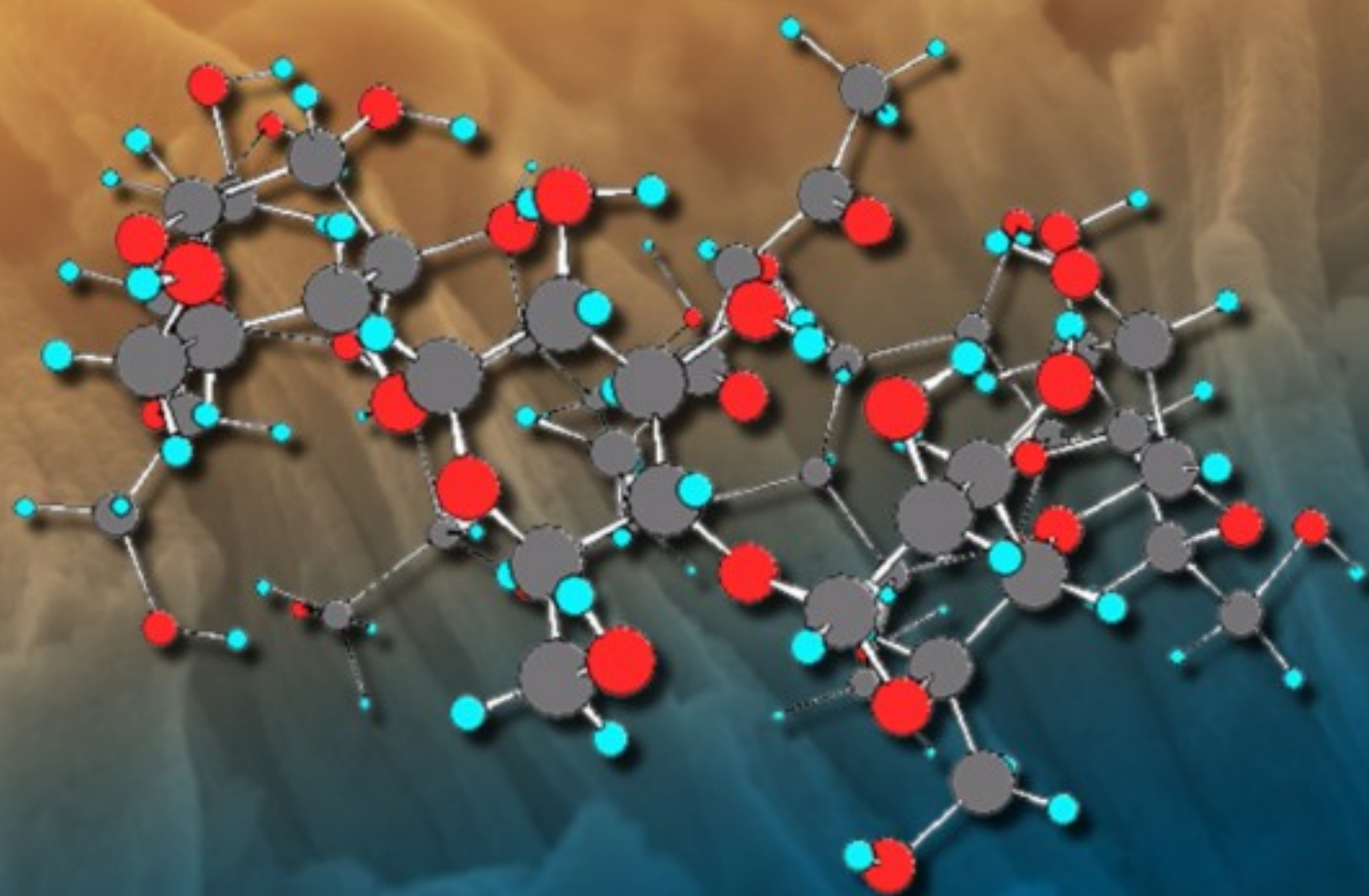


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In-Situ Decoration of Electrostatically Functionalized Multiwalled Carbon Nanotubes with β -Ni(OH)₂ Nanoparticles for Low Temperature NO₂ Detection

