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Analysis of colorimetry using the CIE-L*a*b* system and the photocatalytic activity of photochromic films

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ABSTRACT

This work aims to explain how oxide films are correlated when exposed to UV-A radiation, due to the occurrence of similar phenomena. Thus, thin films of TiO_2 and TiO_2 doped with H_2WO_4 or $Na_2WO_4{\cdot}2H_2O$ were made by spin-coating, heat treated between 650 and 800 °C and evaluated for their optical and photocatalytic properties. The X-ray diffraction technique (XRD) was used to determine the crystalline structure of the films. The photochromism was observed through a spectrophotometer using the CIE-Lab colorimetric method. The analysis of the films photocatalytic behavior was obtained following the photodegradation in 125 ml of methyl orange dye (20 ppm). The results showed that the films presented good photochromic and photocatalytic properties due to the synchronization between the chemical and physical properties of TiO_2 and tungsten.

1. Introduction

Semiconductors are part of the category of materials of high technological relevance and have been widely used in electronic devices, photovoltaic cells, sensors, and photocatalysts since they have excellent electrical, optical and magnetic properties. The number of studies involving these properties has been increasing, aiming to develop new materials [1]. These semiconductors can be used in a variety of ways, including powder and immobilized form [1].

Photochromism in metallic oxides was observed for the first time in 1969, where WO_3 appears bluish in the presence of 0.1 M H_2SO_4. Other metal oxides, such as molybdenum, vanadium, and titanium also exhibit photochromism when exposed to electromagnetic radiation, commonly under UV irradiation and more rarely under visible light [2].

Photochromism causes color changes in the material, which may be of a transparent state (UV absorption only) and a colored condition (visible absorption), or between 2 colored states. It is believed that photochromism occurs by the formation of optical absorption centers created by the disintegration of H_2O molecules incorporated into the film during its deposition or by organic molecules containing O_2 and H_2 adsorbed on its surface [2].

In this context, this work proposes an innovative way to correlate the photochromic and photocatalytic properties of films composed of undoped TiO_2 fibers or doped ones with H_2WO_4 or $Na_2WO_4{\cdot}2H_2O$.

2. Experimental methods

2.1. Electrospinning

The fibers were synthesized by *electrospinning* after the preparation of precursor solutions of TiO_2-P25 Evonik standard, TiO_2, TiO_2/WO_3 and $TiO_2/Na_2WO_4{\cdot}2H_2O$ [3]. 5 ml of each of the four solutions were transferred to a plastic syringe. The syringe was connected to a stainless steel hypodermic needle and attached to the high voltage source. The distance between the needle tip and the cylindrical collector (coated with an aluminum foil to collect the fibers) was 12 cm, the working voltage was 13.5 kV, the flow of the precursor solution was 1.8 mL/h. The fibers produced were collected every 30 min for 4 h. The heat treatment was carried out from 650 up to 800 °C in an electric oven (SANCHIS) using a heating rate of 1.4 °C/min for 1 h.

2.2. Spin-Coating

First, on an ultrasound bath, it was dispersed 0.8 mL of acetylacetone (Sigma-Aldrich) and 0.25 g of each of the synthesized fibers. Then, 8 mL of ethanol (Zeppelin), 0.1 mL of Triton X-100 (Synth) and 0.4 g of polyvinyl butyral (PVB, Sigma-Aldrich) were added to the mixture and magnetically stirred for 5 min. The film was obtained using 5 drops of either one of the said dispersions on glass blades (30 mm x 15 mm) coated with FTO (*Fluorine-Doped Tin Oxide, Xop Glass*) using a Spin Coater TC 100 at 800 rpm.

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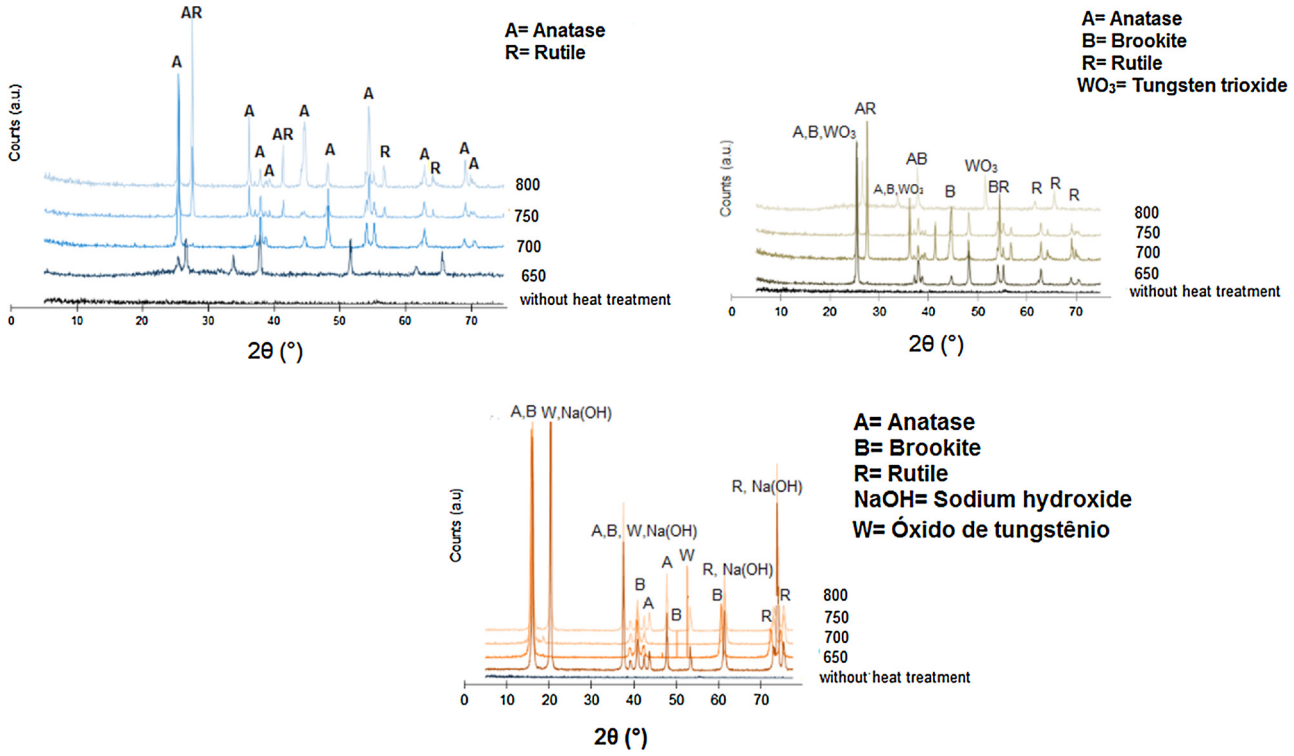

