

Efficiency of a coagulation–flocculation process using *Opuntia ficus-indica* for the treatment of a textile effluent

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ABSTRACT

Coagulation–flocculation is one of the more commonly used techniques for treating industrial effluents. The present work provides the results obtained in the treatment of textile effluent by applying the coagulation–flocculation process with a new reagent derived from cactus *Opuntia ficus-indica* largely found in Morocco. The objective herein is two-fold: to test the efficiency of this technique and establish the action mechanism of this new biodegradable and natural coagulant. The use of this new cactus-based product has shown a very significant effect on turbidity removal (96%), as well as on sludge production reduction (3.3 mL L⁻¹), compared to other chemical coagulants, such as ferric chloride and aluminum sulfate. Settling time however is relatively long (5 h). To assess the mechanism involved in this coagulation–flocculation, we proceeded with global characterization by infrared (IR) spectroscopy and zeta potential measurements on the cactus. These measurements indicated that the colloids associated with the cactus material are negatively charged (at pH = 10.0). The IR spectroscopy analyses revealed in this new material the presence of aromatic groups, such as phenols and aromatic proteins, thus suggesting that Moroccan cactus *Opuntia ficus-indica* rely on a different mechanism than a chemical reagent like metallic salts. The cactus effect likely occurs through a flocculation reaction of a large concentration of metallic hydroxides by tannin molecules of cactus material combined with a “sweep coagulation” mechanism.

Keywords: Coagulation–flocculation; Bioflocculant; *Opuntia ficus-indica*; Zeta potential; Textile industrial effluent

1. Introduction

The toxicity of textile effluents has been the topic of several studies [1–3] and has revealed the need to treat or optimize the treatment of wastewater before discharge into the environment. The coagulation–flocculation process has exhibited great efficiency in eliminating pollution during the treatment of textile effluents [4–7]. Previous studies have demonstrated that the optimization and adjustment of the

effluent physicochemical parameters or the coagulant treatment rate can lead to flocculation and efficient pollutant removal [6–8]. This process may be directly applied to the effluent in order to remove organic materials with colloids or supracolloids without effective reduction of the effluent toxicity [9]. Colloids or supracolloids particles can aggregate and settle out of solution through four basic mechanisms: (i) double-layer compression, (ii) sweep flocculation, (iii) adsorption and charge neutralization and (iv) adsorption and interparticle bridging [10]. The presence of salts can cause

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compression of the double layer, resulting in destabilization of particles whereby repulsive electrostatic interactions are overcome by attractive van der Waals forces (i). Sweep flocculation or enmeshment in the precipitate occurs when precipitating coagulant traps suspended particles within a colloidal floc as it forms or settles (ii) [10]. Destabilization of particles through charge neutralization can occur when suspended particles in solution sorb to oppositely charged ions (iii). Bridging can occur when a coagulant forms a polymer chain that can attach to multiple particles so that particles are bound to the coagulant and need not contact one other (iv) [11,12].

Over the last several years, many studies at the laboratory scale have been conducted to evaluate the efficiency of several coagulants in water treatment, sometimes using pilot unit experiments [13]. The most commonly used in wastewater treatment are the trivalent salts of iron ($\text{FeCl}_3 \cdot \text{Fe}_2(\text{SO}_4)_3$) and aluminum ($\text{Al}_2(\text{SO}_4)_3$) [14,15]. When applying coagulation–flocculation treatment, however, a large amount of sludge may sometimes be generated. This factor must be taken into consideration when choosing the coagulant [16,17]. Furthermore, to minimize the toxic effects of chemical coagulants and respect new stringent regulations, non-toxic and biodegradable coagulants and flocculants have been introduced [18,19], such as chitosan [20], tannin or seed extract from a tree (*Moringa oleifera*) that grows throughout the tropics and subtropics [21]. In this context and to enhance the use of natural substances, we have developed a cactus-based extract as a flocculant. Several studies have shown that cactus possess flocculent properties suitable for water treatment [11,12,22,23]. The reaction mechanisms however are still not well explained nor, have the active molecule's support in the flocculation reaction has not been fully identified. The objectives of this study include examining the efficiency of the flocculation–coagulation process for the treatment of textile effluent, particularly in terms of metal pollution, turbidity, sludge production and color removal. Material characterization by means of infrared (IR) spectroscopy and zeta potential measurements has been conducted to better understand its operating mechanism. Moreover, a comparative study of the cactus with other chemical coagulants is performed. The ultimate goal of this work is to demonstrate the efficiency and the mechanism of action of a new bioflocculant.

