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Vegetable residueof Chayote (*Sechium edule* SW.) as a natural coagulant for treatment of textile wastewater

C. A. Almeida; M. T. F. de Souza; T. K. F. S. Freitas; E. Ambrósio; H. C. L. Geraldino; J. C. Garcia*

Department of Chemistry, State University of Maringa, Maringa, Paraná, Brazil

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ABSTRACT: In this study the vegetable residue of Chayote (*Sechium edule* SW.) has been evaluated as a potential natural coagulant for removing turbidity and chemical oxygen demand (COD) of textile wastewater. Three important factors for the coagulation/flocculation were optimized by response surface methodology (RSM) based on experimental design Box-Behnken. The maximum efficiency of coagulation/flocculation occurred in pH 6.14, $FeCl_3$ dosage 47.40 mg/L and extract of vegetable residue of Chayote dosage 15.00 mg/L getting turbidity removal percentage and COD of 97.95% and 83.84% respectively. The characterization of the vegetable residue of Chayote by Fourier Transform Infrared Spectroscopy (FTIR) and Nuclear Magnetic Resonance of ^{13}C in the solid state (^{13}C NMR) indicated that the residue showed absorption bands and polysaccharides typical signs, distinctive in the case of anionic polyelectrolytes.

Keywords: *Coagulation/flocculation; Response surface methodology; Textile wastewater; Vegetable residue*

INTRODUCTION

The textile companies and laundries have water as one of its main raw materials used in large volumes. It is estimated that to produce 1kg of fabric, is needed 80L of water. In these industries, a large part of the water consumed is not incorporated into the final product and about 80% becomes highly heterogeneous wastewater and pollutant (Garcia *et al.,* 2007; Hydro, 2011; Lotito *et al.,* 2012). These are characterized by being highly colored because they contain high concentrations of salts, suspended solids, biochemical oxygen demand (BOD), chemical oxygen demand (COD) and compounds toxic to humans and the environment, because studies have shown that many textile dyes and byproducts present themselves carcinogenic and/or mutagenic (Peixoto *et al.,* 2013; Yesilada *et al.,* 2013). The pollution of water bodies with these compounds cause, in addition to visual pollution, changes in biological cycles affecting mainly processes of photosynthesis (Pinheiro *et al.,* 2004; Senthilkumar *et al.,* 2011).

*Corresponding Author Email: *jcgmoraes@uem.br*
Tel.: +55 443 011 3674 Fax: +55 443 011 3674

The technique of coagulation / flocculation followed by sedimentation, is widely used in the wastewater treatment process (Prasad, 2009; Aber *et al.,* 2010; Khayet *et al.,* 2011). Coagulation is the process on which the coagulating agent is added to water, reducing the forces which tend to keep separate particles in suspension, and Flocculation is the agglomeration of these particles by means of fluid transport, to form larger particles which can sediment (Ritcher and Netto, 2003). The efficiency of the process of coagulation / flocculation and the cost of operation are influenced by the type of coagulant used, dosage, pH, ionic strength and the content and nature of the organic compounds present in the effluent, being necessary to experimentally determine the best operating conditions for each type of effluent (Delgado *et al.,* 2003; Duan and Gregory, 2003).

Iron salts and aluminum coagulants are the most commonly used in water treatment and wastewater, and $AlCl_3$, $Al_2(SO_4)_3$, $FeCl_3$ and $Fe_2(SO_4)_3$ the most used due to the low cost and greater efficiency in the coagulation process (Yang *et al.,* 2010).

Iron salts are frequently used as coagulants for water treatment, they neutralize the negative charges colloids and form insoluble hydroxides of low solubility iron, can act on a wide pH range. Compared to aluminum iron molecular weight is higher, forming thicker flakes coagulation, significantly reducing the settling time (Matos *et al.,* 2007).

