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# EVALUATION OF THE ANIONIC POLYMER ADDITION IN THE CLARIFICATION OF REAL EFFLUENT FROM THE HYDROPHILIC COTTON BLEACHING

## AVALIAÇÃO DA ADIÇÃO DE POLÍMERO ANIÔNICO NA CLARIFICAÇÃO DE EFLUENTE REAL PROVENIENTE DO ALVEJAMENTO DE ALGODÃO HIDRÓFILO

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## ABSTRACT

Water, essential for maintaining life, is widely used in industrial processes, generating high volumes of industrial liquid effluents. These, if not treated properly end up contaminating water bodies and causing serious damage to the environment. Among the technologies applied to industrial effluent treatment, physical-chemical treatments are usually accompanied by auxiliary products. In this context, the present work aimed to evaluate the influence of anionic polyacrylamide addition as a coagulation aid of industrial effluent clarification from the bleaching process of cotton hydrophilic. Based on preliminary assays, a Central Rotational Composite Design (DCCR) was developed for the coagulant/flocculant combination, evaluating pH, turbidity, and color. The results obtained showed that anionic polymer improved the treatment performance, forming larger, heavier, and more stable flakes, which resulted in better removal of turbidity (29.7%) and color (33.09%) when compared to the treatment without its addition (22% and 25%) respectively. They also speed up the decanting process, reducing the consumption of primary coagulant and contributing to the effluent parameters being following the discharge standards stipulated by legislation, proving its efficiency as a coagulation aid.

**Keywords:** anionic polyacrylamide, flocculation, primary physicochemical treatment.

## RESUMO

A água, essencial para a manutenção da vida, é amplamente utilizado em processos industriais, nos quais geram altos volumes de efluentes líquidos industriais. Estes, se não tratados adequadamente, acabam contaminando corpos hídricos e causando sérios danos ao meio ambiente. Dentre as tecnologias aplicadas no tratamento de efluentes industriais, os tratamentos físico-químicos geralmente são acompanhados de produtos auxiliares. Neste contexto, o presente trabalho teve como objetivo avaliar a influência da adição de poliacrilamida aniônica como auxiliar de coagulação na clarificação de efluente industrial proveniente do processo de branqueamento de algodão hidrofílico. Com base em ensaios preliminares, foi desenvolvido um Planejamento Composto Central Rotacional (DCCR) para a combinação coagulante/floculante, avaliando pH, turbidez e cor. Os resultados obtidos mostraram que o polímero aniônico melhorou o desempenho do tratamento, formando flocos maiores, mais pesados e mais estáveis, o que resultou em melhor remoção de turbidez (29,7%) e cor (33,09%) quando comparado ao tratamento sem sua adição (22% e 25%) respectivamente. Eles também aceleram o processo de decantação, reduzindo o consumo de coagulante primário e contribuindo para que os parâmetros do efluente estejam de acordo com os padrões de lançamento estipulados pela legislação, comprovando sua eficiência como coadjuvante de coagulação.

**Palavras-chave:** poliacrilamida aniônica, floculação, tratamento físico-químico primário

## 1 INTRODUCTION

Water is a natural resource essential for daily life (MAJJED, IFTIKHAR and MUKHTAR, 2024). To be used directly by humans, it must present adequate physical and chemical conditions, be free of substances that are harmful to living beings, and have a satisfactory quality (VALIM and FRUGOLI, 2015). In industrial activity, water is essential for the application and execution of its processes and supply of cooling systems and steam generators. However, its use makes it contaminated, thus giving rise to the so-called liquid effluents (GANDHI, 2004). According to MAJJED, IFTIKHAR and MUKHTAR (2024), the OECD Environmental Outlook to 2050 predicts that manufacturing and industrial production will undergo growth of 400 % expansion, consequently propelling a 55 % increase in global water consumption from 2012 to 2050.

Due to the impacts caused by effluents on the environment, the management and monitoring of treatment plants becomes a challenge (GOFFI et al., 2022). The characteristics of industrial effluents are diverse, as they depend on the nature and size of the industry, the products manufactured, the degree of modernity of the production processes, and the recycling and reuse practices of the generating source (VALIM and FRUGOLI, 2015). Regarding the particularity of each industrial effluent, it is important to develop treatment technologies specific to each industry. It is important to consider, in addition to the particularity of each effluent, financial resources, pollutant removal efficiency, and compliance with current legislation (GOFFI et al., 2022).

