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Synthesis, Characterization and Electrical Studies of ZrV_2O_7/Polyaniline Nanocomposite

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***Abstract*:- Conducting polymers containing inorganic metal oxide materials integrates the materials properties and applications. Chemical oxidation polymerization method was adopted to prepare polyaniline /zirconium vanadate (ZrV_2O_7/PANI) nanocomposite. In situ polymerization reaction was performed for successful synthesis of PANI/ZrV_2O_7 nanocomposite. Structural characterization and morphology of the composite was carried out by X-ray diffraction (XRD) and scanning electron micrograph tools respectively. Particle size and bonding nature of the composite was studied by transmission electron micrograph (TEM) and Fourier transform infrared (FT-IR) tools. Variation of the electrical behavior of the composite sample was also studied.**

Key words: Chemical Oxidation, Nanocomposite, ZrV_2O_7, XRD, SEM, TEM, FT-IR

INTRODUCTION

Conducting polymers are attracted by recent researchers because of its good electrical conductivity, which finds many applications [1-2]. Polyaniline (PANI) is one of the noted conducting polymers and it can be synthesized by the oxidative polymerization of aniline monomer in the presence oxidizing agents [3-4]. Different oxidizing agents, like ammonium persulphate (APS) , hydrogen peroxide etc have been used in the synthetic procedures [5-6]. The formation of PANI composites with inorganic materials provides new synergistic properties that cannot be attained from individual materials [5-7]]. Thermal stability, mechanical strength [29], fire retardant properties and processability can be enhanced by the composite materials [8-10].

When materials like metal oxide nanoparticles combined with PANI matrix, resulting nanocomposites integrates the materials properties and applications. Recent research shows PANI composites has been successfully synthesizing with varied nanomaterials like metal oxides for its commercial applications [11-12].

The present work reports the chemical oxidation polymerization method for the preparation of PANI-ZrV2O7 nanocomposite. As prepared composite was well characterized by various characterization tools. Electrical properties like DC and AC studies of the composite were studied for its electrical behavior.

2. EXPERIMENTAL

2.2. Preparation of ZrV_2O_7-PANI nanocomposites

Zirconium vanadate nanomaterials are prepared as per the literature method [Ref]. ZrV_2O_7 nanocomposite is prepared as follows. 0.1 mol of aniline was dissolved in 1 M HCl to form aniline hydrochloride. Zirconium vanadate is added in the 50 weight percentage of aniline hydrochloride solution with vigorous stirring in order to keep the Zirconium Vanadate suspended in the solution. To this reaction mixture, 0.1 M of ammonium persulphate [$(NH_4)_2S_2O_8$] which acts as the oxidant was added slowly with continuous stirring for 4 – 6 hours at 0 – 5 ^{0} C. The precipitate powder recovered were vacuum filtered and washed with water and acetone. Finally the resultant precipitate was dried in an oven for 24 hours to achieve a constant weight. Lastly all the synthesized composite so obtained above is crushed into fine powder in an agate mortar in the presence of acetone medium. The synthetic scheme is given in figure 1.

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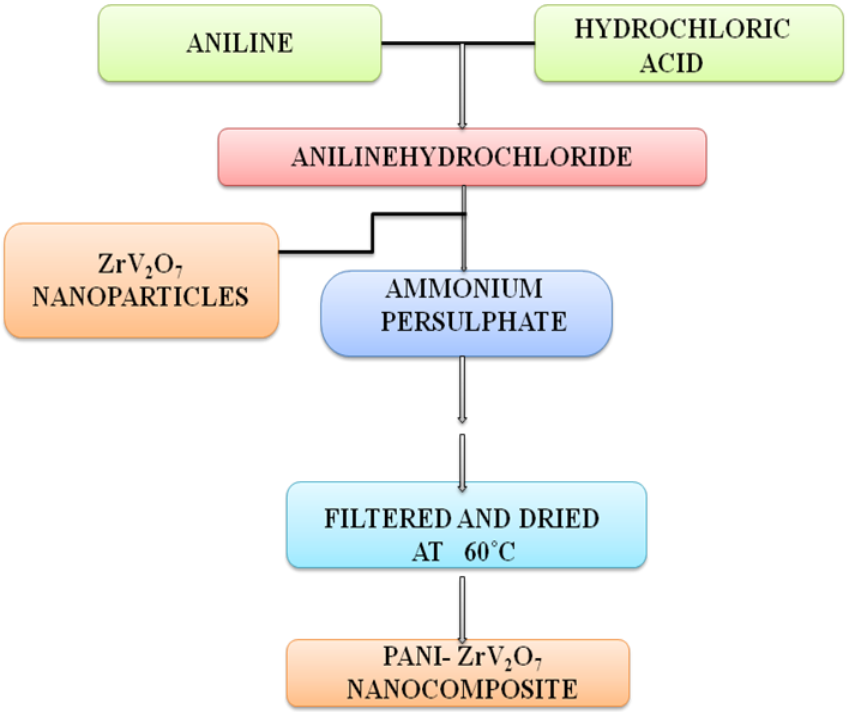

