

Biocomposites of chitosan and Fe/Mn-TiO₂ and its solar photocatalytic activity on amoxicillin degradation

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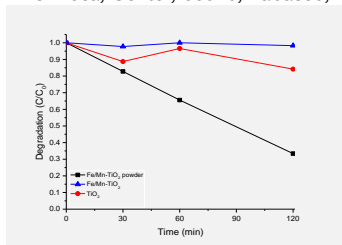
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This study investigates the preparation of biocomposites of chitosan and Fe/Mn-TiO₂ and its efficacy in amoxicillin degradation under solar radiation. Fe and Mn codoped catalysts were prepared by sol-gel method and characterized by XRD and IR. Biocomposites were prepared by casting method. Their photocatalytic perform presents a reduction in the activity of the biocomposites compared to the powders, attributed to limitations in mass transfer and chitosan degradation.

Introduction

Heterogeneous photocatalysis has been well-known process potential for to oxidation of complex organic molecules, like amoxicillin (AMX). This characteristic is promised to implement such a wastewater treatment. However, the catalyst presentation is a challenge. Many strategies have been used to support the catalyst in this process. Chitosan biocomposites have been attractive due to their abundance of availability, low cost, and adsorption properties[1]. In another way, the activation of catalysts against solar radiation is the aim to reach a sustainability process, however many catalysts present low activation with this radiation. Metal co-doping has presented promised results to improve the photocatalytic activity of the catalyst[2]. This work presents the synthesis of Fe/Mn-TiO₂, the preparation of chitosan biocomposite and its performance on amoxicillin degradation under solar radiation.

Material and Methods

Synthesis: Fe/Mn-TiO₂ powders were synthesized by the sol-gel method. Titanium (IV) butoxide was mixed with butyl alcohol. The mixture was stirred for 30 min at room temperature. The pH 3 was adjusted using glacial acetic acid dropped. After 30 min, FeNO₃ and MnSO₄ solutions were dropped to gel formation. The gel was aged for 24 h at room temperature. Posteriorly, the gel was dried at 80°C for 48 h. Finally, the powder obtained was calcined with a ramp of 5°C/min and 450°C for 2h.

Preparation of biocomposites:

Fe/Mn-TiO₂ biocomposites were synthesized by casting method[3]. Acetic acid solution (2 %w/w) was heated to 60°C under magnetic stirring. 0.25 g Fe/Mn-TiO₂ powder was added to the solution. Posteriorly, 0.50 g of commercial chitosan (low molecular weight, Sigma Aldrich) was added, and it was stirred (660 rpm) by 3 h at 60 °C. The mixture was transferred to a glass Petri dish 10 cm in diameter and subjected to 60 °C in a furnace for 24 h. Finally, the Petri dish with the film obtained was kept at room temperature for evaluation and characterization. TiO₂ biocomposite was prepared by similar conditions as control materials.

Characterization:

Fe/Mn-TiO₂ and TiO₂ powders were characterized by X-ray diffraction (XRD), optical microscopy and IR spectroscopy. Cell parameters and cell volume were obtained by UnitCell program. Fe/Mn-TiO₂ and TiO₂ biocomposites were cut in a circular geometry of ~0.5 cm diameter means a commercial single-hole punch hoper and their mass distribution were determined.

Reaction conditions:

In a volume reaction of 10 mL of 10 mg L⁻¹ of AMX, 3 g L⁻¹ of biocomposite or 1 g L⁻¹ of powder was added. The photocatalytic performance of the materials was evaluated under natural solar radiation in June 2024 in CIIT, Apodaca, Nuevo Leon, Mexico (25.771823°, -100.11173°, EPSG: 4326 WGS84) between 10:00 h – 13:00 h approximately. The environmental conditions data were collected from the

Environmental Monitoring System (Sistema de Monitoreo Ambiental) of Nuevo Leon, México of *Apodaca* and *Pesquería* stations. HPLC-DAD followed the AMX degradation. The kinetic of degradation was adjusted by the pseudo-first-order reaction. By multivariable statistics, the correlation between AMX degradation, characterization results, and environmental conditions.

Results and Discussion

Figure 1 shows the XRD patterns of Fe/Mn-TiO₂ and TiO₂. The anatase phase (RRUFF ID R060277) was determined. Fe or Mn oxides phases were not identified. However, a right displacement of Fe/Mn-TiO₂ suggests a co-doping due to Fe and Mn atomic radius are lower than Ti atomic radius. Also, the crystallite size was of 11.5 nm and 10.2 nm to TiO₂ and Fe/Mn-TiO₂ respectively. The *a* and *c* cell parameters and cell volume were 3.78 Å, 9.52 Å and 136.13 Å³ to TiO₂ and 3.78 Å, 9.49 Å and 136.00 Å³ to Fe/Mn-TiO₂, due to a cell contraction by metal co-doping indicated that Fe or Mn are incorporated substitutional into lattice crystalline of TiO₂ [2].

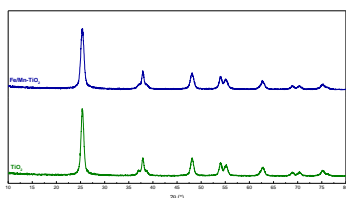


Figure 1. XRD patterns of TiO₂ and Fe/Mn-TiO₂ powders.

The figure in **Graphical abstract** presents the photocatalytic evaluation of powders and biocomposites for 120 minutes. It is observed a reduction in photocatalytic activity of biocomposites

Conclusions

Biocomposites of chitosan and Fe/Mn-TiO₂ show potential for amoxicillin degradation under solar radiation, but their efficiency is lower than that of catalytic powders due to limitations in mass transfer and possible degradation of chitosan.

Acknowledgments

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References

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- [2] C. Hu, C. Liu, K. Du, C. Pang, Z. Zhuo, and Q. Gao, *J. Environ. Chem. Eng.*, 11 (2023) 110440.
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compared to powders, this it attributed to mass transfer limitations. Additionally, chitosan may present a degradation during the degradation. To comprobred this, IR of biocomposites after used were record in **Figure 2** and **Figure 3**, that compared to biocomposites before used, the signals of 1541 cm⁻¹ to 896 cm⁻¹ to Fe/Mn-TiO₂ presented a transmittance reduction.

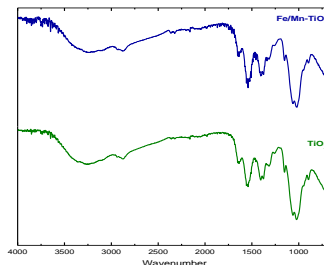


Figure 2. IR spectra of TiO₂ and Fe/Mn-TiO₂ biocomposites.

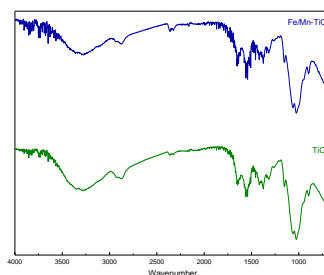


Figure 3. IR spectra of used TiO₂ and Fe/Mn-TiO₂ biocomposites.