

Research Article

Evaluation of Biocoagulant “*Opuntia ficus-indica* and Chitosan” to Improve the Removal of Contaminants in Dairy Effluents through Electrocoagulation

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The dairy industry is classified, worldwide, as one of the main wastewater-generating industries. The objective of the study was to evaluate the effect of parameters on the percentage of turbidity (TB) removal and chemical oxygen demand (COD), using as pretreatment a combined coagulation process using a biocoagulant composed of chitosan and *Opuntia ficus-indica* mucilage (OFI): chitosan-mucilage (Q-M from 1 to 9), and an electrocoagulation process with operational variables of biocoagulant dose (0.025, 0.05 y 0.1 g/L), current density (30, 40 y 50 mA/cm²), and time (10, 15 y 20 minutes). A monopolar glass reactor with aluminum electrodes was assembled by applying the response surface approach methodology. Additionally, using the Box–Behnken mathematical model for the removal of TB and COD, the effects and interactions of the factors and experimental design levels (considering control formulations using triplicates) were evaluated. An analysis of variance was performed to evaluate model responses and optimal conditions. The experimental results showed that the formulation of the compound biocoagulant Q-M-5 in doses of 0.99 g/L achieves 98.44% turbidity removal and 81.72% chemical oxygen demand removal. The dose of the compound biocoagulant has a significant positive effect as does the current density of 30 mA/cm² and time of 19.05 min; that is, its efficiency in removing turbidity and chemical oxygen demand increases when using these parameters. It is concluded that the use of the biocoagulant composed of chitosan and *Opuntia ficus-indica* mucilage (Q-M-5) as the primary treatment of whey wastewater provides evidence for the literature, showing that it is also a low-cost, ecological, and easy-to-apply natural coagulant.

1. Introduction

The removal of contaminants in wastewater is presented as a significant environmental challenge, driven by the growing demand for water. This situation is continually worsening due to the insufficiency of primary sources of this resource, a direct consequence of the rapid growth of the world's population, industries, and increasingly extensive needs [1].

Several studies have been carried out that provide data about procedures to eliminate organic waste, such as chemical, physical, and biological processes, ranging from the identification of indigenous organisms capable of degrading organic matter as a biological method; membrane filtration, adsorption, and coagulation/flocculation as physical methods; and ozonation and advanced oxidation of organic matter as chemical methods [2]. The reuse of this waste is

even applied due to its high organic load, using it as fertilizer and thus giving it a utility [3]. Electrocoagulation (EC), as a physical and chemical treatment, is an effective and promising technique for the elimination of contaminants in wastewater since it removes suspended solids and turbidity [4]. Electrocoagulation as a tool in the removal of waste in contaminated waters is a widely used technique. This electrolytic process requires the application of current to an anode made of materials such as aluminum, iron, copper, or other elements that allow the operation of a reactor; here, it produces the dissolution of the material and forms metal ions (Al^{+3}), active precursors of coagulants; this response enables the interaction with the cathode, which responds by reacting with hydroxyl ions, thus promoting the formation of flocs [5]. The use of electrocoagulation is an efficient method, with various configurations available using materials such as platinum or gold, which are excellent for electrochemical tests. However using materials like aluminum is crucial for establishing a lower-cost and more accessible configuration. Aluminum is much more readily available than other materials and also effectively functions as an electrode [6]. In the search for more accessible and abundant materials, the electrochemical flocculation technique aims to improve by using natural biocoagulants. These organic complexes have gained interest in the last decade because they are less toxic and more eco-friendly compared to conventional chemical coagulants [7, 8]. Coagulants of natural origin have attractive advantages such as renewability, “raw material is easily obtained,” nontoxicity and relative cost-effectiveness, “sludge management and coagulant cost,” unlikely to produce treated water with extreme pH, and highly biodegradable [9, 10]. Natural coagulants can be obtained from different natural sources, such as plants, seeds, marine crustaceans, and biomasses of shellfish and microbial organisms, with natural coagulants of plant origin being the most researched [11]. *Opuntia ficus-indica* (OFI), also known as nopal of the Cactaceae family, is distributed from western Canada to the tip of South America [12]. Chitosan, a biopolymer derived from chitin, has been widely studied for its effectiveness in removing contaminants present in wastewater through electrocoagulation [13, 14], being able to effectively adsorb heavy metals and other contaminants due to its positive charge and the presence of amino groups, which bind to heavy metal ions [14]. The fruits and leaves or cladodes of OFI are commonly consumed as food and are in great demand in various countries; this causes the generation of a large amount of waste [15]. The biocoagulant is extracted from the OFI cladodes and used in liquid suspension or dry matter in wastewater treatment [16]. In the literature, some studies are reported that use biocoagulants such as OFI for the treatment of wastewater [16–21] however, as far as we know, there are still more studies aimed at the use of this biocoagulant in the process combined with electrocoagulation, in addition to the impacts caused by factors such as current density, coagulant dose, and time, which are some of the most important parameters, important that have not been sufficiently reported in the joint investigations to obtain an optimization of the operating parameters [22]. Some methods studied

have peculiarities and can show some advantages and disadvantages in the operational and economic part when taking them to industrial scales. Among the existing methods, electrocoagulation allows to efficiently reduce COD and eliminate other contaminants present in wastewater [23]; likewise, Pacheco et al. [24] used the desirability function to optimize the process at a pH of 10.0, a biocoagulant dose of $4.4 \text{ g}\cdot\text{L}^{-1}$, and a current density of $31.5 \text{ mA}\cdot\text{cm}^{-2}$, showing turbidity removal and COD of 98.0% and 83.8%, respectively. Therefore, wastewater from the dairy industry represents a great threat to the environment, and it is necessary that they be subjected to efficient, low-cost, and easy-to-operate treatment at an industrial level. With the objective of improving the treatment of cheese whey wastewater, this study focused on evaluating and optimizing efficiency through the removal of COD and turbidity (TB). A combined coagulation process was implemented as pretreatment, using different doses of biocoagulant extracted from *Opuntia ficus-indica* in conjunction with commercial chitosan and electrocoagulation with variation in the doses of the compound biocoagulant, current density, and time. The use of aluminum electrodes allowed us to analyze the effect of these operating parameters and their interactions. In this context, a response surface design using the Box–Behnken model was applied to determine all the factors that affect the electrocoagulation process combined with the composite biocoagulant [6, 25]. It is important to highlight that, to date, there have been no previous studies investigating the use of a biocoagulant composed of *Opuntia ficus-indica* and chitosan in the treatment of effluents from the dairy industry. This research gap highlights the novelty and relevance of the present study to the field, providing a valuable contribution to the understanding and optimization of dairy industry wastewater treatment processes.

