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Cassava Wastewater Treatment by Coagulation/Flocculation Using *Moringa oleifera* Seeds

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The present study evaluated the efficiency of starch wastewater treatment through coagulation/flocculation using extracts of *Moringa oleifera* (M.O.) seeds as a natural coagulant. The experiments were performed following a Rotatable Central Composite Design (RCCD). The studied variables were: M.O. concentration and sodium chloride (NaCl) solution concentration. The response variables analysed were: Turbidity, Apparent Colour, COD and Cyanide Ion. The operational conditions optimized by the statistical program were: M.O. concentration of 2,484 mg L^{-1} and saline concentration of 0.9 mol L^{-1}. Under these conditions the removal estimates are 89.16 % (turbidity), 54.43 % (apparent colour), 66.39 % (COD) and 9.9 % (cyanide ion).

1. Introduction

Cassava processing industries, especially those producing starch, have a high polluting potential due to the release of their effluents. Among the main characteristics of this effluent is the high organic load and the presence of cyanide ion, a constituent with high toxic potential. Farms effluents treatments had been done predominantly by systems that use biological mechanisms (Kuczman et al., 2017; Carvalho et al., 2017). However, biological treatment systems present limitations for effluents containing cyanide, as is the case of starch wastewater. Among all plant materials that have been tested over the last few years (Padilha et al., 2013; Jun and Ahmad, 2015; Wolf et al., 2015; Camacho et al., 2015; Teh and Wu, 2014; Daza et al., 2016), the extract obtained from *Moringa oleifera* seeds has shown itself to be one of the most effective as a primary coagulant for wastewater treatment, and it can be compared with conventional coagulants such as those derived from aluminium salts (Bongiovani et al., 2015; Amagloh & Benang, 2009; Lédo et al., 2009; Noor et al., 2018; Ndabigengesere and Narasiah,1998).

There are several studies in the literature that investigate the use potentials of this seed. Prasad (2009) investigated the effects of the dosage, pH and salt concentration for *Moringa oleifera* seed coagulant extraction, for optimization purposes, for colour removal process of distillery effluents. Lédo et al. (2009) investigated the efficiency of two coagulants, aluminium sulphate and *Moringa oleifera* seeds, for turbidity removal in water samples of low turbidity. Due to certain characteristics of starch wastewater, such as high turbidity, colour and colloidal organic matter, the adoption of coagulation/flocculation techniques has become a practice that allows to obtain excellent results, as will be presented throughout the study.

2. Material and methods

2.1 Samples Physicochemical Characterization

The effluent samples used in this study were collected in Zadimel Amidos, which is located in Concordia do Oeste district, in the city of Toledo, Paraná, Brazil. In order to characterize the effluent, the following

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physicochemical parameters were analysed: BOD, COD, Dissolved Phosphorus, Total Nitrogen, Total Solids, pH, Turbidity, Cyanide Ion, Zinc, Cadmium, Copper and Iron.

2.2 *Moringa oleifera* Coagulant Solution Preparation

The preparation of coagulant solution followed a 5 g ratio of peeled M.O. seeds to 100 mL of NaCl salt solution (molar concentrations are shown in Table 1). The procedures followed in this work are described by Formentini-Schmitt et al. (2013) and Lédo et al. (2009).

2.3 Coagulation/Flocculation Assays

For the coagulation/flocculation assays it was used the Jar-Test Micro-controlled equipment, Milan brand, JT-103 model, with capacity for six simultaneous tests. The assays were carried out using a Rotatable Central Composite Design (RCCD), which consists of a complete factorial design (4 assays), a star configuration (4 assays) and the triplicate (3 assays) in the central point, totalizing 11 assays. The data were analysed with STATISTICA™ 8.0 Software.
The studied variables were the concentration of *Moringa oleifera* coagulant and the molar concentration of saline extraction solution of the active principle from *Moringa* seeds. In Table 1 are shown the real and coded ranges used for the tests

Table 1: Real and coded range values used in the RCCD.

