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Coagulation Activity of the Seed Extract from *Zygia cauliflora* (WILLD.) KILLIP applied in Water Treatment

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ABSTRACT

The use of natural coagulants is a promising alternative to replace or assist chemical coagulants due to its numerous advantages. This paper evaluates the effectiveness of a natural coagulant in water treatment. The coagulant was extracted from the *Zygia cauliflora* (Willd.) Killip seed in saline solutions and defatted using 95% ethanol. The tests were conducted using different concentrations of the coagulant (0.1, 0.5, 1.0, 2.0, 3.0, and 4.0 g L^{-1}) and of NaCl (0.0 M, 1.0 M, and 5.0 M). The results showed that the use of 2 g L^{-1} of coagulant and 1.0 M of the salt achieved an effectiveness of 20% and 70%, respectively, of color and turbidity removal. The analysis showed an effectiveness of 70% and 30%, respectively, when removing the UV_{254} compound and DOM (dissolved organic matter). The mechanism for turbidity removal by the coagulants prepared with aqueous solution and with saline solution at 1.0 M of NaCl is supposed to be adsorption and charge neutralization, respectively, following the Freundlich and Langmuir models. However, the coagulant prepared with saline solution at 5.0 M of NaCl tends to form a netlike structure followed by turbidity removal through a sweep coagulation mechanism. Future papers should therefore focus on the use of *Zygia cauliflora* (Willd.) Killip as an alternative for replacement or use as an auxiliary chemical coagulant.

Keywords: Amazon seed, flocculation, natural coagulant.

Atividade Coagulante do Extrato de Sementes de *Zygia cauliflora* (WILLD.) KILLIP no Tratamento de Água

RESUMO

O uso de coagulantes naturais é uma alternativa promissora para substituir ou auxiliar coagulantes químicos devido às suas inúmeras vantagens. Este artigo avalia a eficácia no uso de um coagulante natural no tratamento de água. O coagulante foi extraído da semente de Killip Zygia cauliflora (Willd.) em soluções salinas e desengordurada com etanol a 95%. Os testes foram conduzidos utilizando diferentes concentrações do coagulante (0,1, 0,5, 1,0, 2,0, 3,0 e

4,0 g L^{-1}) e de NaCl (0,0 M, 1,0 M e 5,0 M). Os resultados mostraram que o uso de 2 g L^{-1} de coagulante e 1,0 M de sal alcançou uma eficácia de 20% e 70%, respectivamente, na remoção de cor e turbidez. A análise mostrou uma eficácia de 70% e 30%, respectivamente, na remoção do composto UV254 e DOM (matéria orgânica dissolvida). Além disso, o mecanismo de remoção da turbidez pelos coagulantes preparados com solução aquosa e com solução salina a 1,0 M de NaCl é suposto ser adsorção e neutralização de carga, respectivamente seguindo os modelos de Freundlich e Langmuir. No entanto, o coagulante preparado com solução salina a 5,0 M de NaCl tende a formar uma estrutura semelhante a uma rede seguida pela remoção da turbidez por mecanismo de coagulação por varredura. Portanto, trabalhos futuros devem se concentrar no uso de Zygia cauliflora (Willd.) Killip como uma alternativa para substituição ou uso como coagulante químico auxiliar.

Palavras-chave: coagulante natural, floculação, semente da Amazônia.

1. INTRODUCTION

According to the World Health Organization (WHO) and its member countries, "every person, at any stage of development and socio-economic conditions, has the right to access an adequate supply of potable and safe water". In this context, 'safe' refers to a water supply which does not represent a health risk, with enough quantity to meet all household needs, that is continuously available, and is affordable. If the purpose is to improve public health, such conditions must be considered when defining and maintaining water quality and water supply programs (OPS, 2001).

During the water treatment process, one of the first steps is coagulation, which aims to reduce impurities to make the water potable. Since it is a delicate part of the treatment, a strict control of the dosage of these chemical products is necessary. However, an increase of these dosages is required considering the high demand of potable water and a considerable decrease in the quality of water resources. The decision of coagulant is often based on economic factors, related to suitability, raw water, treatment technology, and cost (Richter *et al.*, 1991).

