

Methylene blue photodegradation employing hexagonal prism-shaped niobium oxide as heterogeneous catalyst: Effect of catalyst dosage, dye concentration, and radiation source

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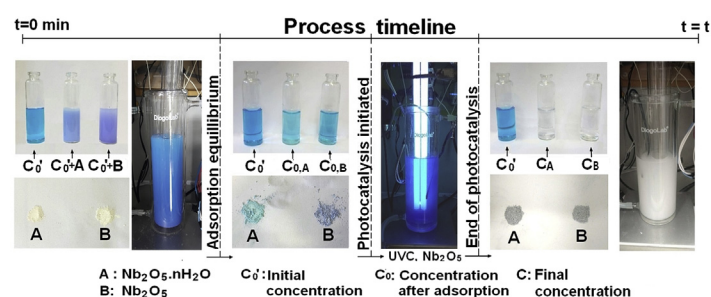
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HIGHLIGHTS

- Nb-based catalysts were prepared using Nb scrap and chips as precursors.
- The feasibility of using Nb-based catalysts for the degradation of MB was studied.
- Niobic acid and niobium oxide showed great performance in the degradation process.
- The highest degradation efficiency is reached using UVC radiation.
- The amount of acid sites, adsorbed H₂O and OH⁻ groups are important for the process.

GRAPHICAL ABSTRACT



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ABSTRACT

This paper evaluates the feasibility of using niobium-based catalysts for the photodegradation of organic dyes. The metal oxides were prepared using niobium scraps and chips as precursors. The semiconductors were characterized by diffuse reflectance spectroscopy, scanning electron microscopy (SEM), X-ray diffraction, infrared and Raman spectroscopy. The as-prepared anhydrous niobium oxide has a pseudohexagonal structure, whereas the hydrated niobium oxide is an amorphous material. The specific surface area of the hydrated niobium oxide is found to be double the area of its calcined counterpart. The photocatalytic efficiency of the materials was evaluated by methylene blue (MB) decomposition, measured by UV-visible spectroscopy. The effects of catalyst dosage and initial dye concentration were investigated in both the adsorption and photocatalysis processes. Increasing the initial MB concentration leads to increase the amount of adsorbed MB but to decrease the photocatalytic efficiency for all materials. In contrast, both the amount of adsorbed dye and the photocatalytic efficiency increase with increasing the catalyst dosage (0.5–2 g L⁻¹). The highest photodegradation efficiency is achieved using UVC radiation. The specific surface area as well as the amount of acid sites, adsorbed water, and OH⁻ surface groups on the catalyst surface demonstrates to be fundamental to the photocatalytic properties of the materials. Furthermore, the photocatalytic mechanism is controlled by superoxide and singlet oxygen species for the hydrated material, whereas the hydroxyl radical is the main active species in the

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photodegradation employing the anhydrous oxide. The hydrated material achieved a complete degradation of the methylene blue.

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1. Introduction

An estimated 700,000 tons of dyes are annually produced worldwide [1]. Slightly more than half of this production is used to manufacture textile dyes, and about 15% is used for dyeing other substrates such as leather and paper. Thus, the effluents that contain dyes represent a considerable source of environmental contamination especially since these substances have toxic and carcinogenic effects at high concentrations [2].

The most conventional methods for purifying wastewater from dyeing processes are based on biological oxidation and physico-chemical phenomena (coagulation, flocculation and adsorption in activated carbon). Nonetheless, these processes suffer from their limited efficiencies due to the high molecular stability and water solubility of the target compounds. Further, the major disadvantage of physicochemical methods is the non-destructive nature of treatment that merely transfers the contaminants from one phase to another [2], thereby generating a new and different type of pollution that essentially requires further treatment [3]. On the other hand, biological methods are not efficient in the treatment of effluents containing toxic and/or non-biodegradable organic substances [4]. In this context, advanced oxidation processes (AOP's) represent an attractive alternative for the treatment of a wide range of organic pollutants in effluents. Heterogeneous photocatalysis, an example of these processes, has earned significant attention in the scientific community due to its versatile application in environmental remediation, virus deactivation, and water splitting [6–8]. The hydroxyl radicals (OH^{\bullet}) formed in this process are highly reactive species that are able to oxidize most organic compounds in industrial effluents. Under optimum conditions, the organic compounds are completely oxidized to CO_2 and H_2O [1].

