IMPEDANCE SPECTROSCOPY BEHAVIORS OF CONDUCTING PANI - SrO NANO COMPOSITES

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Abstract:

The conducting Polyaniline/ Strontium Oxide (PANI-SrO) nanocomposites were synthesized by in-situ polymerization method with 10,20,30,40 and 50 wt.% of SrO in polyaniline. The nanocomposites thus formed were characterized by X-ray diffraction (XRD), Scanning Electron Microscope (SEM) and EDAX. The variation of impedance as a function of applied frequency suggesting nearly Debye-type relaxation mechanism take place. The results obtained for these nanocomposites are of scientific and technological interest.

Keywords: *Polyaniline, Strontium oxide, XRD, SEM, Dielectric, Nanocomposites*

1. Introduction:

The conducting polymer/inorganic nanocomposites having unique physical properties that have attracted more and more attention. Conductive polyaniline (PANI) has been studied extensively because of its ease synthesis, environmental stability, electrical and other properties [1-5]. SrO has been attracted much attention as cathodes in rechargeable battery, selective gas sensors such as H_2S, ammonia because of their high surface area and redox activity. One of the important aspects of the SrO is its layered lamellar structure. Many studies have been conducted to form PANI/SrO composites structurers controlling internal morphology has still remained a challenge. In this paper, authors report synthesis, characterization, AC conductivity, dielectric constant, dielectric loss and impedance spectroscopy of polyamine /SrO nanocomposites [6-11].

2. Experimental Methods:

2.1 Synthesis of Strontium Oxide Nanoparticles and PANI-SrO nanocomposites:

The strontium oxide nanoparticles were synthesized by self – propagating low temperature combustion method, employing strontium oxalate as precursor. The precursor is prepared by dissolving equimolar quantity of ammonium persulphate and oxalic acid in distilled water. This solution monomer aniline was distilled twice before use. Analytical reagent Grade Ammonium Persulfate, Hydrochloric acid (HCL) and Strontium Oxide (SrO) were used for synthesis. The PANI-SrO composites were synthesized by in-situ polymerization. Aniline solution was formed by dissolving aniline (0.1 mol) in 1M HCl. Strontium Oxide was added to aniline solution with vigorous stirring to keep Strontium Oxide suspended in solution. 0.1 M Ammonium persulfate, which acts as the oxidant, was added to this reaction mixture slowly with continuous stirring at 0-5°C, The reaction mixture was kept stirring for 4 to 6 hours. The polyaniline in the form of greenish precipitate was recovered by vacuum filtration and washed with deionized water. To achieve a constant weight, the precipitate was dried for 12 hours in hot air oven at 60°C. In this way polyaniline –Strontium Oxide nanocomposites with 5 different weight percentage of SrO (10,20,30,40, and 50) in polyaniline were synthesized.

3. Characterization:

3.1 X-ray Diffraction (XRD):

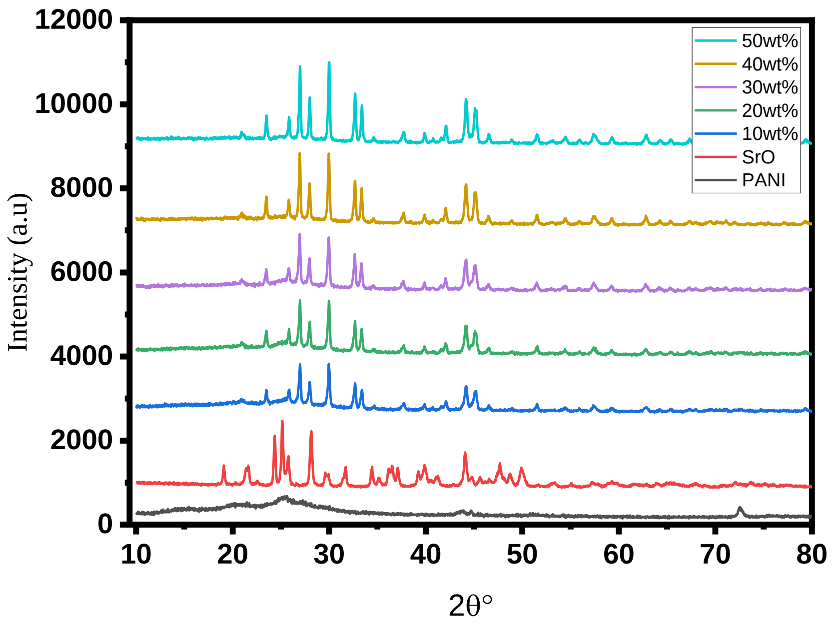


Figure: X-ray diffraction pattern of Pure Polyaniline, SrO and various weight % of polyaniline-SrO nanocomposites.

