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Poly(acrylamide-co-sodium acrylate) Used as a Flocculation Aid Associated with Aluminum Polychloride in the Treatment of Low Turbidity Surface Water by Charge Neutralization

Poli(acrilamida-co-acrilato de sódio) Empregado como Auxiliar de Floculação Associado com Cloreto de Polialumínio no Tratamento de Água Superficial de Baixa Turbidez por Neutralização de Cargas

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The use of flocculation aids is a common practice in the treatment of urban water supply systems, and synthetic polymers containing polyacrylamide are widely used in this context. The aim of this study was to test the effectiveness of poly(acrylamide-co-sodium acrylate) obtained from the polyacrylonitrile recycling process as a flocculation aid employed in ranges of small and large concentrations. The experiments were performed in jar tests using low turbidity surface water. Two concentrations of polymer were tested: a small concentration ranging from 0 to 2.5 mg L^{-1}, and a large concentration varying from 0 to 125 mg L^{-1}. Zeta potential and pH were measured after the application of the reagents, and turbidity after the jar test experiment. The results indicated that 2 mg L^{-1} of the polymer as a flocculation aid increased the removal of turbidity. However, the experiments using larger doses of the reagent interfered with the coagulation-flocculation process, probably due to the high final pH level, charge repulsion, or steric hindrance.

Keywords: Polymer; cross-linked; water treatment.

1. Introduction

Research in the field of treatment of public water supply systems often pertains to technical aspects of providing treated water with assured quality, improvements in natural water management,[1] and process optimization.[2] The main technology employed in this type of process is called conventional treatment, which includes steps such as the addition of a coagulant in a rapid mix unit, followed by flocculation, sedimentation or flotation, filtration, disinfection, and pH correction.[3] One of the most important steps in water treatment is the coagulation-flocculation process, which is responsible for the removal of suspended and colloidal particles. The physicochemical parameter most commonly used to evaluate the efficiency of this treatment is turbidity. Turbidity is a measure of light dispersion caused by particles present in a solution and unsuccessful removal of these particles can interfere with disinfection, as particles may shelter pathogens.[4,5]

The coagulation-flocculation process involves the use of one or more chemicals that destabilize colloidal and suspended particles, allowing their aggregation and the formation of larger flocs, enabling their removal by filtration, sedimentation, centrifugation or flotation.[6] In the context of water treatment for supply purposes, the most studied and discussed mechanisms are adsorption and charge neutralization, as well as sweep coagulation.[7] In adsorption and charge neutralization, ions from the coagulants neutralize the charges on colloids, allowing particle aggregation as the particles are no longer subject to charge repulsion. Therefore, monitoring the Zeta potential is crucial in this approach.[8] On the other hand, the sweep coagulation mechanism is characterized by a higher coagulant dosage, which, combined with specific pH ranges, results in the formation of precipitates in the form of aluminum and iron hydroxides, such as $Al(OH)_3$ and $Fe(OH)_3$. These precipitates physically sweep the colloidal particles, forming larger flocs than those generated by adsorption and charge neutralization, allowing for better sedimentation.[9] The adsorption and charge neutralization mechanism occurs with lower coagulant concentrations and lower pH ranges, making it a more economical process in terms of reagent costs. The flocs formed, in turn, are microflocs, and this method is typically used in direct filtration treatment for water with low turbidity and low organic matter.[10,11] Low-turbidity

raw water is described as difficult to treat, as lower particle concentrations and smaller particle diameters can reduce the collision rate and consequently fail to promote satisfactory particle aggregation, and, consequently, the formation of settleable flocs.[12]

Since coagulation is a highly nonlinear process, it should be evaluated using a pair of coagulant dose and pH level, coagulation diagrams are commonly used to correlate broad pH ranges and coagulant concentrations, observing the resulting residual turbidity.[13,14] PAC is a cationic pre-polymerized inorganic coagulant based on aluminum chloride, and its benefits include the formation of rigid and heavy precipitates, which increase the sedimentation rate; less impact on pH due to the basicity of the reagent; and a lower amount of residual aluminum in the water.[15]

