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DECONTAMINATION OF WASTEWATER CONTAINING DYES USING IRON NIOBATE NANOPARTICLES ($FeNbO_4$)

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ABSTRACT

Increasing industrial progression and population growth have increased environmental concerns, especially the contamination of water resources by persistent organic pollutants, such as dyes. These compounds, widely used in the textile, food and pharmaceutical industries, have high chemical stability, intense coloring and potential toxicity, posing significant risks to health and the environment. Conventional treatment technologies often prove to be ineffective, reinforcing the need for innovative and sustainable alternatives. In this context, Advanced Oxidative Processes (AOPs) emerge as a promising strategy, due to the generation of highly reactive hydroxyl radicals capable of degrading complex molecules. This study reports the hydrothermal synthesis, characterization, and catalytic evaluation of iron niobate ($FeNbO_4$) nanoparticles, with a focus on their application for environmental decontamination. The synthesized material was characterized by X-ray diffraction, confirming its orthorhombic crystalline structure, good stability, and magnetic properties. The catalytic tests were carried out using the azo dye Amaranth as a model pollutant, under visible light irradiation and the presence of hydrogen peroxide. The results showed high degradation efficiency, with rapid color removal in the first 70 minutes, following pseudo-first order kinetics. The material also showed bifunctional surface charge behavior, with two points of zero charge (pH 3.0 and 6.73), favoring the adsorption and degradation of different contaminants. These findings highlight the multifunctional character of $FeNbO_4$, bringing together structural, magnetic and catalytic advantages that qualify it as a promising catalyst in ecotechnological processes. In the future, the aim is to optimize synthesis parameters and expand their application in real effluents, consolidating the potential of $FeNbO_4$ in sustainable remediation technologies.

Keywords: contamination, ferrite-niobium, photocatalysis.

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1 INTRODUCTION

With industrial advancement and population increase, new problems in relation to the restoration of water resources and wastewater treatment are emerging, considering that these factors have resulted in the pollution of water sources and harmful exploitation of the environment new problems in relation to the restoration of water resources and wastewater treatment are emerging, considering that these factors have resulted in the pollution of water sources and harmful exploitation of the environment (Da Silva Bruckmann et al., 2022b, 2025). Furthermore, the progress of (agro) industrial processes has also contributed to the generation of gaseous, liquid, and solid waste with a high concentration of contaminants, which become harmful factors for the environment due to incorrect disposal and inadequate treatment (Salles et al., 2024; Oviedo et al., 2022). Several processes can be used in water treatment, such as filtration, coagulation, and reverse osmosis(Bouzidi et al., 2025; Vidal Zancanaro et al., 2024). In this context, Advanced Oxidative Processes (AOPs) have currently stood out as a promising alternative, as they have the capacity to generate highly reactive hydroxyl radicals, capable of oxidizing and mineralizing complex molecules efficiently (Araújo et al., 2021; Nunes et al., 2024a). Given this scenario, finding more effective catalysts becomes an urgent need. The search for new materials is no longer just a scientific advance and has become a need for society, which requires sustainable solutions to environmental problems (Alves et al., 2017; Santos et al., 2025). With the progress of science, today it is possible to combine different materials and create composites with unique properties, such as electrical, thermal, magnetic, which expand the application alternatives in treatment technologies, making them more modern and efficient. (Bruckmann et al., 2022; Zananaro et al., 2024)

The presence of dyes released by textile, food, pharmaceutical and stationery industries has become one of today's greatest environmental challenges, compromising water quality and posing risks to living beings around the world (Da Silva Bruckmann et al., 2022a; Viana et al., 2025). These organic compounds have high chemical stability, resistance to biological degradation, and strong absorption in the visible region, which gives them an intense color(Nunes et al., 2024b; Periyasamy, 2024; Stefanello et al., 2024). In addition to harming water quality, many dyes present toxicity, mutations and carcinogenic potential, directly affecting aquatic organisms and posing a risk to human health (Bouzidi et al., 2024; Lin et al., 2023; Vargas et al., 2023). Conventional treatment technologies, such as physical-chemical and biological processes, are often not effective in completely removing these persistent pollutants, which reinforces the need for innovative strategies (Barbieri et al., 2024; Salles et al., 2024).

Brazil stands out in the production and refinement of niobium (Nb), in addition to being a pioneer in the development of technologies aimed at its application, which represents great growth potential (Nogueira, 2023). Nb is a transition metal that stands out mainly through its oxides. It appears naturally in the form of niobium oxide (Nb_2O_5), considered a material of great scientific and technological interest due to its peculiar properties. Nb_2O_5 has high thermal and chemical stability, is classified as non-toxic, and has semiconductor behavior, which makes it promising for different applications. This oxide can also crystallize in different polymorphic forms, which directly influence its structural, electronic, and catalytic properties. (Lin et al., 2023; Sun et al., 2021).

