Int. J. Environ. Res., 8(4):1295-1308, Autumn 2014
ISSN: 1735-6865

Use of the Cactus Cladodes Mucilage (Opuntia Ficus Indica) As an Eco-Friendly Flocculants: Process Development and Optimization using Stastical Analysis

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Received 15 March 2014; Revised 6 May 2014; Accepted 9 May 2014

ABSTRACT: The Opuntia ficus indica mucilage was an abundant and a low cost product. In the present paper, it was tested as an eco-friendly flocculants for textile waste water treatment. Its performance as flocculants in decolorization, COD removal and turbidity abatement was checked. The natural product was characterized using UV-Vis and Infra-Red spectroscopy. The study of the effect, followed by an optimization and modeling analysis, of some experimental parameters on the coagulation-flocculation performance, using cactus mucilage combined with aluminum sulfate (as a coagulant), showed that the best conditions for flocculation process were given for a pH equal to 7.25, flocculants dose of about 40 mg/L, flocculation mixing speed of about 30rpm and flocculation mixing time of about 11min. This gives a decolorization equal to 99.84%, COD removal of about 88.76% and turbidity abatement of about 91.66%. The comparison between the flocculation performance of commercial flocculants and the bio-flocculants agent exhibited that the latter has the highest pollution removal.

Key words: Opuntia ficus indica, Bio-flocculants, Optimal conditions, Flocculation performance, Statistical analysis

INTRODUCTION

Textile industries are one of the biggest users of water and complex chemicals during textile processing at various processing stages. The unused materials from the processes are discharged as wastewater that is high in color, biochemical oxygen demand (BOD), chemical oxygen demand (COD), pH, temperature, turbidity and toxic chemicals. The direct discharge of this wastewater into the water bodies pollutes the water and affects the flora and fauna. Effluent from textile industries contains different types of dyes, because of their high molecular weight and complex structures, and shows a very low biodegradability (Hsu and Chiang, 1997; Pala and Tokat, 2002; Kim *et al*. 2004; Gao *et al*,. 2007).

Moreover, biological treatment processes are frequently used to treat textile effluents. These processes are generally efficient for biochemical oxygen demand (BOD) and suspended solids (SS) removal, but they are largely ineffective for removing color from the waste (McKay, 1979; Pagga and Brown, 1986). However, due to the nature of the dyestuffs known as melanoidins, dyes are resistant to biodegradation (Yu Zhou, 2008).

Consequently, treated effluents may contain appreciable amounts of color when discharged. So, several physical and chemical methods have been employed for the removal of dyes (Robinson *et al*., 2001) such as adsorption, oxidation, chemical precipitation, etc. Each has its merits and limitations in application (Gao et al, 2007). But, these procedures have not been widely used due to high cost, formation of hazardous by products and intensive energy requirement (Hai *et al*., 2007).

In this regard, considerable researches have been focused on the removal of color and COD from textile effluents using coagulation-flocculation process (Kim *et al*., 2004; Yu Zhou *et al*., 2008; Ciabattiet *et al*., 2010). In general, these studies involved the use of conventional coagulants, such as alum, lime, ferric chloride, magnesium chloride and ferrous sulfate. Recently, researchers concentrated their studies on the flocculation step using low cost, abundant and non-conventional materials instead of traditional flocculation agents to reduce the toxicity of waste water and the treatment cost. Khiari *et al*., 2010 had evaluated the use of Sodium Carboxyl Methyl Cellulose (CMCNa) prepared from the date palm rachis

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as an eco-friendly flocculants. In addition, plant derived polysaccharides are considered as environmentally friendly flocculants due to their biodegradability, stability and low cost. In this regard, Mishra and Bajpai, 2005, studied the application of a food grade polysaccharides namely Plantagopsyllium mucilage for the removal of vat and reactive dyes. Mishra and Bajpai, 2006, exhibited the flocculation performance of Tamaindus mucilage in the removal of vat and direct dyes. Besides, Anastasakis *et al*., 2009, developed the flocculation behavior of mallow and okra mucilage in waste water treatment. The present paper aims to valorize the cactus (Opuntia ficus indica) cladodes juice as an eco-friendly flocculants. The *Opuntia ficus indica (cactus)* was a cheap and easily available plant. The cactus cladodes are mainly constituted by hetero-polysaccharides with a molecular weight from $2.3.10^4$ to 3.10^6 g/mol (Cardinas *et al*., 1997, Medina-Torres *et al*., 2000). It is a mixture of acidic and neutral polysaccharides consisting primarily of arabinose; galactose; galacturonic acid; rhamnose and xylose (Trachtenberg and Mayer, 1981).

It presented multiples uses such as a food thickener (Cardinas *et al*., 1997), food emulsifier (Medina-Torres *et al*., 2000), bio-coagulant (Miller *et al*., 2008) and as a bio-flocculants (the present work).

In this paper, the use of the Opuntia ficus indica (a species of cactus) as a natural flocculants for treatment of industrial waste water taken from dyeing and finishing unit of DENIM fabrics was investigated. The effects of the main experimental conditions (pH of the effluent, flocculants dose, flocculation mixing speed and flocculation mixing time) on the flocculation treatment performance were studied. Besides, modeling and optimization of some experimental conditions were performed in order to improve the performances of the flocculation process. Comparison between a commercialized anionic and cationic flocculants were investigated. The flocculation process performance in all cases was evaluated by means of the decolorization, the COD removal and the turbidity abatement.

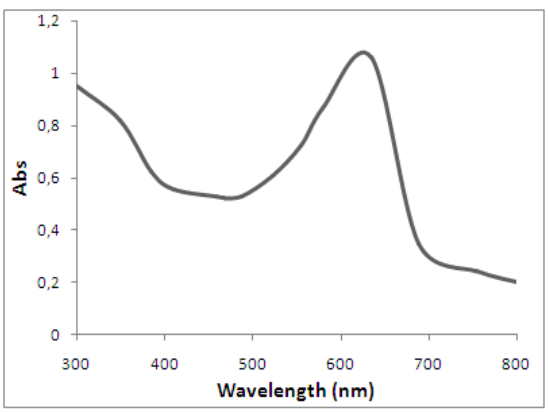


Fig. 1. UV-Vis spectra of different studied dye bath

Table 1. Studied effluent characteristics

pH	**11,9**
Maximum wavelength λ_{max}	630 nm
Abs	10,67
COD (mg O_2/l)	2350
Conductivity (mS/cm)	3,6
Turbidity (NTU)	38

MATERIALS & METHODS

Laboratory tests were conducted on real effluent taken from Tunisian dyeing and finishing unit of DENIM fabrics and conserved at temperature 5°C.