In order to minimize the impacts caused by the use of inorganic coagulants, have as an alternative the use of natural coagulants from microorganisms, animals or plants. In fact, some studies have shown that crop residues can be applied as natural coagulants (Bongiovani *et al.,* 2010). The main natural coagulants studied in researches related to water treatment are the tannins, which can be used as coagulation and flocculation assistants (Heredia *et al.,* 2010) as well as coagulants, such as *Moringa oleífera Lam* (Pritchard *et al.,* 2010), chitosan that is extracted from the shell of seafood (shrimp and lobster) as an auxiliary flocculation (NG *et al.,* 2013), and cactus extract (Souza *et al.,* 2014), and Aloe vera (Borri *et al.,* 2014). All flocculating assistants are used together with metallic coagulant.

The biopolymers are not harmful to the health and are biodegradable, because they are natural products have low cost, reduce the sludge volume generated and depending on the source from which they are extracted, and may be harvested and processed locally (Sanghi *et al.,* 2006; Zhang *et al.,* 2006; Bhatia *et al.,* 2007; Prasad, 2009).

Compared to the use of natural coagulants mentioned above, there are few studies on the use of the residue of chayote in wastewater treatment. Although there any published articles that use vegetable waste as a coagulant potential, Solís *et al.,* 2013 and Villegas-Rosas *et al.,* 2013 used the chayote shells in discoloration of dyes, a process called enzymatic degradation. Chayote is botanically known as *Sechium edule* (SW.), and belongs to the Curcubitaceae family (Saade, 1996). It is native to Central America and Mexico, there are several varieties with different colors (white or cream, light green and dark green), and variations in size, shape, texture and thorns (PBMH, 2008). When chayote is stripped, this releases a kind of "gum" known as mucilage, which on contact with the skin forms a film (film). It was realized that much of that mucilage is present in the shell (residue), and that the more mature the fruit mucilage has more. The bark had a dark green color and some have spines, to be discarded by the consumer and many complain that mucilage solved to find a use for waste testing it as a natural coagulant, since most of the natural coagulant is characterized by being obtained from mucilaginous species (Souza *et al.,* 2014; Borri *et al.,* 2014).

RSM is a collection of statistical technique for designing experiments, building models, evaluating the effects of factors and searching for the optimum conditions of the factors. RSM also quantifies relationships among one or more measured responses and the vital input factors (Barros Neto *et al.,* 2007).

Thus, this study aimed to optimize the extraction process of natural coagulant from the vegetable residue of Chayote (*Sechium edule* SW.), to be used as coagulant and flocculating auxiliary associated with $FeCl_3$ in the treatment of textile wastewater via the process of coagulation / flocculation using RSM to determine the influence of pH and dosage of coagulant, and the interaction between them.

MATERIALS AND METHODS

Textile wastewater

Textile wastewater samples were obtained from a jeans laundry located in northwestern Paraná, Brazil. Those were collected from the equalization pond, after passing through a static sieve to remove fibers and clays from the washing process.

Preparation of the aqueous extract from the vegetable residue of chayote (sechium edule sw.)

The samples of Chayote (*Sechium edule* SW.) were purchased at a local supermarket, these were washed and peeled. To obtain the extract were placed in a food processor, 2.0 g of peels s *in nature* for 100.0 mL of water being shredded for 2 min and then filtered thus obtaining the liquid extract (filtered).

Coagulation/flocculation process

Coagulation tests / flocculation were performed according Eckenfelder (1996) in the device Jar test (model Millan - JT 203/6 micro controlled) using beakers containing 250.0 mL of the sample, these being subjected to 30 seconds of rapid agitation (120 rpm) followed by 15 min of slow agitation (20 rpm) (Souza *et al.,* 2014). After 15 minutes of slow stirring of all jars tests, the device was turned off, waiting the sedimentation of flakes for 30 minutes and collecting the supernatant. For the tests were used stock solution $FeCl_3$ 10% (w/v) i.e., 0.37 mol/L, and the extract solution of vegetable residue of 2% (w/v).

