Synthesis and analysis of Silica/Polyaniline Nanocomposites

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Abstract

The present work describes a new, simple and environmentally friend procedure for preparing Silica/Polyaniline (PANI) hybrid materials by combining the sol–gel method and in situ polymerization of aniline. The synthesis of silica nanocomposites and Silica/PANI nanocomposites was done and their morphological and structural behavior characterization the Silica/PANI nanocomposite were examined under Scanning Electron Microscopy (SEM). For elemental ratio Scanning Electron Microscopy and Energy Dispersion Spectroscopy (SEM and EDS) was conducted which checks the content of individual elements in the sample, Fourier transform infrared spectra(FTIR) depicts graphs between transmittance ratio and wave number, various stretching between different functional groups and observed absorption bands ranges from 1636 to 3405 cm^{-1}. It was also found that the conductivity of the sample gradually increases with the increase in voltage and current.

KeyWords – Silica Nanoparticles, Conducting Polymers, Silica/PANI nanocomposites, Sol-gel.

1. Introduction

Presently, in the recent scenario great attention have been received by nanomaterials due to their optical, electrical chemical and mechanical properties. Nanomaterials are used for ultrasensitive detection of biological species. Various type of materials used to make nanomaterials are nanotubes, semiconductors, carbon nanowires, and nanocomposites are polymers and metals. The materials applications areas involves in optical, chemical sciences applications, bio medical applications and electronic devices. These materials are used for the fabrication uses in high density arrays [1]. A new class of polymers known as Conductive polymers were firstly discovered in 1976. Many polymers are known since 1976 in which significantly polypyrrole, polythiophene, and polyaniline , have gained attention and become the subject of many studies. Encapsulation or blending of inorganic nanoparticles in intrinsically conducting polymers (ICPs) to design nanocomposites is an exciting field where the conjugated polymer matrix [2]. There has been plethora of interest in the last two decades on nanocomposites conjugated polymers because they attains unique electronic and optical properties and their potential applications in a various advanced devices ranging from organic electronics, sensors, batteries, actuators, to electro-optic and electro-chromatic devices. Electrical properties of polymers are modified by adding inorganic nanoparticles within the polymer matrix [3].

PANI is known as the most attractive and appealing conducting polymers because of its special features like low cost, high environmental stability, good electrical conductivity, ease of synthesis, and redox properties are attractive because they are associated with the chain of nitrogen. Polyaniline is a typical phenylene based polymer that have chemically flexible –NH group in a polymer chain supported either side by a phenylene ring. The different feature of PANI as compared to other conjugated polymers is the reversibly tunable redox characteristics which permits the control of electrical conductivity over a wide range by protonation and doping of charge-transfer.

Silica nanoparticles occupy a prominent position in scientific research, because of their easy preparation and their wide uses in various industrial applications, such as catalysis, pigments, pharmacy, electronic and thin film substrates, electronic and thermal insulators, and humidity [4]. Silica nanoparticles have been utilized as electronic substrates, thin films, electrical/ thermal insulators, emulsifiers, stabilizers, etc [5]. To date, Silica/PANI particle composites have mostly been constructed through chemical routes [6].Silica Nanocomposites/PANI have received attention due to their greater surface area which allows the fast distribution of gas molecules in the structure. The properties of the nanocomposites strongly depend on their composition, the size of the particles, interfacial interaction, etc [7].A number of routes/ways are used to prepare nanocomposites of various conductive polymers. The Silica/PANI nanocomposites were synthesized by an in situ chemical oxidative polymerization of aniline in the presence of nano silica [8]. Highly conductive PANI nanostructures are promising materials for batteries and supercapacitors [9].

2. Experimental Details

2.1 Chemical Details

Tetraethylorthosilicate(TEOS), Copper Sulphate ($CuSO_4.5H_2O$), Ammonia solution (NH_3), Methanol (CH_3OH) as an alcohol solvent, 35% Hydrochloric Acid (HCl), aniline (99.5%), sodium laureal sulphate as surfactant.