The Figure. shows X-ray diffraction pattern of Polyaniline. which is amorphous in nature with a board peak centered on 2θ= 25.53 ° which corresponds to (200) diffraction planes of pure PANI and Figure 1(b) shows X-ray Diffraction pattern of PANI-SrO nanocomposites with 50wt.% of SrO in polyaniline. These nanoparticles have shown good crystallinity because of existence of sharp peaks in XRD pattern. The Crystallite size of the synthesized PANI/SrO nanoparticles was calculated using Scherer's formula given by D= 0.9λ/βCosθ where D is the average crystallite size, λ is the wavelength of X-ray (1.5405A) and β is the full width half maximum in radian. The average crystallite size was found to 45nm. In the XRD pattern, different lines attributed to the (100), (111), (200),(002), (201), (012), (112) and (202) planes are good agreement with data of PANI/SrO powder file (ICDD No.00-006-0520) which corresponds to orthorhombic crystalline structure.

Figure1(c) shows XRD pattern of polyaniline-SrO composites of all samples.

By comparing the XRD patterns of composites and SrO, it is confirmed that SrO retains its structure even through it is dispersed in PANI during the polymerization reaction. This indicates that PANI undergoes interfacial interactions with SrO crystallites and presence of SrO and PANI matrix strongly affects the structural behavior of the composites.

3.2 Scanning Electron Micrographs (SEM):

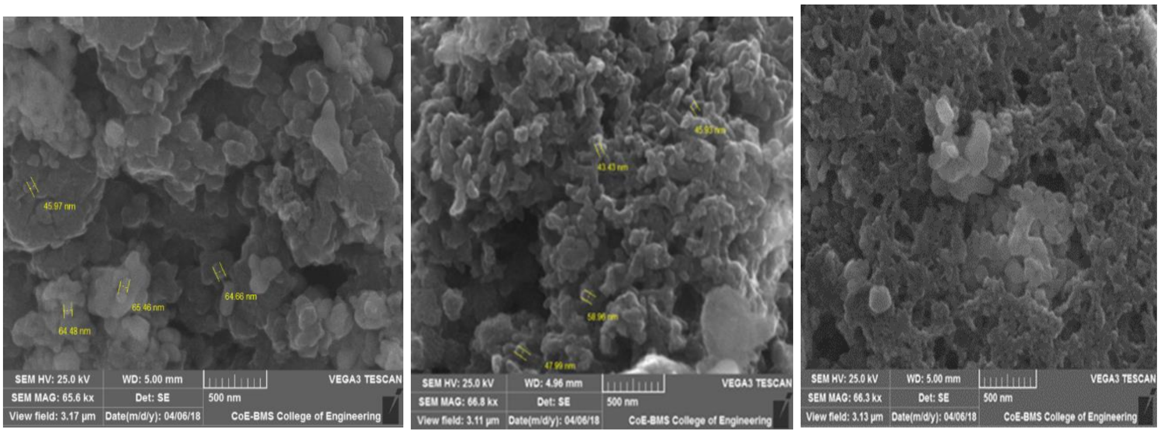
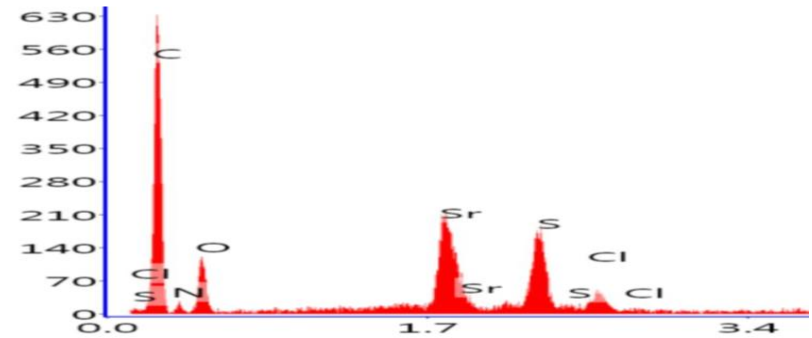


Figure 2 (a) Pure Polyaniline, Figure 2(b) pure Sro Figure 2(c) 50wt.% of polyamine/Sro nanocomposites.

