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Otimização do uso de coagulantes naturais no tratamento de efluentes usando Delineamento Composto Central Rotacional

Optimization of the use of natural coagulants in wastewater treatment using Rotational Central Composite Design

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Resumo

A contaminação dos recursos hídricos por efluentes industriais representa um grande problema ambiental e, no Brasil, a indústria de laticínios é uma das grandes causadoras desses problemas. A composição de seu efluente possui elevada quantidade de matéria orgânica e nutrientes e, portanto, faz-se necessário tratá-los antes do descarte. Este estudo teve como objetivo otimizar o tratamento de efluentes sintéticos de laticínios, simulando o processo de coagulação/floculação, por meio da aplicação de coagulante natural a base de extrato de semente de Moringa oleífera, utilizando o Delineamento Composto Central Rotacional associado à Metodologia de Superfície de Resposta (MSR). As variáveis independentes no projeto foram os tempos de mistura rápida e lenta, a dosagem de coagulante e a concentração de poluentes. Estabeleceu-se um modelo matemático com R^2 de 0,6, no qual ficou comprovada a significância da concentração do poluente e da dosagem do coagulante. A partir da re-parametrização do modelo matemático, considerando apenas essas duas variáveis, obteve-se um ajuste de 74%. Como resultado foi obtida uma eficiência de remoção de 94,9% de turbidez, demonstrando – na primeira análise de aproximação – um resultado satisfatório para o uso de um coagulante natural para remover a turbidez de efluentes de laticínios.

Palavras-Chave: Efluentes de laticínios, Metodologia de Superfície de Resposta, Moringa oleífera.

Abstract

Contamination of water resources by industrial effluents represents a major environmental problem and, in Brazil, the dairy industry is one of the main causes of these problems. The composition of its effluent has a high amount of organic matter and nutrients and, therefore, it is necessary to treat them before disposal. This study aimed to optimize the treatment of synthetic dairy effluents, simulating the coagulation/flocculation process, through the application of a natural coagulant based on Moringa oleifera seed extract, using the Rotational Central Composite Design associated with the Surface Methodology response (MSR). The independent variables in the project were the fast and slow mixing times, the coagulant dosage and the concentration of pollutants. A mathematical model was established with an R^2 *of 0.6, in which the significance of the pollutant concentration and coagulant dosage was proven. From the re-parameterization of the mathematical model, considering only these two variables, an adjustment of 74% was obtained. As a result, a removal efficiency of 94.9% of turbidity was obtained, demonstrating – in the first approximation analysis – a strong result for the use of a natural coagulant to remove turbidity from dairy effluents.*

Key-Words: *Dairy wastewater, Moringa oleifera, Response Surface Methodology.*

1. Introduction

Regarding waste and pollution of available water, preventive measures and awareness-raising on the rational use are urgent. Given the increased frequency of water crises, questions arise about the assumptions, agents, and operation mechanisms of the water system, and the possible causes of the degradation (Pavão and Nascimento, 2019).

Due to the continuous growth of the food industry the waste increased, especially regarding effluents from dairy products, which significantly change water quality. This factor requires for treatment prior to disposal in treatment plants or wasteways, considering the amount of water used during processing and equipment cleaning (Shi *et al*., 2021; Custódio *et al*., 2022).

The liquid effluents generated in the production process and dairies vary according to the industry, and the main pollutants are high contents of organic matter, fats and suspended solids; and organic matter with high concentrations of eutrophication nutrients (Ercin *et al*., 2021; Flach *et al*., 2021). Given the composition of this type of effluent, some conventional treatments result in high sludge volumes, point pollutants such as dyes, or low sedimentation compounds (Cammarota and Freire, 2016).

In the dairy industry, wastewater treatment techniques are generally associated with traditional processes that combine physical (or physicochemical) and biological treatment, ranging from stabilization ponds to aerobic and anaerobic treatments, flocculation, and coagulation treatments (Joshiba *et al.*, 2019).

