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Facile synthesis and characterization of zinc oxide nanoparticles and studies of their catalytic activity towards ultrasound-assisted degradation of metronidazole

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ABSTRACT

A novel and facile approach for synthesis of zinc oxide (ZnO) nanoparticles (NPs) employing homogeneous chemical precipitation followed by hydrothermal heating technique is reported. The present method of synthesis of ZnO NPs is very efficient and cost effective. As-synthesized ZnO NPs were characterized by XRD, FT-IR, EDX, TEM, and N_2 adsorption–desorption (BET) studies. The powder XRD pattern furnished evidence for the formation of hexagonal close packing structure of ZnO NPs having average crystallite size 21.93 nm. The shapes of synthesized ZnO NPs are mostly quasi-cylindrical with sizes 20–50 nm. These ZnO NPs were used as catalysts for the degradation of a pharmaceuticals waste, metronidazole under ultrasound irradiation.

1. Introduction

In the recent years, metal-oxide nanoparticles have been the subject of much interest because of their unusual properties, which often differ from the bulk. These materials have received significant attention as efficient catalysts in many organic reactions due to their high surface area to volume ratio and co-ordination centers which provide a larger number of active sites per unit area in comparison with their heterogeneous counter sites [1–4]. Several methods have been used to prepare ZnO NPs. Some of the important ones are laser ablation, combustion method, electrochemical depositions, sol–gel method, hydrothermal methods, thermal decomposition, chemical vapor deposition, ultrasound, microwave-assisted combustion method, co-precipitation, and mechanical milling [5]. Although each of these methods has its own merits, some of them suffer from drawbacks such as use of expensive chemicals, surfactants, calcination at high temperature etc. Among these various methods, hydrothermal procedure has several advantages as this method does not require high temperature and organic solvents [6]. Moreover, any extra work-up such as grinding and calcination can be avoided and the products can be obtained in high purity and crystallinity.

Attention of the readers may be drawn to the fact that ultrasound irradiation in degrading the organic pollutant has been proved to be an useful tool in accelerating dissolution, enhancing reaction rates, renewing the surface of solid catalysts or reactants etc [7–9]. Thus combination of nanocatalysts with ultrasound irradiation in degradation reactions will certainly open up a new avenue for highly efficient environmentally friendly synthetic protocols.

The presence of pharmaceuticals waste in surface water from industries and personal care products are considered to an emerging environmental issue of concern due to their impact on human health and aquatic life in recent years [7,10]. Therefore, development of newer technologies from removal of pharmaceuticals waste before their release into the environment through degradation will always be important in the present day context. A comprehensive literature study reveals that zinc oxide NPs catalyzed degradation of metronidazole by ultrasound assisted technique does not appear to have reported previously.

As a sequel to our current endeavor [11–14] on the synthesis and application of nanocatalysts, we report herein a new facile synthesis of ZnO NPs and their application as catalyst for ultrasound assisted degradation of metronidazole.

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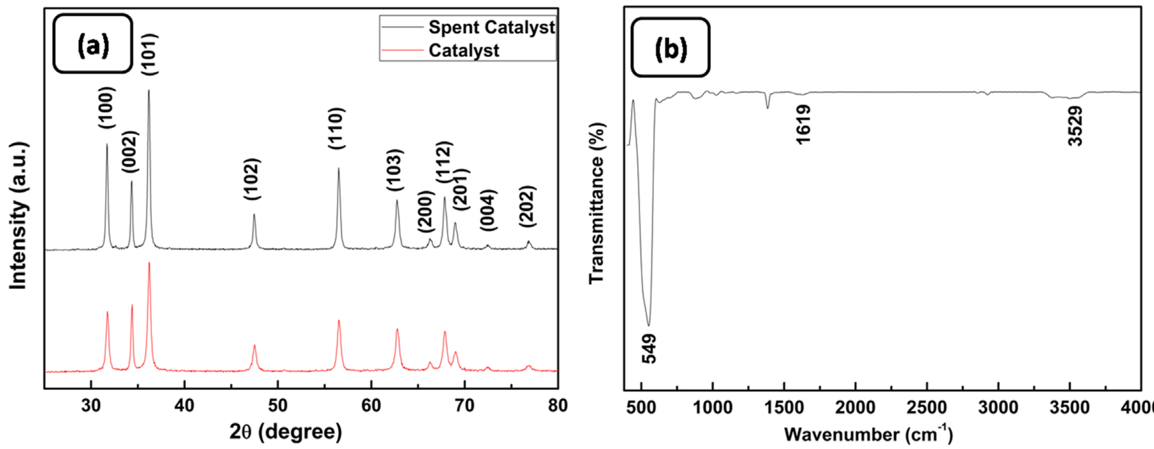

Fig. 1. (a) Powder XRD patterns of ZnO nanocatalysts. (b) FT-IR Spectrum of ZnO NPs.