It is found that from image that polyaniline grains have well interaction with one another. the average size was calculated by using linear intercept formula and it is found to be 45nm to 65nm Figure 2(b) shows the higher resolution SEM image of pure SrO and it is seen to be porous like structure. the average grain size was found to be 45nm to 65nm 200nm. the grains are found to be well interconnected with each other which indicate that they have enough binding energy to combine with neighbors grains or molecules.

Figure 2(b) shows the SEM image of PANI-SrO 50w.t% nanocomposites. It is found to have highly agglomerated chain like structure. the crystallinity of the SrO is seen to decreases with addition of PANI in it. The Average grain size is found to be 45nm to 65nm.

3.3 Energy dispersive X-ray (EDX) analysis:



Element	Weight %	Atomic %
C K	63.51	72.81
N K	9.51	9.43
O K	17.81	15.33
SrL	5.30	0.83
S K	2.99	1.28
CIK	0.80	0.31

EDAX was done to reveal the chemical composition of the samples. Figure shows the SEM and EADX images of PANI, PANI/SrO composites with different weight percentages are shown in the tables.

In order to confirm the existence of chemical composition of the nanoparticles in the nanocomposites, the prepared nanocomposites powder whereas given for EDX analysis. The obtained EDX pattern shows the strong and weak peaks corresponding to N, Sr and Cl atoms, and the results were good agreement with XRD and SEM analysis.

4. Result and Discussion:

Using the values of equivalent parallel capacitance (Cp), Phase angle(δ), Dissipation factor (D) and parallel equivalent resistance(Rp) that are recorded by LCR meter at selected frequency range (f), real and imaginary part of impedance (Z' &Z") have been calculated.

The surface morphology of the all nanocomposites is studied by using Phillips XL30 ESEM scanning electron microscope (SEM). This technique was widely used to know the composition of the materials with the position of profundity of focus (~100 – 200 Å)

4.1 Impedance Spectroscopy:

(a) Real Part of Impedance

From the plots, it is observed that the values of real impedance decreases at higher frequencies region and variation of impedance is depends on the filler concentration in PANI-SrO nanocomposite. The real part of impedance as a function of frequency at room temperature is shown in figure. It is observed that the impedance value decreases with increases in frequency as well as the variation of impedance becomes dependent on filler concentration in polyaniline-SrO composite matrix. Three different processes occur in the multiphase composite system: association of charge carriers, dielectric dispersion because of different dipole rotation and presence of both dielectric dispersion as well as charge carrier's union within measured frequency range. The presence of relaxation peaks in both complex impedances at low frequencies with increase in temperature could be due to space-charge relaxation below 1kHz. Therefore, electrical resistance decreases with decreases in complex impedance

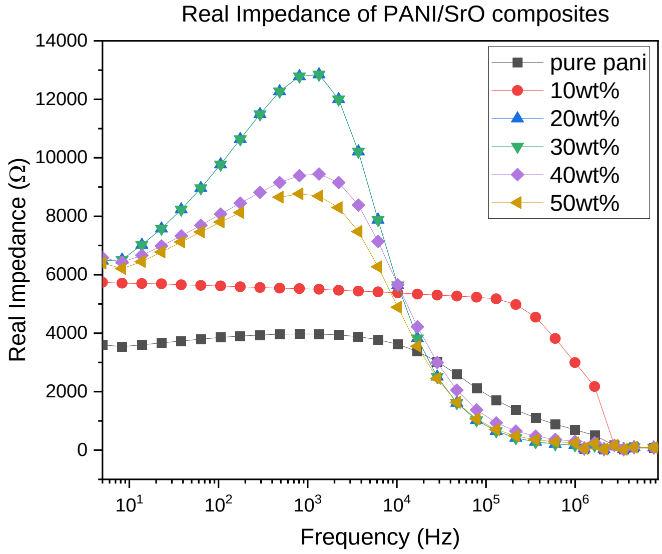


Figure: Shows real part of impedance of PANI-SrO nanocomposites

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(b)Imaginary Part of Impedance

It is clear that the behavior of Z" is dominated by single peak shifts to lower frequencies at 30wt% of PANI/SrO composite and strong dispersion of Z" exists similar to what is known for relaxation system. The frequency corresponding to the peak of Z" (=Z" max) gives the most probable time τm(=1/ωm). The most relaxation time obeys the Arrhenius relation

