Synthesis And Applications Of N-doped Silica Polyaniline Nanocomposites

[1]Harpreet Kaur, [2]Deepak Saini, [3]Anupreet Kaur
[1]Student, [2]Assistant Professor, [3]Assistant Professor
Punjabi University

Abstract **- Although much work has already been done on various aspects of polymer/ silica nanocomposites, more research is required in order to further understanding the complex structure property relationship. Moreover, colloidal polymer/ silica nanocomposites which representation a new category of polymer/silica nanocomposites have attracted growing interest in recent years. Sol-gel method is mostly used in preparation of polymer/ silica nanocomposites particles. Conducting polymers have been found suitable for microelectronic device fabrication due to their excellent electric characteristics and ease of process ability. Among these polymers, Polyaniline has attracted much attention of many researchers due to its ease of synthesis, processibility, good thermal stability and good environment stability. This study involves the synthesis of Silica, PANI, Oxalic Acid nanocomposites conducting with n-type doping. Small amount of Oxalic acid is used in chemical doping to enhance the conductivity of synthesized by oxidative polymerization method. These nanocomposites are characterized using various techniques such as FTIR, SEM analysis and UV Spectra study.**

keywords **- Polyaniline (PANI), Conducting Polymers (CPs), Silica nanocomposites, Oxalic acid.**

Introduction:

Conducting polymers are the synthetic materials which have been a big part of the research that is going on in nanotechnology and applications of these materials are extended in different fields. Polymers possess an advantage over metals that they can be easily processed than metals. Advantages of physical properties of these materials lead to the studies which explored the potential of the conducting polymers in applications of electronics devices, solar cells, energy storage devices, sensors etc. [1-3]. Moreover, colloidal polymer and silica nanocomposites which representation a new category of polymer-silica nanocomposites have attracted growing interest in recent years. Sol-gel method is mostly used in preparation of Polyaniline-Silica nanocomposites particles.

Over the past decade, there has been significant progress in fabricating nanostructured materials with unique properties. Conducting Polymers are polymeric materials that display high conductivities, good electrochemical activity, unique optical properties. CPs have reached special attention as promising candidates in many areas of nanoscience and nanotechnology[4]. CPs also known as conjugated polymers or "synthetic metals" are polymers with highly pie conjugated polymeric chains. The synthesis of conducting polymer films thus recently been a significant subject of intensive research. Conducting polymers are characterized by a conjugated structure of alternating single & double bonds.

Polyaniline (PANI) is a mostly used conducting polymer because the conductivity and resitivity are grown increasing when its samples are doped with different acids [5]. This unique conductivity sets PANI apart from other conducting polymers. High conductivity of the polymers can be achieved through the reduction or oxidation of the polymers with the uptake of either an electron donor or acceptor [6]. Synthesis and analysis are used for check properties of PANI-SiO_2. PANI is regarded as most attractive conducting polymers due to its special features like low cost, high environmental friendly, good electrical conductivity, ease of synthesis and interesting redox properties associated with the chain nitrogen [7].

The most commonly used route for synthesizing silica nano particles is sol-gel method due to its ability to produce nano dispersed with narrow-size distribution nano particles at mild conditions[6]. However, a critical challenge in the preparation of nano composites is the homogeneity in the mixing between the filler & organic components. The nanocomposites are examined by Scanning Electron Microscopy, Fourier Transform Infrared Spectroscopy and UV visible study.

Synthesis of nanocomposites of oxalic acid and Conducting Polymers

Aniline (Loba Chemicals (99% purity)), Copper sulphate (Sd fine), Hydrochloric acid (Qualigens fine chemicals, A R Grade), Ammonia solution in water 28% (Sd Fine Chemicals), TEOS(sigma aldrich), Methanol (Sd fine), Oxalic acid (Loba) were used.

Experimental Procedure:

In this work, the Polyaniline was doped in the presence of methanol. Distilled aniline was used to synthesize Polyaniline (PANI) by chemical oxidation polymerization in acidic medium. Synthesized Polyaniline was dried at room temperature, in air tight vessel. Samples of oxalic acid having ratio 400gm was used.

