

D.C. Conductivity and Humidity Sensing Studies of Polypyrrole/Sodium Metavanadate (Ceramics) Nano Composite

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ABSTRACT: In-situ polymerization of pyrrole was carried out by chemical oxidation method using sodium metavanadate (ceramics) in presence of ammonium persulphate an oxidizing agent to synthesize polypyrrole/sodium metavanadate. The polypyrrole/sodium metavanadate nano composite were synthesized with various compositions viz., 10, 20, 30, 40 and 50 weight percent of sodium metavanadate in polypyrrole. The surface morphologies of these nano composite were analyzed using Scanning Electron Microscopy which discovered the embedment of sodium metavanadate particles in polypyrrole chain. The Fourier Transform Infra-Red Spectroscopy revealed, the stretching frequencies were shifted towards higher frequency side. The powder X-ray diffraction patterns ascertained the exhibition of semi-crystalline behavior. Thermal analysis (TG/DTA) tests illustrated, that nano composite had greater stability than the pure polypyrrole. The temperature dependent D.C. conductivity disclosed that, the sodium metavanadate concentration in polypyrrole was responsible for the variation in the conductivity of the nano composite. The value of the conductivity increased for 40 weight percent of sodium metavanadate in polypyrrole. As percent relative humidity (RH) increases, the resistance of the nano composite decreases which stipulate, the usage of nano composite as humidity sensor.

KEYWORDS: Polypyrrole; Sodium Metavanadate; Nano composite; Conductivity; Sensor.

I. INTRODUCTION

The discovery of electrical conductivity [1] in molecular charge transfer has promoted the development of chemically synthesized conducting polymers [2]. Conducting polymers have found a wide range of applications ranging from rechargeable batteries to smart windows. They have replaced metals in several applications, by virtue of their light weight and greater ease of fabrication [3]. Polypyrrole (PPy) is one of the most studied electronically conducting polymers, which is chemically or electro-chemical synthesized [4]. PPy by virtue of its high electrical conductivity renders its application in variety of electronic devices like batteries, functional electrodes, electro-chromic devices, optical switching devices and sensors [5]. There is a substantial interest in the development of sensors for its application in monitoring and detecting humidity in moisture-sensitive environments, such as glove boxes and clean rooms, thus, humidity sensors have attracted tremendous attention to accomplish the same. Humidity measurement sensors now days have found the wide applications in people's day to day life, modern industry [2-3], agriculture [3-4], medicine [6-7] and scientific research [8-9]. Humidity sensors are subjected to the influence of properties like drift, accuracy, hysteresis or temperature. To overcome such influence new materials like metal oxides [10-11], ceramic [11-12] and polymers [13-14] have been designed as humidity sensors. Humidity sensors with porous ceramics or metal oxides are not commercially suitable, due to their large thickness or a complex integration process [10-12]. Polymer electrolytes have been identified to have better humidity-sensitive characteristics like reliability, ease of processing and low fabrication cost. Hence an attempt is made in this study to use the synthesized PPy as a composite for the humidity sensor and analyse its performance in sensing humidity

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II. MATERIALS AND METHODS

SYNTHESIS

The AR grade (Spectro Chem Pvt. Ltd.) pyrrole was purified by distillation under reduced pressure. 0.3 M pyrrole solution was placed in a beaker kept on ice tray mounted on a magnetic stirrer. To this beaker 0.06 M ammonium persulphate solution was continuously added drop-wise with the help of a burette. Maintaining the beaker temperature in the range of 0°C to 5°C for 5 hours, the reaction was allowed to take place under continuous stirring. The precipitated PPy was filtered from the beaker; filtrate was dried in a hot air oven at 100°C, which yielded 3.6 grams of PPy.