2. Materials and methods

2.1. Material characteristics

The textile effluent samples were extracted at the MultiWash Company (Fez, Morocco). The treatment process was applied to the textile effluent, whose characteristics are given in Table 1.

The cactus was harvested at a wild plantation near Fez in Morocco, geographic coordinate: latitude: 33.979/longitude: -4.863. The cactus pads were washed, and all thorns were removed and dried at 80°C. The material was ground and sieved to obtain a solid powder with a diameter of 0.5–1.00 mm. The powder obtained was then used as a reagent for treating the textile effluent.

Lime ($\text{Ca}(\text{OH})_2$) (>95% Sigma-Aldrich, France), ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) (>99.99% Sigma-Aldrich, France) and

Table 1
Textile effluent characteristics

Characteristics	Values
pH	6.45
Turbidity (NTU)	214
COD (mg L^{-1})	1,266
Color	Blue
Conductivity (mS cm^{-1})	2.13

aluminum sulfate ($\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$) (>97% Fluka, France) of commercial-grade were utilized for the experimental procedure. Lime was prepared in the form of a slurry ($10\text{--}20 \text{ g L}^{-1}$), ferric chloride and aluminum sulfate as a solution ($20\text{--}40 \text{ g L}^{-1}$) using the distilled water in all three cases.

2.2. Jar test experience

Coagulation–flocculation tests were performed using a flocculator (i.e., Jar test). The testing equipment is composed of four stirred reactors (Flocculator Fisher 1198) with a rotation speed of between 0 and 200 rpm. Reactors contain 1 L of textile residual effluent, the mass of cactus extract (from 5.0 to 55.0 mg). Impact of pH (from 4.0 to 12.0) was conducted and adjustment was done by using 1.0 M HCl or 1.0 M NaOH. The volume of bases or acid is negligible (V_{max} added is 10 mL) compared with the volume of effluent (1 L). Different concentrations of the selected coagulant were added to the effluent, using the optimal pH of the coagulant. The mixture was then quickly stirred at 200 rpm for 10 min. Thereafter, the speed was reduced to 30 rpm for 30 min. The last step, using an Erlenmeyer of (1 L) to determine the volume of sludge after a settlement stage of 5 h was carried on.

The coagulation–flocculation process efficiency for treating the effluent was specifically evaluated in terms of turbidity, sludge production, metal contamination and color removal. Sludge generation was assessed according to the settling time of the treated effluent.

After flocculation, turbidity was monitored during the settling stage until reaching a pseudo-balance between the liquid and solid phases. Both settling time and sludge volume were measured once the turbidity in the supernatant became constant.

2.3. Analytical techniques

For all treated effluent samples, we conducted the following physicochemical analyses: pH, turbidity, and absorbance in the range of 200–800 nm spectrum. All these parameters were determined using standard methods for examining wastewater. The turbidity of the wastewater samples was measured using a Hanna Turbidity Meter (HI88713). UV-Vis analyses were performed on a UV-Visible spectrophotometer (UV 2300), and metal elements in both the raw and treated textile effluent were analyzed by inductively coupled plasma atomic emission spectroscopy (ICP-AES ULTIMA 2 instrument (Jobin Yvon, Longjumeau Cedex, France)) after mineralization in aqua regia [24].

Data acquisition and processing were performed using the ICP JY v. 5.2 software (Jobin Yvon). The daily calibration of the monochromator was performed by using the carbon emission lines and each operating wavelength was individually centered before the experiment beginning. The wavelengths used and the appropriate detector voltages, set by analyzing a 100 mg L⁻¹ multielement standard solution [25]. The zeta potential was measured by means of a Malvern Master Zetasizer 3000 device (France).