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Abstract: In situ β -Ni(OH)₂ nanoparticles (NPs) decorated electrostatically functionalized multiwalled carbon nanotubes (f-MWCNTs) composite was fabricated onto flexible substrates by thermal embedding technique for room temperature detection of NO₂ vapors. The MWCNTs were functionalized using electrostatic technique thus introducing hydroxyl functional groups on their surfaces (f-MWCNTs). These bonds were in-situ functionalized with Ni precursors using chemical reduction method hence forming β -Ni(OH)₂ nanoparticles functionalized MWCNTs. The decoration of f-MWCNTs with β -Ni(OH)₂ nanoparticles was confirmed by transmission electron microscopy (TEM), high resolution TEM, scanning electron microscopy (SEM) and X-ray diffraction techniques. The β -Ni(OH)₂ NPs were found to be an average size of 7-10 nm. Thermally embedded films of β -Ni(OH)₂ decorated f-MWCNTs achieved a 38 % change in sensitivity at 75 ppm when exposed to NO₂ vapors in a two-pole format at room temperature. Copyright © 2012 IFSA.

Keywords: Functionalized multiwalled carbon nanotubes, β -Ni(OH)₂ nanoparticles, Electrostatic functionalization, Room temperature NO₂ detection.

1. Introduction

Hybrid nanostructures of nanoparticles (NPs), uniformly distributed on the surfaces of multiwalled carbon nanotubes (MWCNTs) present a new class of nanomaterials that not only exhibit the unique

properties of MWCNTs and nanoparticles individually but also provide a platform to engineer a new class of materials according to the desired applications by taking the advantage of interactions between the nanoparticles and the MWCNTs. NPs decorated MWCNTs have been intensely researched for applications such as nanoelectronics for e.g. supercapacitors [1], chemical sensing [2], biosensing [3], photovoltaic application [4], fuel cells [5] and hydrogen storage. [6]. In general the binding between the NPs and MWCNTs surface can be either covalent or non-covalent. Covalent bonding is usually obtained via the functionalization of MWCNTs with carboxyl (-COOH) [7] or carbonyl (-C=O) through the acid treatment of the MWCNTs [8]. Non-covalent attachment has been achieved using electrostatic directed assembly [9]. However, due to the chemical stability and highly hydrophobic nature of MWCNTs, surface modification of MWCNTs is the first important step for developing high-performance NPs decorated composites and increasing their dispersion stability. Chemical functionalization, such as acid-treatment has been reported to ensure good physical and electrical contact between the tubes and the NPs. Although this process can increase the dispersion property, it can also have a detrimental effect on the conductivity of the composites because of the morphological damage.

Recently, we developed a novel technique to functionalize the surface of MWCNTs with hydroxyl (-OH) and carbonyl (-C=O) bonds on the surface of MWCNTs via electrostatic-force-directed assembly. In this technique, DC based high intensity electric fields near the surface of MWCNTs were generated to ionize atmospheric oxygen to highly reactive native oxygen which was adsorbed on the surface of MWCNTs and generated oxygenated functional bonds on the surface [10]. Nanoparticles decorated MWCNTs are further formed by the chemical reaction of these f-MWCNTs with the precursor salts and reducing agent.

Several NPs have been functionalized to MWCNTs for the applications in NO₂ sensing. Materials like Au, Pt nanoclusters doped MWCNTs, Pd nanoparticles doped MWCNTs etc. have been studied for this application. But, there are several disadvantages linked with this material like advanced equipments required, high temperature detection, costly dopant materials. However, β -Ni(OH)₂ NPs doped MWCNTs act as a strong candidate for NO₂ sensing. β -Ni(OH)₂ has highly stacked lamellar structure and nanosized β -Ni(OH)₂ has high electrochemical performance and room temperature detection strategy. Moreover, the percolation of these particles in the MWCNTs is much better than any other traditional carbon materials which make them a suitable nominee for NO₂ sensing.