Analytical methods

The pH of the samples was determined on a digital pH meter (Hanna Instruments) The turbidity was determined by the nephelometric method and oxygen chemical demand (COD) was determined by a colorimetric method using Closed reflux (APHA, 1998). All assays were carried out in triplicate.

Characteristics of vegetable residue of chayote

The Chayote peels were lyophilized for 72 h and then ground, the FTIR spectrum was obtained using an infrared spectrometer with Fourier transform (Bruker Vertex Model 70V) with an interval ranging between 4000 and 400 cm^{-1}. The spectrum of ^{13}C NMR was obtained using an NMR spectrometer (Varian Model Mercury plus 300), with solid probe CP / MAS 7 mm.

Experimental desing

In this study, Box-Behnken response surface experimental design (BBD) with three factors at three levels was used to optimize and investigate the influence of process variables such as $FeCl_3$ dosage (mg/L), extract of vegetable residue of Chayote dosage (mg/L) and pH on the treatment of textile wastewater using coagulation/flocculation. Process variables and their ranges were determined based on the experimental analysis and they are shown in Table 1. After selection of process (independent) variables and their ranges, experiments were established based on a BBD and the complete design consists of 15 experiments with three center points (used to estimate the experimental error). The total number of experiments was calculated from the following (Equation 1).

$$N = K^2 + K + C_P \quad (1)$$

Where in, N is the number of experiments, K is the number of independent variables (factors) and Cp is the number of central point. For statistical calculations, the process variables were coded at three levels (-1, 0 and +1) and the coding was done by the following Equation 2:

$$x_i = \frac{X_i - X_z}{\Delta X_i} \qquad i = 1,2,3 \ldots k \quad (2)$$

where xi, is the dimensionless value of an independent variable; Xi, the real value of an independent variable; Xz, the real value of an independent variable at the center point; and ΔXi, step change of the real value of the variable i. From the BBD experimental data, a second-order polynomial equation was fitted to correlate the relationship between independent variables and responses. Generalized mathematical form of second-order polynomial equation 3:

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i \le j}^{k} \sum_{j}^{k} \beta_{ij} X_i X_j + \varepsilon \quad (3)$$

where, Y is the response, Xi and Xj are variables (i and j range from 1 to k), β_0 is the model intercept coefficient, β_i is the linear coefficient, β_{ii} is the quadratic coefficient, β_{ij} is the interaction coefficient, k is the number of independent parameters (k= 3 in this study and ε corresponding to the residue left by the model (Kousha *et al.,* 2012; Souza *et al.,* 2014). All experimental data were compiled by the software Design Expert 7.1.3, from the preparation matrix planning, development of the mathematical model, variance analysis and optimization parameters The statistical significance of the model and the terms of regression were evaluated by analysis of variance (ANOVA). Then, the models were used for the construction of 3D response surface contour plots to forecast the relationships between independent and dependent variables. Finally, the optimization process was carried out by response surface methodology.

Table 1. Range of variables and their value for the BBD.

Variables	Symbol	Coded value		
		-1	0	+1
		Actual value		
$FeCl_3$ dosage (mg/L)	X_1	20.0	40.0	60.0
Extract dosage (mg/L)	X_2	8.00	20.0	32.0
pH	X_3	3.00	6.00	9.00

RESULTS AND DISCUSSION

Characterization of vegetable residue

The infrared spectrum (Fig. 1) the following absorption bands were identified 1) ῡ O-H = 3400 cm^{-1}, 2) ῡ C-H (sp3) ≈ 2900 cm^{-1}, 3) ῡ C=O ≈ 1700 cm^{-1} and 4) ῡ C-O ≈ 1000 cm^{-1}. The bands observed suggest the possible existence of carboxylic acid group O-H, C-H of alkane group, group C=O carboxylic acid and single bond C-O (Silverstein *et al.,* 2005).