According to Richter (2009), the most commonly used coagulants are iron and aluminum-based coagulants, due to their reaction with the natural alkalinity of raw water. According to several studies, there is evidence of a relationship between the content of aluminum in treated water and the increasing incidents of neurological diseases. According to Flaten (2001), aluminum plays an important role in the etiology and pathogenesis of Alzheimer's, but this connection is still under debate. Bondy (2010) states that there are analyses that link Parkinson's disease and other chronic neurodegenerative diseases as well.

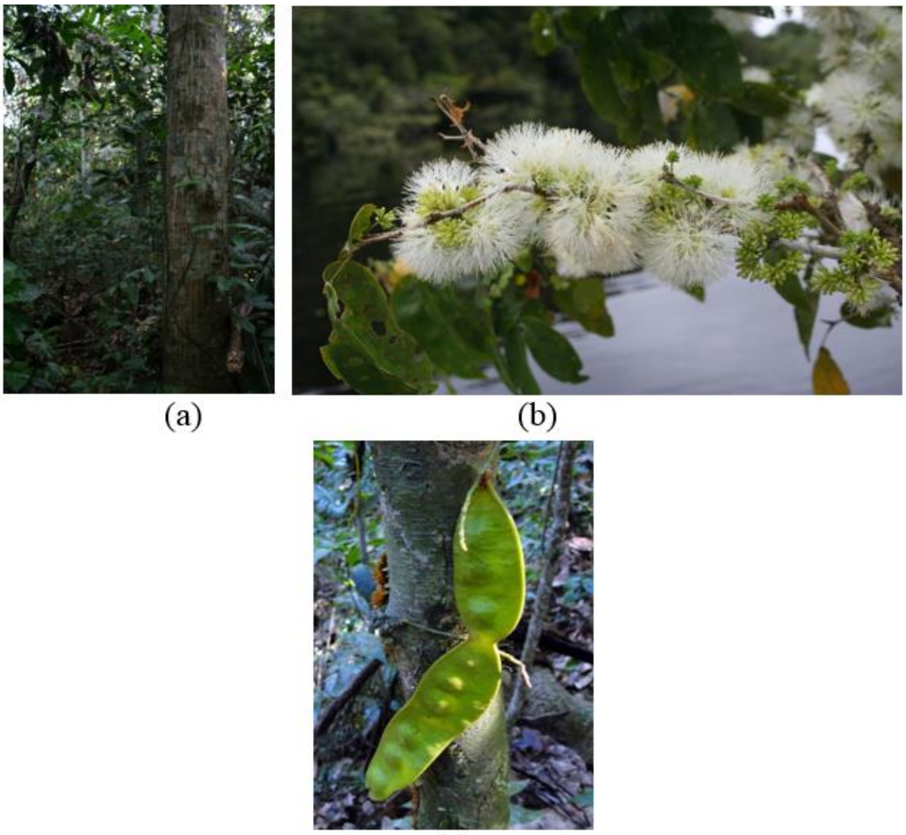
Moreover, the high sensitivity of inorganic coagulants to the water pH and the possibility of secondary contamination of drinking water with traces of toxic synthetic polymeric coagulants or residual iron and aluminum ions are the main challenges for flocculation–coagulation water treatment processes (Bratskaya *et al*., 2004).

In view of these disadvantages, it is desirable to replace chemical coagulants with natural coagulants. Using natural plants as coagulants has many advantages, among which are their profitability, biodegradability, and lack of requirement for a high pH for water treatment (Bratskaya *et al*., 2004).

Thus, the *Moringa oleifera* Lam (moringa) seed has been gaining prominence as a natural coagulant in water treatment since the species contains an edible oil, a water-soluble substance with protein in its organic composition, and can act as an effective coagulant Salazer Gámez *et al*., 2015). However, *Moringa oleifera* is an Indian seed, so its availability could be restrictive for water treatment worldwide. It is therefore important to research the applicability of regional leguminous seeds that could play the same role as *Moringa oleifera Lam*.

Many plants have recently been identified as sources of natural polymers for the treatment of various types of water, including *Plantago ovata*, *Musa* ABB, and *Strychnos potatorum* (Ramavandi, 2014; Tirado *et al*., 2017; Gaikwad and Munavalli, 2019).