Concerning chemical coagulants, Tie *et al.* (2015) highlight that they are expensive in most studies. Moreover, sludge volumes, gross pH alterations, and the release of non-biodegradable residues in the water, make the use of this type of coagulant inadequate for the treatment of effluents since they require a subsequent process to correct the potability parameters (Ndabigensere and Narasiah, 1998; Francisco, 2015). Although chemical coagulants, such as aluminum polychloride (PAC), aluminum sulfate and ferric chloride have been widely studied since it exhibits better efficiency than other coagulants for color and turbidity removal and has smaller temperature and pH dependence (Srivastava *et al.*, 2005; Pisoi, 2011), mainly aluminum sulphate and PAC, one of the major problems of these conventional products is the residual aluminum content present in water after treatment, which has been linked to Alzheimer's disease (Bongiovani *et al.*, 2015).

On the other hand, the use of natural coagulants is one of the treatment alternatives. The main advantages of this type of treatment are the absence of secondary pollution, for example non-biodegradable waste, abundant availability, the low cost and multifunctional behavior (Katayon *et al*., 2006; Bhuptawat *et al*., 2007; Tukki *et al*., 2016). Among the natural coagulants, *Moringa oleifera* (MO) stands out, and it is widely used for water treatment due to the presence of water-soluble cationic proteins (Bhatia *et al*., 2007).

The best advantages of Moringa oleifera are: availability and low cost of production, possibility of using its seeds and shells, and, later, of using the sludge generated by the treatment, since it is biodegradable and can be used as a fertilizer (Alves *et al*., 2010; Keerthi *et al*., 2022; Reddy *et al*., 2010; Vieira *et al.*, 2010). Because of its characteristics MO has been widely used in different types of water treatment alone or combined with other types of coagulants or process to remove turbidity (Lo Monaco *et al*., 2010; De Paula *et al*., 2014; Muniz *et al*., 2015; De Paula *et al*., 2016; Mateus *et al*., 2017; De Paula *et al*., 2018; Valverde *et al*., 2018; Ribeiro *et al*., 2019; Assunção *et al*., 2020).

During the coagulation process in Water Works, the importance of considering the coagulant dosage (chemical or natural) and the kinetic energy applied to the treatments (mixing speed and times) is emphasized (El-Gohary and Tawfik, 2009; Cangela and Benetti,2018; Vaz, 2009).

Therefore, seeking to use the least number of trials, the Rotational Central Composite Design (RCCD) is one of the methods used for optimizing the aforementioned factors, associated with the Response Surface Methodology (RSM), which allows to analyze the parameters simultaneously (Gonçalves *et al*. 2020).

To interrelate one or more responses, dependent variables with numerous factors the independent variables, a group of statistical and mathematical procedures composes the planning performed with Rotational Central Composite Design (RCCD), (Mattietto and Matta, 2012). On the other hand, the Response Surface Methodology is a mixture of mathematical techniques, which refines the adjusted model with the relevant factors to optimize an experiment (Montgomery and Runger, 2012). This methodology has been used in studies for water treatments (Cangela and Bennetti, 2017; De Paula *et al*., 2018; Gonçalves *et al*., 2020).

Considering the lack of studies regarding the optimization of wastewater treatment in food industries with the use of natural coagulants, it is proposed to evaluate the use of *Moringa oleifera* coagulant for the dairy wastewater, and optimize the main factors of mixing times, coagulant dosage and pollutant concentration, by using the Rotational Central Composite Design, associated with the Response Surface Methodology.

2. Materials and Methods

The five steps of the methodology are: experimental design, synthetic water preparing, jar test experiments, obtaining the response surface and equating the surface.