In the present work, we report the synthesis of β -Ni(OH)₂ NPs decorated electrostatically functionalized MWCNTs (f-MWCNTs). An in-situ synthesis method of the nanoparticles through its precursors in the solvent was employed. The morphology of β -Ni(OH)₂ NPs decorated f-MWCNTs (β -Ni(OH)₂-f-MWCNTs) were characterized using EDX (Mapping), SEM, TEM and XRD. The decorated f-MWCNTs were self-assembled on parafilms using thermal embedding technique to fabricate flexible sensors. The β -Ni(OH)₂-f-MWCNTs films were tested for detection of ppm-level nitrogen-dioxide (NO₂) at room temperature. It was found that the conductance of the films increased after NO₂ exposure.

2. Experimental

2.1. Functionalization of MWCNTs

For the present work, MWCNTs were procured from Sigma Aldrich (outer diameter: 10–15 nm, inner diameter: 2–6 nm, length: 0.1–10 mm, purity: >90 % and density: 1.7–2.1 gm/cm³) and hydrated nickel nitrate (NiNO₃·6H₂O) crystals from Merck (M=290.81 g/mol, purity: > 97 %).

A new technique to functionalize the thin films of MWCNTs with oxygenated functional groups using electrostatic charging method, developed by Bhatia et al. [10], was implemented to functionalize the MWCNTs in this study. During the electrostatic functionalization of MWCNTs, due to generation of high intensity electric fields, atmospheric oxygen was ionized to highly reactive native oxygen which was adsorbed on the surface of MWCNTs and generated hydroxyl functional bonds on the surface. The functionalization of the films, f-MWCNTs was confirmed using Fourier transform infrared spectroscopy (FTIR).

2.2. Decoration of f-MWCNTs with Ni(OH)₂ Nanoparticles

A nanocomposite of Ni(OH)₂ NPs decorated f-MWCNTs was synthesized with the in-situ decoration of f-MWCNTs with Ni(OH)₂ nanoparticles using NiNO₃.6H₂O. f-MWCNTs and NiNO₃.6H₂O were taken in a ratio of 3:2 (w:w) in 10 ml dichloroethane and were sonicated for 5 minutes. A solution of 1 g KOH in 5 ml ethane was added drop-wise to the above sonicated solution. The solution was allowed to sonicate for another half an hour. A solution of 0.07 gm of sodium hydrogen borate (NaBH₄) in 10 ml cold ethanol was added drop-wise to the above solution and then finally allowed to sonicate for 7-8 hours in an ice bath to get a homogenous mixture.

2.3 Characterization of Ni(OH)₂ Decorated f-MWCNTs

The chemical analysis the f-MWCNTs decorated with Ni(OH)₂ nanoparticles was carried out by X-Ray Diffraction (XRD) and Fourier Transform Infra Red (FT-IR) techniques. The morphology of the nanocomposite was studied using Scanning Electron Microscopy (SEM)-Zeiss EVO40. The size of Ni(OH)₂ nanoparticles and their distribution on the surface of f-MWCNTs was analyzed using Transmission Electron Microscope (TEM)-JEOL 2100F and EDX (Mapping).