The UV–Vis spectrum of the industrial waste water (diluted 10 times) was recorded using a CECIL 2021 Instruments UV/Vis spectrophotometer and the result was represented in Fig.1.The studied effluent characteristics were given in Table 1.

In the chemical water treatment, the flocculants used were the EPENWATEEXP31/1(PentexQuimica, Espagne), polyacrylamide A_{100}PWG (Kemira Chemicals, France) and cactus mucilage. Their characteristics were represented in Table.2.

TheEPENWATE EXP 31/1, polyacrylamide A_{100}PWG are, respectively, commercial cationic and anionic flocculants used in waste water treatment.

The cactus cladodes were naturally collected in February 2012 from eastern Tunisian region. They were repeatedly washed with distilled water to remove thistles and dirt particles, were sun dried for 3h and cutting into small pieces. The plant was then powdered using domestic mixer and dried at 60°C during 24h. The bio agent was stored in a glass bottle for further use agent without any pre-treatment. The UV–Vis spectrum of the cactus mucilage aqueous fraction (10 times diluted) was recorded using a CECIL 2021 Instruments UV/Vis spectrophotometer.

A Fourier transform infrared (FTIR) spectrum of the bio-flocculants was recorded using a Schimatzu 8400 FTIR Spectrometer (Japan), with the processing software hyper 1.57 using bromide disks. A total of 32 scans for the sample were taken with a resolution of 4/cm, with a range of 4000–400/cm.

The flocculation treatment was combined with coagulation treatment using aluminum sulfate $Al_2(SO_4)_3$(Aldrich, France).

Table 2. Flocculants characteristics

	EPENWATE EXP31/1	POLYACRYLAMIDES A_{100} PWG	Cactus mucilage
Aspect	Powdery, white, granular solid, odorless	Powdery, white, granular solid, odorless	A viscous liquid
Chemical characteristics	Water soluble cationic polymer, present on their chain positive charges, polyelectrolyte	Water soluble anionic polymer, present on their chain negative charges, polyelectrolyte	Non toxic flocculent, biodegradable and not expensive
Density	High	0,75 g/cm^3	1,008 g/cm^3
Molecular weight (g/mol)	2,5.10^5 to 10^6	~5.10^6	2,3.10^4 to 3.10^6

To evaluate the flocculation performance of the cactus mucilage, Jar tests were carried out with special flocculating equipment (Flocculateur W10408, Fisher Bioblock, Germany). The flocculating device was equipped with four breakers to be mixed simultaneously with the same stirring speed (0 to 300rpm). The Jar tests were batch experiments occurring in three steps: the first one was a rapid mixing coagulation (200rpm for 5min) in which 3mL of a coagulant solution (1g/L of aluminum sulfate) was added to 100mL of untreated dyeing waste solution; The second step was a slow mixing flocculation treatment (20-100rpm for 2-20min) in which a fixed quantity of flocculation agent (20-100mg/L) was added; and the third step was a separation of solid and liquid in which the solution was left for sedimentation for half an hour.

After sedimentation, the supernatant was analyzed in terms of percentage of color, chemical oxygen demand COD and turbidity removal.

The absorbance of the supernatant was recorded using a CECIL 2021 Instruments UV/Vis spectrophotometer. The color removal (decolorization (%)) was calculated according to equation Eq (1):

$$decolorization\ (\%) = \frac{Abs_i - Abs_f}{Abs_i} * 100 \qquad (1)$$

Where Abs_i and Abs_f were the absorbance (measured at the maximum wavelength) of the dye bath and the supernatant after coagulation-flocculation treatment, respectively.

The chemical oxygen demand (COD) removal was measured according to the standard method and expressed as CODcr (potassium dichromate as oxidant) (Alpha standard method, 1999). The COD removal (%) was calculated according to equation Eq (2):

$$CODremoval\ (\%) = \frac{COD_i - COD_f}{COD_i} * 100 \qquad (2)$$

Where $COD_i(mgO_2/L)$ and $COD_f(mgO_2/L)$ was the chemical oxygen demand of the effluent before and after coagulation-flocculation treatment, respectively.

The turbidity was determined by a turbid-meter Turb 555IRaccording to the Alpha standard method, 1999.

The turbidity abatement was calculated according to the equation Eq (3):

$$ATur\ (\%) = \frac{Tur_i - Tur_f}{Tur_i} * 100 \qquad (3)$$

Where $Tur_i(NTU)$ and $Tur_f(NTU)$ were the turbidity values of the dye bath solution before and after coagulation-flocculation treatment, respectively.

The coagulation-flocculation process was a pH dependent (Zhou *et al*., 2008, Khiari *et al*., 2010, Sher *et al*., 2013). In this regard, the effect of the pH on the efficiency of the coagulation-flocculation treatment was studied over the pH range from 3 to 10. The pH was adjusted by adding a few drops of 0.5N of NaOH or 0.5N of H_2SO_4. Before the first step of the physic-chemical treatment, the pH of the dyeing solutions was adjusted to 3, 5, 7, 9 and 10. For these experiments, fixed coagulation-flocculation conditions were fixed at: 3mL coagulant dose, 30mg/L flocculants dose, 50rpmflocculation mixing speed, 10min flocculation mixing time.

The effect of the flocculants dose was studied for the bio-flocculants (cactus mucilage) in order to determine the optimal dose. After a rapid mixing coagulation, different flocculants quantities (2, 4, 6, 8 and 10mg) were added to a 100mL of each effluent. The coagulation-flocculation treatment carried out at the optimum pH determined previously (section 2.2.2) with a 50 rpm mixing speed during 10min. The flocculation mixing speed and time were significant parameters for flocculation treatment performance. In fact, series of jar tests were investigated at different mixing velocity

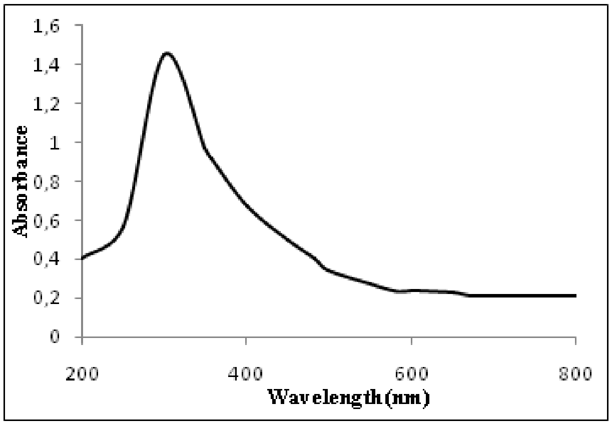


Fig. 2. UV-Vis spectra of the cactus mucilage (10 times diluted)