2.1. Synthetic water preparation

The jar test equipment — Millan brand, model JT303M — was used to execute the tests. To equalize the composition of the samples, minimizing the wastewater composition variables, the methodology proposed by Tchamango et al. (2010), Kushwaha et al. (2010a) and Kushwaha et al. (2010b), was followed, using powered milk (Componesa brand, manufactured by Embaré Industrias Alimentícias S.A.), diluted in 1.5L of tap water, at different concentrations, to generate a constant synthetic dairy wastewater (SDW) compositions throughout the experiments. The homogenization occurred during the preparation of the sample, and then it was transferred to the jars.

2.2. Preparation of the natural coagulant

A methodology adapted from Heredia and Sánchez-Martín (2009), Ndabigensere *et al*., (1995) was used to prepare the coagulant solutions of *Moringa oleifera*. Firstly, the seeds were dried in an oven at 40°C for 48 hours, then it was crushed in a blender with 1 mol NaCl solution in the proportion 5% (mass/volume). The triturated mixture stayed under magnetic stirring for 30 minutes to extract the active compound. After finishing the agitation, vacuum filtration was performed. The final product was stored in a refrigerator, and it was used for a maximum period of 7 days, as recommended by Valverde *et al*. (2018). The dosages were taken directly from the filtered mixture.

2.3. Experimental design

To optimize the responses to the study, the Rotational Central Composite Design (RCCD) was performed, applied to the Response Surface Methodology (RSM). The control factors were: the coagulant dosage (dosage), the fast (FMT) and slow (SMT) mixing times, and the pollutant concentrations (concent.) – four independent variables (k=4). As a response variable, the turbidity removal analysis was compared with the initial sample.

Therefore, the composition of the RCCD was: 16 factorial points (2k), eight axial points (2k) and four replicates at the center point, totalizing 28 experiments. The axial spacing is given by (2k)1/4, which in this case equals two. Table 1 shows the minimum and maximum factorial levels (−1; +1), the center (0), and the minimum and maximum axial points (−2, +2).

To define the concentration levels, the studies of Tchamango *et al*. (2010), Kushwaha *et al*. (2010a) and Kushwaha *et al*. (2010b) was used as basis, with the minimum level 1 g. L^{-1} and the maximum 3 g. L^{-1}. Thus, the milk powder portions were applied by diluting them in 1.5 liters of water, simulating the dairy effluent (SDW). The mixing was carried out until homogenization.

For the coagulant dosage, studies by Bhatia *et al.* (2007) were followed, with modifications in the spacing of the levels: 10 mL minimum and 30 mL maximum.

Based on the studies by Paula (2014), the mixing times were set as: minimum of 10 minutes for slow mixing and 1 minute for fast mixing, and maximum of 30 minutes and 3 minutes, for slow and fast mixing, respectively. Thus, fast rotation was set at 100 rpm and slow rotation at 40 rpm. The sedimentation time was set at 60 minutes.

Once the levels of the control factors were defined, the central composite design for the experiment could be assembled and used as a reference.

3. Results and discussion

According to RCCD definitions, we initiated the jar test by applying the levels in each test. The initial and final turbidity of each sample were measured for their removal analyses, as shown in Table 1.

Table 1. Jar Test Responses.

Factorial	**Run order**	**RCCD**				**Initial Turbidity**	**Responses**	
		Concent.	**SMT**	**FMT**	**DOSAGES**		**Final Turbidity**	**Removal (%)**
		X1	X2	X3	X4			
Complete factorial	1	1.5	15	1.5	15	486	59.10	87.84
	2	2.5	15	1.5	15	987	253.00	74.37
	3	1.5	25	1.5	15	493	42.00	91.48
	4	2.5	25	1.5	15	1000	391.00	60.90
	5	1.5	15	2.5	15	565	75.90	86.57
	6	2.5	15	2.5	15	1000	335.00	66.50
	7	1.5	25	2.5	15	642	82.20	87.20
	8	2.5	25	2.5	15	942	578.00	38.64
	9	1.5	15	1.5	25	522	64.40	87.66
	10	2.5	15	1.5	25	917	60.90	93.36
	11	1.5	25	1.5	25	543	57.20	89.47
	12	2.5	25	1.5	25	712	56.10	92.12
	13	1.5	15	2.5	25	509	51.40	89.90
	14	2.5	15	2.5	25	937	75.50	91.94
	15	1.5	25	2.5	25	581	31.90	94.51
	16	2.5	25	2.5	25	1000	90.20	90.98
Central Points	17	2	20	2	20	902	95.60	89.40
	18	2	20	2	20	839	71.50	91.48
	19	2	20	2	20	902	99.10	89.01
	20	2	20	2	20	755	84.90	88.75