On the other hand, flocculation is the step in which destabilized particles aggregate with each other, by the application of a step with a slower mixing gradient, normally between 70 and 10 s^{-1},[16] called the "slow mixing step". The intention in this step is to form high-weight flocs that can be removed through sedimentation, flotation or filtration.[17] The use of additional reagents to aid the coagulation-flocculation process, also called flocculation or coagulation aids, is suggested to decrease the concentration of coagulants required and to accelerate aggregation, resulting in strong, stable, heavy flocs.[18,19] Zhang *et al.*[12] cite, also, that the use of flocculation aid can be used to prevent residual aluminum in water. Abdul *et al.*[19] cite bentonite, calcium carbonate, anionic and non-ionic polymers as examples of flocculation aids. While the use of linear polymers as flocculation aids is frequently suggested,[20] some authors have studied branched and cross-linked polymers for this aim, observing different behaviors such as higher dosages required.[21,22] Even though biopolymers are associated with enhanced flocculation,[23,24] the most common types of polymers used for this purpose are polyacrylamide-based polymers, thanks to the characteristics of acrylamide monomers, such as their high reactivity, affordable prices, wide availability, and high water solubility.[25]

Poly(acrylamide-co-sodium acrylate), herein referred to as PAAm-NaA, is an example of an acrylamide-based polymer that can be used as a flocculation aid. PAAm-NaA is an anionic cross-linked polymer with superabsorbent behavior,[26] which can be obtained through its monomers or the alkaline hydrolysis of polyacrylonitrile, polyacrylamide or poly(acrylamide-co-acrylonitrile).[27] Cross-linked polymers are employed in many fields, including biomedicine, veterinary medicine, and the food industry, and their behavior as adsorbents of undesirable particles, *e.g.*, dyes or ions such as arsenic, is well known in environmental applications.[28,29] The use of PAAm-NaA obtained from the recycling process of another polymer offers environmental benefits, underscoring the eco-efficiency principle of supporting recycling processes.[30] The purpose of this work was to apply both small and large concentrations of PAAm-NaA obtained from the polyacrylonitrile recycling process as a flocculation aid associated with PACl as a coagulant in the treatment of low turbidity water by the charge neutralization mechanism.

2. Experimental

The raw water employed in the experiments was obtained from Cachoeira do França reservoir, located in the state of São Paulo, Brazil (-23.927816, -47.195354), which is a source used in the public water supply system for Greater São Paulo after being treated in the Vargem Grande water treatment station, with a design flow rate of 6.4 $m^3 s^{-1}$, providing drinking water to around 2 million people. The land surrounding this reservoir is environmentally protected,[30] which explains the turbidity levels lower than 10 NTU of the collected water samples. Natural water with turbidity lower than 20 NTU is described as "low turbidity water" according to Cruz *et al.*[10] The chemical reagents used were PACl FinFlake produced by Finquímica, São Paulo, and PAAm-NaA obtained from the recycling process of polyacrylonitrile, produced by the Granado Institute of Polyacrylonitrile Technology (IGTPAN), São Paulo.

The coagulation was based mainly on the charge neutralization mechanism, with the addition of PACl, and no pH correction. The low turbidity of the samples is one of the characteristics of raw water employed in rapid filters, where adsorption and charge neutralization is mainly employed.[10] This mechanism has advantages such as lower coagulant demand and, consequently, lower cost of chemical reagents.[31] The application of a coagulant without the addition of a reagent for pH correction, will result in the minimum amount of coagulant required to obtain the isoelectric point, since the increase of the pH results in a more negative colloidal system.[32] Since the water studied is treated in a design flow of 6.4 $m^3 s^{-1}$,[30] the attempt to minimize the coagulant dosage by using a polymer as a flocculation aid may result in a large economy in the process. The coagulation following this path is highly dependent on zeta potential,[8] so the concentration of PACl required to reach a zeta potential equal to zero was obtained, also referred to as the isoelectric point, using a Malvern Nano Series Zetasizer zeta potential analyzer.

Following, an analysis was made to determine how the concentration of PAAm-NaA, as a flocculation aid, influences the zeta potential and the pH values. Experiments were carried out in a Floc-Control lab jar test apparatus from PoliControl with six jars, using the concentration of PACl required to reach the isoelectric point, adding PAAm-NaA as a flocculation aid. The reagent was added to the samples in small concentrations ranging from 0 to 2.5 mg L^{-1}, in incremental steps of 0.5 mg L^{-1}. In the range of large concentrations, 0 to 125 mg L^{-1} were added, varying by 25 mg L^{-1} in each jar. The proposed range of small concentrations of 0 to 2.5 mg L^{-1} of PAAm-NaA was adapted from reported concentrations of polymer used

as flocculation aids ranging from 0 to 0.4 mg L^{-1}, 2.0 to 5.0 mg L^{-1}, and 1.0 to 5.0 mg L^{-1}.[33-35] On the other hand, the proposed range of large concentrations was adapted from works that employed polymers as flocculation aids and that reported effective results, such as the concentration of 150 mg L^{-1} of a cross-linked polymer for high turbidity water,[22] and 30 mg L^{-1} for wastewater treatment.[36]

The results were interpreted using linear and quadratic regression, analysis of variance (ANOVA), and Tukey's test.