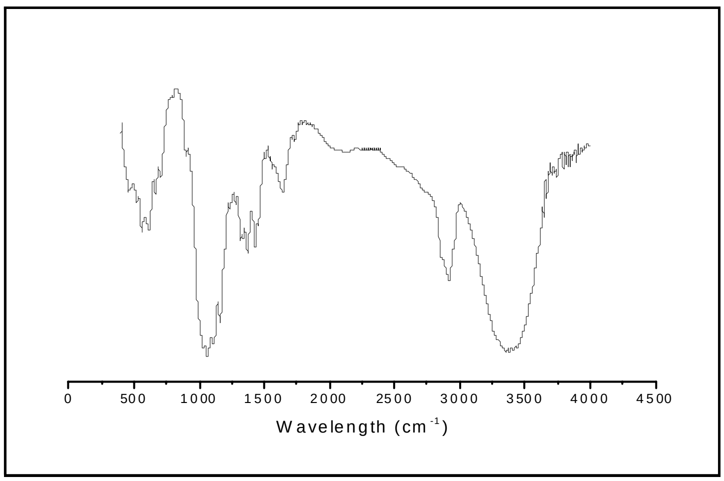


Fig. 3. FT-IR spectra of cactus mucilage

(20, 30, 50, 70 and 100rpm) and time (2, 5, 10, 15 and 20 min). These experiments were carried out at the optimal pH and using the optimal flocculants dose previously determined in 2.2.2 and 2.2.3 sections, respectively.

The optima pH values, optima dosages of the flocculation agent and optimum mixing speed and time were recorded for the maximum decolorization, COD and turbidity removal.

Analysis regression and variance (ANOVA) were used to study the effect of each parameter selected on the obtained results, Minitab (Version 15, State Collegue, PA, USA) was used for the statistical analysis of data. Comparison of means was conducted using ANOVA with Post Hoc Tukey's test at $p<0.05$ (Carmona *et al.*, 2005).

RSULTS & DISCUSSION

The UV–Vis spectrum of the cactus mucilage aqueous fraction (10 times diluted) was represented in Fig.2.It is observed that the bio-agent has no peaks in the visible field.

Fourier-transform infrared spectrum was used to identify the presence of functional groups on the cactus aqueous extract. Fig.3 showed FT-IR spectrum of the bio-flocculants. For Opuntia ficus indica, a broad absorption peaks at around 3200-3500/cm is observed, indicates the presence of carboxylic acid. The absorption band at 2921/cm could be assigned to asymmetric vibration of CH. The stretching vibration band at 2850/cm is due to methoxy group (CH_3-O). The stretching vibration band at 1620/cm is due to asymmetric stretching of the carboxylic C=O double band. A 1430/cm is the peak of phenolic –OH and –

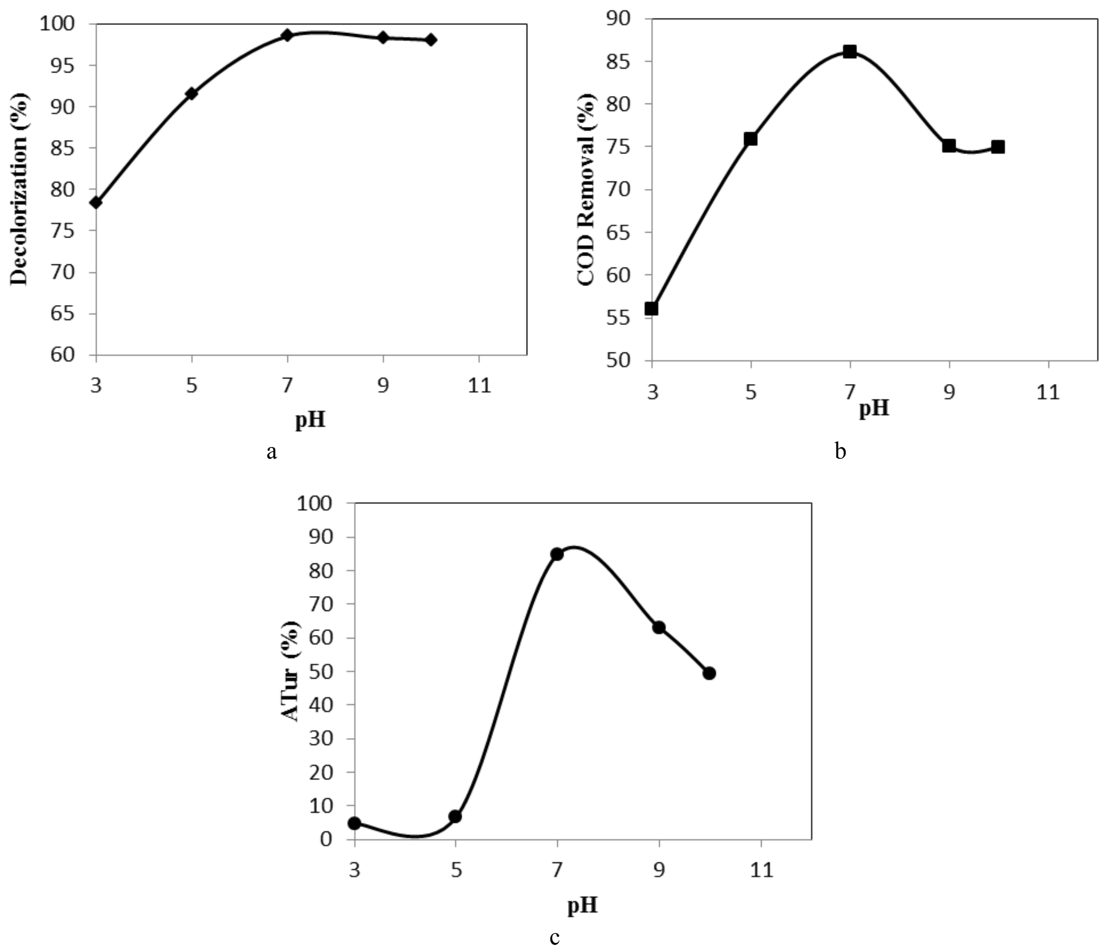


Fig. 4. Effect of the pH on the decolorization, COD removal and Turbidity abatement

C=O stretching of carboxylates. A 1384/cm band is the stretching vibration of –COO. The band at 1072/cm could be due to the vibration of –C-O-C- and –OH of polysaccharides (Barka *et al*., 2013).