In the treatment of industrial effluents, the use of chemical coagulants, such as aluminum sulfate ($Al_2(SO_4)_3 \cdot nH_2O$), widely used in the coagulation phase, stands out due to its good efficiency, low cost, and easy handling (ROCHA, PIMENTEL and NAKAMURA, 2019). However, the application of coagulant agents is not sufficient for the total clarification of the effluent in many situations (LIMA, 2015). High dosages of coagulant result in high operating costs, the need for large physical spaces to store the product, high production of non-biodegradable sludge, and difficulty in its final disposal, making its use unfeasible (ZARA et al., 2012; ROCHA, PIMENTEL and NAKAMURA, 2019). Depending on the quantity dosed in the treatment, when they return to water bodies and are used again by humans, they can be toxic and cause diseases related to dementia and motor coordination (ZIMPEL, 2013). Because of this, polymers, known as flocculants, have been increasingly used as coagulation aids (LEMOS et al., 2017). Compared to coagulants, polymers have several advantages in their use: less sludge generation and improvement in its dehydration conditions, increased size, resistance, and density of flocs, increased flocculation and decantation speed, better clarification results and reduced consumption of primary coagulant (ZIMPEL, 2013; ZARA et al., 2012; BARBALHO, 2017; RIBEIRO, 2018).

Polyacrylamide is a synthetic, water-soluble, flexible-chain, high-molecular-weight polymer that contains the acrylamide monomer as its main constituent. It can be presented as a neutral, anionic, or chemically modified homopolymer (BARBALHO, 2017). Thus, its wide use is due to its versatility in charge density (it can be neutral, anionic, or cationic), obtaining different molar masses, high-viscosity solutions, and formation of strong hydrogen bonds with mineral particles (BARBALHO, 2017). Anionic polyacrylamide polymers are composed of more than 150,000 acrylamide monomers per molecule, typically having molar masses between 12-15 $mg.mol^{-1}$, and act by neutralizing positive electrical charges (ZIMPEL, 2013; RODRIGUES, 2016). They result from the polymerization of the acrylamide monomer via free radical or through copolymerization between two or more monomers, such as acrylamide and acrylic acid (ZIMPEL, 2013; BARBALHO 2017). They are usually efficient within a wide pH range (7 to 14) and in low doses. They are widely used in the treatment of aqueous effluents after being previously treated with hydroxide-forming substances, such as aluminum sulfate (BARBALHO, 2017). They are commonly sold in solid form since they have good solubility in water under agitation and at room temperature (RODRIGUES, 2016;

NASCIMENTO 2017). However, very intense agitation can cause the polymer chains to break, impairing their performance. Therefore, their repair is a step that requires care.

Currently, wastewater treatment techniques such as physical, chemical, or biological processes are used in isolation or combination, in addition to their association with advanced processes (AGUIAR, 2022; SILVA et al., 2023). Traditionally, the treatment of industrial effluents is based on coagulation followed by flocculation and sedimentation, which is an efficient technique, but with the final generation of a large volume of contaminated waste, called sludge. With this in mind, in recent decades, new techniques have been developed, such as treatment by advanced oxidative processes (AOPs), Dissolved Air Flotation (DAF), electrochemical processes (BARROS, 2016; CAMPOS, 2018; AGUIAR, 2022; SILVA et al., 2023), in addition to the replacement of inorganic coagulants, such as aluminum sulfate, by organic ones, such as tannin, extracted from the bark of the black wattle (GUDOSKI and RODRIGUES, 2017). These techniques have been evaluated mainly in the treatment of textile effluents.