2.4. Thin Film Preparation of the Nanocomposite

The film of Ni(OH)₂-f-MWCNTs nanocomposite was fabricated by self-assembly of the nanocomposite by thermal embedding technique on a parafilm. 5 mg of Ni(OH)₂-f-MWCNTs nanocomposite was dispersed in 5 ml of ethanol and sonicated for several hours to provide a uniform suspension. The dispersed solution of nanocomposite was cast on cleaned glass surface and allowed to dry under ambient conditions for several hours. The cast film was then self-assembled on to the surface of non-conducting parafilm by thermal embedding technique while heating at oven temperatures for a few minutes to achieve a desired thickness of about 100 µm of the film. After self-assembly Ni(OH)₂-f-MWCNTs composite on the parafilms, thin strips of specified area were cut from the parafilm for using them as sensing materials with a specific surface area. Electronic grade silver paste was used for wire bonding with the strips. Fig. 1 provides a photographic illustration of the fabrication steps involved in fabricating Ni(OH)₂-f-MWCNTs film onto a parafilm.

2.5. NO₂ Gas Detection

The gas sensing properties of Ni(OH)₂ NP decorated f-MWCNTs film was performed at ambient temperature (25 °C) under 1 atmospheric pressure in a test chamber. The desired parts per million (ppm) levels of the gas were purged in the test chamber by the direct injection method using a gas tight chromatographic syringe. Specifically these ppm levels were kept around 75 ppm. The reproducibility of the results was confirmed by repeating each experiment several times. The on-line data acquisition was carried out with Keithley 6514 Electrometer interfaced with a personal computer as illustrated in

Fig. 2 for measuring the transient change in electrical resistance at the room temperature. The sample was brought to a saturation level in the ambient atmosphere in a closed chamber before the next set of detection measurements were performed.

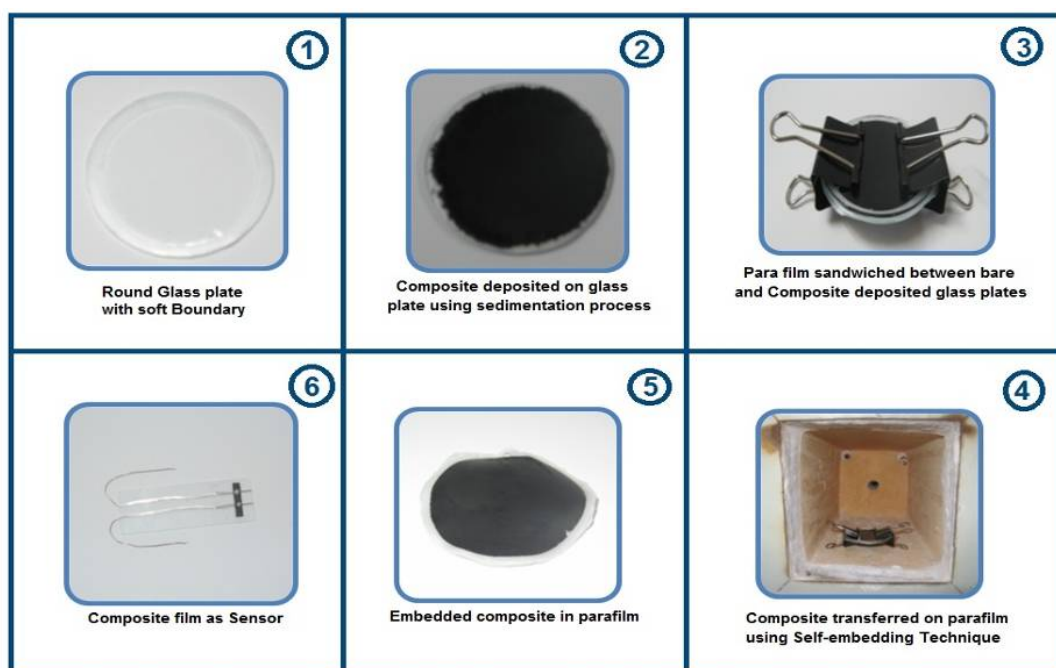


Fig. 1. Photographic illustration of steps involved in the preparation of $\text{Ni}(\text{OH})_2$ -f-MWCNTs nanocomposite films on parafilm using thermal embedding technique.



Fig. 2. Experimental setup for sensing NO_2 vapors at room temperature using Keithley Electrometer interfaced with computer.

