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Synthesis of ultrasound assisted nanostructured photocatalyst (NiO supported over CeO₂) and its application for photocatalytic as well as sonocatalytic dye degradation

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ABSTRACT

The present work focuses on the improved synthesis of bimetallic catalyst, NiO impregnated CeO₂, based on the use of ultrasound assisted approach with comparison with the conventional approach. Morphological characteristics of the catalyst have been established using TEM and BET analysis. In addition XRD and FTIR analysis was also performed to establish the crystallite size and the active functional groups that decide the catalytic activity respectively. It has been established that the catalyst synthesized in the presence of ultrasound showed better catalytic properties than the catalyst obtained using the conventional approach with the specific surface area increasing from 31.41 m²/g to 94.63 m²/g due to cavitation effects. TEM analysis also established the appearance of sharp edged and non-agglomerated nanoparticles in the ultrasound assisted synthesis whereas in the case of conventional approach, agglomerates and higher particle/crystallite size was observed. The catalyst was subsequently used for the catalytic degradation of Brilliant Green dye using UV irradiation and ultrasonic irradiation. The catalytic activity of the catalyst obtained using ultrasound assisted approach was observed to be better than the catalyst obtained using conventional approach. UV induced extent of degradation using the catalyst synthesized in the presence of ultrasound was found to be 82% whereas using conventionally synthesized catalyst, much lower extent of degradation as 40% was obtained. TOC analysis was also performed which confirmed correspondence with the extent of degradation and better results for the ultrasonically obtained catalyst. Overall the ultrasound assisted approach has been demonstrated to be an effective approach for obtaining better quality NiO impregnated CeO₂ catalyst with proven efficacy for decolourization using photocatalysis (higher activity) as well as sonocatalysis.

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1. Introduction

Textile and paper industries are the largest water consuming and water polluting industry as large amount of dyes are used and possibly discharged into effluent streams, if not treated satisfactorily. Conventional methods of treatment such as chemical precipitation/adsorption and biological treatment cannot effectively remove or degrade dyes from effluents and hence developing an effective technique for degradation or complete mineralization is necessary. Now a days, photocatalytic degradation as well as ultrasound induced catalytic degradation of pollutants is becoming a promising and effective approach. Photocatalysis is described as a photo-induced reaction which is accelerated by the presence

of a catalyst. These types of reactions are activated by absorption of a photon with sufficient energy which equals or is higher than the band-gap energy (E_g) of the catalyst [1]. Absorption of photons produces electron-hole pairs on the catalyst surface which in turn can reduce or oxidize the organic materials present in aqueous solutions. The electron-hole pair leads to the formation of hydroxyl radicals (•OH), superoxide radical anions (O₂•⁻), and hydroperoxyl radicals (•OOH), which can effectively drive the degradation of variety of pollutants present in the wastewater. The efficiency of the photocatalysis operation for pollutant degradation also depends on the presence of the oxygen molecules, which either scavenge the conduction band electrons or prevent the recombination of electron-hole pair. The electron in the conduction band can be picked up by the adsorbed pollutant molecules, leading to the formation of radical anions which initiates chain reactions leading to the degradation [2]. The main advantage of applying such a technique is its potential to convert the organic pollutants into nontoxic

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species (CO_2 , H_2O) without any requirement of further separation processes and hence this approach can also be effectively applied for dye degradation. Cavitation is an upcoming technique which has also been applied for the degradation of many pollutants including dye [3]. Cavitation can be described as the formation, growth and collapse of micro-bubbles in liquid leading to localized supercritical conditions i.e. high temperature and pressure pulse (about 1000 atm pressure and about 5000 K temperature). During cavitation bubble collapse, H_2O undergoes thermal dissociation within the vapor phase generating hydroxyl radicals and other oxidizing species depending on the constituents of water. These conditions are favourable for oxidative degradation of pollutants such as pharmaceutical intermediates and dyes [4,5].