For such 0.3 M pyrrole solution, 0.36 grams (resulted from 10% weight of PPy) of sodium metavanadate (NaVO_3) was added and mixed thoroughly maintaining the same physical conditions as mentioned above. To this solution 0.06 M ammonium persulphate was continuously added drop wise with the help of a burette, from which PPy/ NaVO_3 (10%) nano composite was obtained. Similarly, the same procedure was adopted and 20% (by weight of PPy) mounted 0.72 gram, 30% (by weight of PPy) mounted 1.08 gram 40% (by weight of PPy) mounted 1.44 gram and 50% (by weight of PPy) mounted 1.8 gram of NaVO_3 (Sisco Research Lab Ltd.) was added for 0.3 M pyrrole solution which produced the PPy/ NaVO_3 nano composite powder. The pure PPy and PPy/ NaVO_3 nano composite powder were pressed in the form of pellets of 1 cm diameter using hydraulic press. D.C conductivity of this nano-composite was analysed by measuring the current values with respect to voltage from room temperature to 200°C.

CHARACTERIZATION

The surface morphology of pure PPy, PPy/ NaVO_3 (50%) nano composite and NaVO_3 were studied by Scanning Electron Microscope (SEM) images. The Fourier Transfer Infra Red (FTIR) spectra for the pure PPy, PPy/ NaVO_3 (50%) nano composite and NaVO_3 were recorded through the spectrometer in KBr medium at room temperature to study the characteristic stretching frequencies. The X-ray Diffractometer (XRD) patterns for the pure PPy, PPy/ NaVO_3 (weight 50%) nano composite and NaVO_3 were studied on using $\text{Cu } k_{\alpha}$ radiation ($\lambda = 1.5418 \text{ \AA}$) in the 2θ range 20°–80° to understand the characteristic peak of amorphous PPy. Thermal gravo-metric (TG)/Differential Thermal Analysis (DTA) studies were made in the temperature range from 40°C to 740°C at 10°C/min for the pure PPy, PPy/ NaVO_3 (50%) nano composite and NaVO_3 .

III. RESULT AND DISCUSSION

SEM ANALYSIS

Scanning Electron microscopic analysis was carried to understand the surface morphology of nano composite materials.

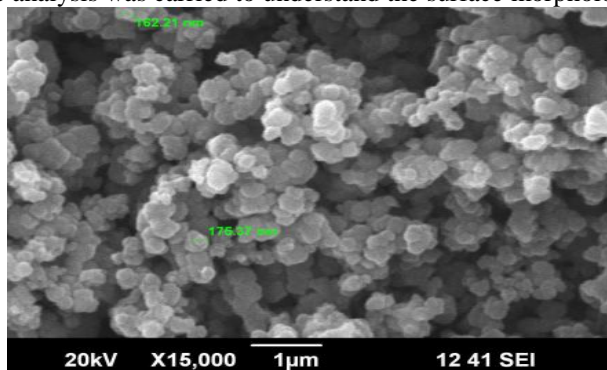


Figure 1.a SEM micrograph of pure PPy

Figure 1.a represents the size and spherical nature of the pure PPy. The elongated chain pattern of the polypyrrole particles was observed. Two particles sizes were measured as 162 nm and 176 nm.

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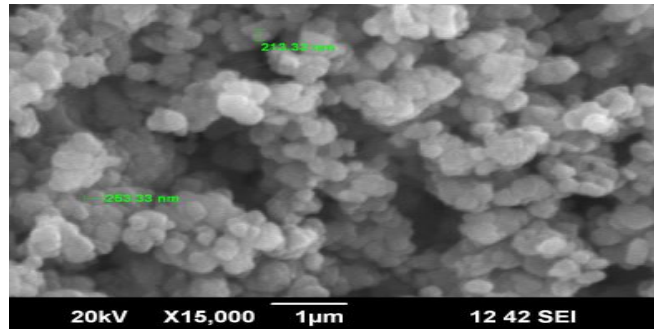


Figure 1.b SEM micrograph of PPy/NaVO₃ (50%) nano composite

Similarly micrograph of the PPy/NaVO₃ (50%) nano composite is represented in Figure 1.b. The particle size was increased to 213 nm and 253 nm due to uniform blending of NaVO₃ particles in PPy chain because of weak inter-particle interaction

