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Decoloration of an Effluent from Textile Industry using *Moringaoleifera* Seed Extract

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Abstract

This research evaluated the removal of textile dyes by the physicochemical process of coagulation-flocculation using a natural coagulant extracted from the seed of the *Moringaoleifera* tree. The seed was collected and two different coagulants extracts were prepared: simple extract and saline extract. For the experimental essays, synthetic samples were prepared in the laboratory using three azo dyes with different chemical structures; direct blue 71, reactive red 2 and acid yellow 23, its decoloration was evaluated individually and in mixtures. Also, a wastewater sampling was carried out in a textile plant located in the municipality of Zinapecuaro, Michoacan and tests of coagulant activity and determination of optimal parameters for the process were carried out, this was accomplished through the simulation of jar test and measurement of absorbance using an ultraviolet-visible light spectrophotometer to determine the percentage of visible color removal.

Keywords:Decoloration; Textile effluents; Dyes; Coagulation-flocculation; *Moringaoleifera* seeds.

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1. Introduction

The textile industry is one of the most active chemical industries and is considered as the main cause of water pollution. Its monthly average production in Mexico reaches 70,000 tons of fiber, which is equivalent to a water consumption of approximately 18,500,000 m^3 [19]. This sector causes strong levels of pollution, mainly in the generation of wastewater containing large amounts of dyes which have complex characteristics and chemical structure and low biodegradability [1]. The textile industry effluents are characterized by extreme fluctuations in parameters, such as: the chemical oxygen demand (COD), biochemical oxygen demand (BOD), pH, color and salinity. Some of the typical parameters of these effluents are: visible color (1100-4500 units), chemical oxygen demand (800 - 1600 mg/L), pH generally alkaline (9-11) and total solids (6000 - 7000 mg/L) [5].

The presence of dyes in water not only alters its aesthetic appearance and causes social conflicts in the communities involved, but it also interferes in the photosynthetic process carried out by some organisms and the exchange of oxygen [2], in addition many dyes are toxic and resistant to degradation processes, even some of them are considered to be carcinogens and mutagens [4]. There is a wide variety of dyes with different chemical structures; among them are azo, anthraquinone and indigo dyes, which in turn can be direct, reactive, disperse, acid etc. The azo dyes are the most commonly and represent 75% of the pigments used by the textile industry [21].

Considering that azo dyes are those of greater use, this research focuses on their decoloration through the physicochemical process of coagulation-flocculation using a natural coagulant extracted from *Moringaoleifera*seeds. A triad of basic or primary colors were chosen; direct Blue 71, acid yellow 23 and reactive red 2, because from them is derived a wide range of shades. In spite of the remarkable technological advance that exists nowadays for the removal of this type of pollutants, it represents a challenge not only for the textile industry, but also for the printing, pharmaceutical, cosmetics and even food industry. The above, mainly due to its complex operation, high cost of treatment and the lack of sustainability in the methods used.

Currently, conventional methods do not achieve the total elimination of these pollutants and sometimes it is necessary to complement a treatment system with several technologies. Generally there are physicochemical, chemical, biological and more recently enzymatic methods for the treatment of wastewater [1, 4]. The most commonly used in the textile industry are chemical methods that use oxidizing agents such as ozone, peroxide of hydrogen and even ultraviolet radiation, these techniques are referred to as "Advanced oxidation processes" [15] and consist of reactions forming chemical oxidation resulting in hydroxyl radicals (OH-) capable of degrading dyes to its mineralization, however its main limiting factors are the high cost, complex operation and in some cases, its long degradation time [1].On the other hand, one of the most commonly methods used in water treatment and purification is the physicochemical process of coagulation-flocculation, in which chemical compounds are used, the most employed are aluminum and iron salts [15]. This physicochemical process is widely used both in developed countries as in developing, for it easy operation and low cost. Nevertheless when applied in textile wastewater, presents some disadvantages, such as the generation of large volumes of sewage sludge and the ineffective decoloration of some soluble dyes [4]. In the area of the textile industry, this mechanism has proved to be inefficient because of its low removal of pollutants. However, this project aims to dispense with the use of these chemical coagulants to evaluate the ability of an organic origin coagulant, which was extracted from the seeds of the *Moringaoleifera* tree instead.