Fig.4.a, b and c showed the removal of dyes as a function of pH for real effluent taken from textile industry. The maximum decolorization percentage (Fig.4.a) was observed at neutral pH although considerable color removal was also obtained at basic pH values. Besides, the higher COD removal (Fi.4.b) was seen at about pH 7 and 8. Also, the turbidity abatement (Fig.4.c) reached its maximum at pH 10. So, the best flocculation performance was achieved when the pH was above 7. The removal efficiency of the color, COD and turbidity were, respectively, 98%, 85% and 84.1% at pH neutral.

The plots in Fig.5.a, b and c showed, respectively, the decolorization, COD removal and the turbidity abatement of the treated effluent versus flocculants dose. It was found that with an increase in flocculants dose up to certain level, the flocculation performance increased and followed by a decreasing trend in dye removal with further increases in dose level.

The most effective dose of the flocculants was found to be 40mg/L at which the higher flocculation performance was observed.This trend (increasing and then decreasing trend) in decolorization, COD and turbidity removal was because of the fact that the optimum amount of mucilage in the suspension caused larger amounts of pollutant particles to aggregate and settle. However, an over amount of the flocculants dose than the optimal in effluent would cause the aggregated particle to respreads and would also disturb particle settling (Chan and Chiang, 1995). This behavior could be explained by the repulsive energy between the polymer and the pollutant after the increasing of the flocculants dose, which causes hindrance in floc formation. (Mishra and Bajpai, 2006)

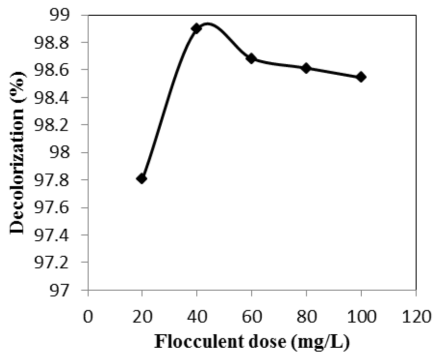


Fig.5.a

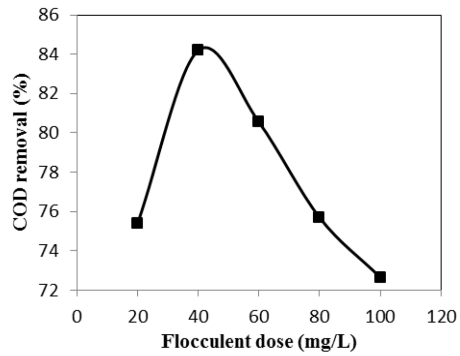


Fig.5.b

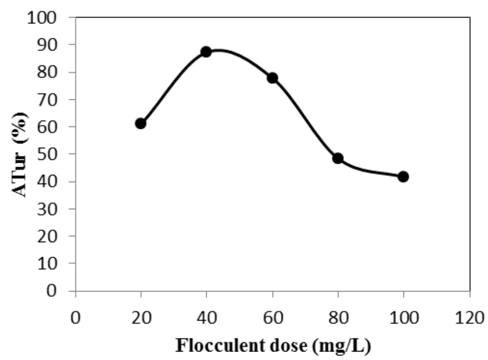


Fig. 5. Effect of the flocculents dose on the decolorization, COD removal and Turbidity abatement

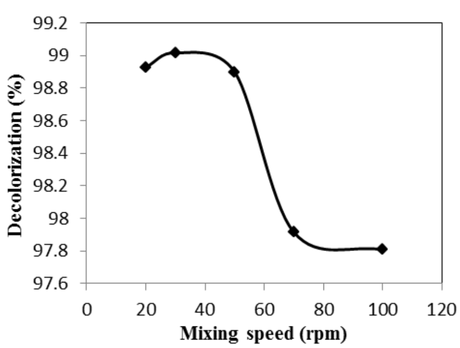


Fig.6.a

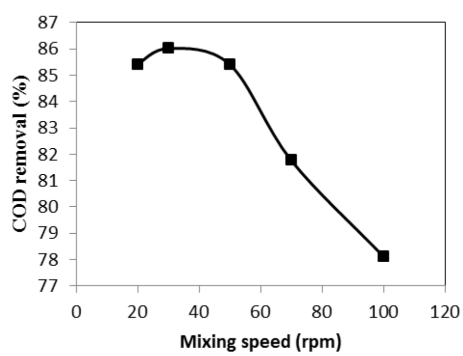


Fig.6.b

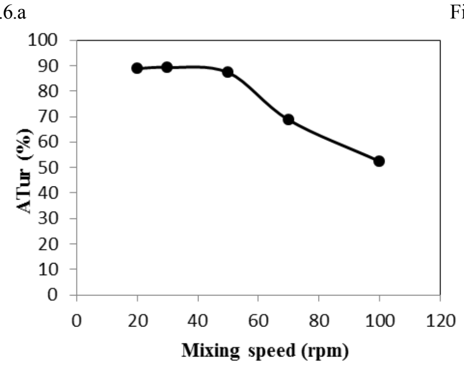


Fig. 6. Effect of the flocculation mixing speed on the decolorization, COD removal and Turbidity abatement

Table 3. Coded actual levels of studied variables and results obtained for a surface design

