

Article

Study of the Sedimentation Parameters of an Iron Ore Tailing from Fundão Dam Using a Tannin-Based Coagulant

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Abstract: From the rupture of the Fundão dam in Mariana–MG, there are tailings still present at the bottom of the plant that must be recovered. The flocculation followed by sedimentation operation can be applied as a unit operation in this recovering process. Instead of using conventional inorganic coagulants, bio-based coagulants offer some advantages, due to their low toxicity and biodegradability. Nonetheless, the use of bio-based coagulants in the mining industry is not established yet, due to the complex parameters that must be taken in consideration. This study analyzes the influence of the pH and flocculant concentration, which are the variables of the 2² full factorial design. The pH value for the batch sedimentation process was defined ranging from 5 to 9. Tanfloc, a tannin-based coagulant, was used as a coagulant agent. The results indicate a strong dependence on the coagulant concentration, and a recommended 15 g/L dosage with pH varying from 6 to 8. From batch sedimentation, it was possible to determine an exponential model for the sedimentation with an excellent fitting ($R^2 = 0.997$). The sedimentation efficiency calculated is 65.6%. These results confirm the potential use of bio-based materials in mining tailing treatments. In addition, they can be used in equipment sizing and simulations of the sedimentation operation.

Keywords: Risoleta Neves Hydroelectric; Candonga; flocculation; natural coagulant



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1. Introduction

The mineral sector has great economic importance for Brazil. According to the Annual Activity Report of IBRAM (Brazilian Mining Institute, Brasília, Brazil), in 2019, it directly represented approximately 4% of the national GDP (Gross Domestic Product), in addition to the indirect impact, since the sector is the most important source of raw material for national industry, which represents 16.8% of GDP. In addition, according to the report, iron ore accounted for 68% of mineral goods exported in 2019 [1].

The tailings resulting from the metal beneficiation and refinement process generate large volumes of mud, which are deposited in containment basins or tailings dams. In November 2015, the country witnessed one of the greatest environmental impacts resulting from mining, the Mariana disaster, where the rupture of the Fundão Dam, in Bento Rodrigues, a district of Mariana, MG, spread around 40 million cubic meters of iron waste in the Rio Doce basin. The impact caused by the avalanche dragging of dense material caused the removal of vegetation and houses, and the impact on the ecosystem was from the immediate reduction of microorganisms, fish and other aquatic animals throughout the bed of the Rio Doce. In addition to these, the disaster also impacted the region's economy, as fishing was prohibited, there was an interruption in the supply of electricity from hydroelectric plants, and there were water shortages in homes and industries [2].

About 115 km from the dam, the Risoleta Neves hydroelectric plant, also known as Candonga, managed to retain around 10.5 million m³ of tailings. Located in the municipality of Santa Cruz do Escalvado, MG, the dam had to be emptied in a hurry to contain the wave of tailings. The material retained in the Candonga dam began to be dredged at

the beginning of 2016, where, through two pumps, it is sent to two cofferdams, so that it can be decanted and later dry stacked at Fazenda Floresta. The waste itself is basically composed of elements from the soil, such as iron, manganese, silica, aluminum and water. It is classified as non-hazardous by the Brazilian Standard for the Classification of Solid Waste (NBR). However, the wave of mud that traveled through the soil and water bodies stirred up contaminants that are the result of agricultural, industrial and urban activity in the region. Like untreated domestic sewage, mercury that over the years has been used in the exploration of gold and chemical components [3].

The procedure of decanting the material is a technique that uses as a premise the gravitational force of the particles in a solid–liquid medium, where the difference in the specific mass between the phases allows the separation. Structures are commonly used that allow the removal of phases after the end of the process where the most concentrated (underflow) is removed by the base region, while the less concentrated is redirected to a spillway (overflow) [4].

The use of flocculation agents helps the sedimentation process, mainly for suspensions with small granulometry particles. Inorganic chemicals (alum, ferric sulfate, ferric chloride) are widely used as coagulant in wastewater treatments. In addition, the use of polymeric materials as a flocculant is also developed. Even though natural components that can act as a biocoagulant are still incipient, natural products can provide some advantages because of their biodegradability and their inertness on water properties [5]. Since there are still not many studies reported, the use of a natural coagulant is considered revolutionary. Allied to the advantages cited before, the economical aspect is also interesting when using natural coagulants, mainly when they are based on residues from agroindustry [6].