In turn, iron niobate ($FeNbO_4$) is a compound formed by the combination of iron and niobium, which has attracted attention due to its multifunctional properties. It has a semiconductor structure, good thermal and chemical stability, as well as magnetic characteristics that expand its field of use. These properties mean that $FeNbO_4$ is used in different applications, such as heterogeneous catalysis, electronic devices, and photocatalytic processes, including the degradation of organic pollutants. Thus, while niobium is valued mainly for its stable and versatile oxides, iron niobate emerges as a composite material capable of bringing together electronic, magnetic and catalytic properties in a single structure, making it promising for advanced technological and environmental applications (Devesa et al., 2024)

Most of the studies on the photocatalytic activity of niobium are focused on the degradation of dyes and organic compounds that can be presented as cations, anions, or neutrals. Because they have highly conjugated π systems, these pollutants have great molecular stability and strong absorption in the visible region, which gives them intense color. These properties make dyes ideal models for evaluating the efficiency of photocatalysts, in addition to representing a relevant environmental problem, as they are often present in bodies of water (Rianjanu et al., 2024; Ribeiro et al., 2025).

Therefore, the present work aims to prepare and characterize magnetic iron niobate ($FeNbO_4$) nanoparticles, investigating their structural, morphological, and functional properties, in order to evaluate their potential for application in innovative ecotechnological processes. The aim is to explore its photocatalytic activity for the degradation of dyes using photo-Fenton, contributing to the development of more efficient and sustainable methods of effluent treatment.

2 METHODOLOGY

2.1 SYNTHESIS AND CHARACTERIZATION OF IRON NIOBATE

The synthesis of iron niobate ($FeNbO_4$) was carried out by modifying the hydrothermal method proposed by Shim et al., (2012). Initially, equimolar solutions of the precursors $Fe(NO_3)_2$ H_2O and $NbCl_5$ were prepared, both with a volume of 50 mL. To ensure complete solubilization and dilution of the compounds, a mixture of absolute ethanol and distilled water was used, since ethanol helps to stabilize the solution and prevents the formation of precipitates.

The solutions obtained were subjected to continuous magnetic stirring for 30 minutes, promoting homogenization. Then, the pH of the solution was adjusted to 10 with the aid of ammonium hydroxide (NH_4OH). After adjusting the pH, the solution was transferred to an autoclave reactor with an internal Teflon chamber, which guarantees corrosion resistance and thermal stability of the system. The reactor was inserted into an oven and subjected to a hydrothermal process at 200 °C for 60 hours; these conditions facilitate the crystallization of $FeNbO_4$. After the hydrothermal reaction, the material obtained went through a successive washing and precipitation cycle using distilled water until a neutral pH was reached. This step is essential for removing residual ions, solvents, and reaction byproducts. Finally, the purified solid was subjected to a calcination process at 100 °C for 24 hours, aiming to structurally stabilize the material and eliminate possible organic residues originating from the precursors.

To evaluate its morphology, crystalline structure, and functional groups, $FeNbO_4$ was characterized by X-ray Diffraction (XRD), and the Zero Charge Potential (PCZ) was performed.

2.2 CATALYTIC TESTS

First, the solution is prepared with distilled water and amaranth CAS n. 915-67-3, $C_{20}H_{11}N_2Na_3O_{10}S_3$, is a dark red azo dye widely used as a model in photocatalytic degradation studies. The proportion used was 50 mg for 1 L, and the pH was adjusted to 3.0 with the help of Hydrochloric Acid (HCL), since the acidic medium favors the efficiency of the photocatalytic process. From this stock solution, the amount necessary for the experiment is removed. To use the iron niobate catalyst, the material was previously ground in a mortar to reduce particle size and ensure a greater contact area between catalyst and solution, also preventing the occurrence of unforeseen events during the kinetics, such as the formation of agglomerates that could compromise the homogeneity of the reaction. In a beaker (250 mL), 100mL of the produced solution and 50mg of the catalyst are inserted, which will be taken to a chamber with a magnetic stirrer with a commercial fluorescent lamp (85 W, Empalux, luminous flux 5195 lm) and another commercial LED lamp (50 W which is equivalent to 74 W of the fluorescent, Empalux, luminous flux 4500 lm) located 10 cm above the surface of the solution. The experiment began with the addition of 120µg of hydrogen peroxide (H_2O_2) to the solution, at which point the light source and magnetic stirrer were also turned on. During the tests, the sample was collected at different times, namely: 0, 5, 10, 20, 30, 40, 50, 60, 70, 80, and 90 minutes. Each sample taken was immediately subjected to centrifugation for 5 minutes, in order to separate the solid catalyst from the solution, and then the spectrophotometer at 525nm was used to determine if it was adsorbing. The discoloration kinetics of the solution was expressed by the ratio C/C_0 (= A/A_0) as a function of time, where: A_0 and A are the absorbances of the initial dye solution and at reaction time t, respectively. Visually, it was observed that, throughout the experiment, the red solution progressively lost its intense color, becoming increasingly lighter until it reached a transparent appearance. The only remaining shade at the end of the process was that of the catalyst in suspension, which was light brown in color, confirming the significant degradation of the dye. This result highlighted the potential of $FeNbO_4$ as a catalyst in heterogeneous photocatalysis processes, especially in the degradation of colored organic compounds present in wastewater.

