

Electrospun self-assembled ZnO nanofibers structures for photocatalytic activity in natural solar radiations to degrade acid fuchsin dye

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ABSTRACT

Zinc oxide (ZnO) nanoparticles, self-assembled in the form of one dimensional ZnO nanofibers were synthesized using electrospinning technique from solution of polyvinyl alcohol (PVA) and zinc acetate followed by calcination at 600°C in oxidizing environment. Scanning Electron Microscope (SEM) analysis demonstrates that morphology of ZnO nanofibers having rough surface and corresponding Energy Dispersive Spectrometry (EDAX) confirmed the Zn: O atomic ratio approximately in 50:50. Transmission electron microscopy (TEM) images clearly demonstrate the rough morphology is due to the self-assembling of ZnO nanoparticles having diameter approximately 50nm. X-ray Diffraction (XRD) reveals the polycrystalline structure and Raman spectra show some shifts in phonon modes. The PL graph show exceptional emission at 342nm due to band-band transition. Under solar radiations as produced ZnO nanofibers degrades the 99% of 25ppm acid fuchsin which proven through UV spectra when compared to blank dye solution. This shows that natural solar radiations are sufficient to excite these self-assembled high surface area ZnO nanofibers to show its photocatalytic activity. Copyright © 2015 VBRI Press.

Keywords: Electrospinning; nanoparticles; nanofibers; solar radiation; photocatalytic activity; self-assembly.

Introduction

ZnO being non-toxic, photoactive and having wide band gap, i.e., 3.4eV at room temperature, is most concerned transition metal oxide with a large exciton binding energy of 60meV [1]. These all provides opportunities for its use in the various applications like renewable energy [2], solar cell electrodes, gas sensors [3], LED lights, waste water treatment due to photocatalytic activity and optoelectronic devices in different forms [4] like nanoparticles, thin films, nanowires. For high photocatalytic activity size of ZnO plays significant role, as in larger size particles photo induced electron holes recombines rapidly which lowers the photocatalytic activity. On the other hand nanoparticles have problem of aggregation and even re-pollute the water, while in case of ZnO thin films the yield is very low to meet the industrial requirement for the water treatment applications. Among all the forms, due to large aspect ratio and high specific surface area, 1D nanostructure has been gaining more attention among researchers. These are very stable and does not aggregates or re-pollute the water also they are proven to be better photocatalytic agent. Although there are many methods for fabrication of 1D structure like sol-gel process [5], sputtering [6], spray pyrolysis[9],

hydrothermal [7, 8] and electrospinning [10, 11], out of these electrospinning is the most fascinating and simple technique to fabricate nanofibers. As compared to other methods it is an inexpensive, versatile and convenient method to form both polymeric [12, 13] and ceramic nanofibers. In this method when high voltage is applied to the polymeric solution, the surface tension gets over by electric field, polymer stretches in the form of fibers and gets collected on conducting collector of opposite charge. A core shell structure can be formed easily by incorporating metal salts in polymeric nanofibers and then sacrificing polymeric nanofibers [10, 14-17] to get pure metal oxide nanofibers [18].

Various inorganic nanostructures, due to their photocatalytic activity, have been tested for dye removal from waste or contaminated water. Dyes are the aqueous pollutants mostly originate from industrial waste in the water. These dyes are being used as main or additive ingredient in pharmaceutical, chemical, petroleum and leather industry and proven to be carcinogenic. Acid fuchsin is the one of such dyes used as an inhibitor of reverse transpose of immunodeficiency virus [19], as a copper corrosion inhibitor [20], in the preparation of organic inorganic hybrid nano-composite [21] and as a

laboratory reagent to stain cytoplasm and nuclei of tissue sections. ZnO in various forms has been proven to be good photocatalytic agent for the wastewater treatment.