Figure 1: Synthetic scheme of Polyaniline – ZrV_2O_7 *nano*composite

Preparation of Pellets

The powders of polyaniline – ZrV_2O_7 nanocomposite so obtained is crushed and ground finely in the presence of acetone medium in agate mortar. This powder is pressed to form pellets of 10 mm diameter and thickness which varies from 1 to 2 mm by applying pressure of 90 MPa in a hydraulic press.

2.3. Characterization

Transmission electron microscopy (TEM) was carried out with Hitachi 7100 and TECH NAI G2 transmission electron microscopes operating respectively at an acceleration voltage of 75 kV and 200 kV. Scanning electron microscopy (SEM) images were obtained with a LEO 1530 microscope operating at an acceleration voltage of 5.0 kV. Fourier transform infrared (FT-IR) spectra were recorded on a Perklinalimer spectrometer. X-ray diffraction measurement was performed with a Philiphs Pro-expert Plus diffractometer.

3. RESULTS AND DISCUSSION

3.1 XRD Analysis

X-ray diffraction pattern of Polyaniline – ZrV_2O_7 composite with 50 wt% of ZrV_2O_7 in polyaniline is shown in figure 2. It is seen that the peaks of zirconium vanadate indicates the crystalline nature of the composite. Most of the Bragg,s reflections observed in the pattern are due to the presence of dispersed zirconium vanadate nanopaticles. The obtained d-spacing values are matches well with standard JCPDS file JCPDS No. 01-088 -0587. The peaks obtained for composite materials are also on the same 2θ values of the zirconium vanadate.

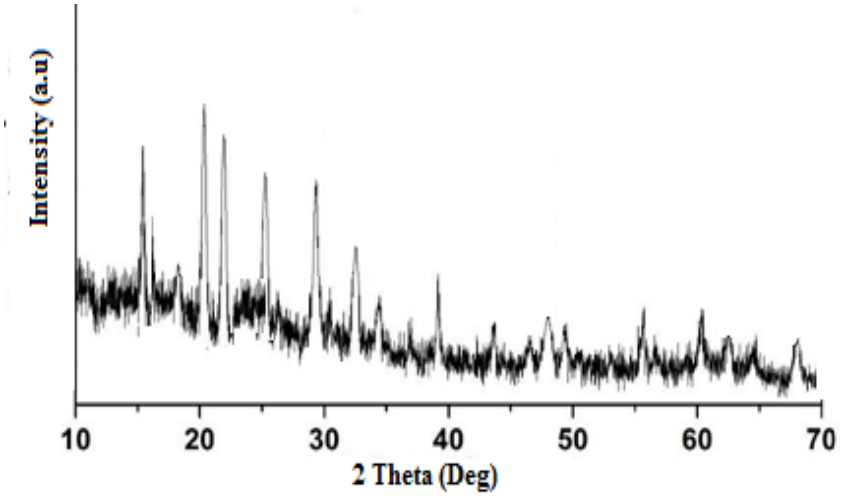

Figure 2: XRD pattern of PANI-ZrV_2O_7 nanocomposite

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3.2 FT-IR ***Analysis***

The polymerization of aniline to its polyaniline with zirconium vanadate nanocomposite was well confirmed by Infrared study. Figure 3 shows FT-IR of as prepared PANI-ZrV2O7 nanocomposite sample. The bands at 1563 and 1481 cm^{-1} are attributed to C=N and C=C stretching mode of vibration for the quinonoid and benzenoid units of PANI. The peaks at 1300 and 1236 cm^{-1} are assigned to C– N stretching mode of benzenoid ring. The peak at 1239 cm^{-1} is the characteristic of the conducting prorogated form of PANI. The bands in the region 1000–1110 cm^{-1} are due to in plane bending vibration of C– H mode. The band at 820 cm^{-1} originates out of plane C– H bending vibration. Peaks below 1000 cm^{-1} are attributed due to the presence of metal oxide complexation in PANI.

