

Sonophotocatalytic treatment of Naphthol Blue Black dye and real textile wastewater using synthesized Fe doped TiO₂



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ABSTRACT

This study examines the treatment of Naphthol-Blue-Black (NBB) dye and real textile wastewater through sonophotocatalytic technique. Fe, TiO₂ and Fe-TiO₂ were synthesized and characterized by SEM, XRD and DRS which revealed its high purity, good doping, nanosize and higher light absorption capacity. The effect of pH, gas content (Ar, O₂, air and N₂), H₂O₂ concentration and catalyst loading were examined. The maximum color removal was observed for initial pH 11, argon gas bubble, 44.1 mmol/L H₂O₂, 3 g/L Fe, 4 g/L TiO₂ and 2.2 g/L Fe-TiO₂. However, 96% was achieved by sonophotocatalytic process for 2.2 g/L 1:7 Fe:TiO₂. Similar methodology for real textile wastewater with Fe-TiO₂ (1:7) could remove 91% of TOC.

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1. Introduction

Advanced oxidation processes (AOPs) have been used as emerging wastewater treatment technologies for various hazardous organic pollutants. Sonophotocatalysis is gaining attention among advanced oxidation process by which the electron hole pairs are generated at high temperature and pressure by the integration of sonolysis, photolysis and semiconductor catalysts resulting in higher hydroxyl radical production [1–4]. Among many semiconductor catalysts, TiO₂ has been identified to possess high degradation efficiency and self-regeneration and therefore, TiO₂ has been used widely as a catalyst in various processes like water purification and also in many applications [5–7]. TiO₂ supported catalysts are found to be more effective as they are inert both chemically and biologically and are photo-stable with high band energy ($E_{bg} = 3.0\text{--}3.2\text{ eV}$) owing to its activity only in the ultraviolet region. The catalytic activity of TiO₂ can be enhanced by tailoring it with some noble metals or metal oxides. The advantages of such modified catalyst mainly depends on the inhibition of the recombination of generated radicals, induction of photoreactions even in solar visible region and thus the metal doped catalyst can be used for any type of degradation studies [8–12]. The degradation capability of doped TiO₂ is based on the catalytic activity which

occur while both sono and photo reactions taking place: (i) Sonolysis of reactants in the presence of catalyst produces more •H and •OH radicals (ii) Photolysis reaction in which the photo irradiation of the catalyst (absorption of photons by the catalyst tends to produce electron hole pairs where the loosely bound electrons at valence band and conduction band gets excited by charge transfer among the available electrons) [13,14].

The degradation of organic pollutants by sonophotocatalysis takes place at the aqueous phase where lot of electrons and holes are available for excitation by the process of cavitation and photooxidation. The catalytic activity is limited by the adsorption of the organic compounds (the complex and/or recalcitrant azo dyes with nitrogen double bond) on the catalytic surface which are difficult to break down into simpler compounds. Other technologies used for dye degradation accomplishes the transfer of these compounds from one form to another form for instance, from azo dyes to amines which are again toxic and carcinogenic to the environment. Most of the primary methods like electrocoagulation, chlorination, ozonation and adsorption methods help in only color removal as they require advanced treatment methods to break this non-destructive dye for achieving complete removal of toxicity from these compounds.

This triggers to utilize the synergistic efficiency of the sonophotocatalytic process by making use of all the radicals, electron hole pairs which are formed during the process. The rapid generation of more radicals can be enabled by introducing a new metal ion to TiO₂. The mechanism of this sonophotocatalysis effect

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consists of the following process (a) activation of the photocatalyst surface (b) enhancement of the mass transport of organic compounds and (c) breakage of aggregation [15–17]. The degradation of azo dye in an aqueous solution using doped catalyst portrays a new type of hybrid technology for environmental remediation [18]. The mineralization and recovery of catalysts employed are more desirable than mere decolorisation in view of environmental safety. The various processes individually have failed to remove the total organic carbon (TOC) [19]. Researchers have found that the combined effect of sonophotocatalysis assisted with green synthesized doped catalyst has been successful in removing the TOC much better than the other process.

In this study, synthesized TiO_2 , green synthesized Fe and Fe doped TiO_2 have been used as catalysts for the sonophotocatalytic degradation of the Naphthol Blue Black (NBB) dye. Initially, a series of experiments were carried out by varying initial solution pH, gas bubbling (argon, nitrogen, oxygen and air) and H_2O_2 concentration to understand the mechanism of degradation of the NBB dye using sonophotolytic technique. Then, the treatment efficiency of the NBB dye with synthesized catalyst such as Fe, TiO_2 and Fe doped TiO_2 with varying ratios was studied. The catalysts were characterized by SEM, XRD and DRS to understand the morphology, purity and light absorption capacity. Finally, based on the results of NBB dye treatment, the sonophotocatalytic technique was applied to real textile wastewater and was reported in terms of total organic carbon variation.

