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Research article

Evaluating the effectiveness of coagulation–flocculation treatment on a wastewater from the moroccan leather tanning industry : An ecological approach

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ABSTRACT

The removal of pollutants from tannery wastewaters, which is renowned for its substantial volumes, intricate composition, and considerable hazards to human health and the environment, is a prominent research area in the field of water treatment. The aim of this study is to employ a bio-coagulant derived from *Parkinsonia aculeata* seeds and a bio-flocculant derived from *Hibiscus esculentus* to minimise the concentration of pollutants in the combined wastewater originating from tanneries. In the course of the research, a thorough physicochemical analysis of the coagulating and flocculating agents, *Parkinsonia aculeata* (PA) and *Hibiscus esculentus* (HE), was performed using techniques such as XRD (X-ray diffraction), FTIR (Fourier-transform infrared spectroscopy), and SEM-EDS (scanning electron microscopy-energy dispersive X-ray spectroscopy). This analysis aimed to determine the composition and characteristics of these biomasses. Subsequently, a comprehensive overview was conducted to summarize the various factors that influence the treatment of tannery wastewater through coagulation/flocculation. This was accomplished by manipulating the target factors and observing their impact on the removal of specific physicochemical parameters such as chemical oxygen demand (COD), electrical conductivity (EC), total chromium (Cr) and Optical density (OD). The variables that were established include pH, dosage of coagulant and flocculant, as well as the speed and duration of agitation in both the fast and slow mixing stages. The experiments were carried out while taking into account the optimal parameters, leading to the near-complete removal of all analyzed pollutants. The optimal requirements for the *Parkinsonia aculeata-Hibiscus esculentus* Coagulation Flocculation System involve adjusting the pH to 8, choosing concentrations of approximately 1.25 g L^{-1} and 0.6 g L^{-1} for the coagulant and flocculant respectively, maintaining a fast speed of 170 rpm for 3 min while keeping the slow agitation at around 30 rpm for 20 min. The removal rates achieved after treating tannery wastewater using the PA-HE coagulant-flocculant combination demonstrate

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high efficacy, with values reaching approximately 100% for TSS, 98.71% for BOD5, 99.93% for COD, 98.88% for NH_4^+, 98.21% for NO_3^-, 90.32% for NO_2^-, 93.13% for SO_4^{2-}, 95.44% for PO_4^{3-}, 96.08% for OD and 60% for total chromium. These results indicate the successful removal of a wide range of pollutants from tannery wastewater through the PA-HE treatment method. In predicting the CF treatment approach, PCA has been employed to preprocess the input data and determine the key variables that impact the process. This can streamline the modeling process and enhance the precision of the predictions.

1. Introduction

Among the contaminants of greatest environmental concern, heavy metals pose high risks to human health due to their known toxicity, carcinogenicity and mobility [1–4]. The aquatic environment is severely affected by the presence of toxic metal ions [5,6], and chromium is one of the major components which is considered to be the most toxic trace element resulting from industrial applications, such as leather tanning [7,8]. Though the leather industry has a significant economic impact, the effluents from tanneries are considered to be one of the most hazardous pollutants in the manufacturing sector [9]. Indeed, they contain solid waste and are laden with hazardous chemicals such as chromium compounds, synthetic tannins, oils, fats, resins, biocides and detergents. Additionally, they also contribute to the emission of certain noxious gases. Excessive pollution from the leather industry remains a significant factor in the deterioration of water quality, particularly with high levels of BOD5, COD, PO_4^{3-} and sulphates which often do not comply with environmental legislation [10–13]. When considering the different approaches to tannery wastewater treatment, coagulation-flocculation is the ideal choice for industrial processes [14,15]. This is because CFS is a basic process in several disciplines, including biochemistry, cheese making and rubber manufacturing [16–18]. Statistical methods have indeed gained attention in water quality modeling, particularly in the context of coagulation flocculation processes. These methods offer the advantage of saving time and effort while reducing errors, making them valuable tools for analyzing and predicting the behavior of these complex systems [19, 20]. Also, CFS has been successfully used in urban wastewater treatment as well as in other industries to eliminate pollutants, that are colloidal in nature. These pollutants possess a high charge, have a small size of less than 1 μm and exhibit a high surface/volume ratio [21]. Due to its practicality, simple design and low energy consumption, this combined technique is considered the most crucial step in solid-liquid separation which promotes the agglomeration of fine colloids into larger particles [22]. In general, the chemicals extensively used in the conventional coagulation-flocculation process are metal salts like alum salts ($Al_2(SO_4)_3$, $AlCl_3$) [23], iron salts ($FeCl_3$, $FeSO_4$) [24], and organic polymers such as acrylamide, acrylic acid and polyacrylamide [25]. In fact, Synthesized Poly (acrylamide-co-acrylic acid) applied for tannery wastewater treatment. Parameters such as color, turbidity, and total dissolved solids (TDS) were measured to assess the effectiveness of these polymers. The results showed a reduction of approximately 96.34%, 99.42%, and 55.45% respectively [26]. Despite their high efficiency, synthetic coagulants and flocculants often produce a secondary solid pollution known as sludge, which is characterized by its low or non-biodegradability and toxic effects [27]. As a result, this category of chemical water purifiers is accused of having negative impacts on humans [28] and ecosystems [29,30]. Furthermore, over the last few years, numerous researchers have investigated the coagulation-flocculation properties of different plant components and extracts obtained from seeds [31,32], leaves [33], stems [34], fruit shells and kernel waste [35] to purify a variety of drinking water [36] and waste water [37], and removal rates for many pollutants are achieving promising results. For example, the replacement of chemical coagulants like aluminum sulfate with environmentally friendly alternatives such as cassava starch and orange peel powder in the treatment of tannery effluents has shown that the efficiency of COD removal has recently reached 38.51%, 37.25%, and 17.97% respectively [38]. The aim of this study is to use a bio-coagulant based on *Parkinsonia aculeata* seeds and a bio-flocculant prepared from *Hibiscus esculentus* to minimise the pollutant load in the composite tannery discharges [39]. In the process, a preliminary study was conducted to detect the optimal values of various parameters that affect the treatment. The study involved varying the target factors to

Table 1
Measured parameters and equipment used.

Parameters	Experimental Equipement
EC ($mS.cm^{-1}$)	Conductivity meter *WTW 82362 Weilheim.*
TSS ($mg.L^{-1}$)	METTLER TOLEDO AT400 precision balance.
BOD5 ($mg\ O_2.L^{-1}$)	OxitopR IS6 type BOD meter
COD ($mg\ O_2.L^{-1}$)	HACH type COD meter
NH_4^+ ($mg.L^{-1}$)	Spectrometer type *BioSpec.mini*
NO_3^- ($mg.L^{-1}$)	
NO_2^- ($mg.L^{-1}$)	
SO_4^{2-} ($mg.L^{-1}$)	
PO_4^{3-} ($mg.L^{-1}$)	
Do	
Color	Visual observation
Cr ($mg.L^{-1}$)	ICP-AES Horiba spectroscopy Jobin Yvon
Ag ($mg.L^{-1}$)	
Cd ($mg.L^{-1}$)	
Ni ($mg.L^{-1}$)	

examine their interaction in removing certain physicochemical parameters like COD, EC, and OD. The established variables include pH, coagulant and flocculant dosage, as well as agitation speed and time in both the fast and slow mixing phases of the process. Principal Component Analysis (PCA) is utilized to decrease the dimensionality of input data by identifying the most crucial features or variables [40,41]. This can be especially beneficial when predicting complex systems, like the coagulation-flocculation process in wastewater treatment [42,43].