Spectral analysis Fourier transform infrared spectroscopy was measured by an infrared spectrometer affinity in the region of 400–4,000 cm⁻¹. Samples were prepared under high pressure between (12–15 tons pressure) with the cactus and KBr powder (KBr > 99% Acros Organics).

3. Results and discussion

3.1. Coagulation–flocculation efficiency

3.1.1. Turbidity removing

This study of treating textile effluent by coagulation–flocculation using a cactus-based product is illustrated in Fig. 1. The turbidity of effluent is 214 NTU (Table 1). After coagulation–flocculation at pH 10.0, the turbidity varies from 68 to 4 NTU for an amount of cactus between 10 to 55 mg L⁻¹. The lowest turbidity value was obtained with a dose of 35 mg L⁻¹ which matches with the lowest value of the volume of sludge (3.5 mL L⁻¹) (Fig. 1a). Fig. 1b shows that the lowest turbidity for 25 and 50 mg L⁻¹ of cactus is pH 10.0. The pH increases all the more as the pH is adjusted from 10.

In a previous work [11], we have obtained similar results with experiments realized at pH 10 in a synthetic effluent with 33 mg L⁻¹ of cactus *Opuntia ficus-indica* and removal efficiency of 96% ± 2. The volume of sludge produced after 5 h of settling was 3.3 mL L⁻¹.

3.1.2. Organic matter and metal removing

Beyond turbidity, and in order to see if organic pollutants from textile residual effluent are also removed. UV-Visible detection is conducted before and after

coagulation–flocculation. Table 2 shows the absorbance in the UV and visible range of the effluent, in the presence of various coagulant cactus doses. Tests were carried out at the optimal pH of 10.0. The raw effluent spectrum shows two broad bands located at wavelengths 290 nm and 675 nm.

The absorbance at 290 nm may be due to conjugated molecules such as proteins, nucleic acid or humic acid [26,27], at 675 nm may be due to the presence of the reactive blue 15 dye [28]. These absorbance bands decrease with the amount of coagulant (from 0 to 33 mg L⁻¹ of cactus). They disappear in the presence of the optimal cactus concentration (33 mg L⁻¹) with more than 90% and 97% of absorbance removals at 290 and 675 nm respectively.

The results of metals analysis in both raw effluent and effluent treated with cactus flocculants are displayed in Table 3. Several metallic elements were present in the effluent: Fe being the most prevalent, followed by Zn, Mn, Cr and, to a lesser extent, Cu and Ni, effluent treatment by coagulation–flocculation found that the process is very suitable for removing copper, chromium and zinc. All solubility products of these metallic hydroxides are very low, the theoretical solubility is less than 10⁻⁸ mole at pH 10 except for Mn with a theoretical residual concentration close to 1 mg/L. For this metal, sorption or coprecipitation mechanisms could explain the reduction of concentration by the coagulation–flocculation treatment. The metal reduction percentage actually exceeded 91%. Let's note that effluent treatment at a basic pH of 10 increases the metal hydroxide deposits while the colloids produced can facilitate the coagulation mechanism.

Table 2
UV and visible absorbance of effluent without and with coagulant

Coagulant dose (mg L ⁻¹)	Absorbance at 290 nm	Absorbance at 675 nm
0	1.75	0.67
25	0.63	0.02
33	0.16	0.02

