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Removal of typical pollutants from water sources of Chouara Tannery by the integration of coagulation–flocculation and activated carbon treatment

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ABSTRACT

This study aims to valorize Chouara Tannery's water sources (the Boukhrareb creek and local underground water) by eliminating some typical pollutants using coagulation–flocculation process followed by activated carbon filtration. For the coagulation–flocculation process, we used ferric chloride ($FeCl_3$) and aluminum sulfate ($Al_2(SO_4)_3$) separately with different dosages to choose the most efficient coagulant. According to the experiment results, analyzed pollutants, heavy metals, and turbidity were well decreased by ($FeCl_3$) with an optimal concentration of 15 mg/L for Boukhrareb creek and 20 mg/L for underground water. In particular, turbidity was reduced with coagulation–flocculation process; it has reached 0.27 NTU for Boukhrareb creek, as well as 1.8 NTU for local underground water after activating carbon filtration. In addition, some toxic heavy metals such as iron, phosphorus, and aluminum were removed up to 99% and mercury for 100% for both water samples. For this reason, this research strongly suggests using ferric chloride as a coagulant–flocculant followed by activated carbon filtration before using them in Chouara Tannery leather production's process.

Keywords: Coagulation–flocculation; Activated carbon filtration; Vegetable tanning; Typical pollutants; Tannery water sources

1. Introduction

Chouara Tannery located in Fez-Morocco (Fig. 1) was built in the 11th century and listed in UNESCO's World Heritage Site due to the fact that it still uses traditional vegetable tanning process (Fig. 2) and avoids the use of chemicals such as chromium. It uses slaughterhouses waste to produce leather but consumes a large quantity of water during its process. The leather tanning industry is significant to the Moroccan economy, especially in Fez city while it participates with 53% in the city's employment, 24% of industrial production, and 23% of benefits [1]. Because of the tannery's needs and dimensions, it uses two water sources nearby Boukhrareb creek and underground water. The first one is under heavy contamination while it receives all the city's industries wastewater and municipal sewage

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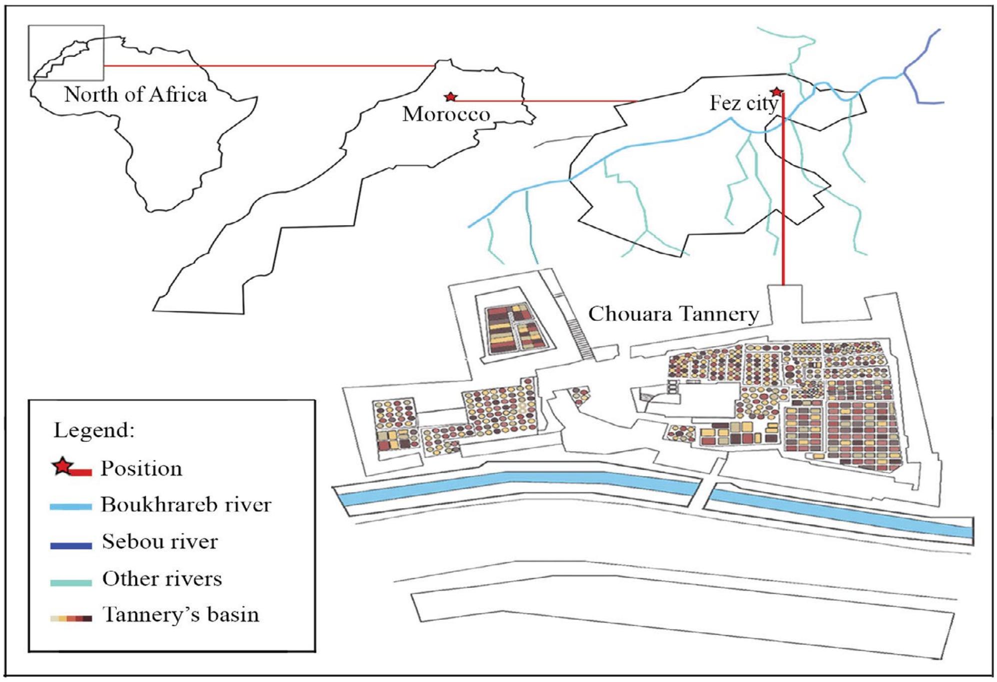