2. Materials and Methods

2.1. Source and Characterization of Cheese Whey Wastewater.

The residual whey effluent samples were obtained from a dairy industry located in Majes, Arequipa-Peru, which processes dairy products such as cheese and yogurt of different types. The sample was taken from a tank that stores cheese whey after the milk curdling process. The characteristics of textile wastewater are shown in Table 1. The residual whey effluent contains a high level of COD due to the organic load and due to the presence of proteins, mineral salts, lipids, and lactose, among others [26].

2.2. Characterization by Fourier Transform Infrared Spectroscopy (FTIR) and Infrared Microscopy.

The samples of chitosan biocoagulant particles (Sigma-Aldrich), cactus mucilage (*Opuntia ficus-indica*), and formulations Q-M-5 were evaluated using a Thermo Scientific brand Nicolet Summit with the KBr pellet method; all samples in the powder were mixed separately with KBr, forming tablets to track the possible interactions between the compounds and the applied ingredients, in the spectrum ranging within the

TABLE 1: Cheese whey wastewater characteristics.

Parameter	Unit	Value
Chemical oxygen demand (COD)	mg/L	40800
Turbidity (TB)	NTU	1073.75

range of 4000–500 cm^{-1} . Likewise, the samples were passed through the Thermo Scientific iN10 Infrared Microscopy (μFTIR) equipment to obtain micrographs of the bio-coagulant composed of *Opuntia ficus-indica* and chitosan.

2.3. *Opuntia ficus-indica* Biocoagulant Extraction. The cladodes of *Opuntia ficus-indica* were washed with water, brushed to remove the spines, peeled to remove as little pulp as possible, then cut into 2-cm pieces, and added to a beaker with distilled water in the proportion 1 : 4 (v/v) [27–29], and the beaker was left to macerate for 24 hours at room temperature. The extract was filtered through a nylon filter mesh with a porosity of 100 μm . The mucilage was precipitated with ethanol (95%, v/v) at a ratio of 1 : 3 (v/v). 95% ethanol extraction was used to dissolve the chlorophyll present in the pads and produce a white mucilage powder [27, 28]. The precipitate was washed three times with ethanol [28] and then dried by evaporation of the solvent in an oven for approximately 24 hours at 50°C, obtaining mucilage crystals, which were stored refrigerated at 4°C.

2.4. Preparation of Biocoagulant Composed of Chitosan and *Opuntia ficus-indica* Q-M. To obtain a biocoagulant composed of chitosan and *Opuntia ficus-indica* (Q-M), we began with the dilution of commercial Sigma-Aldrich chitosan (1% w/v) in 1% (v/v) acetic acid and mucilage of *Opuntia ficus-indica* (0.5% w/v) in distilled water, which were mixed in different proportions according to the formulations shown in Table 2.

To obtain the Q-M compound biocoagulant, the chitosan solution (1% w/v) mixed with *Opuntia ficus-indica* mucilage (0.5% w/v) was used in its different formulations; for this, it was important to add the mucilage solution drop by drop with the help of a burette to avoid precipitation. Then, the mixture was homogenized with a magnetic stirrer at 150 rpm at 60°C for 3 hours [30, 31]. The solution was poured into silicone molds with a circular bottom of 2.5 cm in diameter, and the mixture was spread and left in an oven for 4 hours at 40°C, observing that the solvent evaporated completely [32].

The proportion of chitosan and the biocoagulant of *Opuntia ficus-indica* was varied from the range of 0 to 100% to investigate the optimal combination of chitosan and the biocoagulant of *Opuntia ficus-indica* for the best removal percentage of turbidity and COD. The experiments were performed as described by Chong & Kiew [33] with slight modifications. Different combinations of proportions of chitosan and biocoagulant from *Opuntia ficus-indica* (10/90, 20/80, 30/70, 40/60, 50/50, 60/40, 70/30, 80/20, and 90/10) were investigated. The final turbidity removal percentages obtained in all combinations were compared with the 100% dose of chitosan and *Opuntia ficus-indica* biocoagulant.

TABLE 2: Formulation of the films composed of chitosan and mucilage from *Opuntia ficus-indica*.

Label	Chitosan (%)	Mucilage <i>Opuntia ficus-indica</i> (%)
Q	0	100
M	100	0
Q-M-1	10	90
Q-M-2	20	80
Q-M-3	30	70
Q-M-4	40	60
Q-M-5	50	50
Q-M-6	60	40
Q-M-7	70	30
Q-M-8	80	20
Q-M-9	90	10

2.5. Pretreatment of ARSQ with Biocoagulant Compound Chitosan and Mucilage of *Opuntia ficus-indica* Q-M. The biocoagulation process as pretreatment was carried out in a jar equipment with an automatic controller that allows setting the speed and mixing time. 40 mL of residual whey effluent was placed in each beaker using the coagulation procedure parameters that involved 1 hour of mixing at 250 RPM and 60 min of sedimentation. The doses of the Q-M biocoagulant compound to be used were 0.025, 0.05, 0.1, 0.2, and 0.4 g/L (see Figure 1).

2.6. Electrocoagulation (EC) Process

2.6.1. Cell Preparation. The treatment of dairy effluent by EC was carried out in a 0.1-L monopolar glass batch reactor. Aluminum electrodes were used as the cathode and anode: the electrodes had effective dimensions of 5 × 2.5 cm, and they were placed 1 cm apart and connected in the monopolar parallel connection mode.

2.6.2. Electrode Preparation. Before each experiment, the electrodes were washed with a mixture prepared from aqueous solutions of 100 mL of HCl (36.5%) and 200 mL of hexamethylenetetramine (2.8%) to remove oxide and passivation layers from the electrodes [34].

