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Synthesis and Application of Oxalic Acid Doped Silica-Polyaniline Nanocomposition

[1]Harpreet Kaur, [2]Deepak Saini, [3]Anupreet Kaur
[1]*Department of Electronics & Communications, Punjabi University*
[2]*Basics & Applied Sciences Department, Punjabi University*
Corresponding Author: Harpreet Kaur

Abstract: *Conducting Polymers and their nanocomposites have been known for a while and are used in diverse range of applications. This study involves the synthesis of Silica, PANI, Oxalic Acid nanocomposites conducting with n-type doping. 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 and SEM analysis.*

Keywords: *Polyaniline (PANI), Conducting Polymers (CPs), Silica nanocomposites¸ Oxalic acid.*

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I. Introduction

Nanotechnology is the study of phenomena and fine-tuning of materials at atomic and molecular and macromolecular scales, where properties differ significantly from those at a larger scale. At the nano scale, physical, chemical and biological properties differ from the properties of individual atoms and molecules of bulk matter.

Conducting polymers have been found suitable for microelectronic & nano electronic devices, fabrication due to their excellent electric characteristics & ease of process ability.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[1-3]. 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 and energy storage devices[4]. Many polymers are known since 1976 in which significantly polypyrrole, polythiophene, and polyaniline , have gained attention and become the subject of many studies[5]. Among the polymers, polyaniline (PANI) has been extensively studied due to its excellent conductivity, ease of synthesis, environmental stability, low cost and highly reversible redox properties. Polyaniline is more noble than copper and slightly less noble than silver which is the basis for its broad use in PCB manufacturing and its corrosion protection[6-8].

Polyaniline (PANI) is a unique conducting polymer because of the conductivity that can be achieved through doping that is either protonation or anion uptake in it. This unique conductivity sets PANI apart from other conducting polymers. In addition, the thermal stability and ease of synthesis make PANI one of the most studied conductive polymer systems. Synthesis methods used for preparing Polyaniline are chemical and electrochemical polymerization[9,10]. PANI is regarded as most attractive conducting polymers due to its special features like low cost, high environmental stability, good electrical conductivity, ease of synthesis, and interesting redox properties associated with the chain nitrogen.

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 Polyaniline Silica nanocomposites.

Some applications of Polyaniline such as light emitting diodes, transparent electrodes, corrosion protection of metals, electromagnetic radiation shielding, gas and humidity sensing, battery applications and many more. These nanocomposites are examined by Scanning Electron Microscopy, Fourier transform infrared spectroscopy, UV visible study and XRD patterns[11].

Synthesis of nanocomposites of oxalic acid and Conducting Polymers

Aniline (Loba Chemicals, (99% purity), Coppersulphate (Sdfine), Hydrochloric acid (Qualigens fine chemicals, A R Grade), Ammonia solution in water 28% (Sd Fine Chemicals), TEOS(sigma aldrich), Methanol (Sdfine), Oxalic acid (Loba) were used[4].

Experimental Procedure:

In this present work, the chemical doping in Polyaniline 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 200gm was used.

Silica surface is used because it is can be used to attach polymers or ligands on to the oxalic acid surface. Solution of oxalic acid particles in Polyaniline is prepared. Solution of ammonium hydroxide and then TEOS is added and this solution is stirred for 24 hours to coat the sample. Product of this process is removed using magnets and then filtered out properly[12].

Preparation of Silica Nanoparticles: Silica nanoparticles achieved a definite position in scientific research because they can be easily prepared. A sequential - method was used firstly to prepare the monodispersed silica nanoparticles of uniform size using ultrasonic sol- gel process[11]. The silica particles have therefore obtained by hydrolysis of tetraethylorthosilicate (TEOS) in the medium of methanol and study was done on the effects of different reagents in grain sizes. Particles of various sizes in the 20-460 nm range were synthesized. The ammonia reagents (2.8 to 4 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 examined in scanning electron microscope and transmission electron microscopy[10]. The reaction mixture was stirred for 2 hours and kept for 24 hours at room temperature. It leads to formation of white colored liquid. Later on the mixture was filtered by filtered paper. The obtained precipitate was dried and give white colour nano particles.

Preparation of SiO_2 and PANI: The Silica-Polyaniline was made on the Sol– gel process implemented on SiO_2 nanocomposites, surface modification effect. The Silica-PANI nanocomposites are obtained by silica, aniline, methanol, copper sulphate. 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. It leads to formation of dark green colored liquid. Later on the mixture was filtered by filtered paper. The obtained precipitate was dried and give dark greencolour nano particles[13].

Preparation of Oxalic acid with SiO_2 and PANI: The Silica-PANI particles are obtained by silica, aniline, methanol, copper sulphate and study was done on the effects of different reagents in grain sizes. 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) with oxalic acid (200gm) was used[1,2]. The reaction mixture was stirred for 1 hour and kept for 24 hours at room temperature. It leads to formation of light green colored liquid. Later on the mixture was filtered by filtered paper. The obtained precipitate was dried and give light greencolour nano particles.