2. Material and methods

2.1. Preparation and characterisation of tannery sludge

The tannery effluent was sampled with an adequate volume from an industrial unit located in the Ain nokbi district of Fez. It was stored at a temperature of 4 °C until it was used. Table 1 displays the parameters that were measured to characterise the effluent before and after treatment.

2.2. Preparation and characterisation of coagulation-flocculation agents

Parkinsonea aculeata (PA) seeds were utilized as a coagulant, while *Hisbicus esculentus* (HE) fruits were employed as a bioflocculant. Prior to usage, both substances were washed to remove any impurities or contaminants, dried in an oven at 60 °C for approximately 48 h, pulverized, and sieved to obtain a fraction with a diameter ranging from 100 to 400 μm [44,45]. To prevent re-wetting or moisture contamination, the powders obtained were stored in an airtight container. The biocoagulants were prepared by dissolving a mass of the seed powder in cold water while stirring for 1 h. The stirring ensures even distribution of coagulants in the solution. After the 1 h stirring period, the suspensions were centrifuged at 4000 rpm for 10 min. Then, they were filtered through a 0.45 μm filter paper to remove any remaining solid particles and obtain a mucilaginous solution that is rich in polysaccharide. This solution is used in the coagulation-flocculation process [46]. The biomasses used in this study were characterized using a BRUKER model Vertex 70 Fourier Transform Infrared Spectroscopy (FTIR) coupled with a digital computer. This setup allowed for the analysis of different types of bonds and chemical groups by tracing the spectra between [4000 and 400 cm^{-1}]. Additionally, a Quanta 200 FEI scanning electron microscope equipped with an X-ray Energy Dispersive Spectrometer (EDS) was used for morphological analysis.

2.3. Coagulation-flocculation experiments

Physico-chemical purification by coagulation-flocculation was conducted in the laboratory using a conventional 6-station jar test apparatus of the VELP SCIENTIFICA type (JLT6). Before starting the process, six beakers were prepared and filled with 200 mL of the composite tannery effluent. Afterward, the pH was measured and adjusted using solutions of HCl (1 M) or NaOH (1 M). The PA-based bio-coagulant was added during the rapid mixing phase to achieve small flocs. Subsequently, the HE-based bio-flocculant was added during the slow agitation phase to ensure the formation of large agglomerates. The optimisation tests are carried out by varying the operating parameters within a specific range (Table 2).

2.4. Statistical analysis

In this study, we employ Principal Component Analysis (PCA), a statistical method, to reduce the dimensionality of the dataset. PCA transforms a large number of variables into a smaller set of variables, known as principal components, while retaining most of the information from the original dataset. We conducted the PCA using IBM SPSS Statistics 20 software. Table 3 provides a summary of the abbreviations for the variables measured in this study and their mining.

3. Results and discussion

3.1. Characterisation of the Parkinsonia aculeata composition

3.1.1. Infrared spectroscopy analysis of Parkinsonia aculeata

The FTIR spectrum of the PA plant is displayed in Fig. 1. The outcomes indicate a wide and strong peak at 3310.20 cm^{-1}, which can

Table 2
Selected parameters for improved coagulation flocculation.

Parameters	Interval
pH	2–10
Coagulant concentration ($g.L^{-1}$)	1,25-7,5
Flocculant concentration ($g.L^{-1}$)	0,2-1,2
Coagulation stirring speed (rpm)	150–250
Coagulation contact time (min)	3–9
Flocculation stirring speed (rpm)	15–35
Flocculation contact time (min)	10–60

Table 3
Abbreviations of the variables used in the PCA analysis and their meanings.

abbreviations	Variables measured	abbreviations	Variables measured
COD_pH	Impact of pH on COD removal efficiency	COD_CCT	Impact of Coagulation Contact Time on COD removal efficiency
OD_pH	Impact of pH on OD removal efficiency	OD_CCT	Impact of Coagulation Contact Time on OD removal efficiency
EC_pH	Impact of pH on EC removal efficiency	EC_CCT	Impact of Coagulation Contact Time on EC removal efficiency
COD_CC	Impact of Coagulant Concentration on COD removal efficiency	COD_FC	Impact of Flocculant Concentration on COD removal efficiency
OD_CC	Impact of Coagulant Concentration on OD removal efficiency	OD_FC	Impact of Flocculant Concentration on OD removal efficiency
EC_CC	Impact of Coagulant Concentration on EC removal efficiency	EC_FC	Impact of Flocculant Concentration on EC removal efficiency
COD_CSS	Impact of Coagulation Stirring speed on COD removal efficiency	COD_FSS	Impact of Flocculation Stirring speed on COD removal efficiency
OD_CSS	Impact of Coagulation Stirring speed on OD removal efficiency	OD_FSS	Impact of Flocculation Stirring speed on OD removal efficiency
EC_CSS	Impact of Coagulation Stirring speed on EC removal efficiency	EC_FSS	Impact of Flocculation Stirring speed on EC removal efficiency
COD_FCT	Impact of Flocculation Contact Time on COD removal efficiency	OD_FCT	Impact of Flocculation Contact Time on OD removal efficiency
EC_FCT	Impact of Flocculation Contact Time on EC removal efficiency		

be linked to OH groups [47]. The two narrow bands at 2926.05 and 2855.06 cm^{-1} indicate the asymmetric stretching of aliphatic C–H bonds [47]. The presence of ester functions, lactones or carboxylated groups is confirmed by the intense peak, recorded around 1743.21 cm^{-1} and associated with C=O stretching [47,48]. The infrared bands located at 1653.79 and 1546.47 cm^{-1} may indicate C=C vibrations of the aromatic ring [47,49], while the peaks observed between 1300 and 1000 cm^{-1} suggest C–O stretching vibration, confirming the presence of alcohols, acids, phenolic derivatives, and ethers [47,50]. Finally, the two peaks at 1401.75 and 618.91 cm^{-1}, which correspond to the oscillation of the CH_2 and CH_3 group, could be associated to neutral lipids, proteins, and carbohydrates [47](Table 4). The results obtained partially correlate with many other works reported in the literature [48–50], and they fully correlate with another study conducted in 2016 that characterised the activated carbon obtained from *Parkinsonia aculeata* wood [47].