Fig. 1. Boukhrareb creek, Sebou River, and Chouara Tannery's locations. \

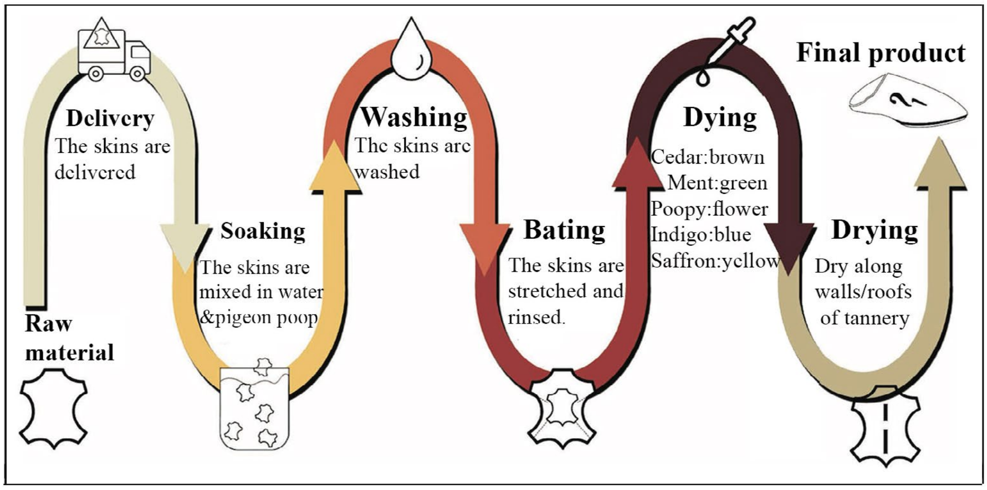

Fig. 2. The vegetable tanning process of Chouara Tannery.

without any treatment to flow directly to the biggest river in Morocco called Sebou [1,2]. This heavy pollution goes through soil until it contaminates the underground water and decreases its quality. Both sources are illustrated in Fig. 1. The analysis results of both sources showed a high level of turbidity, total suspended solids (TSS), chemical oxygen demand (COD), and some heavy metals that affect the tannery's product quality and tanners health as well [1,3]. This represents a serious environmental threat to the aquatic environment and human health [3,4].

Coagulation–flocculation treatment has been submitted among many treatment processes to treat some heavy metals, turbid water with a high content of suspended and colloidal particles [5–8]. It has been known by contributing its mechanism to compress the electrical double layer and to decrease the repulsive potential of colloids in consequence. In addition, it has been recognized by its cost efficiency, time-saving, and the need for small sites [9]. Thus, so far, different experiences have been focused on the performance and the optimization of coagulation–flocculation in treating tannery's wastewater. Haydar and Aziz [10] compared between different coagulants added the appropriate coagulant aid, the chemicals cost decreased by 50% and sludge volume by 60%–70%. In another article, to treat tannery's wastewater, Song et al. [11] used two different coagulants: ferric chloride and aluminum sulfate. Better results were obtained with ferric chloride with 40% removal of COD, 69% for TSS, and 74%–99% for chromium. In spite of that, water quality deterioration has pushed the researches to look for other treatment processes combined with the conventional coagulation–flocculation method, among which granular activated carbon is well-studied particularly for removing pesticides, toxic chemicals, tastes, and odors. Ayoub et al. [12] reported that a high diminution of TSS, phosphorous, turbidity, and chromium was observed using coagulation–flocculation (aluminum sulfate and ferric chloride) followed by activated carbon [12]. Puchana-Rosero et al. [13] demonstrated that tannery's wastewater could be treated by coagulation followed by activated carbon adsorption.

Nevertheless, all reported articles were about treating chemicals and dies of tannery wastewater [14,15], but there was no detailed experiment about using coagulation–flocculation combined with activated carbon to treat tannery's water source. Moreover, the importance of this study consists of the vegetable tanning process of the tannery and the concentration of chromium and heavy metals that has to be improved. In addition, uncertainty remains as to the removal rate of heavy metals when the composition of water varies [16–18].