2.6.3. Configuration and Start-up. Each experiment was performed with 0.05 L of the sample under constant stirring at 100 rpm. The current density was adjusted according to the experimental design (30, 40, and 50 mA/cm^2) using a CC power supply (MESTEK DP3030) operated in a galvanic mode according to the experimental design (see Figure 1). All experiments were performed at room temperature and in triplicate.

2.7. Analytical Methods. To determine the effect of EC on the treatment of cheese whey wastewater, samples were taken at the end of each experiment and the following parameters were analyzed: turbidity (TB) and COD. COD values were determined by the closed reflux dichromate colorimetric method at 600 nm according to standard methods for wastewater analysis [35] using a HACH DR900 colorimeter, and turbidity was measured using a turbidimeter (HANNA

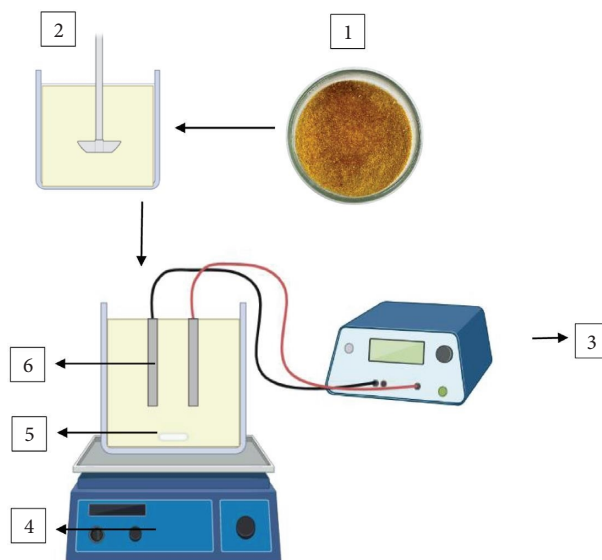


FIGURE 1: Experimental system. (1) Q-M-5 composite biocoagulant, (2) pretreatment with Q-M-5, (3) power supply, (4) stirring plate, (5) magnetic bar, and (6) aluminum electrodes.

HI93703). TB and COD removal efficiencies were calculated using the following equation:

$$RE = \frac{(C_o - C)}{C_o} \times 100, \quad (1)$$

where C_o and C are the initial and final concentrations of the contaminants, respectively.

2.8. Experimental Design and Data Analysis. A response surface model based on the Box–Behnken design (BBD) was used to study the effects of the coagulant dose (X_1), current density (X_2), time (X_3), and the interactions of the factors on the removal of turbidity (TB) and COD. The developed model was also used to optimize the parameters of the TB and COD removal process so that the Box–Behnken design provides the response percentages of removal for both variables, considering three blocks. Table 3 shows the factors and test levels used for model development.

Three sets of experiments of 15 replicates each were considered, for a total of 45 experiments. The experimental data were fitted to a quadratic model for response surface regression, thereby obtaining the regression coefficients. Considering that the response surface methodology (RSM) is used, a second-order model is used [36],

$$Y = \beta_0 + \sum_i \beta_i X_i + \sum_i \beta_{ii} X_i^2 + \sum_i \sum_j \beta_{ij} X_i X_j, \quad (2)$$

where β_0 is the compensatory term, β_i is the linear effect of input factor X_i , β_{ii} is the quadratic effect of factor X_i , and β_{ij} is the linear-linear interaction effect between factors X_i and X_j . Furthermore, Y represents the response variable: TB removal and COD removal.

The experimental data were analyzed using Statgraphics 19 software. The ANOVA table was used to determine the interactions between the process variables and the response

variables. In addition, three-dimensional curves of the response surfaces were created.

2.9. Optimization Procedure. Optimization of TB and COD removal from the residual whey effluent was performed using the statistical software mentioned above. The software optimization module seeks a combination of factor levels that simultaneously satisfy the requirements imposed on each response and factor, with the goal of maximizing this response. Possibly there are two or maximum responses due to the curvature in the response surfaces and their combination in the desirability function. However, by starting from several points in the design space, the chances of finding the “best” local maximum improve.

The Statgraphics optimization module 19 determines the combination of factor levels that simultaneously satisfy the requirements imposed on each response variable and factor. For the working variables, the optimal values for the coagulant dose, current density, and time are sought so that the removal of TB and COD is maximized.

3. Results and Discussion

3.1. Pretreatment of ARSQ with Biocoagulant Compound Chitosan and Nopal Mucilage (*Opuntia ficus-indica*). Figure 2 shows the effect of chitosan formulations with *Opuntia ficus-indica* mucilage (Q-M) on turbidity removal (TB) (%). The Q-M-5 formulation shows maximum TB removal at a dose of 0.05 g/L; using different proportions of chitosan and *Opuntia ficus-indica* mucilage, TB removal (%) is shown at doses of 0.025, 0.05, and 0.1 g/L. The initial turbidity was recorded as 1073.75 NTU before coagulation. The maximum turbidity removal was 94.60% with 50 : 50 Q-M formulation at a dose of 0.05 g/L of composite particles. As for the other formulation ratios 10 : 90, 20 : 80, 30 : 70, 40 : 60, 60 : 40, 70 : 30, 80 : 20, and 90 : 10, turbidity removal (%) of 91.31, 92.61, 93.08, 93.95, 93.98, 91.21, 88.67, and 86.22%, respectively, was obtained. The results show that a 50 : 50

TABLE 3: Independent variables and levels used by the BBD model.

