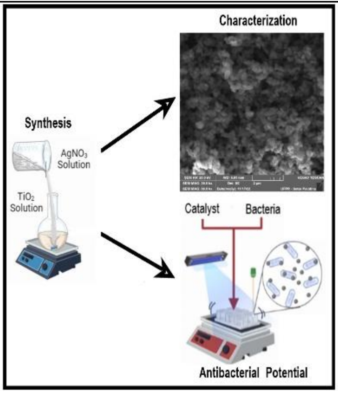
Synthesis, Characterization And Antibacterial Potential Of Ag/TiO$_2$

POSTER
Ph. D.Student: N

Journal: Chemical Engineering Journal

***L. M. S. Colpini*[1]**, *C. Zarzzeka*[1], *J. Goldoni*[2], *F. Marafon*[2], *G. B. da Silva*[3], *D. Manica*[4], *G. G. Lenzi*[5], *R. Brackmann*[6], *M. D. Bagatini*[2] *(1) Federal University of Parana, Pioneiro Street 2153, 85950-000, Palotina, PR, Brazil, ledasaracol@ufpr.br. (2) Federal University of Fronteira Sul, Highway SC 484/Km 02, Southern Border, 89815-899, Chapecó, SC, Brazil. (3) State University of Santa Catarina, Luís de Camões Avenue 2090, Money Account, 88520-000, Lages, SC, Brazil. (4) Federal University of Santa Catarina, University Campus Rector João David Ferreira Lima, Trinity, Florianopolis, SC, Brazil. (5) Federal University Technology of Parana, Doctor Washington Subtil Chueire Street 330, Oak Garden, 84017-220, Ponta Grossa, PR, Brazil. (6) Federal University Tecnology of Parana, Via do Conhecimento, s/n - Km 01 – Fraron, 85503-390, Pato Branco, PR, Brazil.*



The results are based on silver (Ag) (2% and 10%) supported on titanium dioxide (TiO_2) - Ag/TiO_2, synthesized by the excess solvent impregnation method, containing titanium bands and asymmetric vibration of Ti–Ag–O with agglomerated and irregular morphologies as Ag was added. Determination of N_2 physisorption provides that the addition of Ag decreases the specific area of the impregnated mesoporous finds. The studies tested were not photoactivated by white light (9 W), so the antibacterial action is related to the concentration of 10% Ag present in the specific. Similar results were found in the dark test. The test under black light photoactivated all catalysts against *E. coli*, this rapid action originating from reactive oxygen species was proven in the detection test. The impregnated results have rapid and enhanced antimicrobial effects compared to TiO_2, with applications in disinfection devices.

Introduction

Diseases caused by bacteria, viruses, and fungi represent a global threat, which can spread rapidly through contaminated food and water, through the air via sneezing, breathing, and coughing, or through contact with untreated wounds [1]. There are drug-resistant pathogens that can be inactivated by materials activated by light, offering a promising solution against these microorganisms [2], without promoting resistant bacteria [3]. Thus, photocatalysis is based on the ability to generate reactive oxygen species (ROS) from the irradiation of light sources emitting different wavelengths. ROS promotes lipid oxidation and the disruption of the membrane of microorganisms [4]. Among the materials used in photocatalysis is Ag/TiO_2, which can be used against pathogenic bacteria and in antimicrobial coatings for pathogen disinfection, preventing their creation and proliferation [5]. With the aim of exploring new strategies to identify and develop the future generation of drugs or agents to control bacterial infections, this study demonstrates the superior bactericidal effects of Ag/TiO_2 catalysts, thus developing antibacterial materials with a possibility of less expensive and broader application. Thus, efforts were made to synthesize mixed oxides of Ag/TiO_2 (2 and 10% mass ratio of Ag to TiO_2), which were characterized for their physicochemical properties in the action of bacterial inactivation.

Material and Methods

TiO_2 was dissolved in water and silver nitrate - $AgNO_3$ (2 and 10% by mass) was added. The solution was kept in a rotary evaporator for 17 hours at room temperature. Then, the material was dried at 80 °C and calcined at 400 °C for 5 hours. The catalysts 2AgT/I (2% Ag/TiO_2), 10AgT/I (10% Ag/TiO_2), and T (TiO_2) were characterized by: Fourier Transform Infrared Spectroscopy (FT-IR); Scanning Electron Microscopy (SEM) and Nitrogen Physisorption. The microbial activity test was performed with E. coli (NEWP0022) under white light (9 W), black light (36 W), and in the dark. The times tested were 0, 5, 10, and 15 minutes. We employed a protocol established by [6] with adaptations, to detect ROS in bacterial samples using 2′,7′- dichloro fluorescein diacetate (H2DCF-DA). Results were expressed as percentage (%) of ROS relative to 0 min. Statistical analyses were performed using GraphPad Prism 9.0 software, academic trial version. Normality was assessed by Kolmogorov-Smirnov and outliers were excluded by Grubbs' Test.