Run	Coded level of variables				Actuel level of variables				Decolorization	COD removal	ATru
	X1	X2	X3	X4	pH	D (mg/L)	S rpm	T (min)			
1	1	-1	-1	-1	8	20	10	3	63,9	61	50
2	-1	1	-1	1	5	40	10	11	66,4	68	54
3	0	-1	-1	0	6,5	20	10	7	70	66	56
4	-1	1	-1	-1	5	40	10	3	62	64	45
5	-1	-1	-1	0	5	20	10	7	52	50	25
6	0	1	-1	-1	6,5	40	10	3	78	70	65
7	1	1	-1	1	8	40	10	11	97	81	80
8	0	-1	1	-1	6,5	20	30	3	82	73	64
9	-1	1	1	-1	5	40	30	3	68	70	62
10	-1	-1	1	-1	5	20	30	3	58	54	38
11	0	1	1	-1	6,5	40	30	3	90	80	70
12	1	-1	1	1	8	20	30	11	88	72	68
13	1	1	1	1	8	40	30	11	99,4	87	90,3
14	0	1	1	0	6,5	40	30	7	92	82	78
15	1	-1	1	-1	8	20	30	3	76	64	56
16	-1	-1	1	0	5	20	30	7	60	56	40
17	0	-1	-1	-1	6,5	20	10	3	68	63	53
18	-1	-1	-1	1	5	20	10	11	54,3	52	30
19	1	1	-1	0	8	40	10	7	90	78,8	78
20	-1	1	-1	0	5	40	10	7	64	65	50
21	0	-1	1	1	6,5	20	30	11	98,7	86,3	85,2
22	-1	1	1	1	5	40	30	11	76	68	56
23	1	1	-1	-1	8	40	10	3	88,8	71	72
24	-1	1	1	0	5	40	30	7	74	64	52
25	-1	-1	-1	-1	5	20	10	3	50	48	20
26	0	-1	1	0	6,5	20	30	7	97	84	82
27	0	1	-1	0	6,5	40	10	7	84	76	70
28	1	-1	-1	1	8	20	10	11	72,8	70	60
29	1	1	1	-1	8	40	30	3	92	82	74
30	0	1	-1	1	6,5	40	10	11	96,3	83,5	83,2
31	1	-1	-1	0	8	20	10	7	71	68	58
32	0	1	1	1	6,5	40	30	11	99,1	86,1	89,3
33	1	1	1	0	8	40	30	7	98,6	86,4	88,6
34	-1	-1	1	1	5	20	30	11	62	58	43
35	0	-1	-1	1	6,5	20	10	11	72	68	58
36	1	-1	1	0	8	20	30	7	85	70	64

Fig.6.a, b and c showed the removal of dyes as a function of the flocculation mixing speed. From these plots, it is observed that with an increase in mixing speed up to certain level, the flocculation performance increased and followed by a decreasing trend in dye removal with further increases in speed level. Fig.4 showed a transition value around 50rpm. For a lower mixing speed, the decolorization, COD removal and turbidity abatement were relatively high and attained respectively of about 99%, 86% and 89%. However, up to 50rpm, the flocculation treatment performance undergoes a notable decrease to reach 97.8% for the decolorization, 78.11% for the COD removal and 52.36% for the turbidity removal. The higher mixing speed leads to the break of the format aggregated particles (Khiari *et al.*, 2010). The effect of the flocculation mixing time on the flocculation treatment performance was shown

Table 4. Analysis of variance (ANOVA) for developed models

Source	Dgree of Freedom	Decolorization			COD removal			Atur		
		Sum Squares	F-value	P-value	Sum Squares	F-value	P-value	Sum Squares	F-value	P-value
Regression	12	7565,99	33,81	0,000	3809,44	18,91	0,000	10654,8	26,35	0,000
Linear	4	6223,63	83,45	0,000	3032,64	45,17	0,000	8712,25	64,63	0,000
Square	2	1131,91	30,35	0,000	721,64	21,50	0,000	1739,7	25,81	0,000
Interaction	6	20,46	1,87	0,129	55,16	0,55	0,767	202,84	1,00	0,447
Residual Error	23	428,85			386,08			775,1		
Total	35	7993,85			4195,52			11429,9		

Decolorization: R^2=94,64%; R^2(adj)=91,84%

CODremoval: R^2=90,80%; R^2(adj)=86%

ATur: R^2=93,22%; R^2(adj)=89,68%

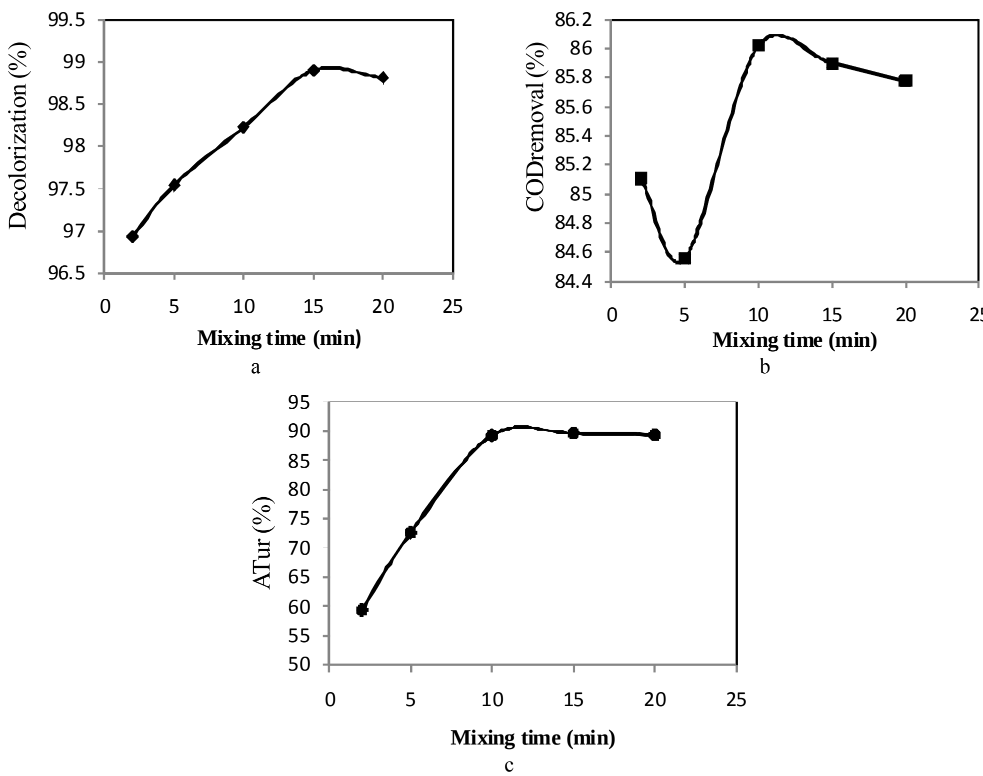


Fig. 7. Effect of the mixing flocculation time on the decolorization, COD removal and Turbidity abatement

Table 5. Cost estimates for flocculation process

Flocculent agent	Cost (TND) per m^3 of effluent	Cost (US$) per m^3 of effluent
Cactus mucilage	0,400	0,244
Polyacrylamide A_{100}PWG	0,600	0,366

in Fig.7.a, b and c. The maximum removal of the pollution was found to be of about 10min. when the mixing time will be higher than 10min, the decolorization, the COD and the turbidity removal were constant. This showed that the maximum performance of the flocculation process was attained for 10min of mixing time. Fig.7.a, b and c showed that the amount of decolorization, COD removal and turbidity abatement increased up to the optimal mixing time and then became constant. A sloped curve indicated the flocculation capacity of the mucilage. Fig.7.b represented three distinct phases: the first one represented the interaction of the pollutant with flocculants, which caused destabilization of the particles in suspensions and they began to flocculate. The second phase indicated the slight decrease of the pollutant removal due to destabilization of the aggregated particles (Agarwal *et al*., 2003). The third phase showed attainment of the stability by the flocs.