The methodology applied in acquiring the data was as follows (identical for all the samples tested): The principle for detecting vapors was based on measuring change in the electrical resistivity of the films when exposed to vapors in the two-pole format under controlled ambient environments. Data acquisition started 5 min prior to purging the test chamber with the desired ppm level of the gas. After, the electrical resistance of the nanocomposite films reached its equilibrium value, in this case lowest resistance under the exposed amount of gas, the films were exposed to the ambient environment and the recovery of the film was then recorded under ambient conditions. After each run of experiment the chamber was flushed for 5 min by a jet of inert gas to remove any residual vapors of the gas. Fifteen

minutes later, a new amount of the same gas was injected, so that the second concentration could be tested based on the above mentioned procedure. Successive injections were repeated until all desired concentrations of the gas were measured. Finally, the films were left either overnight or 4-5 days at ambient temperature to ensure that the films had fully recovered before a new set of measurements could be started with the same films.

The recovery time of the sample was measured after the sample reached its saturation level of resistance and started to recover back to its initial resistance value (Recovery time) when the NO₂ gas effuses out of the chamber. The average percentage sensitivity, %S of the composites to NO₂ vapors was determined using the following equation as discussed in literature [10].

$$\%S = \left[\frac{(R - R_0)}{R_0} \right] \times 100, \quad (1)$$

where R_0 (Ω) is the initial resistance and R (Ω) is the minimum steady state response upon exposure to NO₂ vapors.

3. Results and Discussion

3.1. Characterization

The structural studies of the NP modified f-MWCNTs was carried out using CuK α radiation source of wavelength (λ) 1.54056 Å and the diffraction pattern was recorded for diffraction angle, 2θ , in the range 20-70° using PANalytical X'pert PRO and is shown in Fig 3 (a).