Variable	Level				
	-1.41	-1	0	1	1.41
MO concentration (mg L^{-1})	1977	2100	2400	2700	2823
NaCl concentration (mol L^{-1})	0.3	0.5	1	1.5	1.7

Note: Concentrations were based on preliminary assays results.

For the test conditions a fast mixing rate of 100 rpm was determined for a corresponding fast mixing time of 2 min, followed by a slow mixing speed of 20 rpm for a slow mixing time of 10 min. After 60 minutes of decantation, supernatant aliquots were collected for further Turbidity, COD and Free Cyanide removal analyses.

3. Results and discussion

Results of the physicochemical characterization performed with the wastewater samples and some literature data for comparative purposes are shown in Table 2.

Table 2: Wastewater characterization and some literature data comparison.

Parameter	Results found	Hasan et al. (2014)	Lamo and Menezes (1979)	Fioretto (2001)
BOD (mgO_2 L^{-1})	1,450	–	1,400-34,300	30,000
COD (mgO_2 L^{-1})	2,240	32,000	6,280-51,200	–
Dissolved Phosphorus ($mgPO^{-4}$ L^{-1})	91.83	412	155-598	255.8
Total Nitrogen (mgN L^{-1})	168	165.2	140-1,150	–
Total Solids (mgTS L^{-1})	7,666	6,000	5,800-56,460	70,000
pH	4.1 – 4.8	5	3.8-5.2	4.03
Turbidity (NTU)	1,090 – 1,350	–	–	–
Cyanide Ion (mgCN L^{-1})	16.4 - 23	–	22.0-27.1	–
Zinc (mgZn L^{-1})	0.345	0.962	–	3.7
Cadmium (mgCd L^{-1})	0.01	0.041	–	–
Copper (mgCu L^{-1})	0.01	0.259	–	0.9
Iron (mgFe L^{-1})	7.456	–	–	16.6

According to Fioretto (2001) the cassava starch composition is variable, depending on the cassava variety used, which is correlated with the edaphoclimatic conditions of the place where it is cultivated. Therefore, the differences observed in Table 2 are expected as a function of this characteristic.
The samples pH values were found in the range of 4.1 to 4.8. Fioretto (2001) found a close result, with a mean pH of 4.03. According to Hess (1962), the low pH of the effluent is due to fermentation, with formation of

carbon dioxide, mainly. The biochemical decomposition of glucose produces organic acids, especially pyruvic acid, lactic acid, acetic acid and others, also responsible for the pH decrease.
Results obtained based on the experimental design in terms of the final concentration and reduction percentage are shown in Table 3.

Table 3: Final concentration after treatment through Coagulation/Flocculation.

Assay	Independent variables				Dependent Variables (Final concentration)			
	Coded		Real					
	X_1	X_2	MO conc.	NaCl conc.	COD	Turbidity	Apparent Colour	Cyanide Ion
1	-1	-1	2,100	0.5	1,230 (45.09 %)	*210 (84.44 %)*	2,570 (51.23 %)	21.9 (4.78 %)
2	1	-1	2,700	0.5	1,170 (47.77 %)	198 (85.33 %)	2,680 (49.15 %)	21.3 (7.39 %)
3	-1	1	2,100	1.5	1,740 (22.32 %)	205 (84.81 %)	3,160 (40.04 %)	20.9 (9.13 %)
4	1	1	2,700	1.5	*1,900 (15.18 %)*	193 (85.70 %)	*3,820 (27.51 %)*	21.8 (5.22 %)
5	0	0	2,400	1	770 (65.63 %)	194 (85.63 %)	2,390 (54.65 %)	21 (8.70 %)
6	0	0	2,400	1	*650 (70.98 %)*	189 (86.00 %)	*2,380 (54.84 %)*	20.9 (9.13 %)
7	0	0	2,400	1	800 (64.29 %)	186 (86.22 %)	2,440 (53.70 %)	*20 (13.04 %)*
8	-1.41	0	1,977	1	1,510 (32.59 %)	191 (85.85 %)	2,730 (48.20 %)	21.2 (7.83 %)
9	1.41	0	2,823	1	1,470 (34.38 %)	*146 (89.19 %)*	3,200 (39.28 %)	21.9 (4.78 %)
10	0	-1.41	2,400	0.3	1,600 (28.57 %)	194 (85.63 %)	2,530 (50.02 %)	22.1 (3.91 %)
11	0	1.41	2,400	1.7	1,630 (27.23 %)	199 (85.26 %)	3,740 (29.03 %)	*22.2 (3.48 %)*

