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Melamine-formaldehyde-silica and melamine-silica-cellulose composites in removing Iron and N-ammonia from landfill leachate

Compósitos de melamina-formaldeído-sílica e melamina-sílica-celulose na remoção de ferro e N-amônia do lixiviado de aterro sanitário

Compuestos de melamina-formaldehído-sílice y melamina-sílice-celulosa para eliminar el hierro y el N-amoníaco del lixiviado de vertederos

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Abstract

Melamine-formaldehyde based composites are versatile and can be applied in the treatment of contaminated effluents such as landfill leachate that have a high pollutant load, as they are rich in nitrogen atoms, the sites allow interaction with molecules, atoms or ions of interest. Aiming compares the efficiency of two materials based on the same precursors, evaluating the efficiency of two composites, melamine-silica (PMF-Si) and melamine-silica-cellulose (Cel-M-Si) in removing iron and ammonia nitrogen in landfill leachate. Adsorption kinetics showed that PMF-Si and Cel-M-Si composites adsorb iron from 30 min, with an average removal of ~ 93.4%. Application of Cel-M-Si to removed leachate ca. 75.7% iron and 76.6% ammonia nitrogen. In contrast, it was observed that PMF-Si had a removal efficiency of 70.9% for iron and 55.0% for ammonia nitrogen. The comparative tests allowed to conclude that the composites PMF-Si and Cel-M-Si have potential for the treatment of landfill leachate, being low cost materials and easy synthesis.

Keywords: Cellulose; Landfill leachate; Melamine-silica.

Resumo

Os compósitos à base de melamina-formaldeído são versáteis e podem ser aplicados no tratamento de efluentes contaminados como lixiviado de aterro que possuem alta carga poluente, por serem ricos em átomos de nitrogênio os sítios permitem a interação com moléculas, átomos ou íons de interesse. Visando compara a eficiência de dois materiais a base dos mesmos precursores, avaliou-se a eficiência de dois compósitos, melamina-sílica (PMF-Si) e melamina-sílica-celulose (Cel-M-Si) na remoção de ferro e nitrogênio amoniacal em lixiviado de aterro. A cinética de adsorção mostrou que os compósitos PMF-Si e Cel-M-Si adsorvem ferro a partir de 30 min, com uma remoção média de ~ 93,4%. Aplicação de Cel-M-Si ao lixiviado removido ca. 75,7% de ferro e 76,6% de nitrogênio amoniacal. Em contraste, observou-se que o PMF-Si apresentou uma eficiência de remoção de 70,9% para o ferro e 55,0% para o nitrogênio amoniacal. Os testes comparativos permitiram concluir que os compósitos PMF-Si e Cel-M-Si apresentam potencialidades para o tratamento de lixiviado de aterro, sendo materiais de baixo custo e de fácil síntese.

Palavras-chave: Celulose; Lixiviado de aterro; Melamina-sílica.

Resumen
Los compuestos a base de melamina-formaldehído son versátiles y se pueden aplicar en el tratamiento de efluentes contaminados como lixiviados de vertedero que tienen una alta carga contaminante, ya que son ricos en átomos de nitrógeno, los sitios permiten la interacción con moléculas, átomos o iones de interés. Visanto compara la eficiencia de dos materiales basados en los mismos precursores, evaluando la eficiencia de dos composites, melamina-sílice (PMF-Si) y melamina-sílice-celulosa (Cel-M-Si) en la remoción de hierro y nitrógeno amoniacal en lixiviados de vertedero. . La cinética de adsorción mostró que los compuestos PMF-Si y Cel-M-Si adsorben hierro a partir de 30 minutos, con una eliminación promedio de ~ 93,4%. Aplicación de Cel-M-Si para eliminar el lixiviado aprox. 75,7% de hierro y 76,6% de nitrógeno amoniacal. Por el contrario, se observó que PMF-Si tenía una eficiencia de eliminación del 70,9% para el hierro y del 55,0% para el nitrógeno amoniacal. Las pruebas comparativas permitieron concluir que los composites PMF-Si y Cel-M-Si tienen potencial para el tratamiento de lixiviados de vertedero, siendo materiales de bajo costo y fácil síntesis.
Palabras clave: Celulosa; Lixiviados de vertedero; Melamina-sílice.