2.2 Preparation of Silica Nanoparticles

Silica nanoparticles were prepared by taking liquid Tetraethylorthosilicate (TEOS) of 5ml in which 10ml liquid ammonia was added in the solution. Diluted 5ml of hydrochloric acid was added. 25ml of methanol is also added as a solvent. This mixture of solution was continuously stirred on a magnetic bead stirrer for about 30 minutes. After that the solution was filtered using filter paper. The solution was soaked at room temperature and then the synthesized silica nanoparticles denoted as sample A1 was obtained which was white in color.

2.3 Synthesis of Silica/PANI nanocomposites

The PANI/SiO_2 nanocomposite were synthesized by in situ chemical oxidative polymerization method of aniline in the presence of silica nanoparticles [10]. The value of Silica/PANI nanocomposites was taken. The amount of 900mg of silica nanoparticles was taken and different ratios of weight of methanol and polyaniline were considered. The ratio of silica to polyaniline was 9:1. In this experiment 900mg sample of silica nanoparticles solid powdered form was kept in a beaker in which approximately 10ml (9ml methanol + 1ml polyaniline) was mixed. 2.5g of Copper sulphate powdered form was added in 100ml methanol. The blue green solution was obtained from which 0.5ml of copper sulphate was added in beaker. Then the solution is mixed using magnetic bead stirrer for approximately 20 minutes. After that the solution is filtered and soaked using filter paper. Dark green precipitates are formed which are named as sample A2 and kept in centrifugal bottles for sample analysis.

3. Characterization and Result Details

3.1 SEM Analysis

The sample was firstly analyzed to check the morphology and particle size by scanning electron microscopy (SEM) SEI 20kV at magnification scale of 20000 in range of 1µm at wave distance of 11mm as shown in Figure 1. To

check the crystallography measurement of each chemical amount independently Scanning Electron Microscopy and energy dispersion spectroscopy (SEM and EDS) analysis was done which is depicted in Figure 2 in which spectra as well as energy dispersive spectroscopy. For sample A2 (9:1) the spectrum was in 800 µm. The possible peaks omitted in this case was : 2.160, 8.070, 9.700 keV. All elements were analyzed in a Normalised manner. Number of iterations equals to five. The description of elements, their weight ratio and atomic ratio of sample A2 is shown below in Table 1.

Element	Weight%	Atomic%
C K	26.45	35.05
O K	54.32	54.05
Si K	19.23	10.90
TOTALS	100.00	100.00

Table 1 Description of elements of Sample A2

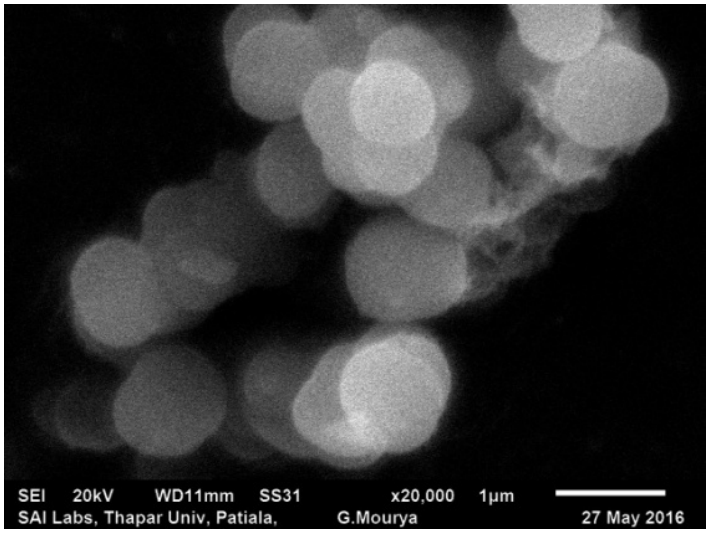

Figure1: Morphology of sample A2-Silica/PANI 9:1 at magnification of 20000 in the range of 1µm.