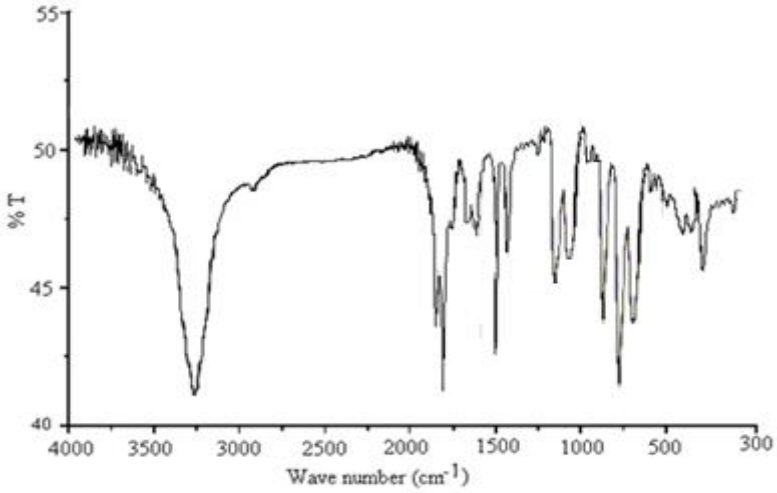

Figure 3: FT-IR spectrum of PANI-ZrV2O7 nanocomposite

3.3 Scanning Electron Microscopy (SEM) Analysis

Figure 4 shows the SEM image of the as prepared PANI-ZrV2O7 nanocomposite. It is observed from the SEM images that, the distribution of metal oxide particles are agglomerated due to the formation of matrix mixing or close complexation. Most of the particles are in spherical shape and are complexed with polymer forms by matriomixing, which enhances the crystalline nature of the PANI.

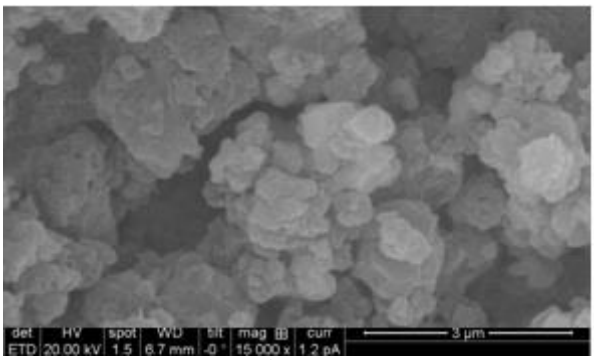

Figure 4: SEM image of PANI-ZrV_2O_7 nanocomposite

3.4 Transmission Electron Microscopy (TEM) Analysis

Figure 5 shows the typical bright filed TEM image of the as synthesized PANI-ZrV2O7 nanocomposite. It can be seen from the image that, the oxide particle are well dispersed and forms polydispersed polymer matrix. Varied particle sizes are observed and most of them are spherical and hexagonal in shape and are in the nano range.

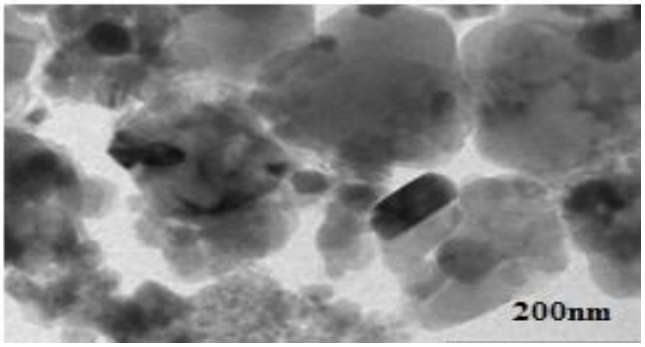

Figure 5: TEM image of PANI-ZrV_2O_7 nanocomposite

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4. ELECTRICAL CONDUCTIVITY STUDY

4.1 DC Conductivity Analysis

Figure 6 shows the variation of DC conductivity as a function of temperature of as prepared PANI-ZrV_2O_7 nanocomposite. The conductivity increases with increase in temperature. The dc conductivity of composite exhibits an exponential behavior in a temperature range 80^0C to 140^0C. Initially the conductivity values are almost constant up to 80^OC and then increases slowly due to behavior of disorder in semi conductor.