Silica surface is used because it is can be used to attach polymers or ligands on to the oxalic acid surface. Oxalic acid is added in solution after 20-30minutes, when polyaniline and silica are added. This whole analysis of samples are done with the help of magneting stirrer. The particles are filtered with the help of filter paper and dried at room temperature in air tight vessel [7].

Preparation of Silica Nanoparticles: Silica nanoparticles are easily prepared with the help of sol-gel method and achieved different apllications in different fields. Sol-gel method was used firstly to prepare the monodispersed silica nanoparticles of uniform [8]. The silica particles are obtained by hydrolysis of tetraethylorthosilicate (TEOS) in the presence of methanol and analysis was done with different reagents in nano sizes. Particles of various sizes in the 20-460 nm range were synthesized. The ammonia reagents (2.8 to 3.8 mol L^{-1}) , methanol (20 to 30 mol L^{-1}) , HCl (0.08 to 0.12 mol L^{-1}) and TEOS (0.012 to 0.12 mol L^{-1}) were used and the particle size has been analysed by scanning electron microscope, fourier transmission electron microscopy and UV visible spectroscopy [9,10]. The synthesised mixture was stirred for 2 hours and kept for 24 hours at room temperature in air tight vessel. It becomes in white colored liquid. Later on the mixture was filtered by filtered paper. The obtained precipitates were dried and give white colour nano particles.

Preparation of SiO_2 and PANI:

The Silica-Polyaniline are synthesis by using the Sol– gel process. The Silica-PANI nanocomposites are obtained by adding particles of silica, aniline, copper sulphate in the methanol, which is used as solvent. Particles of various sizes in the 20-460 nm range were synthesized. The aniline reagents (3.8 to 5 mol L^{-1}) , methanol (30 to 40 mol L^{-1}) , silica (10 to 30gm) and Copper sulfate (10gm) were used[8]. The reaction mixture was stirred for 1 hour and kept for 24 hours at room temperature in air tight vessel. The mixture becomes in dark green colored liquid. After this, the mixture was filtered by filtered paper. After 10hours, the obtained precipitate was dried at room temperature and give dark green colour nano particles[9].

Preparation of Oxalic acid with SiO_2 and PANI:

The Silica-PANI particles are obtained by particles of silica, aniline, copper sulphate in the presence of methanol and synthesis was done in naosized particles. Particles of various sizes in the 20-460 nm range were synthesized. The aniline reagents (3.8 to 5 mol L^{-1}) , methanol (30 to 40 mol L^{-1}) , silica (10 to 30gm) and Copper sulphate (10gm) with oxalic acid (400gm) were used [9]. The reaction mixture was stirred for approx. 1 hour and kept for 24 hours at room temperature. This is done by use of magnetic stirring. It becomes in light green colored liquid. After this, the mixture was filtered by filtered paper. After some hours, the obtained precipitates were dried and give light green colour nano particles.

Results and Discussion

Synthesized particles are analysis with the help of Scanning Electron Microscopy (SEM), Fourier Transform infrared spectroscopy (FTIR) and Ultra Violet Spectra UV- Spectra characterizations. SEM is used to check the size of nanoparticles and SEM uses for focused beams of electrons to render high resolution, three-dimensional images. FTIR is the used to understand the structure of prepared nanoparticles. The FTIR analysis method uses infrared light to scan test samples and observe chemical properties [11]. UV spectra is used for check absorption spectroscopy in the ultraviolet visible spectral region. This UV spectra technique of measurement includes comparison between UV-Vis spectra fitting of the colloidal nanoparticles and theoretical calculation of absorption spectra.

Scanning Electron Microscopy

The samples were firstly analyzed to check the particles size by scanning electron microscopy (SEM). SEM is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. The SEM images show that increasing ratio of oxalic acid concentration can modify the size of nanoparticles. With the increasing of the oxalic acid concentration, the large diameters and higher yields of nanofibers were obtained. In this, Electromagnetic lenses are used to control the path of the electrons. Different types of electrons are emitted from samples upon interacting with the electron beam [12,13]. SEM analysis clearly shows the structure of particles to be nearly spherical and size is about 2.00um. Size of polyaniline nanoparticles is increased with oxalic acid adding in increasing order.

