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Cactus juice as bioflocculant in the coagulation–flocculation process for industrial wastewater treatment: a comparative study with polyacrylamide

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ABSTRACT

Most industries in the world treat their wastewaters with a conventional coagulation–flocculation process using alum as coagulant, polyacrylamide (PAM) as flocculant and lime as coagulant aid. To reduce the use of chemical products in the process, experiments were conducted to substitute the PAM with cactus juice (CJ) as flocculant. From the obtained data, it was concluded that the substitution of PAM with CJ in the coagulation–flocculation process was very effective, compared with PAM. Depending on the wastewater's origin, the bioflocculant showed removal efficiencies of 83.3–88.7% for suspended solids (SS) and 59.1–69.1% for chemical oxygen demand (COD). Lime addition enhanced the coagulation–flocculation process in the presence of CJ similarly to the PAM with efficiencies greater than 90% for both SS and COD. The CJ powder's infrared (IR) spectrum showed the main functional groups present in PAM. It was concluded that CJ as a flocculant fits well with the definition of sustainability and it is appropriate for countries that have regions where cactuses grow naturally.

Key words | bioflocculant, cactus, coagulation–flocculation, industrial wastewater treatment

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INTRODUCTION

Various treatment methods are currently used to treat industrial wastewaters. The choice of the process depends on the wastewater characteristics and on treatment objectives. The use of a chemical treatment process is one of the most common and well-known methods. Among the chemical treatment methods, the most familiar practices include chemical precipitation, coagulation–flocculation and chemical oxidation. For economic considerations, the conventional coagulation–flocculation process (using various chemicals such as salts of iron, aluminum, lime, and polyelectrolytes, etc.) are used worldwide, showing higher treatment performance in terms of organic matter, solid matter and heavy metal removals (Abdel-Shafy *et al.* 1987; Watanabe *et al.* 1993; Szpak *et al.* 1996; Tatsi *et al.* 2003; Ginos *et al.* 2006). The coagulation–flocculation process offers various advantages for the treatment of industrial wastewaters such as lower sensitivity to toxic loadings and higher amounts of organics, easy operation, energy saving, etc. However, it has been demonstrated in numerous studies that chemical products used as coagulants or flocculants (such as aluminium salts, acrylamides, etc.) remain in water after treatment and may induce health problems. For example, it was reported that alum, the most widely used coagulant, may be related to Alzheimer's disease (Martyn *et al.* 1989; Letterman & Pero 1990). Furthermore, monomers of some synthetic organic polymers such as acrylamide may have neurotoxic and carcinogenic properties (Mallevialle *et al.* 1984). Human health risks exist with the use of synthetic flocculants in the treatment of water (Aizawa *et al.* 1990; Letterman & Pero 1990). The neurotoxicity of acrylamides is well documented and other health effects are reported for various organisms such as genotoxic, cancerogenic, reproduction and developmental effects (Dearfield *et al.* 1988). Synthetic polymers, and the undesirable substances that are associated with them, may react with others added for treatment and create by-products with unknown health effects (Ozacar & Sengil 2003). It is recommended to consider the toxic properties of any synthetic polymer considered before adding to water (Aguilar *et al.* 2005). Hence, there is a need, from a human health

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point of view, to consider alternative flocculants in the coagulation–flocculation process.

Therefore, the disposal of the large amounts of sludge generated by the coagulation–flocculation process represents an increasing environmental problem associated with the presence of harmful chemicals (Haarhoff & Cleasby 1988; Ndabigengesere *et al.* 1995; Aguilar *et al.* 2005; Haydar & Aziz 2009; Pritchard *et al.* 2010). Because of the hazards associated with the use of synthetic polymers, a number of natural materials of biological origin have been evaluated for their coagulation–flocculation properties. These natural materials should be available, cost effective, safe for human health and biodegradable. They are derived from seeds, leaves, bark, sap, roots and fruits of plants (bean, cactus, Moringa, maize, etc.) (Ndabigengesere *et al.* 1995; Diaz *et al.* 1999; Zhang *et al.* 2006; Pritchard *et al.* 2009; Antov *et al.* 2010).

In Tunisia, the industries located in the Sfax region treat their wastewater using the conventional coagulation–flocculation process using alum as coagulant, polyacrylamide (PAM) as flocculant and lime as coagulant aid. In order to reduce the chemical products in the process, experiments were conducted to substitute the PAM with cactus juice (CJ) as flocculant. In this regard, a few studies have reported the use of cactus for the turbidity reduction of drinking water and for heavy metal removal from an aqueous solution (Mane *et al.* 2011; Pichler *et al.* 2012). However, to the best of our knowledge, no investigations have examined the feasibility of using this bio-product as flocculant in the conventional flocculation-coagulation process for industrial wastewater treatment.