3. Results and Discussion

Table 1 describes the physicochemical characteristics of the water samples used in the experiments, while Table 2 describes the hydraulic parameters used in the jar tests. The hydraulic parameters, such as duration and gradients, were suggested by Di Bernardo and Paz.[37] Specifically, the sedimentation velocity is recommended to range from 1 to 5 cm min^{-1} to ensure a condition close to a real water treatment station, thus it was chosen the sedimentation velocity of 2.5 cm min^{-1}, the medium point of the suggested range. This velocity represents approximately a sedimentation duration of seven minutes.

Table 1. Physicochemical characteristics of raw surface water.

Parameter	Value
Turbidity (NTU)	3.3 ± 0.4
pH	6.80 ± 0.05
Zeta potential (mV)	-17.5 ± 1.2
Absorbance 254nm (a.u.)	0.079 ± 0.002

Table 2. Hydraulic parameters of the jar tests.

Hydraulic parameter	**Value**
Rapid mixing gradient	600 s^{-1}
Rapid mixing duration	20 s
Slow mixing gradient	70; 40 and 10 s^{-1}
Slow mixing duration	15 min
Sedimentation velocity	2.5 cm min^{-1}

To determine the isoelectric point, 0.5 mg L^{-1} of PACl was added to a raw water sample and its zeta potential was monitored until it reached 0 mV. Figure 1 illustrates the effect of the coagulant on the zeta potential.

The isoelectric point was reached with the use of approximately 2 mg L^{-1} of PACl, and the pH had a small decrease to 6.7. The influence of the polymer on the zeta potential of a sample already neutralized with 2 mg L^{-1} of PACl was then analyzed. Figures 2 and 3 depict the results achieved in the range of small and large concentrations, respectively.

A comparison of the two ranges of concentrations revealed that the magnitude of interference of the polymer on the zeta potential was not linear. For example, the zeta potential obtained for both 2 mg L^{-1} and 25 mg L^{-1} was close to –12 mV, indicating that the effect of the concentration

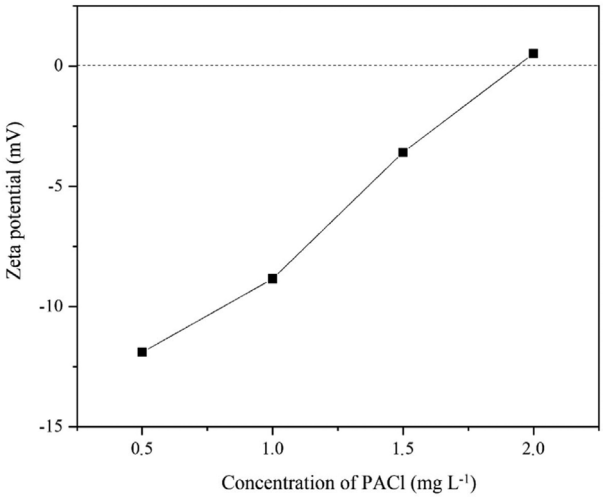

Figure 1. Zeta potential of raw water samples as a function of the concentration of PACl

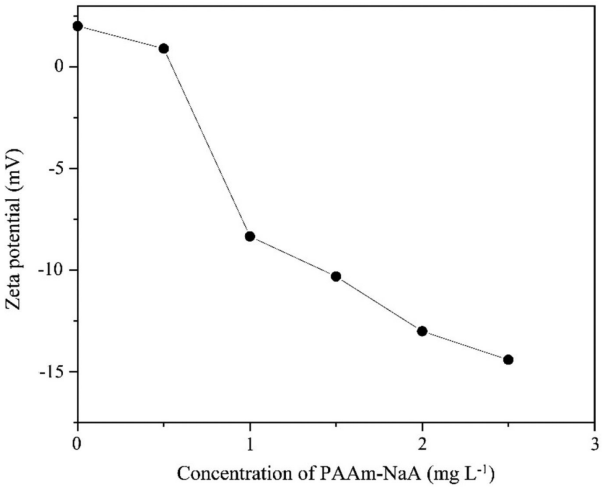

Figure 2. Variation of zeta potential of water samples neutralized with PACl in response to the addition of small concentrations of PAAm-NaA