The use of regional coagulants is studied to evaluate their applicability regarding the difference in coagulation capacity, cheapness, variety, and ease of access. This research has attempted to use *Zygia cauliflora* (Willd.) Killip. seeds as an abundantly available, low-cost, and renewable precursor for producing a coagulant for improving water quality. *Zygia cauliflora* (Willd.) Killip. was considered because it is one of the most abundant species in undisturbed forests and with the highest density throughout the Amazon rainforest, especially in permanent preservation areas of the Moju River Basin (Oliveira *et al*., 2016). *Zygia cauliflora* (Willd.) Killip. trees, flowers and seeds are shown in Figure 1.



(a) (b)

(c)

Figure 1. *Zygia cauliflora* (Willd.) Killip. tree (a), flowers (b) and seeds (c).

Therefore, this paper evaluates the coagulation potential of aqueous and saline solutions obtained from *Zygia cauliflora* (Willd.) Killip. seeds to determine water color, turbidity, and organic compounds absorbed at 254 nm (UV_{254} compounds) and to remove dissolved organic matter (DOM). The effect of the operational parameters including coagulant dosage and NaCl concentration was studied to better understand the coagulation process using *Zygia cauliflora* (Willd.) Killip. seeds.

2. MATERIALS AND METHODS

Raw water samples were collected from a public water treatment system (WTS), Paraná Sanitation Company (Companhia de Saneamento do Paraná, SANEPAR), located in Campo Mourão, State of Paraná, Brazil. The surface water originates from the Campo River Basin.

Characterization before and after the treatment was carried out using quality parameters such as color, turbidity, UV_{254}, and dissolved organic matter (DOM).

2.1. Color

The apparent color was determined based on the procedures described in Standard Methods for the Examination of Water and Wastewater[10] using the Digimed colorimeter equipment, Model DM-COR.

2.2. Turbidity

The turbidity was determined based on the procedures described in Standard Methods for the Examination of Water and Wastewater (APHA *et al.*, 2012) using a turbidimeter (PoliControl equipment, Model AP2000 iR).

2.3. Dissolved organic matter parameters

The UV_{254} and UV_{272} compounds were determined using the HACH spectrophotometer, Model DR5000.

Equation 1 was used to determine the concentration of dissolved organic matter from UV_{272} results, a method proposed previously (Khan *et al*., 2014).

$$\text{DOM (mg L}^{-1}) = 518.93 \times \text{Absorbance (272nm)} + 1.065 \quad (1)$$

The detection of dissolved organic matter (DOM) in water is necessary since it consists of a set of substances originated from the excretion, secretion, and intermediary process of the decomposition of terrestrial and aquatic organisms (Di Bernardo *et al*., 2002), which influences water appearance among other consequences.

2.4. Extraction and preparation of the coagulant

The method used for the extraction and preparation of the coagulant in saline solution was described by Sánchez-Martín *et al.* (2010).

The peels of the *Zygia cauliflora* (Willd.) Killip seeds were manually removed and ground in a domestic blender. The powder was defatted in 95% ethanol with a mass/volume ratio of 1:10 using a magnetic stirrer for 45 minutes at 150 rpm.

The supernatant was manually separated and placed in glass tubes so that the resulting powder would dry in an oven at 60°C for three days.

A coagulant was prepared in distilled water, which was denominated aqueous solution. One gram of the dried seed was added in a glass beaker containing 0.1 L. The coagulant was extracted by turbolysis for 3 minutes and then stirred in a magnetic stirrer for 30 minutes at 150 rpm. The obtained solution was vacuum filtrated on quality filter paper and then on a 0.9 µm pore glass fiber membrane. The dried seeds and turbolysis process are shown in Figure 2.

The same procedure was performed for the saline solutions of 1 M and 5 M.

All tests were performed in duplicate.

Figure 2. Dried seeds (a) and turbolysis process (b).

2.5. Coagulation/flocculation test

The coagulation/flocculation tests were performed on a Jar test device (Nova Ética) with a rotating regulator of mixing rods and jars with capacity of 1 L (Figure 3).

