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Preparation, Characterization and Current Studies of Polypyrrole/Tantalum Pentoxide Composites

Chaluvaraju B V[1], Sangappa K Ganiger[2] and Murugendrappa M V[3]

[1]Department of Physics, Bangalore Institute of Technology, Bangalore-560004, Karnataka, India
[2] Department of Physics, Government Engineering College, Raichur, Karnataka, India
[3] Department of Physics, B.M.S. College of Engineering, Bangalore-560019, Karnataka, India

Abstract

In-situ polymerization of pyrrole (PPy) was carried out with tantalum pentoxide (Ta_2O_5) in the presence of oxidizing agent ammonium persulphate to synthesize polypyrrole/tantalum pentoxide (PPY/Ta_2O_5) composites by chemical oxidation method. The PPY/Ta_2O_5 composites were synthesized with various compositions viz., 10, 20, 30, 40 and 50 wt. % of Ta_2O_5 in pyrrole. The powder X-ray diffraction (XRD) spectrograph, suggests that they exhibit semi-crystalline behavior. The Fourier Transform Infra-Red Spectroscopy (FTIR) reveals the stretching frequencies are shifted towards higher frequency side. The surface morphologies of these composites using Scanning Electron Microscopy (SEM), show that Ta_2O_5 particles are embedded in PPy chain to form multiple phases. Current versus voltage at room temperature studies shown that the metallic behavior of conducting polypyrrole/tantalum pentoxide composites. Current versus temperature at constant voltage studies shown the dimensions of tantalum pentoxide particles in the matrix have a greater influence on the current value.

Keywords: Polypyrrole; Tantalum pentoxide; Current; Voltage; Temperature.

1. Introduction

One fundamental property which normally distinguishes polymers from metals is electrical conductivity. The value of electrical conductivity [1-2] for metals is very high and is generally of the order of 10^4 – 10^6 S cm^{-1}, while for polymers which are generally insulators this value does not exceed 10^{-14} S cm^{-1}. Though the low electrical conductivity of polymers has found its immense use in the manufacture of insulators and dielectric substances, the question of producing polymers which exhibit a conductivity similar to that of metals, has always engaged researchers. During the last two decades, the researchers, through the simple modification of ordinary organic conjugated polymers, have succeeded in preparing polymers with high electrical conductivity called electrically conducting polymers or synthetic metals [3-4].

Tantalum is a white solid that is insoluble in all solvents, but is attacked by strong base and hydrofluoric acid. Orthorhombic and hexagonal phases are known. Ta_2O_5 has a high refractive index, low absorption, inert material, which makes it useful for coatings [5-7].

Tantalum pentoxide has found a variety of uses in electronics due to its high band gap of 3.7 eV [8]. It is used to make capacitors in automotive electronics, cell phones and pagers, electronic circuitry, thin-film components and high-speed tools. In the 1990s, interest grew in the use of tantalum oxide as a high-k dielectric for DRAM capacitor applications [9]. It is used in on-chip MIM capacitors for RF CMOS integrated circuits. Tantalum pentoxide has been utilized in the fabrication of the glass of many photographic lenses due to its high index of refraction.

2. Experimental Details

2.1. Synthesis

The AR grade (Spectro Chem Pvt. Ltd.) pyrrole [10] was purified by distillation under reduced pressure. 0.3 M pyrrole solution was contained in a beaker which was placed in an ice tray mounted on a magnetic stirrer. 0.06 M [11] ammonium persulphate solution was continuously added drop-wise with the help of a burette to the above 0.3 M pyrrole solution. The reaction was allowed for 6 hours under continuous stirring by maintaining a temperature of 0°C to 5°C. The precipitated polypyrrole was filtered and dried in hot air oven and subsequently in a muffle furnace at 100 °C. The yield of the polypyrrole was 3.2 g (to be taken as 100 wt. %).

For 0.3 M pyrrole solution, 0.32 g (10 wt. %) of Ta_2O_5 was added and mixed thoroughly, further 0.06 M ammonium persulphate was continuously added drop-wise with the help of a burette to the above solution to get PPy/Ta_2O_5 10 wt. % composite. Similarly, for 20, 30, 40 and 50 wt. %, 0.64 g, 0.96 g, 1.28 g and 1.6 g of Ta_2O_5 (Sisco Research Lab Ltd.) powder [12] is taken and the above procedure is followed to get PPy/Ta_2O_5 composites.