Synthesis of Polyaniline composite with silica coated oxalic acid is done by in-situ oxidation polymerization of aniline. Aniline solution in methanol is used for polymerization and oxalic acid particles are added in to it while adding cupric sulfate[2]. This is done by magnetic stirring.

II. Results and Discussion

Synthesized particles are studied by performing characterization procedures on to the sample. Scanning Electron Microscopy (SEM), Fourier Transform infrared spectroscopy (FTIR) characterizations are performed. SEM is used to know the morphology and size of nanoparticles. FTIR is the characterization used to understand bond structure of formed nanocomposites[3,5].

Scanning Electron Microscopy

SEM (Scanning Electron Microscopy) is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. Samples of SEM have to be prepared to with stand the vacuum conditions & high energy beam of electrons and have to be of a size that wit fit on the specimen stage. SEM analysis of powdered Oxalic acid-SiO_2-Polyaniline Nanocomposites is obtained. SEM analysis clearly shows the structure of particles to be nearly spherical and size is about 500nm. Size of polyaniline nanoparticles is increased with oxalic acid adding.

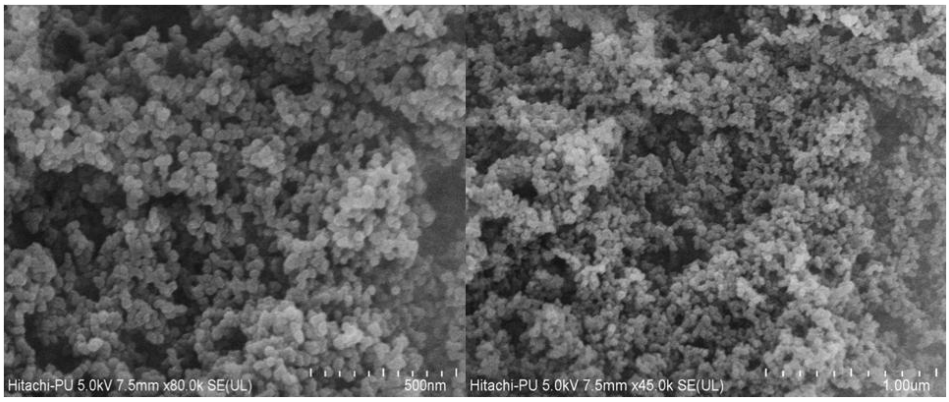

Figure 1: SEM Image of sample of 200gm oxalic acid + SiO_2 + PANI Nanocomposites

Study of FTIR spectra and its analysis

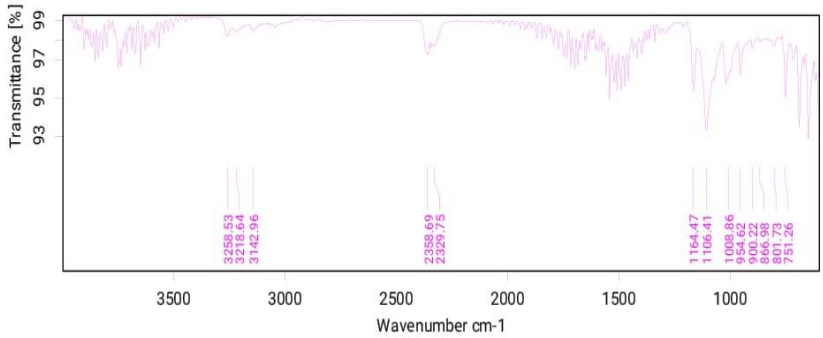

Figure 2. FTIR analysis of SiO_2+ PANI Nanocomposites

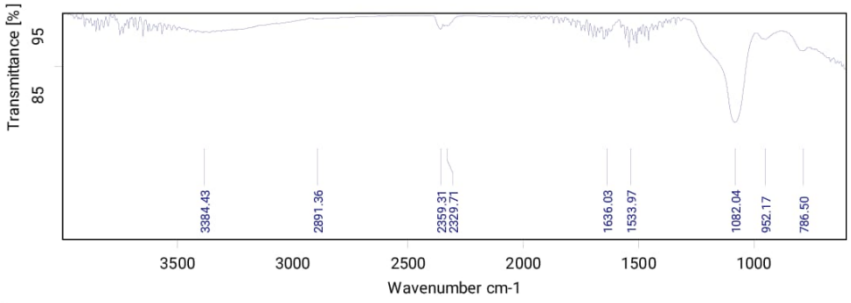

Figure 3. FTIR analysis of SiO_2+ 200gm Oxalic Acid + PANI Nanocomposites

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

FTIR is helpful for characterization of materials. 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.

III. Conclusion

The work comprised of synthesis of Polyaniline composites with Silica coated magnetite particles for modifying optical and electrical properties. SEM characterization reveals the size of particles to be under the 500nm. FTIR analysis confirms the bonding between particles. Silicon oxygen bonds and oxalic acid bonds and Polyaniline bonds are confirming the composite structure. SEM and FTIR spectra of doped PANI indicate the strong interaction of dopant with PANI π-conjugation system which induces structural modifications in the system. The samples are showing direct and indirect band gap, which change with change in dopant's ratio.

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