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Polyaniline-NickelTitanate Nanocomposites: Synthesis and Characterization

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Abstract: *Nickel titanate ($NiTiO_3$) nanoparticles have been synthesized by co-precipitation method. Polyaniline synthesized by the oxidation of aniline in presence of ammonium persulphate Polyaniline-nickel titanate nanocomposites synthesized by embedding different weight percentage of nickel titanate. XRD analysis was used to study the structure and estimate the size of the particles. Scanning electron microscopy was used to study the morphology of composite materials.*

Keywords: *Polyaniline, nickel titanate, co-precipitation , PANI-NT nanocomposites.*

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I. Introduction

Material science is also the study of the properties of solid materials and variation in their properties is due to materials composition and structure. A composite material is made by combining two or more materials have many advantages over monolithic materials, because their mechanical, chemical and electrical properties can be altered as per the requirements for applications. Composites are materials that are combinations of two or more organic or inorganic components. One material serves as a "matrix," which is the material that holds everything together, while the other material serves as reinforcement, in the form of fibers embedded in the matrix. [1].

The polymers having poly-conjugated structures and possess poor electrical conductivity but the oxidized polymers exhibit appreciable electrical conductivity [2-3]. Among the conducting polymers, polyaniline (PANI) have attracted a great deal of attention as is unique among the most researched organic conducting polymer which is easy to synthesize, having good electrical conductivity, fair chemical stability and widely studied for electronic and optical applications[4-5].

Nickel titanate (NT) is an inorganic compound with the chemical formula $NiTiO_3$ is also known as nickel titanium oxide, is a coordination compound between nickel(II), titanium(IV) and oxide ions. It has the appearance of a yellow powder. It is an n-type semiconducting material with a band gap around 2.18 eV, which possess high electrical resistivity and high permittivity. NT has both semiconducting and anti-ferromagnetic behavior.[6-11] This paper presents synthesis of PANI, NT and PANI-NT composites with different composition of NT, XRD characterization and SEM analysis of samples.

II. Materials and Methods

The analytical grade materials were used to synthesize the required samples.

Synthesis of Polyaniline (PANI): PANI was synthesized by the oxidation of aniline in presence of ammonium persulphate. A solution of 0.5 M aniline was prepared in 0.5M HCl. A solution of 0.5M ammonium persulphate was also prepared in distilled water. A known volume of aniline hydrochloride solution was taken in a 1000 ml beaker and is stirred for about five minutes. Now an equivalent quantity of 0.5M ammonium per sulphate is added drop by drop by using a burette and continuously stirred using a magnetic stirrer. The colourless solution of aniline hydrochloride slowly turns to green. After addition of entire quantity of ammonium per sulphate stirring was continued for 15minutes. Precipitate was allowed to settle down and then was filtered. The dark green coloured precipitate of polyaniline was obtained. The precipitate was washed with distilled water several times to remove the impurities. Finally the precipitate was washed with acetone to remove the foreign bodies. Now the precipitate was allowed to dry completely on its own at room temperature. The dried material was grinded to fine powder by using mortar and pestle for about 20 minutes and stored for further process [12].

Synthesis of $NiTiO_3$: The co-precipitation method was adopted to prepare NT nanoparticles. The 0.1M $NiCl.6H_2O$ and of $TiCl_4$ solutions were mixed and stirred for five minutes. A mixed solution of 250ml was

prepared by adding 20ml of 30% H_2O_2 to 15ml of liquid ammonia with remaining quantity of distilled water. A mixture of 250ml each of $TiCl_4$ and $NiCl_2$ mixed in a 1000ml beaker and the above prepared solution was added. The precipitation obtained was filtered, washed and dried. The resultant was the fine powder of $NiTO_3$ which was calcinated at 450^{o}C for two hours and stored for further process.

Synthesis of PANI- NT Nanocomposites: PANI-NT composites were synthesized by adding known wt% of $NiTiO_3$ powder to the polymerization reaction .0.5M of aniline ($C_6H_5NH_2$) solution dissolved in 0.5M HCl and stirred for 5 minutes and 0.5 g (5wt %) of NT nano-powder was added and stirred with magnetic stirrer for about 15 minutes, then ammonium persulphate ($(NH_4)_2S_2O_8$) was added drop by drop. Even after complete addition of ammonium persulphate, stirring was continued for another 30 minutes and allowed the precipitate for about 30-40 minutes to settle down. Now precipitate was filtered and washed with distilled water several times to remove the impurities. Finally washed with acetone and precipitate was dried on its own at room temperature and was grinded for 15 minutes with mortar and pestle. Now the resultant sample was the PANI-NT nanocomposite with 5wt% NT.