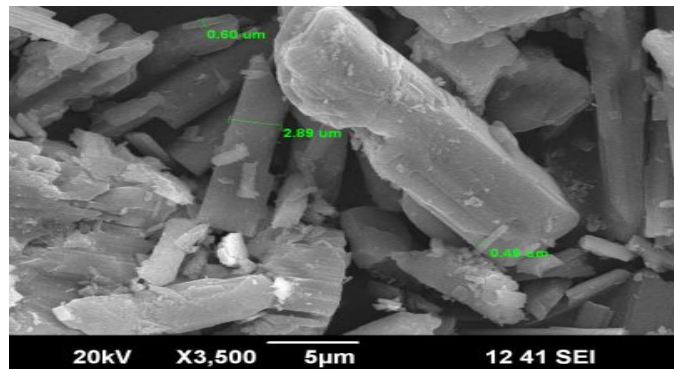


Figure 1.c SEM micrograph of NaVO₃

On the similar means Figure 1.c represents the particle size of NaVO₃, which has the immense size of the particles in comparison with pure PPy and PPy/NaVO₃ (50%) where in the particle size is measured in nm [6-12].

FTIR ANALYSIS

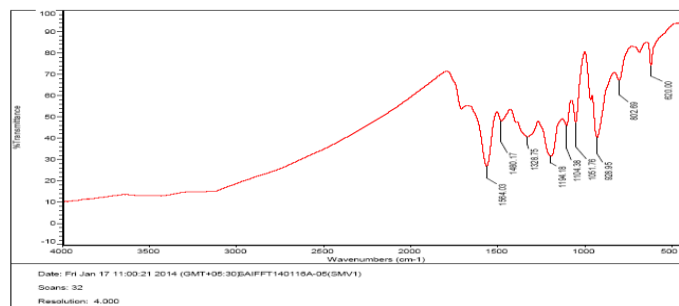


Figure 2 a. FTIR spectrum of pure PPy

The Fourier Transfer Infra Red (FTIR) spectra for pure PPy, PPy/NaVO₃ (50%) nano composite and NaVO₃ was studied. Figure 2.a shows FTIR spectrum of as prepared pure polypyrrole. The characteristic frequencies were observed at 1555 cm⁻¹, 1476 cm⁻¹, 1321 cm⁻¹, 1202 cm⁻¹, 1049 cm⁻¹, 927 cm⁻¹, 797 cm⁻¹, 619 cm⁻¹ for pure PPy.

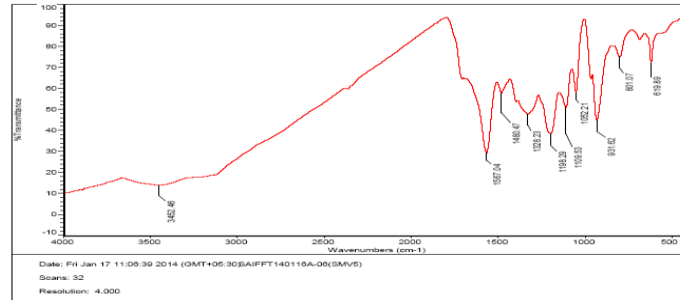


Figure 2 b. FTIR spectrum of PPy/NaVO₃ (50%) nano composite

Figure 2.b shows FTIR spectrum of NaVO₃ diapered polypyrrole nano-composite. The characteristic frequencies for PPy/NaVO₃ (50%) nano composite were observed at 1567 cm⁻¹, 1480 cm⁻¹, 1328 cm⁻¹, 1198 cm⁻¹, 1109 cm⁻¹, 1052 cm⁻¹, 931 cm⁻¹, 801 cm⁻¹ & 619 cm⁻¹ as seen.