3.1.2. *Scanning electron microscopy coupled with EDS of Parkinsonia aculeata*

The SEM analysis of the powder obtained from the dried PA seeds is presented in Fig. 2. The morphology of this material illustrates the presence of irregular structures of various sizes within a heterogeneous and relatively porous matrix. Additionally, the presence of multiple layers creating numerous spaces and forming fine pores is evident. The elemental composition of PA, as analyzed by EDS, is shown in Fig. 2 (e and f). Based on the obtained results, it is clear that this biopolymer has a highly carbonaceous nature. Indeed, Carbon is the primary element, accounting for 52.82% of the total mass, while oxygen is the second most abundant element, with a mass percentage of 45.83%. Additionally, trace amounts of inorganic elements, including K, Al, P, Ca, Mg, and S, are present. The surface micrograph images of PA seeds do not resemble the activated charcoal from the same plant characterized by Comparative research. This may be due to the effect of activation which contributed to the increase in porosity relative to the matrix, as it may be due to the use of a different part of the plant other than the seeds [47]. On the other hand, a study conducted in 2010 reveals a resemblance between the topography of *Moringa oliefera* and *Parkinsonia aculeata*. This similarity can be attributed to the fact that these two plants, both belonging to the Fabaceae family, share a comparable chemical composition [51].

3.2. *Optimisation of coagulation and flocculation*

3.2.1. *Optimisation of coagulation*

3.2.1.1. Effect of pH on treatment performance. Using only PA as a biocoagulant was chosen to assess the impact of different pH levels on the changes in certain parameters, such as COD, OD, and EC, in the combined tannery effluent. The findings are displayed in Fig. 3.

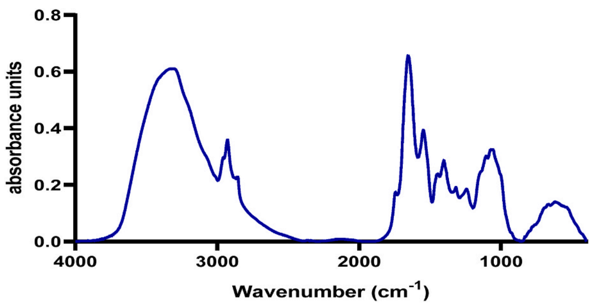

Fig. 1. Infrared spectrum of *Parkinsonisa aculeata* powder.

Table 4
Infrared bands of *Parkinsonia aculeata* (PA) and their attributions.

Position (cm^{-1})	Attributions	References
3310	Elongation vibration of the OH group	[39]
2926	C–H asymmetric vibration	[39]
2855	Asymmetric vibration C–H	[39]
1743	C═O ester elongation vibration/free carboxylic bond vibration	[39,40]
1653	Deformation of water	[39,41]
1546	C═C stretching vibration	[39,41]
1401	CH_2 deformation vibration	[39]
1317	C–H crystalline deformation of amorphous cellulose	[39,42]
1241	C–*O*–C pyranose vibration of hemicellulose	[39,42]
1057	Vibration of the C–O bond	[39,42]
618	Deformation of the O–H group	[39]

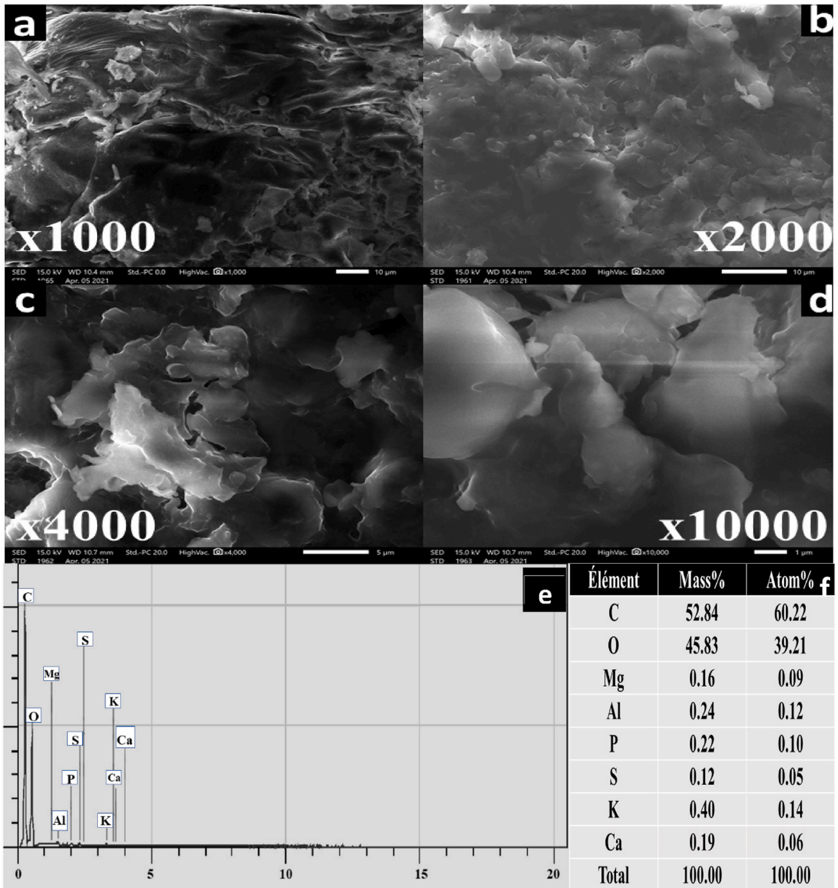

Fig. 2. Parkinsonia aculeata SEM photograph with different magnification powers (a 1000x, b 2000x, c 4000x, d 10000x), and elemental composition analyzed by EDS (e, f).

As observed, the polymer extracted from PA seeds exhibits the lowest COD values at pH 2, 8, and 10, with respective values of 3313.75, 4056.18, and 4068.30 mg $O_2.L^{-1}$. The highest COD value is obtained at pH 2, followed by pH 8. The COD RRs for PA are 79.34, 73.79, 69.9, 74.72 and 74.64% for pH 2, 4, 6, 8 and 10 respectively. Performance optimisation of cassava starch in tannery wastewater treatment show a maximum removal efficiency above 37.5% occurring at an optimum pH range within 3.17 and 9 [38]. In another study, the pH kept constant at 7 allowed Cassava peel and alum to remove 90% of the turbidity [52]. Upon individual analysis of OD removal, it was observed that the values obtained are significantly lower compared to the initial value of 24.7. This demonstrates the effectiveness of PA as a bio-coagulant. It is also noted that these values vary depending on the pH level. The values obtained for pH 2, 4, 6, 8, and 10 are 1.57, 1.56, 2, 1.43, and 1.41 respectively. Notably, pH levels adjusted to 8 and 10 resulted in the highest OD elimination, with a maximum RR of 94.25% and 94.21% respectively. Conversely, the lowest RR of 91.87% was observed at pH 6. Color reduction was found to be reduced up to 96.34% at pH (8.5) in a research using Alum as coagulant in consortium with polymers for

