APPLICATION OF DOSES OF FREEZE-DRIED EXTRACT FROM MORINGA OLEIFERA SEEDS IN WATER TREATMENT

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ABSTRACT

Despite the proven performance and cost-effectiveness of chemical coagulants, natural coagulants are being studied. Among them, Moringa oleifera has been investigated more intensively as it contains proteins that are efficient in the alternative treatment of water for human consumption. The objective of the research was to use water with pH, turbidity and apparent color with levels above that allowed by Brazilian legislation and to treat it with freeze-dried extract of moringa seeds in different doses, selecting the best one for each parameter studied. The study was conducted at the Laboratory for Processing and Storing of Agricultural Products, at the Federal University of Campina Grande, Campina Grande, PB, Brazil. The extract was obtained as a result of the freeze-drying method. Three water quality parameters were evaluated: pH, turbidity and apparent color. Doses of 12, 16, 18 and 24 g/500 mL of freeze-dried extract of moringa seeds were used. Jar tests were performed at 160 rpm. The application of different doses of the freeze-dried extract resulted in pH according to the limits required by Brazilian legislation for drinking water. Based on the best efficiencies in pH, reduction of turbidity and color, doses of 24 g/500 mL were selected for pH and turbidity and 16 g/500 mL for the apparent color.

Palavras-chave: Coagulante natural, Pó, Secagem, Sementes

APLICAÇÃO DE DOSES DE EXTRATO LIOFILIZADO DE SEMENTES DE MORINGA OLEIFERA NO TRATAMENTO DE ÁGUA

RESUMO

Apesar do desempenho e custo-eficácia comprovados dos coagulantes químicos, estão sendo estudados coagulantes naturais, dentre eles, a Moringa oleifera vem sendo investigada mais intensamente, por conter proteínas que são eficientes no tratamento alternativo de águas para consumo humano. Objetivou-se com a pesquisa utilizar água com pH, turbidade e cor aparente com níveis acima do permitido pela legislação brasileira e tratá-la com extrato liofilizado de sementes de moringa em diferentes doses selecionando a melhor para cada parâmetro estudado. Conduziu-se o trabalho no Laboratório de Processamento e Armazenamento de Produtos Agrícolas, da Universidade Federal de Campina Grande, Campina Grande, PB, Brasil. Obteve-se o extrato pelo método de secagem por liofilização. Avaliaram-se três parâmetros de qualidade da água: pH, turbidez e cor aparente. Utilizaram-se as doses de 12, 16, 18 e 24 g/500 mL do extrato liofilizado de sementes de moringa. Realizaram-se os ensaios em Jar Test, a uma rotação de 160 rpm. A aplicação de diferentes doses do extrato liofilizado resultou em pH de acordo com os limites exigidos pela legislação brasileira para água potável. Com base nas melhores eficiências no pH, redução de turbidez e cor selecionou-se as doses de 24 g/500 mL para pH e turbidez e 16 g/500 mL para a cor aparente.
INTRODUCTION

The consumption of drinking water is essential for human survival. Zhang et al. (2013) mention that in recent years the problems related to the pollution of water sources have worsened. Domestic, industrial and agricultural effluents are major sources of pollutants and require appropriate techniques for the treatment of water destined for the human consumption.

Generally, the first stage of water treatment is coagulation, which probably significantly influences the next stages of treatment. The purpose of coagulation and flocculation, as a consequence, is to significantly increase the sedimentation speed of colloids present in water (Franco et al., 2017). Coagulation is largely affected by factors such as turbidity, pH, amount of coagulant and mixing conditions, and it may result in an uneconomical and harmful process carried out with a high concentration of coagulant or unnecessary doses of pH correctors, or even with the insufficient removal of particles (Mateus et al., 2017). Given the importance of this separation process, it is essential to develop studies on the behavior of doses of natural coagulants at this stage.

Over the years, interest in the use of natural coagulants in water treatment has increased due to their advantages over chemical coagulants. One of these natural coagulants is obtained from the seeds of Moringa oleifera Lam., which contains active agents with excellent activity and coagulant properties (Muyibi et al., 2013). The efficiency of moringa seed, as a natural coagulant for water treatment, has already been proven by authors such as Madrona et al. (2010), Vieira et al. (2010), Nishi et al. (2011) and Schmitt et al. (2018). In addition, the moringa’s ability to treat water for public supply, removing turbidity and color, was also verified by practical experiences (Muniz et al., 2015).