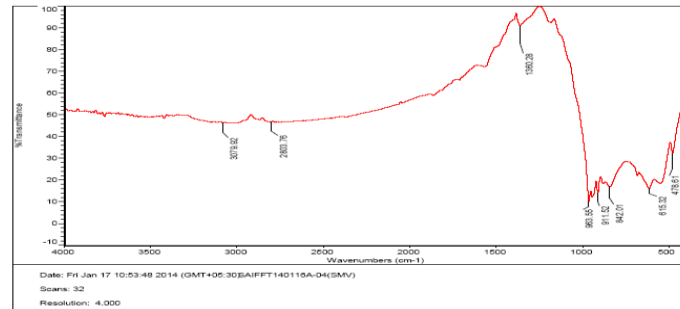


Figure 2 c. FTIR spectrum of NaVO₃

Similarly the characteristic frequencies for NaVO₃ were observed at 1518 cm⁻¹, 1360 cm⁻¹, 1049 cm⁻¹, 963 cm⁻¹, 911 cm⁻¹, 842 cm⁻¹ & 615 cm⁻¹ as seen in Figure 2.c. From this outcome it can be attributed that, the presence of C=N stretching, N-H bending deformation, C-N stretching and C-H bending deformation frequencies were shifted towards higher frequency side for the PPy/NaVO₃ (50%) nano composite. This indicates that, there is a homogeneous distribution of NaVO₃ particles in the polymeric chain due to the Van der Waals interaction between the PPy chain and NaVO₃ [6-7, 10-14].

XRD ANALYSIS

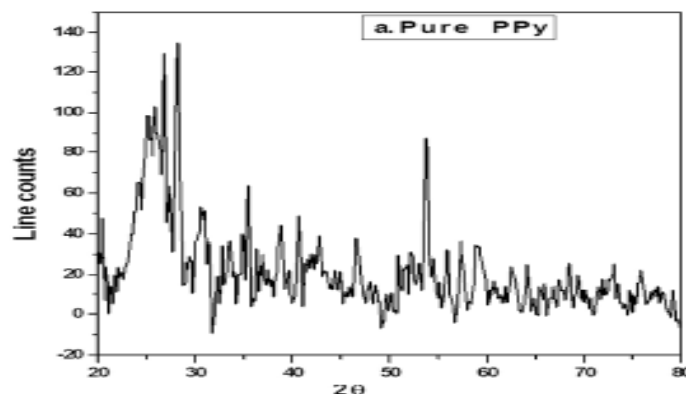


Figure 3 a. XRD pattern of pure PPy

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The X-ray Diffractometer (XRD) patterns for the pure PPy, PPy/NaVO₃ (weight 50%) nano composite and NaVO₃ were studied on using Cu K_α radiation ($\lambda = 1.5418 \text{ \AA}$) in the 2θ range $20^\circ - 80^\circ$ to understand the characteristic peak of amorphous PPy. Figure 3.a represents the XRD pattern of the pure PPy which has a broad peak at about $2\theta = 25^\circ$. The main peaks were observed with 2θ at $17.6^\circ, 22.53^\circ, 24.53^\circ, 25.72^\circ, 27.21^\circ, 28.18^\circ, 31.93^\circ, 33.9^\circ, 46.09^\circ, 48.78^\circ$ and 50.36° with the inter-planar spacing (d) $5.03 \text{ \AA}, 3.94 \text{ \AA}, 3.62 \text{ \AA}, 3.46 \text{ \AA}, 3.27 \text{ \AA}, 3.16 \text{ \AA}, 2.8 \text{ \AA}, 2.64 \text{ \AA}, 1.96 \text{ \AA}, 1.86 \text{ \AA}$ and 1.81 \AA respectively