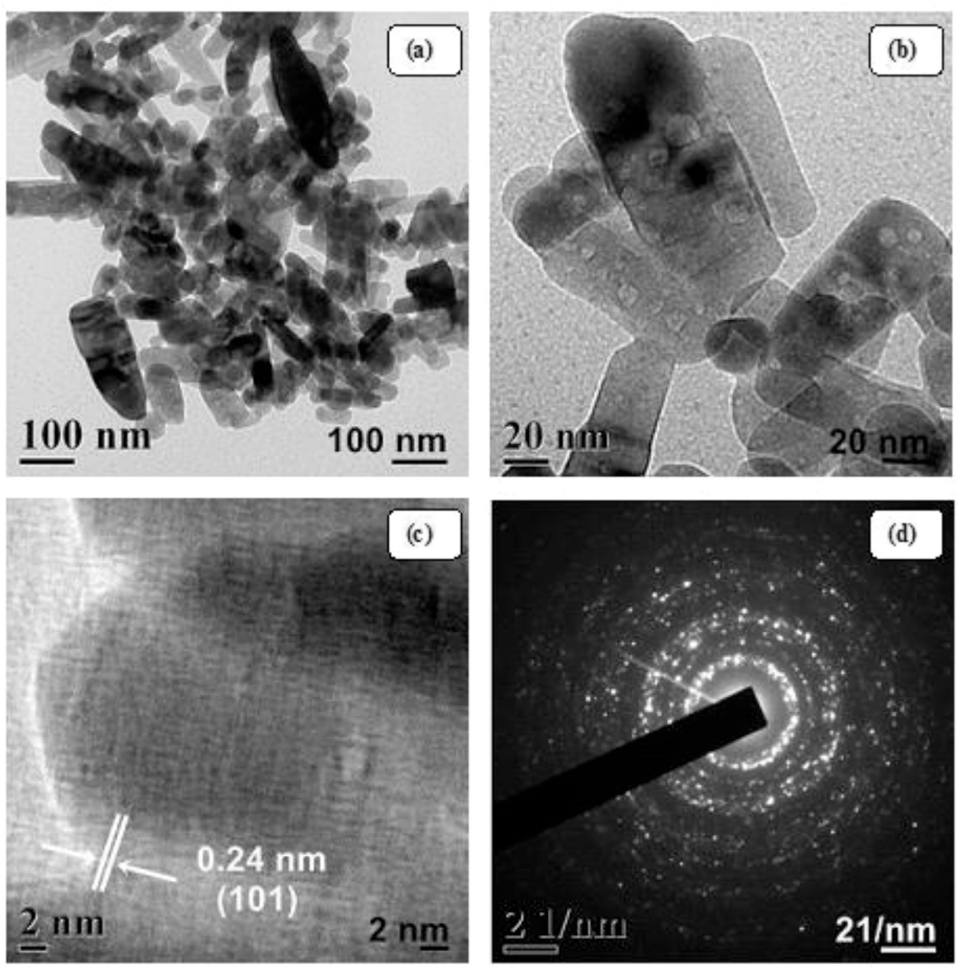

Fig. 2. (a, b) TEM images (c) HRTEM image and (d) ED pattern of ZnO nanocatalyst.

2. Experimental

2.1. Materials and physical measurements

Zinc chloride ($ZnCl_2$) and tributylamine ($C_{12}H_{27}N$) were purchased from Merck India Ltd. FT-IR spectrum was recorded on KBr matrix with Bruker 3000 Hyperion Microscope with Vertex 80 FT-IR system. XRD measurements were carried out on a Bruker AXS D8-Advance powder X-ray diffractometer with Cu-Kα radiation ($\lambda = 1.5418$ Å) with a scan speed of 2°/min. Transmission electron microscopy images were obtained on a JEOL, JEM2100 equipment. The sample powders were dispersed in ethanol under sonication and TEM grids were prepared using a few drops of the dispersion followed by drying in air. Sonication was performed in Qsonica Q700 sonicator with a frequency 20 kHz and at a nominal power of 250 W.