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The pure PPy and PPy/ Ta_2O_5 powder was pressed in the form of pellets of 10 mm diameter using hydraulic press. The conducting silver paste was applied to the pellets of synthesized composites to act as electrodes.

2.2. Characterization

The X-ray diffraction patterns of PPy/Ta_2O_5 composites were recorded on X-ray Diffractometer (Bruker AXS D8 Advance) [11-14, 16-17] using Cu $k_\propto$ radiation (λ = 1.5418 Å) in the 2θ range 20°– 80°. The FTIR [11-14, 16-17] spectra of the PPy/Ta_2O_5 composites were recorded on IR Affinity-1 (Shimadzu, Japan) spectrometer in KBr medium at room temperature. The SEM [11-14, 17] images of PPy/Ta_2O_5 composites were investigated using Scanning Electron Microscope (Jeol 6390LV). The current values were noted down as varied values of voltage at room temperature and the current values were noted down as temperature values were decreasing from 200°C till 30°C.

3. Results and Discussion

3.1. FTIR Analysis

The FTIR spectra of pure PPy, PPy/Ta_2O_5 (20%) composite & Ta_2O_5 are shown in Figure 1. The characteristic stretching frequencies are observed at 1546.91 cm^{-1}, 1467.83 cm^{-1}, 1300.02 cm^{-1}, 1045.42 cm^{-1}, 966.34 cm^{-1}, 912.33 cm^{-1}, 792.74 cm^{-1}, 680.87 cm^{-1} and 617.22 cm^{-1} may be attributed due to the presence of C = N stretching, N – H bending deformation, C – N stretching and C – H bending deformation frequencies. In comparison with pure PPy, for PPy/Ta_2O_5 composites the stretching frequencies are shifted towards higher frequency side. This indicates that, there is homogeneous distribution of Ta_2O_5 particles in the polymeric chain due to the Van der Walls interaction between polymeric chain and Ta_2O_5 [13-14].

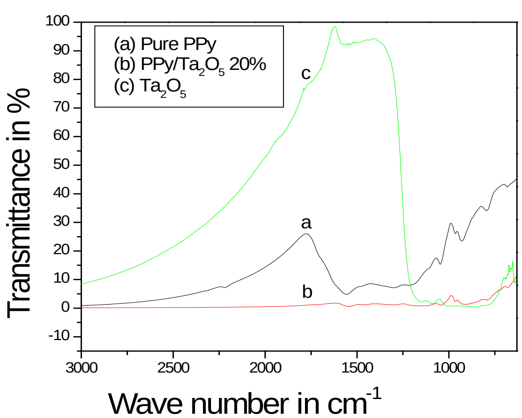

Figure 1 FTIR spectra of (a). pure PPy, (b). PPy/Ta_2O_5 (20 wt. %) composite and (c). Ta_2O_5

3.2. XRD Analysis

Figure 2.a represents the X-ray diffraction pattern of pure polypyrrole, which has a broad peak at about 2θ = 25°, shows a characteristic peak of amorphous polypyrrole. Figure 2.b represents XRD pattern of PPy/Ta_2O_5 (20 wt. %) composite. Characteristic peaks are indexed by lattice parameter values. Main peaks are observed with 2θ at 22.65°, 28.09°, 36.49°, 46.47°, 49.55°, 55.29°, 58.26°, 63.43° and 70.40° with respect to inter-planar spacing (d) 3.92 Å, 3.17 Å, 2.46 Å, 1.96 Å, 1.83 Å, 1.65 Å, 1.58 Å, 1.46 Å and 1.33 Å. Careful analysis of X-ray diffraction of PPy/Ta_2O_5 (20 wt. %) composite suggests that it exhibits semi-crystalline behavior. Figure 2.c presents XRD pattern of Ta_2O_5 revealing the partial crystalline nature [11-12, 14].