1. Introduction

Nitrogen-rich organic compounds are of great importance as they have applications in various sectors of technology, since nitrogen atoms have sites that allow interaction with molecules, atoms or ions of interest (Bretterbauer et al., 2012; Shin et al., 2011; Vareda et al., 2020). One of the organic compounds that fits perfectly with the characteristics that favor the adsorption process is the melamine-formaldehyde polymer (PMF), where melamine works as a source of nitrogen (Liu et al., 2019).

PMF is a material that is easy to synthesize, has a high surface area, low cost, and is chemically stable (Bretterbauer et al., 2012; Nakanishi et al., 2021; Tan et al., 2013). In addition, PFM is a versatile polymer, as it has the ability to efficiently remove both anionic and cationic dyes, and can also be regenerated via an advanced oxidation process (Wang et al., 2016). On the other hand, the inclusion of other materials, such as silica nanoparticles, in the PMF may favor greater adsorption efficiency for species of interest, as is the case verified in the adsorption of Cu(II) and $Cr_2O_7^{2-}$ reported by Avan et al., 2021. Thus, it has been common the emergence of new investigations on the use of PMF to remove dyes and metals from wastewater (Avan et al., 2021; J. Li et al., 2017; Nakanishi et al., 2021; Tan et al., 2013).

Recently, Santos et al., 2021 synthesized PMF decorated with silica nanoparticles (PMF-Si), which showed good adsorption capacity for the methylene blue dye. Mesquita Junior et al., 2021 applied the Cel-M-Si composite containing cellulose originating from *Mangifera indica* for adsorption of methylene blue and found that there is a removal of *ca.* 89% of the dye with a contact time of 30 min. The use of cellulose together with melamine and silica aims to improve the ability of the PMF composite in the adsorption process for metals and other compounds of interest, since cellulose is a widely explored material for application as an adsorbent (Mohammed et al., 2021).

In addition to the overwhelming need to remove dyes from industrial wastewater, there is also a strong call for processes to remove ammonia and iron from solid waste leachate. In this sense, the use of cheap materials such as the PMF-Si composite and the Cel-M-Si containing cellulose can be useful, if they have the ability to remove contaminating species. Iron, for example, is an important metal in the human body, but its excess causes problems such as deposition, encrustation, appearance of rusty bacteria that are harmful to the environment, in addition to being toxic to living beings, if inferred in high amounts (Hijazi et al., 2020; Rehman et al., 2018).

The recommended limit of iron concentration in domestic water is 0.3 mg/L (Standard Test Methods for Iron in Water). samples with concentrations ranging from 0.1 mg/L to 1000 µg/L (Standard Test Methods for Iron in Water) On the other hand, ammonia is a highly soluble substance and difficult to remove, causing toxicity to living organisms and harming the environment (Yan et al., 2020). As a result, efforts are made to advance the development of methods for the detection and quantification of ammonia dissolved in water (Standard Test Methods for Ammonia Nitrogen In Water), but also ways to mitigate environmental pollution (Hasanoĝlu et al., 2010; Zhang et al., 2020).

In Brazil, the National Environmental Council provides for the conditions and standards for the release of effluents, with the maximum concentration of total ammonia nitrogen being 20 mg/L, while the maximum concentration of dissolved iron allowed is 15 mg/L (Oppong-Anane et al., 2018). Thus, it is evident that the treatment of leachate from environments such as, for example, landfills and stabilization ponds need attention with adequate treatment to remove not only ammonia and iron, but also many other species of contaminating materials.

In this work, we will emphasize the study of iron and ammonia removal from solid waste leachate. In view of the potential applications of PMF composites for removing dyes and metals from wastewater, we sought to investigate the efficiency of PMF-Si and Cel-M-Si composites containing cellulose for removing iron and ammoniacal nitrogen from contaminated water. Therefore, in this work we present the adsorption tests for removal of iron and ammoniacal nitrogen, in addition to the microscopic characterizations and thermogravimetric analysis of the composites PMF-Si and Cel-M-Si.