TiO_2 [6,7], ZnO [8] and more recently, NiO [9,10] and Ag_2CrO_4 [11], have been considered as efficient catalysts for application in water treatment due to their higher photocatalytic activity and excellent stability during the operation. NiO is a p-type semiconductor with a wide band gap between valence and conduction band, making it specifically suitable as a photocatalyst (the band-gap energy ≈ 3.62 eV). However nickel suffers drawbacks such as possible aggregation which significantly reduces the surface area and leads to the closure of catalytically active sites [12]. To overcome the aggregation problem, the catalyst is usually supported over commonly used supports such as carbon, zirconia, ceria, alumina, titania, etc. [13–16]. CeO_2 is reported as an effective support for Ni and widely used due to its strong metal support interaction nature with Ni [17]. Use of CeO_2 as support material is seen to improve the dispersion and increase the active phase and performance of the catalyst. Ultrasound assisted synthesis can also give catalyst with better morphology and activity [18–22]. With a simple modification in reaction conditions, various forms of nanostructured materials can be synthesized, including metals, alloys, oxides, sulfides, carbides, and nanostructured supported catalysts. Initial application of ultrasound to the mixture can help to achieve homogeneous nucleation with subsequent application leading to enhanced growth of the crystals. Acoustic cavitation near solid–liquid interface (heterogeneous system) is asymmetric and associated with generation of the high-speed micro jets which impact the solid surface and create cleaning and deagglomeration effects. The potential energy of the collapsed bubble is converted into kinetic energy of the micro jet with velocities around few hundreds of meters per second. Due to these high intensity jets with turbulence, the application of low frequency ultrasonic irradiation can yield nanoparticles whose sizes are much lower than the size obtained using conventional approach [18–22]. The possibility of the radical formation due to the ultrasound also assists the process of formation of nanomaterials [20] especially for processes based on reactions. The use of ultrasound during the preparation of metal oxides has several advantages such as the possibility of obtaining well-crystallized material without using high temperatures or obtaining controlled nanoparticle morphologies. The objective of the present work was to investigate the efficacy of ultrasound for obtaining NiO supported over nanosized CeO_2 catalyst with possible improvements in the morphological properties and catalytic activity.

Brilliant Green dye used in the investigation as a model pollutant is triphenyl nitrogen containing cationic dye. The exact structure of the brilliant green dye has been given in Fig. 1. Brilliant green is chemically known as ammonium, (4-(4-(diethylamino)- α -phenylbenzylidene)-2,5-cyclohexadien-1-ylidene)diethylammonium sulfate ($\text{C}_{27}\text{H}_{34}\text{N}_2\text{O}_4\text{S}$) with λ_{max} of 625 nm and molecular weight of 482.6 g/mol. This dye is listed as a priority pollutant due to its carcinogenic nature [23]. It is used to color synthetic fibers and silk biological stain, in paper printing applications, as a dermatological agent, veterinary medicine, and as an additive to poultry feed

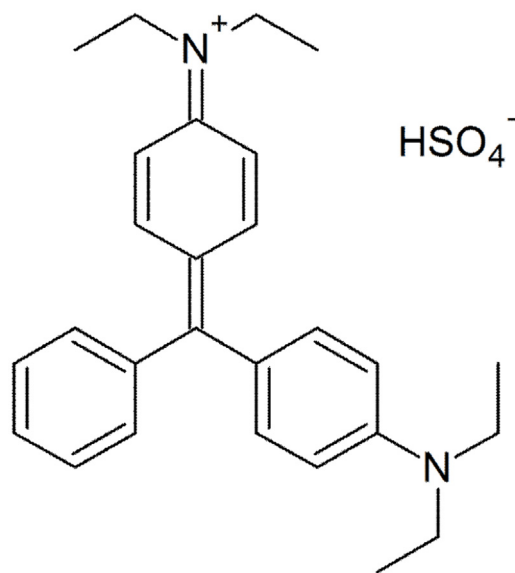


Fig. 1. Structure of Brilliant Green dye.

to inhibit propagation of intestinal parasites and fungus. Being highly toxic for humans and animals, it is imperative to find novel treatment approaches for efficient degradation. There have been some earlier reports of application of cavitation reactors for the treatment of BG such as combination of cavitation and oxidizing agents (H_2O_2 , $\text{Na}_2\text{S}_2\text{O}_8$ and NaOCl) [23], sonolysis alone [24], sonocatalysis involving polyacrylic acid hydrogel [25], sonophotocatalytic oxidation [26] and photocatalytic oxidation [27]. BG dye degradation studies have also been reported using different doped catalysts and using hybrid methods [28,29]. Present research work deals with application of NiO impregnated over CeO_2 in photocatalytic and sonocatalytic degradation of BG dye for the first time with an objective of comparing the efficacy of two types of catalysts obtained using the ultrasound assisted approach and the conventional approach as well as the two modes of irradiation in the actual degradation.

2. Materials and methods

2.1. Materials

Cerium nitrate hexahydrate ($\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$), nickel nitrate hexahydrate ($\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$), sodium hydroxide (NaOH), and PEG-8000 (polyethylene glycol) were obtained from Hi-media, Mumbai. Dry powder of Brilliant green dye was obtained from Dyestuff department at Institute of Chemical Technology, Mumbai.

2.2. Equipment

Ultrasonic probe sonicator with diameter of 13 mm obtained from Dakshin, Mumbai was used for catalyst synthesis as well as sonocatalytic degradation. Two UV tube (Philips TUV 8 W/G8T5), having power rating of 8 W and dominant emitting wavelength of 254 nm were used as a source of ultraviolet irradiation for the photocatalytic degradation. Quartz glass reactor was used for studies involving UV irradiated dye degradation and was equipped with magnetic stirring. During sonocatalytic dye degradation using ultrasonic probe, glass reactor is used along with magnetic stirring. The temperature was always controlled around 30°C using cold water bath.

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