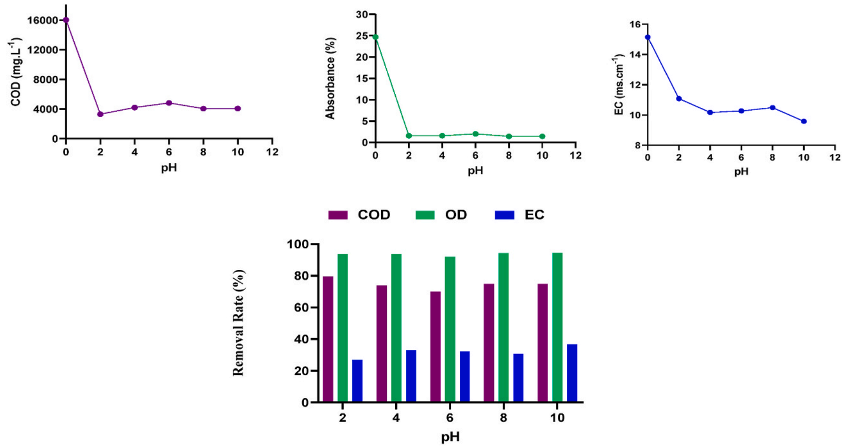

Fig. 3. Effect of several pH values on the variation of COD, absorbance and electrical conductivity. **Operating conditions:** treated volume = 200 mL, (pH = 2–10), [PA] = 3.75 g L^{-1}, coagulation speed = 150 rpm, coagulation time = 5 min, [HE] = 0.8 g L^{-1}, flocculation speed = 25 rpm, flocculation time = 20 min.

treating tannery wastewater [26]. The removal of the electrical charge also depends on the initial pH of the coagulation. Based on the obtained results, a low elimination is observed at the highly acidic pH of 2 with a minimum RR of 26.81, within the pH range of 4–8. The EC concentration increases from 10.19 to 10.49 mS cm^{-1} as the pH increases. Additionally, at a highly basic pH of 10, the removal of this parameter is very appreciable with a maximum RR of 36.72%. Generally, the removal of the three parameters is very different according to the pH used. The removal of coloration and EC exhibits promising results at pH = 10, while the ideal pH for COD removal is 2. However, this pH value is excessively acidic and can lead to further pollution by acidifying the treated effluent. Furthermore, a noticeable orange coloration appeared after one week of settling the treated effluent at pH = 2. This may be due to the oxidation of trivalent Chromium to hexavalent Chromium, which is extremely toxic and carcinogenic, Therefore, in our research, we selected pH 8 as it showed a significant RR of OD, EC and especially COD. The excellent performance of PA at acidic pH can be explained by the abundant presence of H^+ ions in the medium. These ions help neutralize the negative charge of the colloids and promote coagulation through Van der Waals forces. At pH = 10 the excess of OH^- ions likely leads to competition between the adsorption sites and the pollutants in the composite tannery effluent. Turbidity removal from tannery wastewater using graphene oxide-ferric oxide nanocomposites as an adsorbent was reduced up to 95% at pH 6 [53]. Therefore, it can be inferred that coagulation occurs through the inter-particle bridging mechanism. Bibliographic studies indicate that when natural polymers are used as coagulants, different coagulation mechanisms may be expected [54,55]. Research on coagulation has demonstrated that the optimal pH for pollutant removal varies depending on the type of particle studied, implying that it can be acidic, basic, or neutral [54]. Furthermore, other studies have indicated that the initial pH significantly influences particle surface charge and coagulation behavior [56,57].

3.2.1.2. Effect of Parkinsonia aculeata concentration on treatment performance. The removal efficiency of COD, staining and electrical conductivity under different dosages of PA extracted biocoagulant are shown on Fig. 4. Based on the analysis of the COD variation

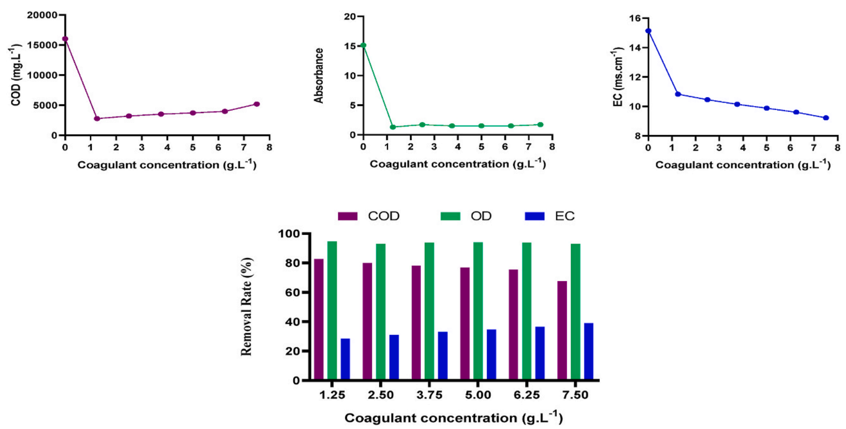

Fig. 4. Effect of diferent concentrations of PA on the variation of COD, absorbance and electrical conductivity. **Operating conditions:** treated volume = 200 mL pH = 8, ([PA] = 1.25–7.5 g L^{-1}), coagulation speed = 150 rpm, coagulation time = 5 min, [HE] = 0.8 g L^{-1}, flocculation speed = 25 rpm, flocculation time = 20 min.

results, it was found that increasing the concentration of biocoagulant leads to a significant increase in the COD of the composite tanning effluent. The respective values for the concentrations of 1.25, 2.5, 3.75, 5, 6.25, and 7.5 g L^{-1} are 3213.75, 3516.78, 3731.93, 3971.33, and 5195.57 mg $O_2.L^{-1}$. Therefore, the optimal dose that gave a maximum RR of 82% was 1.25 g L^{-1}. The decrease in COD concentration of the tannery wastewater is probably due to the fact that the PA polymer contains coagulant proteins and polysaccharides. The charged ions provided by these molecules adsorb and neutralize the counter charged colloids and promote their agglomeration. In contrast, increasing the PA concentration above 1.25 g L^{-1} most probably causes competition between the excess relegated ions and colloidal particles with opposite charge. The PA coloration removal performance did not vary significantly when the PA dose was increased from 1.25 to 7.5 g L^{-1}, and the RR generally showed values above 90%, with a maximum RR of 94.73 for the 1.25 g L^{-1} concentration and a minimum RR of 93.92 for the 5 g L^{-1} concentration. However, the effect of varying the concentration of PA on the removal of electrical conductivity shows an inverse effect compared to the removal of COD, in other words, increasing the concentration of biocoagulant from 1.25 to 7.5 g. L^{-1} gradually reduced the concentration of mineral load from 15.14 to 9.23 mS cm^{-1}, and the RR obtained are 30.91, 32.95, 34.74, 36.52, 39.03% for the concentrations of 1.25, 2.5, 3.75, 5, 6.25, 7.5 g L^{-1}, respectively. The conductivity removal rates show low values; this is apparently due to the fact that the electric phase of the effluent is dominated by ions that have the same charge as colloids, which is the cause of repulsive forces. In the same context, increasing the concentration of PA means increasing the total number of counter ions; this increases the chance of oppositely charged molecules to agglomerate with the polymer used, after neutralisation. The results of the study concerning Cassia Fistula seed gum as a biocoagulant for the treatment of wastewater from fish canneries are partially similar to the results found in our research, regarding COD removal. Researchers reported that the COD removal efficiency gradually decreases when the concentration of Cassia Fistula gum increases. These results also show that the PA polymer used in our study has a high efficiency compared to Cassia Fistula gum and the maximum COD RRs are 82 and 59.41%, respectively [58]. The investigation reveals that the removal of certain parameters increases as the concentration of cactus juice coagulant increases. These findings are consistent with the results of electrical conductivity removal [59]. Other studies also support these results, as they found that cactus juice significantly reduced the coloration of vegetable oil refinery wastewater. The maximum rate of discoloration was approximately 95%, which closely aligns with our research findings of 94.73% [60].