2. Materials and Methods

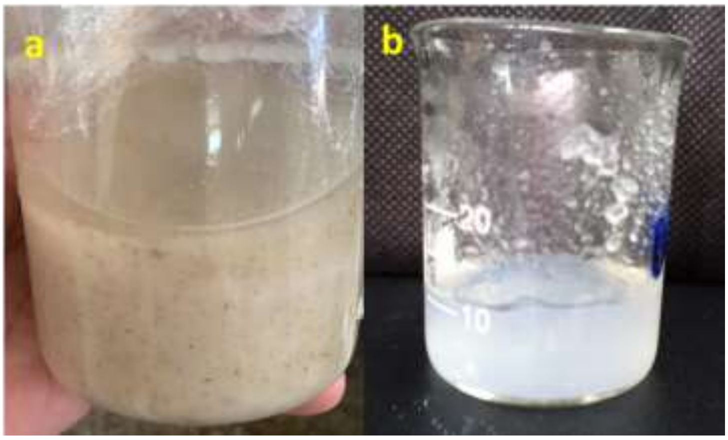
2.1 Methodology

This work presents a case study based on the comparison of two composites based on melamine and silica in the treatment of effluents (Köche, 2011; Pereira et al., 2018; Yin, 2001). Composites were approached by Santos et al., 2021 and Mesquita Junior et al., 2021.

2.2 Synthesis of PMF-Si and Cel-M-Si composites

The synthesis procedure for the melamine-formaldehyde-silica composite (PMF-Si) is in accordance with Santos et al., 2021 and the melamine-formaldehyde-silica-cellulose (Cel-M-Si) compound is in agreement with Mesquita Junior et al., 2021. Figure 1 presents digital photos of the synthesized materials. In Fig. 1a is the composite Cel-M-Si and in Fig. 1b it is possible to see the image of the hydrogel PMF-Si.

Figure 1 - Images of composites (a) Cel-M-Si and (b) PMF-Si.



Source: Authors (2021).

2.3 Landfill leachate sample

The collection of the landfill leachate was carried out in Teresina-PI (5°09’46.09’S, 42°45’02.12’W). The leachate was stored in 2 L polypropylene vials, protected from light and at room temperature. The sample did not undergo any previous treatment, such as decantation, filtration or chemical treatment, before mixing with the composites.

2.4 Characterization and methods

The formation of PMF-Si and Cel-M-Si composites was confirmed and discussed in previous publications (Mesquita Junior et al., 2021; Santos et al., 2021). The morphology of the composites was elucidated by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Both composites were also subjected to thermogravimetric analysis (TG-DTG). The characterization procedures can be found in more detail in Mesquita Junior et al., 2021 for the Cel-M-Si composite, and in Santos et al., 2021 for the PMF-Si hydrogel.

2.5 Iron and ammonia nitrogen

The high content of iron present in the leachate causes the contamination of groundwater, therefore, the removal of the mineral is of fundamental importance. When the slurry is in the maturation phase, there is a reduction in the biodegradable fraction and a considerable increase in ammonia nitrogen. Thus, the proliferation of microorganisms is unfeasible, which makes conventional biological treatment difficult. Firstly, the efficiency of the composite in the adsorption of iron was studied in a $FeCl_3$ solution (7 ppm) at the contact times of 10, 30, 60 and 90 min.

The evaluation of iron removal in landfill leachate was performed based on the method developed by Oppong-Anane et al., 2018, with some modifications. Ammonia removal was performed in line with the methodology adapted from González-Cortés et al., 2021. Thus, 1 g of Cel-M-Si was added to 100 mL of landfill leachate solution, and 10 g hydrated (1g dry) of PMF-Si in 100 mL of landfill leachate solution, both in separate weaklings. The mixtures of the two composites and the landfill leachate were stirred at 180 rpm for 30 min using the Solab equipment, model SL – 180/D. Subsequently, the landfill leachate samples with the Cel-M-Si compound were centrifuged for 4 min at 4000 rpm and the landfill leachate samples with the PMF-Si hydrogel were centrifuged for 10 min at 4000 rpm, both using the DAIKI centrifuge, Spinplus Titan model. The supernatants of the two samples were collected and the measurements of iron and ammonia nitrogen were carried out in a spectrophotometer (HACH – DR3900). Ammonia nitrogen was determined by the Nessler method according to the reagents developed by Hach. The adsorption capacity of the composites was calculated with Equation 1:

$$\textit{adsorption capacity}\ (\%) = \left(\frac{C_0 - C}{C_0}\right) \times 100 \qquad \text{Equation 1}$$

Where C_0 is the initial concentration of the analyte and C is the concentration recorded at the end of the adsorption process.

