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Research paper

Evaluation of methylene blue-removal by adsorbents recovered from industrial activity

Avaliação da remoção de azul de metileno por adsorventes recuperados de atividades industriais

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ABSTRACT: The study evaluated the application capacity of materials recovered from mining activities (nano hematite recovered from acid mine drainage and basaltic remineralizer (BR) (rock powder) discarded from quarries) as adsorbents in removing methylene blue dye. The adsorbent materials were characterized and had their zero-charge point determined, with pH_{PCZ} 6.5 for rock powder and 5.5 for hematite. The results showed that the basaltic remineralizer contains 51.1% silicon dioxide, 15.2% hematite, and 13.2% alumina. The analysis also indicated that the levels of heavy metals in the rock powder were within the limits established by CONAMA resolution 420/2008, suggesting safety in its application in the environment. The adsorption tests were performed in batches, using 0.5 g of adsorbent in 100 mL of 10 mg. L^{-1} methylene blue solution. The experiments were performed in triplicate in the pH range of 2 to 6. The dye removal capacity was satisfactory, with hematite removing an average of 1.73 mg. g^{-1} and basaltic rock powder 2.08 mg. g^{-1}. The greater efficiency of basaltic rock powder, even with a smaller amount of hematite (approximately 0.08 g), can be attributed to the concomitant presence of alumina, offering more active sites for dye adsorption. This study suggests new possibilities for removing textile dyes from effluents and adds commercial value to basaltic rock powder, a byproduct of the mining industry, and nanoparticles recovered from industrial activity.

Keywords: Mining waste, nano iron oxide, basalt, adsorption, textile dyes.

RESUMO: *O estudo avaliou a capacidade de aplicação de materiais recuperados de atividade de mineração (nano hematita recuperada de drenagem ácida de minas e remineralizador basáltico (pó de rocha) descartado de pedreiras), como adsorventes na remoção do corante azul de metileno. Os materiais adsorventes foram caracterizados e tiveram ponto de carga zero determinado, sendo* pH_{PCZ} *6,5 para o pó de rocha e 5,5 para hematita. Os resultados mostraram que o remineralizador basáltico contém 51,1% de dióxido de silício, 15,2% de hematita e 13,2% de alumina. A análise também indicou que os níveis de metais pesados do pó de rocha estavam dentro dos limites estabelecidos pela resolução 420/2008 do CONAMA, sugerindo segurança em sua aplicação no ambiente. Os ensaios de adsorção ocorreram em batelada, utilizando 0,5 g de adsorvente em 100 mL de solução de azul de metileno 10* $mg.L^{-1}$*. Os experimentos foram feitos em triplicada na faixa de pH de 2 a 6. A capacidade de remoção do corante foi satisfatória, com a hematita removendo em média 1,73* $mg.g^{-1}$ *e o pó de rocha basáltica 2,08* $mg.g^{-1}$*. A maior eficiência do pó de rocha basáltica, mesmo com menor quantidade de hematita (0,08g aproximadamente) pode ser atribuída à presença concomitante de alumina, oferecendo mais sítios ativos para a adsorção do corante. Este estudo sugere novas possibilidades para a remoção de corantes têxteis de efluentes e agrega valor comercial ao pó de rocha basáltica, um subproduto da indústria de mineração e às nanopartículas recuperadas de atividade industrial.*

Palavras-chave*: Resíduos de mineração, nano óxido de ferro, basalto, adsorção, corantes têxteis.*

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INTRODUCTION

The search for environmental solutions in contemporary society causes the emergence of technologies that impact significant changes in the population from a social, economic, and cultural point of view (Tomkelski; Scremin; Fagan, 2019). Industrial waste, which is normally discarded, represents a potential raw material for producing low-cost high value-added materials, such as nanoparticles (Flores *et al*., 2012; Rauen *et al.*, 2021).

Nanoscience is the area that studies physical-chemical phenomena on the nanometric scale. In terms of dimension, the nanometer (nm) is equivalent to a billionth part (10^{-9}) of a meter (Rai, 2013). Nanoparticles are nanoscale particles with great importance in the study of nanoscience. Nanoparticle research includes the study of iron nanoparticles as adsorbents or nanocatalysts used in healthcare, effluent treatment, and adsorption/photocatalysis (Vinícius-Araújo *et al*., 2024; Kołodyńska *et al.,* 2016; Ramírez-Sánchez; Bandala, 2016).