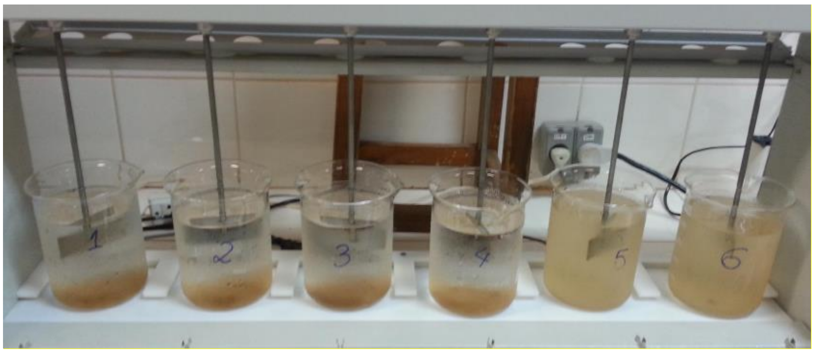


Figure 3. Coagulation/flocculation performed on Jar test equipment.

The use of the Jar Test allows the determination of the ideal dosage of coagulant or coagulation aid necessary to cause raw water clarification (Di Bernardo *et al*., 2002).

Predetermined amounts of coagulant solution were added to each jar (Table 1), using an automatic pipette. The water used was at room temperature, without any pH correction or adjustment.

Table 1. Coagulant concentration solution in each jar.

	Jar 1	Jar 2	Jar 3	Jar 4	Jar 5	Jar 6
Coagulant concentration (mg L^{-1})	0.1	0.5	1.0	2.0	3.0	4.0

The rapid mixing was set at 1 minute, with gradient speed of 120 rpm, while the slow mixing was set at 15 minutes at 60 rpm. The settling time was established at 15 minutes. The methodology used for the tests was described in Di Bernardo *et al*. (2002).

Subsequently, the color, turbidity, UV_{254} compounds, and DOM were measured to verify the efficiency of the process by comparing the results with raw water.

2.6. Mechanism evaluation

In order to evaluate the mechanism of coagulation/flocculation process using aqueous and saline solutions obtained from *Zygia cauliflora* (Willd.) Killip, Langmuir and Freundlich isotherms were employed to describe adsorption equilibrium. This evaluation was performed using turbidity removal data and was adapted from Fahmi *et al.* (2011), Mateus *et al*. (2020), Zhou *et al*. (2018) and Sia *et al.* (2020).

The theoretically derived Langmuir model assumes adsorption energies are uniform and independent of surface coverage and complete coverage of surface by a monolayer of adsorbate indicates maximum adsorption. The linear form of Langmuir isotherm is given in Equation 2:

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{q_m K_l C_e} \tag{2}$$

Where:

q_e = ratio of turbidity adsorbed to the dosage of coagulant employed
C_e = equilibrium concentration of turbidity
q_m = maximum adsorption capacity of the coagulant employed
K_l= Langmuir constant, related to the energy of adsorption

The value of Langmuir constants (slope) and maximum adsorption capacity (intercept) were obtained from the linear correlation plots between $1/q_e$ with $1/C_e$.

On the other hand, the Freundlich model is empirical, encompassing the heterogeneity of adsorbent surface, the exponential distribution sites and their energies. The linear equation of the Freundlich isotherm is given in Equation 3:

$$lq_e = lK_f + \frac{1}{n} lC_e \quad (3)$$

Where:

q_e = ratio of turbidity adsorbed to the dosage of coagulant employed
C_e = equilibrium concentration of turbidity
K_f = Freundlich constant indicative of the relative adsorption capacity of the adsorbent
1/n = constant that indicates the adsorption intensity

The value of Freundlich constants (slope) and adsorption intensity (intercept) were obtained from the linear correlation plots between ln q_e with ln C_e.

3. RESULTS AND DISCUSSION

3.1. Characterization of the raw water

The results of the raw water characterizations are presented in Table 2 according to color, turbidity, UV_{254} compounds, and DOM.

Table 2. Results of the physical-chemical analysis of the raw water.

	Raw water
Color (uH)	92.2
Turbidity (NTU)	76.3
UV_{254}(abs)	1.367
DOM (mg L^{-1})	1,183.2

According to Gaikwad *et al*. (2019), the composition of typical raw water samples collected from a river has a turbidity of 5-50 NTU. The turbidity of the raw water collected in this study was 76.3 NTU, which can be considered high.

On the other hand, the raw water characteristics are similar to reports found in literature. Ramavandi (2014) evaluated a coagulant extracted from *Platago ovata* and used a raw water with turbidity of 76 NTU, TOC (total organic carbon) of 8.6 mg L^{-1} and DOC of 4.02 mg L^{-1}.[8] Lima *et al*. (2017) used raw water with color of 174 UC and turbidity of 97 NTU when studying *Abelmoschus esculentus* as a flocculation aid. Malik (2018) used raw water with higher levels of turbidity, 250 NTU.