Results and Discussion

The FT-IR spectra of AgT/I showed a slight shift of peaks around 660–700 cm^{-1}, indicating the asymmetric vibration of the Ti-Ag-O bond. The micrograph of the T catalyst exhibited a homogeneous and spherical aspect. However, as the Ag doping increased in the AgT/I catalysts, sponge-like and irregular clusters formed, originating from the aggregation process. Type II isotherms were obtained for the impregnated catalysts, thus characterizing mesoporous materials. Regarding the antimicrobial activity tests, graph D, **Figure 1**, showed bactericidal action from the 10-minute mark due to the Ag concentration that produces ROS, which accumulated near bacterial membranes, causing leakage and death. Graph 1H yielded similar results, corroborating with the tests in clear LED light. An increase in colony number (0-10 min) was observed in graph 1J, indicating that UV light repaired E. coli due to a sublethal dose, thus promoting its cellular regeneration. There was a decline in colony number for the T catalysts (Figure 1J) and 2AgT/I (Figure 1K) from the 15-minute mark and for the 10AgT/I catalyst (Figure 1L) from the 5-minute mark, indicating photoactivation by black light, suggesting antibacterial action. This effect was caused by increased ROS generation, surpassing E. coli photorepair. The 2AgT/I (Figure 1K) and 10AgT/I (Figure 1L) catalysts exhibit more pronounced photocatalytic effects than the T catalyst (Figures 1J). The AgT/I catalysts prove to be more effective due to the synergistic effect of Ag and TiO2, thus resulting in the combination of ROS production, Ag release, and also greater particle stability. **Figure 2** demonstrates an increase in intracellular ROS generation values of E. coli for the 10AgT/I catalyst from the 5-minute mark, thus confirming the data presented in Figure 1L, i.e., ROS were generated from photocatalytic effects, demonstrating the effects of antibacterial activity. ROS accumulate and overload the antioxidant defense mechanisms of bacteria, causing membrane damage and cell death.

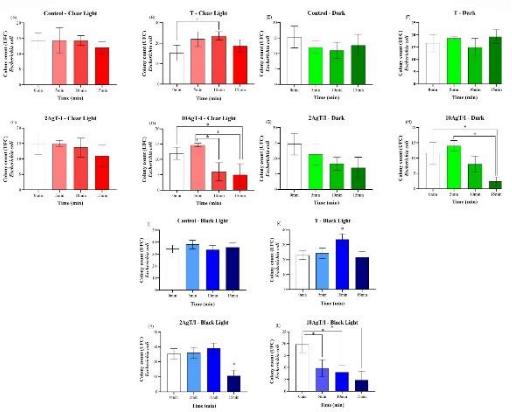
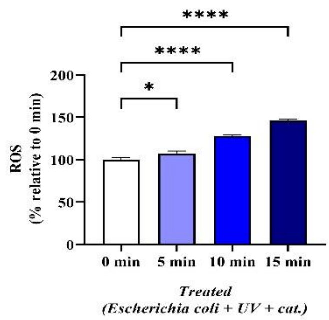


Figure 1. Antibacterial action against E. coli of the control (A, E, and I) and the T catalysts (B, F, and J), 2AgT/I (C, G, and K), and 10AgT/I (D, H, and L) in the clear test (A-D), dark (E-H), and black light (I-L), according to the time variation of 0, 5, 10, and 15 minutes. Asterisks indicate $p < 0.05$ (one-way ANOVA, Tukey post-test).

Figure 2.



Intracellular ROS generation values of E. coli from the ROS detection assay by H2DCF-DA for the 10AgT/I catalyst. Asterisks indicate $p < 0.05$ (one-way ANOVA, Tukey post-test).

Conclusions

The AgT/I catalysts exhibit promising antimicrobial effects, with the potential for application in rapid bacterial disinfection devices, offering the possibility of less expensive and broader application in antibacterial therapy.

References

[1] D. Rodríguez-Lázaro, N. Cook, F.M. Ruggeri, J. Sellwood, A. Nasser, M.S.J. Nascimento, M. D'Agostino, R. Santos, J.C. Saiz, A. Rzeżutka, A. Bosch, R. Gironés, A. Carducci, M. Muscillo, K. Kovač, M. Diez-Valcarce, A.Vantarakis, C.-H.von Bonsdorff, A.M. de Roda Husman, M. Hernández, W.H.M. van der Poel, *FEMS Microbiol. Rev.*, 36 (2012), 786.

[2] P. Makvandi, C. Wang, E.N. Zare, A. Borzacchiello, L. Niu, F.R. Tay, *Adv. Funct. Mater.*,30, (2020).

[3] V.G. Benatto, J.P.A. de Jesus, A.A. de Castro, L.C. Assis, T.C. Ramalho, F.A.La Porta, *Mater. Today Commun.*, 34 (2023), 105192.

[4] M. Fioreze, E.P.Santos dos, N.Schmachtenberg,: *Rev. Eletrônica em Gestão, Educ. e Tecnol. Ambient.* 18, (2014).

[5] A.Kumar, P. Choudhary, A. Kumar, P.H.C. Camargo, V. Krishnan, *Small*, 18, (2022).

[6] C. Liao, Y. Li, S.C. Tjong, *Nanomaterials.* 10 (2020), 124.