



Influence of jute template on the surface, optical and photocatalytic properties of sol-gel derived mesoporous zinc oxide

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ABSTRACT

Utility of bio-templates in the synthesis of crystalline solids is one of the preferred routes in green synthesis. The present study explores jute fiber as a biotemplate for the sol-gel synthesis of zinc oxide. AT-IR of the synthesized sample clearly revealed the chelation between the metal and active groups of jute. The surface area enhancement and reduction in crystallite size in the templated samples were confirmed from BET and XRD analyses. ZnO prepared using 0.5 g/3 g of precursor possessed higher average crystallite size than the other samples. Surprisingly, the same sample also had a high surface area due to its distinct spherical morphology, highlighting the influence of morphology on the surface area of ZnO. Jute templated ZnO exhibited significant photocatalytic activity for degradation of crystal violet dye under visible light. Thus, jute has been proved to be an efficient template for the sol-gel synthesis of ZnO with controlled crystallite characteristics.

1. Introduction

Uncontrolled pollution of air, water and other natural resources is a potential threat to the well being of life on earth. Among the various aquatic pollutants, industrial effluents contribute significantly to the dwindling of quality and quantity of potable water [1,2]. Metal oxide semiconductor mediated photocatalysis is one of the efficient in-house water treatment processes used to tackle water contamination. In this work, crystal violet, a cationic triphenyl methane dye, has been chosen as a model pollutant. Crystal violet finds potential use as a colourant in applications such as, textile dyeing, ballpoint pen, paper, leather, additives, foodstuffs, cosmetics and in laboratory based cell viability assays [3]. Its presence in the industrial effluents is both aesthetically unacceptable as well as a health hazard as the dye has been proven to cause adverse mutagenic effects in living tissues, in turn, leading to cancer. Therefore, it is important to analyse the abatement of this pollutant through photocatalytic degradation.

A variety of semiconductor oxide and sulphide materials have been explored for the removal of organic pollutants from aqueous medium, among which TiO₂ and ZnO have been extensively studied [4,5]. The efficiency of ZnO as a photocatalyst for the degradation of various pollutants such as acid Black 1 [6], acid violet 7 [7], reactive orange 4 [8] and bromothymol blue [9] has been widely studied. ZnO with the wide band gap of 3.37 eV and large exciton binding energy of 60 MeV attests to be superior to commercial Degussa P25 in certain cases, as ZnO has high quantum efficiency and absorption in solar region [10–

13]. In general, the surface properties and the morphology of the semiconductor have significant influence on its photocatalytic activity. The optical and surface characteristics of ZnO have been reported to be enhanced through coupling ZnO with other semiconductor oxides and sulphides [14,15], doping with metal/non-metal [16,17], co-doping [18] and template-assisted synthesis [31]. Among the different methods adopted, template assisted synthesis has been one of the effective routes to obtain mesoporous ZnO of desired morphology and surface area. Many of the conventionally employed physical and chemical templates are inefficient and involve complex synthetic processes [19]. Bio-inspired synthesis of ZnO has garnered special focus in recent times, as it often provides cost-effective and environmentally benign alternative to the contemporary chemical routes and facilitates the formation of customized, morphology-controlled structures [20–22]. To date, biotemplates such as rice powder, wood, egg shell albumen, butterfly wings, apple, silk, DNA and mango core inner shell have been used for the synthesis of ZnO to obtain surface controlled materials [23–30]. Apple-pectin mediated synthesis of double caged pea-nut shaped ZnO has been synthesized by hydrothermal method with improved photocatalytic efficiency in the UV region [31]. Dong et al. [32] achieved highly porous hierarchical ZnO films with the surface area of 9 m²/g by using egg shell membrane as template. Using rice as biotemplate, Donya Ramimoghadam et al. [23] synthesized ZnO with the enhanced surface area of up to 22 m²/g, and in addition they reported the lignocellulosic biowaste [33], rice straw as the template for the synthesis of TiO₂ with enhanced photocatalytic efficiency. Wang

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et al. [34] reported the synthesis of mesoporous ZnO with improved photocatalytic efficiency in the visible region using modified lignin as the template. Reported studies on the use of biotemplates for mesoporous ZnO synthesis have been limited. In this context, the present work aimed to synthesize ZnO nanoparticles using jute, a natural fiber, as a template for the first time. The major components of the jute fiber are cellulose, hemicellulose and lignin [35]. Jute fiber has a number of surface active groups that are capable of chelating with metal ions [36]. In the present study, it was expected that such chelation between jute and Zn metal of ZnO precursor would serve as the nucleation site for the formation of ZnO, simultaneously controlling the agglomeration. The effect of jute template on the morphology, surface area and photocatalytic efficiency of synthesized ZnO was explored in this study and the results are presented below.

2. Experimental details

Jute fiber was supplied by National Jute Board (NJB), Chennai. Crystal violet dye (CI-42555) from Sigma–Aldrich, analytical grade Zinc acetate dihydrate, oxalic acid, Sodium hydroxide, Sulphuric acid and ethanol from Fischer Scientific and Zinc Oxide from Merck were used as received without any further purification process.

3 g of zinc acetate was dissolved in 90 ml of ethanol, to which finely chopped jute fibers (in four different ratios) were added and stirred for half an hour. 3 g of oxalic acid dissolved in ethanol was added drop by drop to the above mixture with constant stirring for 3 h. The resultant colloidal solution was left undisturbed for 24 h for gelation. The gel obtained was washed, dried in oven at 100 °C and then calcined at 500 °C for 5 h. Control ZnO was prepared through the same procedure without adding jute template. The samples were labeled Z, ZJ1, ZJ2, ZJ3 and ZJ4 denoting the weight of jute to be 0, 0.25, 0.5, 1 and 1.5 g respectively. [37].