3 RESULTS AND DISCUSSION

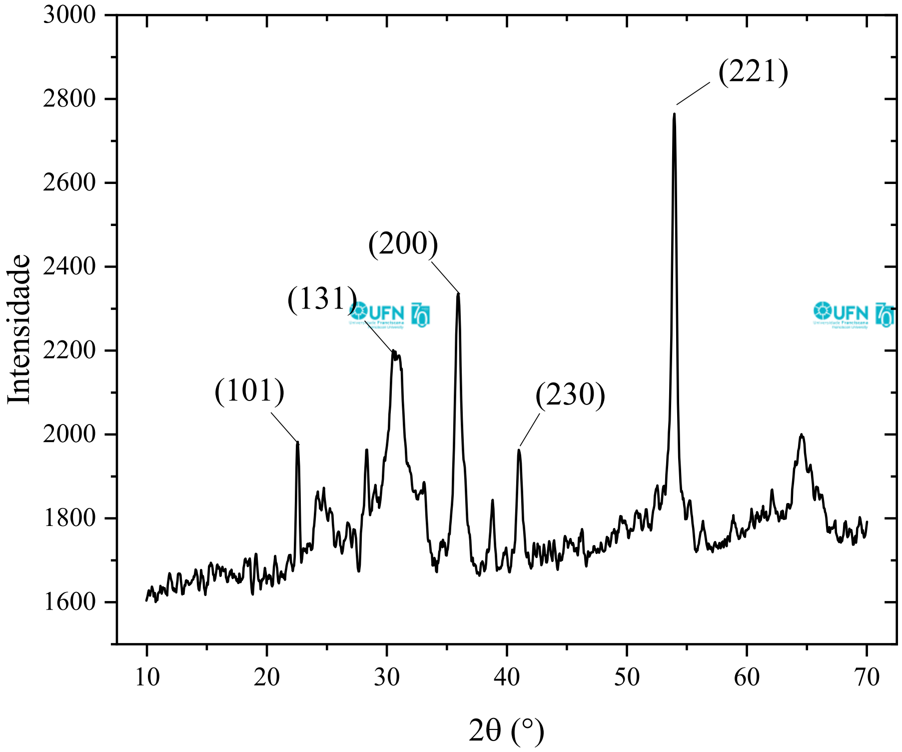
3.1 X-RAY DIFFRACTION (XRD)

One of the instrumental analysis techniques used in the characterization of iron niobate is X-ray Diffraction, where diffraction methods directly measure the distance between parallel planes of points in the crystalline lattice. This information is used to determine the lattice parameters of a crystal. Diffraction methods also measure the angles between planes (Rhoden et al., 2021)

X-ray diffraction analysis was carried out with the aim of confirming the formation of iron niobate ($FeNbO_4$) obtained experimentally (Figure 1). The diffractogram obtained presents characteristic peaks at 22.53°; 30.47°; 35.94°; 41.0° and 53.97°, which are in agreement with data reported in the literature, therefore confirming the formation of the compound with a well-defined crystalline structure. These angles correspond to the crystallographic planes (101), (131), (200), (230), and (221), characteristic of the orthorhombic crystal system, showing that the synthesized material has good crystallinity (Dhak et al., 2011; Khaliq et al., 2024)

The result obtained presents the most intense peaks that are related to the (200) and (221) planes, indicating that these crystalline directions have a greater degree of structural ordering, which may be associated with the predominance of certain orientations during crystal growth (De Andrade et al., 2020).