About the treatment of effluents from the bleaching process of hydrophilic cotton for pharmaceutical, medicinal, and hygienic purposes, little has been studied. However, the similarity of this to the effluents generated after primary processing stages in the textile industry allows new treatment alternatives for the textile industry to be (re)considered for the effluent in question. This suggests the importance of carrying out further studies on the subject. It is important to carry out research involving real industrial effluent, to optimize materials and experimental and process conditions, and to comply with current legislation regarding the release of treated effluent into nature, as well as its reuse (SILVA et al., 2023). Therefore, this work aims to evaluate the performance and influence of the anionic polyacrylamide polymer, compared to aluminum sulfate, in industrial effluent clarification from the hydrophilic cotton bleaching process.

## 2 MATERIAL AND METHODS

### 2.1 Effluent collection

The effluent used in this study comes from a cotton bleaching industry in Itajaí Valley, Santa Catarina, Brazil, and was collected at 36.9°C ± 2°C in the equalization tank. This effluent was stored in 5 L and 50 L plastic drums previously sanitized with water and neutral detergent. The effluent was then taken to the SAMAE Laboratory (Brusque, Santa Catarina), where physical-chemical analyses were performed.

## 2.2 Physicochemical analysis

The physical-chemical parameters analyzed in the crude effluent (CE) and treated effluent (TE) were pH, turbidity, and color (Table 1). All parameters were analyzed in triplicate.

Table 1. Parameters analyzed and methodology used

| Parameter | Equipment | Measurement method |
|---|---|---|
| pH | pHmeter HACH Sension$^+$ pH 31 | Potentiometric |
| Turbidity (NTU) | Turbidimeter HACH 2100 Q | Nephelometric |
| Color (Pt-Co) | Spectrophotometer HACH DR 6000 | Spectrophotometric |

Source: The author (2024)

## 2.2 Coagulation, flocculation, and decantation tests

The coagulation, flocculation, and decantation tests were performed in the Jar-Test (Milan 203 M), which has 6 jars with a volumetric capacity of 2 L each and a multiple distributor that simultaneously doses the products in all jars. The chemical products used were: liquid iron-free aluminum sulfate coagulant (QUIMISA) and anionic polyacrylamide-based polymer (AP 1234 - BUSCHLE & LEPPER).

To perform the assays, the aluminum sulfate solution was prepared at a final concentration of 1%; and the polymer solution was prepared at a concentration of 0.08%, adding 0.64 g of polymer to 800 g of distilled water under mechanical stirring until complete dissolution and homogenization (adapted from NTS 233 Ver. 1 – SABESP, 2020). The solution was prepared and immediately used, resulting in 5 ppm of aluminum sulfate and 0.8 ppm of anionic polymer solution.

The conditions for performing the fast mix (coagulation), slow mix (flocculation), and decantation steps for the tests performed (Table 2) were adapted from Vaz et al. (2010) and Dumke et al. (2015). In addition to the physical-chemical analyses performed on the supernatant liquid of each effluent sample, the assays had their performance visually evaluated for floc formation and clarification. All assays were performed in triplicate.

Table 2. Conditions for carrying out tests in Jar-Test

| Step | Velocity | Time |
|---|---|---|
| Fast mix | 121 rpm | 1 min |
| Slow mix | 20 rpm | 15 min |
| Decantation | 0 rpm | 20 min |

Source: The author (2024)

## 2.4 Preliminary tests for aluminum sulfate coagulant contents

To determine the best dosage of aluminum sulfate coagulant (5, 10, 15, 20, 25, or 30 ppm equivalent to 1 mL, 2 mL, 3 mL, 4 mL, 5 mL, or 6 mL) and evaluate its performance in the clarification stage, 6 tests and 1 control were carried out, from the addition of 2 L of crude effluent in each of the 6 jars of the Jar Test under the conditions of Table 2. The aluminum sulfate concentration that presented the best result was selected as the central point of the coagulant dosage in the factorial design.

## 2.5 Factorial design

A $2^2$-factorial design, including 4 axial points and 4 repetitions at the central point, totaling 12 assays, was used to evaluate pH, turbidity, and color based on the variation of aluminum sulfate and anionic polymer (Table 3 and Table 4). The central point assay condition for aluminum sulfate was defined by the best result obtained in the preliminary assay for aluminum sulfate. The central point assay condition for the anionic polymer was defined as 2 mL, according literature (adapted from RODRIGUES, 2016).