3.2. Results of the tests with Zygia cauliflora (Willd.) Killip as a potential natural coagulant

Figure 4 presents the results of color, turbidity, UV_{254nm} absorption compounds, and DOM removal obtained with the coagulants prepared from *Zygia cauliflora* (Willd.) Killip in different dosages and different concentrations of NaCl. The results with saline solutions were similar, showing no considerable variations between 1.0 M and 5.0 M solutions. The results of removing the color, turbidity, and UV_{254} were approximately 20%, 70%, and 70%, respectively, with no considerable differences between the dosage employed. Despite this, the coagulant prepared with 5.0 M of NaCl removed no DOM at any dosage, while the coagulant prepared with 1.0 M of NaCl removed approximately 21% of DOM at all dosages.

On the other hand, the coagulant obtained from the aqueous solution removed no color, while removing turbidity, UV_{254}, and DOM at approximately 45%, 70%, and 30%, respectively.

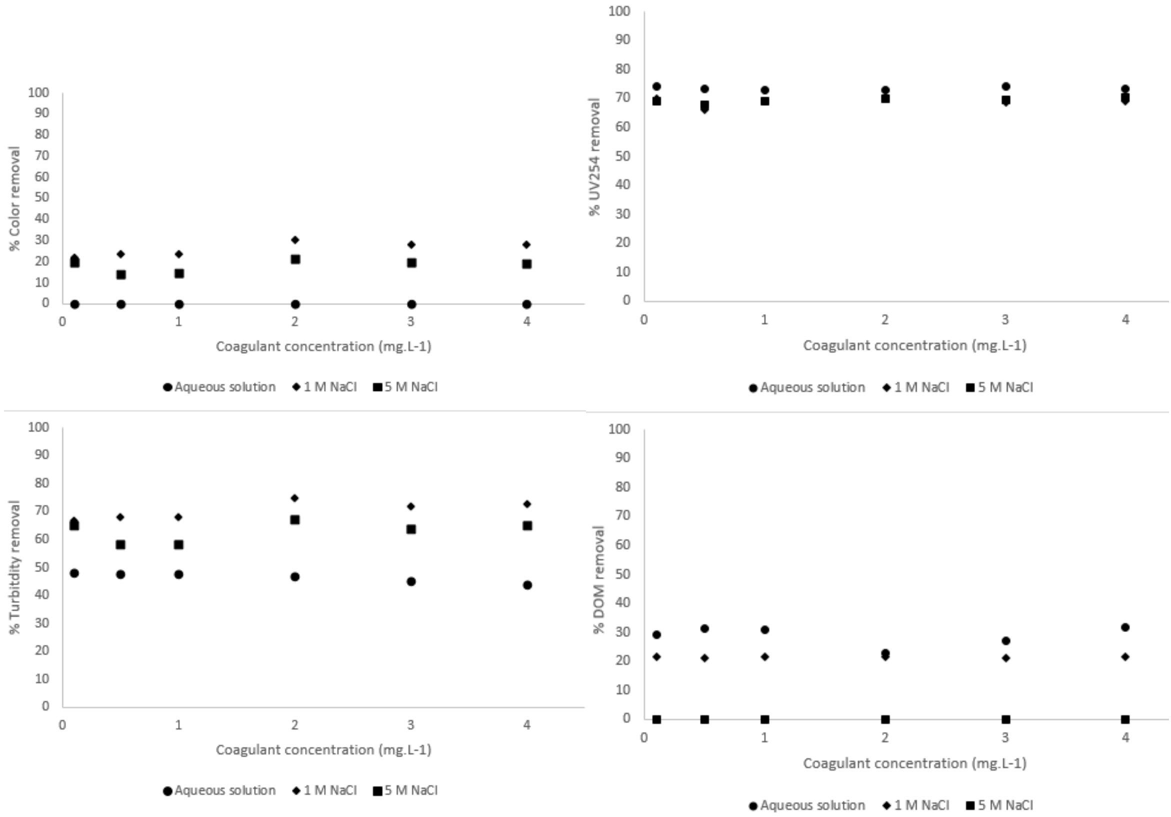


Figure 4. Efficiency of removal of parameters using different dosages of coagulants prepared from *Zygia cauliflora* (Willd.) Killip.

