Measurement 67 (2015) 1–9

Contents lists available at ScienceDirect

Measurement

journal homepage: www.elsevier.com/locate/measurement

Synthesis of simple and novel biocomposite doped nanocrystalline tin oxide and its humidity sensing properties

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ARTICLE INFO

Article history:
Received 25 March 2014
Received in revised form 7 December 2014
Accepted 3 February 2015
Available online 20 February 2015

Keywords:
Hygrophila Spinosa T. Anders
SnO_2
Mixed metal oxide
Negative photoconductivity
Biocomposite

ABSTRACT

The *Hygrophila Spinosa* T. Anders (HST) plant seed that contains mineral constituents were doped in SnO_2 prepared by the conventional method. The structural parameters, functional groups, surface morphology, elemental analysis and optical behavior of the prepared samples was made using X-ray diffraction, Fourier transform-infra red, scanning electron microscope, energy dispersive X-ray analysis, diffuse reflectance spectroscopy techniques respectively have been studied. XRD-surface area for all the samples was obtained from the Williamson–Hall plot. Photoconductivity studies were carried out for all the samples prepared with different HST composition in SnO_2. The negative photoconductivity was observed for the HST doped SnO_2 and HS samples, except pristine SnO_2. The SH2 sample with high specific surface area shows good response and recovery characteristics toward humidity.

1. Introduction

In the field of material science, it is interesting to study the structural, electrical and optical properties. Tin-oxygen system has two stable oxides of tetragonal modification, namely SnO and SnO_2. SnO is not widely applied as SnO_2 due to its low thermodynamic stability in condensed state [1]. The crystalline SnO_2 is a n-type semiconductor of tetragonal rutile structure with a wide band gap of 3.2–3.6 eV at room temperature, and it exhibits photoelectric properties. Hence it is considered to be an important metal oxide. SnO_2 has great potential in different areas of technical application such as gas sensor, photosensor, antistatic coating, opto electronic devices, solar cells, catalyst support, electrode materials and photovoltaic devices [2–4]. SnO_2 has the characteristics of high optical transparency in the visible range (97%), low resistivity (10^{-4}–10^{6} ohm cm) and excellent chemical stability [5]. The pristine SnO_2, due to its low resistivity, do not abruptly change the resistance values at higher RH (Relative Humidity). Therefore, it is important to bring the measurable changes in physical properties such as electrical conductivity of SnO_2, by altering the surface properties to find many applications in wide areas and this can achieved by element doping. Researchers have reported [6–13] in improving the humidity sensitivity of SnO_2 by doping La^{3+}, Ce^{3+}, K^{+}, Pd, TeO_2, poly (o-anisidine), In, Zr, Pt, and Sb. However, either the high cost or the less availability of many of these dopants is a disadvantage to use them. Hence, we focused our present work to dope the mineral oxides derived from bioresources. For this purpose, the seeds of *Hygrophila Spinosa* T. Anders, a well known medicinal plant rich in mineral content and found abundantly in the paddy fields and marsh areas, are used. This plant is widely distributed and the seeds are commercially available throughout India, Sri Lanka, Burma, Malaysia and Nepal [14]. The roots, leaves and seeds of this plant

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http://dx.doi.org/10.1016/j.measurement.2015.02.004
0263-2241/

find a prominent place in Ayurveda and Unani medicines. The seeds of *Hygrophila Spinosa* T. Anders (HST) contain minerals like Ca, Mg, K, Fe, Cu, etc. [15]. Hitherto, no research work has been undertaken to explore the possible effects of the addition of mineral contents of *Hygrophila Spinosa* T. Anders onto SnO_2 toward gas or humidity sensitivity detection. Hence, the main intention of the present work is to study the variation shown in the trend of properties on addition of *Hygrophila Spinosa* T. Anders seeds into SnO_2. The prepared samples was extensively characterized by thermogravimetric analysis, X-ray diffraction, FT-IR spectroscopy, scanning electron microscopy, energy dispersive X-ray analysis, optical, photoconductivity, response and recovery properties.

2. Experimental details

2.1. Materials

Analytical grade $SnCl_2{\cdot}2H_2O$ and Poly Vinyl Alcohol (PVA) were purchased from SD fine chemicals, India. Deep brown colored *Hygrophila Spinosa* T. Anders seeds (HST) that were approximately 2 mm diameter in size were obtained from the local ayurvedic medical store located at Chennai, Tamil Nadu, India.