**Table 3. Planning levels for each independent variable**

| Variable | Levels | | | | |
|---|---|---|---|---|---|
| | (-√2) | (-1) | (0) | (+1) | (+√2) |
| Aluminum Sulphate | 3.2 mL<br>16 ppm | 4 mL<br>20 ppm | 6.0 mL<br>30 ppm | 8 mL<br>40 ppm | 8.8 mL<br>44 ppm |
| Anionic polymer | 0.59 mL<br>0.233 ppm | 1.0 mL<br>0.4 ppm | 2.0 mL<br>0.8 ppm | 3.0 mL<br>1.2 ppm | 3.41 mL<br>1.364 ppm |

Source: The author (2024)

**Table 4. Central composite planning matrix with actual and coded levels**

| Assay | Aluminum Sulphate | | Anionic Polymer | |
|---|---|---|---|---|
| | Encoded | Real | Encoded | Real |
| 1 | (-1) | 4 mL | (-1) | 1.0 mL |
| 2 | (+1) | 8 mL | (-1) | 1.0 mL |
| 3 | (-1) | 4 mL | (+1) | 3.0 mL |
| 4 | (+1) | 8 mL | (+1) | 3.0 mL |
| 5 | (-√2) | 3.2 mL | 0 | 2.0 mL |
| 6 | (+√2) | 8.8 mL | 0 | 2.0 mL |
| 7 | 0 | 6.0 mL | (-√2) | 0.59 mL |
| 8 | 0 | 6.0 mL | (+√2) | 3.41 mL |
| 9 | 0 | 6.0 mL | 0 | 2.0 mL |
| 10 | 0 | 6.0 mL | 0 | 2.0 mL |
| 11 | 0 | 6.0 mL | 0 | 2.0 mL |
| 12 | 0 | 6.0 mL | 0 | 2.0 mL |

Source: The author (2024)

### 2.6 Data analysis

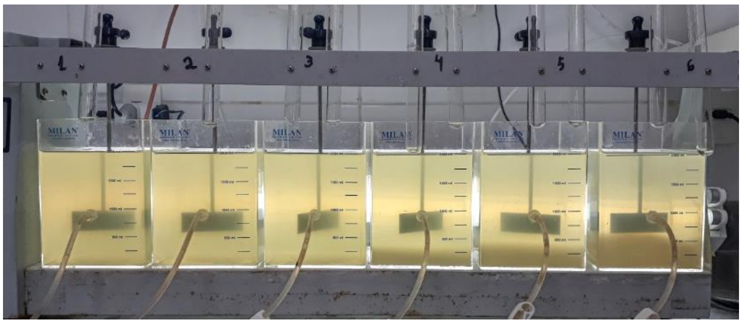
The results obtained from crude effluent analysis and after the preliminary treatment were evaluated using Microsoft Excel Software, considering their respective means and standard deviation. For the experimental planning, in addition to the initial treatment of the data in Microsoft Excel Software, the results obtained were also processed in GNU Octave Software (adapted from PEREIRA and PEREIRA-FILHO, 2018).

## 3 RESULTS AND DISCUSSION

### 3.1 Physicochemical analysis

The effluent used in this study was collected industrially after passing through preliminary treatment and consisted of a rotary sieve and an equalization tank, responsible for promoting the homogenization of the effluent for treatment. The characterization of the pH, turbidity, and color parameters of the crude effluent (CE) and after treatment (ET) in the different assays, changing the amount of aluminum sulfate, as well as the discharge standards established by legislation, can be observed in Figure 1 and Table 5.

Figure 1. Preliminary test with aluminum sulfate after decantation



Assays 1 to 6 (from left to right) with aluminum sulfate contents: 1 mL, 2 mL, 3 mL, 4 mL, 5 mL and 6 mL.