Factor	Unit	Origin factor	Factor code		
			-1	0	+1
Coagulant dose (CDo)	g/L	X_1	0.025	0.05	0.1
Current density (CD)	mA/cm^2	X_2	30	40	50
Time (T)	min	X_3	10	15	20

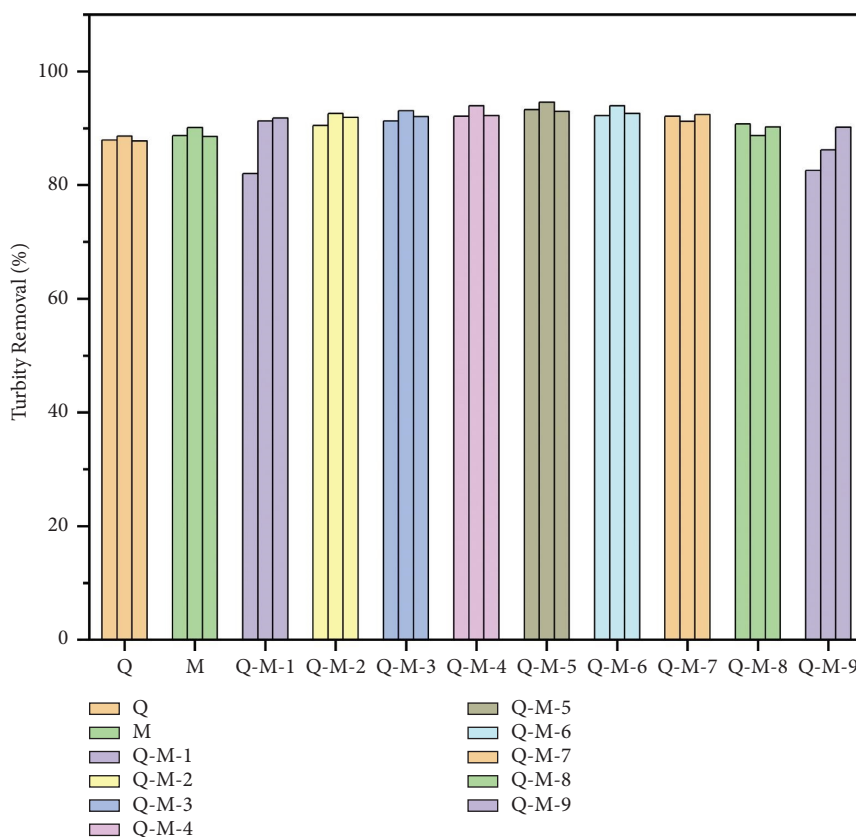


FIGURE 2: Turbidity removal using different proportions of Q-M (1–9) at doses of 0.025 (first bar), 0.05 (second bar), and 0.1 g/L (third bar).

ratio of chitosan:*Opuntia ficus-indica* (Q-M-5) mucilage increased the % turbidity removal.

Subsequently, an ANOVA was carried out where the Q-M-5 formulation (50 : 50) of chitosan:*Opuntia ficus-indica* mucilage was used in different doses of 0.025, 0.05, 0.1, 0.2, and 0.4 g/L, showing that between the blocks studied, there is a significant difference, as shown in Figure 3. The results are that at a dose of 0.05 g/L, a turbidity removal of 94.60% is obtained.

3.2. Characterization by FTIR and Infrared Microscopy of the Q-M-5 Compound. Figure 4 shows the FTIR spectra of nopal mucilage (*Opuntia ficus-indica*) in comparison with the spectrum of the optimal formulation Q-M-5, where the bands are observed between the range of 3500 cm^{-1} and 3200 cm^{-1} ; a sharp peak is presented, which is attributed to the stretching of the O-H bond of multiple hydroxyl groups associated with molecules that are present in both the nopal mucilage sample and the Q-M-5 formulation. As well as the vibration stretching of the N-H group [37]. In both spectrum a band is recorded between the 1600 cm^{-1} and 1700 cm^{-1}

peak, due to the C=O vibration with the stretching of COOH groups, this is attributed to the presence of proteins, water and bound amides [3, 38]; it is observed in the same way in the mucilage spectrum of *Opuntia ficus-indica* as in that of Q-M-5. The stretching of the vibration band at 1100 cm^{-1} to 1350 cm^{-1} is due to the asymmetric stretching of the carboxylic COO double bond and saccharide units, where it is observed more prominently and intensely in the spectrum of the mucilage of *Opuntia ficus-indica* and not in the Q-M-5 [38]. The transmittance values of the peaks from 1200 cm^{-1} to 900 are recognized as carbohydrates.

The vibrational stretching of the peak at 871.468 cm^{-1} is attributed to SO. The peaks in the wavenumber region lower than 700 cm^{-1} could be attributed to the extension of the halogenates [39]; likewise, Basiak et al. [40], Durazzo et al. [41], and Choque-Quispe et al. [37] mentioned that the broadband between 1500 and 800 cm^{-1} is known as the fingerprint of the material, and the functional groups -OH, C-O, -CH₂, and C-O-C predominate, which include molecules of sugars, alcohols, and organic acids.

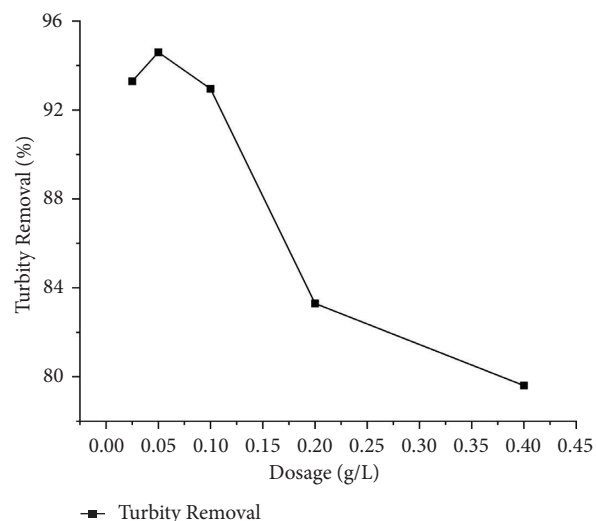


FIGURE 3: Effect of the Q-M-5 dose on turbidity removal.