2.1. Characterizations

Thermal stability and degradation temperature of the as-synthesized samples were analyzed using a SDT Q 600 US analyzer (ASTM E1131). The attenuated transformed infrared spectroscopy (Perkin Elmer) was employed to study the interaction between the template and the metal oxide. The physical state, crystallite size and the purity of the catalyst were studied using a Bruker AXS D8 Advance X-ray diffractometer. The morphology and elemental composition of the synthesized catalysts were observed by Scanning Electron Microscope (Hitachi S-3400 N) equipped with energy dispersive X-ray microanalysis (EDX) (HORIBA-EMAX EDAX). The morphology and average particle size of the Z and ZJ2 catalysts were examined through Transmission Electron Microscope using FEI Tecnai G2 S-twin instrument with a UHR pole piece. The samples were dispersed in chloroform, sonicated and deposited on carbon coated copper grids for TEM analysis. A DRS-UV spectrophotometer (Shimadzu UV 2450) was used to study absorption maximum of the synthesized samples using BaSO₄ as a reference. A Quadrasorb station 2 instrument surface area analyzer was used to determine the surface area of the sample. The extent of mineralization of the crystal violet dye was measured using Total organic carbon analyzer (Shimadzu TOC-V CPN).

2.2. Evaluation of photocatalytic activity

To investigate the photocatalytic efficiency of the synthesized ZnO samples, experiments were conducted in a Heber single tungsten lamp (500 W) visible photoreactor with the emission wavelength of 400 nm. In a typical run, 100 ml of 25 μM solution of crystal violet dye loaded with 0.1 g of the catalyst was stirred in dark for 30 min to allow for adsorption-desorption equilibrium and then irradiated under visible light. Aliquots of the dye solution were withdrawn at regular intervals, centrifuged and the supernatant was analyzed using a UV/visible

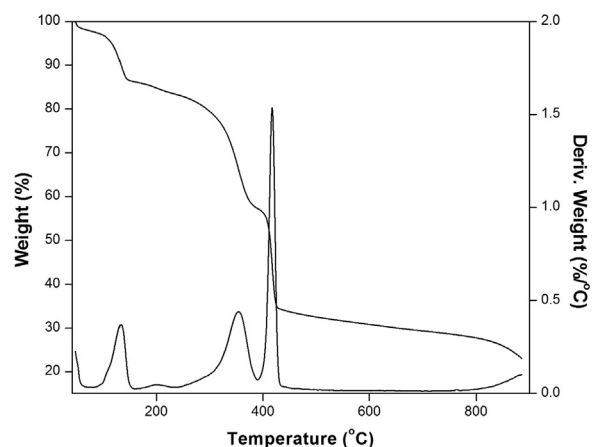


Fig. 1. TGA and DTG graphs of as synthesized ZJ2.

spectrophotometer at the absorption maximum of crystal violet (595 nm).

3. Result and discussion

3.1. Thermal characterisation

The TG and DTG curve of the as-synthesized ZJ2 nanoparticle is displayed in Fig. 1. The major contents of jute fiber are cellulose, hemicelluloses and lignin [35]. The initial degradation of extending till 200 °C was due to desorption of the surface-adsorbed water molecules with the weight loss of 12.48%. The next stage of degradation starting from 200 to 370 °C was due to the degradation of hemicelluloses [38] and some of the lignin content of the jute fiber accompanied by the weight loss of 25.10%. Brebu et al. [39] reported that the decomposition of lignin occurred over a wide range of temperature (200–500 °C). The weight loss of 24.84% was due to the decomposition of cellulose ranging from 380 to 420 °C [40] ensuing to the third stage of degradation. No significant change in weight was observed beyond this temperature. Thus, it can be inferred that a calcination temperature of 500 °C was sufficient for the removal jute template to obtain pure ZnO.

3.2. Spectral characterisation

The AT-IR spectrum of raw jute (a), as synthesized (b) and calcined ZJ2 (c) and non-templated ZnO (d) are presented in Fig. 2. The peaks at 3400 and 1059 cm⁻¹ in raw jute (RJ) can be attributed to –OH stretching vibrations and O–C–O stretching vibration of cellulose backbone, respectively. In the as-synthesized ZnO sample, while the intensity of the –OH peak increased, that of the O–C–O peak became weaker. This may have been due to the disturbance of O–C–O linkages in the cellulose moieties and the consequent formation of intra molecular hydrogen bonding in jute [41]. The peak at 1634 cm⁻¹ in RJ assigned to carbonyl group of jute fiber, got shifted to the lower wave number (1625 cm⁻¹) in as-synthesized, jute-templated ZnO. This could have been due to the formation of chelating bond between the carbonyl group of the fiber and Zn metal ion [32,42], since jute fiber has the ability to form a chelating complex with metal ions [36]. However, the calcined sample exhibited only the characteristic ZnO peaks similar to those of control ZnO, confirming the formation of pure ZnO without any impurities at the calcination temperature employed.

Fig. 3 shows the XRD patterns of calcined ZnO synthesized with and without jute fiber template. The diffraction patterns of the raw jute fiber and the as-synthesized sample are also depicted as an inset in Fig. 3. Crystallite sizes of all the synthesized samples were calculated using Debye–Scherrer formula and listed in Table 1 [43]. The resultant prominent peaks of ZnO were found to be in good agreement with the

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