2.6 Statistical analysis

The statistical treatment of the experiments and data interpretation were performed using Excel 2016 and Origin 8.5 software. All analyzes were performed in triplicate and results expressed as mean plus standard deviation. All comparisons were performed with a confidence level of 95% and the significant difference between the mean values was assessed using the Tukey test.

3. Results and Discussions

3.1 TG and DTG of PMF-Si and Cel-M-Si composites

The TG and DTG of the PMF-Si and Cel-M-Si materials are shown in Figure 2. The PMF-Si showed a significant loss of surface water in the temperature range of 45-100 ºC. On the other hand, the elimination of water retained in the composite structure and bound to the silanol groups occurred between temperatures 150-270 °C. In the Cel-M-Si compound the removal

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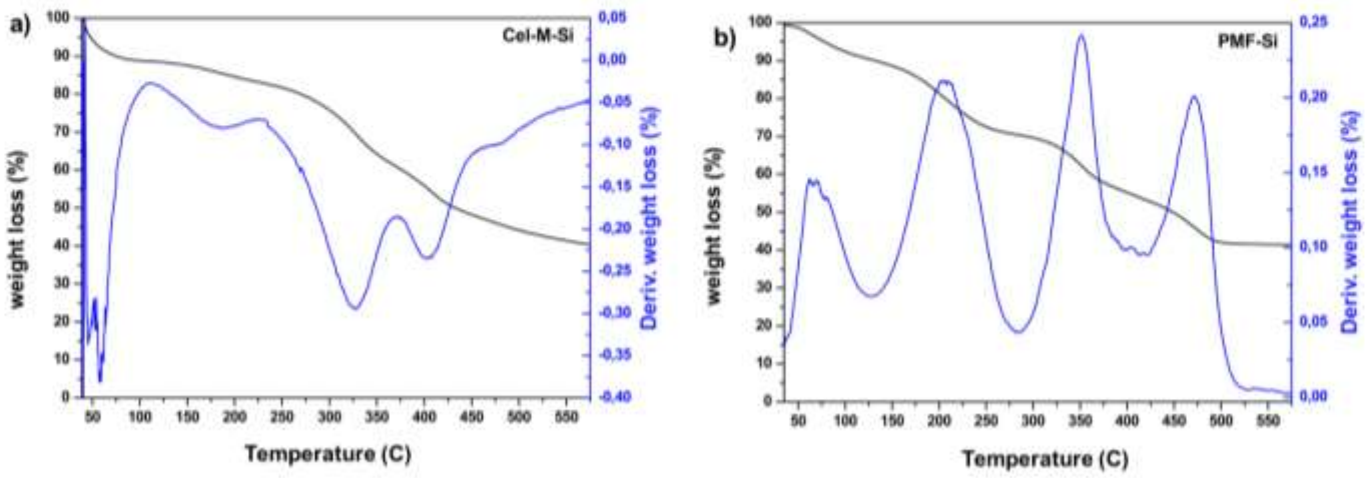
of water only occurs in the temperature range of 45-100 °C. As PMF-Si is a hydrogel, it has a greater amount of water retained on its surface as well as between its interstices. The PMF-Si composite presents two more events, one between the temperatures of 282-418 ºC which is attributed to the degradation of the melamine-formaldehyde polymer and the loss of mass of glycerin, and the last event between the temperatures 418-525 ºC, attributed to the thermal decomposition of PMF fibers. The resulting mass of the melamine-silica composite is ~42%. In the Cel-M-Si composite, the event between temperatures 265-450 ºC is attributed to the loss of mass caused by the degradation of PMF and cellulose. Between temperatures 375-510 ºC there was mass loss related to the degradation of organic compounds of Cel-M-Si. The final mass percentage of the Cel-M-Si composite is 65%, which is the highest percentage of waste compared to PMF-Si. This is due to the presence of cellulose ash in addition to silica.