To investigate ZnO nanofibers or other forms for its dye degradation ability from waste water, a number of UV sources have been used such as UV lamp, tungsten lamp [10], mercury lamp [16], halogen lamp [22] etc. These sources are costly and can be effective in lab stage but not viable for industries use. However, solar photocatalysis is a cost effective and sustainable treatment due to the utilization of solar energy (an abundant source of energy). Our sun emits ultra-violet radiations of all wavelengths. Shortest wavelength UV radiations (100-280) are germicidal and completely absorbed by OZON layer and atmosphere, while medium waves (280-315nm) also absorbed mostly by atmosphere so these UV radiations are not available for chemical reactions. Only the large wavelength UV radiation (315-400nm) reaches to the earth, although these have very less energy to ionize atoms but can cause chemical reactions. ZnO having band gap 3.37 or less can be easily excited to take part in chemical reactions using these UV radiations. Till now none have reported or investigated solar radiation (which is a natural source) for dye degradation using as produced ZnO nanofibers. In present investigation, the energy from these natural radiations is found to be quite sufficient to excite ZnO nanofibers to catalyze the dye degradation and exhibit photocatalytic activity.

Experimental

Polyvinyl alcohol (PVA) cold (M.wt. 125,000) and zinc acetate dihydrate ($Zn(ac)_2 \cdot 2H_2O$) were purchased from CDH, New Delhi. Fuchsin acid dye having 99.8% purity was procured from fisher scientific. $Zn(ac)_2$ and PVA cold (Mwt:1,25000) were dissolved in distill water in a ratio of (3:4) by sonication followed by magnetic stirring 15h for uniform mixing. A clear homogeneous solution obtained was electrospun by optimizing processing parameters to get bead free and uniform fibers by using electrospinning equipment (ESPIN-NANO, procured from Physics Instrument Company, Chennai)[23, 24].

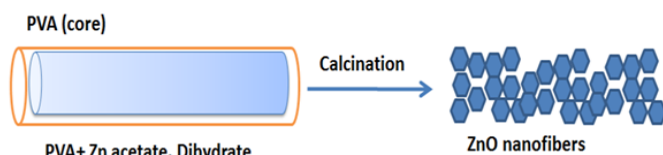


Fig. 1. Schematics of ZnO nanofibers formation from PVA and $Zn(ac)_2$ core-shell structure.

The parameters were kept constant as 0.1ml/h flow rate, tip to collector distance 20cm and drum collector speed 2000 rpm. Thus formed fibers were calcinated at 600°C in isothermal heating furnace upto 4 hours in air. A solution of 25ppm acid fuchsin in water was prepared by serial dilutions of stock solution (1mg/ml).

Characterizations

Morphology of nanofibers formed before and after calcination were characterized using scanning electron

microscope (SEM, VP-EVO MA-10, Carl Zeiss, UK). The ZnO nanofibers were characterized by high resolution transmission electron microscope (HRTEM, JEOL 2200 FS with a Cs-corrector). Raman studies of PVA- $Zn(ac)_2$ and ZnO nanofibers were carried out using a Renishaw Via Reflex Raman spectrometer, UK, with an excitation laser source of 514nm wavelength. The Raman spectra were recorded from 100 cm^{-1} to 3000 cm^{-1} with 5 mW laser power and 10 s of exposure time. The crystal structure of ZnO was studied by X-ray diffraction (XRD, D-8 Advanced Bruker diffractometer) using CuK_{α} radiation ($\lambda = 1.5418 \text{ \AA}$). Optical properties of solid ZnO nanofibers were studied using Photoluminescence spectrometer (Perkin-Elmer, Model No.LS-55).

Photocatalytic characterization

The solutions with and without ZnO nanofibers were studied at different conditions using UV spectrophotometer (Model: 160 UV, Shimadzu). The degradation of dye was confirmed by reduction in peak intensity at 546.5 nm (abs. wavelength for fuchsin acid).

Results and discussion

Fig. 2 illustrated the morphology of PVA- $Zn(ac)_2$ electrospun nanofibers observed by SEM. The SEM image of PVA- $Zn(ac)_2$ nanofibers mat shows the wide variation in the diameter of nanofibers. There is some $Zn(ac)_2$ particles also visible on the surface of nanofibers which fall as droplet during the electrospinning.