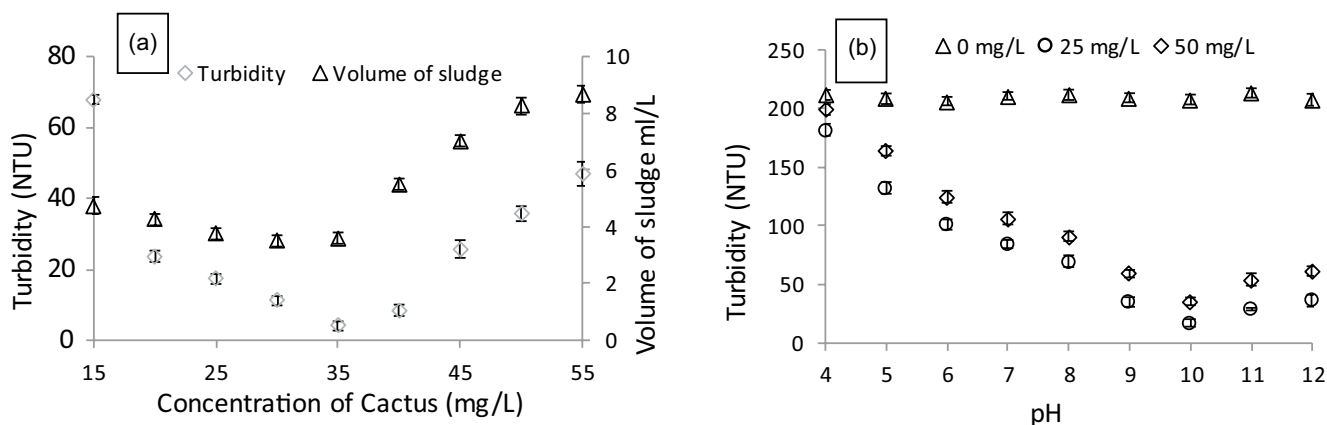


Fig. 1. (a) Evolution of turbidity and sludge volume depending on the dose of cactus (from 15 to 55 mg L⁻¹) at pH 10 and (b) Evolution of turbidity depending on the pH for different doses of cactus (0, 25 and 50 mg L⁻¹).

3.2. Mechanism

To clarify the cactus action mechanism, we proceeded with an infrared spectrophotometric characterization. The results obtained by IR spectroscopy using the KBr method indicated the presence of several peaks (Fig. 2). We observed a peak between 3,200 and 3,500 cm^{-1} , which reveals the existence of carboxylic acids. On the other hand, the vibration bands at 1,620 cm^{-1} indicate the presence of C=O groups, while the vibration at 1,430 cm^{-1} is characteristic of

phenol groups [29]. Lastly, the peaks at 1,320 and 1,050 cm^{-1} prove the presence of aromatic proteins and polysaccharides respectively [30].

Fig. 3a shows the variation of zeta potential vs. pH in the case of a cactus solution 30 mg L^{-1} : as pH increases, the zeta potential becomes more highly negative (3.31 mV at pH = 3.0. moving to -21.8 mV at pH = 12.0). The pH corresponding to the isoelectric point equals 4.1; Fig. 3b provides the variation in zeta potential vs. cactus dose (mg L^{-1}) in the case of textile effluent treatment. The increase in dose

Table 3
Concentrations of metallic elements in the raw effluent and after treatment

Metals (mg L^{-1})	Fe	Cr	Zn	Mn	Ni	Cu
Raw effluent	2.61	0.094	0.36	0.15	0.03	0.09
pKsp (25°C)	39	31	17	13	16	20
Treated effluent	0.32	0.008	0.03	0.06	0.02	0.009
Limits of detection ($\mu\text{g L}^{-1}$)	0.41 ± 0.01	0.08 ± 0.04	0.09 ± 0.01	0.32 ± 0.02	0.81 ± 0.01	0.26 ± 0.01

