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EMERGING TRENDS IN SCIENCE AND TECHNOLOGY (ETIST-2021)

27th October 2021

Jointly Organized by
Department of Biological Science, Physical Science and Computational Science

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A nations's growth is in proportion to education and intelligence spread among the masses. Having this idealistic vision, two great philanthropists late. S.P. Nallamuthu Gounder and Late. Arutchelver Padmabhushan Dr.N.Mahalingam formed an organization called Pollachi Kalvi Kazhagam, which started NGM College in 1957, to impart holistic education with an objective to cater to the higher educational needs of those who wish to aspire for excellence in knowledge and values. The College has achieved greater academic distinctions with the introduction of autonomous system from the academic year 1987-88. The college has been Re-Accredited by NAAC and it is ISO 9001 : 2015 Certified Institution. The total student strength is around 6000. Having celebrated its Diamond Jubilee in 2017, the college has blossomed into a premier Post-Graduate and Research Institution, offering 26 UG, 12 PG, 13 M.Phil and 10 Ph.D Programmes, apart from Diploma and Certificate Courses. The college has been ranked within Top 100 (72nd Rank) in India by NIRF 2021.

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ACETONE SENSING PROPERTY OF MESOPOROUS ZnO NANOSTRUCTURES

M. Chitra[1] - K. Uthayarani[1*]- N. Neelakandeswari[2] and E. K. Girija[3]

ABSTRACT: Mesoporous zinc oxide nanostructures synthesized via sol-gel route using rice husk as template is subjected to acetone sensing property at room temperature. The functional group analysis of the prepared sample was identified by infrared spectroscopic tool. The structure and morphology of the nanostructures were characterized by X-ray diffraction, scanning electron microscope (SEM), transmission electron microscope (TEM) and Nitrogen adsorption–desorption analyses. SEM and TEM observations revealed the formation of spherical zinc oxide nanoparticles over the interwoven fibrous network. The porous network of the synthesized material helps in transportation of acetone vapour even at room temperature.

Keywords: sensors, acetone, room temperature, porous materials, ZnO, biotemplate

1. INTRODUCTION

Acetone, an explosive and toxic gas, needs to be detected because when inhaled it causes headache, fatigue and harmfulness to nerve system [1]. The widespread detection and continuous monitoring of such an organic pollutant requires a low-cost, fast, sensitive, selective and stable sensing device [2]. Traditional sensing technologies reported earlier suffer from poor selectivity [3]. But, chemiresisitive sensors which employ metal oxide semiconductors (MOS) as functional materials have a great demand nowadays to overcome the shortcoming of unselectivity [4]. Thus the controlled synthesis of such active materials with different shape and morphology has attracted considerable interest so that a portable sensor can be obtained with simple fabrication technology. Among various MOS, zinc oxide (ZnO) has proven to be an efficient ethanol sensor at room temperature in our earlier reports [5]. Especially, extensive studies are available on porous ZnO nanostructures which favours quick diffusion of gases and thereby enhances the surface reaction to take place at a higher rate [6]. Hence, in this present work, mesoporous ZnO nanostructures were synthesised via sol-gel route using rice husk as the biotemplate, characterised by various state-of-the-art techniques and tested for acetone sensing at room temperature.

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2. **EXPERIMENTAL DETAILS**

The starting materials, zinc chloride ($ZnCl_2$), hexamethyltetramine (HMT) purchased from Rankem were of analytical grade and were used without further purification. Rice husk to be used as the biotemplate was purchased from local rice mill. Calculated quantity of rice husk was soaked in 0.1 M aqueous zinc chloride solution for 24 h at room temperature. The pH of the solution was maintained at 9 by adding aqueous HMT solution to get zinc ions templated over the rice husk. Soaked solution were filtered, washed, dried at room temperature and calcined at 400 ºC for 3 hrs. The obtained rice husk templated zinc oxide was labelled as ZRH. The flowchart for the synthesis of ZnO nanostructures using rice husk template is presented in Figure 1. Thus the synthesised biotemplated ZnO nanostructures with porous nature has already been reported for ethanol sensing at room temperature [5].

