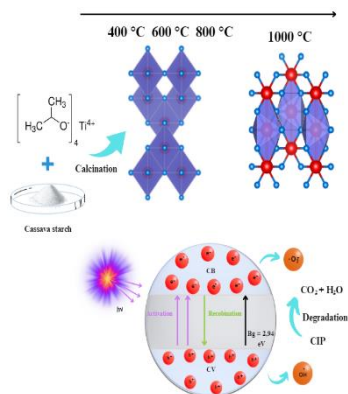


Development of an Efficient Photocatalyst Over a Wide Temperature Range for Pollutant Degradation: Exploring the Synergy between Cassava Gum and TiO₂

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Polysaccharides are emerging as a green alternative to modify the properties of semiconductors. In this study, we investigated the effect of cassava gum on the synthesis of TiO₂, varying the calcination temperatures, and analyzed its implications on the oxide properties and pollutant degradation. Analyses by X-ray diffraction, scanning electron microscopy, and diffuse reflectance spectroscopy highlighted the influence of both the polysaccharide and calcination temperature on the formation of crystalline phases, morphology, and optical properties such as the energy gap. Photocatalytic tests revealed a significant degradation (70.6% in 120 min) of Ciprofloxacin (CIP) under ultraviolet (UV) irradiation using 0.5 g/L of the TC400 catalyst (TiO₂ modified by the polysaccharide and synthesized at 400 °C), outperforming photolysis (10.63%).

Introduction

The increasing urbanization and industrialization have led to an alarming increase in the release of pollutants into the environment, posing significant challenges to water quality and public health [1]. Among the various classes of pollutants, pharmaceutical residues emerge as a particular concern due to their potential adverse effects on aquatic ecosystems and human health [2]. The persistence of these compounds in the environment is a cause for concern, as many demonstrate resistance to natural degradation processes, potentially negatively influencing essential biological processes, such as the increase in antimicrobial resistance [3].

In this context, photocatalysis is a promising approach for removing organic pollutants from wastewater [4]. Titanium dioxide (TiO₂) has been widely explored as a photocatalyst. However, to maximize efficiency and overcome the limitations associated with pure TiO₂, the search for more sustainable and environmentally friendly synthesis methods has gained prominence, driven by the need to develop materials with lower environmental impact. Thus, using cassava gum/starch (*Manihot* et al.) as a precursor in modifying TiO₂ appears to be an innovative and promising approach. Starch, an abundant, renewable, and biodegradable natural polysaccharide, presents properties that make it attractive for applications in photocatalytic materials [5].

This study aims to explore the influence of cassava gum on the synthesis of TiO₂ at different temperatures and to examine its impact on the resulting material's structural, morphological, surface, and optical properties. By understanding how the synergy between cassava gum and TiO₂ can

optimize the performance of the photocatalyst, we hope to contribute to the advancement of water treatment technologies and environmental remediation, providing more effective and sustainable solutions for the reduction of water pollution.

Material and Methods

The components used in the synthesis and photocatalytic activity included Ethyl Alcohol 99.8% (Aldrich), Ciprofloxacin (Dinâmica), Titanium isopropoxide 97% (Aldrich), Cassava gum (registered under number A923A49 in the National System for Management of Genetic Heritage and Associated Traditional Knowledge), and Ultrapure water. The samples were synthesized using the sol-gel method [1]. They are called TCX (T-TiO₂, C-cassava starch, X-calcination temperature). Initially, the mixture of 1 g of cassava starch with 100.0 mL of ethyl alcohol was kept under stirring for 30 minutes. After 30 minutes, 6.0 mL of ultrapure water was added slowly and left stirring for another 30 minutes; then, the solution was left to rest for 24 hours. After resting, the solution was dried in an oven at 75 °C. X-ray Diffraction characterized the material at different temperatures (XRD), Scanning Electron Microscopy (SEM), and diffuse reflection spectroscopy (DRS).

Photocatalytic studies used 0.5 g L⁻¹ of samples to degrade the CIP drug (1.0 x 10⁻⁵ mol L⁻¹) in aqueous solution. The solutions were irradiated using a commercial lamp (160 W) without a bulb as a source of UV radiation. The reactor used to contain the solution was coupled to a thermostatic bath and maintained at a constant temperature (25.0 ± 0.2 °C). Absorption in the dark for 30 min was measured before starting the photocatalytic test. The collected aliquots were centrifuged and verified using an

Agilent Technologies Cary 60 UV-Vis spectrophotometer in the 200 to 800 nm range. Photocatalytic efficiency was determined using Equation 1:

$$\% \text{Degradation} = (C - C_0) / C_0 \times 100 \quad (1)$$

C_0 represents the initial concentration of CIP, and C represents the drug concentration at different times during the experiment.