2. Materials and Methods

The general experimental methodology to carry out this investigation is made up of six main stages, they are shown in figure 1.

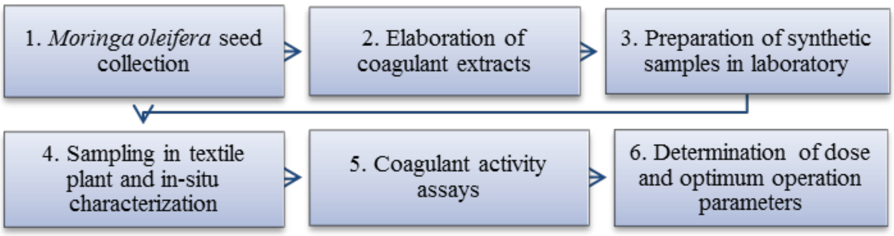

Figure 1.General diagram of the experimental methodology

2.1. Moringa oleiferaseedcollection

Moringa seed was collected manually directly from the dried pods of trees in some lands of the municipality of La Huacana, Michoacan, located to the South of the State, in the coordinates 18 ° 58' North latitude and 101 ° 48' West longitude, at an altitude of 480 meters above sea level. It borders to the North with the municipalities of Nuevo Urecho and Ario, to the East by Turicato, to the South by Churumuco and Arteaga and to the West by Mugica and Apatzingan. Its distance to the State capital is 161 km. Its climate is tropical with rains in summer and in some parts dry steppe. It has an annual rainfall of 800 millimeters and temperatures ranging from 10 to 54° C.

2.2. Elaboration of coagulantextracts

In order to analyze the efficiency of two different extracts, there was prepared a simple extract and a saline extract, both as explained below.

- Simple extract:

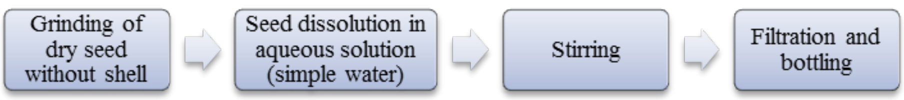

Figure 2.Diagram of the production process of simple extract coagulant

To prepare 1 L of simple extract coagulant in aqueous solution, fisrt of all Moringa seeds were collected and dried. Then, they were reduced to powder in a domestic mill. The 50 g of powder were dissolved on 1 L of distilled water by mixing vigorously for 45 min with a magnetic stirrer. Then, the mixture was filtered and used on coagulation tests without further purification.

- Salineextract:

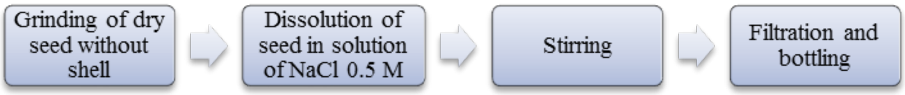

Figure 3.Diagram of the production process of saline extract coagulant

To prepare 1 L of saline extract coagulant in aqueous solution, fisrt of all Moringa seeds were collected and dried. Then, they were reduced to powder in a domestic mill. The 50 g of powder were dissolved on 1 L of NaCl 0.5M solution by mixing vigorously for 45 min with a magnetic stirrer. Then, the mixture was filtered and used on coagulation tests without further purification.The extract obtained, in both cases, is a clear liquid as shown in figure 4, and it must be stored and labeled in an airtight container and preserved at a temperature of 4 °C.

Figure 4. Elaboration of coagulantextract

2.3. Preparation of synthetic samples in laboratory

In order to test the coagulants extracts, in a first stage, synthetic samples were prepared in the laboratory using three types of dyes, all of azo type but with a classification of different use; direct blue 71, reactive red 2 and acid yellow 23, were tested separately and as mixtures.

Different volumes of synthetic samples were prepared in volumetric flasks, always with a known concentration dye, which ranged between 30 and 50 ppm for experimental purposes before to the analysis of a textile effluent with the objective to obtain preliminary data on appropriate doses of coagulant.