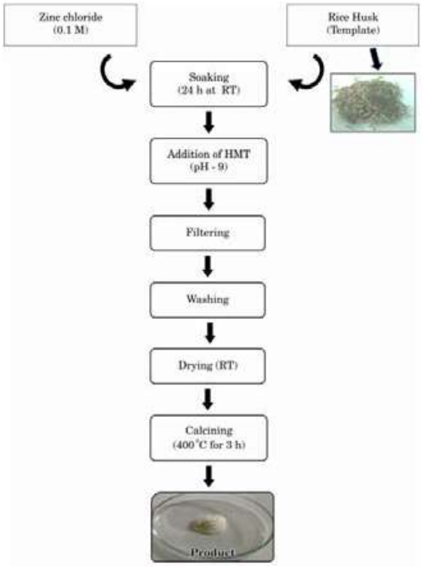

Figure 1 Flowchart for the synthesis of ZnO using rice husk

FT-IR spectra were recorded for ZRH synthesized before and after calcination using a Perkin Elmer RX1 FT-IR spectrophotometer by KBr pellet technique in the range between 400 cm^{-1} - 4000 cm^{-1}. The powder X-ray diffraction (XRD) pattern of the samples was recorded using PANALYTICAL X – Ray diffractometer in 2θ ranging from 5° - 80°. The morphology was recorded using JOEL Model JSM – 6390LV Scanning Electron Microscope (SEM) and the elemental composition was analysed by Energy Dispersive X-ray (EDAX) analysis attachment. High resolution Transmission Electron Microscopic image (HRTEM) of ZRH was recorded using JOEL JEM 2100 Transmission microscope. Pore size distribution (PSD) analysis was carried from the Barrett–Joyner–Halenda (BJH) plot obtained with a Micromeritics apparatus. ZRH was tested towards sensing of acetone using the gas sensing apparatus [5]. ZRH pellet is exposed to air and acetone consecutively at room temperature. The change in resistance is calculated from the output voltage for the constant current using Agilent source meter. From the resistance value, the sensitivity was calculated using the formula [7] -

$$\text{Sensitivity} = \frac{\left|R_{air} - R_{acetone}\right|}{R_{air}} \text{ x } 100\,\% \qquad \text{..................} \; (1)$$

where R_{air} is the resistance of the sample in air and $R_{acetone}$ is the resistance of the sample when exposed to acetone vapour.

3. RESULTS AND DISCUSSION

Figure 2 (a) and 2 (b) shows the FT-IR spectrum of ZRH before and after calcination respectively. The bands obtained for ZRH before calcination reveals the presence of certain functional groups of the template. The bands between 3750 cm^{-1} to 3250 cm^{-1} correspond to the – OH stretching vibration present in both the template and $Zn(OH)_2$. The bands between 3000 cm^{-1} and 2500 cm^{-1} correspond to the C-H stretching of alkanes [8] which might be exclusively due to the presence of cellulose structure of the template. The bands between 1750 cm^{-1} and 1500 cm^{-1} correspond to the – OH bending vibration of both the cellulose structure and $Zn(OH)_2$. The bands at 906 cm^{-1} and 746 cm^{-1} correspond to the Si – O vibrations of the template. After calcination, most of the bands corresponding to the organic molecules disappear and this shows the complete decomposition of the template. Thus it is understood from the spectrum of ZRH, decomposition of the rice husk template due to calcination and the presence of metal oxide has been identified. The bands between 3500 cm^{-1} and 3700 cm^{-1} are due to the stretching vibration and the bands between 1500 cm^{-1} and 1670 cm^{-1} occur due to the bending vibration of surface hydroxide. The bands at 897 cm^{-1} and 738 cm^{-1} correspond to M-O deformation vibration and match well with the reported values of ZnO [9, 10].

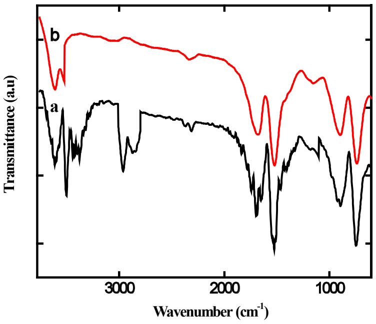

Figure 2 FT-IR spectra of ZRH (a) before and (b) after calcination

Figure 3 (a) shows the XRD pattern of ZRH which shows sharp peaks at 31.45°, 34.11°, 36.03°, 47.19°, 56.33° in addition to the broader peak at 23 °. These sharp peaks correspond to the hexagonal phase of wurtzite structured ZnO [ICDD file - 36 – 1451] and the existence of these sharp peaks along with the broader peak indicates the presence of crystalline phase (ZnO) onto the matrix (rice husk). Crystallization had been taken place during calcination.