Starting from the ^{13}C NMR (Fig. 2) It was observed the presence of six peaks, as in ^{13}C NMR each peak

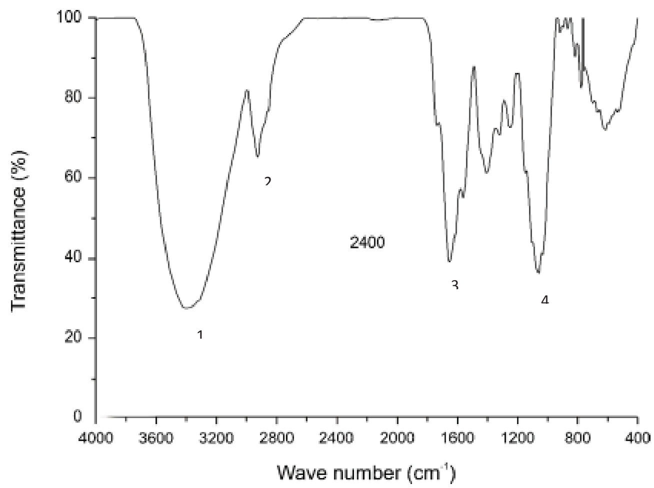

Fig. 1: FT-IR spectra of vegetable residual of chayote (Sechium edule SW.).

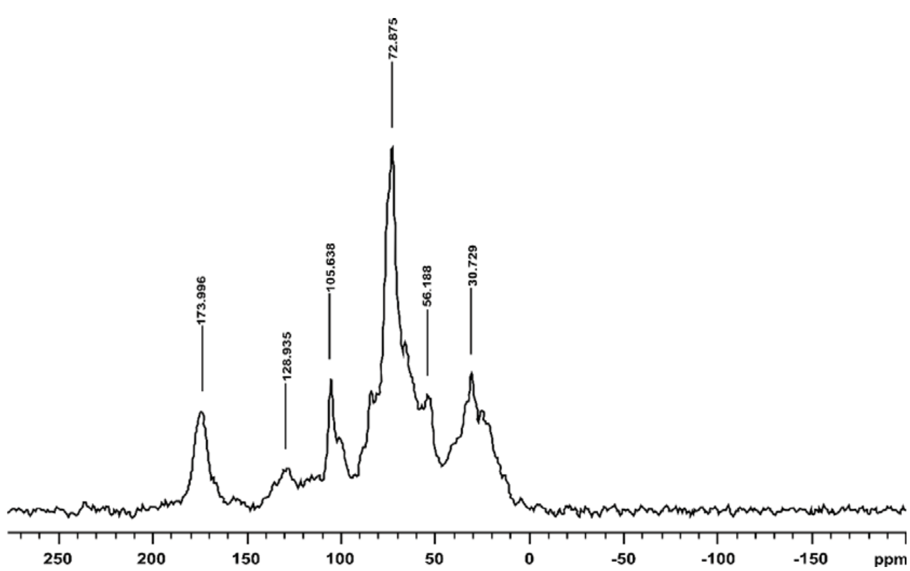

Fig 2: 13C NMR spectra of vegetable residual of chayote (Sechium edule SW.).

corresponding to a single carbon when the molecule is not symmetrical, at first it is assumed that the sample has 6 carbons in its structure, and that these carbons in principle comprise the monomeric structure of the polymer.

Signals were detected at a range of 30-54 ppm corresponding to aliphatic carbons, in the region of wide signals 73 ppm corresponding to C-OH, in the region of 105 ppm corresponding to C-O-C and in the region of 173 ppm of carbonyl (possibly derived from the carboxylic acid), all these signals indicate the presence of polysaccharides. These results show that the identification of compounds formers of the extract is very important in view of their application as natural polymer, which due to their structural characteristics, promotes greater adsorption of colloidal particles (Silverstein *et al.,* 2005).