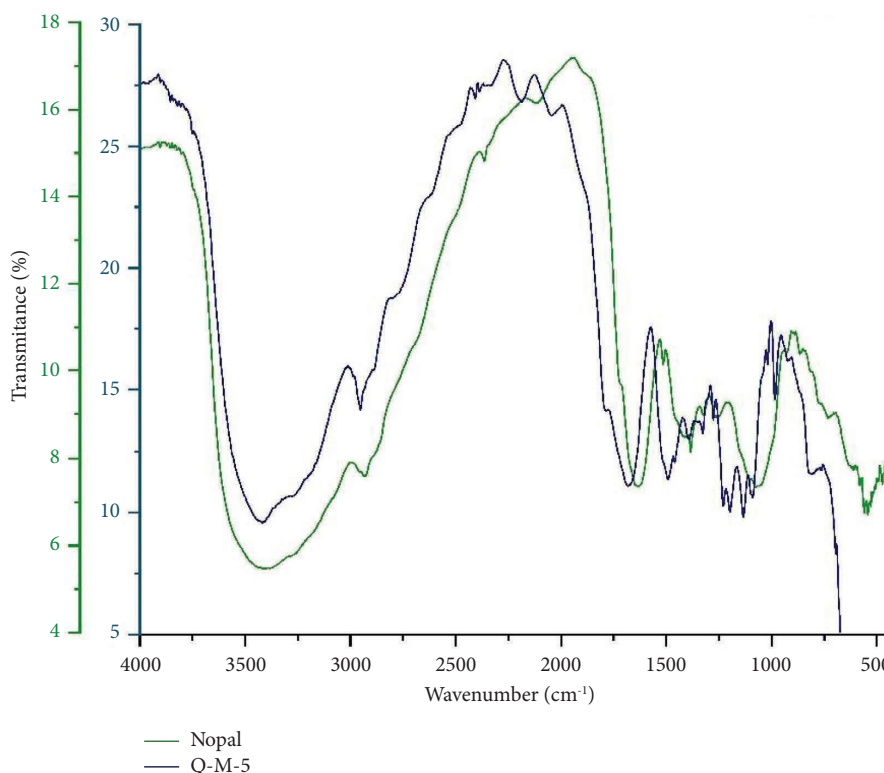


FIGURE 4: FTIR spectrum of the nopal mucilage (*Opuntia ficus-indica*) vs the Q-M-5 mucilage.

The analyses of the Q-M-5 spectrum show characteristic bands; in some works, the bands of isolated chitosan are shown with a broadband approximately between 3200 and 3374 cm^{-1} for the stretching vibration of the OH and NH groups, 1661 cm^{-1} for amide I, 1595 cm^{-1} for amide II, and 1380 cm^{-1} for amide III, as reported by Abdel-Rahman [42] and Kaya [43]. When compared to commercial chitosan, the asymmetric stretching of the C-O-C bridge and the stretching at 1080 cm^{-1} for the -CO bond, as noted by [44], are characteristic of its structure. Benhabiles [45] and Kumari [46] mentioned that

the results obtained provide an important alternative towards the production of chitin and chitosan; this is due to the operating conditions and analysis techniques used, which is why they consider the following important parameters: temperature, concentration, and reaction time in each stage, as well as increasing the range of factors. Our spectrum shows a broad band between 3500 cm^{-1} and 3200 cm^{-1} characteristic of chitosan and stretching that denotes the presence of amides I and II) at 1600 cm^{-1} and 1500 cm^{-1} respectively, keeping some similarities with previous works.

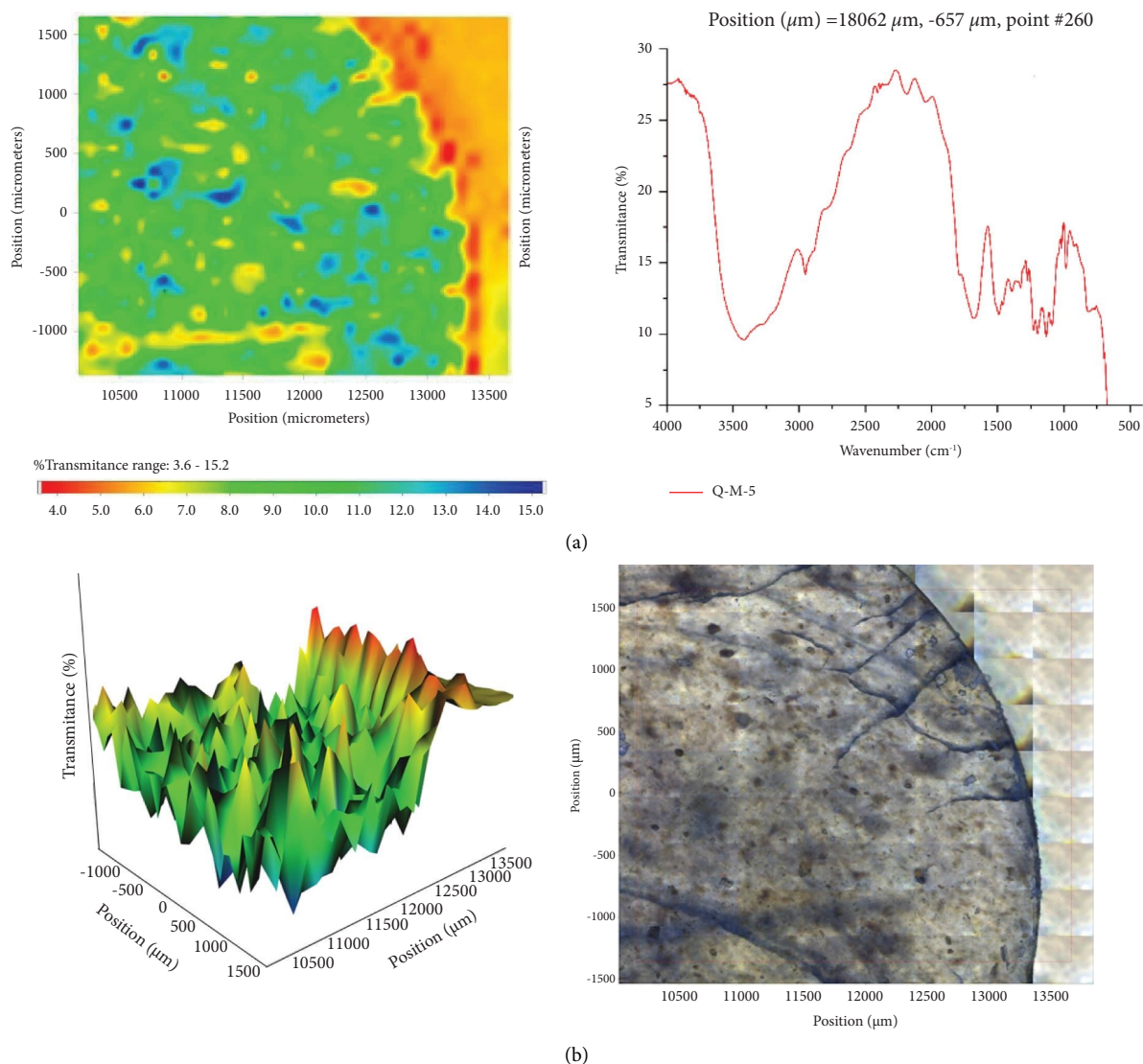


FIGURE 5: FTIR spectrum of compound Q-M-5 (a, b).

