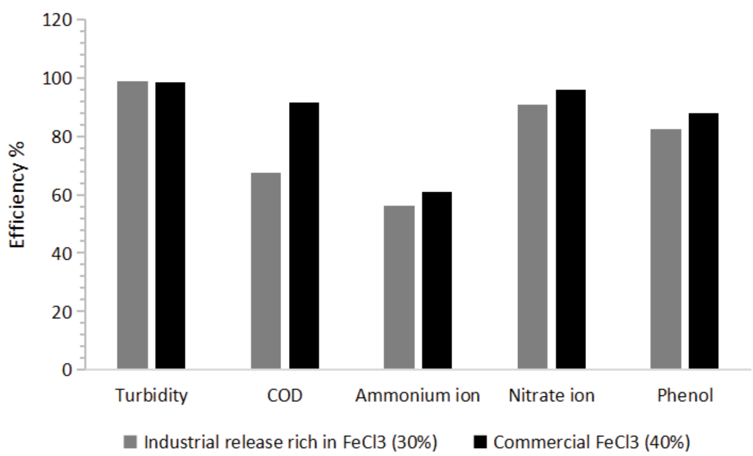

Fig. 7. A comparison between the effect of the industrial release rich in $FeCl_3$ (30%) and commercial $FeCl_3$ (40%) on the reduction of turbidity, COD, phenol, nitrate and ammonium ions.

of industrial waste at the lowest cost, which presents valuable economic and environmental aspects. In addition, the company responsible for producing $FeCl_3$-rich releases operates 24 h a day, producing a large volume of $FeCl_3$-rich liquids.

This study shows that the recycling of liquid industrial waste offers an excellent solution to treat in turn the wastewater with the lowest cost, generating hence environmental and economic benefits. The present work has focused on the valorization of the steel industry effluents and their uses as effective coagulants for the treatment of the fuel washing wastewater.

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