Source: The author (2024)

Table 5. Analysis of pH, turbidity, and color of CE and preliminary assays

| | Assays | pH | Turbidity (NTU) | Color (mg Pt-Co/L) |
|---|---|---|---|---|
| | CE | 11.13 ± 0.01 | 103.8 ± 3.1 | 325 ± 2 |
| Assays | 1 | 10.43 ± 0.01 | 103.2 ± 4.1 | 320 ± 1 |
| | 2 | 10.52 ± 0.01 | 102.7 ± 0.6 | 305 ± 1 |
| | 3 | 9.56 ± 0.01 | 93.9 ± 1.0 | 283 ± 2 |
| | 4 | 9.08 ± 0.01 | 84.5 ± 0.5 | 260 ± 1 |
| | 5 | 8.64 ± 0.02 | 90.3 ± 1.2 | 274 ± 1 |
| | 6 | 7.42 ± 0.02 | 80.9 ± 1.6 | 244 ± 1 |
| Release Pattern (Class 2) | CONAMA 357/05 | 6.0 – 9.0* | 100* | 75* |
| | CONAMA 430/11 | 5.0 - 9.0 | NE | NE |
| | State Law 14675 | 6.0 – 9.0 | NE | NE |

CE = crude effluent; * Changed by CONAMA 430/11. NE – Not specified. Source: The author (2024)

Preliminary assays are fundamental for determining initial parameters in industrial processes (NEVES, 2016). Preliminary assays using only aluminum sulfate do not show visible clot formation (Figure 1) in assays 1 and 2, nor were they clarified. This fact may be associated with the lower volumes of coagulant used, as well as observed in studies by Dassan et al. (2015), where lower coagulant dosages resulted in lower turbidity removal efficiencies in industrial washing effluents. In addition to the coagulant dosage used, the contact time also shows an influence on color and turbidity removal (BAPTISTA et al., 2014). In this study (Figure 1), the decantation time (20 minutes) was the same for all coagulant concentrations evaluated.

The efficiency of coagulation/flocculation processes can be measured by the size and characteristics of the flocs formed, the amount of flocculated matter, and the clarification of the supernatant liquid (BARBALHO, 2017). These characteristics were visible in assays 4, 5, and 6 (Figure 1), which also showed parameters more suited to environmental regulations regarding pH, turbidity, and color (Table 5). pH control is essential for the performance of the coagulant's action (RIBEIRO, 2018). In the case of aluminum sulfate, for example, a pH below 5.5 or above 8.5 is not recommended, as it makes the aluminum ions soluble in the medium, compromising the efficiency of coagulation (ROSALINO, 2011). Ideally, after treatment, the effluent from an industry should meet all the regulations relevant to its location. Thus, the pH values obtained for assays 5 and 6 meet the Resolutions for release into water bodies in force in the region of the present study (5.0 – 9.0) - CONAMA 430/11 and CONSEMA Resolution No. 181/2021 (SILVA, 2015; NEVES, 2016; ZANITH, 2016).

The Itajaí-Mirim River, into which the effluent under study is discharged, is considered by State Ordinance 24/1979 as Class 2 (SILVEIRA, 2007). According to CONAMA 357/05, discharge standards for Class 2 rivers should not exceed 100 NTU of turbidity and 75 mg Pt-Co/L of true color. However, with the implementation of CONAMA 430/11, the discharge standards for these parameters are no longer quantified. In turn, CONSEMA No. 181/2021 also does not establish turbidity and color limits. What is required, however, is that the discharge of effluents into Class 2 rivers, even if treated, does not impair their quality, that is, does not cause changes in the color and turbidity of the receiving water body (LEME, 2019).