Figure 5.Azo dyes used for the preparation of synthetic samples

2.4. Sampling in textile plant and in-situ characterization

The sampling was carried out in a place of the plant which can be considered as an open channel and collector that receives water from other lines and that located just before three artificial lagoons where wastewater discharges are arranged before they are dischaged to a river that encompasses the municipality of Zinapecuaro and Querendaro. A simple wastewater sample was taken corresponding to the washing process that is performed after textile dyeing.

The wastewater sample was collected directly from the discharge pipe with the sampler, where it was immediately characterized with a portable equipment for temperature, pH, oxygen dissolved, total and dissolved solids, electrical conductivity, salinity and ORP (redox potential); then, the samples were stored in plastic bottles.

2.5. Coagulantactivityassays

In order to determine the coagulant-flocculant ability of *Moringaoleifera* extract, jar test procedures were simulated in laboratory; the aim was to evaluate the decoloration capacity of textile effluent for both types synthetic samples (simple dyes and mixtures), according to the following scheme (figure 6):

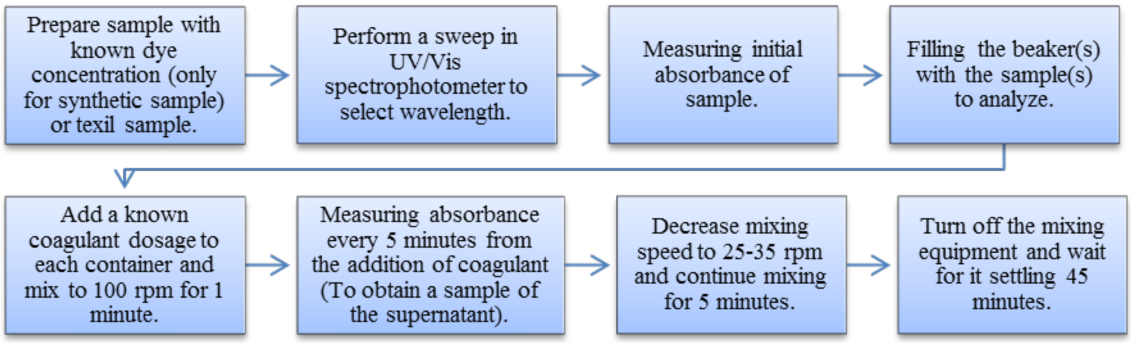

Figure 6.Methodology for testing of coagulant activity

3. Results and discussion

3.1 Coagulantactivityassays

- Directblue 71 dye:

Direct dye with a concentration of 30 ppm was tested with four different dosages and the results of the measurements of absorbance are shown in table 1.

Table 1.Decoloration of direct Blue 71 with different coagulant dosages

Time (min)	Coagulant dosages (mL/L)			
	5	20	25	30
0	1.712	1.762	1.708	1.759
2	1.635	2.173	2.632	2.997
5	1.658	2.111	1.471	1.058
10	1.608	0.955	1.079	1.048
15	1.603	0.587	0.542	0.966
20	1.592	0.485	0.533	0.725
25	1.592	0.432	0.472	0.677
30	1.591	0.307	0.328	0.562
35	1.592	0.288	0.259	0.529
40	1.59	0.168	0.172	0.512
45	1.589	0.092	0.109	0.419

The following graph shows the previous results (Table 1). The curves with doses of 20 and 25ml/L showed a similar behavior, where both doses showed up to 95% of visible color removal.