Adsorption studies the ability of a solid (adsorbent) to adhere to its surface other substances (adsorbates) present in fluids. This adhesion allows the separation of compounds from this mixture (Nascimento *et al*., 2020) and is therefore well studied for removing pollutants (Martins *et al*., 2023).

Substances with adsorbent properties can have organic (and co-products), synthetic, and mineral origins.

According to Staub (2019), the hematite nanoparticulate used in this study, recovered from acid drainage of mines (Carbonífera Criciúma) has a considerable adsorption potential, even with its reduced surface area compared to other commercial adsorbents. Besides the high adsorption capacity, iron oxides are physicochemically stable, are not toxic to the environment, and have a low production cost (Silva; Pineda; Bergamasco, 2014; Rauen *et al*., 2021). Basaltic remineralizer (BR), the other material used as adsorbent in this study, has a considerable hematite content in its composition. This remineralizer also comes from mining activity, a quarry in southwestern Paraná, and is usually discarded or used only in agriculture.

Methylene blue is a dye widely studied in adsorption processes due to its high adhesion to solids. It is used as a model compound to investigate the adsorption of organic contaminants in aqueous solutions (Di Bernardo; Dantas, 2005).

In this context, we aimed to evaluate the capacity of materials originating from mining residues (basaltic remineralizer and nano hematite) as adsorbents for removing methylene blue in an aqueous solution.

MATERIAL AND METHODS

OBTAINING AND CHARACTERIZATION OF ADSORBENTS

The basaltic rock samples (**Figure 1**) were collected in a quarry in Francisco Beltrão, Paraná, Brazil, where distropheric red latosol soil (LVd) from basaltic spill predominates. This soil has considerable depth, mainly in the flat portions of the relief, where its composition is clayey, homogeneous, porous, and structured around slopes (Brito *et al.*, 2006).

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Figure 1. Sample of Basaltic Remineralizer

The samples were homogenized in sieves with a mesh size of 0.30 mm (ABNT, 2024). X-ray fluorescence analysis was used to quantify the main oxides present in basaltic rock-hematite, alumina, calcium oxide, magnesium, potassium, phosphorus, and manganese-, which was performed at the laboratory SGS Geosol, located in Belo Horizonte, Minas Gerais, Brazil.

The iron oxide nanoparticulate (**Figure 2**) was obtained by acid drainage recovery of mines in a coal mine of Criciúma, Santa Catarina, Brazil, and provided by the Laboratory for Environmental Studies - LEMA/UFSC. No transformation process was performed beyond calcination of the hematite sample at 450 °C for 4 hours to convert goethite into hematite in a furnace Fornitec F1, located at the UTFPR campus in Toledo, Paraná. The characterization parameters are presented in **Table 1**.

Figure 2. Sample of hematite nanoparticulate

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Table 1. Physicochemical and textural parameters of nano-hematite

Parameters	Nano hematite
BET surface area, $m^2.g^{-1}$	66.8
Pore volume, $cm^3.g^{-1}$	0.43
Mean pore diameter, nm	25.5
Dimensions, nm	520x363
Morphology	Acicular
Crystalline phase	Hematite
Size of crystallite, nm	13.1
Zero-point of charge, pH_{ZPC}	5.5±0.5

Source: Adapted from Rauen *et al.*, 2021.

Before the adsorption tests, we analyzed basaltic remineralizer and hematite samples using infrared spectroscopy from tablets produced using previously desiccated potassium bromide (KBr). The infrared region spectrum was recorded by the Perkin Elmer Spectrum 65 spectrophotometer, located in the Analysis Center of UTFPR Campus Toledo.