In the same manner, PANI- NT nanocomposites with 10wt%, 15 wt%, and 20wt% of NT were synthesized. The prepared PANI- NT nanocomposites were characterized by XRD and SEM.

III. Results and Discussion

The Figures-1 and 2 respectively representing the XRD patterns of pure PANI and NT nanoparticles. The XRD pattern of pure PANI shows three broad peaks at 2θ values 10.5^o, 20.5^o and 25.5^o and indicates its semi-crystalline structure nature. The XRD pattern of NT reveals crystalline structure of the sample formation of tetragonal phase of $NiTiO_3$, which is approved by the appearance of X-ray reflections at 2θ values 10.797^o, 25.354^o, 39.293^o, 35.752^o and 54.287^o (JCPDS 75-3757). The average crystallites sizes of NT particle have been estimted by Scherer's formula:

$$\mathbf{d} = \frac{\mathbf{k\lambda}}{\boldsymbol{\beta}\mathbf{cos\theta}}$$

where **d** is the crystallite size, a constant **k**=0.94, **λ** is the wavelength of radiation, **β** is the full width at half maximum (FWHM) of the diffracted peak and **θ** is the angle of diffraction. The average crystallites size was estimated at 34.77nm.

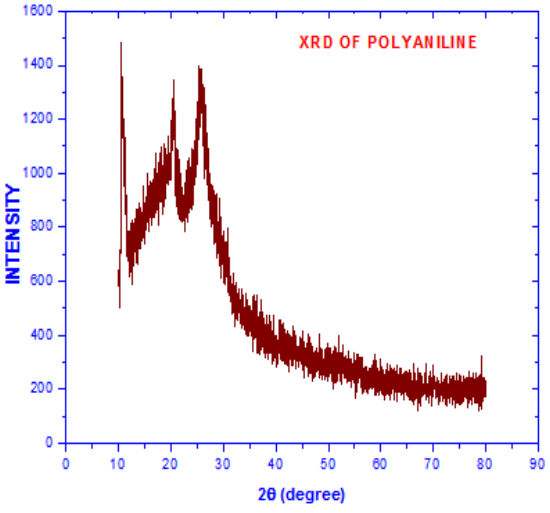

Figure 1: XRD pattern of pure PANI

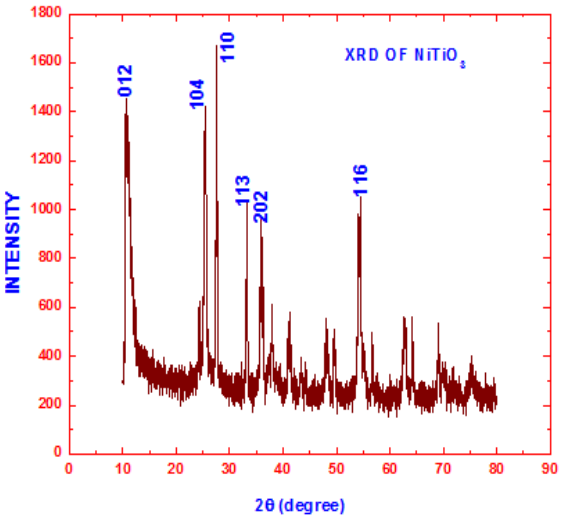

Figure 2: XRD pattern of pure NT

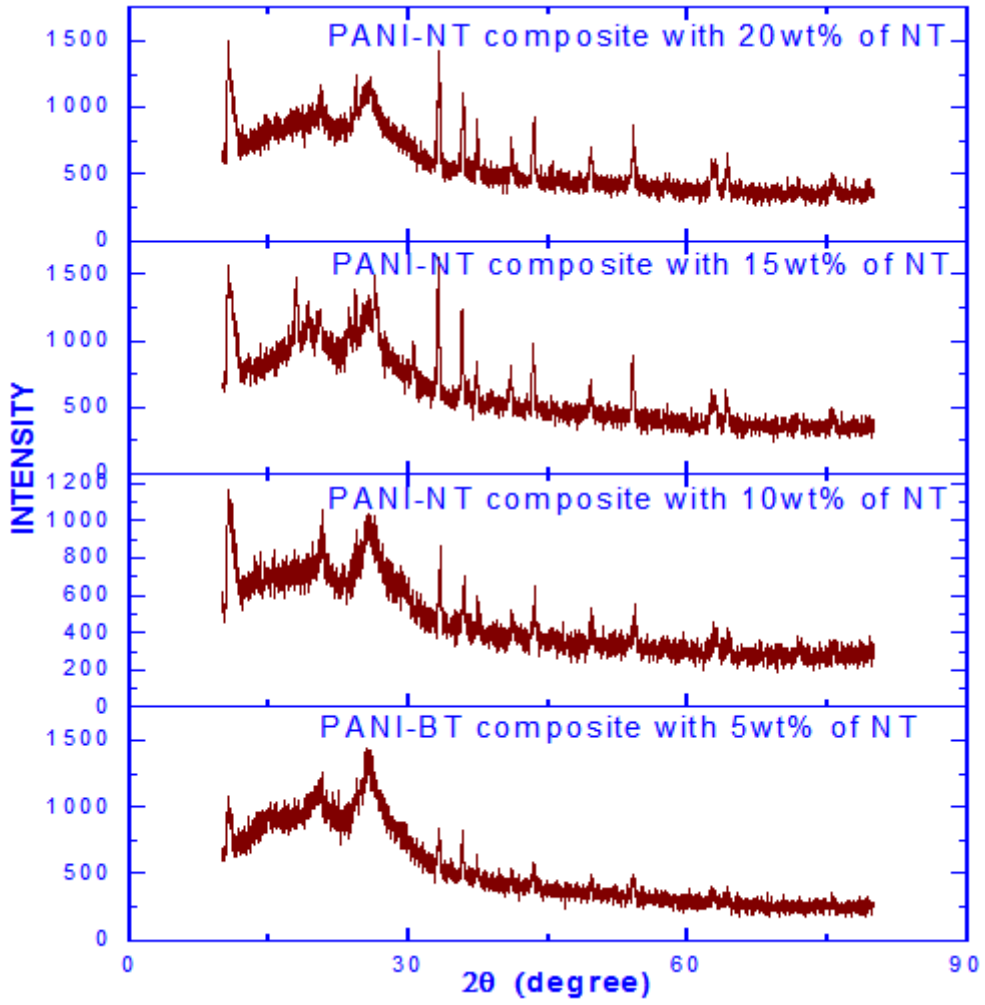

Figure 3: XRD pattern of (a) NT naono material & PANI-NT nanocomposites with (b) 5wt% NT, (c) 10wt% NT, (d) 15wt% NT & (e) 20wt% NT

The X-ray diffraction patterns of NT and PANI-NT nanocomposites are as shown in Figure-3.The long peak of NT XRD pattern at 33.20^{o} can be seen in all prepared PANI-NT composites which reveals that NT retained its structure even though dispersed in PANI during polymerization reaction, the height of the peak with increase in wt% of NT. The interaction between PANI and NT does not affect on appearance of broad peak of PANI reveals the formation of semi-crystalline composites. This result reveals the formation of PANI-NT nanocomposites and the estimated particle sizes are as in the following table.

PANI-NT COMPOSITES	5wt% of NT	10wt% of NT	15wt% of NT	20wt% of NT
PARTICLE SIZE	19.08nm	20.20nm	19.99nm	20.13nm