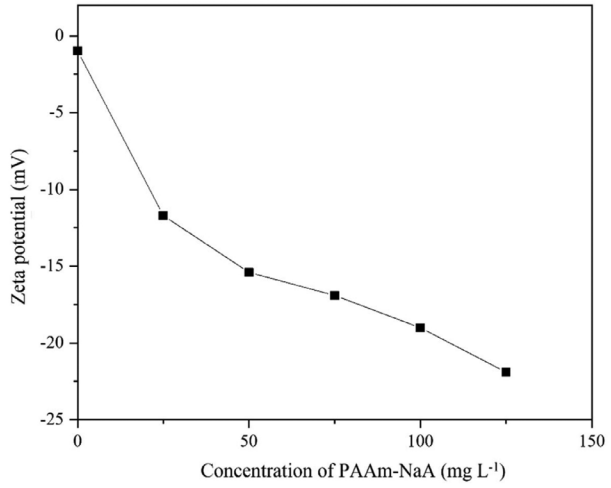

Figure 3. Variation of zeta potential of water samples neutralized with PACl in response to the addition of large concentrations of PAAm-NaA

of polymer on the zeta potential was not proportional to the concentration. Electrophoresis, which has been used as a method to measure the zeta potential, is suitable for diluted systems, but results may be affected by an increase in the concentration of high molecular mass cross-linked polymers such as PAAm-NaA.[38] Additionally, variables such as fluid viscosity, dielectric constant and temperature influence the physical relationship between zeta potential and particle mobility, affecting the measurement of the parameter.[8] Viscosity is expected to be affected by high concentrations of PAAm-NaA, a polymer whose behavior is viscoelastic.[39] Moreover, steric hindrance, which is related to the concentration of polyacrylamide superabsorbent polymers,[40] may affect the outcome of electrophoresis.[41]

Figures 4 and 5 illustrate the effect of the addition of the small and large concentrations of polymer on the variation in pH levels.

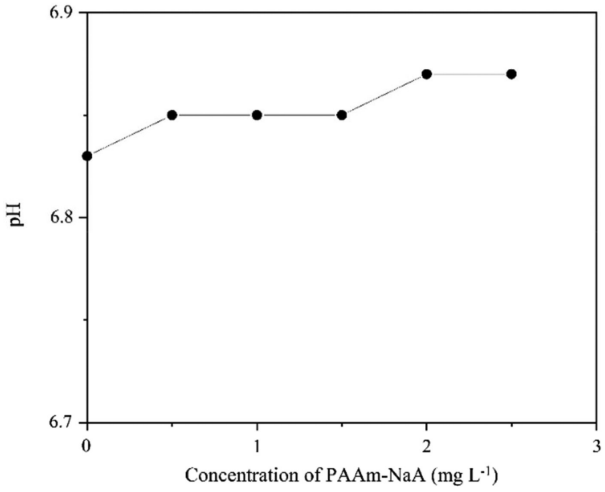

Figure 4. Influence of the addition of small concentrations of PAAm-NaA on the pH level of neutralized surface water

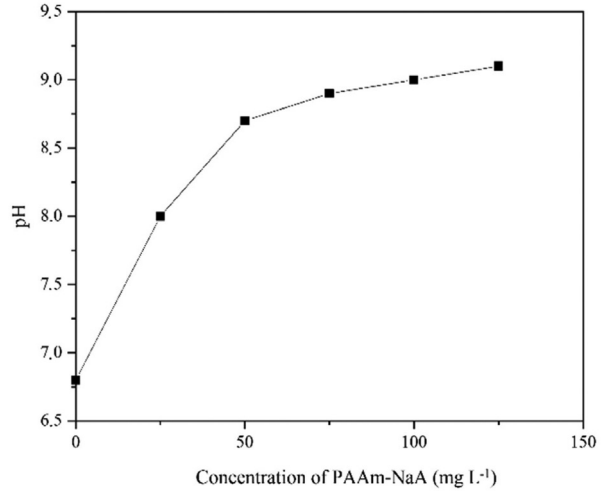

Figure 5. Influence of the addition of large concentrations of PAAm-NaA on the pH level of neutralized surface water

As can be seen, the pH level did not vary significantly in response to the addition of small concentrations of polymer, but with the addition of larger concentrations, the pH level increased from approximately 6.8 to above 9.0.