The analysis of temperature dependent conductivity data suggest that the charge transport mechanism in PANI can be explained by the variable range hopping (VRH) model.

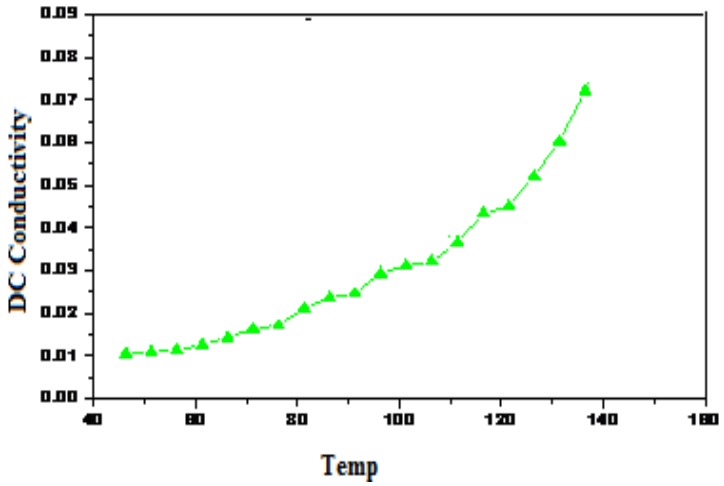

Figure 6: DC conductivity trace of PANI-ZrV_2O_7 nanocomposite.

4.2 AC Conductivity Analysis

Figure 7 shows the variation of ac conductivity as a function of frequency of PANI-ZrV_2O_7 nanocomposite. The conductivity increases with increase in frequency is observed in the figure. The ac conductivity of composite shows almost constant conductivity in the frequency range 10^2 Hz to 10^4 Hz. After this, the conductivity increase with increase in the frequency especially in the 10^5 – 10^6 Hz range. Lattice polarization around a charge in localized state may be responsible for multiple phases of conductivity in the composite

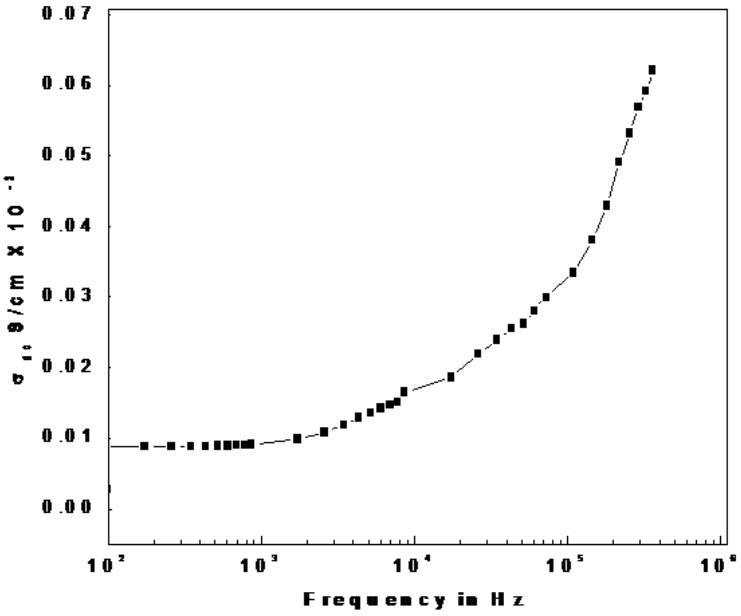

Figure 7: AC conductivity trace of PANI-ZrV_2O_7 nanocomposite

5. CONCLUSIONS

Chemical oxidation polymerization method was used for the successful preparation of metal oxide/polyaniline nanocomposite. The enhanced crystalline nature of the composite is observed due to insertion of inorganic metal oxide materials. Both dc and ac conductivities carried over the composite shows the presence of polarons as charge carriers and confirm the extended chain length of polyaniline.

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