

This study investigates the synthesis and characterization of TiO₂-babassu mesocarp polysaccharide composites for environmental remediation applications. Optical, structural, and textural analyses demonstrate the composite's efficacy in degrading organic pollutants, with significant degradation rates observed in photocatalytic testing. The composites exhibit promising characteristics, including a band gap value of 2.99 eV, mesoporous structure, and efficient degradation of methylene blue dye. These findings underscore the potential of TiO₂-babassu mesocarp composites as sustainable solutions for mitigating environmental pollution.

Introduction

Organic pollutants from diverse sources pose a significant global environmental threat, necessitating effective remediation strategies. Advanced Oxidative Processes (AOPs), particularly TiO₂-based heterogeneous photocatalysis, offer the potential to degrade such contaminants by generating reactive oxygen radicals. However, TiO₂'s efficacy is hindered by limitations like low efficiency [1].

Incorporating TiO₂ into natural polysaccharide matrices, like babassu mesocarp, presents a promising solution due to benefits such as biocompatibility and structural adaptability [2].

This study aims to synthesize TiO₂ with babassu coconut mesocarp polysaccharide, evaluate its crystalline structure stability at different temperatures, and evaluate its efficiency in degrading methylene blue dye.

Material and Methods

The babassu mesocarp was obtained from the Babcoall project, which was incubated by UFPI/INEAGRO. TTIP from Sigma-Aldrich and ethyl alcohol from Dynamic served as TiO₂ precursors. Methylene blue (97.0%) from Dynamic was used for photocatalysis. TiO₂ composites were prepared via sol-gel method with 1% (m/v) babassu mesocarp, TTIP, polysaccharide solution, and ultrapure water. After drying at ~75 °C, samples were calcined at 400 °C for 2 h. Composites containing babassu were labeled as 1BBT. TiO₂ was also synthesized without polysaccharide using the same method.

Optical characteristics analyses were conducted using Diffuse Reflectance Spectroscopy, employing the Shimadzu UV-2600 spectrometer. The sample band gap was determined using the conventional Kubelka-Munk method and the Tauc equation (Equation 1) from spectra obtained by the Shimadzu UV-Vis spectrometer UV-2550.

$$\alpha h\nu \propto (h\nu - E_g)^n \quad (1)$$

Where α represents the absorption coefficient, h is the Planck constant, ν is the radiation frequency, E_g indicates the band gap energy, and n refers to the type of electronic transition involved, where the value of 1/2 is employed.

XRD tests with temperature variation were performed using a Bruker D8 Advance powder diffractometer with an Anton Paar XRK900 reaction temperature chamber. Textural properties were analyzed via N₂ adsorption-desorption at 77 K using Quantachrome equipment. The BET method determined the surface area, while pore volume distribution and diameter were estimated using the BJH method from N₂ adsorption curves.

A 12.0×10⁻⁶ mol L⁻¹ MB dye solution was used for photocatalytic testing with a catalyst concentration of 0.5 g L⁻¹. Equilibrium was reached in 30 minutes in the dark. Tests were performed in a 100 mL borosilicate reactor at 25°C using a 160 W commercial mercury vapor lamp emitting UV light.

Results and Discussion

Figure 1 shows the band gap value of the material obtained using the Tauc plot method. It is noted that the obtained value of 2.99 eV is slightly lower than that reported in the literature for pure TiO₂, as this material typically exhibits a band gap value of around 3.2 eV for the anatase phase [3].

In Figure 2, the formation of the TiO₂ crystalline structure is observed starting from 200°C, with peaks becoming clearer from 300°C onwards. As the complete decomposition of the babassu mesocarp occurs above 500°C, the peaks become more intense, attributed to its semi-crystalline nature. The material remained in the anatase phase between 300°C and 700°C, with the rutile phase of TiO₂ forming above this temperature.

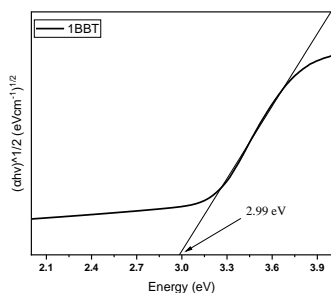


Figure 1. Band gap energy is determined by Touc's plot method.