Figure 2 - TG and DTG of composites (a) Cel-M-Si and (b) PMF-Si.

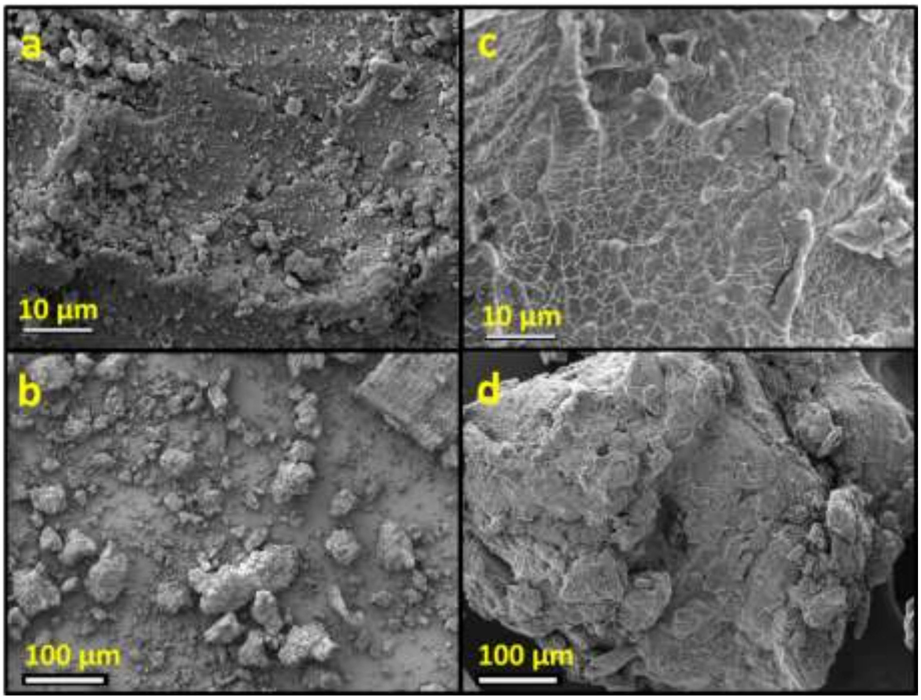


Source: Authors (2021).

3.2 Scanning Electron Microscopy of Composites

Figure 3 shows SEM images of the two materials, Cel-M-Si and PMF-Si. Figure 3a shows the Cel-M-Si material that has a heterogeneous distribution of spherical agglomerates containing silica and melamine on the surface of the fibers. Figure 3b, with greater distance from the material (100 µm), shows in the upper right corner, the presence of a cellulose particle. Figure 3c shows the morphology of the PMF-Si with a rough structure with the appearance of dry human skin, a fact justified by the large loss of water in the preparation of the PMF-Si sample for microscopic analysis.

Figure 3 - Scanning electron microscopy of composites (a and b) Cel-M-Si (c and d) PMF-Si.