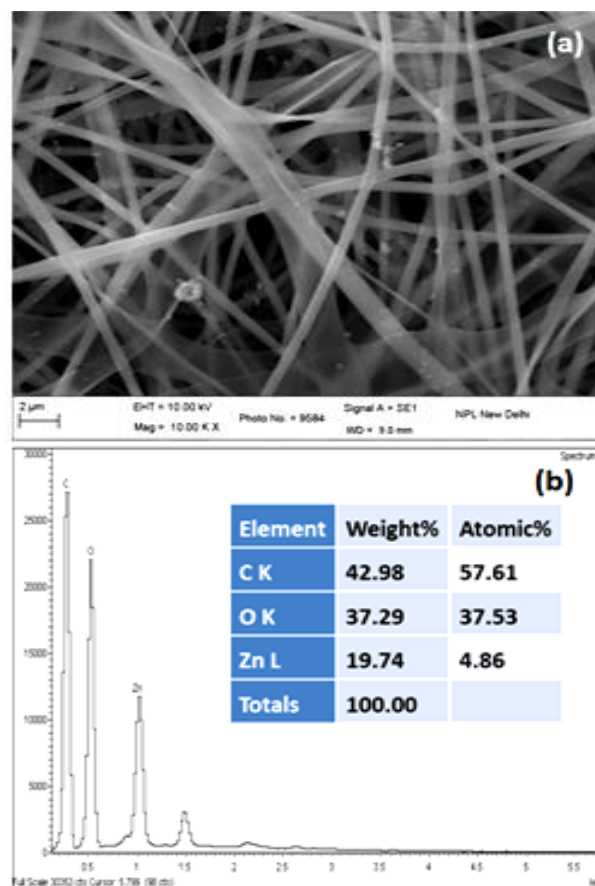


Fig. 2. (a) SEM image and (b)EDAX of PVA- $Zn(ac)_2$ nanofibers.

It can be seen that many nanofibers become flat which may be due to the slow drying of solvent in nanofibers. It is formerly proven that water solvent evaporation rate is slower than other organic solvent; this is due to the increased viscosity. The nanofiber collected on the collector due to the sudden impact, they get flattened[25]. In our case viscosity increases due to the $\text{Zn}(\text{ac})_2$. The morphology of nanofibers signify zinc acetate particles embedded in PVA fibers and the corresponding energy dispersive X-ray analysis (EDAX) shows the presence of carbon, zinc and oxygen collectively from PVA and $\text{Zn}(\text{ac})_2$ (Fig. 2b).

Fig. 3(a) shows SEM image of ZnO nanofibers derived after calcinations of PVA-Zn(ac)₂ composite fibers in air. During calcinations at 600°C, PVA got degraded and only ZnO remains in the structure.

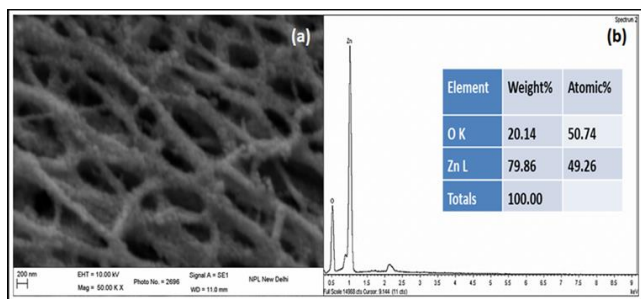


Fig. 3. (a) SEM image and (b) EDAX of ZnO nanofibers.

These ZnO nanofibers have very rough surface and diameter is in range of 160-180 nm. During calcinations ZnO is formed from $\text{Zn}(\text{ac})_2$ dehydrate salt. This has been evidenced in elemental analysis by EDAX in Fig. 3(b). This shows pure Zinc and Oxygen present in nanofibrous structure in ratio of 50:50 in atomic %. It seems like particles of ZnO are assembled to form a fibrous structure.