Figure 1: XRD of $FeNbO_4$

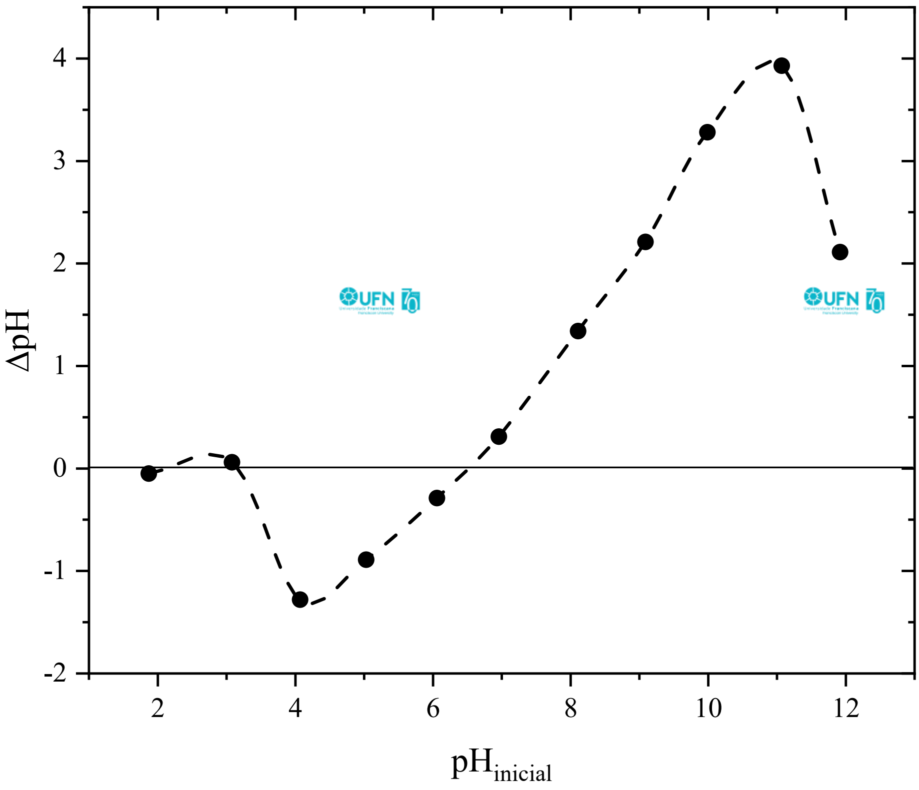


Source: Author construction (2025)

3.2 ZERO CHARGE POTENTIAL (PZC)

The results obtained for the variation in the pH of the medium in the presence of the catalyst are shown in Figure 2. Firstly, it is possible to observe that the material has two pHpzc points, one that occurs around pH 3 and the other that appears around 6.73. This behavior is consistent with the biphasic nature of the material and indicates the presence of many types of surface sites. In a strongly acidic environment, the first pHpzc is justified by the more pronounced acidic nature of the hydroxyl groups linked to iron, while the second point, which is close to neutrality, is caused by the less acidic nature of the groups linked to niobium. These properties are important in photocatalytic processes because the surface charge of the semiconductor directly affects the adsorption of target species and the interaction with oxidizing agents such as O_2 or H_2O_2 (Hamid, 2024; Kosmulski, 2023). This property expands the window of catalytic action and promotes the attraction of cationic contaminants in situations close to neutrality, as well as anionic contaminants in acidic environments. The dual acid-base nature of iron niobate can therefore increase photocatalytic efficiency by encouraging the production of reactive radicals under various operating circumstances (He et al., 2024)

Figure 2: Variation in the pH of the medium according to the initial pH



Source: Author construction (2025)

3.3 CATALYTIC TESTS GRAPH

Figure 3 presents the result of the decolorization efficiency for the 50 mg L^{-1} solution of the azo dye Amaranth, using magnetic iron niobate nanoparticles, in the presence of visible artificial light with a fluorescent lamp. It is worth noting that preliminary adsorption tests were carried out (without hydrogen peroxide and without an irradiation source) and showed negligible efficiencies in removing the dye from the solution. (Oliveira et al., 2016; Muhammad et al., 2025; Oliveira et al., 2019).High catalytic efficiency was observed for magnetic iron niobate nanoparticles, when comparing it with other catalysts in works reported in the literature for the degradation of the azo dye Amaranth (Rehman et al., 2021)