In the range of small concentrations of polymer, the jar test experiment was performed with six concentrations ranging from 0 to 2.5 mg L^{-1}, with four repetitions. It was observed visually flocs formed in all samples, with no apparent coagulation disturbance. After the residual turbidity was determined, a linear regression was performed. The sample without PAAm-NaA is also referred to as "zero." Table 3 lists the results of residual turbidity, while Figure 6 illustrates the data dispersion and linear regression.

Table 3. Residual turbidity at the isoelectric point after jar test experiments using small concentrations of PAAm-NaA.

PAAm-NaA concentration (mg L^{-1})	Repetition | Turbidity (NTU)			
	1	2	3	4
T_1 = 0.0	2.98	2.88	3.02	3.04
T_2 = 0.5	2.96	2.74	2.76	2.97
T_3 = 1.0	2.02	2.33	2.69	2.73
T_4 = 1.5	2.12	2.50	2.73	2.36
T_5 = 2.0	1.95	2.47	2.44	2.00
T_6 = 2.5	2.09	2.68	2.75	2.09

Note that the linear regression is significant, at 5% of significance, given the p-value < 0.05. However, the R^2 of 0.4477 indicates that the y variable is partially explained by the x variable. Additionally, the Pearson correlation is –0.669, which indicates a moderate correlation among the variables.[42] A quadratic regression was applied to find a better-fitting statistical model, whose results are depicted in Figure 7.

The quadratic regression was also significant, at 5% of significance, given the p-value < 0.05, and the R^2 was 0.5468, which is slightly higher than the R^2 of the linear regression. The better fit of the quadratic model indicates a possible minimum point, followed by an inverse tendency in response to increasing concentrations of PAAm-NaA.

Another approach used for the statistical interpretation of the results was an analysis of variance (ANOVA), which indicated the significant effects of the treatments. This was followed by the Tukey test, which pinpointed the significant mean differences among the treatments. The boxplot chart in Figure 8 shows the Tukey test results, where different letters indicate means with significant difference at 5% of significance.

The results of the Tukey test indicated that only the concentration of 2.0 mg L^{-1} of polymer resulted in a significant difference, at 5% of significance, between the zero sample and the ones treated with the addition of PAAm-NaA. On the other hand, the use of 2.5 mg L^{-1} resulted in a final turbidity level that did not differ significantly from the zero sample, as indicated by the minimum point, followed by the increase in final turbidity, tendency expected given the quadratic regression better fitting in comparison with the linear regression. The Tukey test results included data dispersion and identified the treated

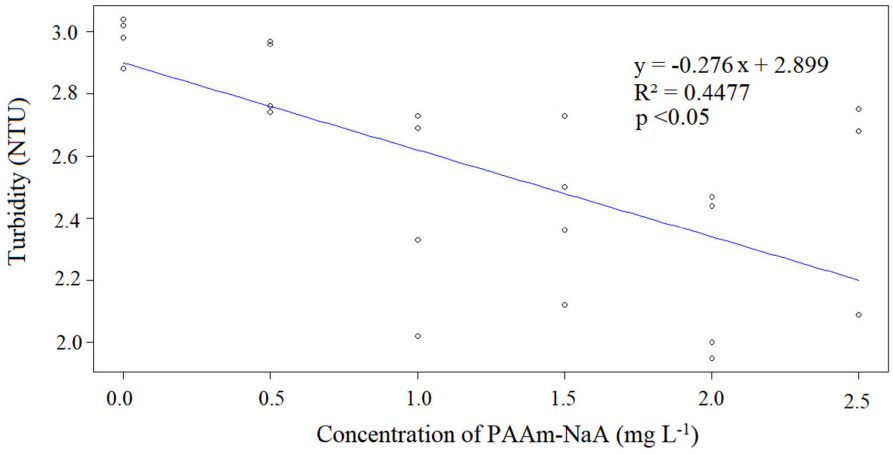

Figure 6. Linear regression of the residual turbidity after jar test experiments using small concentrations of PAAm-NaA as flocculation aid