Therefore, this tannery aims to have 100% environmental products that can be recycled several times while it uses vegetable tanning process but its polluted water sources obstruct it. In this study, we opted for treating Chouara Tannery's two water sources, which could be used directly after the chosen treatment in order to remain in an ecological process with no toxic chemicals (Chouara Tannery's objective). Furthermore, due to the Chouara Tannery's land dimension, we needed a hybrid treatment process that fits in a small space, time-efficient, economically advisable, and easily operated [19]. Besides, it will not damage the heritage of the place. For those reasons, we recommended the use of coagulation–flocculation treatment followed by activated carbon filtration to eliminate analyzed pollutants and toxic heavy metals especially mercury.

2. Materials and methods

2.1. Sample collection

The first step of our process is collecting samples from Chouara Tannery. It is important to mention that during the collecting of all water samples, the closed bottles were dipped in each kind of water at the depth of 0.7–0.9 m [20]. After that, the bottles were opened inside and were closed again to bring them out to the surface. Before collecting the water samples, all the sampling bottles were rinsed in the laboratory with distilled waste and rinsed once again with sampled water.

In the first process of the tannery, the creek water was taken straight from the creek by a pump. For the tanning process, the underground water was put in a big basin by a pump for easy use. Therefore, the creek water samples were taken directly from the creek, from the same point where the pump is located and the underground water samples were taken forthrightly from the basin. They are collected for physiochemical, organic, inorganic, biological analyses, and treatment essays. These samples were placed in different bottles depending on the way of use and transported to the laboratory promptly. Both, the creek water and the underground water were stored at 4°C in a deep freezer and used in the experiments without any dilution. However, this transformation generates large amounts of polluted water containing solid waste, organic skin, salts, and chemicals that are discharged either in the sewer system or in public pipes or nature [21].

2.2. Coagulation–flocculation

To evaluate the overall pollutant removal performance, it was necessary to optimize parameters that effect directly the process efficiency. Considerable researchers had suggested that the most crucial parameters in coagulation–flocculation are those in the concentration added in coagulants and pH [22–24]. Therefore, aluminum sulfate ($Al_2(SO_4)_3$) and ferric chloride ($FeCl_3$) were used as coagulant and flocculant respectively. Those two chemicals (Aladdin, China) were used separately in coagulation–flocculation treatment of Boukhrareb creek and underground water samples to find out the most appropriate one for each sample. Also, the chemicals were prepared as solutions (1% w/v) by dissolving in distilled water to achieve 100 mL. The temperature of the laboratory was recorded at 25°C during the jar test. An equal volume (1 L) of the samples was measured in six stirrers of the jar test apparatus (Phipps and Bird, Inc., USA) [25,26]. Jar test performed as follows: for coagulation, we fixed the stirrer operating at the "flash mix" with an acceleration relatively 120 rpm fix for 2 min followed by flocculation (where chemicals are added) at 25 rpm for 30 min. In the case of aluminum sulfate, concentrations applied for underground water were 5, 10, 15, 20, 25, and 30 ml/L for creek water samples were 20, 25, 30, 35, 40, and 45 ml/L. In the same way, ferric chloride was tested with concentrations 5, 10, 15, 20, 25, and 30 ml/L in underground water and 15, 20, 25, 30, 35, and 40 ml for creek water samples. Then, the samples were settled for 30 min to fully settle all the floc. Next, the supernatants of the treated sample were taken to analyze its turbidity, pH, and some heavy metals. According to treated samples analysis results, the best results of each chemical were taken to activated carbon filtration.