To define each adsorbent's zero-point of charge (pH_{ZPC}), we used the 11-point methodology, adapted from Regalbuto and Robles (2004 cited in Silva *et al.,* 2015). Initially, sodium chloride (NaCl) solutions were prepared at 0.1 mol.L^{-1} and different pHs (1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, and 12). For each adsorbent, 500 mg were accurately measured and transferred, along with 100 mL of each NaCl solution (0.1 mol.L^{-1}) to Erlenmeyer flasks that were kept at constant stirring on a Solar shaker table SL-180/DT for 24 hours at 100 rpm. Afterward, the samples were filtered and pH values were measured.

ADSORPTION ASSAYS

The adsorption tests were conducted in batches with constant temperature (100 rpm, 25 °C) on a Logen LS 4500 shaker. The same conditions were applied to both adsorbents. All the tests were done in triplicate.

Each Erlenmeyer flask received 100 mL of 10 mg.L^{-1} methylene blue solution and 0.5 g of adsorbent.

To verify the effect of pH on the removal capacity of methylene blue, the tests were performed in triplicate at each pH value (2, 3, 4, 5, and 6).

After 360 min of stirring, the samples were filtered through a 0.45-μm hydrophilic PVDF (Polyvinylidene Fluoride) membrane. The concentrations of methylene blue were then determined with a Kazuaki model UV-vis spectrophotometer (IL-0082) at a wavelength of 660 nm (calibration curve made previously).

RESULTS AND DISCUSSIONS

CHARACTERIZATION OF ADSORBENTS

Figure 3 shows the reading of the infrared spectrophotometer for the basaltic remineralizer. The spectrum presents a peak in the region of 1000 cm^{-1}, indicating the

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stretch attributed to the Silicon-Oxygen (Si-O) bending. The peaks in 590 and 740 cm^{-1} indicate the presence of Fe-O broadband connection (Fan *et al.,* 2020*).*

Infrared spectroscopy for the hematite nanoparticulate (**Figure 4**) showed peaks indicating the presence of water and iron clusters at 538-467cm^{-1}, corresponding to the vibration of the Fe-O bond, as can be observed in **Figures 3** and **4**.

Figure 3. Infrared spectroscopy with Fourier transform (FTIR) for a sample of basaltic remineralizer

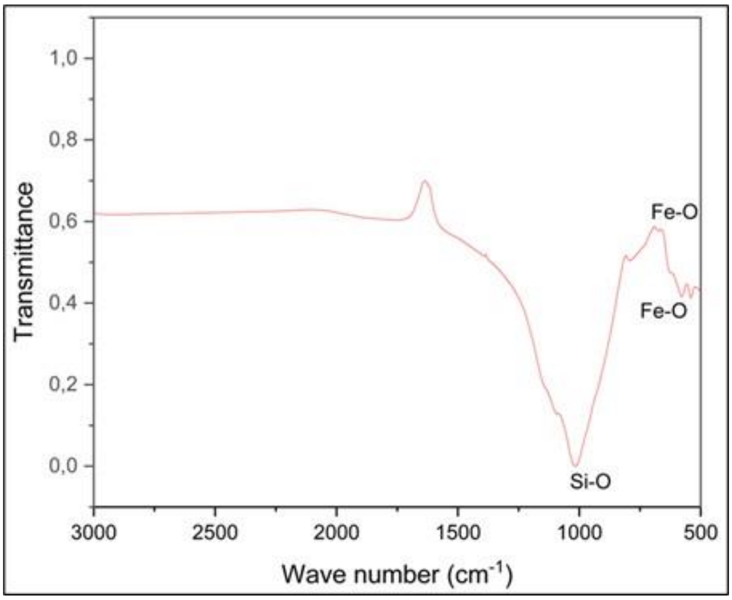
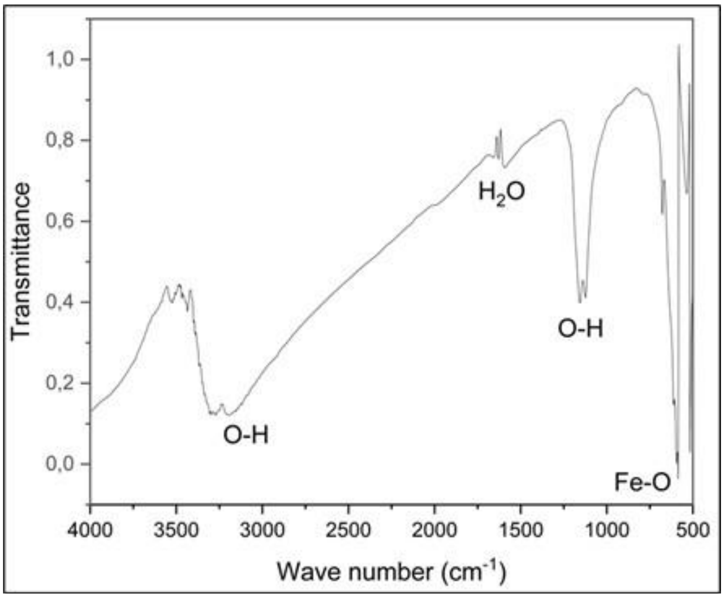