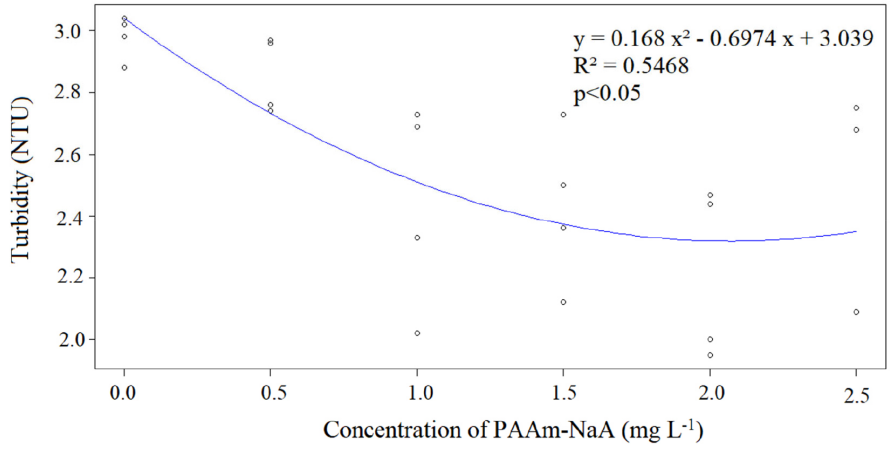

Figure 7. Quadratic regression of the residual turbidity after jar test experiments using small concentrations of PAAm-NaA as flocculation aid

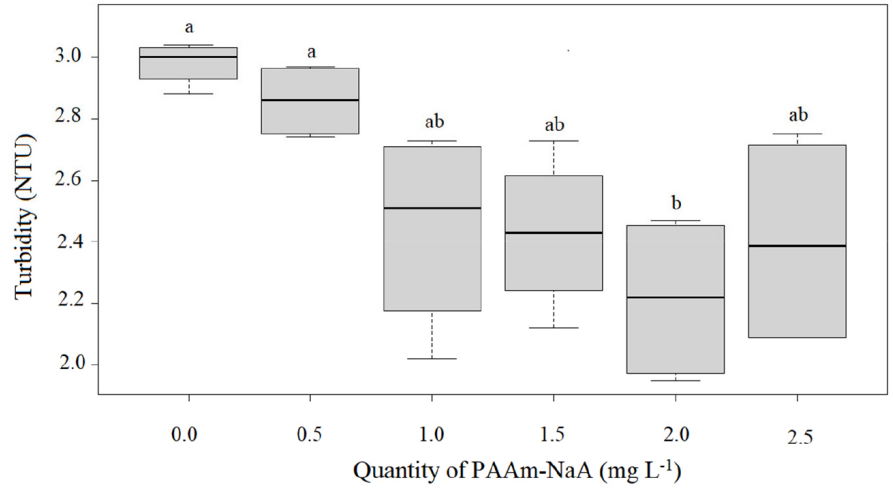

Figure 8. Boxplot chart of turbidity removal in jar test experiments using small concentrations of PAAm-NaA, including Tukey test results

sample with the highest turbidity removal, thus proving to be more useful than the linear and quadratic regression approaches employed to interpret the data, which provided low R^2 values.

The behavior of an optimum concentration of polymer as a flocculation aid has also been examined by other authors, and it has been reported that the use of excessive concentrations of polymer leave behind residual amounts in

treated water due to insufficient adsorption sites or available bridges.[43] In addition, the reflocculation behavior of broken particles reportedly improved in response to optimum concentrations of polyacrylamide-based polymers, although larger concentrations interfered with the final floc size.[35]

It is possible to note that, for the 2.0 mg L^{-1} concentration of PAAm-NaA, even with a mean residual turbidity value significantly lower than the other treatments, the reduction was only around 33% of the initial turbidity. This is partially expected, since low turbidity waters are often related to flocs that are small, light, loose, and difficult to precipitate.[12] The sedimentation velocity employed, in this case of 2.5 cm min^{-1}, also influences the low turbidity removal observed, since it is a medium sedimentation velocity of the proposed range,[37] and lower velocities provide higher residual turbidity removal for jar tests experiments in the same conditions.[14]

A jar test experiment was then carried out using the range of larger concentrations of PAAm-NaA from 0 to 125 mg L^{-1} in water samples coagulated with PACl at the isoelectric point. Figure 9 shows final turbidity as a function of the concentration of polymer.