Since cactus is abundant in Tunisia and does not attract much commercial interest due to lack of knowledge about its alimentary properties, it seems worthwhile investigating the use of this natural product in wastewater treatment. Hence, the objective of this study was to evaluate the efficiency of using CJ to substitute for PAM as flocculant for the treatment of wastewaters from the food and glue industries. The process's removal performance was evaluated mainly through monitoring suspended solids (SS) and chemical oxygen demand (COD).

MATERIALS AND METHODS

Cactuses sampling and characterisation

Cactuses (belonging to the genus Opuntia), were sampled from the Sfax region (Tunisia). Samples were ground with a grinder, filtered using a gauze compress and then supplemented with distilled water (10%). This operation produced a liquid called CJ, which was stored at 4 °C until used. CJ was subjected to chemical characterisation in accordance with AOAC (1995) methods. CJ pH was measured with a pH-meter (model 420A, Orion, USA). Dry weight was quantified by drying the CJ at 105 °C, lipids were determined by Soxhlet extraction, nitrogen was quantified by the Kjeldhal procedure and ash content by incineration in a muffle furnace (model 1400, Thermolyne, USA) at 550 °C. Proteins were calculated using a rate of 6.25% nitrogen to protein (AOAC 1995). Carbohydrates were calculated by difference using the formula 100% − (water + proteins + lipids + ash). The structure of CJ powder (CJP) (obtained by drying CJ at 105 °C) was also investigated using Fourier transform infrared (FTIR). An FTIR spectrum was obtained by a Nicolet Nexus FTIR spectrometer (Thermo Electron Corporation, USA) using the KBr method. The CJP spectrum was comparable with that for PAM.

Industrial wastewater sampling and characterisation

Wastewater samples were collected from two different industries: a confectionery manufacturer (WWFI) and a glue manufacturer (WWGI) located in the Sfax region, Tunisia. In order to minimise the changes in their characteristics, the wastewater samples were stored at 4°C until use. The pH was measured with a pH meter (Orion 420A) as mentioned above. SS, total Kjeldahl nitrogen (TKN) and COD were determined according to *Standard Methods* (APHA 1992). Absorbance values were measured using a spectrophotometer (UVmini-1240 Shimadzu, Japan) at the maximum absorption bands of the samples (310 and 308 nm, respectively, for WWFI and WWGI). The colour of the wastewater samples was notified based on visual observation.

Coagulation–flocculation assays

Non-ionic PAM (PAM: Shandong Sanfeng Group Co., Ltd, China), the commonly used polyelectrolyte in the industry, and CJ were tested as flocculants. They were added to the beakers at the flocculation stage in the presence of alum (Suvchem, Mobay, India) used as the coagulant at optimum doses. Coagulation–flocculation experiments were conducted using the jar-test procedure (Stuart SW6, UK, equipped with six beakers of 1L volume). The jar-test procedure consisted of rapid stirring for a period of 3 min at 150 rpm followed by 17 min slow stirring at 60 rpm. After a settling period of 30 min, the COD, SS and pH were determined in supernatant samples (APHA 1992). Once the optimum flocculant (PAM or CJ) doses were determined, the effect of lime (a local product) as coagulant aid was tested using the same jar-test procedure.

RESULTS AND DISCUSSION

Wastewater characterisation

The characteristics of the two types of wastewater used in the study are presented in Table 1. Samples from the two industries are different from each other with respect to both physical and chemical characteristics. WWFI showed a yellowish colour whereas WWGI was whitish. Maximum absorbance values were, respectively, 1.65 and 3.5 for WWFI and WWGI, suggesting that most of the organic pollutant load of these two wastewaters absorb light at the UV range. The composition is strongly influenced by the nature of the industry. Samples showed pH values of 4.9 and 6.7, respectively for WWFI and WWGI. The SS were nearly the same (270 and 290 mg/L, respectively for WWFI and WWGI). The COD of WWGI was much higher (99,200 mg/L) than WWFI (2,376 mg/L), representing nearly a 42-fold ratio. However, the WWFI had a higher TKN (140 mg/L) than WWGI (28 mg/L), a 5-fold ratio. This indicates that the differences in the wastewater composition are likely to affect the coagulation–flocculation process.