Results and Discussion

The X-ray diffractogram presented in Figures 1a and 1b, obtained from the sample calcined at 400 °C, revealed distinct peaks at 25° (101), 38° (004), 48° (200), 54° (105), 56° (211) and 63° (203), indicative of the presence of the anatase phase of TiO₂. As the calcination temperature increased, the intensity of these peaks increased, accompanied by the ap

The morphological analysis, illustrated in Figure 1c for TC400, highlighted the spherical shape of the nanoparticles. The EDS analysis, presented in Figure 1d, revealed characteristic Ti peaks, confirming the adequate synthesis of the NPs.

The band gap determination carried out based on data obtained by the DRS technique, showed a decrease in this value as the calcination temperature increased, with the TC1000 sample presenting the lowest value (2.94 eV).

Figure 1e displays the evolution of photocatalysis using the TC400 sample over different time intervals. In turn, Figure 1f demonstrates the degradation of CIP, with a reduction of approximately 10.63% under photolysis and 70.6% when subjected to photocatalysis of the material over 120 minutes.

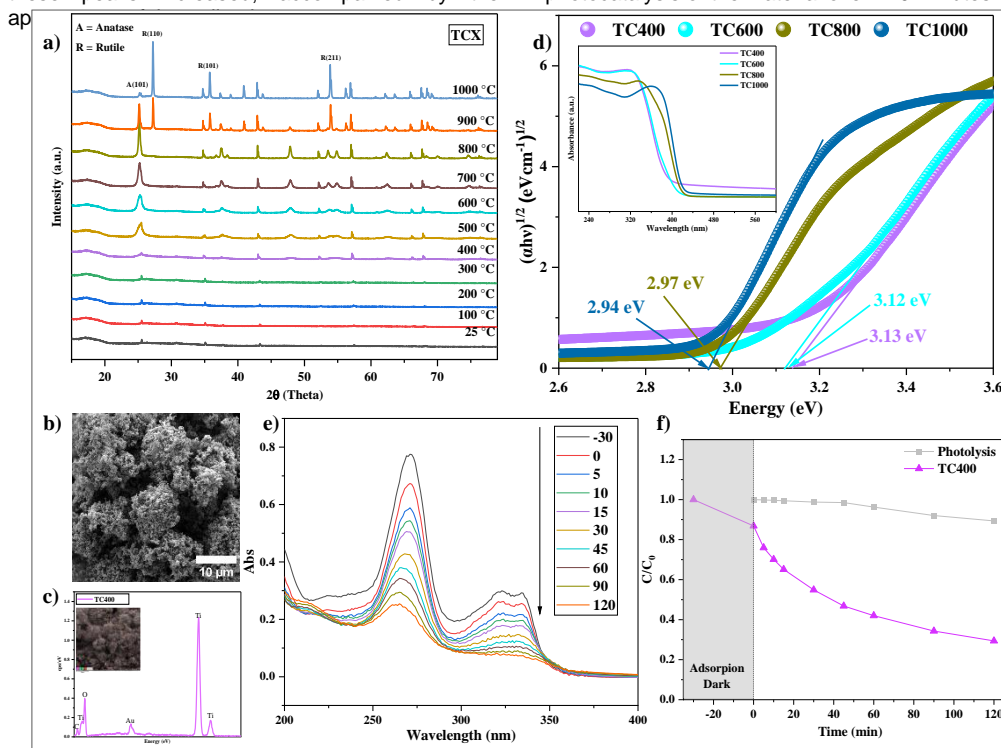


Figure 1. a) DRX, b) MEV, c) EDS, d) band gap, e) absorbance of aliquots, f) Concentration vs time.

Conclusions

TiO₂ nanoparticles were successfully synthesized from natural reagents. The calcination temperature at 400 °C allowed the formation of the anatase phase, which was confirmed by XRD. The images (SEM) demonstrate the formation of spherical nanoparticles. The material showed excellent photocatalytic activity for the degradation of methylene blue.

Acknowledgments

Feel free to contact the organizing committee for any further information.

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