Natural polymers such as cellulose, chitin, gelatin, alginate, lignin and other starch derivatives are the most commonly used strategies in wastewater treatments. Not only as coagulants, but as filters, adsorbents or membranes. These materials are interesting due to advantages on the residue disposal [7]. In the mining industry, the main interests are based on heavy metal removal and turbidity decreasing. Both are related to the water quality after the ore processing.

Modified starch based on quinoa (*Chenopodium quinoa*) was used to remove heavy metals such as Cd, Hg, Pb and As and oil fractions from wastewaters. The coagulant reduced turbidity and up to 85% of heavy metals from synthetic aqueous systems [8]. *Opuntia ficus-indica* mucilage as a coagulant material presented a removal capacity higher than 90% for iron (Fe) and manganese (Mn) metals. This study relates a strong dependence on the system pH for the ions removal and turbidity decreasing using a Mexican river water sample [9]. Some plant-based coagulants based on *Moringa oleifera*, *Strychnos potatorum* Linn, *Plantago ovate*, *Trigonella foenum graecum* and *Opuntia ficus indica* are potential substitutes for chemicals. In this study is presented a landscape on the utilization of natural coagulants and the economic issues related to them. The utilization in small batch operations is highlighted, which confirms the necessity of upscaling studies [10].

The combination of natural and inorganic coagulants is also a viable alternative. The use of *Moringa oleifera* seed extract, zeolite, ferric chloride, chitosan and aluminum sulphate coagulants through the coagulation, flocculation and sedimentation process was analyzed. The dosage of 4000 mg/L of *Moringa oleifera* extract was optimized for this complex system. This study confirms the possibility and the difficulty to establish optimal process conditions [11].

Tannins are polyphenolic compounds that can be found in wood, the bark of trees, leaves, buds, stems, fruits, seeds, roots and plant galls. A complete review on the use of tannin-based coagulant is offered by Tomasi et al. (2022). The authors bring effluents from different industries such as cosmetic, laundries, textiles, food and landfills. They also remark the freedom from toxicity and the safety for the human health. Nonetheless, the economic issue and adoption on a large scale are still threats that must be coped with [12].

Grape (*Vitis vinifera*) seed powder was used to remove chromium (VI) ions from wastewater. The study optimized the sedimentation conditions. Optimized values of pH

and the coagulant dosage found were 4.53 and 0.5 g/L, respectively. The polyphenolic character from the grape seeds is responsible for guaranteeing the interaction with the metallic compound [13].

An intelligent alternative to remove aqueous Cr (VI) is the employment of tannic acid as coagulant. This material forms an organometallic Cr (III) complex by redox reaction. This triggering mechanism precipitates chromium in a pH-dependent system. The complex is possible to be obtained due to the polyphenol character of the tannin [14]. Tannic acid from *Acacia mearnsii* and ammonium hydroxide react to produce a formaldehyde-free coagulant. To reduce turbidity and remove color from a humic acid synthetic residue, a statistical approach was adopted. The satisfactory results reduced turbidity in 100% and the color removal of 89.9% [15]. Condensed and hydrolysable tannins, extracted from the flower of *Musa* sp., were used in the processing of iron ore. Using the response surface methodology, the system containing between 15 and 35% of solids was processed, and the turbidity reduction was over 97% [16].

The flocculation agent Tanfloc is a material derived from black wattle (*Acacia mearnsii*) with great potential in the treatment of mining wastewater [17]. However, the ideal concentration and the best pH range vary according to the flocculant used. Thus, this work presents, through a factorial design, the best concentration of tannin as a flocculant and the best pH range so that the sedimentation process is optimized.

2. Materials and Methods

Waste from the Candonga dam, in the city of Santa Cruz do Escalvado, Minas Gerais, was used. The waste from the desliming and flotation process, iron ore concentration stages, comes from the rupture of the Fundão dam, in the municipality of Mariana in 2015. The material used was dry, and was manually homogenized, undoing the larger aggregates. Elementary chemical characterization was performed using X-ray dispersive spectroscopy (EDX) (Model Vega 3, Tescan, Brno, Czech Republic). The acquisitions were performed in six different points of the raw solid sample for the metals present in the sample.