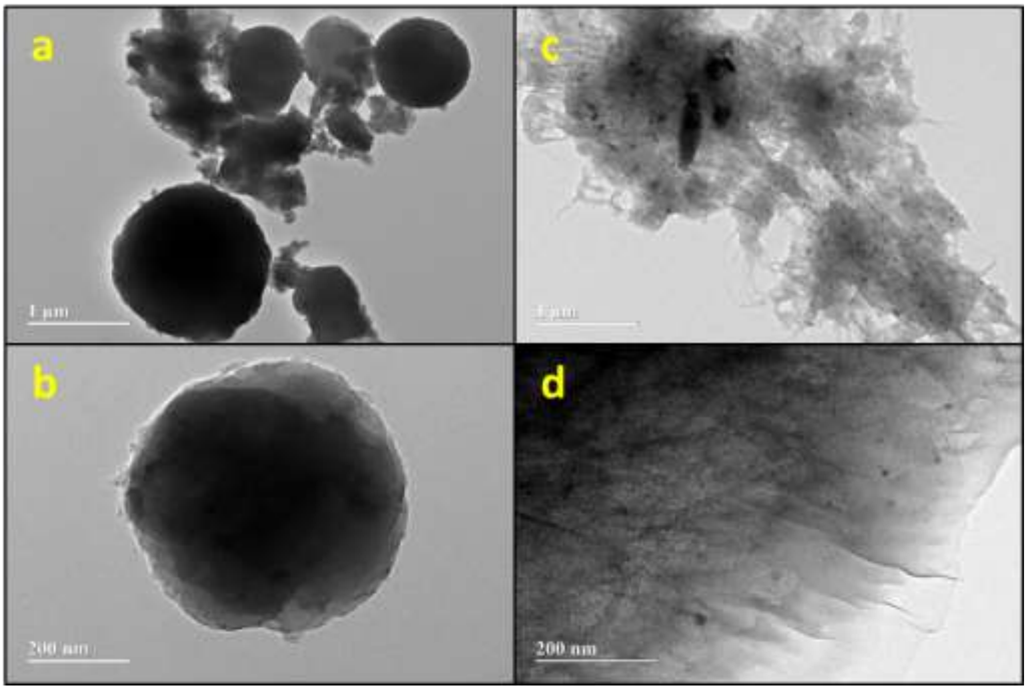
Source: Authors (2021).

Figure 3d, on the 100 µm scale, shows hydrogel particles with a spongy appearance. This morphology occurs because PMF-Si has in its structure the melamine-formaldehyde polymer, a 3D material capable of crosslinking and forming fibers that encompass silica nanoparticles with a large amount of water, as confirmed in thermogravimetric analysis (Merline et al., 2013). This disposition is also confirmed by the PMF images obtained by Schwarz and Weber, 2017. When comparing the micrographs of Cel-M-Si and PMF-Si materials, it can be concluded that the hydrogel has a morphology that allows greater water retention, but has a smaller amount of dispersed particles on its surface.

3.3 Transmission electron microscopy of composites

Figure 4 shows the transmission electron microscopy (TEM) of the Cel-M-Si (Figure 4a and 4b) and PMF-Si (Figure 4c and 4d) materials. The micrometric scale Cel-M-Si composite presents approximate particles forming spherical agglomerates. The PMF-Si composite shows the hydrogel containing silica nanoparticles. Silica nanoparticles are homogeneously distributed within the PMF matrix. The presence of silica nanoparticles and not agglomerates, in addition to the uniform distribution, allows the PMF-Si composite to have better stability and better water absorption capacity by the hydrogel (Schwarz & Weber, 2015). Figure 4d at 200 nm scale shows in the upper left corner the network structure of the PMF present in the hydrogel. This 3D structure is responsible for supporting the mass of water present in the hydrogel (Baniasadi et al., 2021). Furthermore, it is possible to verify the presence of some Si nanoparticles that are adsorbed to the PMF fibers.

Figure 4 - Transmission electron microscopy of (a and b) Cel-M-Si and (c and d) PMF-Si.



Source: Authors (2021).

3.4 Removal of iron and ammoniacal

Removal of iron and ammonia-N by PMF-Si and Cel-M-Si composites was performed by direct adsorption. The composites have in common the reagents melamine, formaldehyde and silica. However, the synthesis route and the products formed are different. The evaluation of iron removal by composites was carried out firstly from the study of the contact time necessary for effecting adsorption. The analysis was performed in a univariate manner in a $FeCl_3$ solution at times of 10, 30, 60 and 90 min. Table 1 shows that the composites had a removal percentage greater than 90% for all contact times evaluated, which maximum was reached in 90 min with respective removal of 97.4% for Cel-M-Si and 95.2% for the PMF-Si.

Table 1 - Iron removal from $FeCl_3$ solution.

Time (min)	PMF-Si-Cel Removal (%)	PMF-Si Removal (%)
10	92.5 ± 0.1	92.0 ± 0.1
30	92.5 ± 0.1	93.4 ± 0.1
60	95.5 ± 0.2	94.7 ± 0.7
90	97.4 ± 0.1	95.2 ± 0.1

Source: Authors (2021).