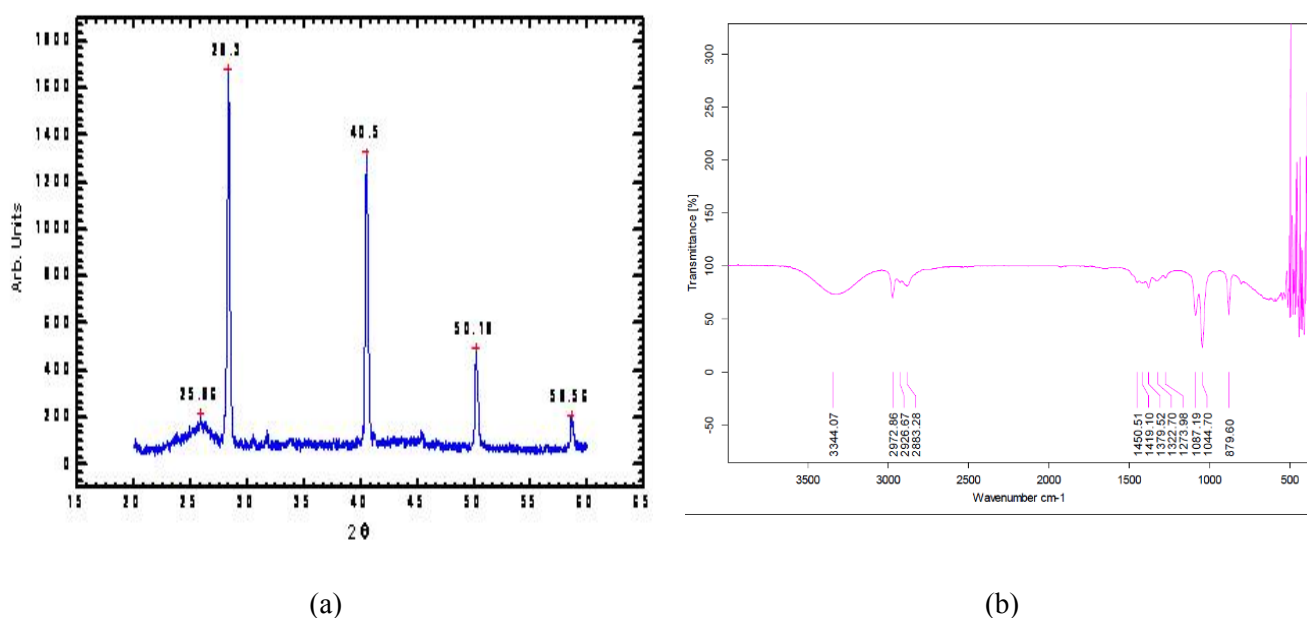


Fig. 3. a) XRD of f-MWCNTs decorated with β -Ni(OH)₂ nanoparticles, b) FTIR spectrum of β -Ni(OH)₂-f-MWCNTs.

The diffraction peaks present at 50.488° and 59.113° correspond to (102) and (110) planes of β -Ni(OH)₂ respectively (JCPDS File No. 14-0117). Peak at 25.8° belongs to (002) plane of MWCNTs. Thus, the XRD pattern suggested that there was an in-situ decoration of f-MWCNTs with β -Ni(OH)₂.

The theoretical calculations were conducted for the measurement of the crystallite size of β -Ni(OH)₂ using Scherrer formula which showed a crystallite size of approximately 10 nm at 40.5°

$$d = \frac{K\lambda}{(b \cdot \cos \theta)}, \quad (2)$$

where d represents the crystallite size, K is a constant, λ is the wavelength of the incident radiation, b is full width half maximum at a particular value of 2θ and θ is the diffraction angle [11].

An Insight into the chemical bonding was obtained by FTIR technique, performed using Bruker Alpha-E in the range 400 to 4000 cm⁻¹. The FTIR spectrum is shown in Fig. 3 (b). The peak at 879.60 indicates the presence of Ni-O-CO₂ vibration [12]. 1044.70 and 1087.19 vibrations are ascribed to broad C-O bonds [13]. The vibrational values at 2883.28 and 2972.86 denote the CH₃, CH₂ and C-H stretching [14], 3344.07 peaks correspond to the O-H linkage [15].

The surface morphology of the β -Ni(OH)₂ f-MWCNTs was studied by Field Emission Scanning electron Microscope. FESEM analysis of the sample was conducted using Zeiss EVO40 and is shown in Fig 4 (a). The SEM images showed uniform dispersion of β -Ni(OH)₂ nanoparticles on the surface of f-MWCNTs. Fig 4 (b) shows the EDX (Mapping) profile of the nanocomposite. The profile showed an even dispersion of Ni and Oxygen elements in the sample, confirming the presence of nickel nanoparticles with their hydroxide/oxide linkage presence with MWCNTs.

A bright-field TEM image using JEOL 2100F of nickel hydroxide particles attached with a high magnification is shown in Fig. 4 (c) and 4 (d). The images confirmed homogenous attachment of β -Ni(OH)₂ NPs on the surface of f-MWCNTs as well as impregnation inside the nanotubes. This observation was confirmed by the lumen size seen in the figure that was approximately same as the particle size seen in the picture. The size of β -Ni(OH)₂ particles varied from 7 to 10 nm. Most particles were quasi-spherical in shape and uniformly disperse on the surface of MWCNTs and inside the tubes. This proved that our decoration method was able to make the attached metal particles uniformly deposited onto MWCNTs.

3.2. Response of β -Ni(OH)₂-f-MWCNTs Films Towards NO₂ Gas

The response of β -Ni(OH)₂-f-MWCNTs nanocomposite sensor is shown in Fig. 5. The sample was placed in a closed chamber for detection of NO₂ gas. The sample at ambient conditions showed a resistance of 187 Ω . On purging 75 ppm of NO₂ gas in the chamber, the resistance value decreased to 115 Ω (approx.). Therefore, for 75 ppm of NO₂ gas the sensitivity is of about 38 %. The reduction in the resistance accounts to electron-rich nature of NO₂ gas which when purged on the sample, donates its electrons to functionalized β -Ni(OH)₂-f-MWCNTs which enhances the flow of current thereby reducing resistance of the composite.