2.3. Activated carbon filtration

To make a filtration column, 50 g of granular activated carbon (Sinopharm Chemical Reagent Co., Ltd., China) was washed by distilled water four times and then filled into a glass chromatography column (16 mm inner diameter and 300 mm long) that was previously inserted with 15 g of degreasing cotton (Sinopharm Chemical Reagent Co., Ltd., China). Characteristics of the granular activated carbon are measured as follows: the total surface area was 500–1,000 m^2/g; apparent density was 450 kg/m^3 and the iodine number was 1,104 mg/g. With the optimum dosage of each coagulant, effluent from the sedimentation tank entered into the filtration column and filtrated samples were collected for analyses. Before each filtration experiment, the activated carbon with the filtration column was washed four times and then rinsed by the sample to be filtered another time. During the filtration, the flow rate was fixed at 5 mL/min.

2.4. Apparatus and analytical method

All analyses were conducted in duplicate according to Standard Methods [27] and the same results were reported. COD was tested by a photometer (PALINTEST 8000). Biochemical oxygen demand (BOD_5) was analyzed by colorimetric method using a spectrophotometer. Conductivity was analyzed by a CONSORT K 912 probe. The pH was examined by a pH meter (GPHR 1400). For turbidity, we used the Nephelometer (HANNA Instruments TB200). TSS was examined by evaporation (OHAUS MB35).

3. Results and discussions

The analyses of all water samples and coagulation–flocculation treatment followed by activated carbon filtration were done in a laboratory. Fig. 3 summarizes all processes described before.

3.1. Physicochemical characteristics of raw water sources and tannery

3.1.1. Wastewater

A physicochemical analysis was done to creek's raw water and underground raw water in order to know the pollution degree of each one. The analysis results are illustrated in Table 1 below.

An excessive concentration of all characteristics is observed from the Table above. In other words, a higher amount of COD means that both samples have oxidizable organic materials. This reduces the dissolved oxygen quantity that may lead to an anaerobic condition. COD is higher than BOD while COD contains both biodegradable and non-biodegradable substances, however, BOD includes only biodegradable substances. So, those results demonstrate heavy pollution existing because of organic matters and indicating the under severe threats of the aquatic life, which was inconsistent with the observation of other researchers [2,3]. The total solid waste of both samples is high and it can be seen in eye scale while the colors of the samples are too dark that block the light and lead to less oxygen, resulting in various problems such as reducing tannery's leather quality efficiency and aquatic life of the creek. In addition, the high concentration of TSS can be the result of the increase of bacteria, nutrients, pesticides, and metals amounts in both water samples. High results of the turbidity are because of the intensity of scattered light and the creek's turbidity is due to the discharge of the entire city's wastewater to the creek without any preceding treatment. The creek water's pH is more than seven because it is slightly alkaline and contains a lot of OH^- concentration. A higher pH affects people's health; it could cause skin and eye irritations and increase the risk of mobilized toxic metals that can be absorbed by humans. In the tannery process, tanners have to be inside the water to treat the skin. As a result, tanners tend to have many skin problems because of this polluted source.

The heavy metal analyses of both samples hold a large quantity of Mercury and this is due to the discharge of wastewater of some city industries that use this toxic chemical. This causes several environmental and health issues such as death of aquatic life, algal blooms, habitat destruction from sedimentation, debris, other short and long term toxicity from chemical contaminants. The other toxic heavy metals are almost non-existent. Other typical pollutants such as iron (Fe), phosphorus (P), and aluminum (Al) have to be decreased.

3.2. Coagulant–flocculation experiment

3.2.1. Turbidity and pH analysis after coagulation–flocculation experiment

After coagulation–flocculation and sedimentation, upper supernatants were drawn to samples using a pipet to examine their turbidity and pH of treated samples. Extracted samples were placed in tubes to avoid the generation of air bubbles and to be analyzed [28]. Fig. 4 illustrates the turbidity and pH of the creek water samples