TABLE 4: Box–Behnken experimental design and experimental data obtained for the removal of TB and COD from whey wastewater.

Essay	Coagulant dosage (g/L)	Current density (mA/cm^2)	Time (min)	% removal of TB	% removal of COD
1	0.025	30	15	78.33 ± 0.97	72.73 ± 0.45
2	0.025	40	10	76.84 ± 0.54	71.16 ± 0.27
3	0.025	40	20	71.86 ± 0.24	71.17 ± 0.11
4	0.025	50	15	76.80 ± 0.75	72.33 ± 0.42
5	0.05	30	10	66.12 ± 1.6	59.16 ± 0.37
6	0.05	30	20	69.01 ± 0.11	56.51 ± 0.32
7	0.05	40	15	65.40 ± 0.15	62.85 ± 0.66
8	0.05	40	15	65.94 ± 0.73	63.15 ± 0.38
9	0.05	40	15	66.80 ± 1.21	62.32 ± 0.24
10	0.05	50	10	66.87 ± 0.08	61.79 ± 0.35
11	0.05	50	20	70.76 ± 0.27	58.51 ± 0.14
12	0.1	30	15	94.15 ± 0.94	84.99 ± 0.27
13	0.1	40	10	94.49 ± 0.22	61.41 ± 0.49
14	0.1	40	20	96.68 ± 0.58	71.65 ± 0.34
15	0.1	50	15	83.61 ± 0.54	69.22 ± 0.46

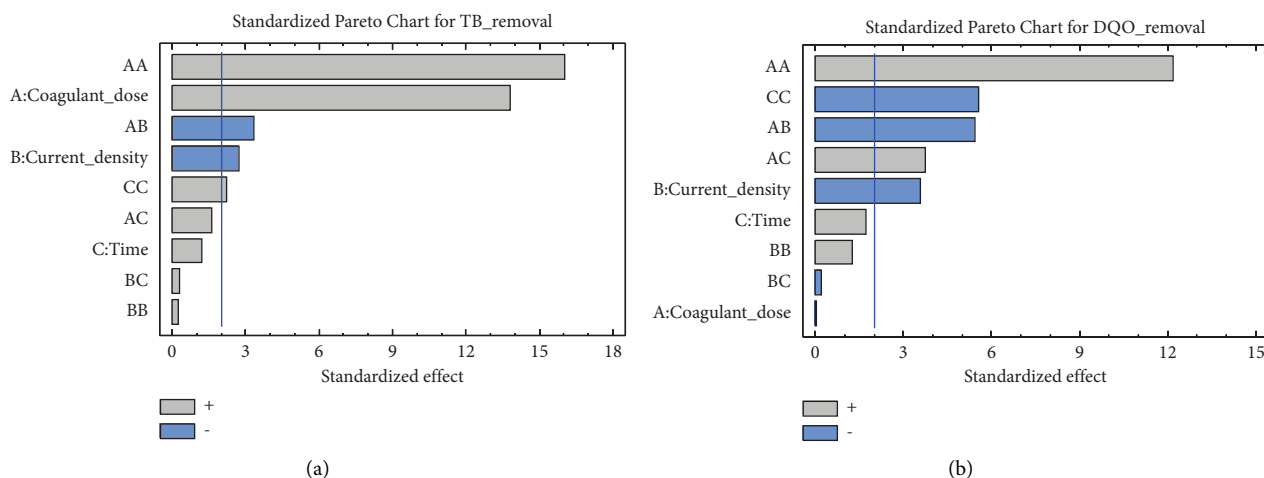


FIGURE 6: Pareto effects that correspond to the regression carried out for removal of (a) TB and (b) COD.

Figures 5(a) and 5(b) show the spectrum obtained by FTIR of the Q-M-5 combination. To obtain micrographs, the OMNIC™ PICTA software was used, and a search was performed in the personalized libraries at 10 random pixels within the mucilage particles of *Opuntia ficus-indica*. The FTIR mapping and micrography (Figures 5(a) and 5(b)) show us a spectral integration from 4000 to 400 cm^{-1} in percentage of transmittance [47].

The heat map determines by colour the transmittance range and the signal intensity by infrared microscopy at each point, showing that the blue areas present in the mapping have a % transmittance of 98.8% in the peak with the most intense signal, while the red areas represent the lowest signals; this area is understood as empty signals, that is, those that do not have a sample.

3.3. Standardized Pareto Effects. Table 4 shows the TB and COD removal values, as well as the removal efficiency observed in each test. The Pareto diagrams (Figures 6(a) and 6(b)) indicate the variables that are significant in the process of removing TB and COD from the whey effluent.

According to Pareto diagrams (Figures 6(a) and 6(b)), the statistical importance of each factor in the removal of TB and COD from the whey effluent can be compared. The dose of the compound biocoagulant is the factor that most influences the removal of contaminants from the whey effluent although all factors are statistically significant ($p \leq 0.05$). Increasing the dose of the compound biocoagulant and time increases the removal of TB and COD from the whey effluent.

The dose of the compound biocoagulant Q-M-5 and the time of the treatment process of the residual whey effluent can have a significant effect on the elimination of TB and COD, as shown statistically in Figure 6.

However, increasing the values of these parameters may have a negative effect on TB and COD removal in the case of residual whey effluent. Although the current density and dose combination of the composite biocoagulant are important, their effect is of minor importance compared to the effect that the dose of the composite biocoagulant has on the removal of TB and COD. Therefore, it is recommended to use a minimum amount of AB, BB, and AC to remove these contaminants from the residual whey effluent, using pre-treatment in the composite biocoagulant Q-M-5 followed by the electrocoagulation process. An excessive dose of the biocoagulant can reverse the surface charge of the colloidal particles, causing dispersion and consequently decreasing the efficiency of the coagulant [48–50]. Optimization of the biocoagulant dose is essential to ensure high efficiency using electrocoagulation in the treatment of waste effluents.