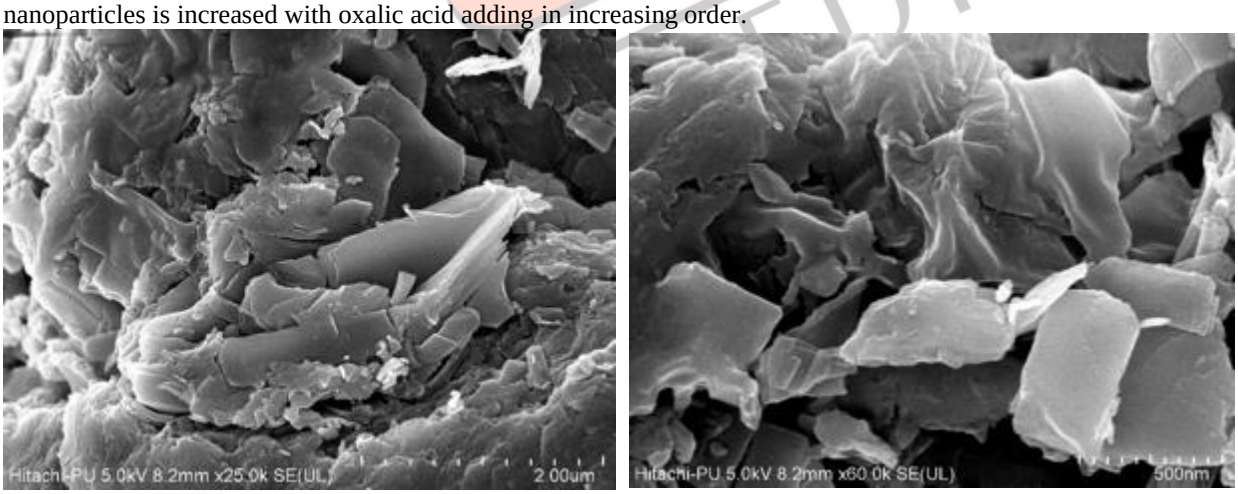

Figure 1: SEM Image of sample of 400gm oxalic acid + SiO_2 + PANI Nanocomposites

Study of FTIR spectra and its analysis

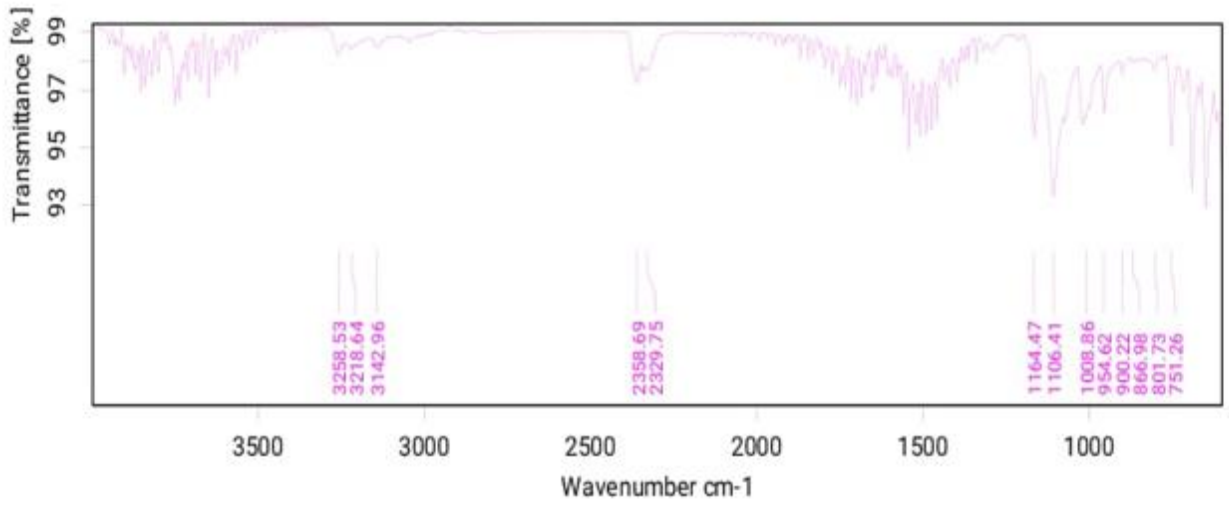

Figure 2. FTIR analysis of SiO_2 + PANI Nanocomposites

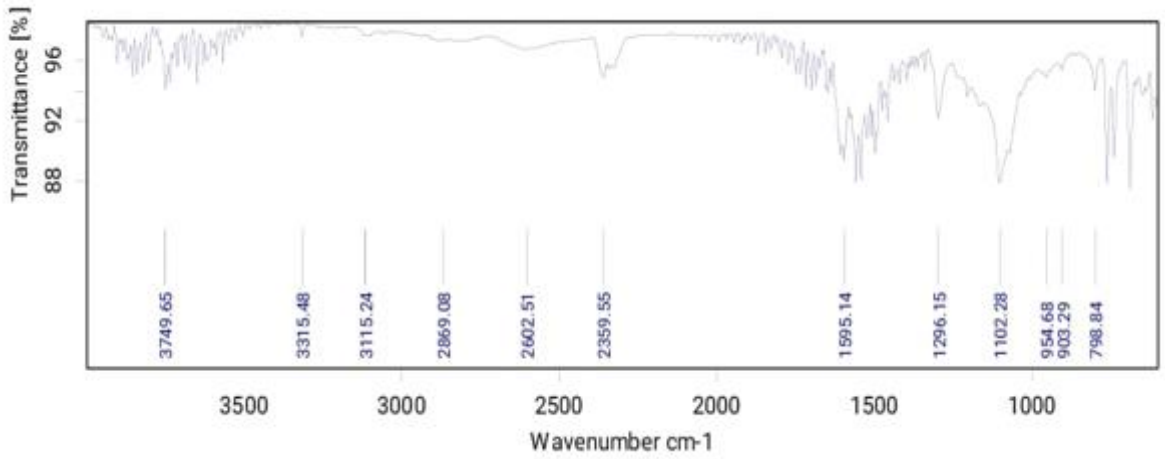

Figure 3. FTIR analysis of SiO_2 + 400gm Oxalic Acid + PANI Nanocomposites

FTIR is helpful for characterization of materials and check chemical properties of particles. FTIR Spectra was recorded on an Agilent Cary 630 FTIR Spectrophotometer. The stretching of different functional groups are shown below in Table of different functional groups, their standard absorption band range and observed absorption bands. Observed absorption bands are depicts the graph between transmittance(%) and wavenumber(cm^{-1}).

Figure 5. Shows FTIR spectra of pure Polyaniline prepared by using oxalic acid at room temperature. The characteristics peaks confirm the formation of Polyaniline.

Functional Groups	Standard Absorption Band Range (cm^{-1})	Observed Absorption Bands (cm^{-1})	Probable Assignments
OH	3650-3200	3384.43	OH stretching
C-H	3000-2800 900-500	2359.31 786	C-H stretching and C-H bending
Si-O	1111-801	952	Presence of Silica
C=N	1700-1600	1636	C=N stretching in imine
C=C	1600-1650	1594.70	C=C bond stretch in aromatic double bond

UV SPECTRA
400gm oxalic acid

Wavelength	**Absorbance**
1. 400	0.639
2. 410	0.649
3. 415	0.620
4. 420	0.603

UV Visible result is becomes in decreasing order, when we increase absorbance rate of sample. By measuring the absorbance at specific wavelength, the impurities of particles can be detected.

CONCLUSION

The synthesis of nanocomposite particles are used to enhance the electronics and electrical field. The Silica-Polyaniline composites are used to analysis the study with Oxalic acid. SEM characterization reveals the size of particles to be under the 2.00um. FTIR analysis confirms the Silicon oxygen, oxalic acid bonds and polyaniline bonds by the composite structures. UV spectra is used for check absorption spectroscopy in the ultraviolet visible spectral region. Polyaniline is mostly used due environmental stability. The aim of the present work is to understand and prepare conducting polymer materials having improved conductivity so that they can be used in electronic devices. The experimentation results shown that SiO_2 nanoparticles are being encapsulated by PANI with very strong effect on the morphology of nanocomposites.

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