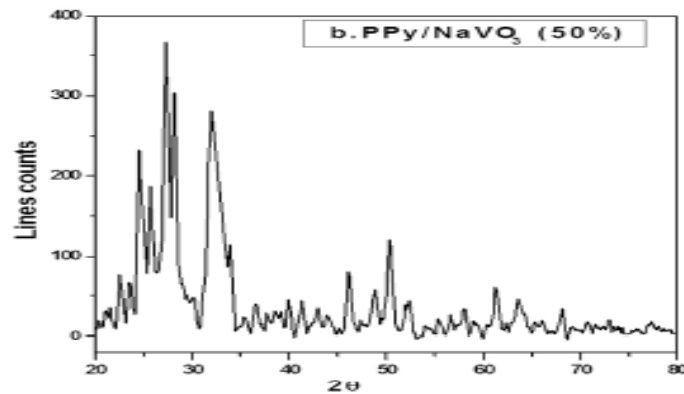


Figure 3 b. XRD pattern of PPy/NaVO₃ (50%) nano composite

Figure 3.b shows XRD pattern of NaVO₃ dispersed polypyrrole nanocomposite. On comparison of XRD pattern of NaVO₃, pure polypyrrole and PPy/NaVO₃ sample. The composite pattern shows peaks of the both NaVO₃ and polypyrrole samples. In addition to this shift of some peaks of both samples confirms of polymer composite.

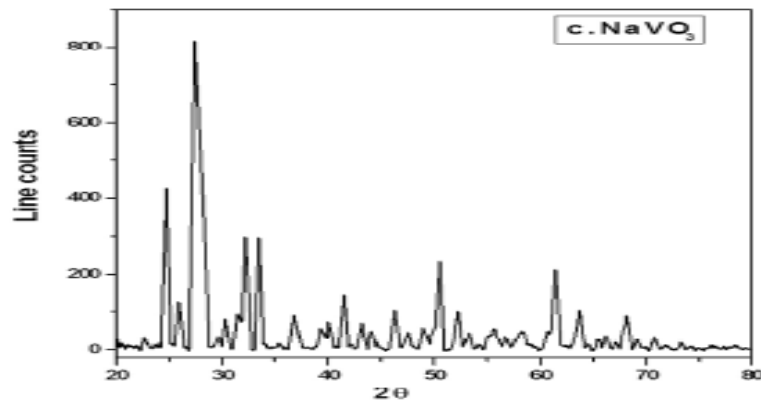


Figure 3 c. XRD pattern of NaVO₃

Figure 3.c shows XRD pattern of NaVO₃ samples. The pattern shows more Bragg's reflection due to crystalline nature of the samples. 2θ values of the samples are matches well with 2θ values of NaVO₃. This shows the confirmation of the sample formation.

TG/DTA ANALYSIS

Thermal gravo-metric (TG)/Differential Thermal Analysis (DTA) studies were made in the temperature range from 40°C to 740°C at $10^\circ\text{C}/\text{min}$ for the pure PPy, PPy/NaVO₃ (50%) nano composite and NaVO₃. Figures 4.a-4.c show the graphs of derivative weight (mg/min) Vs temperature for the pure PPy, PPy/NaVO₃ (weight 50%) nano composite and NaVO₃ respectively..

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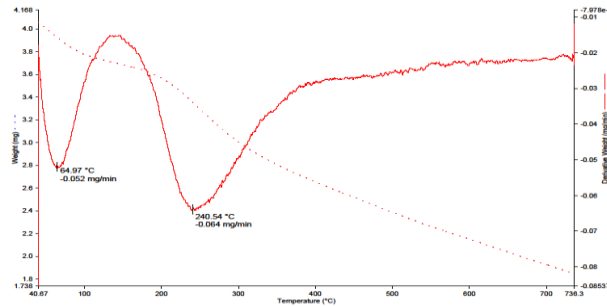


Figure 4 a. TG/DTA micrograph of pure PPy.

For the pure PPy, 0.052 mg/min is decomposed at 65°C mg/min and 0.064 mg/min is decomposed at 240°C with respect to total weight of the sample i.e. 4.057 mg.