3.4. Regression Models: Generation, Calibration, and Analysis of Variance. The following equations show the generated second-order polynomial models that refer to the removal of TB and ARSQ COD as a function of current density, biocoagulant dose, and pH:

$$\begin{aligned} \text{TB Removal} = & 114,678 - 1026,28 \cdot CDo + 0,0517222 \cdot CD - 2,83003 \cdot T + 11413,1 \cdot CDo^2 \\ & - 7,22667 \cdot CDo \cdot CD + 7,04491 \cdot CDo \cdot T + 0,00199306 \cdot CD^2 + 0,00503333 \cdot CD \cdot T + 0,0777389 \cdot T^2, \end{aligned} \quad (3)$$

$$\begin{aligned} \text{DQO Removal} = & 58,5308 - 864,855 \cdot CDo - 0,301351 \cdot CD + 5,16443 \cdot T + 8762,89 \cdot CDo^2 \\ & - 11,8989 \cdot CDo \cdot CD + 16,3375 \cdot CDo \cdot T + 0,0109292 \cdot CD^2 - 0,0032 \cdot CD \cdot T - 0,194917 \cdot T^2. \end{aligned} \quad (4)$$

TABLE 5: Box–Behnken experimental design and experimental data obtained, including predicted values, for the removal of TB and COD from whey wastewater.

Essay	Coagulant dosage (g/L)	Current density (mA/cm ²)	Time (min)	% removal of TB	% removal of COD	% predicted removal of TB	% predicted removal of COD
1	0.025	30	15	78.33 ± 0.97	72.73 ± 0.45	74.03	72.55
2	0.025	40	10	76.84 ± 0.54	71.16 ± 0.27	77.43	70.88
3	0.025	40	20	71.86 ± 0.24	71.17 ± 0.11	76.23	66.85
4	0.025	50	15	76.80 ± 0.75	72.33 ± 0.42	76.15	77.1
5	0.05	30	10	66.12 ± 1.6	59.16 ± 0.37	68.91	59.5
6	0.05	30	20	69.01 ± 0.11	56.51 ± 0.32	68.96	59.88
7	0.05	40	15	65.40 ± 0.15	62.85 ± 0.66	66.05	62.77
8	0.05	40	15	65.94 ± 0.73	63.15 ± 0.38	66.05	62.77
9	0.05	40	15	66.80 ± 1.21	62.32 ± 0.24	66.05	62.77
10	0.05	50	10	66.87 ± 0.08	61.79 ± 0.35	66.91	58.42
11	0.05	50	20	70.76 ± 0.27	58.51 ± 0.14	67.97	58.16
12	0.1	30	15	94.15 ± 0.94	84.99 ± 0.27	95.72	81.45
13	0.1	40	10	94.49 ± 0.22	61.41 ± 0.49	91.06	64.72
14	0.1	40	20	96.68 ± 0.58	71.65 ± 0.34	95.14	72.95
15	0.1	50	15	83.61 ± 0.54	69.22 ± 0.46	87	68.15

TABLE 6: ANOVA of TB and COD removal.

Source	TB removal			COD removal		
	Sum of squares	<i>p</i> value	Observation	Sum of squares	<i>p</i> value	Observation
X_1 : coagulant dose	1588.770	0.000	S	0.005	0.981	NS
X_2 : current density	62.073	0.010	S	109.053	0.001	S
X_3 : time	11.784	0.243	NS	25.143	0.095	NS
X_1X_1	2144.510	0.000	S	1264.200	0.000	S
X_1X_2	93.025	0.002	S	252.198	0.000	S
X_1X_3	22.101	0.113	NS	118.861	0.001	S
X_2X_2	0.440	0.820	NS	13.231	0.221	NS
X_2X_3	0.760	0.764	NS	0.307	0.850	NS
X_3X_3	41.839	0.032	S	263.025	0.000	S
Total error	274.945			280.339		
Total (corr.)	5237.980			2406.700		

Note. S: significant and NS: not significant.