Coagulation–flocculation results

Determination of the optimum dosage for flocculants

With alum, optimum doses of 4 and 5 g/L were used for WWFI and WWGI, respectively, as determined in a preliminary study (data not shown); the jar-test experiments were performed using CJ as flocculant with doses varying between 0.056 and 0.729 g/L (g dry weight of CJ). Similar experiments were conducted with the PAM at doses varying between 0.004 and 0.244 g/L. In the presence of the bioflocculant for both wastewater samples, the pH decreased at the beginning and remained almost stable with the increase of the bioflocculant doses (Figure 1(a)). This falling pH behaviour is similar to PAM for WWGI, but less for CJ for WWFI. This confirms the demonstrated fact that polyelectrolytes have little or no effect on the pH and generally do not require pH adjustment for effective use (Qian *et al.* 2004).

Table 1 | WWFI and WWGI characteristics

Parameters	WWFI[a]	WWGI[b]	Discharge standards[c]
pH	4.94	6.70	6.5-9
SS (mg/l)	230	270	400
COD (mg /l)	2,376	99,200	1,000
TKN (mg/l)	140	28	100
Absorbance[d]	1.65	3.5	
Colour (visual)	Yellowish	Whitish	–

[a]WWFI: wastewater from food industry.
[b]WWGI: wastewater from glue industry.
[c]Tunisian wastewater standard discharge in sewer system (INNORPI 1989).
[d]Absorbance was determined at 308 and 310 nm, respectively, for WWFI and WWGI.

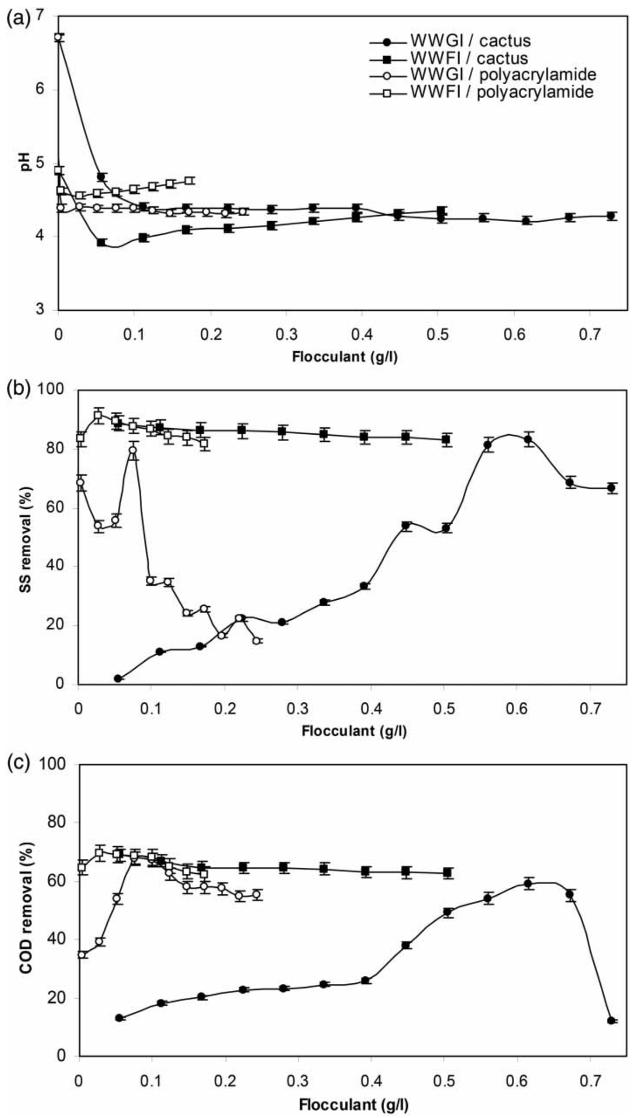

Figure 1 | Coagulation–flocculation performance using CJ and PAM as flocculant: (a) pH variation; (b) SS removal; (c) COD removal.