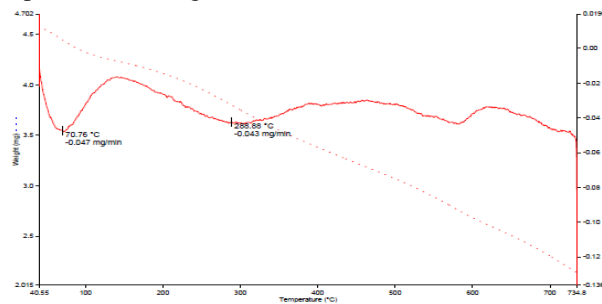


Figure 4 b. TG/DTA micrograph of PPy/NaVO₃ (50%) nano composite

For the PPy/NaVO₃ (weight 50%) nano composite, 0.047 mg/min is decomposed at 71°C mg/min and 0.043 mg/min is decomposed at 289°C with respect to total weight of the sample i.e.4.580 mg. It is found that at 740°C, the weight loss caused by the volatilization of the small molecules in the PPy/NaVO₃ (50%) nano composite at different temperatures is slow as compared to that of the pure PPy which is thus an indication of its higher stability. This clearly proves that insertion of NaVO₃ into PPy forms a nano composite with increased thermal stability [15-23].

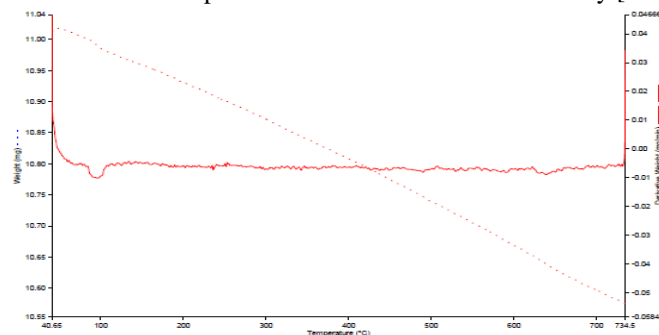


Figure 4.c. TG/DTA micrograph of NaVO₃

Thermal gravo-metric (TG)/Differential Thermal Analysis (DTA) studies were made in the temperature range from 40°C to 740°C at 10°C/min for the NaVO₃ as shown in Figure 4.c.

IV. D C CONDUCTIVITY STUDY

It is observed that, the conductivity increases with temperature. Figure 5.a and 5.b shows the multiple phases of conductivity and it is clearly depicted in the graph that, the value of conductivity increased for 40% weight of NaVO₃ in PPy. This can be attributed to the extended chain length of polypyrrole which facilitates the hopping of charge

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carriers when the content of NaVO_3 is increased. In the contrast the same response was not observed for 50% weight, since more number of charge carriers hoped between favorable localized sites at that weight. In all the nano composite, the conductivity increased with respect to the temperature when compared with the pure PPy only at 40% weight of NaVO_3 in PPy, which is thus the percolation threshold for this nano composite. The nano composite obey percolation theory, this can be attributed due to the distribution of NaVO_3 particles in PPy due to hopping of the charge carriers [24-30].

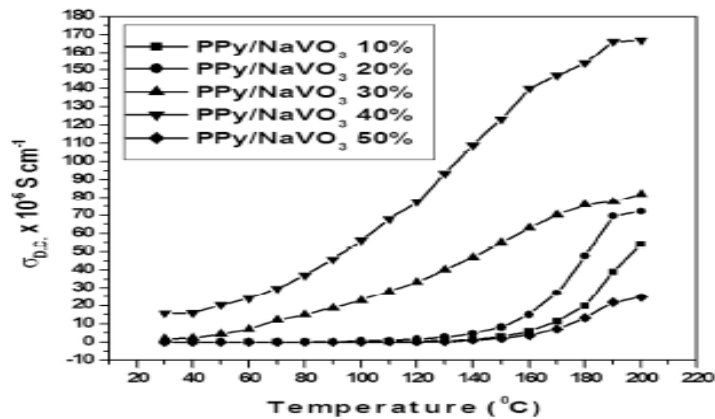


Fig. 5.a Variation of D.C. Conductivity as a function of temperature for the PPy/ NaVO_3 nano composite