When compared to chemical coagulants, M. oleifera has a number of advantages such as: it does not require pH and alkalinity adjustments; it does not cause corrosion problems; it is low-cost, biodegradable and produces a low volume of sludge (Bongiovani et al., 2013; Adeniran et al., 2017). In addition, several studies using the moringa seed solution have shown that its seeds are not toxic to humans and animals (Váz et al., 2010).

According to a study by Muyibi and Evison (1995), the efficiency of the moringa in the treatment of water was a result of the increase in coagulant dose. The reverse was observed by Santos et al. (2013) in which the use of moringa seed in lower concentrations together with a filtration process resulted in high efficiencies of turbidity removal, whereas high concentrations, as observed by Lo Monaco et al. (2012), are used in wastewater treatment, in which the turbidity values are significantly higher than in natural waters. Valverde et al. (2018) when using higher proportions of moringa in the coagulation/flocculation and sedimentation processes observed lower efficiencies for removing apparent color and turbidity.

Proteins called lectins, present in the moringa seeds, and which are responsible for the water coagulation/flocculation process, lose their effect in a short time (Katayon et al., 2006). In this regard, techniques should be adopted to preserve this effect for a long period of time. In materials that are sensitive to heat, such as moringa, the selection of an appropriate drying method is necessary (Hamid et al., 2016). Among the drying methods, freeze-drying is used to guarantee microbiological stability, reduce deterioration due to chemical reactions, facilitate storage and reduce transportation costs (Salazar et al., 2017).

Thus, the objective of the research was to use water with pH, turbidity and apparent color with levels above that allowed by Brazilian legislation and to treat it with freeze-dried extract of moringa seeds in different doses, selecting the best for each parameter studied.

MATERIAL AND METHODS

Obtaining freeze-dried powder

This study was conducted at the Agricultural Products Processing and Storage Laboratory, at the Federal University of Campina Grande, Campina Grande, Paraíba, Brazil. M. oleifera seeds were manually peeled and crushed in a domestic blender. A benchtop freeze dryer at -54 °C was used to obtain the extract, at a pressure of 110 mmHg for 72 h.
Obtaining water artificially

Three water quality parameters were evaluated: pH obtained by adding 0.25 g of sodium carbonate to distilled water in order to obtain a higher value than allowed, turbidity and apparent color obtained with the application of 0.5 g of clay (Table 1). The maximum permitted values correspond to those established by Ordinance No. 5 of the Ministry of Health (BRASIL, 2017).

Coagulant dosage test

Doses of 12, 16, 18 and 24 g/500 mL of the freeze-dried extract of the moringa seeds were used. Jar Test equipment was used in the coagulation/flocculation and water sedimentation tests. There were two different times in the process ($T_1 = 3$ min: homogenization of the samples); ($T_2 = 10$ min: coagulation/flocculation), at a rotation of 160 rpm according to a methodology adapted from Pereira et al. (2015).

After the end of the coagulation/flocculation process, the Jar Test was turned off and the samples were kept at rest for 45 min, so that the material could settle. Then, 50 ml of each sample was taken for the analysis of the parameters: pH in direct reading of the samples in digital pH meter; turbidity using a microprocessed digital turbidimeter model DLT-WV and apparent color in a color analyzer by the Platinum-Cobalt method, adapted from the Standard Methods for the Examination of Water and Wastewater (EATON et al., 2005).

The efficiency of the coagulant for each parameter was calculated according to the methodology adapted from Fayos et al. (2016), in which the initial and final values were replaced by the value of the respective parameter analyzed, Eq. 1.

\[
\text{Coagulant efficiency (\%)} = \left( \frac{\text{Initial parameter value} - \text{Final parameter value}}{\text{Initial parameter value}} \right) \times 100
\]

Table 1. Initial water characterization

| Parameters                  | Initial average value | MAV\(^1\) |
|-----------------------------|-----------------------|-----------|
| pH                          | 11.2                  | 9.5       |
| Turbidity (NTU)             | 289                   | 5         |
| Apparent color uH (mg Pt-Co/L) | 500             | 15        |

\(^1\)Maximum Allowable Value (BRASIL, 2017).

Statistical analysis

The experimental design was completely randomized, with four treatments and three replications. The data were subjected to analysis of variance and the averages were compared using the Tukey test at 5% probability, using the Assistat 7.7 program (SILVA; AZEVEDO, 2016).