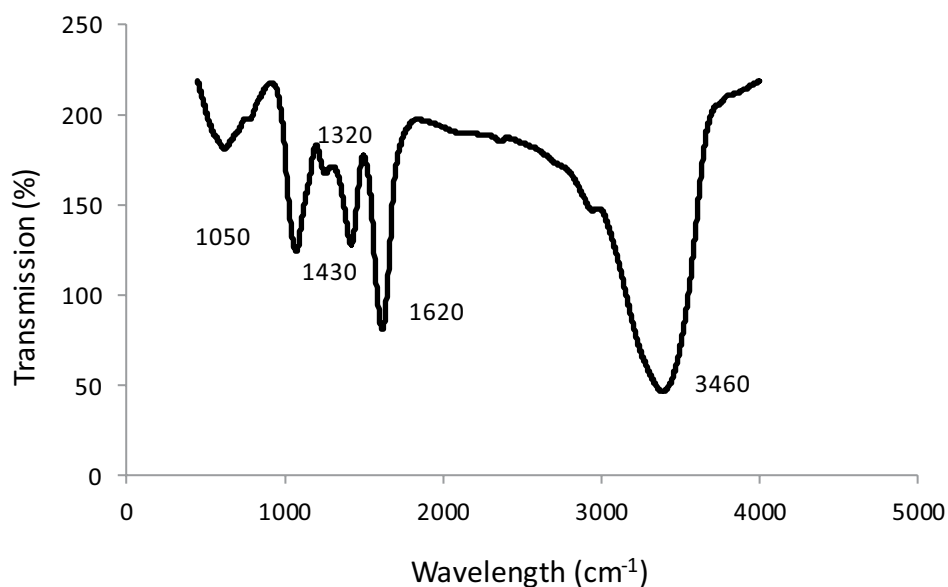


Fig. 2. Infrared spectrum of cactus material.

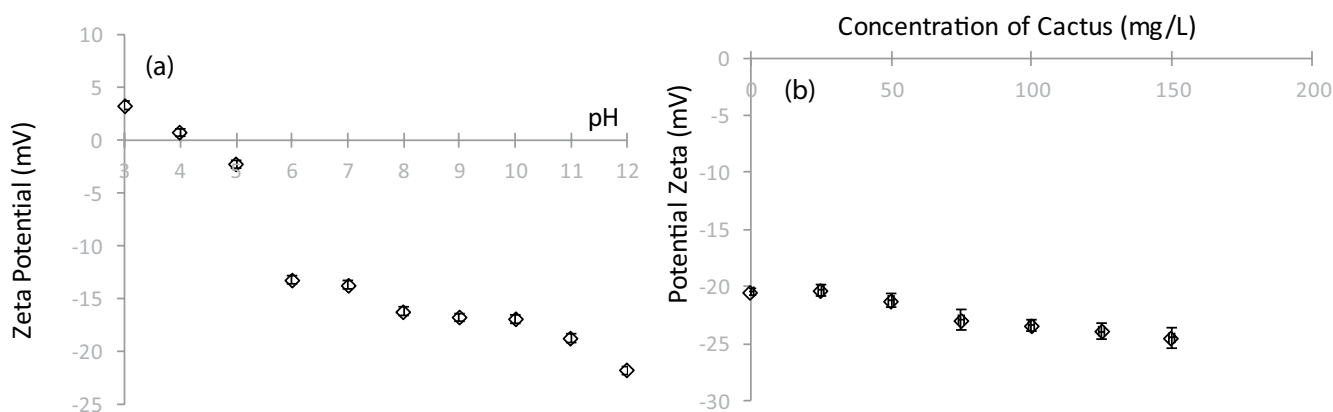


Fig. 3. Variation of zeta potential as a function of (a) pH and (b) dose cactus material.

causes a low decrease of zeta potential. The zeta potential measurements showed that the colloids associated with the cactus-based product were negatively charged (at pH = 10.0) and for all pH values the synthetic water solution with clay presents negative colloids [22].

As observed with the metallic quantification, all metals precipitate at pH 10 with a very high colloidal concentration and the appearance of a mechanism of sweep coagulation. Anion exchange was proposed for an explanation of the coagulation reaction between the tannin of cactus

