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Sonophotocatalytic degradation of Orange II dye using low cost photocatalyst

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ABSTRACT

The photocatalytic and sonophotocatalytic degradation of Orange II was investigated using a synthesized catalyst with low cost materials. Iron (III) oxide-titanium (IV) oxide (1% Fe) photocatalyst was synthesized by sol-gel method and pure titanium dioxide was used as photocatalytic reference material. Their crystallographic structures were determined by X-ray diffraction. The surface area and pore diameter was studied by Brunauer–Emmett–Teller method (N_2 adsorption/desorption). The samples were characterized by Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Analysis (EDX). SEM images show the formation of particles with spherical geometry. Synergistic effects of ultrasound and photocatalysis were demonstrated. Three different ultrasonic frequencies were tested 250 kHz, 500 kHz and 1000 kHz. The best catalytic activity (higher Orange II degradation efficiency) was obtained at 500 kHz. Test results show that it is possible the total decolourisation of the Orange II dye in aqueous media by the sonophotocatalytic process using visible light, without producing hazardous by-products. The process can be considered as a potential environmentally friendly technique for organic synthetic dyes degradation.

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1. Introduction

From food and clothing to cosmetics and drugs, color is a chief aspect of industrialized products (Pereira and Alves, 2012). Such demand has a serious environmental cost, because industrial production and use of colorants requires large amounts of water that is polluted throughout the process (Kant, 2012). Such industrial effluents have long become a serious environmental issue (Ratana and Padhi, 2012.). Due to their solubility, organic synthetic dyes are among the major contributors to water pollution (Zaharia and Suteu, 2012). About 50% of the world production of dyes is azo dyes (Heng et al., 2014), such as Orange II. Industrial wastewaters containing dyes have a high environmental footprint because they are chemically stable, non-biodegradable and potentially carcinogenic (Zaharia and Suteu, 2012). Dyes-containing effluents also carry other harmful chemicals, which form a poisonous mixture that is not chemically inert (Kant, 2012). Therefore direct discharge is undesirable because such effluents are toxic, carcinogenic or mutagenic (Chong et al., 2010). Even if a treatment process before discharge existed, conventional water treatments are not designed to remove synthetic dyes and their degradation by-products (Chuan et al., 2012). Dyes also affect photosynthesis by decreasing light penetration, reducing the amount of dissolved oxygen by blocking the oxygen interchange at the surface and increasing the biochemical oxygen demand (Kant, 2012). Moreover, due to their chemical stability azo dyes cannot be removed efficiently by classical wastewater treatments (Heng et al., 2014).

Consequently, there is an urgent need to develop effective water treatments that reduce water pollution and increase recycling and reuse. In order to remove dyes from industrial wastewaters, several advanced oxidation processes (AOPs) have been tried (Chong et al., 2010). Among the various AOPs, ultrasonic assisted treatments have

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proved effectiveness to remove organic pollutants (Arslan-Alaton, 2003). Pollutants such as textile dyes, aromatic and phenolic compounds, cholorobenzene, bisphenol-A and carboxylic acid can be removed from wastewaters by sonochemical oxidation (Dükkancı et al., 2014). During the ultrasonic irradiation cavitation bubbles are formed and they collapse producing high temperatures and pressures. Under these conditions, hydroxyl, hydrogen and organic radicals are generated, as shown in equation (1) (Arslan-Alaton, 2003).

$$H_2O\,(g) + R \rightarrow H^{\bullet}(g) + {}^{\bullet}OH + R^{\bullet}\,(g) \quad (1)$$

Some gaseous organic compounds are destroyed by pyrolisis within the bubbles. In addition, hydroxyl radicals produce hydrogen peroxide (Ozdemir et al., 2011). Although ultrasound has been used for decolourisation and degradation of textile dyes, it has been difficult to completely mineralize the dye (Siddique et al., 2014). In recent years several studies have investigated the modification of TiO_2 photocatalyst by adding transition metal impurities. In the case of Ti(IV)/Fe(III) mixed oxide, studies have shown significant photoactivity improvement compared to TiO_2 (Sánchez et al., 2007). It has been shown that there is a photoelectron transfer from TiO_2 to Fe_2O_3, which promotes oxygen reduction (Moniz et al., 2014). Iron-doped TiO_2 prepared by impregnation and coprecipitation methods, has shown significant photocatalytic activity using visible light. One of the problems of photocatalysts is that their efficiency and photocatalytic degradation rates are reduced due to deposits on its surface that block active sites (Adewuyi, 2005). By adding ultrasound to the photocatalytic process, mass transfer rate is improved and the blocked sites on the photocatalyst are released. Another favourable effect is the increment of surface area of the catalyst due to breakup of its particles which increases the number of active sites (Zhong et al., 2011). Therefore, the combination of ultrasound and photocatalysis has a synergistic effect (Ma et al., 2006). For the particular case of Orange II, ultrasonic degradation has been studied (Dükkancı et al., 2012) and it has also been combined with heterogeneous Fenton processes (Zhang et al., 2009). In this work, sonophotocatalysis is applied to degrade Orange II using Fe_2O_3-TiO_2. Following such ideas, in this work Orange II degradation is studied by combining a photocatalytic process with an ultrasonic treatment in a range of frequencies that has not been previously explored in order to analyze the influence of the ultrasonic frequency in the process. Initially, only photocatalytic tests were carried out using TiO_2 and Fe_2O_3-TiO_2, applying visible and UV light. Next, ultrasound was irradiated in order to enhance the performance of the processes that used Fe_2O_3-TiO_2 as catalyst. Since Fe_2O_3-TiO_2 is activated by visible light, it was preferred to implement the sonophotocatalytic process. Visible light activation results in a more practical implementation of the process and also makes it environmentally friendly. Ultrasonic frequency was varied as 250 kHz, 500 kHz and 1000 kHz, in search of a better outcome. Although successful treatment processes are reported in the literature, toxicological studies of treated water are seldom performed. In our work, toxicity tests are performed to verify that the degradation does not generate harmful by-products. The Fe_2O_3-TiO_2 samples were characterized by BET measurement, SEM and diffuse-reflectance UV-vis spectroscopy.