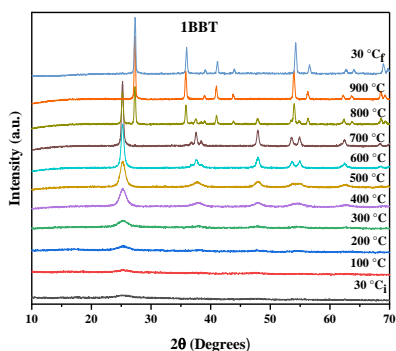


Figure 2. X-ray diffraction with temperature variation of the 1BBT composite.

In Figure 3, the 1BBT nanocomposite exhibited isotherms resembling type IV and H2-type hysteresis. This isotherm shape is characteristic of mesoporous materials [4]. The values of specific surface area, pore volume, and average pore diameter are observed in Table 1, with the respective values being $95.41 \text{ m}^2 \text{ g}^{-1}$, $0.11 \text{ cm}^3 \text{ g}^{-1}$, and 4.60 nm .

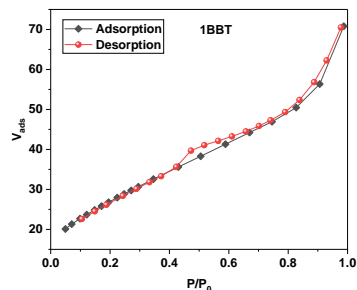


Figure 3. Adsorption-desorption isotherm of 1BBT.

In Figure 4, the degradation rate of methylene blue dye through photocatalysis and photolysis is observed. It was noted that through the photocatalysis process, the dye exhibited a degradation percentage of 69.34%, while in photolysis, the percentage was only 2.97%. The catalyst absorbs light, generating reactive species that initiate the degradation process of the chain molecule by forming electron-hole pairs. The hole in the photocatalyst's conduction band produces the hydroxyl radical, while the electron in the valence band can react with molecular oxygen to produce the superoxide ion radical [5].

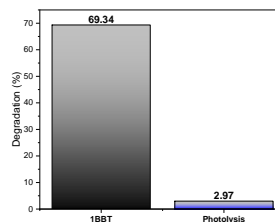


Figure 4. Discoloration rate for Methylene blue solution.

Table 1. Specific surface area, pore volume, and pore diameter distribution of the 1BBT composite.

Sample	Specific Surface Area ($\text{m}^2 \text{ g}^{-1}$)	Pore volume ($\text{cm}^3 \text{ g}^{-1}$)	Average Pore Diameter (nm)
1BBT	95.41	0.11	4.60

Conclusions

This study demonstrated the efficacy of TiO_2 -babassu mesocarp polysaccharide composites in degrading organic pollutants, with promising results in optical, structural, and textural analyses. The photocatalytic testing revealed a significant degradation rate of methylene blue dye, highlighting the potential of these composites for environmental remediation. These findings contribute valuable insights towards developing sustainable solutions for addressing environmental pollution challenges.

Acknowledgments

LIMAV, PPGCM, INCT POLISSACARÍDEOS, UFPI, FAPEPI, CAPES, CNPq.

References

- [1] F.C. Monteiro, I.D.L. Guimaraes, P. de Almeida Rodrigues, J.V. da Anunciação de Pinho, C.A. Conte-Junior. J Photochem Photobiol, A Chem 437 (2023) 114497.
- [2] J.M. Abisharani, S. Devikala, R. Dinesh Kumar, M. Arthanareeswari, P. Kamaraj, Mater Today Proc 14 (2019) 302.
- [3] F. Mughal, M. Muhyuddin, M. Rashid, T. Ahmed, M.A. Akram, M.A. Basit, Chem Phys Lett 717 (2019) 69.
- [4] Preeti, S. Banerjee, A. Debnath, V. Singh, Environ Nanotechnol Monit Manag 15 (2021) 100459.
- [5] F.P. Araujo, P. Trigueiro, L.M.C. Honório, D.M. Oliveira, L.C. Almeida, R.P. Garcia, A.O. Lobo, W. Cantanhêde, E.C. Silva-Filho, J.A. Osajima, Int J Biol Macromol 165 (2020) 2813.