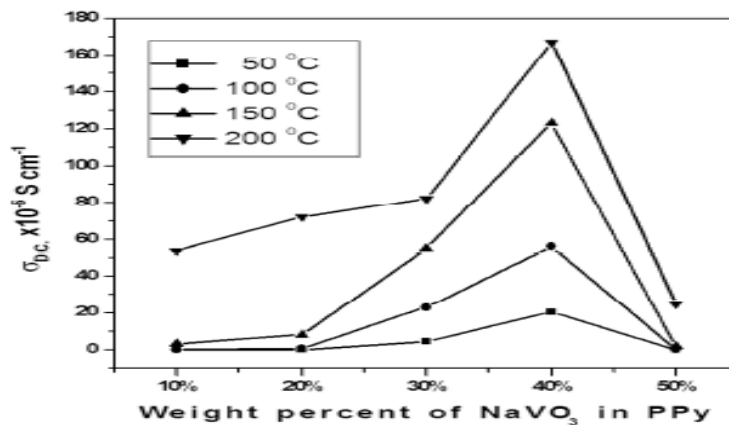


Fig. 5.b Variation of D.C. conductivity as a function of weight percent of NaVO_3 in pure PPy

V. HUMIDITY SENSING STUDY

Polymers have been identified to have better humidity-sensitive characteristics like reliability, ease of processing and low fabrication cost. The nano composite prepared in this study was analyzed for humidity sensing. Figure 6 shows that, the resistance decreases with increase in percentage relative humidity, this reveals, and the suitability of PPy/ NaVO_3 nano composite as an excellent humidity sensor. The decrease in the resistance with increasing humidity can be attributed to the mobility of the NaVO_3 ion which is loosely attached to the polymer chain by weak van der Waals forces. At low humidity, the mobility of the NaVO_3 ion is restricted; this can be attributed to the fact that under dry conditions, the polymer chains would tend to curl up into compact coil form. On the contrary, at high humidity, the polymer absorbs water molecules and the polymer chaining takes place, followed by the uncurling of the compact coil into straight chains that are aligned with respect to each other. This geometry of the polymer is favorable for enhanced mobility of the NaVO_3 ion [31-38].

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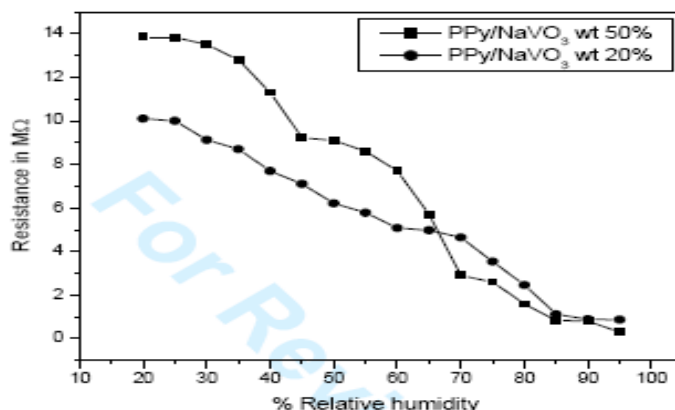


Fig. 6 Resistance as a function of humidity response for PPy/NaVO₃ nano composites

VI. CONCLUSION

The PPy/NaVO₃ nano composite were synthesized to tailor the transport properties. Detailed characterizations of the nano composite were carried out using SEM, FTIR, XRD and TG/DTA techniques. The results of D.C. conductivity of the PPy/NaVO₃ nano composite have shown a strong dependence on the weight percent of NaVO₃ in the pure PPy. The threshold value was found to be 40 % weight. PPy/NaVO₃ nano composite have shown that the resistance drops down with the increment in percentage relative humidity, thus making it a very good humidity sensor.

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