Considering these results, the coagulants prepared with a saline solution at 1.0 M of NaCl were more efficient than those prepared with a 5.0 M solution. Furthermore, better results were obtained for the coagulant concentration of 2 g L^{-1}, even when compared to concentrations of 3 g L^{-1} and 4 g L^{-1}, which also showed reasonable results of color and turbidity removal. Thus, a more satisfactory efficiency was obtained for the saline solution with 1.0 M concentration of NaCl for the analyzed parameters, presenting 30.4% and 74.5% of removal, respectively, for color and turbidity.

Moreover, the saline solutions presented greater efficiency in removing color and turbidity when compared with the aqueous solution, as observed by Madrona *et al*. (2010), Nkurunziza *et al*. (2009), and Nishi *et al*. (2011).

Nkurunziza *et al*. (2009) reached 99.8% of turbidity removal using *Moringa oleifera* coagulant extracted with saline solution at 1.0 M, while Nishi *et al*. (2011) obtained 96.4% of color removal in the same conditions. These results suggest that *Zygia cauliflora* (Willd.) Killip could be applied as a coagulation/flocculation aid, instead of a primary coagulant.

The aqueous solution presented better UV_{254} compound and DOM removal, removing 74.3% of UV_{254} compounds at the concentration of 3.0 mg.L^{-1} and 31.7% of DOM at the concentration of 4.0 mg L^{-1}.

The presence of NaCl in the water showed that increasing the ionic strength results in a greater amount of extracted compounds, but does not affect coagulation activity, which can be observed in the results of UV_{254} and DOM removal (Kukic *et al*. 2015).

Previous work found in the literature shows that the coagulant obtained from *Plantago ovata* at a dosage of 0.250 mg L^{-1} showed turbidity removal of 36% and 99%, respectively, when using crude and treated extract (Ramavandi, 2014).

Tirado *et al*. (2017) applied higher dosages (5-200 mg L^{-1}) of starch obtained from *Musa* ABB as a coagulant and achieved color and turbidity removals from 90 to 99%.[9] The difference in results when compared to this work could be due to the higher dosages used by the authors.

Valverde *et al*. (2018) evaluated the synergistic effect between *Moringa oleifera* and APC (aluminum polychloride) for coagulation/flocculation and observed lower removal efficiencies at greater proportions of moringa, with 70.1%, 75.1%, and 56.7% for color, turbidity, and UV_{254} absorption, respectively, which suggests that natural coagulants present lower removal efficiencies. Furthermore, Valverde *et al*. (2018) obtained a turbidity removal close to that observed in this work with the coagulant prepared with saline solutions, from 65 to 70%. Moreover, the removal of UV_{254} compounds achieved in this paper are higher, at 70%, for aqueous and saline solutions than the results observed by Valverde *et al*. (2018), even though the authors employed a synthetic coagulant in association with *Moringa oleifera*.

3.3. Mechanism evaluation results

Figures 5 and 6 show the respective results of Langmuir and Freundlich adsorption equilibrium models for turbidity removal, and Table 3 presents the R^2 obtained for each model and coagulant condition.

It was observed that the coagulants prepared with aqueous solution and with a saline solution at 1.0 M of NaCl show positive correlation with Langmuir and Freundlich models. The coagulant prepared in aqueous solution presented R^2 of 0.77 and 0.98, respectively, for the Langmuir and Freundlich models, while the coagulant prepared with a saline solution at 1.0 M of NaCl showed R^2 of 0.63 for and 0.54, respectively, for the Langmuir and Freundlich models. This indicates that adsorption and charge neutralization mechanisms are most likely involved in the processes (Fahmi *et al*., 2011).