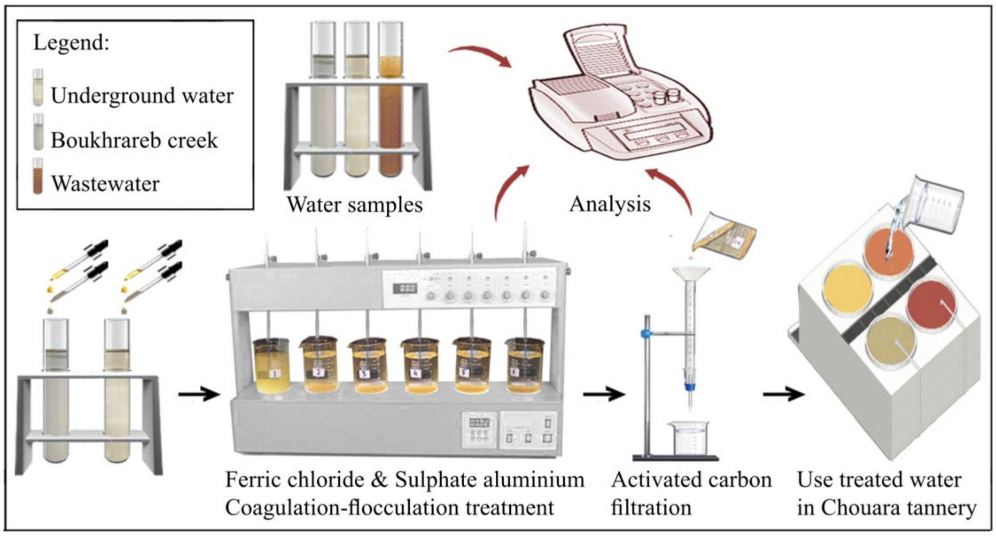

Fig. 3. Schematic description of the whole process.

treated by six different dosages of ferric chloride (Fig. 4a) and aluminum sulfate (Fig. 4b) ranging from 5 to 30 mg/L. Fig. 5 shows underground water samples results of the same experiment ranging from 15 to 45 mg/L.

We can observe that the underground water turbidity decreased to 0.53 NTU with a pH of 7.5 after using ferric chloride as a coagulant flocculant and 0.53 NTU with a pH of 7.3 after using aluminum sulfate as a flocculant. For creek water turbidity and pH, they reduced to 2.4 NTU and 7.5 after using ferric chloride as a flocculant and 4.0 NTU with a pH of 7.4 using aluminum sulfate respectively. Even if we used different flocculants and dosages in both samples, pH was remained between neutral and slightly alkaline which is close to the optimum value of 7.5 [29]. This can be explained as the coagulant dosage was beneficial to pollutants elimination and helped to keep a low concentration of flocculant cation after coagulation–flocculation [30,31]. According to these data, ferric chloride performed better than aluminum sulfate at the specified conditions. This can be explained by ferric chloride's color of Fe^{3+} [31] and its superior efficiency in removing turbidity and pH while once the coagulant flocculant dose increases the turbidity and pH decrease until it reaches their optimal [32].

To conclude, the optimum coagulant flocculant for the underground water dose was 15 mg/L with ferric chloride and 20 mg/L with aluminum sulfate. For the creek water sample, the optimum concentration was 20 mg/L for ferric chloride and 40 mg/L with aluminum sulfate. This jar test experiment was satisfying in this case; while we could have a trans-lucid watercolor compared with before and reduced the dissolved substances in water.

Table 1
Composition of the creek water underground water

Parameters	Boukhrareb creek raw water	Underground raw water
COD (mg/L)	2,950	2,570
TSS (mg/L)	96	47
pH	8.1	7.9
BOD_5 (mg/L)	110	105
Turbidity (NTU)	1,093	1,204
Hg (mg/L)	6.800	8.100
Zn (mg/L)	0.299	0.105
Mn (mg/L)	0.123	0.037
Cu (mg/L)	0.09	0.148
Pb (mg/L)	0.021	<0.004
Ni (mg/L)	0.017	0.008
Cr (mg/L)	0.015	<0.004
As (mg/L)	<0.004	0.005
Se (mg/L)	<0.004	<0.004
Cd (mg/L)	<0.001	<0.001
Fe (mg/L)	3.606	0.037
Al (mg/L)	2.830	0.180
Ba (mg/L)	0.128	0.044
B (mg/L)	0.080	<0.004
Ba (mg/L)	0.128	0.044
P (mg/L)	3.200	1.090