3.2.1.3. Effect of coagulation stirring speed on purification performance. A set of tests was carried out with an optimum pH of 8, an optimum PA dosage of 1.25 g L^{-1} to examine the effect of agitation speed on the removal efficiency of COD, OD and EC (Fig. 5). The results of these tests over a range of agitation speeds that vary between 150 and 250 rpm show a slight increase in the COD value with each increase in agitation speed, and the values found are 2541.03, 2598.60, 2607.69, 2713.75 mg $O_2.L^{-1}$ for speeds of 150, 170, 200, 250 rpm respectively. The findings also indicate that the highest percentage of COD removal was achieved at 150 rpm, with a value of 84.16%. This speed facilitated the effective dispersion of the coagulant, promoting the aggregation of colloids and the formation of large coagulates. However, exceeding the optimal agitation speed resulted in a decrease in the removal rates. This is likely due to the fragmentation of the large coagulates into smaller fragments that are difficult to settle. In another study, wastewater from the Nam Phong swine slaughterhouse was treated. The results indicated that both Alum and PAC achieved a removal rate of over 90% for color, turbidity, and COD, using a stirring speed of 75 (RPM) [61]. The OD removed does not vary greatly with increasing coagulation speed, and the obtained values generally fluctuate between 2.09 and 2.19. Similarly, the RRs exhibit relatively high values, but they are still too close to each other. Moreover, the percentages found are 91.09, 91.59, 91.5, 91.31% for speeds of 150, 170, 200, 250 rpm. The results expressed as percentages of elimination for minimizing electrical conductivity, show very low RRs of less than 30%, and the speed of 200 rpm is the one that allowed for maximum EC elimination. Indeed, this parameter decreased from 15.14 in the raw effluent to 10.67 in the treated effluent. It can be assumed that the inability of the PA to reduce the overall rate of electrical conductivity is due to the increase in electrical charge in the medium after the release of the bio-coagulant ions. It can be assumed that increasing the

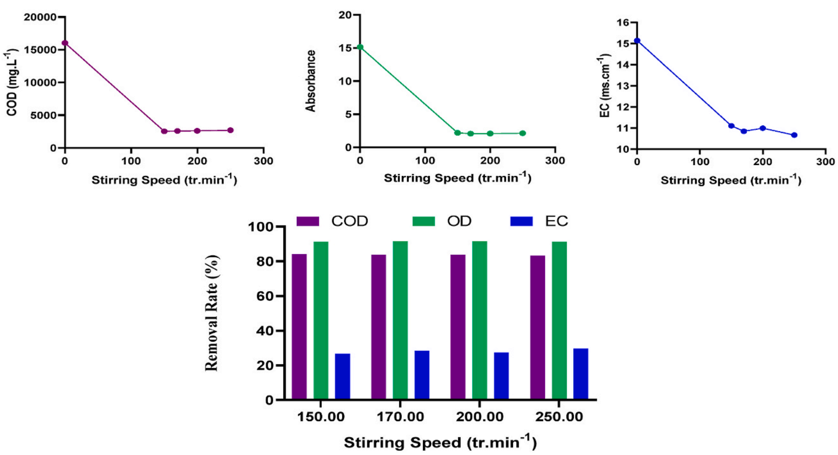

Fig. 5. Effect of different coagulation stirring speeds on the variation of COD, absorbance and electrical conductivity. **Operating conditions:** treated volume = 200 mL, pH = 8, [PA] = 1.25 g L^{-1}, (coagulation speed = 150–250 rpm), coagulation time = 5 min, [HE] = 0.8 g L^{-1}, flocculation speed = 25 rpm, flocculation time = 20 min.

stirring speed also affects the release of PA ions, which minimises the overall charge. According to a study published in 2017, a high mixing speed causes polymer chain breakage, limiting the size of the formed coagulates [62]. In contrast, a study carried out in 2020 found no difference in the effect of stirring speed on COD removal through the adsorption process [63]. Anteneh et al. reported in 2014 that mixing speeds between 50 and 120 rpm progressively reduce COD and color by up to 58–72% and 63–81%, respectively. The best performance for reducing these two parameters, with a removal rate (RR) of 83% and 90%, is achieved at a speed of 150 rpm. However, the authors observed that further increasing the stirring speed to 200 rpm decreases the performance in COD and color reduction [64]. These findings are in perfect conformity with the current research.

3.2.1.4. Effect of coagulation stirring time on purification performance. Fig. 6 shows the effect of coagulation stirring time on the removal of COD, colour and EC using a PA dosage of 1.25 g L^{-1}. For this purpose, coagulation stirring times of 3, 5, 7 and 9 min were chosen. As can be seen from the curves in Fig. 6, the coagulation time affects the removal of COD and EC in the same way. Indeed, both parameters increase proportionally with increasing the duration of coagulation and the maximum RRs are 83.74 and 28.07% respectively. These values were achieved after a minimum of 3 min of stirring. Conversely, after stirring for 9 min, both COD and EC showed a decrease in percentages of 82.89% and 27.08% respectively. However, there was only a slight improvement in terms of color values, and the removal rates remained around 89%. These results are consistent with other literature studies, which have also shown that the highest removal efficiency of 96.7% is obtained after 90 s of rapid stirring, Increasing the stirring time to 120 s results in a removal efficiency of 96% [65]. Other researchers have conducted studies on the treatment of textile effluent using the coagulation-flocculation process. They have examined the impact of different stirring times (1, 2, 3, 4, and 5 min) on the removal of COD and color. It was observed that a stirring time of 1 min was sufficient to achieve a higher removal rate, reaching 60.9% and 82.1% [66]. A study in 2018 found that a stirring time of 3 min was optimal for coagulation using various coagulants, and the efficiency of coagulation slightly decreased with longer stirring times [67].