These results were analyzed using the software Minitab 15 offers a collection of software, support materials and services that enables to manage and improve the quality of processes (D'Agostino and Stephens, 1986). It is used for calculating basic statistics and for simple estimation and hypothesis. In this paper, a factorial experimental design methodology was employed to estimate the influence of the different variables of the flocculation process. Using the factorial design, we investigated the effects of the experimental factors and the interactions between those factors. The advantages of factorial experiments include the relatively low cost, the reduced experiments number and the increased possibilities to evaluate interactions among the variables. The full factorial design was chosen for the purpose of evaluating the importance of pH of the effluent, dose of the flocculants, mixing flocculation speed and mixing flocculation time on the decolorization, COD removal and turbidity abatement of the treated wastewater.

Response surface methods are used to examine relationship between one or more response variables and a set of quantitative experimental variables or factors (Khuri and Cornel, 1987). The factors considered in this study are: the pH of the effluent (5, 6.5 and 8), the flocculants dose (20 and 40 mg/L), the mixing flocculation speed (10 and 30 rpm) and the mixing flocculation time (3, 7 and 11 min), whereas the experimental results to treat are the decolorization, the COD removal and the turbidity abatement of the treated effluent. The factors values are chosen from the effect study of each factors developed in the previous part.

The experimental surface plan to model is described in Table.3, its regression analysis by a quadratique model leads to the following equations:

$$Decolorization\,(\%) = 86{,}05 + 11{,}491 * pH + 6{,}525 * d(mg/L) + 5{,}425 * S(rpm) + 4{,}387 * t(min) - 11{,}875 * pH^2 - 0{,}687 * t(min)^2 + 1{,}458 * pH * d(mg/L) + 0{,}258 * pH * S(rpm) + 0{,}987 * pH * t(min) - 1{,}947 * d(mg/L) * S(rpm) + 0{,}229 * d(mg/L) * t(min) + 0{,}379 * S(rpm) * t(min)$$

;WithR^2=94.64% (4)

$$CODremoval\,(\%) = 76{,}838 + 7{,}258 * pH + 5{,}541 * d(mg/L) + 3{,}319 * S(rpm) + 3{,}392 * t(min) - 9{,}483 * pH^2 - 0{,}52 * t(min)^2 + 0{,}0083 * pH * d(mg/L) + 0{,}3583 * pH * S(rpm) + +1{,}375 * pH * t(min) - 0{,}641 * d(mg/L) * S(rpm) - 0{,}279 * d(mg/L) * t(min) - 0{,}462 * S(rpm) * t(min)$$

; With R^2= 90.80% (5)

$$ATur(\%) = 71{,}619 + 13{,}495 * pH + 8{,}533 * d(mg/L) + 5{,}366 * S(rpm) + 5{,}333 * t(min) - 14{,}729 * pH^2 - 0{,}716 * t(min)^2 + 0{,}162 * pH * d(mg/L) - 1{,}004 * pH * S(rpm) + 1{,}768 * pH * t(min) - 1{,}866 * d(mg/L) * S(rpm) + 0{,}066 * d(mg/L) * t(min) + 0{,}316 * S(rpm) * t(min)$$

; With R^2= 93.22% (6)

Where pH was the pH of the effluent, d (mg/L) was the flocculants dose, S (rpm) was the mixing flocculation speed, t (min) was the mixing flocculation time, and decolorization (%), COD removal (%) and ATur (%) were calculated, respectively, according to the equation Eq (1), Eq (2), Eq (3), mentioned in section 2.2.2. For the regression equation of the decolorization, COD removal and turbidity abatement, it was found that the squared multiple correlation coefficients "R^2" were respectively 94.64%, 90.80% and 93.22%. So, we can conclude that the models obtained for the responses had a very good predictability (R^2=100% represents a perfect predictability) (Ben Ticha *et al*., 2013).

In order to determine the significant main and the interaction effects of the factors influencing the performance of the flocculation treatment using bio-product, an analysis of variance (ANOVA) was performed using Student's t-test $p<0.05$ (Carmona *et al*., 2005). P-value is the probability value that is used to determine the statistically significant effects in the mathematical model. The importance of the data can be judged by the P-values, with values closer to zero denoting greater significance. For 95% confidence level the P-value should be less than 0.05 for the effect of the considered statistically significant (Srinivasan and Viraraghavan, 2010). The variance analysis of the decolorization, COD removal and the turbidity abatement parameters were given respectively in Table.4. According to Table.4, the obtained P-values of the pH of the effluent (pH), the flocculants dose (d (mg/L)), the flocculation mixing speed (S (rpm)) and the flocculation mixing time (t (min)) varied from 0.000 to 0.001 for the studied responses. So, it seems that the studied factors are statistically significant. However, the P-values of the interactions varied from P= 0.066 to P= 0.992. So, the factor interactions are statistically insignificant with 95% confidence level.

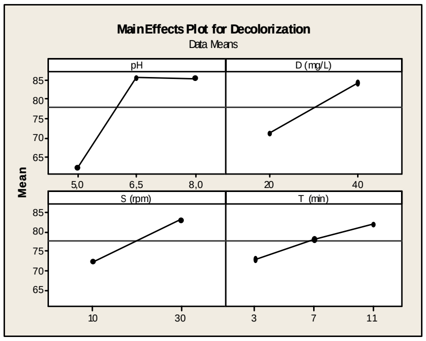
The main effects of each factor (pH, flocculants dose, mixing flocculation speed, and mixing flocculation time) on the flocculation treatment performance were shown in Fig.8.a, b and c. A main effect occurs when the mean response changes across the levels of a factor. It is used to compare the relative strength of the effects across factors (Ben Ticha *et al*., 2013).

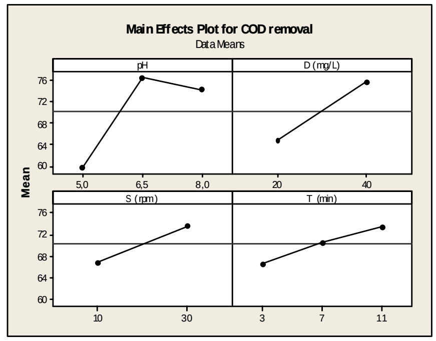
Analyzing the graphs of Fig.8, it seems that the behavior of these factors varies from ones response to another. But, it is clear that the pH of the effluent was the most important variable on the flocculation treatment performance.