Optimum dosages and treatment performances (SS and COD removals) were achieved for each wastewater sample (Figures 1(b) and 1(c)). Generally, results depict that the overall removal efficiencies of the SS and COD increased with an increase of both CJ and PAM dosages until reaching an optimal value, depending on the wastewater type. In the case of WWFI, the optimum dose for cactus was obtained at the lowest dosage tested (0.056 g/L), giving removal efficiencies of 88.7 and 69.1%, respectively, for SS and COD. However, for WWGI, the removal of SS and COD increased slowly, reaching the optimum at 0.616 g/L, with efficiencies of 83.3% (for SS) and 59.1% (for COD). Above the optimum dosages, COD and SS removals decreased differently depending on the wastewater. The decline may be the result of solids re-suspension at these concentrations (>0.056 and >0.616 g/L, respectively, for WWFI and WWGI). The high concentrations of the bioflocculant might confer positive charges on the particle surface (a positive zeta potential) allowing redispersion of the particles as reported for polyelectrolytes (Kemmer 1988). Experiments performed with PAM (Figures 1(b) and 1(c)) showed removal efficiencies of 91.3% and 69.6%, respectively, for SS and COD in the case of WWFI obtained at an optimum dose of 0.028 g/L. However, in the case of WWGI, the obtained PAM optimum of 0.076 g/L allowed 79.6 and 67.5%, respectively, for SS and COD removal.

Table 2 summarises the performance of both CJ and PAM as flocculants. Interestingly, CJ performances appear similar or slightly lower in most assays to those obtained using PAM, except in the case of WWGI, which showed SS removal (83.3%) slightly superior to that obtained with PAM (79.6%). However, the optimum dosages of CJ for WWFI (0.056 g/L) and WWGI (0.616 g/L) were high compared to those obtained for PAM (0.028 and 0.076 g/L, respectively, for WWFI and WWGI). This observation may be due to the fact that the flocculation process was related to the original concentration of the SS of the wastewater, and to the addition of a suitable amount of flocculant, which resulted in a high degree of flocculation. This fact is supported by the finding of Barany & Szepesszentgyorgyi (2004). In this respect, a 93% COD removal was obtained by Elleuch et al. (2014) using 1 g/L of praestol and 180 mg/L of $Al_2(SO_4)_3$ $18H_2O$ as flocculant and coagulant, respectively, during the treatment of oil-bottle washing water. For pasta industry effluent, the use of 40 mg/L of PAM allowed an elimination of 77.78% of COD (Khannous et al. 2011). Generally, the application of coagulation and flocculation processes can lead to a significant reduction of SS and COD for wastewater from many industries such as pulp and paper, food processing, textiles, pharmaceutical, petrochemical, etc. (Lee et al. 2012). However, the utilisation of polyelectrolyte depends on what kind of effluent is being treated; for instance, a polyelectrolyte used for the treatment of food processing wastewater might not work properly on treating other industrial effluents, since the physicochemical characteristics of effluents such as pH, colour and the predominant type of pollutants affect the polyelectrolyte action, and consequently the necessary dose (Goloba et al. 2005; Zhu et al. 2011). Although CJ optimum dosages were higher (two and eight times higher than PAM for WWFI and WWGI, respectively) and might allow the production of greater amounts of sludge, its use remains attractive because it is safe for human health and biodegradable. Moreover, it may lead to a beneficial economical gain by reducing the import of high-cost chemicals (Altaher & Alghamdi 2011; Saritha 2012).

Table 2 | Optimum dosages and efficiencies of flocculants (CJ and PAM) and lime used as coagulant aid for WWFI and WWGI

	CJ		PAM	
	WWFI[a]	WWGI[b]	WWFI	WWGI
Flocculants				
Optimum dosage (g/L)	0.056	0.616	0.028	0.076
SS removal (%)	88.7	83.3	91.3	79.6
COD removal (%)	69.1	59.1	69.6	67.5
pH at the optimum dosage	3.92	4.21	4.56	4.39
Lime + flocculants at optimum doses				
Lime optimum dosage (g/L)	7.12	7.12	5.70	6.41
SS removal (%)	92.2	90.3	93.5	90.7
COD removal (%)	95.6	82.1	95.9	86.3
pH at the optimum dosage	11.60	8.90	10.93	8.15

[a]WWFI: wastewater from food industry.
[b]WWGI: wastewater from glue industry.

Determination of the optimum dosage of lime

Coagulant aids may improve the coagulation–flocculation process, taking into consideration that its use should not greatly increase water treatment costs. The selection of the coagulant aid should also take into consideration other factors such as preparation, accessibility and available legislation related to its use. In wastewater treatment processes, the lime dosage should be controlled and lowered in order to reduce the produced amounts of sludge and problems associated with its disposal. In Tunisia, lime is widely used in industrial wastewater treatment as coagulant aid.