3.2.2. Optimisation of flocculation

3.2.2.1. Effect of flocculant concentration on tannery effluent treatment. Fig. 7 illustrates the primary dose range of HE for eliminating certain pollutants from the tannery effluent while maintaining a constant pH of 8. The observations in the figure indicate that the removal of COD increases as the concentration of HE rises within the range of 0.2–0.60.2-0.6 g L^{-1}, reaching an optimal value of 2753.15 mg O_2.L^{-1} for a dose of 0.6 g L^{-1}. The COD removal efficiency decreased beyond this range, and the RRs in terms of COD are 80.70, 81.35, 82.84, 81.61, 80.87 and 51.77% for the concentrations of 0.2, 0.4, 0.6, 0.8, 1 and 1.2 g L^{-1} respectively. The concentration of HE did not influence the efficiency of colour removal dramatically. The values found for HE concentrations of 0.2, 0.4, 0.6, 0.8, 1 and 1.2 g L^{-1} are 0.473, 0.47, 0.575, 0.5, 0.494 and 0.542 respectively, with a maximum RR of 98.08 % for the minimum concentration of 0.2 g L^{-1}. Unlike COD abatement, the EC removal increases as the HE concentration increased from 0.2 to 1.2 g L^{-1}, and the maximum RRs were obtained in the dose range of 1–1.2 g L^{-1} with percentages of 31.77 and 31.17 %. These findings align with a previous study, which suggested that the impact of flocculant dose on heavy metal removal can be categorised into 3 phases:

- A phase indicating low removal using a minimum amount of flocculant.
- A second phase corresponds to an increase of the dosage within a reasonable range, allowing for the acceleration of metallic trace elements removal.
- A final phase is recognised by the excessive dosage of the flocculant, causing a disorder of the reaction system and limiting the removal action [68].

The effectiveness of HE mucilage and powder as a bioflocculant in effluent treatment has been reported in several studies. Research

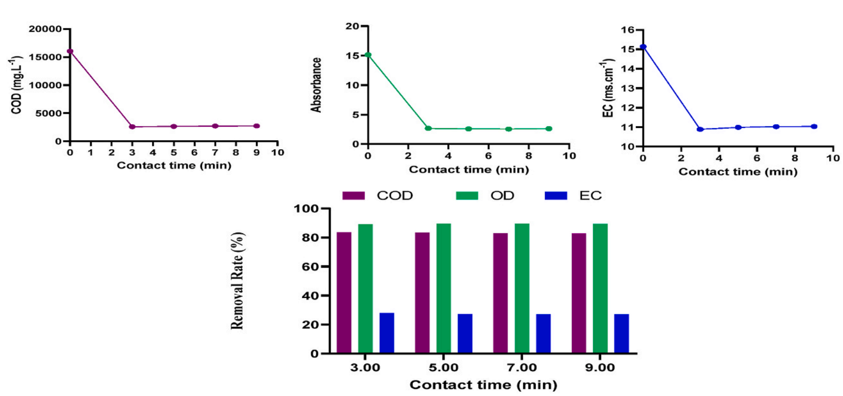

Fig. 6. Effect of different coagulation stirring periods on the variation of COD, absorbance and electrical conductivity. **Operating conditions:** treated volume = 200 mL, pH = 8, [PA] = 1.25 g L^{-1}, coagulation speed = 150 rpm, (coagulation time = 3–9 min), [HE] = 0.8 g L^{-1}, flocculation speed = 25 rpm, flocculation time = 20 min.

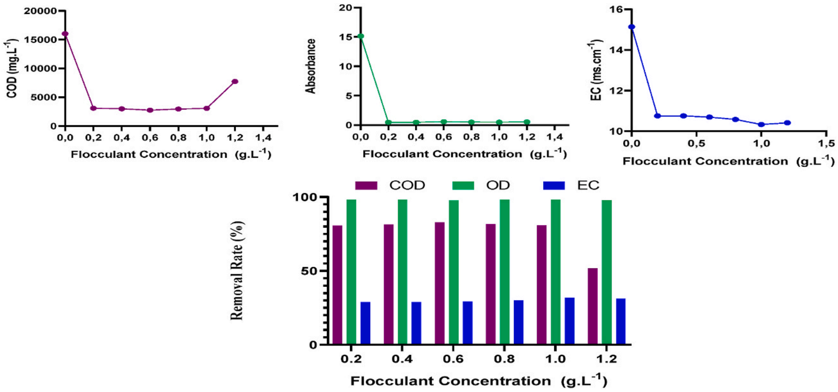

Fig. 7. Effect of different concentrations of (HE) on the variation of COD, absorbance and electrical conductivity. **Operating conditions**: treated volume = 200 mL, pH = 8, [PA] = 1.25 g L^{-1}, coagulation speed = 150 rpm, coagulation time = 3 min, ([HE] = 0.2–1.2 g L^{-1}), flocculation speed = 25 rpm, flocculation time = 20 min.

in 2019 noted that HE powder significantly reduces turbidity, with greater reductions observed at higher concentrations of the bio-flocculant. Furthermore, an extremely high dosage of flocculant increases the electrostatic repulsion in the reaction system by augmenting the excessive negative charge present in the macromolecular chain [69].

3.2.2.2. Effect of flocculation speed on tannery effluent treatment. The results of COD, colour and electrical conductivity removal from the tanning wastewater as a function of flocculation speed are presented in Fig. 8. The findings indicate that the COD concentration decreases progressively with the increase of the flocculation speed in the range of 10–30 rpm. The values obtained are 3334.96, 2780.42, 2531.93 and 1683.45 mg O_2.L^{-1} for the speeds of 15, 20, 25 and 30 rpm. However, an increase in the COD can be observed when the agitation speed is increased to 35 rpm. The RRs represents a maximum value of 97.51% at a stirring speed of 20 rpm. However, above this speed, the percentage of OD removal tends to vary more slowly and reaches stable values, ranging between 96.26 and 96.27%. The RRs for electrical conductivity are 31.10, 29.92, 30.58, 30.71 and 30.77% respectively for stirring speeds of 15, 20, 25, 30, and 35 rpm. No significant difference was observed between the values obtained; nevertheless, the speed of 15 showed the highest efficiency for EC abatement. These results are in agreement with the published literature, which also shows that the agitation speed in the flocculation phase should be neither too low nor too high, to prevent sedimentation and flake breakage [70]. In the study carried out by Kazi et al. on "Tannery wastewater treatment using natural coagulants", the stirring speed for flocculation is the same as the optimal speed for COD removal in our research, which is 30 rpm [71].