For a sensing material, average response time, τ_{90} (sec) is the time in which the sensor response is enhanced by 90 % of its initial value in NO₂ vapor environment and average decay time, τ_{decay} (sec) is the time in which the sensors were able to retrieve back to 90 % of its initial resistance value. On exposure to 75 ppm of NO₂ vapors on a sample containing 60 % amount of β -Ni(OH)₂ by weight, a sensitivity of about 38 % was observed in the samples. The average τ_{90} of the sample was observed to be 65.48 sec and the average τ_{decay} of the sample was observed to be 2751.5 sec.

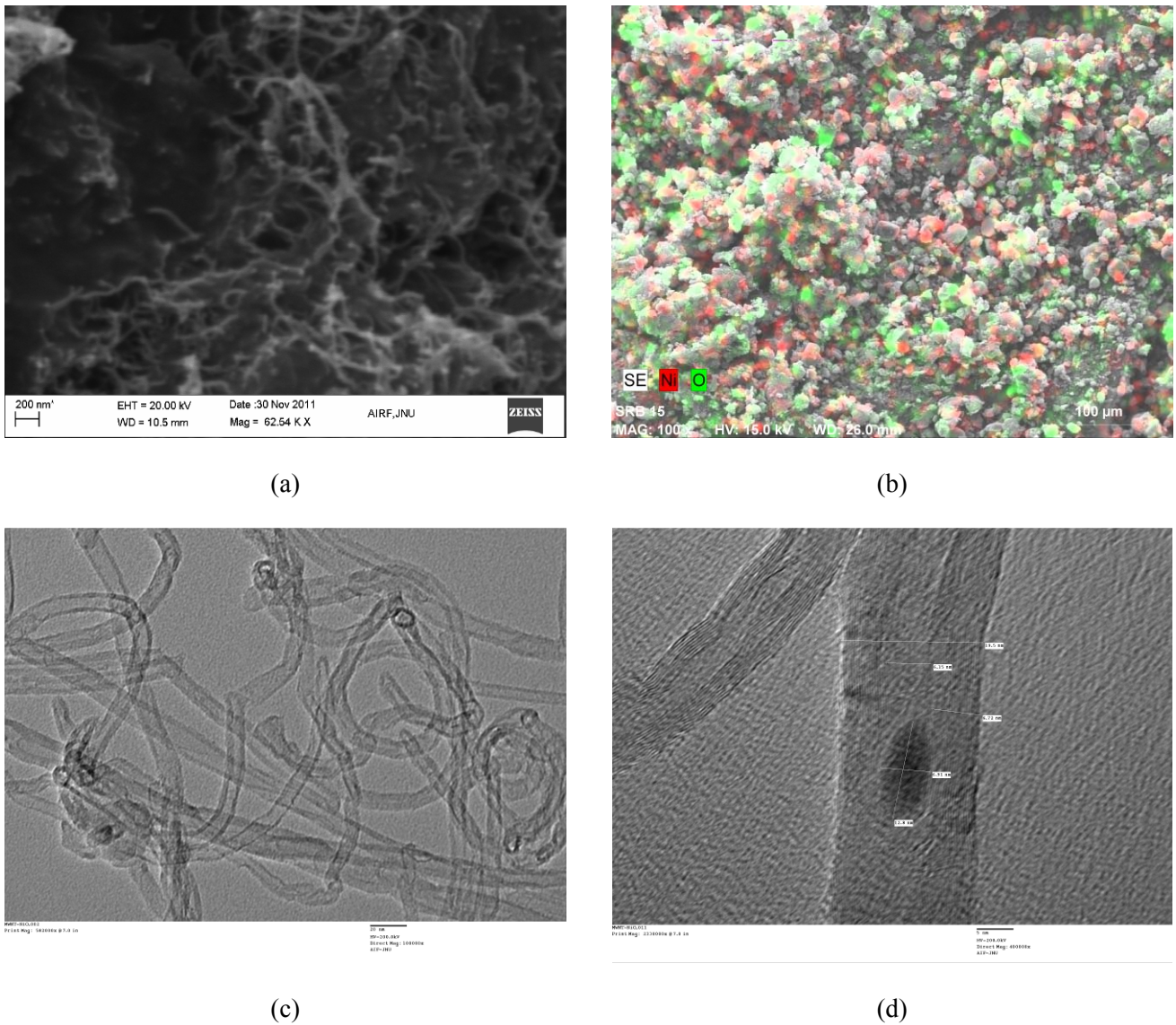