Fig. 4 shows TEM images of ZnO nanofibers with the rough surface, in which ZnO particles of different sizes assembled in the form of fibrous structure (Fig. 4a). The nanoparticles of size 50-60nm interconnected to each other, may can be due to the diffusion. Fig. 4(b) shows the magnified TEM image showing ZnO nanoparticles attached to each other.

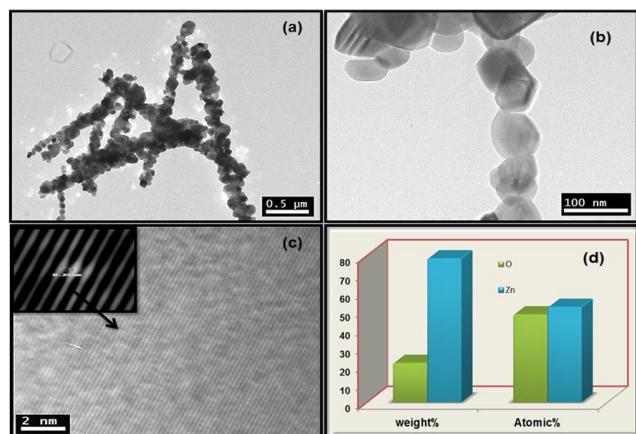


Fig. 4. TEM image of ZnO nanofibers (a) at low magnification (b) at higher magnification (c) HRTEM of single ZnO particle (d) EDAX of ZnO.

These are in the form of self-assembled directly through minimization of Gibbs free energy. The minimization of free energy is a result of weaker intermolecular force between self-assembled ZnO particles. So the forces acting between ZnO nanoparticles are weak intermolecular forces like vander waal or weak polar forces of attraction. There is some balance of attractive and repulsive forces which allows them to organize in the form of fibers. Fig. 4(c) shows the HRTEM image of ZnO particles having d-spacing 0.24nm which can be further confirmation by XRD reported in the next section with its polycrystalline character. EDAX of ZnO nanofibers (in situ TEM) revealing presence of only Zn and O molecules in Fig. 4(d).

Fig. 5 shows the Raman spectra of the PVA-Zn(ac)₂ and ZnO nanofibers membrane. Raman spectra of PVA-Zn(ac)₂ in Fig.5(a) shows two major peaks ~ 1436cm⁻¹ attributed to -CH and -OH bending and ~ 2919cm⁻¹ is due to the stretching vibrations of -CH and -CH₂ in PVA and Zn(ac)₂ both. While after calcinations at 600°C, the ZnO nanofibers are evidence for the clear phonon modes (Fig. 5b).

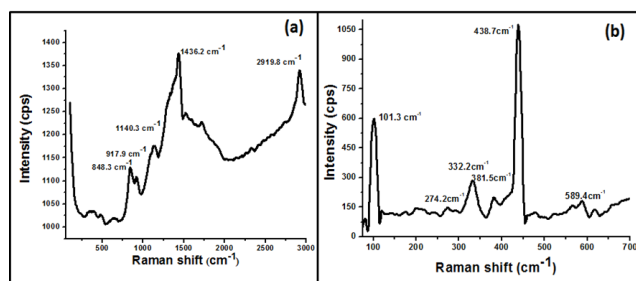


Fig. 5. RAMAN spectra of (a) PVA-Zn(ac)₂ nanofibers and (b) ZnO nanofibers.

There are eight sets of zone centre optical phonons, according to Group theory out of which two for the transverse optical (TO) phonons, two for longitudinal optical (LO) phonon, two low and high phonon mode as E₂ are Raman active. The E₂(high) phonon mode is most prominent around 438 cm⁻¹ is characteristic of the wurtzite phase particularly[26]. The peak near 101 cm⁻¹ is characteristic of E₂ low phonon mode[27]. However there is a +7cm⁻¹ shift in both second order Raman (originating from zone boundary phonon 3E_{2H}-E_{2L}) and A_{1T} appearing at 332 and 381cm⁻¹ instead of 339 and 388cm⁻¹. Also a +6 cm⁻¹ shift is found in E₁(LO) Raman active phonon mode. As nanostructures formed the different morphologies may have different internal strains due to growth in different directions. In our case Raman shift may be due to different morphology i.e. ZnO nanoparticles assembled in to nanofibers form.