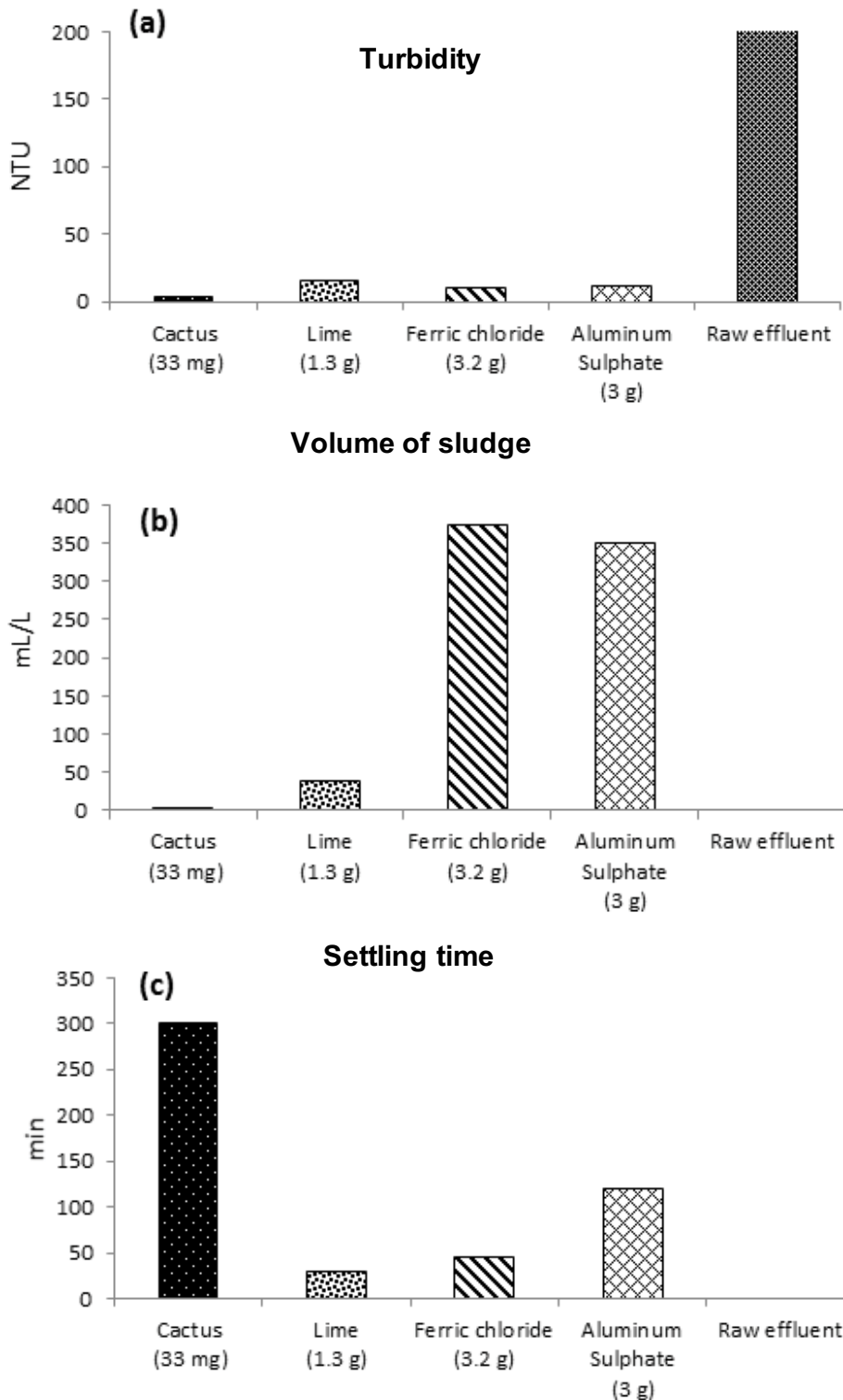


Fig. 4. Evolution of the turbidity (a), the volume of sludge (b), and the settling time (c) according to the type of coagulants.

material and the numerous colloids (metallic hydroxides and clay) [22].

However, it is possible that naturally existing ions present in the cactus itself may provide the ionic strength necessary for compression of the double layer and therefore coagulation. However, theoretical analysis of cactus *O. ficus-indica* inorganic cation content and experimental analysis from conductivity measurements [31] of water treated with cactus *O. ficus-indica* indicate that the ionic strength provided by cactus *O. ficus-indica* itself is not high enough to cause coagulation as a result of double-layer compression.