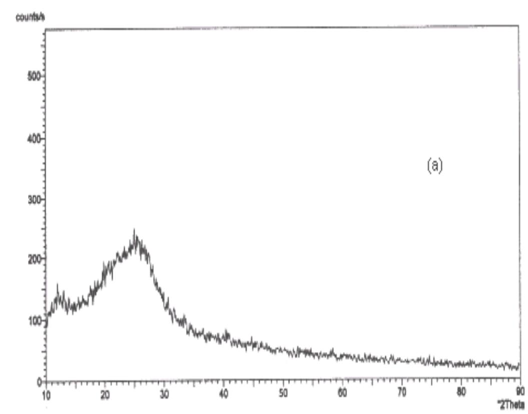

Figure 2.a X-Ray Diffraction pattern of pure PPy

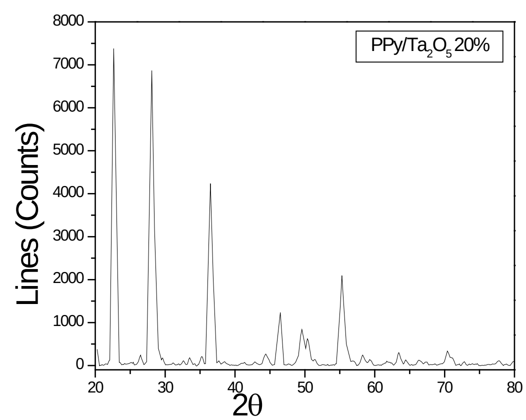

Figure 2.b X-Ray Diffraction pattern of

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PPy/Ta_2O_5 (20 wt. %) composite

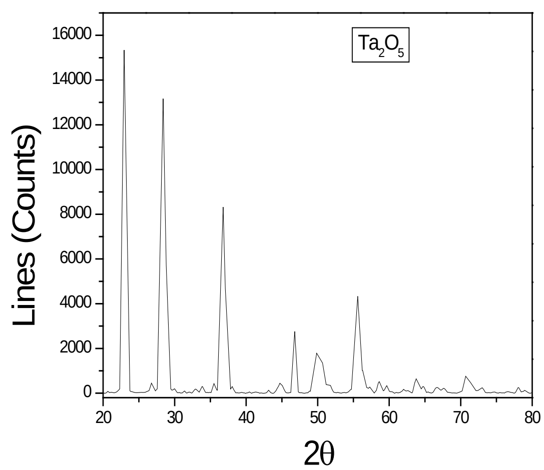

Figure 2.c X-Ray Diffraction pattern of Ta_2O_5

3.3. SEM Analysis

Figure 3.a, 3.b and 3.c show SEM micrographs of pure PPy, PPy/Ta_2O_5 (20 wt. %) composite and Ta_2O_5. It is seen clearly from the SEM micrograph of polypyrrole that, it has clusters of spherical shaped particles. The polypyrrole particles with elongated chain pattern are observed. The chemically polymerized polypyrrole samples prepared with polypyrrole powder have a much larger specific surface than the electrochemically polymerized film. A granular morphology of the polypyrrole particle structures is measured from SEM photographs and is found to be about 1 μm in diameter, which is consistent with other reports. A very high magnification of the SEM images show the presence of hemi spherical nature of polymer as clusters in the composite. Ta_2O_5 particles are embedded in PPy chain to form multiple phases, presumably because of weak inter-particle interactions [11-12, 14].