Figure 4. Infrared spectroscopy with Fourier transform (FTIR) for samples of hematite nanoparticles



The obtained bands are very similar to those obtained by Yousefi, Golikand and Mashhadizadeh (2013), who synthesized hematite in the laboratory, and Nogueira *et al.,*

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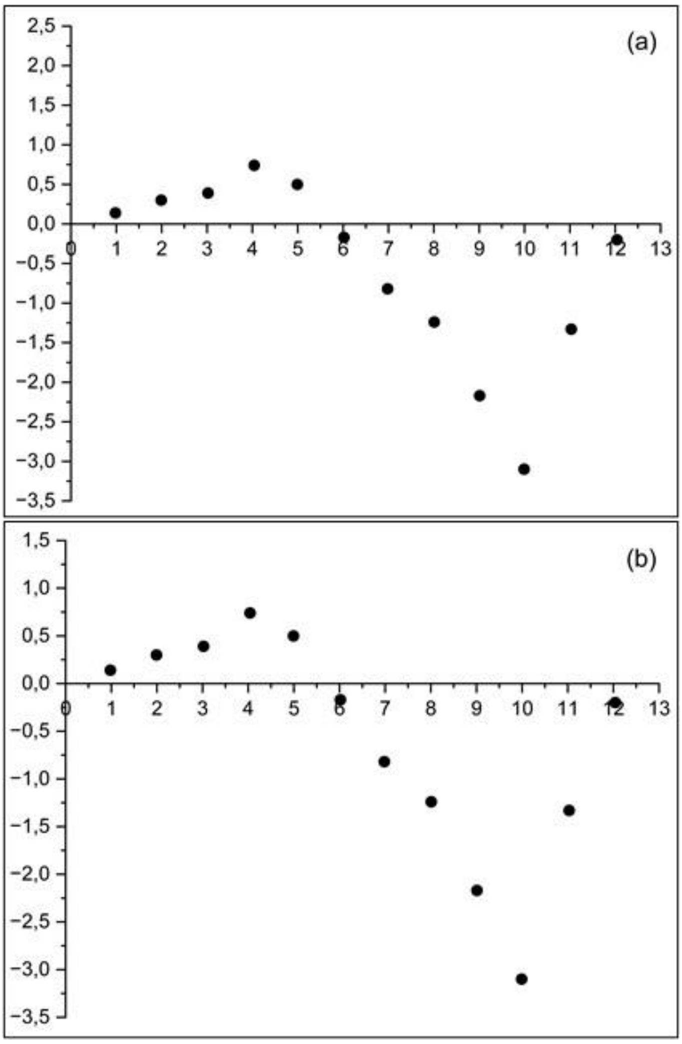
(2011), who determined the presence of hematite in raw clay material collected in San Juan, Argentina.

In the adsorption process, several materials are used as adsorbents, and it is essential to characterize them for a correct application. One of the most important aspects of this characterization is the zero-point of charge (ZPC), which represents the pH at which the material's surface has a neutral charge, i.e., remains constant. Cation adsorption is favored when the pH of the solution is above the ZPC, whereas anion adsorption occurs more efficiently when the pH is below the ZPC (Perez; Fields; Teixeira, 2017).

Figure 5. Zero-point of charge (pH_{ZPC}) for (a) basaltic remineralizer and (b) nano hematite



For the pH range considered, the values of the adsorbents' pH_{ZPC} were determined by the intersection points of the respective curves with the x-axis. Thus, the values are 6.5 and 5.5 for basaltic remineralizer and nano-hematite, respectively.

