



Chemical sensing, thermal stability, electrochemistry and electrical conductivity of silver nanoparticles decorated and polypyrrole enwrapped boron nitride nanocomposite



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ABSTRACT

In the present work, a nanocomposite of boron nitride (BN) and silver nanoparticles (Ag) enwrapped by polypyrrole (PPy) has been synthesized for first time via *in situ* chemical oxidative polymerization of pyrrole using ferric chloride. The structures of synthesized FeCl₃ doped PPy, BN/Ag and PPy/Ag@BN nanocomposites were confirmed by fourier transform infrared spectroscopy, x-ray diffraction, thermogravimetric analysis, field emission scanning electron microscopy and transmittance electron microscopy technique. Our investigations showed that the electrical response of PPy/Ag@BN nanocomposite which contains a conductor (Ag), an insulator (BN) and a semiconductor (PPy), was greater than that of polypyrrole. The electrochemical supercapacitive performance shows that FeCl₃ doped PPy has higher capacitance than PPy/Ag@BN which might be due to higher conductivity of PPy as PPy/Ag@BN shows poor conductivity due to the insulating nature of BN. The newly synthesized nanocomposite showed rapid CO₂ sensing and significantly improved DC electrical conductivity.

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1. Introduction

A central goal in polymer synthesis is to directly convert simple chemical building blocks into useful materials. While a wide variety of interesting and potentially important structurally complex polymers have been discovered through recent research efforts, for example, biopolymers, advanced polymer networks, responsive materials and so on, their synthesis via traditional methods can sufficiently be complicated to limit their accessibility especially with the efficiency often demanded in polymer synthesis. One area where structure complexity has proven particularly significantly in the field of π -conjugated polymers. The development of polyheterocycles such as polypyrrole [1], polythiophene [2,3], and others [4–6] and their copolymers has revitalised how scientists create a host of organic electronics, such as semiconductors, photovoltaic devices or sensors [7–10].

Conducting polymer-based nanocomposites containing metallic or semiconducting nanoparticles provide exciting systems to investigate with the possibility of designing device functionality [11]. There are lot of studies on synthesis, characterization and

applications of nanocomposites with diverse combinations of conducting polymers such as polypyrrole, polyaniline, poly(3,4-ethylenedioxythiophene) and metallic/semiconducting nanoparticles [12–15]. The composites combining two conducting components, a metal and an organic semiconductor, are expected to exhibit a good level of electrical conductivity as well as tunable physical, chemical and responsive properties [16–19]. Among them, polypyrrole and polyaniline composites have most frequently been studied such as energy storage devices [20,21].

The physical properties of boron nitride (BN) are mostly governed by its atomic structure. BN is isoelectronic with graphite and therefore h-BN is also known as “white graphite.” h-BN consists of a layered structure comprising a network of (BN)₃ rings. Although, graphite has metallic conductivity, BN is an insulator due to the covalent interlayer bonding of the boron and nitrogen atoms which localizes the free electrons. This also explains the difference in colors of graphite (black) and BN (white) [22]. Recently, BN has attracted considerable attention for its several unique features such as hardness, high-temperature stability, high thermal conductivity, high melting point, high volume resistivity, high corrosion resistance, high dielectric breakdown strength, good resistance to oxidation, and its chemical inertness. It also has wide range of applications ranging from optical storage to medical treatment,

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photocatalysis, optoelectronic devices, coatings and electrical insulation [23,24].

In view of above mentioned properties, herein we describe our efforts towards the development of polypyrrole enwrapped BN/Ag with concentric core/shell structure. Compared with the corresponding single and two component samples, the three component PPy-BN-Ag system (one component i.e. PPy, two component i.e. BN/Ag and three component i.e. PPy/Ag@BN) exhibited enhanced electrical conductivity which may be due to fast charge carriers transfer rate. Moreover, thermal stability of electrical conductivity has increased impressively. To the best of our knowledge, this is the first attempt to induce such electrical conductivity in a non-conducting boron nitride with simultaneous enhancement in electrical conductivity via decoration of Ag nanoparticles enwrapped by polypyrrole. Therefore, BN modified, polypyrrole enwrapped materials may prove to be an exciting area of research as due to the synergism between the constituents the resulting PPy nanocomposite is expected to have unique properties such as enhanced DC electrical conductivity and electrochemical studies.

2. Experimental

2.1. Materials

Pyrrole 99% (Spectrochem, India), ferric chloride anhydrous (Fisher Scientific, India), boron nitride (MK Nano, Canada), silver nitrate (Sigma, USA), polyvinylpyrrolidone (Sigma-Aldrich), N,N-dimethylformamide (CDH, India) and methanol (E. Merck, India) were used as received. The water used in these experiments was double distilled.

2.2. Synthesis of PPy, BN/Ag and PPy/Ag@BN

FeCl₃ doped PPy was synthesised by oxidative polymerization method. To a solution of pyrrole (0.05 mol) in 100 mL distilled water was added drop wise a solution of ferric chloride (0.05 mol) in 100 mL distilled water. The reaction mixture was then stirred continuously for about 20 h resulting in the formation of black colored solid. The product thus formed was filtered and washed several times with distilled water, methanol and kept in an air oven at 80 °C for 6 h.