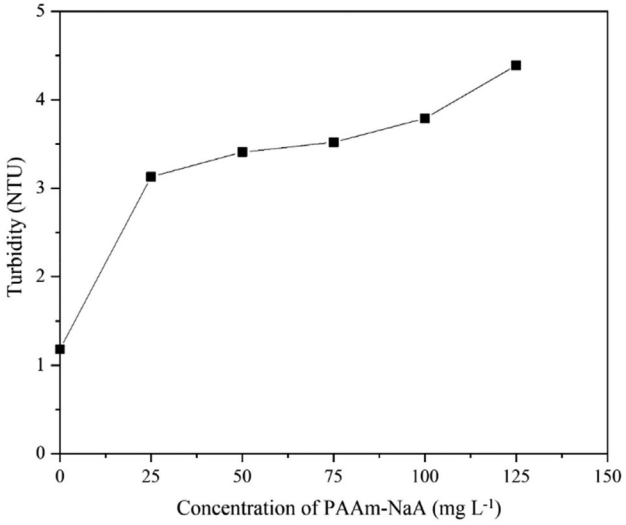

Figure 9. Residual turbidity after jar test experiments using large concentrations of PAAm-NaA

It was found visually and based on the final measured turbidity shown in Figure 9 that coagulation failed to occur when larger concentrations of polymer were used, unlike the sample without reagent (the zero sample). This absence of coagulation in the presence of larger concentrations of flocculation aid can be attributed to some factors, including the distance of the expected favorable situation of the coagulation by charge neutralization, and also the repulsion caused by the polymer: the pH increases significantly and the zeta potential becomes highly negative with the addition of large concentrations of PAAm-NaA, as stated in Figures 3 and 5, and the charge neutralization mechanism occurs under acidic conditions at very low coagulant concentrations, to ensure the aluminum in its soluble form and achieving the isoelectric point; the charge interference caused by a highly negative zeta potential in this pH, considering that the PACl concentration is fixed, possibly causes charge repulsion in particles formed;[7,11] additionally, steric hindrance was observed in treatments with high concentrations of a polyacrylamide-based super-absorbent polymers, indicating that the large concentrations of flocculation aid may cause physical disturbance.[41,44]

Some authors observed similar behavior, for example, Habibi *et al.*[45] observed the hindrance of the coagulation-flocculation process in a steel factory effluent when higher doses of polyacrylamide were applied, and Feng *et al.*[46] observed mutual repulsion of encapsulated particles when large concentrations of charged polyacrylamide-based polymers are applied in the treatment of high-turbidity water treatment.

Given that the literature focuses mainly on linear polymers as flocculation aids,[47] there is a knowledge gap concerning the use of cross-linked polymers for this purpose. A large concentration of a cross-linked polymer was reportedly effective in the treatment of high turbidity water,[22] which is contrary to the results obtained in this work. A more in-depth investigation is needed of the poor flocculation performance for large concentrations observed here, including differences in the quality of raw water and the characteristics of the polymer, such as the influence of electrical charge and pH level.

4. Conclusions

It was concluded that the use of 2 mg L^{-1} of PAAm-NaA produced better results of turbidity removal in water samples with low turbidity, coagulated with PACl at the isoelectric point and without pH adjustment, in comparison with samples without the flocculation aid. Since the polymer employed here came from the polyacrylonitrile recycling process, its use may provide environmental benefits. The statistical results of the Tukey test were more relevant to this study than those provided by linear and quadratic regression, since the R^2 of the regressions was unsatisfactory, thus representing a useful procedure for the evaluation of jar test data. Concentrations ranging from 25 to 125 mg L^{-1} of the polymer employed as a flocculation aid interfered in coagulation. This behavior may be explained by the interference in the coagulation mechanism in a high pH level, charge interference, steric hindrance, or the characteristics of the low turbidity raw water. Further studies with lower sedimentation velocity are suggested for better visualization of significantly different results among the treatments.

While this work covers the effect of the polymer as a flocculation aid associated with PACl in the treatment of low-turbidity waters through the charge neutralization mechanism without the addition of an alkalizing agent, a deeper understanding of the process requires studies related to different coagulation mechanisms, for several combinations of coagulation concentration and pH, and

also considering the treatment of high-turbidity raw water or effluents. Other variables, such as apparent color, sludge formation, and residual aluminum, are sanitary parameters of interest concerning the current potability standards in Brazil,[48] and their monitoring would provide additional information on the effectiveness of the treatment.

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