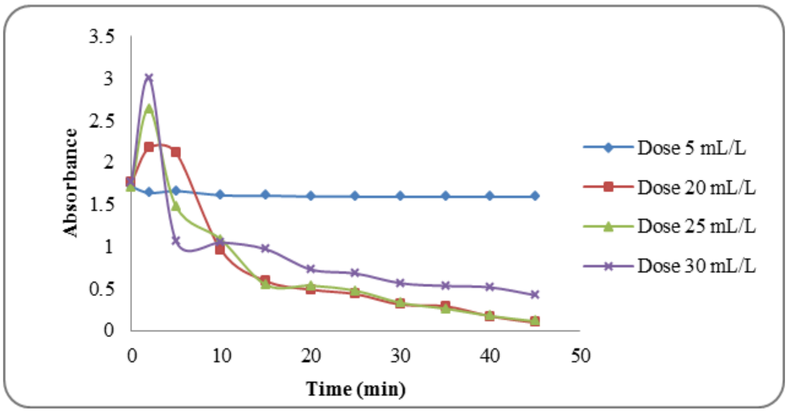

Figure 7.Kinetics of decoloration of direct blue 71

As illustrated in figure 7, the dose of 5mL/L is inefficient and the decoloration is almost zero, on the other hand, with doses above 20 mL/L there is a possible restabilization of colloidal particles that prevents an appropriate coagulation, flocculation and sedimentation. The following pictures shows different stages and results of the decoloration process of direct blue 71 dye (figure 8, 9, 10 and 11).

Figure 8.Synthetic sample of direct blue 71 without treatment

Figure 9.Synthetic sample of direct blue 71 five minutes after the addition of the coagulant

Figure 10. Formation of flocs

Figure 11.Decolorationprocess of direct blue 71 dye with 20 mL/L coagulant dosage

- Mixtures of two and three dyes:
  - Direct blue 71 (DB71) + Acid yellow 23 (AY23):

A synthetic sample was prepared with two dyes, one direct and other acid, each one with 30 ppm concentration, this mixture was tested, following several preliminary studies with a dose of 25 mL/L of coagulant, visible color removal for this case was only 46%, results are shown in table 2.

  - Direct Blue 71 + Acid yellow 23 + Reactive Red 2 (RR2):

In the same way, a mixture of three dyes was prepared: direct, acid and reactive dyes with individual concentrations of 30 ppm and a dosage of coagulant 25 mL/L was applied, the percentage of visible color removal reached 82%, results are enclosed in table 2.

Table 2.Decoloration of two mixtures of dyes (direct + acid, direct + acid + reactive) with coagulant dosage of 25 mL/L

Mixture direct blue 71 (30ppm) + acid yellow 23 (30ppm)		**Mixture direct blue 71 (30ppm) + acid yellow 23 (30ppm) + reactive red (30ppm)**	
Dosage = 25 mL/L		**Dosage = 25 mL/L**	
Time (min)	**Absorbance**	**Time (min)**	**Absorbance**
0	1.577	0	0.601
2	2.07	2	1.336
5	2.067	5	0.257
10	1.439	10	0.204
15	0.938	15	0.18
20	0.917	20	0.179
25	0.917	25	0.178
30	0.914	30	0.16
35	0.878	35	0.159
40	0.872	40	0.146
45	0.847	45	0.106

The results of the previous table are expressed graphically for both mixes as shown below in figure 12. Figures 13 to 20 show pictures of coagulation process and sedimentation for mixtures of two and three dyes, as mentioned above.

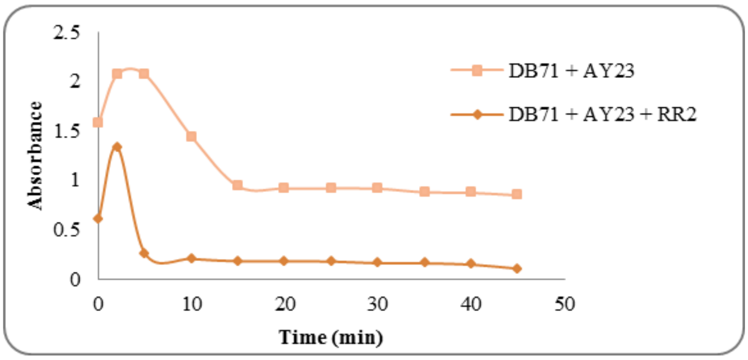

Figure 12.Decolorationprocess of dyes mixtures

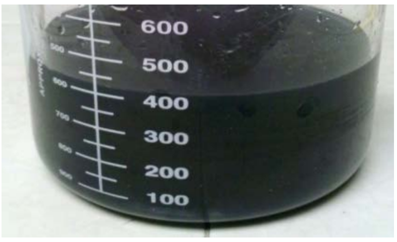

Figure 13.Synthetic sample of direct blue 71 and acid yellow 23 without treatment.