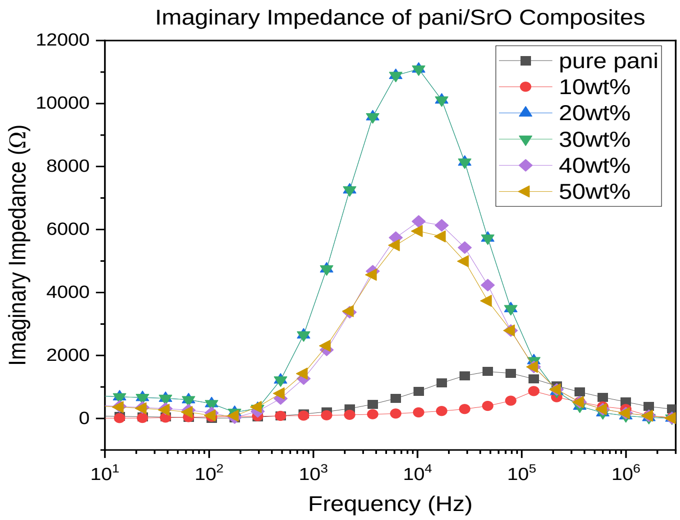

Figure: Shows the frequency dependence of the imaginary part of the impedance(Z'') of polyaniline/SrO composites.

4.2 Cole-Cole plot

Figure Shows the variation of real impedance as function of frequency Vs imaginary part of impedance (Cole-Cole plot) of PANI/SrO composites. It is observed from the above figure that the semi circles of Cole-Cole plot suggest that dominance of Debye type relaxation and Cole-Cole plot exhibits semicircles inverted, where it initially decreases and attains minimum further it increases to maximum and further it decreases. Therefore, it suggests that nearly Debye type relaxation has been confirmed. It is observed that resistance of the composites is inconsistent because of the change in the allotment of SrO particles in polyaniline. A mechanism for the charge motion through the pressed pellets of conducting polymers was hypothesized.

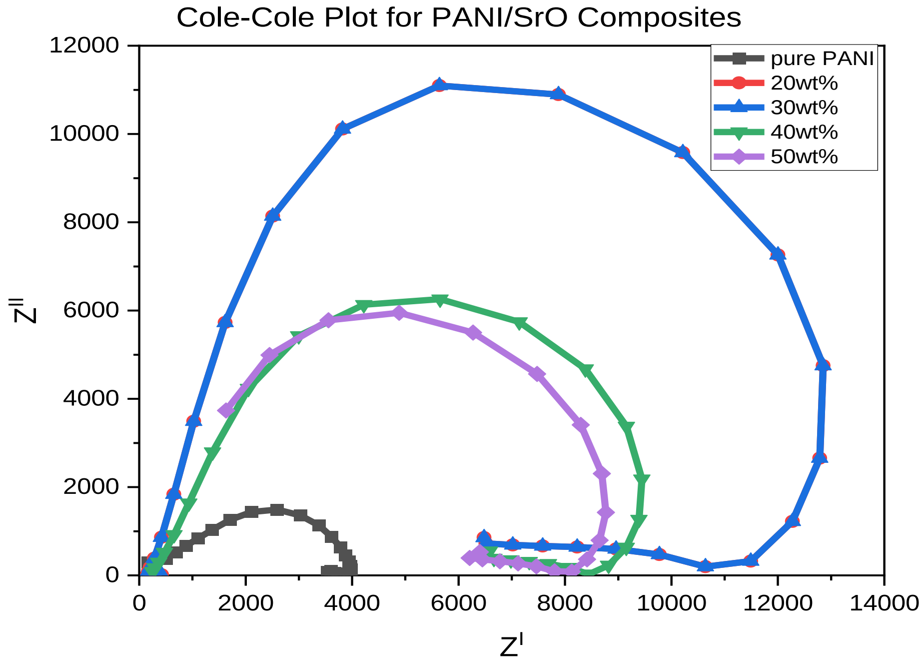

Figure: Shows Cole- Cole plot of polyaniline/SrO nanocomposites

5. Conclusion:

Polyaniline- SrO nanocomposites were prepared by in-situ polymerization method, The XRD pattern confirms size and crystallite of nanocomposites. The SEM image shows the presence of SrO nanoparticles which are uniformly distributed throughout the nanocomposites sample. Variation of impedance as a function of frequency suggests nearly Debye type relaxation, Cole-Cole Plots supports the observed phenomenon of Debye-type relaxation.

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