The flocculating agent used was Tanfloc, a compound based on tannins from black wattle. A solution of Tanfloc flocculating agent was prepared using 3.5 g of Tanfloc in 50 mL of deionized water. Sulfuric acid and sodium hydroxide 1M solutions were used to prepare the liquid media. A 2² full factorial design was adopted, where the parameters to be analyzed were the concentration of the polymer and the pH of the solution. It used the factorial design as a way to study the operating conditions for the sedimentation of mineral waste, and in analyzing the interactions between these variables. According to [18], the flocculating agent Tanfloc is more effective in the pH ranges from 4.5 to 8. The pH equal to 7 was defined as the central point. For the 2² model, the variables were codified in terms of -1, 0 and +1. The lowest values of the analyzed parameter received the lowest value, -1, and the highest value received +1. For the central values, 0 are defined, as can be seen in Table 1. The response variable is the overflow final turbidity. Statistica 7.0 (StatSoft, Hamburg, Germany) software was used for data processing. In addition, an analysis of variance (ANOVA) is performed to evaluate the correlation between the variables and the clarification.

Table 1. Full factorial design variables from the experimental design varying pH and flocculant concentration.

Experiment	Coded Values		Parameter Values	
	pH	Coagulant Concentration	pH	Coagulant Concentration (g/L)
1	-1	-1	6	5
2	-1	+1	6	15
3	+1	-1	8	5
4	+1	+1	8	15
5	0	0	7	10
6	0	0	7	10
7	0	0	7	10

For the tests was used a solid concentration of 37.5 g/L of tailings in the suspension, based on the study by Santos et al. (2018) [19]. It was obtained using 40 mL of liquid and 1.5 g of solids. The pH corrections (from 6 to 8) on the liquid phase were performed before the solid addition, in order to keep the concentration constant. After being added to the falcon tubes, they were manually shaken together for about 1 min. In order for the final system to obtain an adequate concentration of the flocculant (from 5 to 10 g/L), each falcon tube received the coagulant solution, which, after being added, was stirred and left to rest for another hour. After the predicted time, samples of 5 mL of the clarified material were collected using a graduated pipette, diluted and analyzed for turbidity.

The turbidity values were read using the Policontrol AP2000 turbidimeter (Policontrol, Diadema, Brazil), at room temperature 21 °C. Due to equipment limitations, it was necessary to dilute the clarified solution in 15 mL of deionized water. Representation of the concentration, indicated by the nephelometric turbidity factor scale (NTU), is obtained by Equation (1):

$$y = \beta_0 + \beta_1 \times X_1 + \beta_2 X_2 + \beta_{12} X_1 X_2 \quad (1)$$

where y is the NTU value (dependent variable), β_1 and β_2 are the coefficients for pH (X_1) and coagulant concentration (X_2) and β_{12} for the interaction between them. After treating the results, the response surfaces were generated using the full factorial design.

Based on the results of the factorial design, a batch sedimentation is performed in triplicate in a beaker containing 37.5 g/L of waste. Using the jar test module, the samples were shaken for five minutes. After agitation, the suspensions are transferred to three glass graduated cylinders, where the interface heights were collected every 10 min, and the turbidity values read after completing 24 h of sedimentation. Samples of clarified and sludge after completing 24 h were collected and weighed and the values of the Petri dish, which were stored in an oven at 105 °C, discounted. Thus, it was possible to verify the mass of water, which following the density value 1 g/cm³, and it was possible to describe the concentration of suspended solids and in the sludge.

A mathematical exponential model, obtained from Origin 2018, is proposed for the process. The efficiency is calculated by Equation (2):

$$\text{Efficiency (\%)} = [1 - (\text{Concentration overflow} / \text{Concentration feed})] \times 100 \quad (2)$$

3. Results and Discussion

The elementary chemical analysis from the EDX is shown in Table 2. The sample is based in iron and silica materials. The sample comes from the iron ore mining industry, so it is expected to have a high iron concentration. The iron is found in the oxide forms. Silica is present in the sample as sand (SiO₂). A considerable level of aluminum is present in the sample. Traces of potassium, manganese and calcium were observed. The presence of oxides can guarantee the interaction with the tannin. Figure 1 shows images of the tubes after 1 h of sedimentation. The turbidity values read in NTU of the samples are shown in Table 3, corresponding to each pH distribution and flocculant concentration.

