

UV AND VISIBLE LIGHT-INDUCED DEGRADATION OF METHYL ORANGE BY TiO₂ NANOTUBES/Ag NANOPARTICLES SYNTHESIZED BY MICROWAVE ASSISTED CHEMISTRY.

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TiO₂-based materials are widely used in photocatalysis due to their high reactivity, physical and chemical stability, low toxicity and cost. In particular, TiO₂ nanotubes (NTs) have been used recently because they are effective for the photocatalytic degradation of various organic contaminants. However, TiO₂ is active under UV active light limiting their wide spread use under solar irradiation. Several methods have been developed to synthesize nanostructured crystalline TiO₂, including sol-gel technique, hydrothermal process, anodization, and so on. Besides these established synthesis methods, the hydrothermal process assisted by microwave has been shown to be superior due to rapid and homogeneous heating and fast kinetics of crystallization¹. In this study, a green and facile one-step microwave-assisted reaction is reported which is capable of synthesize TiO₂ NTs or Ag nanoparticles (NPs) that can be combined in a second step to produce a visible light active photocatalyst.

TiO₂ NTs and Ag NPs were synthesized by microwave irradiation using a commercial MARS 6 and a standard Panasonic apparatus respectively. P25 (EVONIK) material was used as a TiO₂ precursor for the synthesis of TiO₂ NTs in 9 mol L⁻¹ NaOH aqueous solution. A power of 300 W during 2 h at 180 °C was used. Finally, TiO₂ NTs were washed, neutralized and annealed at 400 °C for 3 h in air atmosphere to obtain anatase structure. Silver NPs were prepared from the desired solution mixture in a Teflon home-made closed reactor using a power of 1600 W (2450 MHz). Ag NPs were stabilized with polyvinylpyrrolidone (PVP) or polyacrylamide (PAM). TiO₂ NTs and Ag NPs were characterized by TEM, SEM (EDX), XRD, XPS, BET and UV-diffuse spectroscopy. Photocatalytic experiments under UV or visible light irradiation were carried out in a quartz photochemical reactor. The change in concentration of the dyes was monitored regularly by measuring the absorbance at 465 nm as previously described². Unfiltered or filtered light ($\lambda \geq 400$ nm) from a high pressure Xe/Hg lamp of 150 W (Sciencetech Inc.) was used as excitation source. Metal Orange (MO) was used as prototype dye to test the photodegradation activity.

TEM images of the TiO₂ NTs with lengths between 50-500 nm, internal diameters of about 3.7 nm and wall thickness of 2.1-5.5 nm were prepared. Ag NPs of sizes between 1.8 nm (PAM) and a multimodal distribution 0.9-11.9 nm (PVP) were loaded on the TiO₂ NTs by simple immersion process. After thermal treatment at 450 °C for 1 h, the photocatalysts were tested for degradation of MO aqueous solutions. Pure TiO₂ NTs photocatalytic degradation rate contrasted with the activity of the TiO₂ NTs loaded with Ag NPs. Under UV-vis irradiation the TiO₂ NTs/Ag NPs photocatalyst degraded the MO to complete decolouration of the solution in 3 h of irradiation. Additionally, irradiation of this system with $\lambda \geq 400$ nm shows an efficient photodegradation process without direct photochemical activation of TiO₂.

The results presented here showed the potential of the prepared photocatalyst for degradation of dyes using solar radiation.

1. V. Polshettiwar, et. al, Green Chemistry 12, 743 (2010)
2. C. W. Backes, et. al, C. W, J. Braz. Chem. Soc. 25, 2417 (2014).

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