3.2.2. Heavy metals dosage

The samples with the optimum dosage of each coagulant were taken to analyze the removal efficiency of heavy metals and typical pollutants illustrated in Tables 2 and 3. According to creek water sample results, mercury and chromium decreased with the amount of 99.99% with both coagulants. Aluminum was reduced by 99% with ferric chloride and 91% with aluminum sulfate, iron decreased by 90%, and 97% and phosphorus removed by 84% and 84% respectively. For underground water sample results, mercury was removed by 99% with both coagulants; aluminum was reduced by 95% with ferric chloride and 22% with aluminum sulfate while we used residual aluminum sulfate, chromium, and iron reduced by 98% and phosphorus by 65% with both coagulants. Apart from heavy metals, COD decreased in both water samples, it dropped by 86%, TSS

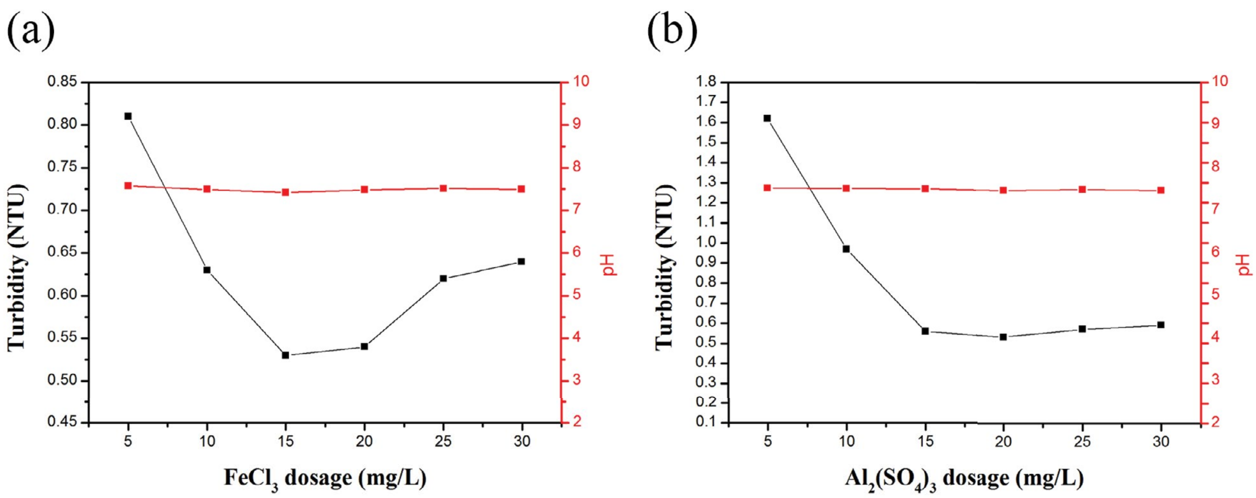

Fig. 4. Turbidity and pH of underground water samples treated by different dosages for each flocculant.