Table 2. Elementary chemical composition for the solid sample.

Analyte	% Weight
Fe	51.411
Si	36.945
Al	11.021
K	0.343
Mn	0.176
Ca	0.104

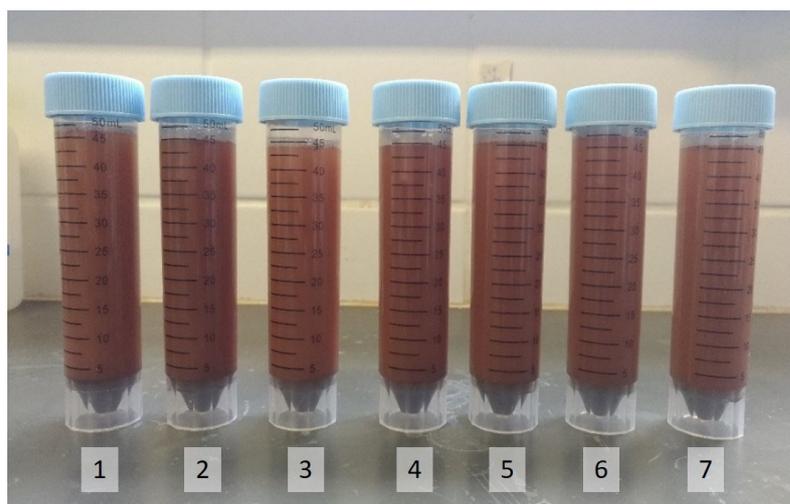


Figure 1. Sedimentation test in falcon tubes after 1 h of sedimentation in the presence of Tanfloc coagulant according to the experimental design (1–7).

Table 3. Overflow turbidity analysis performed after sedimentation under different conditions of pH and coagulant concentrations.

Experiment	Parameter Values		Turbidity (NTU)
	pH	Coagulant Concentration (g/L)	
1	6	5	196
2	6	15	143
3	8	5	201
4	8	15	142
5	7	10	171
6	7	10	172
7	7	10	171

Figure 2 represents the response surface for the statistical planning with repetition at the central point, where the color variation refers to the turbidity of the clarified product, noting that for solutions with higher polymer concentration, there is a lower turbidity value, and thus, clarification with less suspended solids.

It shows the response surface for turbidity values in relation to pH versus Tanfloc concentration. It is observed that pH values between 6 and 8 do not have a significant effect on the sedimentation of the tailings, presenting linear behavior. The ANOVA data (Table 4) corroborate what has already been presented, where the pH variation does not directly influence the turbidity.

Table 4. ANOVA table for the parameter analysis.

Parameters	SS ¹	DF ²	MS ³	F ⁴	p ⁵
pH	4.000	1	4.000	6.462	0.084535
Coagulant concentration *	3136.000	1	3136.000	5065.846	0.000006
pH + Coagulant concentration *	9.000	1	9.000	14.538	0.031725
Error	1.857	3	0.619		
Total	3150.857	6			
R ² = 0.994					

¹ SS, sum of squares. ² DF, degrees of freedom. ³ MS, mean square. ⁴ F, F ratio; ⁵ p, p value. * Statistically significant for a 95% level of confidence. If the level of confidence is 95%, p = 0.05, then the result is significant.