In the analysis of the removal of iron and nitrogen-ammonia, a time of 30 min was used for both composites, as longer times did not show a significant increase in Fe removal. After verifying the effectiveness of the composites in removing iron from the $FeCl_3$ solution, tests were carried out on the raw landfill leachate. The landfill leachate presented an initial concentration of 14.5 ppm of iron and, after treatment, about 75.7% of the total iron content that was removed by the Cel-M-Si. The PMF-Si composite removed about 70.1% of the total iron content. These values demonstrate that composite materials have a high adsorptive capacity for dispersed iron in landfill leachate. When compared to the $FeCl_3$ solution, the removal was lower, due to the landfill leachate being a complex matrix and not having undergone pre-treatment. Furthermore, iron adsorption can be inhibited by competition with other elements present in the leachate. This effect of decreasing adsorption by the presence of other species has an antagonistic effect and occurs when there is the presence of two or more solutes in the mixture, exerting negative interference in the adsorption of the species of interest, resulting in a decrease in the adsorption capacity (Melo et al., 2019).

(M. Li et al., 2020) studied the adsorption of iron on cellulose compounds, and reported that several groups containing oxygen, such as C-O, C=O, C-O-C, are related to the adsorption process. In FTIR analysis it was observed by Sarkar & Sarkar, 2017 bands at 500-588 cm-1 corresponding to the vibrations of Fe-O bonds in cellulose nanocomposites. Iron removal is also related to electrostatic interactions between amine groups present in the melamine of PMF (Kabir et al., 2018). Compounds rich in hydroxyl groups (-OH) or oxygen with negative charge such as the silanolate group (Si-O-) present in silica nanoparticles originated from the neutralization of silicate in the formation of the composite PMF-Si and the composite Cel-M-Si, are promising for iron removal.

In addition to heavy metals, the toxicity of landfill leachate can be attributed to a wide variety of compounds present and high concentrations of ammoniacal nitrogen. This is an important pollutant, mainly due to the different oxidation states in which nitrogen is found and the favoring of the eutrophication process, which results in a decrease in dissolved oxygen in water (González-Cortés et al., 2021; Miranda et al., 2021). The high content of ammonia nitrogen makes water treatment more difficult. Therefore, the removal of this contaminant was also studied.

The initial samples of the landfill leachate, before treatment with the composites, presented ammonia concentration around 100.0 ± 2.0 ppm. This value is about 5 times the maximum value allowed by CONAMA, which is 20 ppm (CONAMA, 2011). After treatment with the composite Cel-M-Si, a decrease of around 76.6% of the initial concentration of ammonia nitrogen was observed, and a reduction of 55.0% after treatment with the composite PMF-Si. With this result, it is possible to infer that the composites presented in this work can be used in the treatment of water with a high content of ammoniacal nitrogen.

In the literature, several methods are presented for the removal of ammoniacal nitrogen in leachate samples, for example, biological treatments with activated aerobic sludge, anaerobic digestion, air purging, ion exchange, coagulation-flocculation, precipitation and adsorption techniques (González-Cortés et al., 2021; Miranda et al., 2021). However, there are no reports of the use of melamine-formaldehyde-silica-based composites, nor the use of cellulose from *Mangifera indica* pruning.

4. Conclusions

From the application of PMF-Si and Cel-M-Si composites, we observe that iron and ammonia nitrogen are removed from landfill leachate by adsorption. Both composites were efficient in removing iron and ammonia nitrogen with a contact time of 30 min. Cel-M-Si promoted a reduction of 75.7% of total iron and 76.6% of ammonia nitrogen, with final concentration values of 3.6 and 23.5 ppm, respectively. The PMF-Si composite showed 70.9% efficiency for removing iron and 55.0% for removing ammonia nitrogen with final concentration values of 4.67 and 45.0 ppm, respectively. The performance of comparative tests allowed us to conclude that melamine-formaldehyde-silica-based composites have potential for the treatment of landfill leachate. As future perspectives, aimed at the application of composites in the removal of other contaminating metals and in the treatment of wastewater from the textile industry. In view of the potential of melamine-silica-based composites in the treatment of effluents, this applicability should be thoroughly investigated, increasing the spectrum of harmful compounds present in effluents, in addition to studies on the optimization of variables, such as composite mass , temperature, and the union with other materials to increase the efficiency of effluent treatment.

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