Other studies have determined, for the synthesized hematite nanoparticulate (Shrimali *et al.,* 2016), the approximate value of 6.2, whereas for the remineralizer from the mining industry, Hawerroth (2020) found a zero-point of charge of 6.8 for remineralizing intrusive igneous granite (pink granite).

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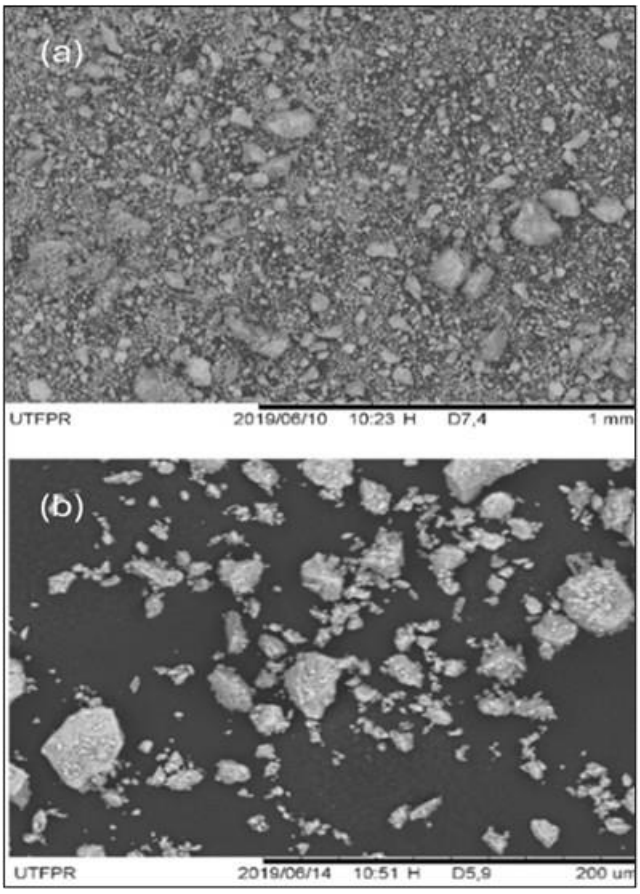
Scanning electron microscopy (SEM) is a technique that provides information about the surface morphology of materials, which is directly related to their adsorption capacity.

Figure 6 shows the images at scales 1:1 mm (a) and 1:200 µm (b) for nano-hematite.

Figure 6. Scanning Electron Microscopy (SEM) of the Hematite nanoparticulate. (a) 1:1 mm; (b) 1:200 µm



Its morphology can be described as amorphous with dispersed particles with heterogeneous clusters. In their research, Shrimali *et al*. (2016) found, with the SEM images, smooth and uniform surfaces of iron oxide particles. In contrast, Moreira *et al*. (2017) found uniform spherical and monodispersed nanoparticles for iron oxide, indicating that the process of obtaining hematite can significantly change its surface morphology.

After quantifying the components through the techniques of loss on ignition and X-ray fluorescence, it was possible to determine the concentration of silicon dioxide (SiO_2), other oxides in the sample, and the amount of heavy metals, respectively (**Table 2**).

This determination shows that, in addition to the hematite (Fe_2O_3), other substances may also influence the remineralizer's capacity as an adsorbent.

A large amount of SiO_2 (51.1%) was observed in the basaltic remineralizer sample (**Table 1**). SiO_2 is present in practically all rocks, sands, and soils, and silicates (a group of silicon oxides) represent about 27.7% of the earth's crust (Mori; Santos; Sobral, 2007). Another oxide with significant content (13.2%) was alumina (Al_2O_3), an aluminum oxide with great commercial importance and the main source of aluminum through the conversion of

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bauxite (Manhães; De Holanda, 2008). Hematite represents 15.2% of the total composition of basaltic remineralizer.