Fig. 1. XRD pattern of (a) TiO_2, (b) TiO_2/WO_3 and (c) $TiO_2/Na_2WO_4.2H_2O$ films.

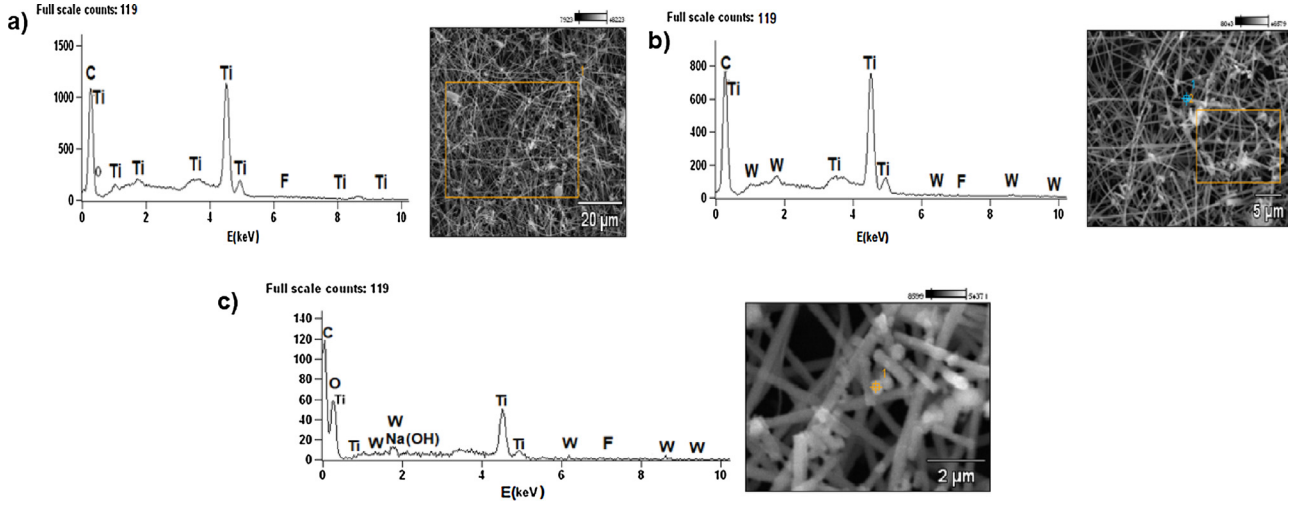

Fig. 2. Scanning electron microscopy (SEM) images of (a) TiO_2, (b) TiO_2/WO_3 and (c) $TiO_2/Na_2WO_4{\cdot}2H_2O$ film surfaces.

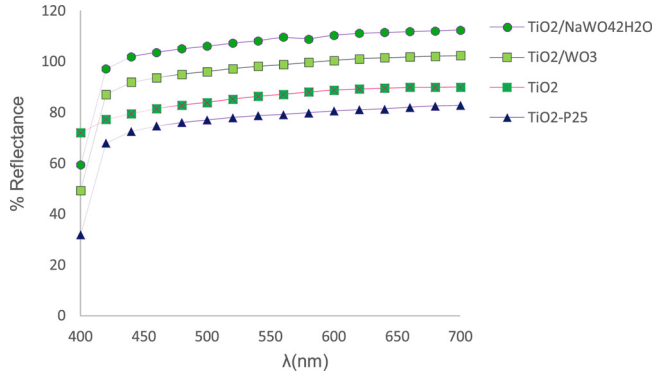

Fig. 3. Light reflected by synthesized films.

2.3. Characterization

A PHILIPS diffractometer with CuKα radiation, using a voltage of 40 kV and 40 mA, equipped with the X'PERT HightScore software, was used to identify the phases present in the fibers. A scanning electron microscope (SEM, JEOL JSM 6060) equipped with a characteristic X-ray detector (EDS) was used to evaluate the morphology of the fibers and to identify the presence of Na, W, Ti and O atoms in the films depending on the composition of the precursors. An UV–vis-NIR (Cary 5000) double-beam spectrophotometer was used to determine the band gap of the samples using the Kubelka-Munk correlation. The colorimetry was analyzed by a Konica-Minolta spectrophotometer, CM 2600 d, with an integrated sphere and an ultraviolet filter. The illuminant used was the D65, and the color measures simulated an observer at 10°. The photoactivity of the films was analyzed by a Cary Agilent 7000