### 3.2 Coagulation, flocculation, and decantation assays

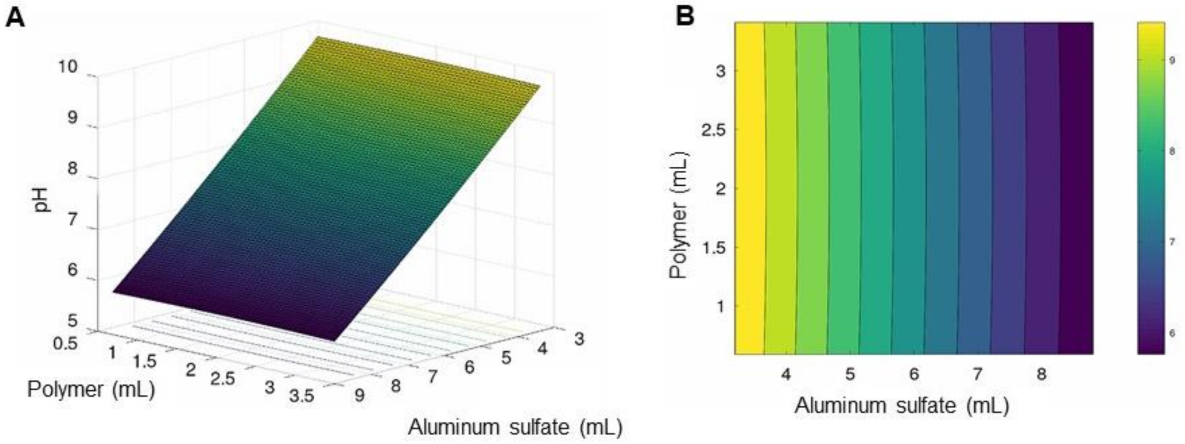
Values of pH, turbidity removal, and effluent color as a function of the variation in the dosages of aluminum sulfate and anionic polymer obtained by factorial design are shown in Table 6 and Figures 2, 3, and 4.

Table 6 - Results of pH, turbidity, and color analyses of the experimental design

| **Assays** | **pH** | **Turbidity (NTU)** | **Color (mg Pt-Co/L)** |
|---|---|---|---|
| 1 | 9.20 ± 0.01 | 90.7 ± 1.2 | 274 ± 1 |
| 2 | 6.16 ± 0.02 | 71.9 ± 0.3 | 214 ± 1 |
| 3 | 9.20 ± 0.01 | 89.0 ± 0.4 | 269 ± 1 |
| 4 | 6.25 ± 0.01 | 67.3 ± 0.2 | 198 ± 1 |
| 5 | 9.62 ± 0.02 | 92.7 ± 0.8 | 278 ± 2 |
| 6 | 5.88 ± 0.01 | 62.4 ± 0.2 | 194 ± 1 |
| 7 | 7.74 ± 0.02 | 78.9 ± 0.4 | 239 ± 1 |
| 8 | 7.68 ± 0.01 | 73.0 ± 0.3 | 218 ± 1 |
| 9 | 7.63 ± 0.01 | 75.6 ± 0.4 | 223 ± 1 |
| 10 | 7.55 ± 0.01 | 72.8 ± 0.2 | 214 ± 1 |
| 11 | 7.75 ± 0.01 | 78.8 ± 0.5 | 236 ± 1 |
| 12 | 7.95 ± 0.01 | 76.3 ± 0.1 | 232 ± 1 |
| CONAMA 357/05 | 6.0 – 9.0* | 100* | 75* |
| CONAMA 430/11 | 5.0 - 9.0 | NE | NE |
| CONSEMA nº 181/2021 | 6.0 - 9.0 | NE | NE |

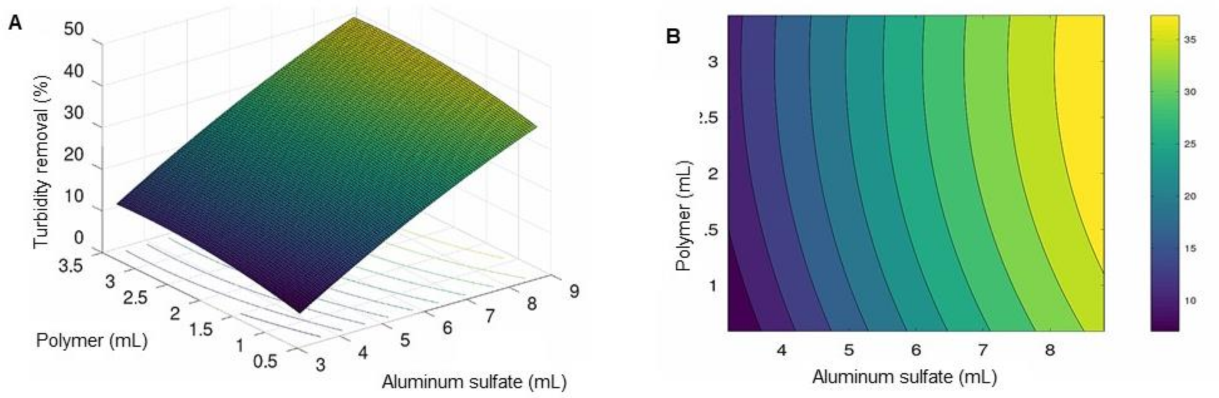
* Changed by CONAMA 430/11. NE – Not specified. Source: The author (2024)