Table 2. Quantification of macro elements of the Basaltic Remineralizer

Macro elements	Content (%)
SiO_2	51.1
Fe_2O_3	15.2
Al_2O_3	13.2
CaO	9.86
MgO	5.97
K_2O	1.03
P_2O_5	0.28
MnO	0.22
Other components	3.14

Samples collected in the Triângulo Mineiro and characterized by Pires and Cardoso (2022) had similar contents of the main oxides, such as 48.91% SiO_2, 15.14% alumina, and 13.24% hematite. For basaltic remineralizer samples, Edward (2016) found 42.77% silicon dioxide, 11.59% alumina, and 23.10% hematite.

The heavy metal quantification of the remineralizer samples presented satisfactory results based on the CONAMA resolution 420/2008.

The CONAMA resolution 420/2008 references potentially toxic elements and ensures they do not threaten human and animal health. **Table 3** presents the optical emission spectrometry analysis (ICP-OES) for the remineralizer and compares the data with the minimum values determined by the resolution. The results show that this remineralizer can be applied in natural environments without negative consequences.

Table 3. Optical emission spectrometry (ICP-OES) analysis for heavy metal quantification of the basaltic remineralizer

Heavy metals	Remineralizer	Research Value - Industrial
Cu	253.8	600
Zn	63	2000
Cr	33	400
Ba	54	750
Ni	26.8	130
Mo	0.53	120
Co	23.7	90
Se	<1	***

***Undeclared limit of quantification

Rauen *et al*. (2021) also assessed the toxicity of hematite nanoparticles. The results

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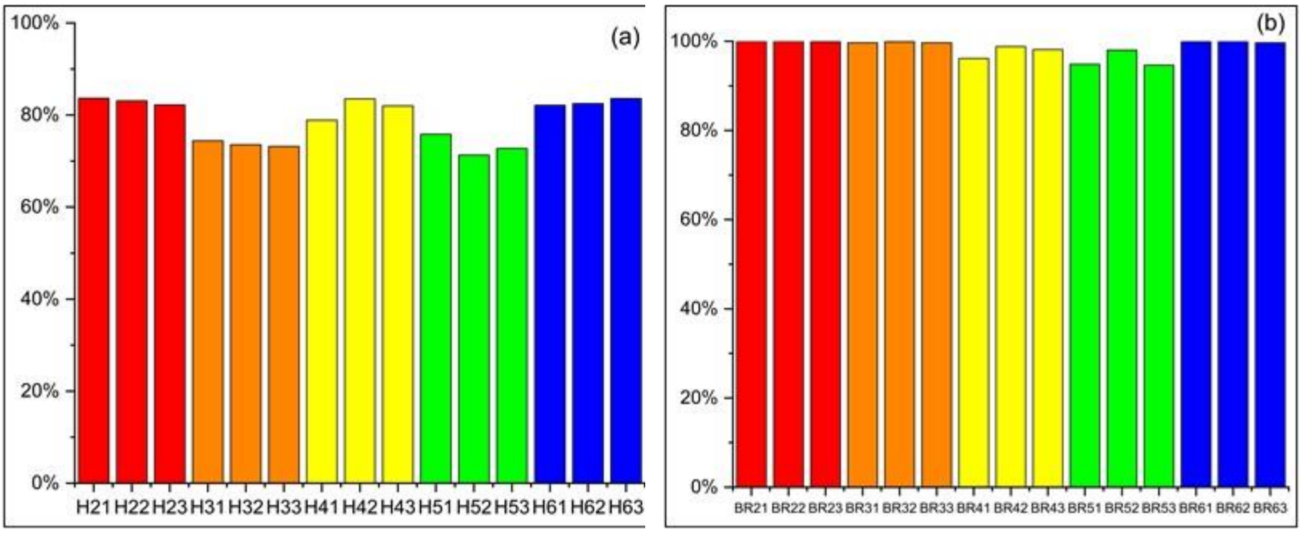
showed that this material, iron nanoparticles from industrial waste, can be used as a catalyst without adverse effects on the environment.

ADSORPTION ASSAYS

To verify the application of materials recovered from industrial activities in the recovery of impacted environments, adsorption tests were performed using basaltic remineralizer and nano hematite as adsorbents.

With the sample readings performed in a UV-vis spectrophotometer, the adsorption capacity and the percentage of removal of each adsorbent could be defined. **Figure 7** shows the removal capacity of adsorbents at each pH by hematite (a) and basaltic remineralizer (b).