2. Experimental procedure

2.1. Synthesis and characterization

TiO_2 was synthesized by the sol-gel method (May-Lozano et al., 2016), in a three-necked flask; butanol (Aldrich) was mixed with titanium isopropoxide (Aldrich) at room temperature, and then water was poured dropwise into the solution. The H_2O/alkoxide molar ratio used was 16, the solution was kept under stirring for 2 h. The samples were dried at 120 °C in oven for 2 d and finally calcined in a muffle at 450 °C for 3 h. Synthesis of Fe_2O_3-TiO_2 (1% Fe) photocatalyst used was performed by the impregnation method (Ganesh et al., 2012). Fe_2O_3 nanoparticles (Aldrich) were dissolved in 2 mL of 2-propanol. Subsequently, in an agate mortar, 1 g of TiO_2 together with the dissolution of Fe_2O_3 were placed and triturated for 1 h. The powders obtained were dried at 90 °C for 24 h and calcined at 400 °C for 2 h. The surface areas were calculated by BET method and the pore volume was determined by the Barrett–Joiner–Halenda (BJH) method. The study was obtained on a Micromeritics ASAP 2020 instrument at a temperature below −198 °C. Prior to the adsorption experiments, samples were treated at 180 °C for 16 h. The optical properties of the samples were analyzed by UV-vis diffuse reflectance spectroscopy (DRS). The band gap was obtained from the diffuse UV-vis data for the samples using the Kubelka-Munk function by plotting the transformed equation $(F(R)^{*}h\upsilon)^{1/2}$ versus the energy of light and extrapolating the linear region of the transformed spectra to zero. The particles range size and morphology of the samples were studied using a scanning electron microscopy (SEM, ZEISS, SUPRA 55VP).

2.2. Treatment processes

The photocatalytic and sonophotocatalytic treatments were carried out in a stirred batch reactor with operation volume of 100 mL, at 20 °C, with an initial concentration of 10(mg)(L^{-1}) of Orange II dye and 0.03 g of photocatalyst. Orange II decolourisation was monitored by UV-Vis spectra at 483 nm. Fig. 1, shows the Orange II structure and one example of spectra obtained for the Orange II during the decolourisation; in this case, with sonophotocatalytic treatment at different treatment times.

Fig. 2, shows the diagram of the entire system. A simple cooling system was used to keep the reactor temperature at 20 °C. The pH was monitored throughout the process, showing no change from its initial value (7.0). Initial tests were conducted without catalyst and applying only light, but the dye was not degraded.

Then, the reactor was irradiated using an UV LED lamp (5 W, UltraViolet 100 LED Torch) with a central wavelength of 395 nm. On the other hand, photocatalysis with visible light was tried using an LED floodlight (IP65, 4500 Lm) of 50 W. The gap between the lamp and the liquid surface was 3.0 cm. The temperature was maintained at 20 °C by the recirculation of cool water. Moreover, in some tests 0.025 mL of H_2O_2 as oxidizing reagent was added to evaluate its effect on the photocatalytic Orange II degradation. The same light conditions of the photocatalytic reaction were used in the sonophotocatalytic degradation. The sonophotocatalytic treatments were carried out applying ultrasonic irradiation at 250, 500 and 1000 kHz for 1 h. For each case, the power density was 35 WL^{-1}.

2.3. Toxicological studies

Respirometry technique was used to evaluate the toxicity of the by-products, which could be formed during the sonophotocatalytic process (BM-EvoRespirometer). Activated sludge from an aerated bioreactor of an actual wastewater treatment plant was used for this test. A bioreactor vessel (1 L capacity) was used to evaluate the actual dynamic respiration rate (Rs) and the oxygen consumption (C.O). Three different tests were performed: 1) only sodium acetate as a soluble readily biodegradable substrate, 2) the biodegradable substrate with the aqueous solution of Orange II before the decolourisation and 3) the biodegradable substrate with the aqueous

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