Regression model and statistical analysis

The relationships between removal of turbidity (Y_1) and removal of COD (Y_2) and three independent variables ($FeCl_3$ dosage (X_1), extract dosage (X_2) and pH (X_3)) were studied. The experimental design listed in Table 2 also provides the responses such as

Table 2: BBD and experimental results

Run	$FeCl_3$ dosage (mg/L)	Extract dosage (mg/L)	pH	Turbidity removal (%)	COD removal (%)
1	20.0 (-1)	8.00 (-1)	6.00 (0)	94.09	75.72
2	60.0 (1)	8.00 (-1)	6.00 (0)	97.61	78.48
3	20.0 (-1)	32.0 (1)	6.00 (0)	93.48	72.95
4	60.0 (1)	32.0 (1)	6.00 (0)	97.17	70.88
5	20.0 (-1)	20.0 (0)	3.00 (-1)	94.67	63.27
6	60.0 (1)	20.0 (0)	3.00 (-1)	95.29	69.49
7	20.0 (-1)	20.0 (0)	9.00 (1)	90.49	68.80
8	60.0 (1)	20.0 (0)	9.00 (1)	94.32	73.99
9	40.0 (0)	8.00 (-1)	3.00 (-1)	93.36	69.84
10	40.0 (0)	32.0 (1)	3.00 (-1)	96.13	66.38
11	40.0 (0)	8.00 (-1)	9.00 (1)	97.18	74.95
12	40.0 (0)	32.0 (1)	9.00 (1)	92.03	62.92
13	40.0 (0)	20.0 (0)	6.00 (0)	97.14	80.21
14	40.0 (0)	20.0 (0)	6.00 (0)	98.18	87.13
15	40.0 (0)	20.0 (0)	6.00 (0)	97.18	81.59

removal of turbidity (Y_1), removal of COD (Y_2) each experimental were conducted in random order to prevent statistical distortion, that is, atypical deviations associated with combinations of levels that do not exist. Without randomization, the errors may seem smaller than they actually are (Barros *et al.,* 2007). The response obtained in Table 2 was correlated with the three independent variables using a polynomial equation, Equation 3. Least squares regression was used to fit the obtained data to Equation 3. The best fit models in the coded factors in equations 4 and 5.

$$Y_1 = +97.50 + 1.46X_1 - 0.43X_2 - 0.68X_3 + 0.043X_1X_2 + 0.80X_1X_3 - 1.98X_2X_3 - 1.45{X_1}^2 - 0.46{X_2}^2 - 2.36{X_3}^2 \quad (4)$$

$$Y_2 = +82.98 + 1.51X_1 - 3.23X_2 + 1.46X_3 - 1.21X_1X_2 - 0.26X_1X_3 - 2.14X_2X_3 - 4.05{X_1}^2 - 4.42\,{X_2}^2 - 10.04\,{X_3}^2 \quad (5)$$

In order to investigate the adequacy of the developed mathematical models, ANOVA evaluations are made and shown in Table 3, imply that the developed models can describe the coagulation/ flocculation process significantly. To measure how well the suggested model fit the experimental data, the parameters F-value, R^2, p-value, and lack of fit were used (Ferreira *et al.,* 2007; Mishra *et al.,* 2008; Amenaghawon *et al.,* 2013). As can be seen in Table 3, F-values of responses implied that the quadratic model is significant. Moreover, each term in the model was also examined for significance and the p-value smaller than 0.05 implies that the corresponding model term is highly significant. From Table 3, it is clear that the linear and quadratic term $FeCl_3$ dosage, the quadratic term of the effluent pH and the interaction between the extract dosage and the pH of the effluent were the factors that most influenced ($P < 0.05$) the removal of turbidity and the linear and quadratic terms of the $FeCl_3$ dosage were not significant ($P > 0.05$), indicating that the $FeCl_3$ dosage established for the

Table 3: ANOVA table for responses.