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Table 1. Jar Test Responses (continue).

Factorial	Run order	RCCD				Initial Turbidity	Responses	
		Concent.	SMT	FMT	DOSAGES		Final Turbidity	Removal (%)
		X1	X2	X3	X4			
Axial Points	21	1	20	2	20	397	82.10	79.32
	22	3	20	2	20	1000	755.00	24.50
	23	2	10	2	20	725	91.50	87.38
	24	2	30	2	20	712	105.00	85.25
	25	2	20	1	20	706	85.20	87.93
	26	2	20	3	20	906	91.90	89.86
	27	2	20	2	10	940	995.00	−5.85
	28	2	20	2	30	788	40.20	94.90

In many assays, we observed more than 80% of turbidity removal and the maximum value of 94.9% of removal (assay 28), excepting the value obtained in the assay 27, where turbidity increased. Assunção *et al*. (2020) obtained a turbidity reduction of 87.9% for Synthetic effluents with turbidity of 240, treated with 80 mg.L^{-1} MO extract, whereas Mateus *et al*. (2017) obtained mean of 96% removal efficiency in the treatment with *Moringa oleifera* (MO) followed by the microfiltration (MF) and nanofiltration (NF) process in dairy wastewater treatment. It is important to highlight that in none of these cited works an optimization methodology was applied.

The number in assay 27 is a consequence of the low dosage of the coagulant that causes very small and low specific weight flocs, impairing its sedimentation. One of the important factors in this process is the size and specific weight of the particles generated in the previous step since they depend on gravity to decant. When analyzing the suspended materials, the turbidity reading may increase due to non-decanted flocs, which is an important factor.

Given the results, the analysis could be made by response surface. First, the linear and quadratic factors of X1 (concentration) and X4 (dosage) were used. Considering 0.57 as the adjusted R² (R squared) value for the model, we sought to re-parameterize the model, using only significant variables.

The Table 2 presents the coefficients of the model.

Table 2. Analysis of the re-parametric quadratic model

Characteristic	Turbidity removal model analysis				
	Estimated Coefficient	Standard Error	t-value	Pr (>|t|)	Significance
Intersection	94.3585	4.1713	22.6185	0.000	***
X1 (L)	−8.9775	2.6926	−3.3342	0.003	**
X4 (L)	14.0808	2.6926	5.2295	0.000	***
X1:X4	7.4712	3.977	2.2656	0.034	*
X1 (Q)	−7.7516	2.5544	−3.0346	0.006	**
X4 (Q)	−9.5979	2.5544	−3.7574	0.001	**

Legend: (L) Linear Effect; (Q) Quadratic Effect

Therefore, the significance of the interaction between pollutant concentration and dosage of coagulant, and also the quadratic effect increased. The new R² of the model was 0.69, a value considerably better compared to the previously obtained. Thus, we obtained Equation 1:

$$Y(X)=94.3585-8.9775X1+14.0808X4-7.7516X1^2-9.5979X4^2$$

Y(X) = removal of turbidity (%); X1 = initial pollutant concentration (g.L^{-1}); X4 = coagulant dosage (mL).

We used the analysis of variance (ANOVA) (Table 3) to validate the model. The calculated F value (Fcalc) should be greater than the tabulated F value, considering the number of degrees of freedom and the significance level α=0.05(5%).