Figure 7. Capacity of removing methylene blue by nano-hematite - (a) and basaltic remineralizer (b)



For sample classification purposes, the first letter indicates which adsorbent is used: H for hematite and P for remineralizer. The second character indicates the solution's pH (1 for pH 1, 2 for pH 2, [...]) and the last character indicates the point of the triplicate (0 for reference - without added adsorbent mass - 1, 2, or 3 for each point of the triplicate.

The result shows that both adsorbents had a high adsorption capacity throughout the pH range, and the remineralizer achieved almost 100% removal rates of methylene blue, whereas nano-hematite showed rates of 80% to 90%. This result converges to the zero-point of charge values found, and anion adsorption is favored in pH values below pH_{ZPC}.

At pH 2, the adsorption values were 2.08 $mg.g^{-1}$ and 1.73 $mg.g^{-1}$ for remineralizer and nano-hematite, respectively. The greater removal capacity of methylene blue by the remineralizer can be explained by the presence of other oxides with adsorbent capacity in the sample, such as aluminum oxide, identified by ICP-OES. Aluminum oxide is amphoteric and acts as a catalyst in several reactions, and its presence can speed up the adsorption process (Salla, 2017). As the heterogeneous catalysis process has an adsorption step, the molecules of methylene blue may have adsorbed on the surface of this oxide, as pointed out by the research of catalytic photodegradation of methylene blue based on the silica-structured alumina by Pereira (2017).

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In other studies of methylene blue adsorption from composts of humic-hematite acids, El Gaayda *et al*. (2022) obtained an adsorption capacity of 4.15 $mg.g^{-1}$ for isolated hematite and 13.40 $mg.g^{-1}$ for hematite-humic acid composite. For dye removal using leaching residue containing iron oxides such as goethite and hematite, Çetintas (2021) obtained an adsorption capacity of 3.07 $mg.g^{-1}$ to remove methylene blue from zeolite (silicon- and alumina-rich mineral), and Silva (2013) achieved a maximum adsorption capacity of 40.26 $mg.g^{-1}$.

The results showed that these materials recovered from industrial activities can be used as adsorbents with a positive response in removing contaminants in water media.

Southwestern Paraná, for example, is inserted in the area of occurrence of basalts, which are alkaline rocks rich in materials such as Ca, Mg, and K. These elements are important for the soil because they are inserted in the group of macronutrients, elements that plants need in high quantity. Remineralizers are obtained in the region as waste from quarries and therefore have great environmental importance since they become environmental assets rather than liabilities.

CONCLUSIONS

Our study assessed the potential of using basaltic remineralizer and nano-hematite recovered from industrial activities as adsorbents.

FTIR analysis showed characteristic peaks in the infrared region indicating the presence of iron and silicon in the basaltic remineralizer sample and iron and water in the hematite sample.

The loss on ignition defined the content of silicon dioxide at 51.1%. From the X-ray fluorescence analysis, we obtained the content of 15.2% hematite and 13.2% alumina in the basaltic remineralizer sample. The ICP-OES method returned results of 253.8 ppm copper, 63 ppm zinc, 33 ppm chromium, 54 ppm barium, 26.8 ppm nickel, 0.53 ppm molybdenum, and 23.7 ppm cobalt in the basaltic remineralizer sample.

Scanning electron microscopy demonstrated that the hematite nanoparticulate has an amorphous morphology with dispersed particles and heterogeneous clusters.

For the removal capacity of methylene blue at pH 2, we obtained the mean value of 2.08 $mg.g^{-1}$ for basaltic remineralizer and 1.73 $mg.g^{-1}$ for hematite nanoparticulate. The higher value for the remineralizer is linked to the presence of other components, such as alumina, in its composition.

Thus, it was possible to characterize these materials and evaluate the removal capacity of methylene blue dye via the adsorption process. The results were satisfactory and present a great potential for study, especially regarding the remineralizer, which can be used directly. On the other hand, the nano hematite recovered from acid drainage of mines requires many processes to obtain it. This assigns value and commercial application to the basaltic remineralizer.

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