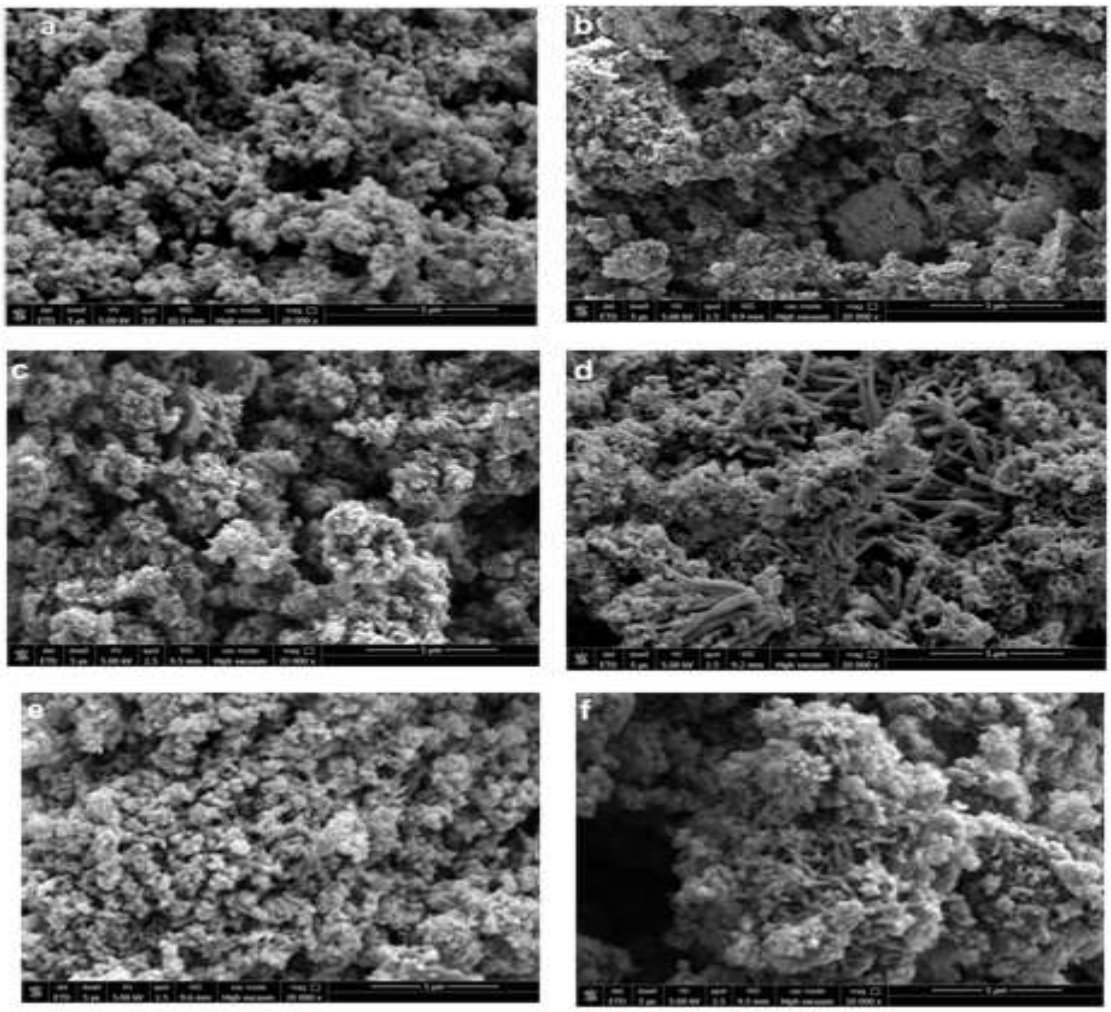

Figure 4: SEM images of (a)PANI,(b) NT (C)PANI-NT nanocomposite with 5wt% of NT, (d)PANI-NT nanocomposite with 0wt% of NT, (e)PANI-NT nanocomposite with 15wt% of NT & (C)PANI-NT nanocomposite with 20wt% of NT

The SEM images of PANI, NT and PANI-NT nanocomposites were obtained to analyze their surface morphology. The SEM photographs of (a) PANI (b) NT and (c-f) PANI-NT composites of magnification 20kx are presented in figure-4. Figure- 4(a) shows SEM image of PANI indicates micro-porous structure.Figure-4(b) is the SEM image of NT indicates the existence of its crystalline structure The Figures-4(c),(d), (e) and (f) are the are the respective SEM images of 5 wt % , 10 wt % , 15 wt % and 20 wt % PANI-NT nanocomposites. The increase in wt% of NT results in increase of the crystallinity of composite materials. The wide dispersion of filler particles (NT) in the matrix (PANI), confirms the formation of nanocomposites.

IV. Conclusion

Polyaniline and $NiTiO_3$ nanoparticles have been synthesized successfully. Polyaniline - $NiTiO_3$ nanocomposites have been successfully synthesized by in situ chemical polymerization of aniline using ammonium persulphate as oxidizing agent by co-precipitation method. The particle sizes were estimated ffrom XRD plots and surface morphology analyzed from SEM images.

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