



Photodiscoloration of textile reactive dyes on Ni/TiO₂ prepared by the impregnation method: Effect of calcination temperature

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ABSTRACT

In this work, we report the preparation of Ni/TiO₂ (2 and 5 wt%) catalysts by the impregnation method and calcinations temperature (250, 300 and 400 °C) for the discoloration of dyes reactive orange-122, reactive blue-19 and reactive yellow-145 under artificial and solar light. The characterization techniques used were: N₂ adsorption measurements (BET method), scanning electron microscopy (SEM) with energy dispersive X-ray (EDS), temperature programmed reduction (TPR), X-ray diffraction (XRD), thermogravimetric analyzer (TGA) and photoacoustic spectroscopy (PAS). The experimental results under artificial light indicate that the photocatalyst 5% Ni/TiO₂ calcined at 400 °C discolored dye reactive yellow-145 aqueous solutions 91.78% in 8 h, in contrast to 2% Ni/TiO₂ calcined at 400 °C with 41.82% to dye reactive blue-19 and 5% Ni/TiO₂ calcined at 250 °C to dye reactive orange-122 with 48.20%. The photocatalyst 5% Ni/TiO₂ calcined at 400 °C under solar irradiation in 60 min, is more efficient in the discolored dye reactive orange-122 aqueous solutions 92.16%.

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Introduction

The ever increasing industrial activity driven by the population growth raises concerns about the potential environmental impact of various activities.

Special attention must be paid to the continued contamination of natural water sources, which have historically received a great variety of wastes from homes and industries, such as from the textile industry [1–3].

Textile effluents typically present a high organic load of difficult degradation, mainly due to residual dyes from incomplete textile fiber dye fixation [1,4–6].

Reactive dyes have an electrophilic group that can make covalent bonds with hydroxyl groups of cellulose fibers, amino groups, hydroxyl, protein fiber thiols and polyamide amino groups. There are numerous reactive dyes; however, the main ones have the azo and anthraquinone functions as chromophore groups and chlorotriazinile and sulphato ethyl sulphonyl groups as reactive groups [7].

Heterogeneous photocatalysis, which belongs to Advanced Oxidation Processes (AOP), this emerging destructive technology ends up leading to the total mineralization of organic pollutants to CO₂ and H₂O [1,6,8–10] with the use of semiconductor catalysts. TiO₂ is the most studied mainly due to its high photochemical stability in a broad pH range, its low cost, and its nontoxicity [1,11–14].

The technique of metal ion-doped into TiO₂ has been widely studied, the transitional metal ions such as nickel, chromium, iron, vanadium, and zinc have been used for increasing the photocatalytic activity [13–15]. To this end, the aim of the present investigation is to compare TiO₂ the effects in the presence of metal on the catalyst surface (2 and 5 wt% Ni) and the calcination temperatures (250, 300 and 400 °C) for the discoloration of dyes reactive orange-122, reactive blue-19 and reactive yellow-145 by artificial and solar light.

Materials and methods

Catalysts preparation

In all experiments, chemicals of analytical grade and double quartz distilled water (resistivity ~18 MΩ cm) were used.

Ni/TiO₂ catalysts were prepared by the impregnation method according to the procedure described below.

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Synthesis of Ni/TiO₂

Ni/TiO₂ (2 and 5 wt%) was prepared which used TiO₂ (supplied by Vetec with over 98% purity) and NiCl₂·6H₂O (supplied by Synth with over 98% purity) with doubly distilled water solutions of the above metal precursor (Ni). The solution was stirred at room temperature in rotating evaporator for 17 h. The obtained solution was then dried in air at 120 °C for 21 h. The final catalysts were obtained after calcined in a muffle at 250, 300 and 400 °C for 5 h.

Characterization of the catalysts

N₂ adsorption measurements

N₂ adsorption measurements: specific surface area (*S*_o), average pore volume (*V*_p), and average pore diameter (*d*_p) of the catalysts obtained were determined in a Quantachrome Corporation model Nova 1200. The *S*_o values were determined by the Brunauer, Emmett, and Teller (BET) method by submitting the samples to previous activation at 150 °C in vacuum for 2 h.

Scanning electron microscopy (SEM) with energy dispersive X-ray (SEM/EDS)

Micrographs of the catalysts were obtained in a Scanning Electron Microscope Leo Stereoscan, model-440 equipped with an energy dispersive X-ray microsound (EDS). The samples were fixed to the surface of a double-face adhesive tape and coated with a gold layer. Acceleration tension was 30 kV.

Temperature programmed reduction (TPR)

The measurements were conducted in a home-built TPR apparatus and were carried out in a quartz tube packed with the material sample inside. The calcined samples were heated from room temperature to 1000 °C at heating rate of 10 °C/min in a 1.75% H₂/98.25% Ar gas flow at the rate of 30 mL/min. The thermocouple was located in the central position of the bed and the results were obtained from the hydrogen consumption, which is recorded by a thermal conductivity detector.

X-ray diffraction (XRD)

Diffraction patterns of the catalysts were obtained with a Shimadzu XRD 6000 apparatus. Copper emission line (Cu Kα, λ = 0.154 nm) with emission tube acceleration of 30 kV, current of 20 mA, and scanning velocity of 2° 2θ min⁻¹ was used as a radiation source.

Thermogravimetric analyzer (TGA)

The developed samples were analyzed in a SHIMADZU-Thermogravimetric Analyzer-Model TGA-50. Approximately 10 mg of calcined catalysts were submitted to a heating rate of 10 °C/min from room temperature to 1000 °C, while an inert gas, such as nitrogen, passed through the samples at 20 mL/min.

Photoacoustic spectroscopy (PAS)

The photoacoustic spectroscopy measurements in the UV–vis spectral regions were performed using a lab-made experimental set up. The monochromatic light was obtained from a 1000-W xenon arc lamp (Oriel Corporation 68820) and a monochromator, also from Oriel Instruments, model 77250. The light beam was modulated with a mechanical chopper, Stanford Research Systems SR540. A lab-made photoacoustic cell was designed to have a very low volume. It was made of aluminum block, machined to hold samples with maximum dimensions of about 5 mm in diameter and 1 mm in thickness, which allows light to enter through a high transparent quartz window of 6 mm diameter and 2 mm in thickness. The microphone chamber was 15 mm away and connected to the sample holder room by means of a 1-mm diameter duct.

The capacitive microphone which was used is a very sensitive 12-mm diameter Bruel & Kjaer model 2639 with a high gain of 50 mV/Pa and flat frequency response from 1 Hz to 10 kHz. The lock-in amplifier was from EG & G Instruments, model 5110. All the photoacoustic spectra were obtained at a modulation frequency of 20 Hz and recorded between 220 and 720 nm. Data were acquired by a personal computer and the PAS spectra were normalized with respect to the carbon black signal.

The band gap energies were determined by using Eq. (1):

$$\lambda = \frac{hc}{E_{gap}} = \frac{1240}{E_{gap}} \quad (1)$$

where *E*_{gap} is the band gap energy in units of eV, which was obtained by using the direct method, i.e., *m* = 2.

Photocatalytic tests

The reactive dyes orange-122, blue-19 and yellow-145 were purchased from Chemical Texpal Ltd., located in Valinhos (São Paulo, Brazil). Their structure and maxima absorption wavelength are shown in Table 1 [16–18]. Fig. 1 illustrates the reactor used in

Table 1
Molecular structure of reactive dyes.

Dye	λ _{max} (nm)	Chemical structure
C.I. Blue-19	590	
C.I. Orange-122	486	
C.I. Yellow-145	425	

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