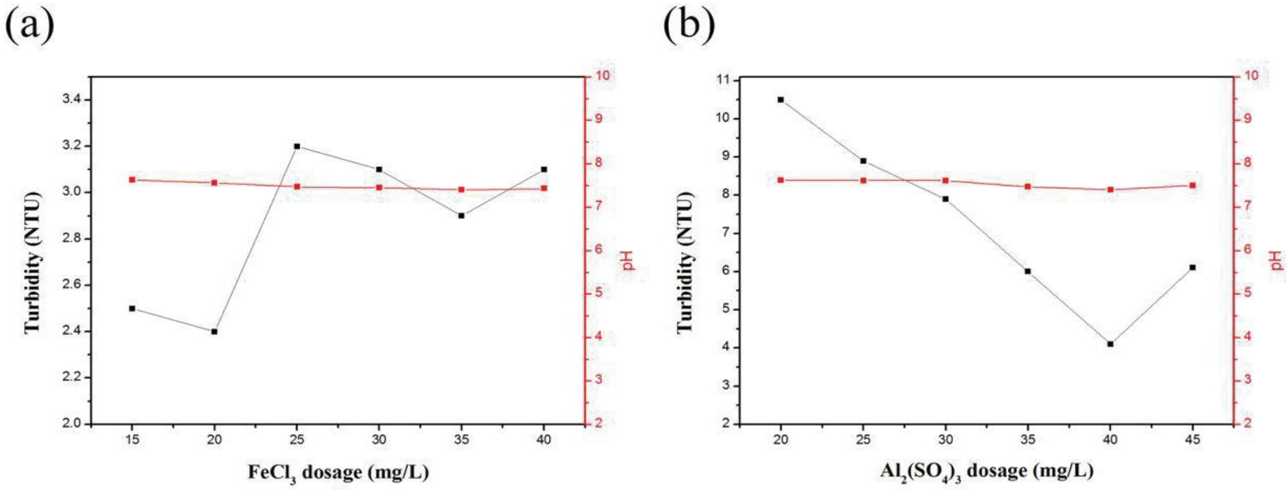

Fig. 5. Turbidity and pH of creek water samples treated by different dosages for each flocculant.

Table 2
The concentration of typical pollutants in creek water after coagulation-flocculation with different coagulants

Parameters	Coagulant	
	Ferric chloride	Aluminium sulfate
COD (mg/L)	400	550
TSS (mg/L)	9.0	10
BOD_5 (mg/L)	50	65
Hg (mg/L)	0.0003	0.0030
Cr (mg/L)	0.0002	0.0002
Fe (mg/L)	0.0904	0.0275
P (mg/L)	0.5067	0.5372
Al (mg/L)	0.0394	0.2728

Table 3
The concentration of typical pollutants in underground water after coagulation–flocculation with different coagulants

Parameters	Coagulant	
	Ferric chloride	Aluminium sulfate
COD (mg/L)	350	415
TSS (mg/L)	5.0	7.0
BOD_5 (mg/L)	40	50
Hg (mg/L)	0.0002	0.0030
Cr (mg/L)	0.0001	0.0003
Fe (mg/L)	0.0156	0.0466
P (mg/L)	0.6487	0.6375
Al (mg/L)	0.1410	0.1968

reduced by 90% using ferric chloride, and 89% with aluminum sulfate, BOD_5 only with 55% using ferric chloride and 62% with aluminum sulfate. This has been established through a variety of studies [33–35], while colloidal metallic hydroxides and organic compounds are removed during coagulation–flocculation treatment. This was because the coagulant added promotes the agglomeration of small particles into bigger sizes to become suitable for settlement. As a conclusion, both samples delivered a good performance for reducing organic pollutants and heavy metals analyzed with ferric chloride and aluminum sulfate.

3.3. Activated carbon filtration

To purify water for treatment, it needs to go through the coagulation–flocculation process using a coagulant. In spite of the fact that this step decreases dissolved typical pollutants and suspended substances in the water, after coagulation–flocculation treatment, the concentration of toxic mercury almost disappeared. Therefore, to ensure the improvement of water quality, a successive filtration treatment with activated carbon was added. The optimum result of coagulation–flocculation treatment of both water source samples went through activated carbon filtration. The results procured by the analysis of both samples after activated carbon filtration are shown in Table 4.

As observed in Table 4, high turbidity elimination was achieved using carbon filtration. The underground water sample with ferric chloride reduced from 0.53 to 0.27 NTU and with aluminum sulfate decreased from 0.52 to 0.30 NTU after activated carbon treatment. For the creek water sample with ferric chloride, it reduced from 2.40 to 1.80 NTU and from 4.0 to 3.1 NTU after coagulation with aluminum sulfate. These results showed that the activated carbon is an effective adsorbent because of its porosity and large surface area in which contaminants may be adsorbed. We also analyzed some typical pollutants in the effluent of activated carbon filtration, and the results are demonstrated in Tables 5 and 6.