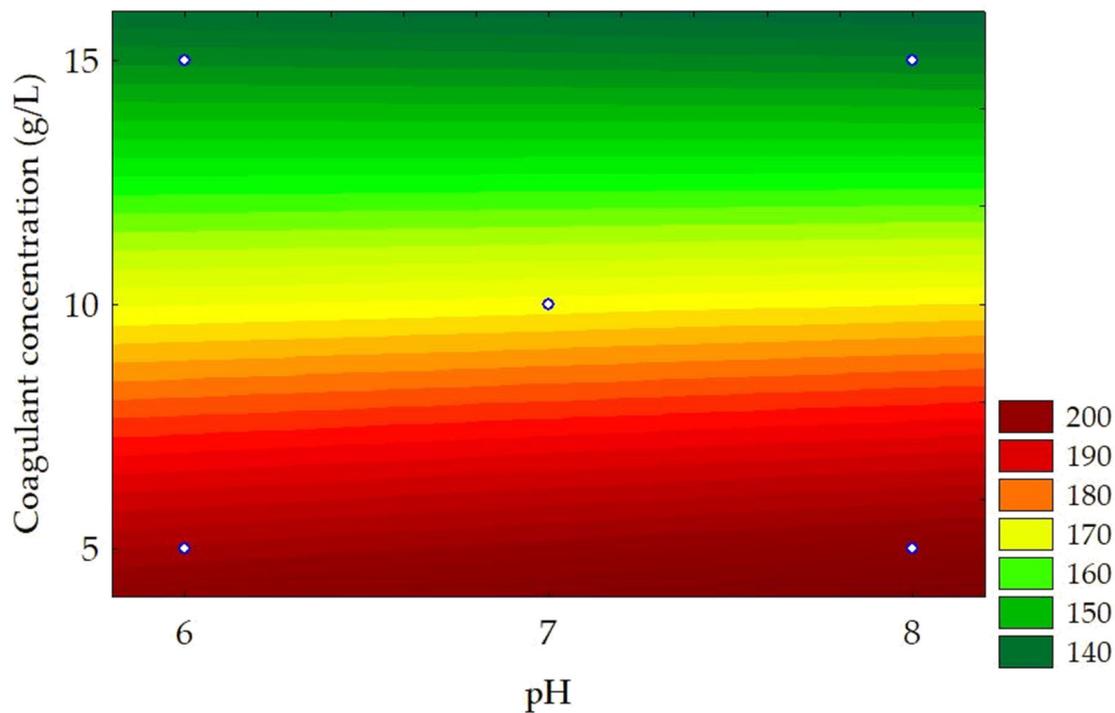


Figure 2. Response surface for the turbidity analysis from the overflow varying pH and coagulant.

Analyzing Table 4, it is clear that within the levels adopted, the pH variation itself does not influence the sedimentation process of the tailings, while the concentration of the polymer used has an inversely proportional relationship with the turbidity. In addition, it is noted that the interaction of the parameters adopted jointly influences the process, that is, the interaction of the pH with the flocculant is relevant for the sedimentation. The interaction can be explained by the influence of pH on the phenolic structures of the tannin. The pH variation can change the chemical structure of the tannin, more specifically, alter the hydroxyls present in its benzene structure, causing the denaturation of the polymer, thus increasing the solubility of the compound in alkaline pH, making it difficult to perceive its denaturation. Hydrogen ions also affect the charges of the colloids, determining the nature of the flakes formed [20,21]. Based on the values given by Statistica software, the turbidity value can be expressed through Equation (3), being valid for the pH range and concentration of Tanfloc using coded values shown in Table 1.

$$\text{NTU} = 170.9 - 28.0[\text{pH}] - 1.5[\text{pH}] [\text{Coagulant concentration}] \quad (3)$$

The ANOVA Table also presents the probability (p) of having a superior or extreme statistic in relation to that verified by the test. As the significance level was 5% (p value under 0.05), it shows that for the analyzed data, the pH is not significant. That is, it means that the treatments of the independent variables explain almost completely the turbidity variation (99.94%), being presented by the sum of the treatments. From the expression and the turbidity values, it can be seen that for higher values of pH and concentration of Tanfloc, there is a lower value of turbidity of the clarified product.

From previous studies using iron ore particles, it was identified that the system pH possesses a strong effect on the sedimentation. The study observed only the flocculation procedure, with agitation and an initial solid content of 130 g/L. It was possible to significantly reduce the turbidity by more than 97%. It means that the tannin-based coagulant presented an excellent result [16].

Thus, based on the statistical designs of the experiments, it is possible to establish conditions for the operation of the sedimentation. Maximum values of pH equal to 8 and concentration of 15 g/L were defined for the batch sedimentation test, since they presented the lowest turbidity value. Using the results of the statistical design of the experiments, batch sedimentation was performed. Based on the data collected, it was possible to construct the sedimentation curve, represented by Figure 3.

