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Photocatalytic CO_2 conversion: sol-gel and aerosolization synthesis of Cu_xO-modified TiO_2 over 3D printed supports

Pascual. J.[1], Altieri, T.[2], Lombardo, M. V.[2], Montesinos, V. N.[1,3], Quici, N.[1,3]

(1) Centro Tecnologías Químicas – Dpto. de Ingeniería Química, FRBA, UTN, Medrano 951, CABA, Argentina
(2) Gerencia de Química - INN – CNEA, CONICET, Av. Gral. Paz 1499, Villa Maipú, Argentina,
(3) Gerencia de Química – CNEA, Av. Gral. Paz 1499, Villa Maipú, Argentina.
natalia.quici@gmail.com

Cu_xO-modified TiO_2 photocatalysts are powerful materials for conversion of CO_2 in methane. However, it is necessary to develop scalable synthesis routes that led to photocatalysts with improved conversion efficiency, elevated photocatalytic activity and low environmental impact.

3D printing facilitates the construction of precise geometrically-controlled reactors in short production times that can speed up the thinking-designing-production cycle of reactors minimizing waste generation.

In this work we synthesized Cu_xO-modified TiO_2 photocatalysts following two different routes: (1) modification of commercial TiO_2 by surface precipitation of Cu^{2+} (Cu_xO-TiO_2) and (2) one-pot aerosolization of TiO_2 and Cu_xO precursors (Cu_xO@AerTiO$_2$). The solids were characterized by diffuse reflectance spectroscopy, SEM and XRD. Exploratory experiments for impregnation of photocatalysts in PET monoliths were undertaken.

The modification of commercial TiO_2 (Aeroxide P25 or Hombikat UV100) was carried out by dropwise addition of 62.9 mL of a 0.05 M Cu^{2+} solution to 400 mL of a 12.5 g/L of TiO_2 suspension in NaOH 0.25 M under continuous stirring. After 4 h, the solid was filtered, washed, dried at 80 °C overnight and, finally, calcinated at 400 °C for 2 h. The effect of copper salt ($CuCl_2$ vs. $CuNO_3$) and Cu loading (0.28, 1 and 5 Cu/Ti At%) was studied. XRD patterns clearly showed the presence of CuO in all cases over the P25 samples, in addition to the anatase and rutile typical signals for the base photocatalyst. When $CuCl_2$ was used as Cu_xO precursor, well defined nanoparticles of 80 nm where obtained. The synthesis with $CuNO_3$ gave a smooth homogeneous aspect to the solid surface with no distinguishable nanoparticles.

The synthesis through aerosolization was based on the one described in the work of Zelcer et al. [1]. Briefly, a solution containing 1.39 g acetylacetone, 1.39 g of glacial acetyc acid, 0.34 g of Ti-iPrOH, 0.0145 g of $Cu(NO_3)_2.3H_2O$ and 0.2 g of Pluronics F127 in 43 mL of MilliQ water was continuously fed to a Büchi B-290 spray drier with a peristaltic pump at a flow rate of 3 mL/min. The liquid was atomized at 220 °C in a two-fluid nozzle with a secondary air-flow of 473 L/h. After collection of the material from the cyclone of the spray drier, the solid was calcined at 440 °C for 4 h.

The band-gaps of the synthetized photocatalysts were calculated by the Tauc method being 3.13 eV, 3.17 eV and 3.15 eV for the Cu_xO-P25, Cu_xO-UV100 and Cu_xO@AerTiO$_2$ powders, respectively.

Square-based monolith of 3 × 1.5 × 3 cm with 15% of PET loading were impregnated by dip-coating in suspensions of 1 – 20 g/L of P25. Once dried at 50°C overnight, all of them were irradiated from one side with a 365 nm UV-LED and the UV-light intensity was measured before and after passing through the impregnated monolith by a 365 nm radiometer. The use of 1 g/L of TiO_2 was found as the best option as it absorbs 90% of the UV-light, against the 92.3% absorbed by the monolith impregnated in the 20 g/L suspension. In all cases, the diffuse reflectance spectrum obtained for the Cu-modified TiO_2 impregnated supports matched the spectrum of the respective powders.

The solids synthesized presented promising structural and optical properties and, currently, XRD and SEM analysis are being completed, together with porosimetry for all solids to have broader and comparative information.

References

1. *Zelcer, A., Franceschini, E.A., Lombardo, M.V., Lanterna, A.E., Soler-Illia, G.J.A.A. (2020) A general method to produce mesoporous oxide spherical particles through an aerosol method from aqueous solutions, Journal of Sol-Gel Science and Technology*, 94, 195–204.