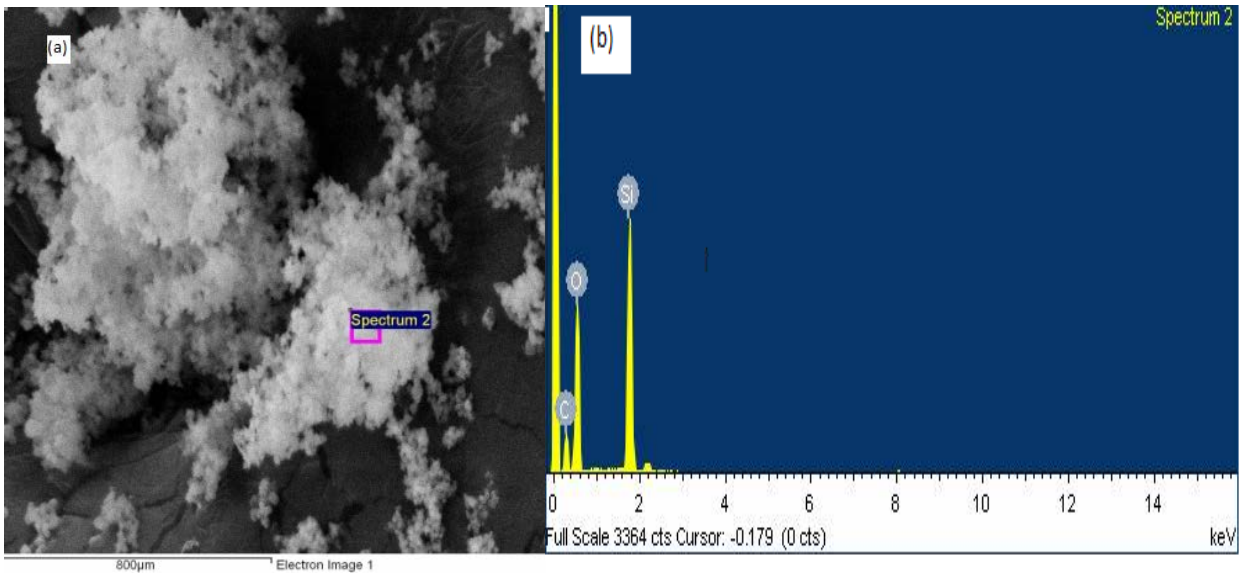

Figure 2:(a) A2 (9:1) spectrum in the range of 800µm. **(b)** Different peaks obtained of carbon,oxygen and silica.

3.2 Study of FTIR spectra and its analysis

FTIR Spectra was recorded on an Agilent Cary 630 FTIR Spectrophotometer. The stretchings of different functional groups are shown below in Table 2 of different functional groups, their standard absorption band range and observed absorption bands. Observed absorption bands are Figure 3 Depicts the graph between transmittance and wavenumber. Different Peaks can be observed in which the highest peak is observed in 2200-2000 interval whereas the lowest peak is in 1200-1000 interval.

Functional Groups	Standard Absorption Band Range (cm^{-1})	Observed Absorption Bands (cm^{-1})	Probable Assignments
OH	3650-3200	3271	OH stretching
C-H	3000-2800 900-500	2834 798	C-H stretching and C-H bending
Si-O	1111-801	956	Presence of Silica
C=N	1700-1600	1629	C=N stretching in imine
C=C	1600-1650	1629	C=C bond stretch in aromatic double bond

Table 2: Representing Functional Groups,their Standard absorption band range,Observed Absorption bands and Probable Assignments.