In this part of the study, experiments were carried out with lime doses ranging from 1.425–10.68 g/L. Generally, lime addition made the sample more alkaline (Figure 2(a)). For samples conducted with CJ at a lime dose of 7.12 g/L, the wastewater pH reached 11.6 (for WWFI) and 8.9 (for WWGI). Therefore, the pH should be neutralised before discharging the wastewater in the sewer system. In the case of WWFI, SS and COD removals reached 92.2% and 95.6%, respectively (Table 2). However, for WWGI, the removal effectiveness was slightly lower with values of 90.3% and 82.1%, respectively for SS and COD (Table 2, Figures 2(b) and 2(c)). Furthermore, when the lime dose was increased (>to 7.12 g/L), the removals for both SS and COD were not improved and decreased in slightly different manners, depending on the wastewater samples (WWFI or WWGI). This indicated an optimum dose for lime of 7.12 g/L for both, with CJ as flocculant. The combined use of lime and CJ leads to more efficiency of SS and COD removals similar to that obtained in the presence of PAM. However, with PAM, the lime doses were slightly lower with values of 5.7 and 6.41 g/L, respectively, for WWFI and WWGI (Table 2). Interestingly, after the coagulation–flocculation process, both effluents were visually colourless Similar to the above-mentioned results, other studies (Ginos *et al.* 2006; Mehta 2012) have revealed that the use of lime as coagulant aid in the flocculation–coagulation process for industrial wastewater can lead to significant reductions of SS, turbidity and COD. However, optimum lime dose and removal rates varied depending on the coagulant/flocculant materials and the effluent (Ginos *et al.* 2006; Mehta 2012). For example, Georgiou *et al.* (2003) have applied lime (at 800 mg/L) and ferrous sulfate (at 1,000 mg/L) for the treatment of cotton textile wastewater, and observed 95 and 70% of colour and COD removal, respectively.

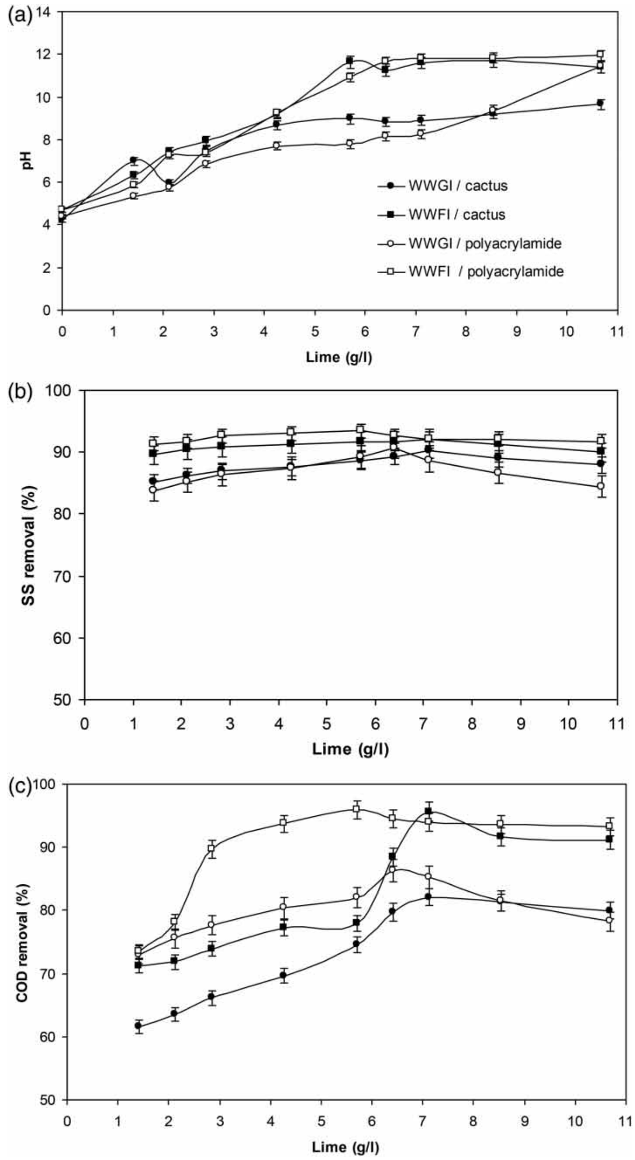

Figure 2 | The effect of lime on coagulation–flocculation performance in the presence of optimum doses of CJ (0.056 and 0.616 g/L, respectively, for WWFI and WWG1) and PAM (0.028 and 0.076 g/L, respectively, for WWFI and WWG1) used as flocculant: (a) pH variations; (b) SS removal; (c) COD removal.