Figure 2. Influence of aluminum sulfate and polymer dosages on pH variation



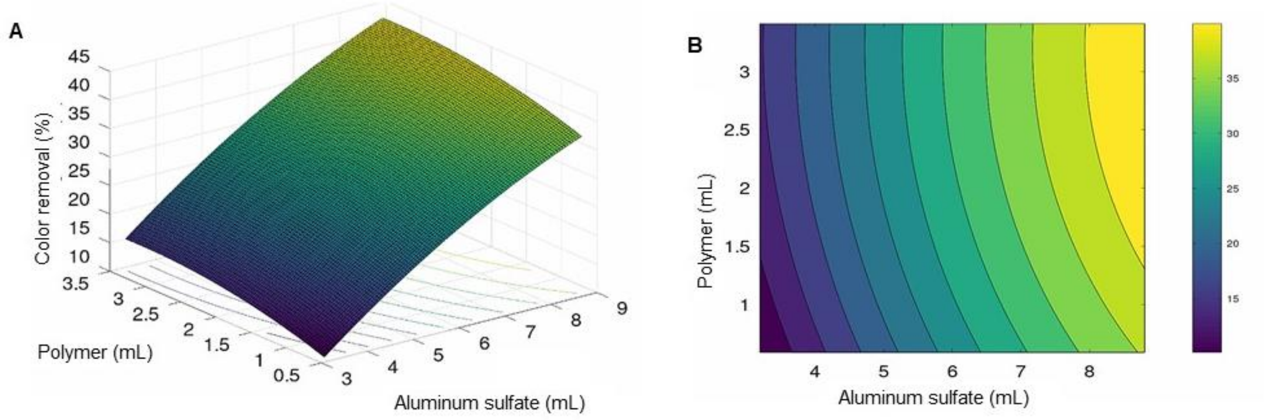
(A) = Response surface, (B) = contour plot. Source: The author (2024)

Figure 3. Influence of aluminum sulfate and polymer dosages on turbidity removal (%)



(A) = Response surface, (B) = contour plot. Source: The author (2024)

Figure 4. Influence of aluminum sulfate and polymer dosages on color removal (%)



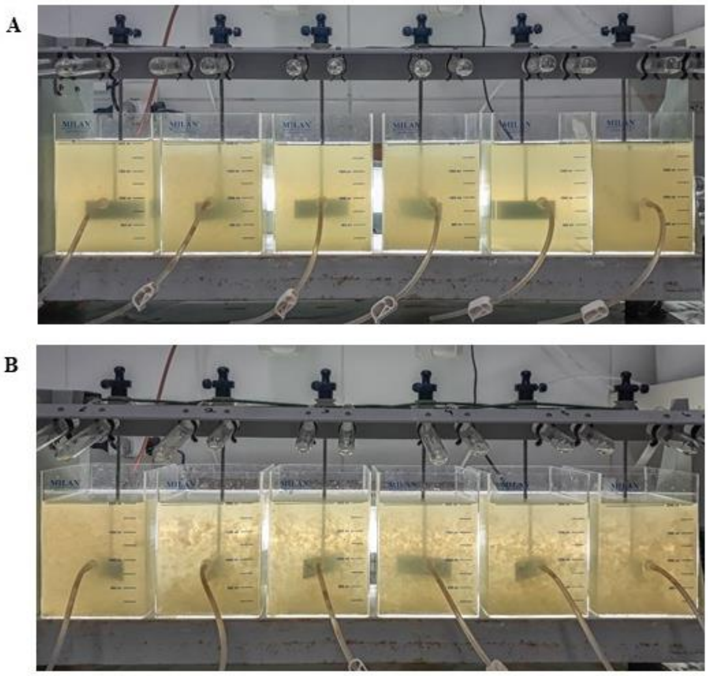
(A) = Response surface, (B) = contour plot. Source: The author (2024)