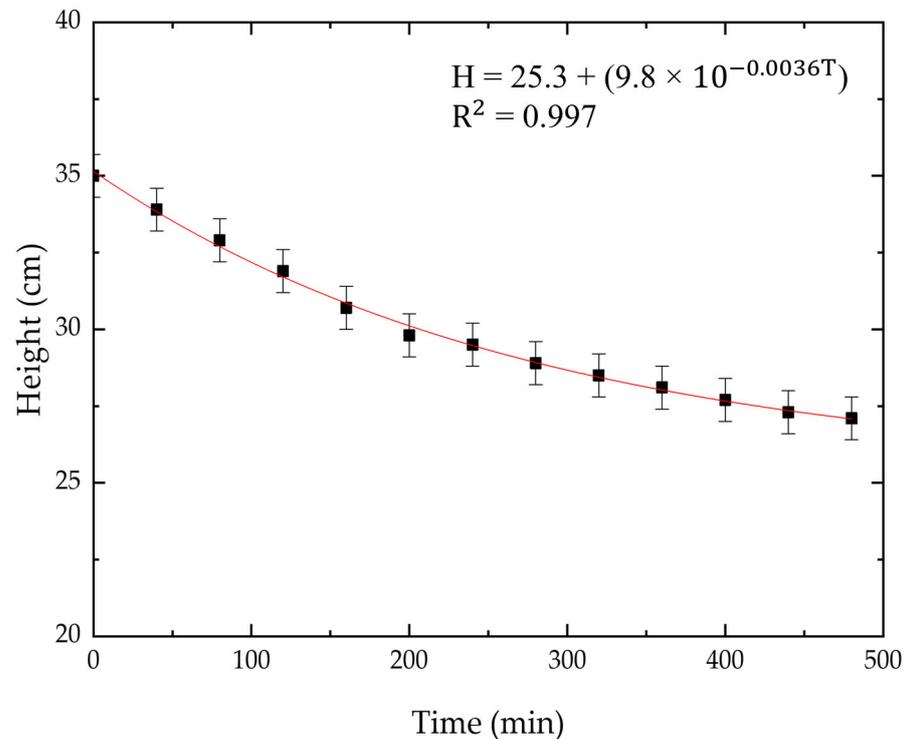


Figure 3. Batch sedimentation for ph 8 and coagulant concentration of 15 g/L.

In the literature, the models that describe the sedimentation graph of the interface height versus time are predominantly adjusted to an exponential function. Although exponential fits manage to express well the behavior of particles in a batch sedimentation, there are cases where power law functions fit better [22]. The model fits in an exponential model and the calculated R^2 (0.997) indicates a strong correlation.

The clarified product had a concentration of 12.9 g/L and the sludge 263.5 g/L. It represents an efficiency of 65.6%. Based on the interface height and time data, the plotted graph, based on Roberts' methodology, showed the model's critical point. This value, which represents the inflection of the clarification curve by the thickening curve, was noted at a time of 200 min with a height of 29.8 cm from the interface, with the sedimentation velocity and critical concentration equal to 0.009 cm/min and 39.5 g/L.

4. Conclusions

With this work, it was possible to define the best parameters so that the iron ore tailings present in the Risoleta Neves Hydroelectric Power Plant settle and generate a clarified product with a lower concentration of suspended solids. For the range of adopted pH values, it was evident that in central values, between 6 and 8, the variation in the clarification results was not as expressive. By applying the 2^2 factorial design, also varying the concentration of the tannin flocculant, it was possible to verify this relationship. By statistical analysis, the dependent variable, turbidity, responds directly to only one of the two independent variables. The concentration of tannin, the natural flocculant used in the study, directly influences the results, where a higher concentration of this ensures lower turbidity of the clarified product. However, the interaction between the analyzed

parameters, pH and polymer concentration, also proved to be relatively significant, which can be explained by the influence of hydrogen ions on the chemical structure of tannin, more specifically, their reaction with benzene hydroxyls. In the statistical analysis with two parameters and repetition at the central point, the highest values were more efficient, with pH equal to 8 and concentration equal to 15 g/L. Thus, batch sedimentation was proposed to verify clarified and sludge concentrations, and a 65.6% efficiency.

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