	Y_1		Y_2	
Source	F-value	P-value	F-value	P-value
Model	7.70	0.0184[a]	7.44	0.0287[a]
X_1	17.56	0.0086[a]	16.96	0.2465[b]
X_2	1.53	0.2715[b]	1.47	0.0378[a]
X_3	3.82	0.1080[b]	3.69	0.2610[b]
X_1X_2	7.444E-003	0.9334[b]	7.444E-003	0.4913[b]
X_1X_3	2.66	0.1637[b]	2.57	0.8799[b]
X_2X_3	16.20	0.0101[a]	15.65	0.2458[b]
${X_1}^2$	7.99	0.0368[a]	7.72	0.0625[b]
${X_2}^2$	0.83	0.4052[b]	0.80	0.0481[a]
${X_3}^2$	21.27	0.0058[a]	20.54	0.0020[a]
Lack of fit	4.01	0.2062[b]	1.38	0.6504

[a] Significant ($P < 0.05$); [b] not significant ($P > 0.05$); X_1: ferric chloride dosage, mg/L; X_2: extract dosage, mg/L; X_3: pH; Y_1: removal of turbidity and Y_2: removal of COD.

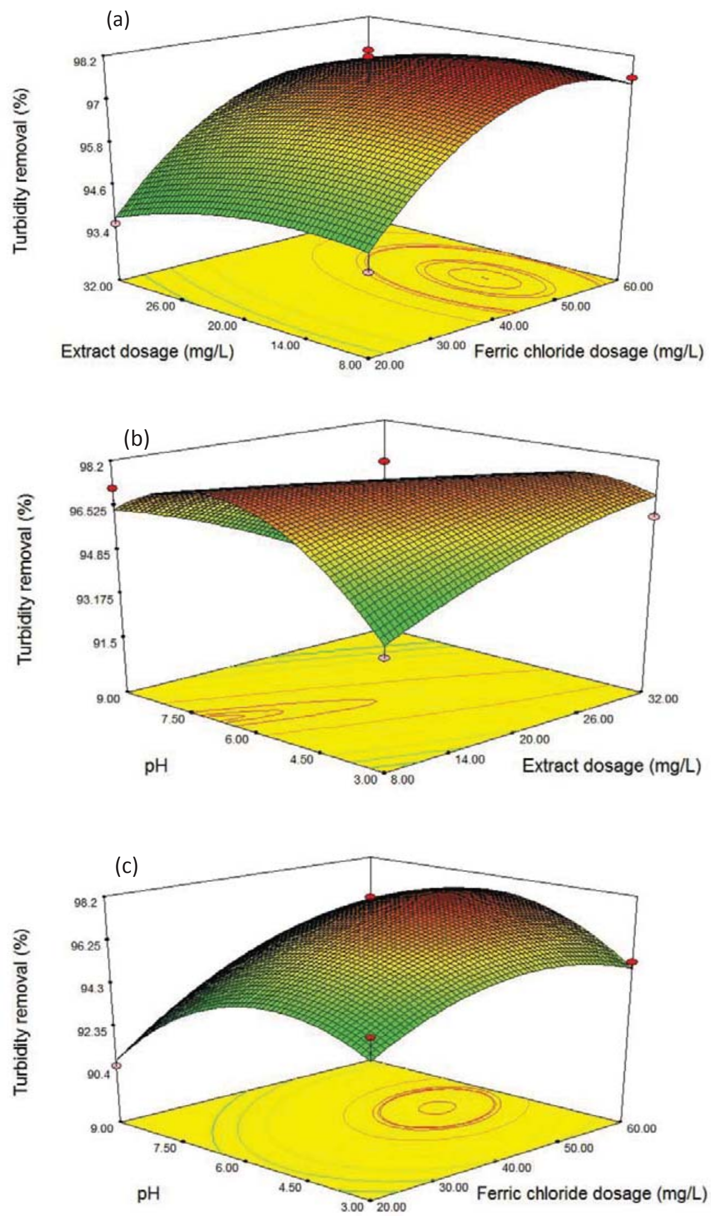

Fig. 3. Response surface plots (a-c) showing the effects of variables (X_1: ferric chloride dosage, mg/L; X_2: extract dosage, mg/L and X3: pH) on the turbidity removal.

central delineation limit (40.00 mg/L) It proved to be sufficient for the process, in the removal response of COD. Furthermore, the lack of fit F-values between 1 and 2, indicating that lack of fit is not significant relative to pure error, i.e. the model fits the experimental data and the predictive can be used for any combination of the values of the variables. Actual values are data for each specific run from Table 2, and predicted values are created by the model, Equations (4) and (5). The values of total determination coefficients (R^2), observed for responses Y_1 (removal of turbidity %) and Y_2(removal of COD %) were 0.9327 and 0.9184, respectively, suggesting a good fit of the model to experimental data.