It can be observed that (Figure 2A, Figure 2B) the pH variation of the effluent studied is not influenced by the dosage of the anionic polymer acrylamide, but rather by the dosage of aluminum sulfate. This is because aluminum sulfate, when undergoing hydrolysis reactions, consumes the alkalinity present in the medium (ZARA et al., 2012), that is, the higher its dosage, the greater the reduction in pH. According to the data shown in the response surface (Figure 2A), it can be noted that to obtain effluents with pH values within the limits stipulated by legislation (Table 6), the dosage of aluminum sulfate should be between 4 mL and approximately 8 mL, obtaining pH values close to neutrality when 6 mL of coagulant is used.

Regarding turbidity, (Figure 3A; Figure 3B) the polymer and aluminum sulfate influence its removal. Higher turbidity removal percentages (35%) are observed when using dosages higher than 1.25 mL of polymer and close to 8 mL of aluminum sulfate. However, the use of 6 mL of aluminum sulfate alone provided a 22% turbidity removal (Table 5, test 6). This shows that its combined use with the polymer begins to be effective from the proportion of 6 mL of aluminum sulfate and 1.5 mL of polymer (25% turbidity removal), as shown in Figure 3B. This characteristic is also observed in the color of the treated effluent (Figure 4A; Figure 4B), where higher color removal percentages (40%) can be obtained by polymer concentrations from 1.25 mL and close to 8 mL of aluminum sulfate. The addition of the polymer aids in the effluent clarification process (ZIMPEL, 2013; BARBALHO 2017). However, its use has proven effective when combined with levels above 6 mL of aluminum sulfate.

The addition of flocculants in treatment processes favors the increase in particles during the flocculation stage due to the formation of chemical bridges, resulting from their long chains (BARBALHO, 2017). This characteristic was observed in the factorial design assays (Figure 5A and Figure 5B), where the formation of flakes after the addition of the polymer increased when compared to the preliminary assays (Figure 1), with better flakes formation between assays 7 and 12 (Figure 5B). Although the polymer proved effective in combinations with levels above 6 mL of aluminum sulfate, the decantation stage was much faster. While in preliminary assay 6 (Table 4), the time required for good decantation was 20 min, in assay 7 of the factorial design (Table 4), containing the same dosage of coagulant, but with the addition of polymer, the time required for decantation was 45 seconds. In experiment 7 of the factorial design (Table 6), the levels of turbidity and color removal also showed better percentages, reaching 29.70% and 33.09% respectively, compared to 22% and 25% removal when the coagulant was used alone. Dumke et al. (2015) also recorded better results in the removal of color and turbidity from water after the addition of polymer in the treatment.

Figure 5. Effluent treated with aluminum sulfate and anionic polymer under the conditions of the central composite factorial design



(A) = Assays 1 to 6 of the design (from left to right), (B) = Assays 7 to 12 of the design (from left to right).
Source: The author (2024)

Although the results from experiments with polymer addition were generally better than those with aluminum sulfate alone in this study, not all of the pH, turbidity, and color results obtained fell within the limits established by law (Table 6). Thus, based on the response surfaces and contour plots obtained for each parameter analyzed, it is possible to observe more clearly the influence of the coagulant and flocculant on the treatment, as well as determine ideal dosages for obtaining an efficient treatment that meets the standards of the law.

## 4 CONCLUSIONS

The addition of an anionic polymer based on polyacrylamide had a positive influence on the treatment of hydrophilic cotton effluent, promoting improvement in the removal of color and turbidity, with little influence on the pH of the medium. The results obtained about the parameters evaluated in this study could not be achieved with only the aluminum sulfate coagulant within the range studied, without the addition of an anionic polymer. The contribution of the polymer to the formation and growth of flocs in the flocculation process was extremely noticeable, being able to significantly reduce the time required in the decantation and sludge formation stage. Thus, its efficiency as a coagulation aid in the treatment of effluent from hydrophilic cotton bleaching processes is proven.

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