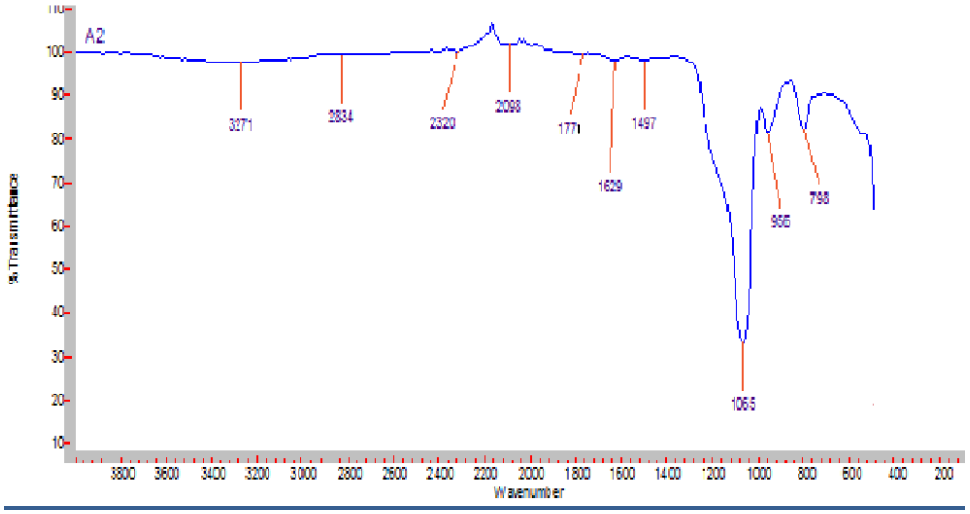

Figure 3: FTIR Spectra of A2 Sample

3.3 Study of conductivity of Silica/PANI Nanocomposites

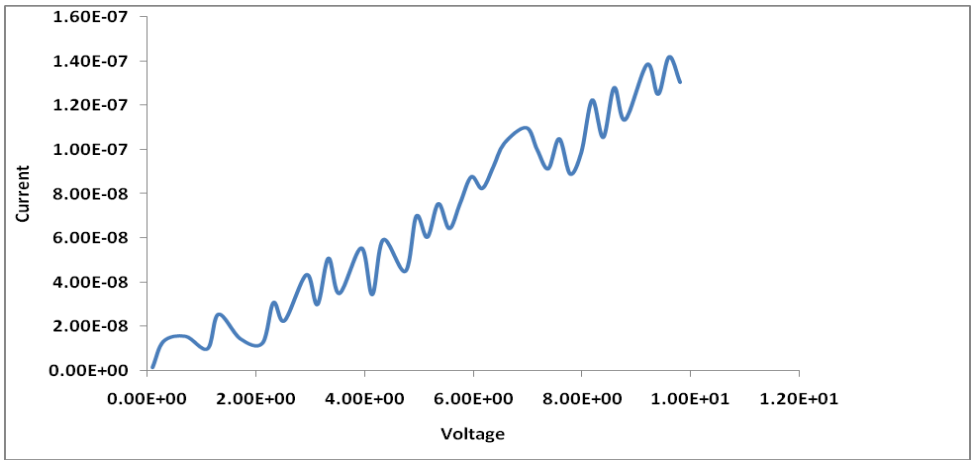

Figure 4: I-V or Graphical Representation of Conductivity of Sample A2

Two probe technique was the basic idea behind checking the electrical conductivities of the samples. For this purpose the pellets of round shaped were made of thickness 0.69mm at room temperature of 25^0C and compression moulding machine was used. The resistivity was computed using equation:

$$\text{Resistivity}(\delta)\ \text{ohm}-\text{cm} = \frac{4.532Vt}{I}$$

$$\text{Conductivity}(\sigma)\ \text{S/cm} = \frac{1}{\delta}$$

where the voltage applied is V volts, t is the temperature, the current flowing is I in the sample and difference between the probes is calculated as S distance. The strings in the above graphs shows that the Silica/PANI nanocomposites exhibits metallic behavior. As the voltage is increased the current is also increased. Due to larger surface area of silica nanocomposites and larger aspect ratio the conductivity of Silica/PANI nanocomposites is enhanced. These larger areas permits as an effective percolative conducting bridges [11].Due to high concentration of SiO_2 enhances the conductivity which further leads to inclination of mobility of electrons mobility in the composite system. This may be attributed to the doping effect associated with SiO_2 nanoparticles that were believed to help induce the formation of a more efficient network for charge transport, thus enhancing the conductivity of the composites [10].

4. Conclusion

It can be predicted from the SEM analysis that Silica/PANI Nanocomposite of A2 sample at magnification of 20000 are obtained in the range of 1000nm range. SEM and EDS shows the exact amount of concentration of elements present in the sample. Further Stretching of bonds between different elements is examined under FTIR which also depicts the absorption bands observation at different peaks. It was also concluded that the conductivity is enhanced as compared to pure PANI due to highly concentrated SiO_2 and as the voltage is increased current is also hiked which leads to more conductivity.

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