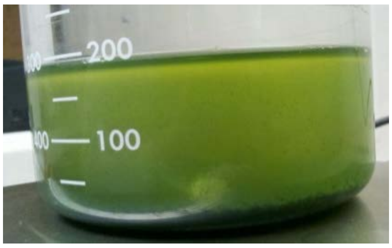

Figure 14.Synthetic sample of direct blue 71 and acid yellow 23 twenty minutes after the addition of the coagulant

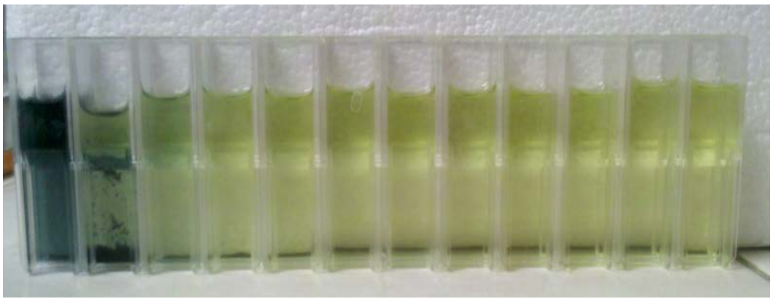

Figure 15.Decolorationprocess of the mixture direct blue 71 and acid yellow 23 with 25 mL/L coagulant dosage

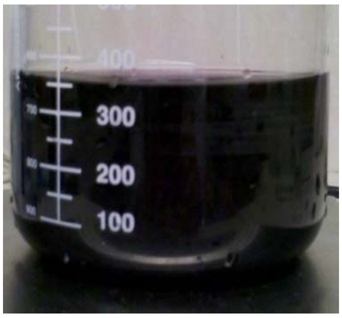

Figure 16.Synthetic sample of DB71, AY23 and RR2 without treatment

Figure 17.Synthetic mixture at the time of the addition of coagulant

Figure 18. Formation of flocs

Figure 19.Synthetic mixture after twenty minutes of sedimentation

Figure 20.Decolorationprocess of the mixture direct blue 71, acid yellow 23 and reactive red with 25 mL/L coagulant dosage

- Real sample of textile plant:

After taking the sample in the textile plant some basic parameters were measured in the site by means of portable equipment, parameters that were also evaluated after treatment with coagulant of *Moringa*, the results were the following (Table 3):

Table 3.Preliminary characterization of textile wastewater

Parameter	**Without treatment**	**With treatment**
Temperature	25.5°C	18.5°C
pH	9.5	7.3
Dissolved oxygen	1.92 ppm	----
Total dissolved solids	309 ppm	32 ppm

In addition, the same experiments as for synthetic samples were conducted. First of all, the full process of coagulation-flocculation-sedimentation of the textile wastewater sample was evaluated for 45 minutes with absorbance measurements every 5 minutes to analyze the behavior of the curve of decoloration with a dosage of 20 mL/L which results are shown individually in figure 21. In the case of textile effluent with the above mentioned dosage, visible color removal percentage was 80%. Subsequently, the same sample was subjected to other three different doses of coagulant; 10, 30 and 40 mL/L, where only the initial and the last after 45 minutes absorbances were measured, the results are showed in figure 22, it could be observed that with doses greater than 30 mL/L there is a phenomenon of restabilization and saturation of colloidal particles which increase the value of the absorbance at the end of the treatment.

Figures 23 to 26 show textile wastewater coagulation-floculation and sedimentation process. In terms of the efficiency of two different extracts (simple and saline), according to data of experiments carried out in triplicate for each dye and their mixtures, it can be concluded that the saline extract is slightly more efficient than the simple extract; this difference is minimal as shown on the figure 27, however this is attributed to the increase of positive charges that act directly on the destabilization of the negatives colloids which are in this case azo dyes.