Fig. 6(a) shows the XRD diffraction pattern of as obtained ZnO nanofibers after calcination. The XRD spectra shows various diffraction peaks at 31.78°(100),34.44° (002), 36.28° (101), 47.58° (102), 56.62° (110), 62.92° (103),66.46° (200),68.04° (112) and69.12°(201).

All the diffraction peaks are well matched with the hexagonal wurtzite crystal structure of ZnO (JCPDS 89-1397).

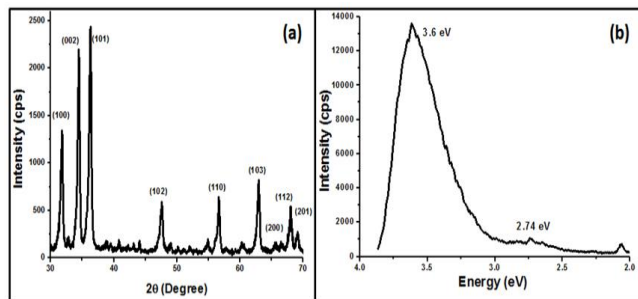


Fig. 6. (a) XRD pattern and (b) Photoluminescence behavior of ZnO nanofibers.

The peak at 36.28° (101 plane) is found to be most prominent which translates to a d-spacing of 2.47 \AA [28]. This agrees the d-spacing calculated from HRTEM in Fig. 4. The crystallite size was calculated for (101) plane was 22nm which is approximately half to the nanoparticle size i.e. 30-40 nm (using TEM). The solid self-assembled ZnO nanofibers membrane is characterize for optical behavior. PL spectra (Fig. 6b) show two emissions at the room temperature from ZnO nanofibers on excitation at wavelength in UV region 300 nm.

It demonstrates an ultraviolet emission at 342nm (3.625 eV) and blue emission at 451 nm (2.74) in visible region. A small peak at 600 nm (2.06eV) is harmonic to excitation wavelength of 300 nm. In the literature the emission of ZnO shown at 380-383 nm (i.e. 3.37eV) but in our case a blue shift of 40 nm is found in PL spectra by emission at 342 nm, that may be due to the Burstein-Moss effect, the band-band transition is expected to be enhanced rather than by free exciton (380nm), and so shift toward higher energy [29]. Also the emission at in visible region (451nm) is very low which shows surface defects transition is negligible, hence surface defects are very low in as prepared ZnO nanofibers by electrospinning followed by calcination.

To study the degradation of fuchsin dye, 25ppm acid fuchsin dye solution is prepared and divided in three equal parts named as solution [a], [b] and [c]. Solution [a] consists of as such dye only and solution [b] and [c] with 5mg ZnO nanofibers. Solution[b] having ZnO nanofibers is kept in the dark, while solution [c] having ZnO nanofibers is exposed to sunlight for 2.5 h.

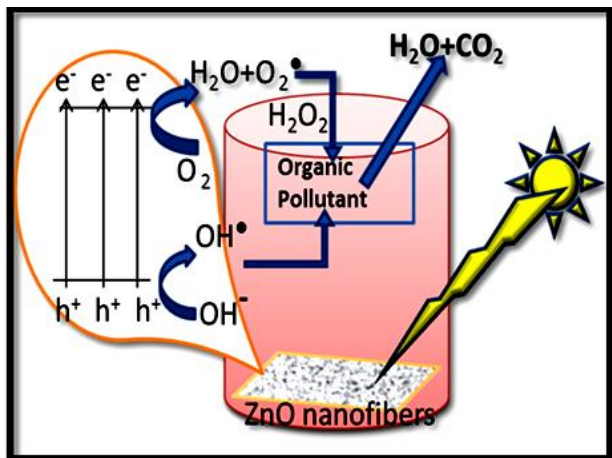


Fig. 7. Schematic of ZnO nanofibers activation and dye degradation mechanism.