The interaction effect plots are also studied and are represented in Fig.9.a, b and c. The interaction between factors occurs when the change in response from the low level to the high level of one factor is

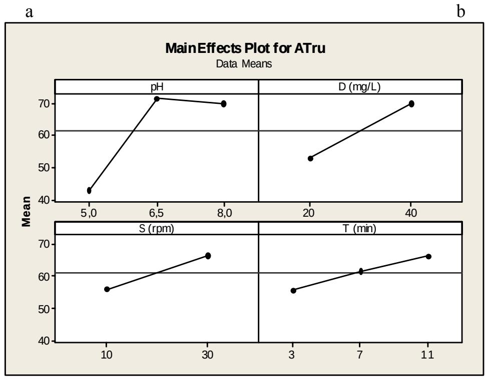
Table 6.The flocculation process performance of different textile waste water using different bio-flocculants

Reference	Type of waste water	Type of flocculent	Optimum dose of flocculent (mg/L)	Decolorization	COD Change	Turbidity abatement
This work	Textile waste water	Cactus mucilage	40	99,84%	88,76% Decrease	91,66%
Anastasakis *et al*, 2009	Synthetic waste water	Malva sylvestris mucilage	12	-	80% increase	96,3-97,4%
Anastasakis *et al*, 2009	Synthetic waste water	Hibiscus esculentus seedpods	5	-	190% increase	93-97,3%
Mishra and Bajpai, 2006	Direct fast scarlet	Tamarindus mucilage	10	60%	-	96%
Khiari *et al*, 2010	Textile waste water	CMCNa	100	-	-	93%
Khiari *et al*, 2010	Textile waste water	CMCNa	100	-	-	85%
Fang *et al*, 2010	Acid black 1	Lignin-base cationic flocculent	75	97,1% Decrease	-	89,3%
Fang *et al*, 2010	Reactive Red 2	Lignin-base cationic flocculent	50	98,3% Decrease	-	96,3%
Fang *et al*, 2010	Direct Red 23	Lignin-base cationic flocculent	35	99,5% Decrease	-	93,5%



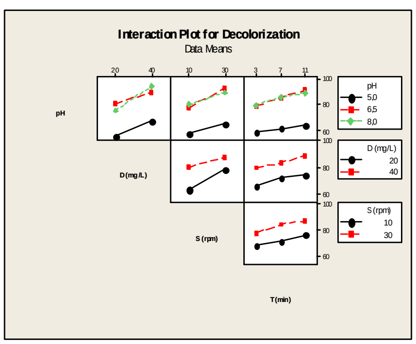


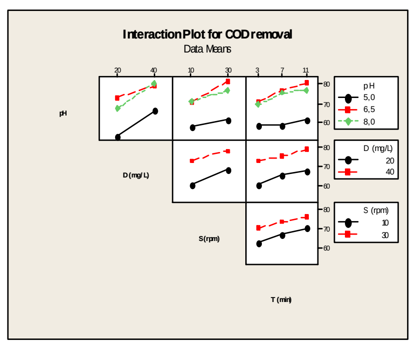
a b



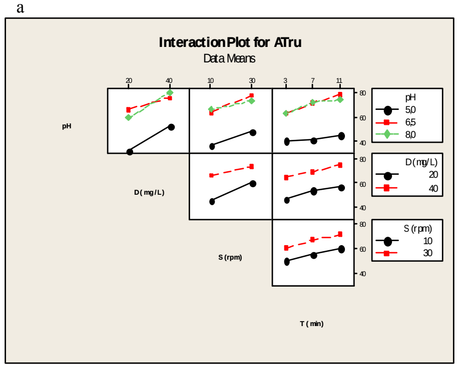
c

Fig 8. Analysis of the main effects plot of the decolorization (Fig.8.a), COD removal (Fig.8.b) and Turbidity abatement (Fig.8.c)





a



c

Fig. 9. Analysis of interaction plot of the decolorization (Fig.9.a), COD removal (Fig.9.b) and Turbidity abatement (Fig.9.c)