Fig. 4. a) FESEM image f-MWCNTs decorated with β -Ni(OH)₂ nanoparticles, b) EDX (Mapping) of Ni and O in the sample, c) TEM image of the f-MWCNTs showing decoration of β -Ni(OH)₂ nanoparticles on the walls of MWCNTs, d) TEM image of the f-MWCNTs showing insertion of β -Ni(OH)₂ nanoparticles inside the lumen of f-MWCNTs.

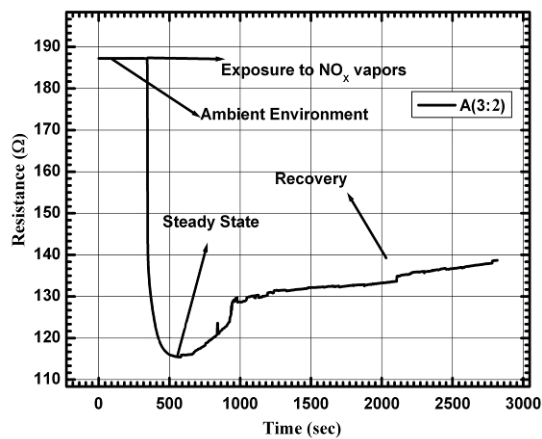


Fig. 5. Response curve of β -Ni(OH)₂-f-MWCNTs nanocomposite embedded film in flexible parafilm at 75 ppm level of NO₂ vapors.

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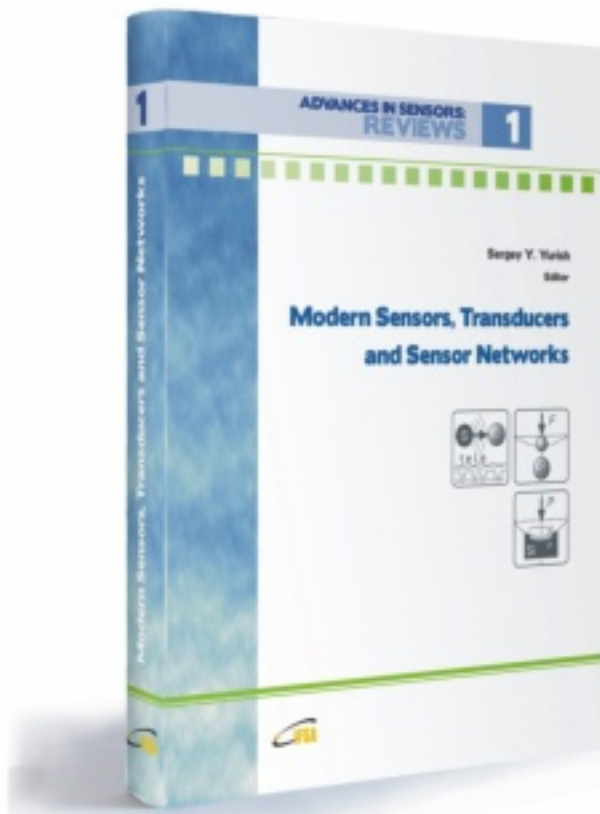
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