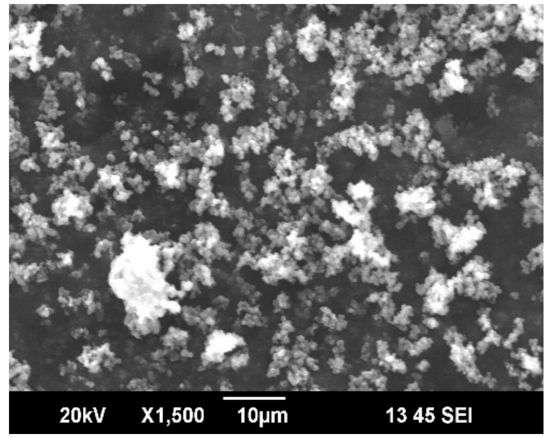

Figure 3.a SEM micrographs of pure PPy

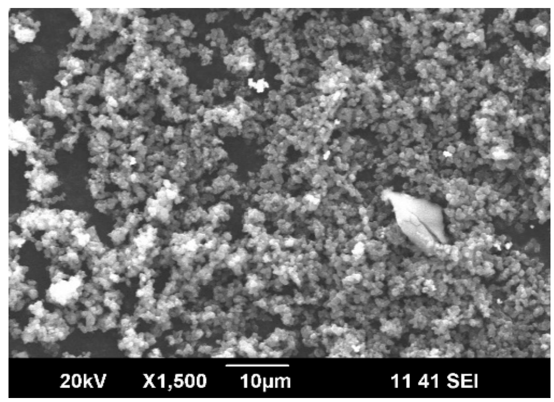

Figure 3.b SEM micrographs of PPy/Ta_2O_5 (20%) composite

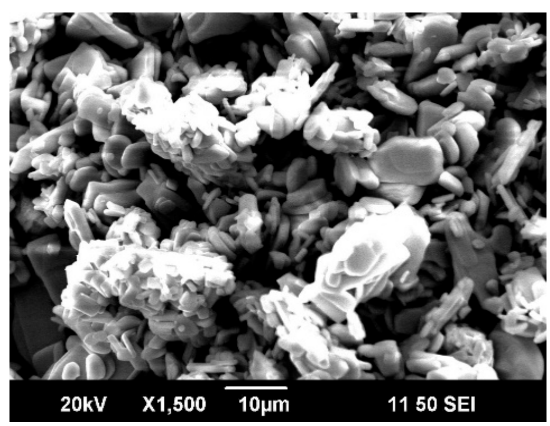

Figure 3.c SEM micrographs of Ta_2O_5

3.4. Current Studies

Current versus voltage at room temperature plot is as shown in Figure 4. As voltage increases current also increases showing the metallic behavior of PPy/Ta_2O_5 composites. At higher temperatures, the I-V characteristic for PPy/Ta_2O_5 composites develops a strong temperature dependence. Linearity of the I-V characteristic increases as temperature increases. The increase in current is due to the variation in distribution of Ta_2O_5 particles which may be supporting for more number of charge carriers to hop between favorable localized sites causing increase in current.

The plot of current versus temperature at constant voltage is shown in Figure 5. As temperature decreases from 200°C, current is decreases almost linearly till 150°C. After this temperature, current is almost constant till the room temperature. This shows that the dimensions of tantalum pentoxide particles in the matrix have a greater influence on the current value [18].

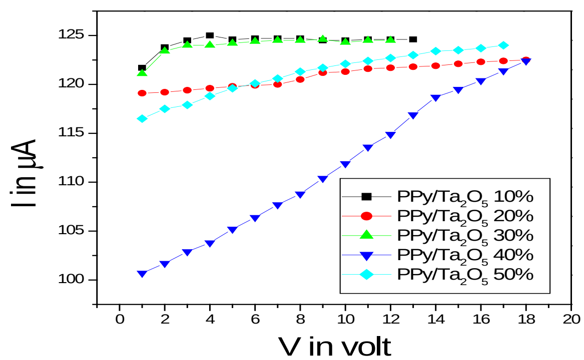

Figure 4 Current versus Voltage at Room Temperature

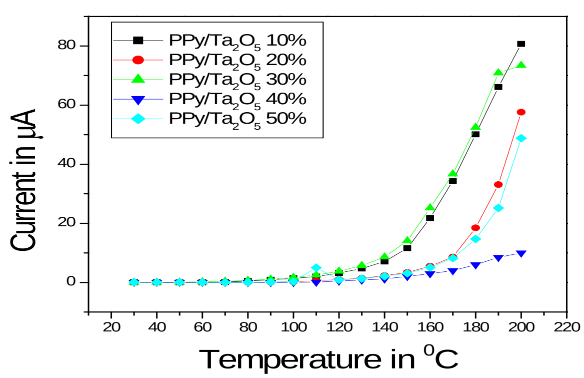

Figure 5 Current vs Temperature at constant voltage

4. Conclusions

Efforts have been made to synthesis the polypyrrole/tantalum pentoxide composites to tailor the current studies. Detailed characterizations of the composites were carried out using the SEM, XRD and FTIR techniques. As voltage increases current also increases showing the metallic behavior of conducting polypyrrole and its composites. As temperature decreases, current is also decreases. Current values were different for polypyrrole/tantalum pentoxide composites. This shown that the dimensions of tantalum pentoxide particles in the matrix have a greater influence on the current value. Polypyrrole/tantalum pentoxide composites may find applications in sensors.

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