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The value of Fcalc obtained for the percentage removal of turbidity was 12.41, higher than the tabulated value of 2.80, which indicates the model to be highly significant (p-value less than 0.00001), confirming, therefore, its validation.

Table 3. ANOVA analysis for the model

Source of variation	Sum of Squares	Degree of Freedom	Mean Square	Fcalc
Regression	10187.4	4	5093.7	12.41
Residual	4721.0	23	205.3	
Total	14908.4	27		

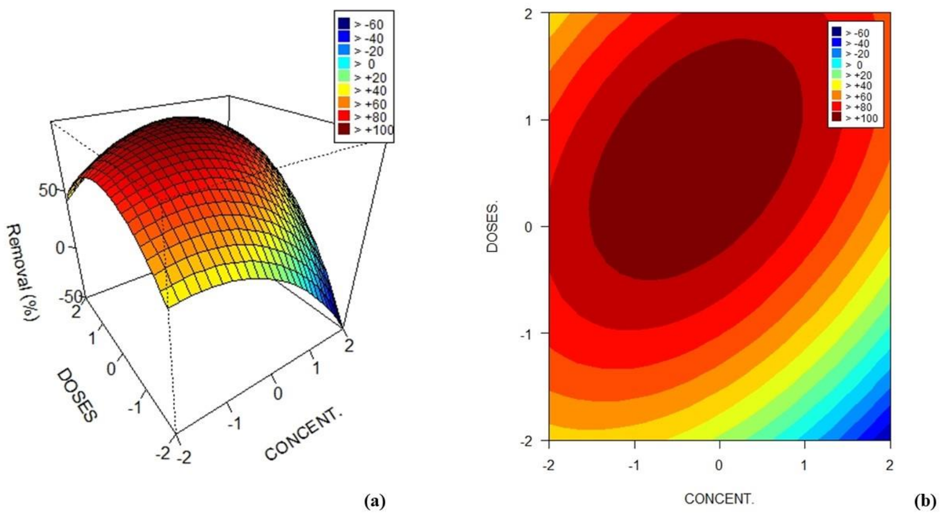
Y(x): $F_{4.23;0.05} = 2.80$; $R^2 = 0.68$, p-value <0.00001

On the Figure 1(a) is shown an increasing removal of turbidity at lower milk power concentrations and higher dosage. The best points are in the red area of the graph.

For a better analysis, we can use the contour plot from Figure 1(b) to determine the optimal point of the variables.

The optimum point is in the darker region. Thus, by using the R software (R Foundation, 2020) we determined that the optimal point is represented by the concentration of pollutants equal to 1.86 g.L^{-1} for a dosage of 23.13 mL of coagulant.

Figure 1. Response Surface and Contour plot of the model



(a) (b)

In: (a) Response Surface; (b) Contour plot

4. Conclusion

The coagulation and flocculation process is still the most widely used for economic and practical reasons. Due to the negative factors arising from using chemical coagulants, such as the high cost and the chemical residues left during the treatment process, the use of natural coagulants, for example Moringa oleifera seeds, presents itself as an innovative alternative.

This study evaluates the use of *Moringa oleifera* coagulant for the dairy wastewater and optimize the treatment by using the Rotational Central Composite Design, associated with the Response Surface Methodology. It was obtained as satisfactory result of first approximation. The coagulant used was efficient in removing turbidity from synthetic dairy wastewaters, with a removal value of 94.90%. Despite high removal values, the turbidity values of the samples remained high (between 30 and 800 uT), requiring further treatment after the coagulation process. Nevertheless, this study enabled the elaboration of the model for treating these effluents, based on synthetic water.

The adoption of optimization processes can reduce the use of natural coagulants in the treatment of effluents, as well as increase its efficiency, reducing its cost.

As future recommendations, we suggest using the model developed based on synthetic wastewater on real wastewaters from dairy products, refining the model and including analysis for other parameters, such as the removal of nutrients and organic matter.

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