After activated carbon filtration of all samples, all analyzed characteristics were reduced. The first percentage that will be given is activated carbon filtration of Boukhrareb creek sample with ferric chloride and the second one with aluminum sulfate. COD reduced by 96.61% and 95.76%, TSS decreased by 93.73% and 92.29%, BOD_5

Table 4
Turbidity and pH of samples from both water sources after activated carbon filtration with different coagulants used in the previous coagulation–flocculation process

Water sources	Parameters	Coagulants used in the previous coagulation–flocculation process	
		Ferric chloride	Aluminium sulfate
	Turbidity (NTU)	1.80	3.10
Creek water	pH	7.50	7.50
Underground water	Turbidity (NTU)	0.27	0.30
	pH	7.40	7.50

Table 5
The concentration of typical pollutants in creek water after activated carbon filtration with different coagulants used in the previous coagulation–flocculation process

Coagulants used in the previous coagulation–flocculation process		
Parameters	Ferric chloride	Aluminium sulfate
COD (mg/L)	100.0	120.0
TSS (mg/L)	6.000	7.400
BOD_5 (mg/L)	40.00	45.00
Hg (mg/L)	0.0001	0.0001
Cr (mg/L)	0.0001	0.0001
Fe (mg/L)	0.0005	0.0007
P (mg/L)	0.0101	0.0072
Al (mg/L)	0.0002	0.0018

Table 6
The concentration of typical pollutants in underground water after activated carbon filtration with different coagulants used in the previous coagulation–flocculation process

Coagulants used in the previous coagulation–flocculation process		
Parameters	Ferric chloride	Aluminium sulfate
COD (mg/L)	89.00	98.00
TSS (mg/L)	4.000	5.000
BOD_5 (mg/L)	35.00	37.00
Hg (mg/L)	0.0001	0.0001
Cr (mg/L)	0.0001	0.0001
Fe (mg/L)	0.001	0.001
P (mg/L)	0.006	0.007
Al (mg/L)	0.007	0.011

removed by 63.63% and 59.09% respectively. Also, iron reduced by 99.86% and 99.80%, phosphorus decreased by 99.68% and 99.33% and aluminum removed by 99.99% and 96.52% respectively. However, mercury and chromium concentrations were removed by 99.99% which means that they became almost non-existent after activated carbon filtration. For underground water sample, COD removed by 96.53% and 96.18%, TSS eliminated by 91.48% and 89.36%, BOD_5 decreased by 66.66% and 64.76%, iron decreased by 97.29% in both samples, phosphorus was removed by 99.44% and 99.35% and aluminum reduced by 96.11% and 98.99%. This means that the adsorption of toxic heavy metals ions found using commercial activated carbon [27] helped us to get the best results because of its activation while it helps the reduction of heavy metals and the reduction of the color into natural watercolor. This shows that activated carbon filtration was necessary for effective adsorption of the metal ions. In general terms, the expansion of the concentration of the toxic heavy metals is due to the deduction of the oxygen (O_2) while we have the absorption of a substance by a living organism which means by activated sludge microorganism. The toxic heavy metals hold the role of restraining the biodegradation microorganisms activities processes. Afterward, the uptake of O_2 is done by microorganisms in order to reduce the substrates that were degraded. So, from the outcome got from the analyses, a positive reduction of toxicity scale was shown. Our research illustrated that the Hg was one of the most adsorbed and toxic to activated sludge microorganisms. But still, through comparing the raw results with after activated carbon results we can understand that the fact of using coagulation–flocculation with ferric chloride or aluminum sulfate followed by activated carbon offered very good results. The quantities of heavy metals that existed previously in the samples are almost inexistent.

4. Conclusion and future outlook

Coagulation–flocculation followed by activated carbon filtration was used to treat Chouara Tannery's water source. Using ferric chloride as a coagulant gave us an optimum concentration and better turbidity, heavy metals, and other typical analyzed pollutants results for both sources samples comparing it with aluminum sulfate in the process of coagulation–flocculation and activated carbon filtration. To conclude, this technique proved that it can remove most of the heavy metals and reduce the turbidity and COD as well as it showed a good solution to this tannery problem. For this reason, our study highlight is on the application of this technique for this case. Also, it is easy to use, simple, and very cost-effective while the activated carbon can be used several times and it will improve the product's leather quality to obtain an eco-friendly product and protect tanner's health. In addition to that, the importance of this case pushed us to design a summary of the process flow sheet studied in reality demonstrated in Fig. 6.

Fig. 6. Summary design of the process flow sheet studied in reality.

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