Note: In parentheses are the respective reduction percentages.

As can be seen in Table 3 results show that the highest turbidity reduction was 89.19 % corresponding to assay number 9 (coagulant concentration of 2,823 mg L^{-1} and molar concentration 1 mol L^{-1} NaCl solution). The lowest reduction for this parameter was assigned to assay number 1 (2,100 mg L^{-1} and 0.5 mol L^{-1}), with 84.44 %. The use of chitosan to obtain a percentage of turbidity reduction of 71.6 %, using 170 mg L^{-1} of chitosan under acidic pH, was observed by Souza & Pawlowsky (2011). The sufficient time for the flakes sedimentation was 20 minutes. For this removal percentage in the present study it would be necessary around 30 minutes to obtain similar results using a concentration of 2,823 mg L^{-1} of Moringa.
Lima et al. (2009) added tannin and flocculation auxiliaries. The authors, through a factorial design, evaluated the effect of pH (4.6; 6.3 and 8.0) on turbidity removal process. Results of the tests showed that the maximum reduction efficiency achieved was 50 % (from 800 to 400 NTU) under the conditions of basic pH (8.0) and tannin concentration of 0.2 mL L^{-1}, while the present study worked under acidic pH conditions, without any adjustments, obtaining a significantly higher removal percentage.
As already pointed out in previous items, pH of the wastewater used in this study was in the range of 4.1 to 4.8. This pH range could justify the lower removals observed for apparent colour in this study in relation to the other ones found in the literature. This consideration is made based on Madrona et al. (2010), so that the authors found better results under pH 8.0 using surface water sample, while in pH range from 4.0 to 6.0 the colour removal was significantly lower. The mean pH values of Formentini-Schmitt et al. (2013) and Paterniani et al. (2009) were 7.2 and 6.5, respectively, all above the current study range.
The coagulation assays point to the great capacity of organic load removal, expressed here by the COD parameter. This is confirmed by assay number 6 (2,400 mg L^{-1} and 1 mol L^{-1}), which presented a percentage of 70.98 %. The lowest removal was performed in assay number 4 (2,700 mg L^{-1} and 1.5 mol L^{-1}), with 15.18 % efficiency. The mathematical model that describes the behaviour of the process is presented in Equation 1:

$$\mathrm{COD}\ (\%) = 0{,}67 - 0{,}072\mathrm{NaCl} - 0{,}162\mathrm{MO}^2 - 0{,}193\mathrm{NaCl}^2 \quad (1)$$

The explanation for the good results of COD removal through coagulation process finds consistent grounds in Hess's (1962) considerations. The author states that most of the organic matter in these wastewaters is in the

form of dissolved or colloidal suspension, so that the adoption of simple or natural decantation treatment techniques do not achieve desired effects, which in turn are possible by coagulation/flocculation techniques.
In one of the studies that denote the ability to remove COD by coagulation is the investigation developed by Lima et al. (2009). The authors were able to obtain a COD reduction efficiency of 91 % for the treated cassava starch wastewater (from 33,440 to 3,040 $mgO_2\ L^{-1}$) under conditions of pH 8 and in the presence of tannin 1.0 mL L^{-1} and Polipan 0.030 mg L^{-1}.
The response surface and contour plot for the COD removal process are shown in Figures 1 (a) and (b).