3.2.2.3. Effect of flocculation time on tannery effluent treatment. The results concerning the effect of slow mixing time (10, 20, 30, 40 and 50 min) on the removal of COD, EC and colour are shown in Fig. 9. From the above findings, it is evident that the gradual increase in flocculation time has a slight impact on the removal efficiency of these three parameters. Indeed, the optimum removal of COD was obtained after 20 min of slow agitation, resulting in a maximum RR of 88.89%. This value stabilised for timings of 30, 40 and 50 min to reach a minimum percentage of 86.41% after 1 h of flocculation. As can be seen from the curves. The OD values for the times of 10, 20, 30, 40, 50, and 60 min are 0.841, 0.937, 1.079, 1.012, 0.859, and 0.965 respectively. The conductivity values for the same times are 13.6, 13.76, 13.81, 13.79, 13.64, and 13.77. As a result, it can be concluded that the time required for a more effective removal of these

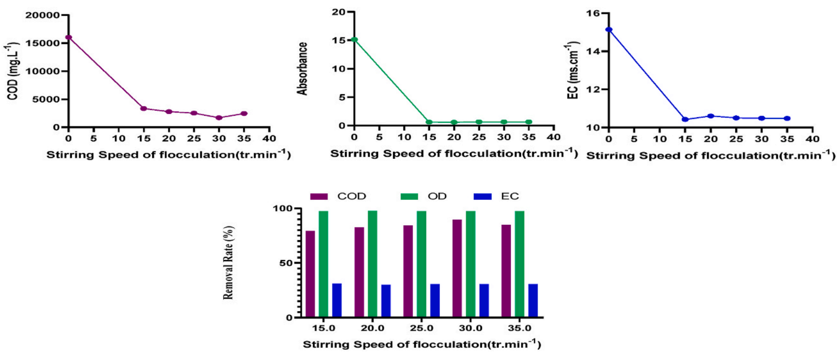

Fig. 8. Effect of different flocculation stirring speeds on the variation of COD, absorbance and electrical conductivity. **Operating conditions:** treated volume = 200 mL, pH = 8, [PA] = 1.25 g L^{-1}, coagulation speed = 150 rpm, coagulation time = 3 min, [HE] = 0.6 g L^{-1}, (flocculation speed = 15–35 rpm), flocculation time = 20 min.

two factors is only 10 min, and the maximum RRs are 96.81 and 20.46%. In contrast to the coagulation phase, which does not require much time, the gathered results indicate that extending the agitation duration is essential during flocculation to guarantee the creation of denser and more durable flocs. This, in turn, facilitates their decantation during the settling phase. A recent study found that turbidity and total suspended solids were removed by approximately 98% within 15 min of coagulation [72]. Another study, which examined the use of sprayed okra pod in paint wastewater treatment, determined that 30 min was the optimal time for the flocculation process [73]. Additionally, a study that investigated the impact of physical variables on flocculation efficiency found that a slow stirring time of 10 min was sufficient, as there was no significant difference observed beyond this duration [74].

3.3. Optimised coagulation-flocculation system for tannery effluent treatment

Physicochemical characterization results in Table 5 demonstrate the high efficiency of the CF system used for TEM purification. The calculated (RR) after TEM treatment by PA-HE are as follows: TSS - 100%, BOD5 - 98.71%, COD - 99.93%, NH_4^+- 98.88%, NO_3^-- 98.21%, NO_2^- - 90.32%, SO_4^{2-} - 93.13%, PO_4^{3-} - 95.44%, Cr - 60 %, and OD - 96.08%. It is worth noting that the TEM effluent at the system inlet has a complex composition with high levels of organic and mineral matter. These values obtained exceed the Moroccan standards, but they decrease significantly after treatment, leading to an improvement in effluent quality. The results obtained in the current study surpass the findings of a study conducted on the optimisation of tannery wastewater treatment conditions using chitosan as a coagulant. The reductions achieved in COD, suspended matter, and chloride concentrations are significant, at 68.90%, 95.24%, and 39.72% respectively [75]. In another study, the focus was on optimizing the coagulation-flocculation treatment of artisanal tannery wastewater. The results show that at an optimal pH of 6.09 and an aluminum sulfate dosage of 11.60 g/L, significant removal efficiencies were achieved, with a maximum COD removal of 38.51%, TSS removal of 76.05%, and turbidity removal of 79.64% [76]. The study on optimizing coagulation parameters for tannery effluents found that a combination of aluminium sulfate and ferric chloride is effective in clarifying tannery wastewater by reducing 86% for colour removal. However, the reduction of TSS in the treated effluent is not significant enough, with a RR of only 38–46%. Applying centrifugation after coagulation improves TSS removal by an additional 15% [77]. This issue represents one of the challenges at the treatment level for the mentioned system.

3.4. Statistical treatment of the data using PCA

The results of the PCA analysis are visualized in Fig. 10, which likely illustrates how various parameters of CFS affect the removal rates of COD, OD, and EC in the tannery wastewater treatment. PCA can assist in identifying the chemical parameters or combinations most strongly linked to coagulation flocculation properties, offering valuable insights for future research or practical applications [42, 43]. Outcomes from PCA show that Principal Component 1 (PC1) captures 44.87% of the total variance and is primarily influenced by (OD_CCT, COD_pH, OD_CSS, EC_FCT, OD_FCT, EC_CCT, EC_FSS, COD_CCT, and OD_pH), while Principal Component 2 (PC2) captures 19.13% with a strong dominance of (EC_CC, COD_CC, EC_FC, COD_FSS, OD_FSS, COD_CSS, COD_CCT, COD_FC, EC_CSS, and EC_CCT). This indicates that these two factors together represent 64% of the original dataset's variability. Furthermore, it implies a loss of about 36% of the information, referring to the unaccounted portion of the original data's variability. The findings suggest that the data can be divided into four distinct regions based on similarity. Furthermore, the statement indicates that dissimilarities are observed with respect to PC1 and PC2. This implies that the variables within group A (COD_CCT, EC_CCT, EC_FCT, COD_pH, and OD_FCT) and the variables within group B (OD_FSS, COD_CSS, OD_CC, and OD_FC) are positively correlated within themselves and with each other, and this correlation pattern is reflected in the two principal components. Similarly, variables within groups C (OD_CSS and OD_CCT) and D (EC_pH, EC_CSS, and COD_FSS) are also correlated within their respective groups but exhibit a negative score with respect to the two components 1 and 2. Certain variables show differences when analyzed in relation to the first and second factors. COD-CC and COD-FC are positively correlated with PC2 and negatively correlated with PC1. These variables move in opposite directions when PC1 and PC2 change. Conversely, EC_FSS and OD_pH are positively correlated with PC1 and negatively correlated with PC2, indicating a similar opposite relationship. EC_FC and COD_FCT have a modest relationship with PC1 and PC2, respectively. In contrast, EC_CC is negatively correlated with both two principal components, suggesting an inverse relationship between them.