2. Materials and methods

2.1. Materials

Naphthol Blue Black (Sigma–Aldrich), concentrated H_2SO_4 (Merck), NaOH (Merck), hydrogen peroxide (30%, Merck), titanium *tetra-iso-propoxide* (Merck), ethanol (Merck), $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (Merck), argon gas (99.99%), oxygen gas (99.99%), nitrogen gas (99.99%) (Priyam gas agency, Trichy, India), air (compressed air) and green tea leaves (purchased from local market) were used for this study. Double distilled water was used for preparing all the solutions.

2.2. Experimental setup

The experimental setup for sonophotocatalysis consists of Phillips UV-C (Philips, 256 nm) lamp of 4W placed over the reactor of 500 mL volume made of borosilicate glass. The reactor was placed in the ultrasound bath (Dakshin, Mumbai) filled with double distilled water which operates at 37 kHz frequency with an

operating power of 200 W and has the provision for water circulation. The water level in the ultrasound bath, the reactor position and the reactor immersion height in the ultrasound bath were maintained same for all the studies. All the experiments were carried out in the setup as schematically represented in Fig 1.

2.3. Methods

A stock solution of 1000 mg/L concentration of the NBB dye solution was prepared using 0.1 g of NBB dissolved in 100 mL distilled water. The mixture was then stirred for 10 min with the help of magnetic stirrer and stored in dark for further solution preparation of required concentration. The parametric studies were carried out with initial pH, cavitation bubble content and hydrogen peroxide concentration variations. In order to study the effect of initial pH on sonophotolysis of the NBB dye solution, the pH of the dye solution was adjusted either using 0.1N concentrated H_2SO_4 or 0.1N NaOH and was monitored with full featured multiparameter instrument (YSI Inc., Professional Plus, USA). The gas content in the cavitation bubble was varied by purging the required gas such as nitrogen, oxygen, argon and air at a flow rate of 1 LPM for 5 min for the saturation of the gas in the NBB dye solution just prior to sonophotolysis. In the case of H_2O_2 addition study, the H_2O_2 of required quantity was added to the NBB dye solution just before the start of the sonophotolysis experiment in order to avoid any pre-oxidation reaction. All the color removal studies were carried out for 1 hour unless otherwise mentioned and 400 mL of the reaction solution was used. The variation in dye color change at different time intervals (15, 30, 45 and 60 min) was monitored by taking intermittent dye solution and measuring its absorbance value (at 618 nm the λ_{max} for NBB dye) in a UV–vis spectrophotometer (Merck Spectroquant Pharo 100). The percent dye color removal was calculated based on the absorbance value of the dye using Eq. (1).

$$\% \text{ Colour removal} = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

where, C_i —initial concentration and C_f —final concentration after treatment.

2.4. Synthesis of catalysts

For the synthesis of Fe [20], 2 g of green tea leaves was taken and brewed it for 10 min. in 100 mL of Millipore water at 80 °C. The solution was then cooled down and filtered through Whatman filter paper no.1. Then, 13.9 g of FeSO_4 was added to the filtered

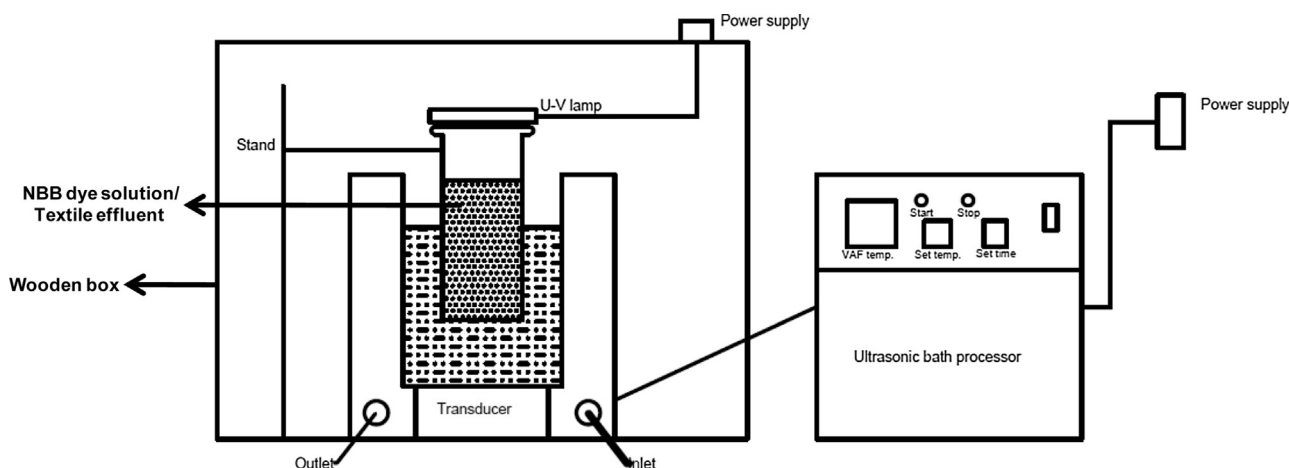


Fig. 1. Schematic of sonophotolytic experimental setup.

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