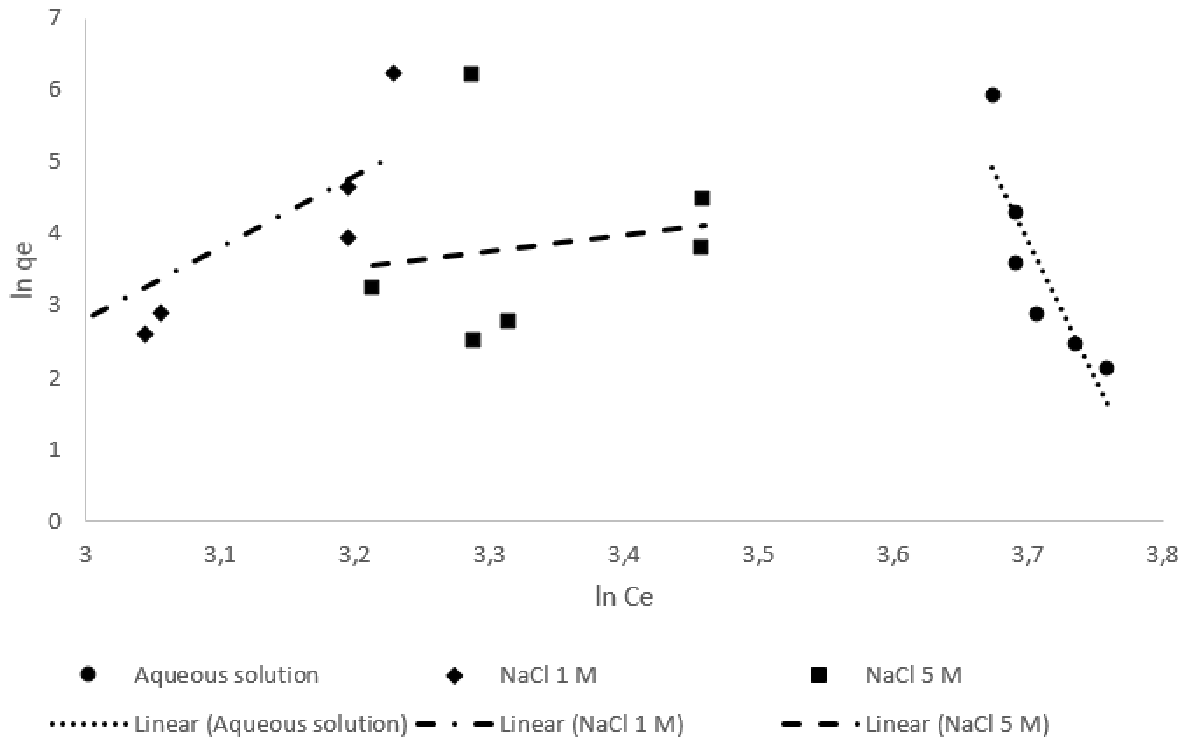


Figure 5. Langmuir adsorption isotherm for turbidity removal.