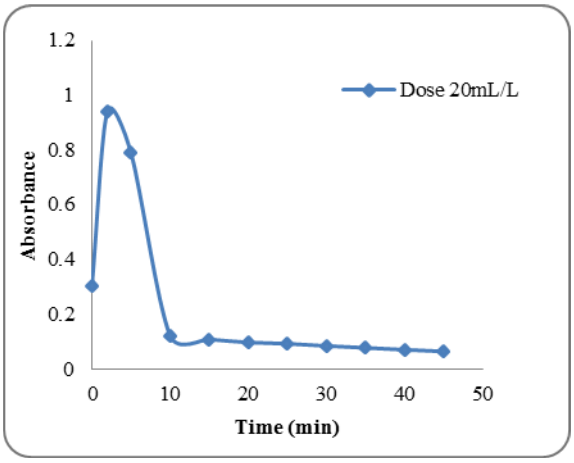

Figure 21.Decolorationprocess of a textile effluent with 20mL/L coagulant dosage

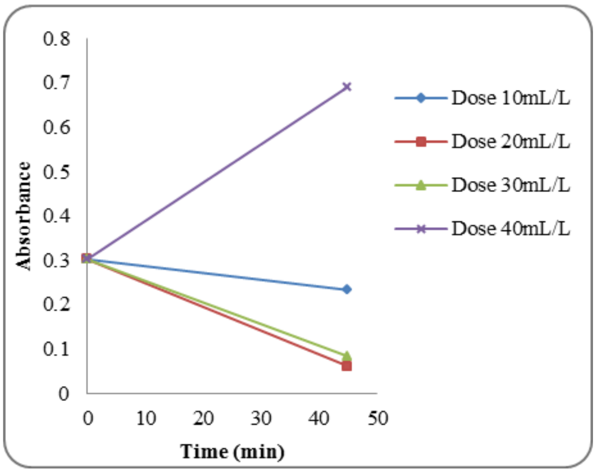

Figure 22.Decolorationprocess of a textile effluent with different coagulant dosage

Figure 23. Textile wastewater sample without treatment

Figure 24. Textile wastewater sample five minutes after the addition of coagulant

Figure 25.Textile wastewater with coagulant and its sedimentation after 45 minutes

Figure 26.Decolorationprocess of textile wastewater with 20 mL/L coagulant dosage

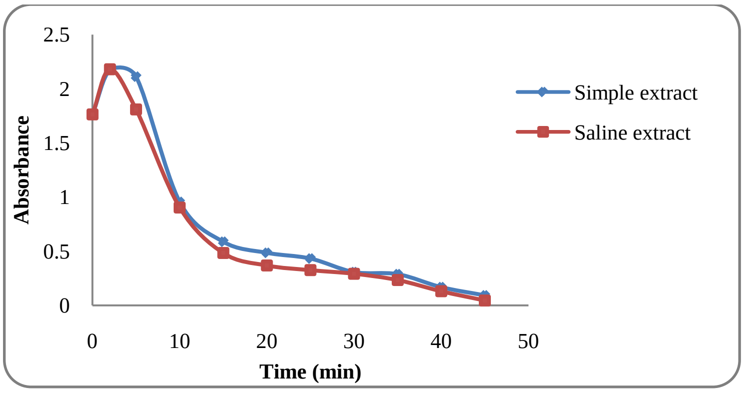

Figure 27.Decolorationprocess with simple and saline extract.

4. Conclusions

In general, it can be concluded that the use of *Moringaoleifera* seed extract as natural coagulant for the treatment of textile wastewater is technical, economic and environmentally viable. The methodology for the elaboration of coagulants extracts is very simple and economical; it does not imply the use of reagents or complex equipment. The process is highly efficient for azo dyes because it can remove up to 95% of the visible color in a period of time that varies between 30 to 45 minutes. According with studies performed for direct, acid and reactive azo dyes, the optimum coagulant dosage ranges is between 20 and 25ml/L. The flocculant capacity of the proteins contained in the seed could be comparable with a synthetic cationic polymer coagulant and its activity is based on the selective adsorption of suspended material on the surface of the molecule, due to its high density of positive charge. The results allow us to infer that the coagulation with *Moringaoleifera* seed extracts are not controlled by electrostatic forces, but by adsorption and formation of bridges. This enables to suggest their direct application in a wastewater treatment plant, without the need to adjust the pH of the water during the process.

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