Boron Nitride and silver nitrate (1:1 M ratio) were put into 50 mL teflon-lined stainless steel autoclave containing 30 mL of N,N-dimethylformamide (DMF). 0.1 mmol PVP was then added into the above solution and the autoclave was kept at 160 °C for 8 h. The autoclave was then cooled to room temperature and product was isolated by centrifugation. The obtained product was washed with deionized water and absolute ethanol for three times and finally dried at 80 °C for 12 h. In this reaction, DMF was used as a solvent as well as reducing agent to reduce Ag⁺ to Ag. PVP was added as a stabilizer.

Pyrrole (0.05 mol) in 100 mL distilled water and BN/Ag (500 mg, previously sonicated for one hour) in 100 mL distilled water were mixed and stirred continuously for 10 min. A solution of ferric chloride (0.05 mol) in 100 mL distilled water was poured dropwise into the mixture. The reaction mixture was then stirred continuously for about 12 h resulting into the formation of black colored solid compound. The product PPy/Ag@BN was filtered and washed several times with distilled water and methanol and kept in an air oven at 80 °C for 6 h. For electrical conductivity measurements, 150 mg material from each sample was pelletized at room temperature with the help of a hydraulic pressure instrument at 80 kN pressure applied for 15 min.

3. Characterization

The morphology, structure and chemical composition of the FeCl₃ doped PPy, BN/Ag and PPy/Ag@BN were studied by a variety of techniques. The fourier transform infrared spectroscopy (FTIR) carried out using a Perkin-Elmer 1725 instrument. X-ray powder diffraction (XRD) data were recorded by Bruker D8 diffractometer with Cu K α radiation at 1.540 Å in the range of 5° ≤ 2 θ ≤ 70° at 40 kV. Thermogravimetric analysis (TG, DTA, DTG) was performed on the selected samples of the nanocomposites by Perkin Elmer (Pyris Diamond) instrument, heating the samples from ~24 °C to ~1000 °C at the rate of 10 °C min⁻¹ in nitrogen atmosphere at the flow rate of 200 mL min⁻¹. Field emission scanning electron microscopy (FESEM) and Energy dispersive x-ray analysis (EDX) carried out using a LEO 435-VF. Transmittance electron microscopy (TEM) was done using Technai G2 20 S-TWIN. PPy and PPy/Ag@BN were studied in terms of DC electrical conductivity retention by isothermal and cyclic ageing technique. A four-in-line probe with a temperature controller was used for thermal stability, PID-200 (Scientific Equipment, Roorkee, India) was used to measure the DC electrical conductivity measurements and its temperature dependence. The DC electrical conductivity was calculated by using the following equation:

$$\sigma = \frac{\left[\ln 2 \left(\frac{2S}{W} \right) \right]}{\left[2\pi S \left(\frac{V}{I} \right) \right]} \quad (1)$$

where I, V, W and S are the current (A), voltage (V), thickness of the pellet (cm) and probe spacing (cm) respectively and σ is the conductivity (Scm⁻¹) [25]. In isothermal stability testing, the pellets were heated at 50 °C, 70 °C, 90 °C, 110 °C and 130 °C in an air oven and the DC electrical conductivity was measured at an interval of 5 min in the accelerated ageing experiments. In the case of cyclic ageing technique, DC conductivity measurements were taken 5 times at an interval of about 80 min within the temperature range of 50–150 °C. For the electrochemical cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) studies, the methodology and details of sample preparation can be seen elsewhere [26]. For electrical conductivity and sensing measurements, 150 mg of each sample was pelletized at room temperature with the help of a hydraulic pressure instrument at 80 kN pressure applied for 15 min. The relative humidity was about 40% and room temperature was ~25 °C. All the measurements were performed in a laboratory fuming chamber.

4. Results and discussion

4.1. FTIR spectroscopic study

Fig. 1 shows the FT-IR spectra of as prepared FeCl₃ doped PPy, BN/Ag and PPy/Ag@BN. The absorption peak at 3424 cm⁻¹ was assigned to N–H stretching vibrations of PPy. The bands of characteristic stretching frequencies for pyrrole ring C=C, C=N and C–N were obtained at 1542, 1466, 1299 cm⁻¹ respectively. The band at 915 cm⁻¹ correspond to C=N⁺–C stretching and has been attributed to the polaron band characteristic of the doping, suggesting that the PPy support is in its oxidized state and contains positively charge entities (N⁺) [27]. The absorptions band obtained around 1371 cm⁻¹ may be attributed to BN stretching of BN/Ag [28]. In case of PPy/Ag@BN nanocomposite, the N–H, C=C, C=N and C–N stretching frequencies were slightly shifted and were present at 3414, 1535, 1456 and 1291 cm⁻¹ respectively. The characteristic

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