2.2. Biosynthesis of HST doped SnO_2

The weight ratios of $SnCl_2$: HST chosen were 1.0:0.0, 0.75:0.25, 0.5:0.5, 0.25:0.75 and 0.0:1.0 and were labeled as SH0, SH1, SH2, SH3 and HS respectively. Prior to preparation, the HST seeds were cleaned well to remove the dust, and subjected to dry-grinding. The ground HST of an appropriate weight was taken in a separate beaker and distilled water was added to it slowly under continuous stirring. During the continuous stirring, HST turned a gelly substance and settled at the bottom of the beaker. After settling, the excess water was drained out and the gel was washed several times in the distilled water until the water after wash was very clear. The after-wash water was again drained out completely. $SnCl_2{\cdot}2H_2O$ was dissolved in distilled water in a separate beaker and was added to that HST gel kept in another beaker and stirred thoroughly for about 5 h to form a uniform gelly mixture. This HST – $SnCl_2{\cdot}2H_2O$ gelly mixture was kept in hot air oven at 110 °C overnight, which resulted in a dark brown dry substance. It was later calcined at 600 °C for 5 h at the heating rate of 5 °C/min, in order to bring about a thermal decomposition, phase transition and removal of

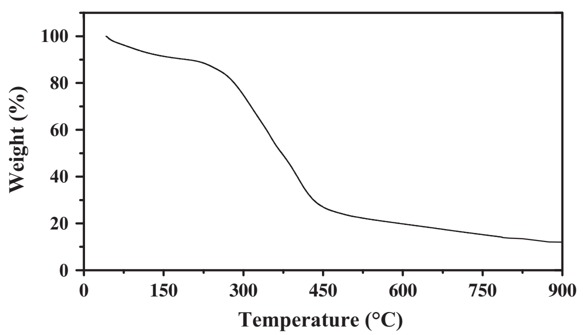

Fig. 1. Thermogravimetric analysis of *Hygrophila Spinosa* T. Anders (HST).

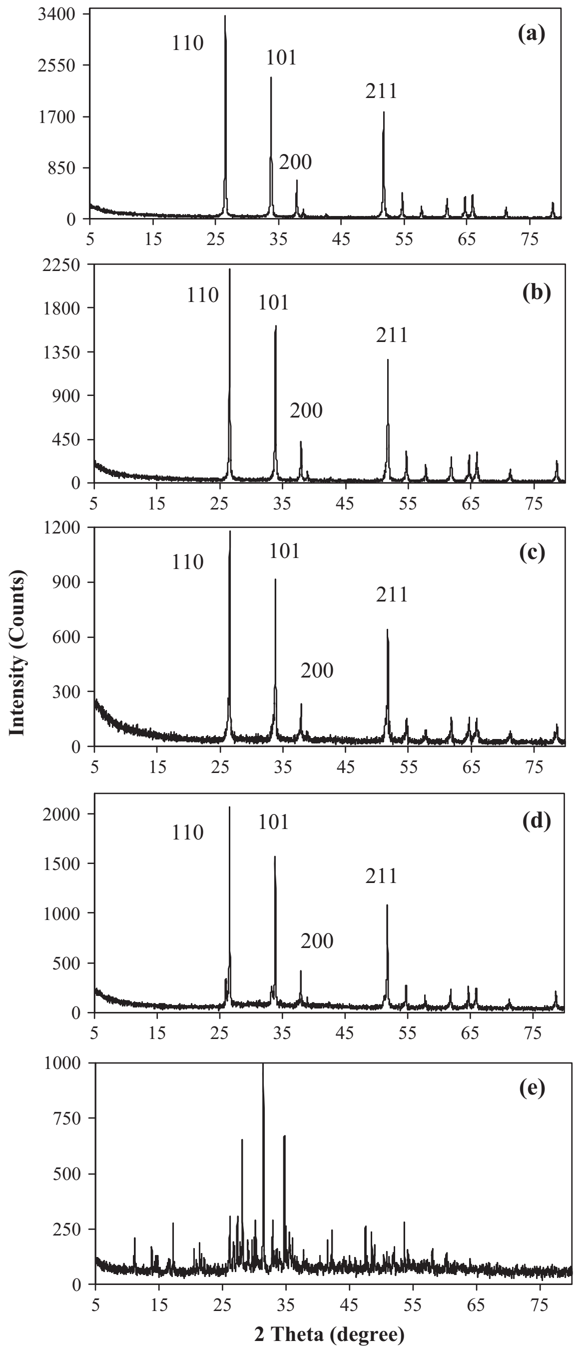

Fig. 2. XRD patterns of (a) SH0, (b) SH1, (c) SH2, (d) SH3, and (e) HS samples.