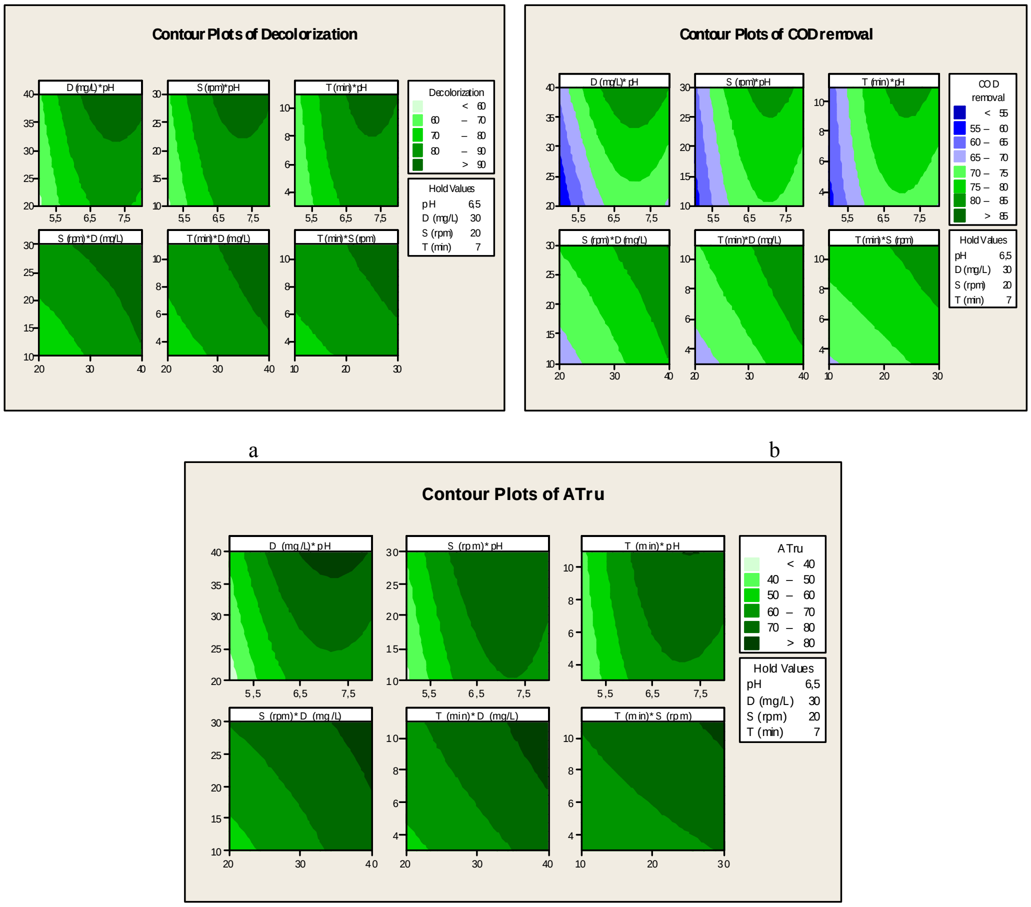


Fig. 10. Contour plots of responses

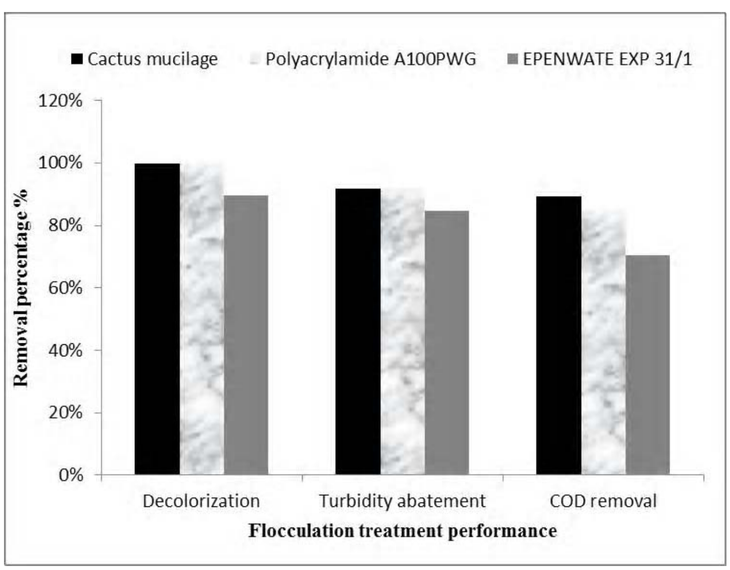


Fig. 11. Comparison of the flocculation treatment performance between natural and commercial flocculants

different from the change in response at the same two levels of a second factor (Ben Ticha *et al*., 2013). From graphs of Fig.9, the equations Eq (4), Eq (5) and Eq (6) and the variance analysis (ANOVA), it can be seen that there is no significant interactions between all others factors. Contour plots, selected in such way as to demonstrate the main effect of individual variables, together with those representing the most significant variable combinations, and were performed. These plots (Fig.10) show the variations in decolorization, COD removal and turbidity abatement, as a result of selecting different values of two variables while the variables for the other variables are held constant. Analysis of the contour plots may be used to identify an optimized solution for the studied response.

From Fig.10, it can be seen that a pH of about 7.2, a flocculants dose between 35 to 40mg/L, a mixing time of about 10min and a mixing speed of about 25 to 30rpmare the optimum conditions for an efficient flocculation treatment. The optimal conditions of the flocculation treatment with cactus mucilage were predicted by response optimizer tool of Minitab 15 software for maximized response. The optimal level of the selected factors were as follows: pH of the effluent of about 7.25, flocculants dose of about 40 mg/L, flocculation mixing speed of about 30rpm and flocculation mixing time of about 11min leading in theory to a decolorization equal to 99.89%, COD removal of about 89.25% and Turbidity abatement above 92.18% with an overall desirability value equal to 99.4%.

The validation experiment was performed to verify the accuracy of the model. Validation tests were carried out at the optimum conditions described in section 3.2.7 (pH of the effluent of about 7.25, flocculants dose of about 40mg/L, flocculation mixing speed of about 30rpm and flocculation mixing time of about 11min).

The experiments were conducted in triplicates and the average value was calculated. In theory, the optimum values under these optimum conditions of the decolorization, COD removal and Turbidity abatement were respectively 99.89%, 89.25% and 92.18%, whereas, the experimental values obtained were respectively 99.84%, 88.76% and 91.66%.

By the comparison of the mean values of the effluent treated obtained and the predicted values, it is obvious that the model is validated. Natural flocculants, mainly polysaccharides, are considered environmentally friendly in comparison with inorganic and organic coagulants due to their biodegradability (Mishra and Bajpai, 2006). However, a comparative study of two commercialized flocculants (EPENWATE EXP31/1 and Polyacrylamide A_{100}PWG) and the cactus mucilage was carried out. As shown in Fig.11, the performance of the cactus mucilage was higher than the achieved by EPENWATE EXP 31/1 and equal to obtained by the A_{100}PWG. It can be deduced that the obtained flocs using cactus mucilage and the A_{100}PWG as flocculants are bigger and heavier than those arising from the system based on the EPENWATE 31/1. The flocculation performance of the studied agents can be classified (from the highest to the lowest) as:

Cactus mucilage~A_{100}PWG>EPENWATE EXP 31/1

From the economic point of view, flocculation process cost of natural agent is influenced by production costs of the bio-flocculants extraction and the flocculation process energy. A comparative study was done between flocculation treatment costs using natural agent and commercial flocculants in terms of flocculation process prices. The results are shown in Table.5. From Table.5 and Fig.11, it is obvious that the cost of flocculation treatment using commercial agent was greater than natural process with equal performance. In this regard, Table.6 compares the results obtained in this research to those obtained in other studies where natural flocculants were used.

CONCLUSION

From the present set of experiments, flocculation, using Opuntia ficus indica mucilage as bio-flocculants for pollution removal, was proved to be a simple and efficient treatment from an economic and technical point of view. The flocculation process performance was evaluated via the measurement of the decolorization, the COD removal and the turbidity abatement after treatment. A modeling and optimization flocculation conditions were carried out using ANOVA response. The optimal flocculation pH, dose of flocculants, flocculation mixing speed and flocculation mixing time were, respectively, about 7, 40mg/L, 30rpm and 11 min. At the optimal conditions, the decolorization, the COD removal and the turbidity abatement were, respectively, 99.84%, 88.76% and 91.66%. The comparative study with commercial flocculants (EPENWATE EXP 31/1, Polyacrylamide A_{100}PWG) showed that the Opuntia ficus indica mucilage had a better performance. In this paper, a new bio-flocculants and abundant agent was found for textile waste water treatment with a low cost.

ACKNOWLEDGEMENTS

The authors are particularly grateful to Mr. Mohamed Touzi the G.D of SITEX (Société Industrielle des Textiles), Mr. Moez Kechida the development engineer in SITEX and all the technical and administrative staff of this company for their assistance and valuable contribution.

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