When solar radiation of desired energy (wavelength) fall on ZnO nanofibers, electrons get excited to conduction band leaving holes (+ve charge carriers) in valance band. These electrons and holes generated during the photo catalysis can degrade almost all types of organic, inorganic, and microbial contaminants, due to their high redox potentials. Holes react with hydroxyl ion (OH^-), to form hydroxyl free radical (OH^\cdot). In the same way photo generated electrons helps to form superoxide anion free radicals (O_2^\cdot), But these may form H_2O_2 when react with water and evolved out of reaction medium. Free radicals OH^\cdot and O_2^\cdot produced in the solution then reacts with organic pollutants (dyes) and degrades the conjugated bonds and as a result decolorizes it. The H_2O and CO_2 produced as byproducts which are harmless. In this way water purification occurs with ZnO nanofibers. The schematic of mechanism of degradation of fuchsin dye is illustrated in Fig. 7.

The Fig. 8 shows UV spectra of all the three solutions along with distill water as blank in which dye solution is prepared.

The solar radiation as a source of photons for the activation of catalyst is used. It shows the clear difference in absorbance in as such acid fuchsin (25ppm) dye solution and same containing ZnO nanofibers. The UV spectrum of as such dye solution shows absorbance at 546.5 nm with absorbance value 1.0 while in case of solution[b] shows absorbance of 0.701. Solution[c] (i.e. exposed to solar radiation) shows absorbance 0.187 which is near to pure distills water i.e. 0.1824 in (curve d). This shows the dye degradation is 99% when exposed to solar radiation although in dark dye also degraded to 37% as compared to fresh dye solution.

Also there is one peak found around 340nm but only in both the samples containing both ZnO and dye which suggest some intermediate structure formation due to the reaction between ZnO and fuchsin acid.

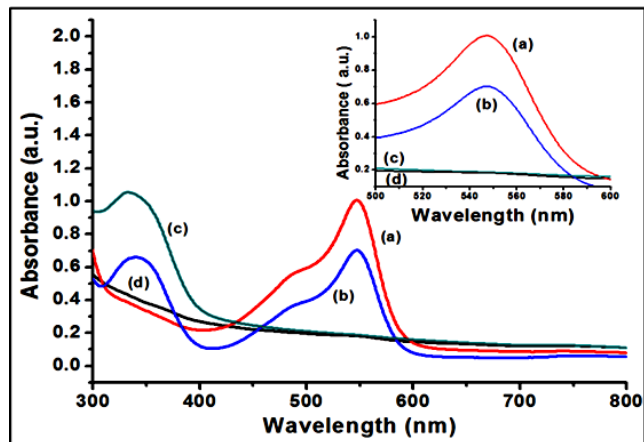


Fig. 8. UV spectrum of acid fuchsin dye 25ppm (a) as such (b) with ZnO nanofibers in dark, (c) with ZnO exposed to solar radiation and (d) distill water as blank.

Conclusion

The self-assembled ZnO nanofibrous structure is derived from the electrospun $\text{PVA}/\text{Zn}(\text{ac})_2$ nanofibers after

calcinations at 600°C. The different characterization technique evident ZnO nanoparticles assembled by weak vander Waal type interactions during calcinations process. The XRD and TEM investigation reveals its wurtzite structure. The optical behavior by PL spectra demonstrates an ultraviolet emission at 342nm (3.625 eV) and blue emission at 451 nm (2.74 eV) in visible region. The blue shift of self-assembled nanofiber by 40nm in PL spectra is due to the Burstein-Moss effect, the band-band transition is expected to be enhanced. Also the emission at in visible region (2.74 eV) is very low which shows surface defects transition is negligible in ZnO nanofibers fabricated by electrospinning technique. The photo degradation of acid fuchsin present in waste water under solar radiation demonstrates 99 % degradation within 2.5 hr time. Thus self-assembled ZnO nanofibrous structure can be promising cost effective and sustainable solar photocatalyst for different applications.

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