CJ characterisation

The CJ showed an acid pH value of 4.75. The Kjeldahl protein and lipid concentrations were 3.32 ± 0.37 and $5.5 \pm 0.62\%$ (w/dw), respectively. The calculated carbohydrate value represented the highest portion, about $73.42 \pm 1.16\%$ w/dw. The carbohydrates may contain L-arabinose, D-galactose, L-rhamnose, D-xylose and galacturonic acid as reported by various studies on the mucilage of *Cactus opuntia* (Swati & Govindan 2005; Vijayaraghavan *et al.* 2011). Consequently, the high coagulation level of cactus may be attributed to the complex carbohydrate contained in CJ, which has great water retention capacity (Vijayaraghavan *et al.* 2011). It was reported by Swati & Govindan (2005) that galacturonic acid is possibly the active ingredient that ensures the coagulation capability of cactus (*Opuntia* spp.). Insight into the chemical structures of CJ was obtained using IR spectroscopy (Figure 3) and compared to PAM. Similarly to PAM, the main functional groups of CJP included carboxyl (–COOH), hydroxyl (–OH) and amino or amine ($–NH_2$) groups, as well as hydrogen bonds, which are the preferred groups for the flocculation process (Table 3). The existence of the carboxylic group may be related to the presence of polygalacturonic acid (the partial deprotonation of the carboxylic functional group in aqueous

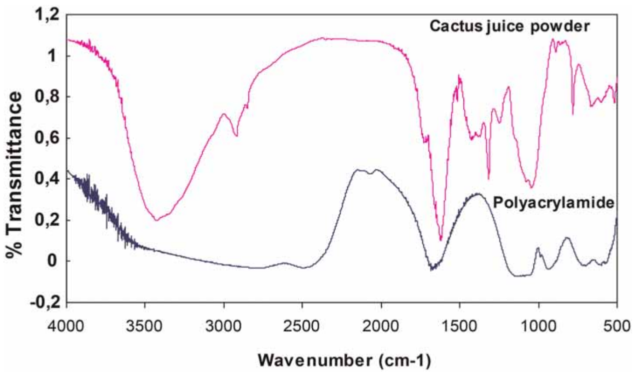

Figure 3 | FTIR spectra of CJP and PAM.

Table 3 | Main functional groups in CJP and PAM from FTIR spectrum analysis

Vibration type	Wave number (cm^{-1})
Hydroxyl and amines (O–H and N–H stretching)	3,400
Carboxyl (C = O stretching)	1,600
Methoxyl (C–O stretching of alcoholic groups)	1,050–1,150

solution implies the chemisorption between charged particles and the –COO– group). Moreover, the presence of –OH groups along the polymeric chain of polygalacturonic acid assumes possible intra-molecular interactions that may distort the relative linearity of the chain (Vijayaraghavan *et al.* 2011). The presence of the $–NH_2$ group confirmed the presence of glycoproteins as reported by Madjoub *et al.* (2001) for Opuntia mucilage. Interestingly, the similar properties of CJ and PAM are confirmed by the presence of the same functionality.

CONCLUSIONS

Synthetic chemicals used in conventional coagulation–flocculation are often costly, may not be available locally, and may have detrimental effects on health and the environment. To overcome these problems, an important objective is to find sustainable alternative technologies using natural products. According to the present study, CJ is a promising natural material to substitute for PAM, the chemical commonly used in wastewater stations as flocculant. Interestingly, cactus is much cheaper than synthetic flocculants. In Tunisia, this plant is abundant and has little commercial use. Moreover, cactuses have no known harmful effects, and, as a natural material, they fit well with the definition of sustainability, making them appropriate for regions of the world conducive to the growth of cactuses. To the best of our knowledge, this is the first study showing the possibility of substituting PAM with a natural, low-cost flocculant from cactuses during the coagulation–flocculation process of wastewater generated by two different industries. The CJ process effectively removed SS and COD with efficiencies greater than 90%, similar to PAM. Interestingly, the CJ IR spectrum showed the same main functional groups as those present in PAM. It seems that CJ has a wider optimum dosage range for flocculation in various colloidal suspensions, depending on the wastewater origin. However, it is still necessary to explore the advantage of CJ and perform tests in pilot scale.

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