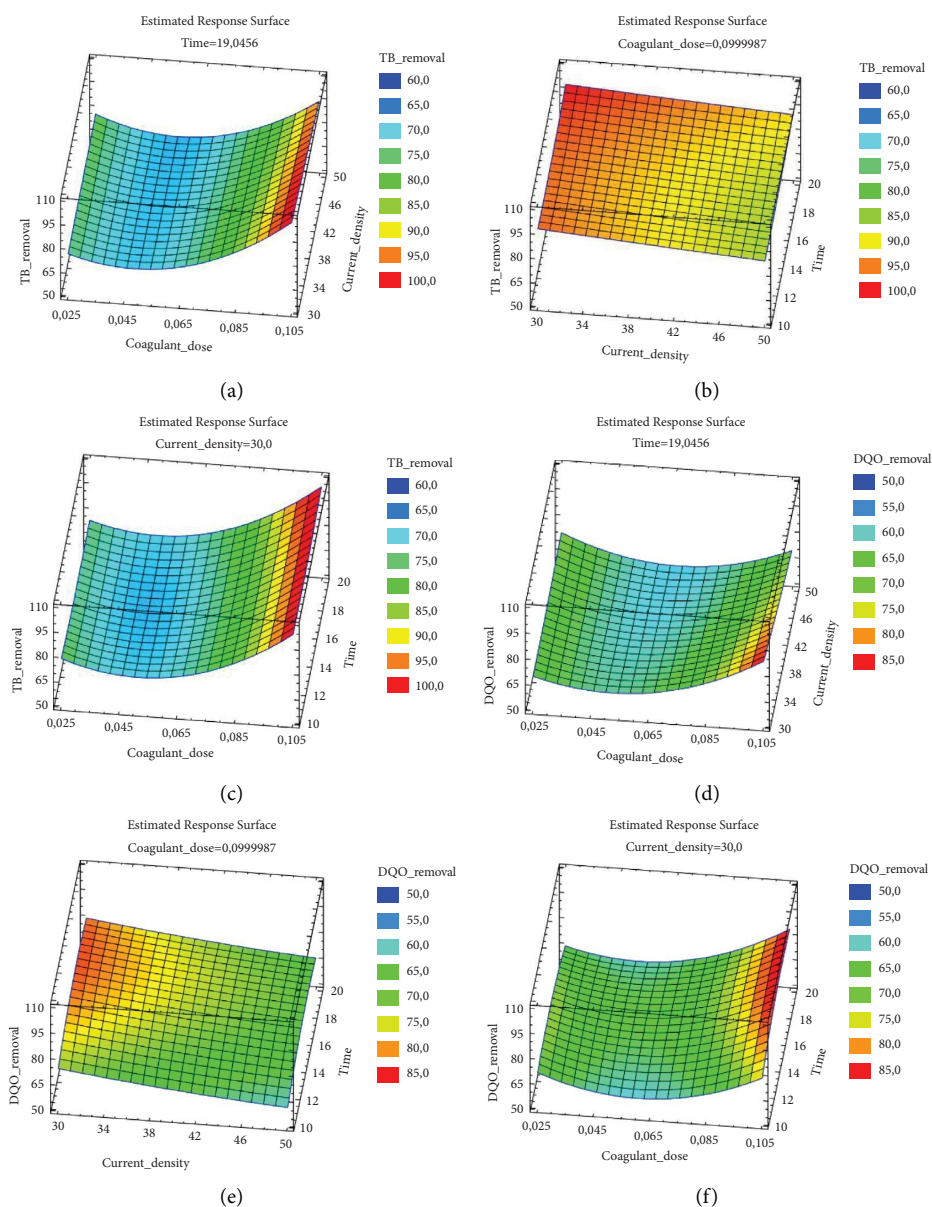


FIGURE 7: Three-dimensional response surfaces for turbidity values (a–c) and COD removals (d–f).

The positive signs in the coefficients represent a synergistic impact, and the negative coefficients represent an antagonistic effect [51]. A negative impact of the coagulant dose on the removal of TB and COD is observed. Table 5 shows the TB and COD removal values along with the predicted values obtained with regressions (3) and (4).

Table 6 shows the results of the analysis of variance (ANOVA) for the parameters of the response surface regression model using the experimental results for TB and COD removal.

The proposed statistical model presented adequate adjustment coefficients: $R^2 = 88.35\%$ for COD removal, allowing a removal of 81.72% to be achieved, and $R^2 = 94.75\%$ for TB removal, allowing a removal of 98.44% to be achieved, with a coagulant dose of 0.0999987 g/L, a current density of 30 mA/cm², and a time of 19.0456 minutes.

Figure 7 shows the response surface for the percentage removal of COD and TB as functions of CDo, CD, and T. The variable CDo has a greater influence than CD and T on the removal of COD and TB. It can also be seen that when the CD is lower, a greater removal of COD and TB is achieved.

The resulting response surfaces determine the regression polynomials, equations (3) and (4) provide a detailed view of how different values of coagulant dose, current density, and time provide different results for TB removal percentages and COD. Like the Pareto diagram (Figure 6(a)), the response surfaces of Figures 7(a), 7(b), 7(c) confirm that the coagulant dose is the most significant and important factor in the TB removal process: The increase in the value of this factor leads to an increase in the TB removal rate. Particularly, Figure 7(b) shows that when the coagulant dose is established to be equal to the optimal value 0.0999987 g/L determined with the optimization module of the Statgraphics software, the TB removal percentage is greater than 85% for different values of current density and time; this can be seen in Figure 7(c).

On the other hand, the response surfaces of Figures 7(d) and 7(e) show that the current density must be minimum to maximize the percentage of COD removal, which supports the calculation of 30 mA/cm² as the optimal value for this factor; a similar interpretation of this factor for TB removal can be seen in Figures 7(a) and 7(b). Additionally, although the time factor turns out to be nonsignificant for the removal of TB and COD, the surfaces show that the increase in time implies an increase in the percentage of removal of both variables, which explains why the optimization process determined that the time should be 19.04 minutes.

Increasing the current density can improve the coagulation efficiency due to greater production of metal ions, which promotes the formation of hydroxides. However, too much current density can lead to high energy costs and the generation of unwanted byproducts [52]. Each study uses different optimization methods and evaluates different combinations of parameters, demonstrating that the optimization of the electrocoagulation process in dairy wastewater is a complex topic, which requires an individualized approach. However, all studies conclude that electrocoagulation is a promising technique for treating dairy

wastewater and the optimization of parameters can be improved.

The dosage of coagulants or flocculants in the turbidity water for turbidity removal was another important variable that affects the turbidity removal efficiency. The result revealed that the turbidity of the residual whey effluent decreased with the dose of 0.05 g/L of chitosan:*Opuntia ficus-indica* mucilage. However, beyond the dose of 0.05 g/L, a decreasing profile in the percentage of turbidity removal was observed. Therefore, in this study, the optimal turbidity removal performance occurred with 0.05 g/L of Q-M-5, which resulted in a turbidity removal percentage of 94.60% in the treated effluent.

4. Conclusions

In the present study, experiments were conducted to evaluate and optimize the cheese whey wastewater treatment by a biocoagulant composed of *Opuntia ficus-indica* and chitosan (Q-M-5) as the primary treatment followed by an electrocoagulation process. The Q-M-5 biocoagulant was used without chemical modification. The turbidity removal and chemical oxygen demand were evaluated and optimized using the response surface methodology based on the Box–Behnken design to optimize the biocoagulant dose, current density, and time. The results indicate that the biocoagulant dose, current density, and pH were significant ($p \leq 0.05$) in the removal of turbidity and COD. The efficiency of the contaminant removal depended on the dose of the biocoagulant that obtained a high treatment efficiency. The optimal biocoagulation dose point of Q-M-5 was equal to 0.09 g/L biocoagulant dose at 30 mA/cm² current density and 19.04 minutes. The turbidity and COD removal efficiencies observed with the optimized operating parameters were 98.44% and 81.72%, respectively. The use of the Q-M-5 biocoagulant as the primary treatment of cheese whey wastewater provides evidence of being effective, in addition to being a natural, low-cost, ecological, and easy-to-apply coagulant. For future research, it is suggested to evaluate the process of primary coagulation with the biocoagulant followed by electrocoagulation on a pilot scale to improve the application in the treatment of whey and cheese wastewater.

Data Availability

The data are available upon request from the corresponding author.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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