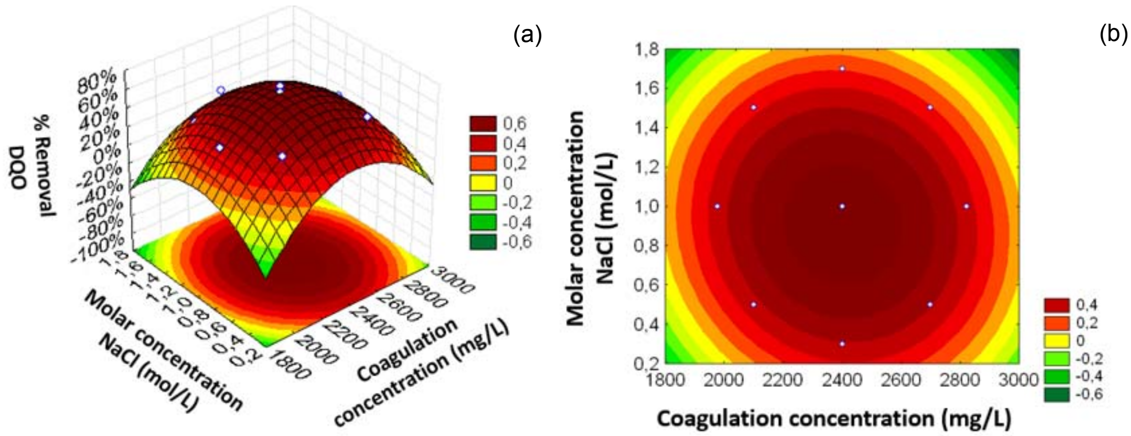

Figure 1: (a) Response surface a (b) contour plot for COD removal.

Oliveira et al. (2001) using Aluminium Polychloride (50 mg L^{-1}) as a coagulant, obtained a COD reduction rate of around 86 % (from 14,866 to 1,960 mg L^{-1} COD), a percentage considerably closer to that of the present study. It is important to highlight that for coagulation assays Oliveira et al. (2001) have used flocculation auxiliaries, specifically a natural polyelectrolyte used at a concentration of 2.5 mg L^{-1}, which represents an additional cost to the process. In addition, it was necessary to adjust the pH of the effluent to 8.0, as a function of the narrower range of performance of this type of coagulant, which is not the case for Moringa, which can be used in a wide range of pH without affecting in large scale its efficiency.
Souza & Pawlosky (2011) in their studies of cassava starch wastewater by-products recovery, through coagulation using chitosan as a coagulant, found a maximum COD reduction of 23.5 % (from 85,400 to 65,330 $mgO_2\ L^{-1}$). To obtain this percentage the required sedimentation time was 20 minutes, which in terms of the present study would represent a removal of approximately 20 %, almost identical to that found by the authors.
According to Bhatia et al. (2007) and Bongiovani et al. (2015), the volume of sludge produced using M.O. coagulant is considerably lower compared to the aluminium (Oliveira et al., 2001) and presents no problem to be discarded, since according to Katayon et al. (2006) and Daza et al. (2016), sludge generated from Moringa and other natural coagulants is easily biodegraded, presenting low environmental risk.
Thus, by analysing COD removal values, it is seen that Moringa coagulation treatment can be used as the primary treatment for cassava starch effluent. This, according to Souza & Pawlowsky (1998), would significantly reduce the pollutant load of this wastewater and consequently the costs for the treatment, which are due to the need to acquire large areas for the construction of waste stabilization ponds, whose size is also dependent on the organic load of the effluent.
Coagulation assays results indicate that the method used is able to remove the cyanide ion, but not very expressive, presenting a removal range from 3.48 % to 13.04 %. The highest percentage in this reduction was found in assay number 7 (2,400 mg L^{-1} and 1 mol L^{-1}), and the lowest efficiency was found in assay number 11 (2,400 mg L^{-1} and 1.7 mol L^{-1}).
Xu et al. (2005) evaluated the removal of free cyanide from praziquantel wastewater (anthelmintic drug). In this study, the authors propose the joint use of two treatment technologies - coagulation/flocculation + GMA (gas membrane absorption). The authors conducted coagulation assays using different concentrations of PAC. At the end of the tests, the following optimal coagulation conditions were observed: 0.5 g L^{-1} of PAC; pH 10; rapid mixing time of 3 minutes with 140 rpm, slow mixing time at 40 rpm, with a settling time of 15 minutes. The free cyanide removal result was considered by the authors as not significant, with a final concentration of 3,154 mg L^{-1}, against a concentration of the raw wastewater of 3,180 mg L^{-1}, that is, a reduction of less than 1 %. On the other hand, the GMA system was able to remove 99.9 % of free cyanide.