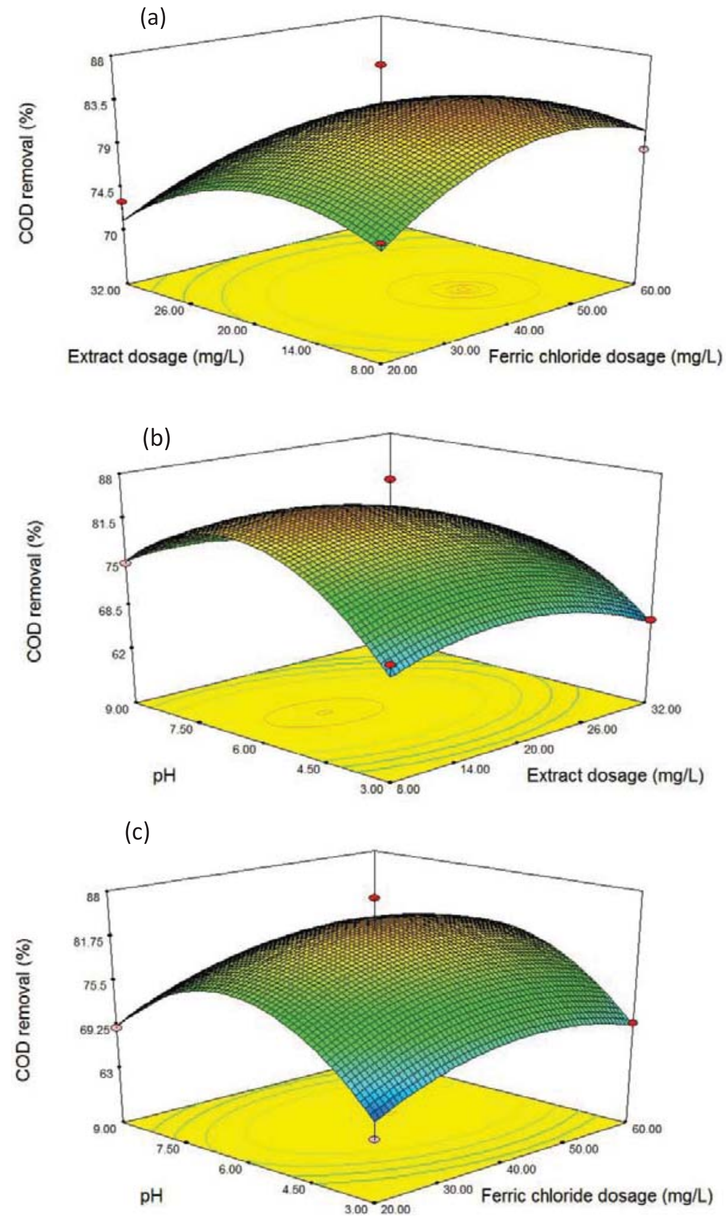

Fig. 3. Response surface plots (a-c) showing the effects of variables (X_1: ferric chloride dosage, mg/L; X_2: extract dosage, mg/L and X3: pH) on the turbidity removal.