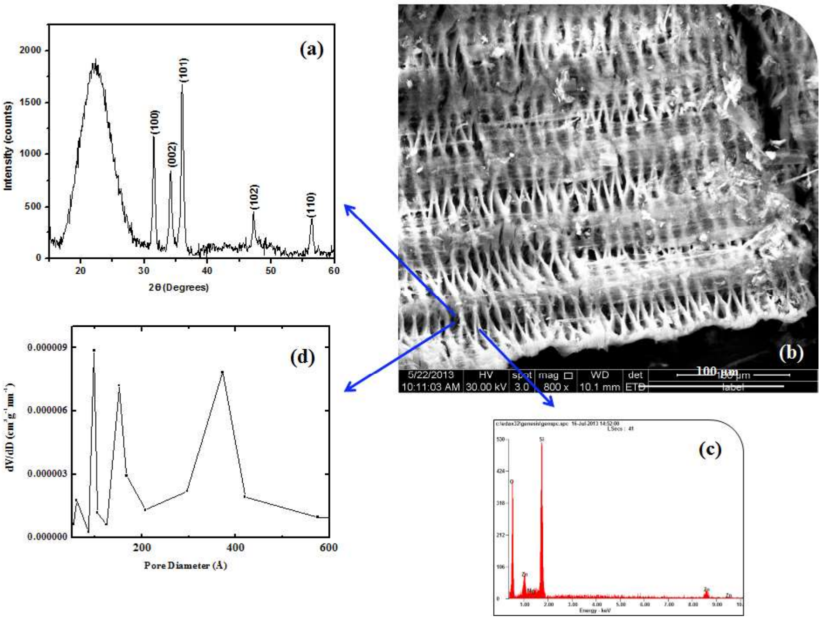

Figure 3 (a) XRD pattern (b) SEM image (c) EDAX spectrum and (d) Pore size distribution plot of RH

SEM image of ZRH shown in Figure 3 (b) reveals the interwoven fibre like morphology and EDAX of ZRH shown in Figure 3 (c) reveals the presence of the oxides of Si and Mg acting as a support for ZnO in ZRH which is also evidenced from XRD. These textured nanostructures of ZRH possess enormous pores of different sizes ranging from 10 - 50 nm which is in accordance with the pore size distribution plot shown in Figure 3 (d). All the pores are not loaded uniformly during the incorporation of ZnO onto the residual template which is in correlation with the multiple peaks obtained from PSD plot. HRTEM image of ZRH shows loaded spherical ZnO nanoparticles (around 25 nm) over the interwoven fibrous network (inset of Figure. 4 (a)).

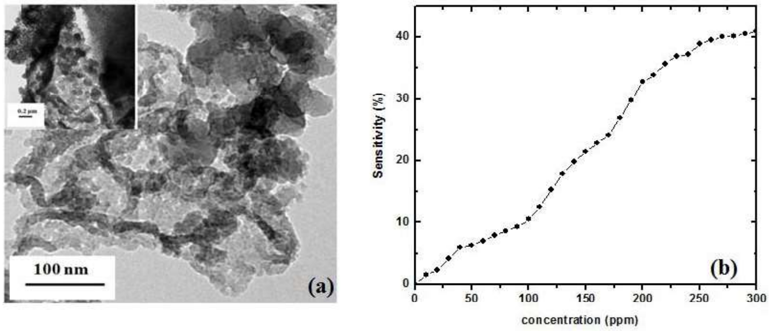

Figure 4 (a) HRTEM image of ZRH and coalescent spherical ZnO nanoparticles (inset) and (b) Sensitivity plot of RH

4. SENSITIVITY OF ZRH TOWARDS ACETONE PAPER LAYOUT DETAILS

Initially, when ZRH is surrounded by air, oxygen molecules will adsorb on the fiborous network to generate chemisorbed oxygen species (O^{2-}, O^{2-} and O^{-}) by capturing electrons from the conductance band and results in high resistance. When acetone is injected from 0 ppm to 300 ppm in steps of 10 ppm, the adsorbed oxygen species reacts with the reducing gas and the electrons are released to the fibrous network resulting in the reduction of resistance. ZRH reverts back again to its resistance on exposure to air. Thus the sensitivity of ZRH towards acetone increases linearly to 40.33% with increase in the concentration from 0 to 250 ppm. At low concentration of acetone, the larger response is attributed to the formation of unimolecular layer of gas molecules on the surface of the material which would interact with the surface more actively [11] and above 250 ppm, the formation of multilayers of gas molecules would result in saturation of gas response. Hence, the mass reactive sites and excellent permeability of porous ZRH assists in creating active sites on the surface and eventually enhance their activity towards acetone at room temperature.

5. CONCLUSION

Mesoporous ZnO nanostructures prepared via sol-gel route using the biotemplate, rice husk. The open channels and porous surface of ZnO enhances the surface accessibility and thereby felicitates acetone sensing even at room temperature.

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