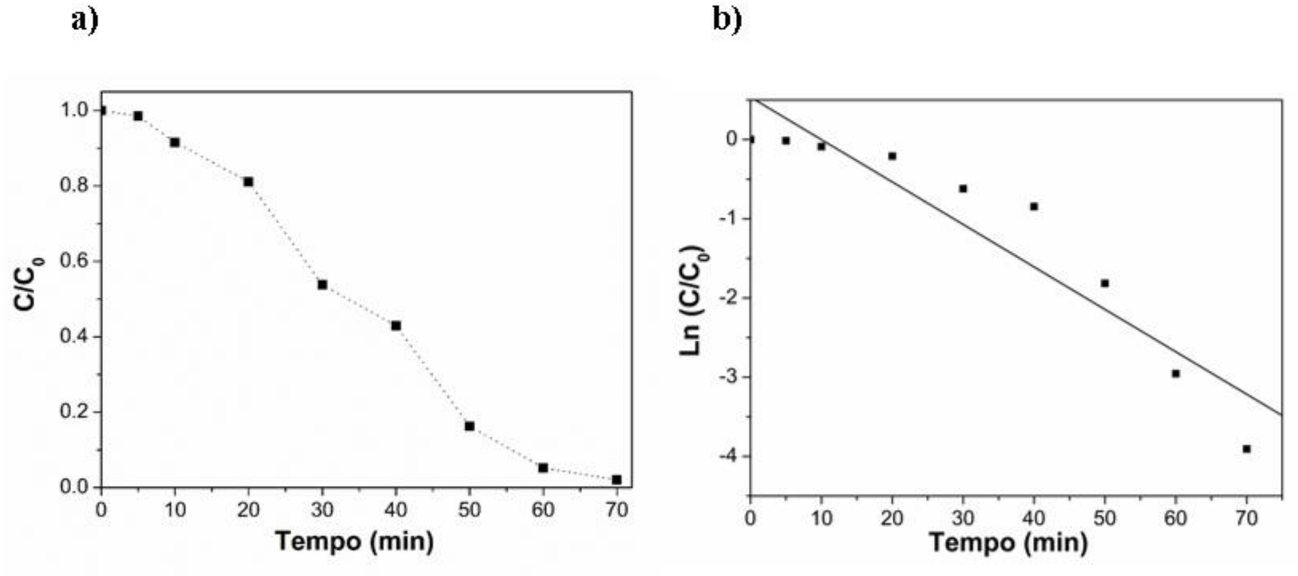
The results showed a significant decrease in the dye concentration, especially in the first 40 minutes of occurrence, followed by a tendency towards stabilization, reducing the handling of most of the molecules present.

To better understand the process, the data were adjusted to the pseudo-first order kinetic model, described by the research:

$$ln\left(\frac{C_0}{C_t}\right) = k_d t$$

Where C_0 is the initial concentration of the dye, C_t is the concentration over time and k_d is the rate constant for dye decolorization, determined from the slope of the respective straight lines. The fit showed good linearity, suggesting that the degradation of amaranth in the presence of $FeNbO_4$ predominantly follows pseudo-first order kinetics, which is consistent with the proposed mechanism for heterogeneous photocatalytic processes mediated by hydroxyl radicals (•OH).

Figure 33: Decolorization efficiency of the azo dye Amaranth (a) ln (C/Co) versus reaction time (b).



Source: Author construction (2025)

4 CONCLUSION

Hydrothermal synthesis proved to be an efficient route for obtaining iron niobate ($FeNbO_4$) nanoparticles, resulting in a material with good crystallinity, thermal stability and relevant magnetic properties. The XRD confirmed the formation of the desired phase, demonstrating the suitability of the method used. In photocatalytic tests, $FeNbO_4$ demonstrated significant performance in the degradation of the azo dye amaranth, achieving a significant reduction in color in a short period of time and following pseudo-first order kinetics. These results indicate that the material brings together structural and functional characteristics that qualify it as a promising catalyst for ecotechnological processes aimed at treating industrial effluents.

In addition to confirming the potential of $FeNbO_4$ as a sustainable alternative for the degradation of organic pollutants, this work paves the way for future investigations involving the optimization of the synthesis, the study of different reaction conditions, and the application of the material in real wastewater treatment systems, expanding its practical and environmental impact.

In the future, it is planned to characterize the material by nitrogen adsorption/desorption (BET), scanning electron microscopy coupled to energy dispersive X-ray spectroscopy, and diffuse reflectance spectroscopy (band-gap), and subsequently, evaluate its catalytic activity through heterogeneous photo-Fenton and Photocatalysis reactions in synthetic industrial effluent solutions. Therefore, the present work applies nanotechnology around wastewater treatment, meeting the Sustainable Development Goals (SDG).

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