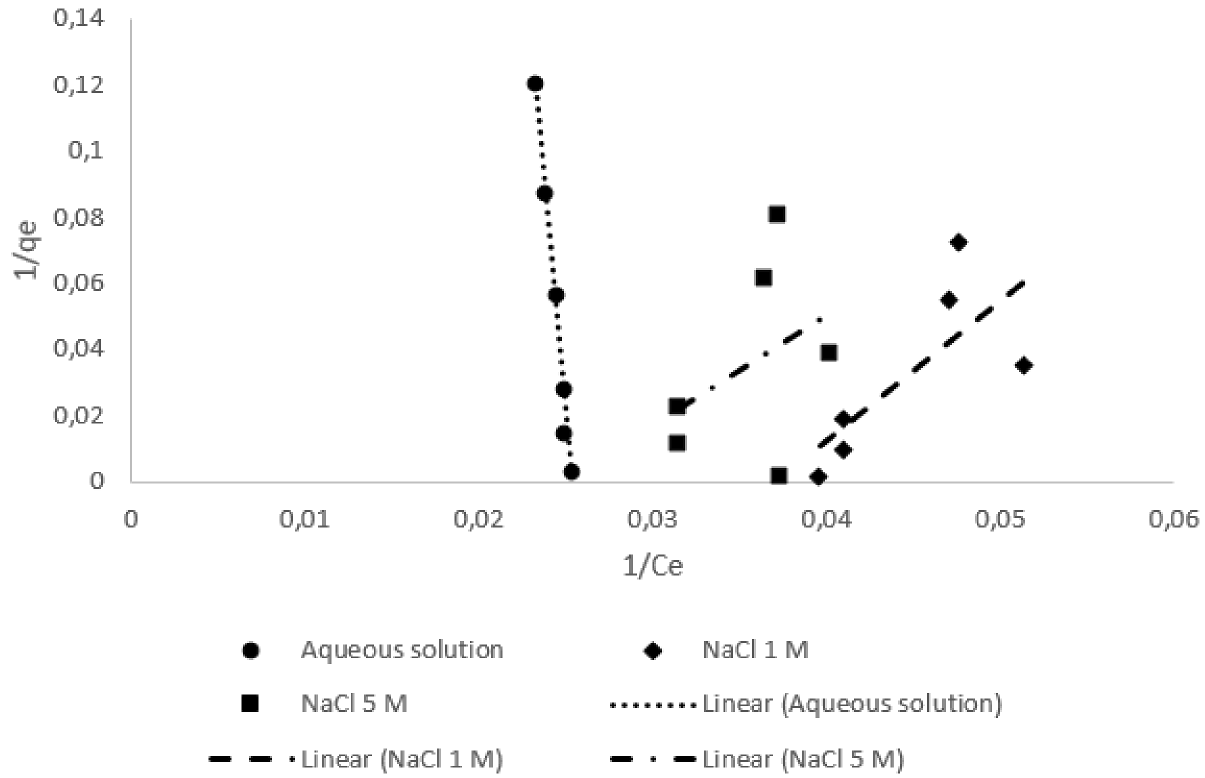


Figure 6. Freundlich adsorption isotherm for turbidity removal.

Table 3. R^2 results for Langmuir and Freundlich models applied to turbidity removal.

Coagulant condition	R^2 (Langmuir)	R^2 (Freundlich)
Aqueous solution	0.77	0.98
NaCl 1 M	0.63	0.54
NaCl 5 M	0.03	0.15

Furthermore, the mechanism for turbidity removal for the coagulant obtained from *Zygia cauliflora* (Willd.) Killip. Extract in aqueous solution was found to be due to adsorption, approximating to the Freundlich type (>R^2), which indicates that adsorption occurs on heterogeneous surface and the adsorption energy decreases logarithmically, as the surface of adsorbent becomes covered by the solute (Fahmi *et al*., 2011; Mateus *et al*., 2020; Zhou *et al*., 2018; Sia *et al*., 2020).

With the experimental data presented, it is possible to observe that the Langmuir model presented a better fit (>R^2) for the coagulant with a saline solution at 1.0 M of NaCl, suggesting homogeneous and monolayer adsorption characterizing a more selective adsorption process, as reported by Mateus *et al*. (2020). The decay in R^2 values when compared with aqueous solution suggests that the mechanisms involved tend to change when saline solution is used to extract the coagulant from *Zygia cauliflora* (Willd.) Killip. seeds.

However, these models were not found to fit for the coagulant prepared with a saline solution at 5.0 M of NaCl, which presented R^2 for the Langmuir model of 0.03 and for the Freundlich model of 0.15. This occurrence indicates that the coagulant obtained from *Zygia cauliflora* (Willd.) Killip. at higher saline concentrations more probably form a netlike structure followed by turbidity removal through a sweep coagulation mechanism, as previously described (Fahmi *et al*., 2011).

4. CONCLUSIONS

This study showed that the concentration of NaCl slightly influenced the removal of water parameters such as color, turbidity, UV_{254} compounds, and DOM using the coagulants obtained from *Zygia cauliflora* (Willd.) Killip. The optimum dosage of coagulant for color and turbidity removal was 2.0 mg L^{-1} with 1.0 M NaCl, which is very low and removed 21% of DOM from treated water. The removal of color, turbidity, UV_{254} compounds, and DOM were 30.4%, 74.5%, 70.1%, and 21.5%, respectively, in this optimum dosage.

Furthermore, aqueous solution in concentrations of 3.0 and 4.0 mg L^{-1} removed UV_{254} compounds and DOM more efficiently, with values of 74.3% and 31.7%, respectively.

Moreover, the mechanism for turbidity removal by the coagulants prepared with aqueous solution and with a saline solution at 1.0 M of NaCl is supposed to be adsorption and charge neutralization, with the adsorption isotherm following the Freundlich model for aqueous solution and the Langmuir model for saline solution at 1.0 M of NaCl. Therefore, the coagulant prepared with a saline solution at 5.0 M of NaCl tends to form a netlike structure followed by turbidity removal through sweep coagulation mechanism.

According to the data presented, the *Zygia cauliflora* (Willd.) Killip seed can be used as a natural coagulant and is a good alternative to improve water quality for human supply.

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