Results obtained by Xu et al. (2005), as well as those of the present study, conclude that the influence of coagulation on cyanide ion removal is inefficient.
Because it has more than one response variable - turbidity, apparent colour, COD and cyanide ion - and it is interesting to find the optimum operational values of the independent variables (coagulant concentration and saline solution molar concentration) that simultaneously satisfy all dependent variables, the STATISTICA™ software uses the Desirability function approach. With this artifice, the simultaneous optimization of all response variables is maximized in a single value, called global desirability, as follows:

- Coagulant solution concentration: 2,484.6 mg L^{-1};
- Saline solution concentration (NaCl): approximately 0.9 mol L^{-1}.

Under these conditions the 4 response variables - turbidity, apparent colour, COD and cyanide ion - should show removal values of 89.16 %, 54.43 %, 66.39 % and 9.9 %, respectively.
It has been known that the action of *Moringa oleifera* as a coagulant is attributed to soluble cationic proteins. However, overdosages of this coagulant may affect the efficiency of the coagulation process. It is possible to observe this trend in Table 3, which shows that for the parameters Apparent Colour, COD and Cyanide Ion the ideal coagulant concentration was 2,400 mg L^{-1}, which resides in the central portion of the studied range from 1,977 to 2,823 mg L^{-1}, that is, higher values induced a lower efficiency.
Ndabigengesere et al. (1995) found that the degree of coagulation, measured by Zeta potential, by the use of *Moringa*-based coagulants may be affected by overdosages. The authors observed that additions above the optimal dosage of 500 mg L^{-1} - Zeta potential of 0 mV (maximum degree of coagulation) - resulted in a reversal of the zeta potential to +4 mV, which represents an excess of positive ions in the solution, causing the colloids to become positively charged. From the point of view of saline solution molar concentration , the fact that 1.0 mol L^{-1} concentration has been identified as the most efficient in the removal studies for all parameters can be attributed to the salt concentration levels used for extraction of the active principle of Moringa. The explanation for this, according to Prasad (2009), may probably be the effect known as "salting-out", which has the principle that proteins are less soluble in high concentrations of salt. Madrona et al. (2010) analysed different KCL solutions concentrations (0.01, 0.1 and 1.0 mol L^{-1}) in their tests. The authors observed that with KCL concentration of 0.1 mol L^{-1} the M.O. solution had the same amount of protein as in the MO solution extracted with 1.0 mol L^{-1} KCl, although the one with best results was the MO solution extracted with 1.0 mol L^{-1} KCl.

4. Conclusions

The main conclusions from the results are:

- Maximum removals found for the response variables Turbidity, Apparent Colour, Cyanide Ion and COD were 89.19 %, 54.84 %, 13.04 % and 70.98 %, respectively.
- The assay conditions for the maximum removals were: 2,400 mg L^{-1} M.O. concentration for the variables Apparent Colour, COD and Cyanide Ion. In case of Turbidity the ideal M.O. concentration for removal within the range studied was 2,823 mg L^{-1}. In terms of saline solution, the ideal concentration was 1.0 mol L^{-1}.

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