RESULTS AND DISCUSSION

Figure 1 shows that, as the doses of the freeze-dried extract increased, the pH of the water significantly decreased, with the dose of 24 g/500 mL having the lowest value (8.8). From the application of 16 g/500 mL, the pH of the water is within the limits allowed by Brazilian legislation for drinking water, which establishes a pH value of 6 to 9.5 (BRASIL, 2017). Therefore, only 12 g/500 mL of the doses applied resulted in less efficiency, as it presented a result higher than that required.

Adeniran et al. (2017), when studying the pH of wastewater, initially obtained pH 9.6 and after treatment with control doses, 2, 4 and 6 g of moringa seeds the pH value decreased to 9.21, 7.8, 7.57 and 7.1, respectively. According to Santos et al. (2016), the range considered ideal for the pH is between 7.2 to 7.6 and a limit value of 8.0; the pH below 7.0 can cause irritation to the skin and eyes, as well as corrosion problems in equipment. Above its ideal, it reduces the effectiveness of chlorine and can cause problems such as incrustations in the tubes and other parts of the water circulation system resulting in water cloudy.

The results of applying different doses to remove turbidity from water are shown in Figure 2. The doses showed a significant difference. The 24 g/500 mL dose was the one that showed the
highest efficiency in removing turbidity (60.66%), followed by the dose of 16 g/500 mL (56.72%). The other doses showed an efficiency of 46.20 and 51.08%. Among the results obtained in research developed by Bongiovani et al. (2013), the statistical analysis also indicated that the different concentrations of the coagulant obtained from *M. oleifera* were statistically different with an optimal dosage of 50 mg/L. Values higher than those obtained in the present study were reported by Nkurunziza et al. (2009) that, when using saline extract for water treatment with 50 NTU turbidity, found 87.5% turbidity removal percentage.

Ordinance No. 5/2017 of the Ministry of Health establishes a maximum value of 5.0 NTU for turbidity in drinking water (BRASIL, 2017). Thus, the use of a coagulant based on the turbidity extract of *M. oleifera* seeds proved to be efficient in removing turbidity, but not effective.

Muniz et al. (2015), when evaluating the effect of moringa seeds used in shells and without shells in reducing water turbidity with different levels of turbidity, observed that the efficiency of moringa seeds in removing turbidity varied according to the initial turbidity levels of water samples. The samples with initial turbidity of 70, 250 and 400 NTU suffered reductions greater than or equal to 89.5, 95.5 and 97.5%, respectively, in relation to the initial turbidity, from the concentration of 200 mg/L of moringa seeds.

Mangale et al. (2012) obtained an optimum dose of 50 mg/L of seed powder of *M. oleifera* using low turbidity water (<50 NTU) obtaining turbidity below 5 NTU at the end. Franco et al. (2012), evaluating filtration with and without pretreatment, observed that filtration with pretreatment had greater removal efficiency (89% of turbidity and 86% of color) if compared to filtration without receiving it (62% of color and turbidity). It is worth mentioning that filtration is one of the important steps in water treatment, but it was not applied in this research; therefore, it is necessary to add a filtration stage to improve the quality of the treated water.

The results obtained for the apparent color of the water are shown in Figure 3. Unlike what happened with the pH and turbidity, the dose that showed greater efficiency in removing the apparent color was 16 g/500 mL. This showed a reduction of 61.33% when compared to the initial apparent color 500 (mg Pt Co/L).

The application of 12 g/500 mL showed a lower efficiency of 45.33%; the other dosages, 18 and 24 g/500 mL, showed efficiency of 52.66 and 55.33%, respectively. A similar result was verified by Valverde et al. (2018), when using higher proportions of moringa extract as a coagulant, which resulted in lower efficiencies of removing turbidity.

![Figure 1](image-url)
apparent color and turbidity, 70.1 and 56.7%, respectively.

According to Renault et al. (2009), natural coagulants are more advantageous than inorganic compounds because they produce denser and more compact flakes, reduce the required inorganic coagulant dose and produce smaller amounts of sludge. This statement corroborates Mohamed et al. (2015) when observing that freeze drying and extraction of moringa by using potassium chloride and nitrate decrease the optimal coagulant doses by 28 and 18%, respectively.

**CONCLUSIONS**

- The application of different doses of freeze-dried extract of moringa seeds in water treatment resulted in pH according to the limits required by Brazilian legislation for drinking water.
- The dose of 24 g/500 mL is recommended to be applied in water treatment because it provides a pH closer to the ideal for drinking water.
The parameters of turbidity and apparent color showed higher values at the end of the research than the ones required by Brazilian legislation for drinking water, with doses of 24 and 16 g/500 mL, respectively, being the most suitable for reducing the parameters during water treatment.

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