Influence of $FeCl_3$ dosage

Statistical analysis of the experimental data identified $FeCl_3$ dosage (X_1) as the most important variable in the response analysis. As shown in Equations (4) and (5) and Table 3, $FeCl_3$ dosage has a large, linear, positive effect on the removal of turbidity (Y_1) and removal of COD (Y_2). A 3D response surface plot for the responses in Figs. 3 (A) and 4 (A) depicts the change of removal of turbidity (Y_1) and removal of COD (Y_2) with varying $FeCl_3$ dosage and extract dosage, plotted for the case where pH of 6.00. From the results, it is found that removal of turbidity (Y1) is increased with increasing $FeCl_3$ dosage and removal of COD (Y_2) is increased with $FeCl_3$ dosage in the central point (40.00 mg/L) throughout the coagulation/flocculation process.

Influence of extract dosage

Statistical analysis of the experimental data identified extract dosage (X_2) as the most important variable in the coagulation/flocculation process of textile wastewater. As shown in Equations (4) and (5) and Table 3, extract dosage has a large, linear positive effect on removal of turbidity (Y_1) and quadratic positive effect on removal of removal of COD (Y_2). A 3D response surface plot for the responses in Figs. 3 (B) and 4 (B) depicts the change of removal of turbidity (Y_1) and removal of COD (Y_2) with varying extract dosage and pH, plotted for the case where $FeCl_3$ dosage of 40.00 mg/L. From the results, it is found that removal of turbidity (Y_1) is increased with increasing extract dosage and removal of COD (Y_2) is increased with extract dosage in the central point (20.00 mg/L) throughout the coagulation/flocculation process of laundry industry.

Influence of pH

pH has a linear negative effect on the removal of turbidity (Y_1) and linear positive effect removal of COD (Y_2) in coagulation/flocculation process. The quadratic influence of this factor has significant negative effect on responses. Figs. 3 (C) and 4 (C) shows the influence of $FeCl_3$ dosage and pH on removal of turbidity (Y_1) and removal of COD (Y_2) in coagulation/flocculation process for the case where the extract dosage is 20.00 mg/L. By constant pH in 6.00 removal of turbidity (Y_1) and removal of COD (Y_2) the maximum removal.

Process of optimization

The critical point (great) for each response surface model was found by solving the partial derivatives of Equations 4 and 5.

The values obtained for the critical point were as follows: $FeCl_3$ dosage = 49.86 mg/L, extract dosage = 15.12 mg/L and pH = 5.95 for removal of turbidity and $FeCl_3$ dosage = 44.93 mg/L of the extract dosage = 14.88 mg/L and pH = 6.33 for COD removal.

The working conditions were established as an average between the great values that were found for each removal. These values were $FeCl_3$ dosage = 47.40 mg/L, extract dosage= 15.00 mg/L and pH = 6.14 having as a response 97.95% removal of turbidity and 83.84% of COD.

CONCLUSION

The aqueous extract of vegetable residue of Chayote proved to be a potential natural coagulant. The characterization by FTIR and ^{13}C NMR of this residue showed absorption bands and typical polysaccharides signals.

In addition to the presence of hydroxyl and carbonyl groups and by the behavior displayed by the extract during the study was typical anionic polyelectrolytes which were used as flocculation aids when associated with primary coagulants. Through the experimental design it was possible to predict the best conditions to work with the association of coagulants, obtaining optimal values as: FeCl3 dosage = 47.40 mg/L, extract dosage = 15.00 mg/L and pH = 6.14.

RSM optimization was conducted to investigate the actual applicability of extract of vegetable residue of chayote as a natural coagulant using the response surface method. The analysis of variance (ANOVA) showed that the data fitted the quadratic model and that the extract dosage was significant for the percent removal of COD and turbidity. The best conditions obtained for the variables were: FeCl3 dosage = 47.40 mg L^{-1}, extract dosage = 15.00 mg L^{-1} and pH = 6.14 having as a response 97.95% removal of turbidity and 83.84% of COD.

It is concluded that the biopolymer derived from extract of vegetable residue of Chayote showed up as a coagulant potential. Natural polymers do not present risks to human health, are cheap, renewable sources, non-toxic, highly biodegradable and fully available for the treatment of effluent ecological in an efficient manner.

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