Nowadays, the most widely used photocatalyst in degradation of organic dyes is TiO_2 , given its strong oxidizing power, chemical stability, low cost, and non-toxicity [8]. However, the large-scale applications of TiO_2 are hampered by its low efficiency under solar radiation, high recombination rates, and the difficulty of its separation and recovery [9]. Therefore, the development of new photocatalysts and methods that enhance the photocatalytic efficiencies of the currently used materials has strongly attracted the interest of several researchers [5,10,11].

From this perspective, niobium oxide and niobic acid are considered to be good alternatives to the TiO_2 photocatalyst. These materials absorb radiation in the UV region (similar to TiO_2) and show stability and resistance to corrosion in a wide range of acidic and basic systems [12,13]. They also have high catalytic activities and useful photocatalytic properties [13–16]. In addition, these materials can be easily separated from an effluent, since their hydrocolloids are not stable in water [17–19].

The advantages of using low-cost syntheses in the production of niobium-based catalysts from niobium scraps are the reductions of waste volumes from mining and mineral processing activities, disposal costs, and environmental impacts [20]. Moreover, the use of scraps and chips for catalyst synthesis has emerged as a potential alternative to reduce the catalyst preparation costs and to add value to these residues by transforming them into attractive materials for prospective applications in wastewater treatment.

Thus, the feasibility of using niobium oxide synthesized by the

conversion of niobium scraps in the photodegradation of organic dyes was studied in this work. Additionally, a systematic investigation was performed to assess the effect of the catalyst dosage, dye concentration, and radiation source on the photocatalytic process.

2. Materials and methods

2.1. Synthesis of $Nb_2O_5 \cdot nH_2O$ and Nb_2O_5

Hydrated niobium (V) oxide ($Nb_2O_5 \cdot nH_2O$) was prepared by the dissolution of metallic niobium (3 g) in an acidic mixture of HNO_3 (8 mL, 65% w/w) and HF (10 mL, 40% w/w). After complete solubilization, 80 mL of an NH_4OH solution (1:3 v/v) was added under stirring until the precipitation of the hydrated metal oxide was completed. The material was then filtered, washed with deionized water to $pH = 7$, and dried in an oven at $100^\circ C$ until achieving a constant mass. Anhydrous niobium oxide (Nb_2O_5) was obtained by heat treatment of $Nb_2O_5 \cdot nH_2O$ at a temperature of $550^\circ C$ for 6 h.

2.2. Characterization

Crystalline structure and the presence of inorganic impurity were investigated by X-ray diffraction (XRD), using a PANalytical X'Pert PRO MPD 3060 diffractometer, equipped with an X'celerator detector, operating at 40 kV and 30 mA with $MoK\alpha$ radiation. XRD patterns were collected in the range of 2θ from 10 to 70° at step size of 0.026° .

Sample morphology was observed using a Scanning Electron Microscope equipped with a field emission gun (FEG-SEM), TESCAN model MIRA 3.

UV–vis diffused reflectance (DR) spectra were performed on a UV–vis spectrophotometer (Shimadzu UV-2600) with an integrating sphere attachment.

The infrared spectra were acquired by Fourier transform infrared spectroscopy using a universal attenuated total reflectance sensor (FTIR-UATR) (Perkin Elmer Spectrum, model Frontier). The FTIR spectrum was an average of 16 scans at a speed of 2 s per scan at a range of 450 – 4000 cm^{-1} . The resolution of the spectrometer was set to 4 cm^{-1} .

Raman spectra of the materials were collected with a LabRAM HR Evolution model Raman Spectrophotometer (Horiba) equipped with a camera and using an Ar laser (532 nm). The spectra were taken between 50 and 2000 cm^{-1} with acquisition time of 30 s, 2 cycles and a slit size of $100\text{ }\mu\text{m}$.

Nitrogen adsorption isotherm at 77 K was determined in an Autosorb-1MP gas analyzer (Quantachrome Instruments). The BET surface area was determined using the data from the isotherm by the Brunauer, Emmett, and Teller (BET) equation. The samples were outgassed overnight at $100^\circ C$ prior to adsorption measurements.

2.3. Evaluation of the catalytic activity in the photodegradation of methylene blue

The tests for the determination of the photocatalytic activity were performed in a jacketed reactor with a height of 30 cm high and an internal diameter 10 cm. A PL-L lamp was placed inside a quartz tube (4 cm in diameter and 40 cm in length) with a closed

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