4. Conclusion

In this paper, we have demonstrated the potential of using sustainable biomass for the physico-chemical treatment of effluent from a modern tannery. The physicochemical characterization of the coagulating and flocculating agents, namely PA and HE, using XRD, FTIR, and SEM-EDS, revealed the high carbon content of these biomasses. Furthermore, the optimisation results emphasized the importance of selecting the appropriate operating parameters to achieve optimal outcomes and ensure the proper functioning of the treatment process. Additionally, conducting the experiments with the optimal parameters resulted in the almost complete elimination of all analyzed pollutants, with the exception of EC and COD, which still complied with the Moroccan legislative standards after treatment.

Data availability

The data used to support the findings of this study are available from the corresponding author upon request.

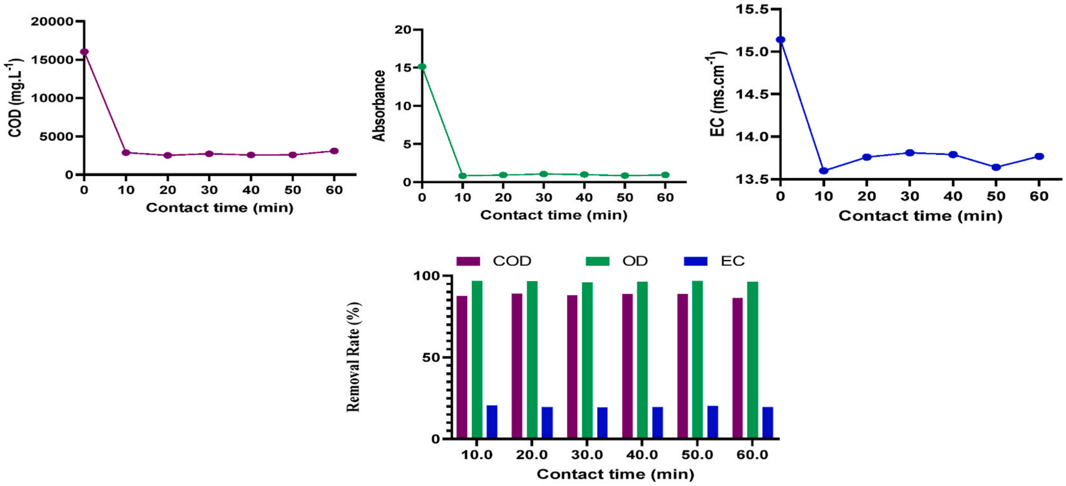

Fig. 9. Effect of different flocculation stirring periods on the variation of COD, absorbance and electrical conductivity. **Operating conditions:** treated volume = 200 mL, pH = 8, [PA] = 1.25 g L^{-1}, coagulation speed = 170 rpm, coagulation time = 3 min, [HE] = 0.6 g L^{-1}, flocculation speed = 30 rpm, (flocculation time = 10–60 min).

Table 5
Physico-chemical characterisation of the tannery effluent after treatment.

Parameters	Raw effluent	PA treated effluent	RR %
CE ($mS.cm^{-1}$)	24.15	20.61	14.65
TSS ($mg.L^{-1}$)	6020.4	0	100
BOD5 (mg $O_2.L^{-1}$)	3345.75	43	98.71
COD (mg $O_2.L^{-1}$)	37613.52	2546.6	93.22
NH_4^+ ($mg.L^{-1}$)	114.8	1.28	98.88
NO_3^- ($mg.L^{-1}$)	68.4	1.22	98.21
NO_2^- ($mg.L^{-1}$)	11.5	1113	90.32
SO_4^{2-} ($mg.L^{-1}$)	6761.6	464.16	93.13
PO_4^{3-} ($mg.L^{-1}$)	8.72	0.397	95.44
OD	23.73	0.93	96.08
Cr	20	8	60
Ag	<0.01	0	100
Cd	<0.01	0	100
Ni	<0.01	0	100

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CRediT authorship contribution statement

Ghita El Mouhri: Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Methodology, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Ibtissame Elmansouri:** Methodology, Formal analysis, Conceptualization. **Halima Amakdouf:** Visualization, Methodology, Formal analysis. **Hajar Belhassan:** Methodology, Formal analysis. **Rabie Kachkoul:** Validation, Software, Formal analysis. **Fatima Ezzahra El oumari:** Visualization. **Mohammed Merzouki:** Visualization, Validation, Supervision. **Anissa Lahrichi:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Methodology, Formal analysis, Conceptualization.

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.

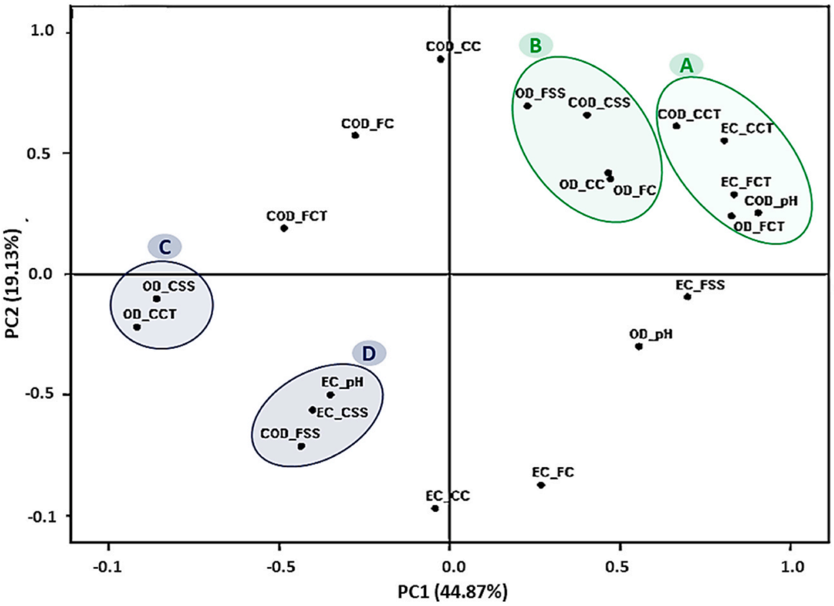

Fig. 10. Principal Component Analysis (PCA) plot for the input variable used for PA-HE treatment efficiency prediction.

Nomenclatures

BOD5	Biochemical Oxygen Demand
CF	Coagulation-Flocculation
CFS	Coagulation-Flocculation System
COD	Chemical Oxygen Demand
Cr	Total chromium
EC	Electrical Conductivity
FTIR	Fourier-Transform Infrared Spectroscopy
HE	*Hibiscus esculentus*
NH_4^+	Ammonium
NO_3^-:	Nitrate
NO_2^-:	Nitrite
OD	Optical Density (Absorbance)
PA	*Parkinsonia aculeata*
PA-HE	*Parkinsonia Aculeata-Hibiscus Esculentus*
PCA	Principal Component Analysis
PO_4^{3-}:	Phosphate
rpm	rotation per minute
RR	Removal Rate
SEM-EDS	Scanning Electron Microscopy-Energy Dispersive X-ray Spectroscopy
SO_4^{2-}:	Sulfate
TEM	Mixed Effluent from the Tannery
TSS	Total Suspended Solids
XRD	X-ray diffraction

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