The zeta potential measurements showed that the colloids associated with the cactus-based product were negatively charged (at pH = 10.0). Infrared spectroscopy exhibited the presence of aromatic groups, such as phenols and aromatic proteins. This analysis suggested that the plant material operates through a mechanism different from that of chemical coagulants. Cactus action probably occurs through a flocculation mechanism with adsorption and bridging between particles along the lines of a “sweep coagulation” mechanism. Furthermore, the cactus *O. ficus-indica* with a negatively charged backbone (carboxyl and hydroxyl groups from phenol) at pH 10.0 allowed the polymer molecules to be extended into solution and produce loops and tails to promote bridging of flocs, as other bio-flocculants, such as tannin and anionic cellulose [32].

3.3. Comparison of cactus with metal salts flocculants

Fig. 4 shows the evolution in parameters (turbidity, sludge volume production and settling time) of the treated effluent depending on coagulant type. The cactus was compared with three coagulants, including aluminum sulfate ($\text{Al}_2(\text{SO}_4)_3$), ferric chloride (FeCl_3) and lime ($\text{Ca}(\text{OH})_2$).

The optimal dose obtained by aluminum sulfate has been estimated at 3 g L^{-1} with an efficiency of turbidity removal of 98%. The sludge production after 2 h of settling was 360 mL L^{-1} . The lime allows a turbidity elimination of 93% for an optimal dose of 1.3 g L^{-1} . It seems to be the best suitable for low production of decanted sludge, just after 30 min for settling. The ferric chloride allows turbidity elimination and produced the same quantity of decanted sludge such as aluminum sulfate (380 mL L^{-1}). The optimal dose obtained has been estimated at 3.2 g L^{-1} . These results demonstrate that the relation between the volume of sludge produced and the amount of coagulant introduced is globally the same for both the ferric chloride and aluminum sulfate coagulants and it is higher compared with the one from the cactus extract. As regards the lime coagulant, the sludge volume decreases to 40 mL L^{-1} for lime and less with cactus material (3.3 mL L^{-1}) but the effect on turbidity is less than FeCl_3 and $\text{Al}_2(\text{SO}_4)_3$. The use of the vegetal material has eliminated turbidity to a similar extent as the ferric chloride and aluminum sulfate 96%. The main difference between the two classes of coagulant (chemical and natural) is more distinct in the difference of both the amount of added coagulant 100 times higher for chemical coagulant than for cactus and sludge production. Indeed, we observed that in the case of cactus, the optimal dose and sludge volume are very small compared to those of the

other chemical coagulants. The settling velocity, however, is much faster in the case of lime, followed by ferric chloride, aluminum sulfate and lastly the cactus. Accordingly, other bio-flocculants, such as tannin and sodium alginate, have a very slow settling time [32], which is probably due to a slow micro aggregation of the flocs.

4. Conclusion

Flocculation–coagulation is an unavoidable process for reducing the turbidity of wastewater. The cactus *O. ficus-indica* was tested as a coagulant for textile industrial effluents. For test conditions (200 rpm for 10 min and then 30 rpm for 30 min), the method is optimum for a cactus dose of 33 mg L^{-1} at pH 10.0 removal is 98% for turbidity, >90% of UV-Visible absorbance (dissolved organic matter and dye), 91% for metals (Fe, Zn, Mn, Cr, Cu, Ni). Compared to the metal salts ($\text{Ca}(\text{OH})_2$, $\text{Al}_2(\text{SO}_4)_3$, FeCl_3) widely used in coagulation–flocculation (i) the coagulant dose is 60–90 times lower and (ii) the volume of sludge after settling is 50–400 times lower.

Zetametric analyses have suggested that the cactus material operates through a mechanism different from that of metal salt coagulants, with its action likely occurring through a flocculation mechanism with adsorption and bridging between particles along the lines of a “sweep coagulation” mechanism. Ionized groups amine, carboxyl, phenol detected by IF spectroscopy could be involved in those mechanisms.

In addition, the use of cactus *O. ficus-indica* as a bio-flocculant is an economic solution and providing an environmental-friendly approach for the sustainable development of bio-based renewable energy in the future.

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