



Structural Characterization of TiO₂ Doped Polyaniline

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ABSTRACT: Conducting TiO₂/Polyaniline (TiO₂/PANI) polymer composite were successfully synthesized by chemical oxidative polymerization method. These synthesized polymers were characterized by x-ray diffraction (XRD), scanning electron microscope (SEM). Amorphous nature of pure PANI is turned to crystalline nature as increasing doping concentration from 0 to 40%. SEM micrographs exhibit agglomeration and as composition increases there is an increment in the particle size. Fourier transform infrared spectroscopy study showed the bonding and vibrations and confirms the composite of TiO₂ in PANI.

Keywords: Polyaniline/TiO₂ Composite, SEM micrographs, XRD, FTIR.

I. INTRODUCTION

The synthesis of conducting polymers has been accomplished by oxidising or reducing process either through chemical doping [1] or electrochemical doping [2]. Various applications of conducting polymers have been proposed as transducers of biosensor [3], electrodes of rechargeable batteries [4], artificial nerves and muscles [5], gas sensors [6], solid electrolytic capacitor, diodes and transistors [7], anti-static electromagnetic shielding [8], and biomedical applications [9]. Polyaniline (PANI) continues to attract considerable attention because its electrical and optical properties can be changed by oxidation and protonation of the amine nitrogen atoms. The protonation and deprotonation and various other physico-chemical properties of polyaniline is due to the presence of the –NH– group. There are several reports of polyaniline found in the literature over the decades about the structure and constitutional aspect of aniline polymerization [10]. The primary structure describes the connectivity of the atoms. The secondary structure describes the three dimensional shape due to short range non-bonded interactions, such as backbone twisting. Our aim is to synthesize pure polyaniline and TiO₂ doped polyaniline to enhance the structural properties of the conducting polymer.

II. MATERIALS AND METHOD

A. Preparation of polyaniline

For the synthesis of polyaniline first we have to choose a raw material which gives a conducting polymer. Aniline (0.2M) solution dissolved in 1M aqueous solution of HCl at a room temperature with a constant mechanical stirrer for 2hours to ensure completion of the reaction this gives aniline hydrochloride followed by this ammonium per sulphate (0.2M) was added up drop wise to a stirred solution. The time of initial colouration of mixing of the reactant depend upon the temperature and concentration of the proteic acid. During the polymerization reaction, HCl was used as a proteic acid and the temperature was mentained at 0 to 5^o C by using a freezing mixture. The end product was a green colour precipitate. This precipitate was filtered, washed with de-ionised water, with acetone in order to remove the oligomers and excess ammonium persulphete and (0.1M) Hydrochloric acid to remove the Cl⁻ ions and unreacted aniline. At last precipitate was dried in a hot air oven for 24hours at a temperature of 50^o C to achieve a constant mass.

B. Preparation of TiO₂/PANI component

Synthesis of PANI/TiO₂ composite carried out by polarization in situ. Aniline (0.2M) was dissolved in hydrochloric acid (1M) and stirred for 2 hours to form hydrochloric.

TiO₂ was added in the mass fraction to the above solution with vigorous stirring in order to keep the TiO₂ homogeneously suspended in the solution. 0.2M ammonium persulphate was added to this mixture which acts as an oxidant was slowly added drop wise with continuous stirring at 5°C for 6 hours to completely polymerize the monomer aniline the precipitate was filtered, washed with deionized water and finally dried in a hot air oven for 24 hours to achieve the constant mass. In this way, PANI/TiO₂

composite containing various mass fraction of TiO₂ (5%,10%,20%,30%,40%) in PANI were synthesized.

III. RESULT AND DISCUSSIONS

A. X-ray diffraction analysis

Figure 1 shows the XRD pattern of pure polyaniline (pani) and TiO₂ doped polyaniline with different concentration like (5%, 10%, 20%, 30% and 40%). Pattern of PANI exhibits broad peak in the range of 20-27 degree and it has amorphous nature.

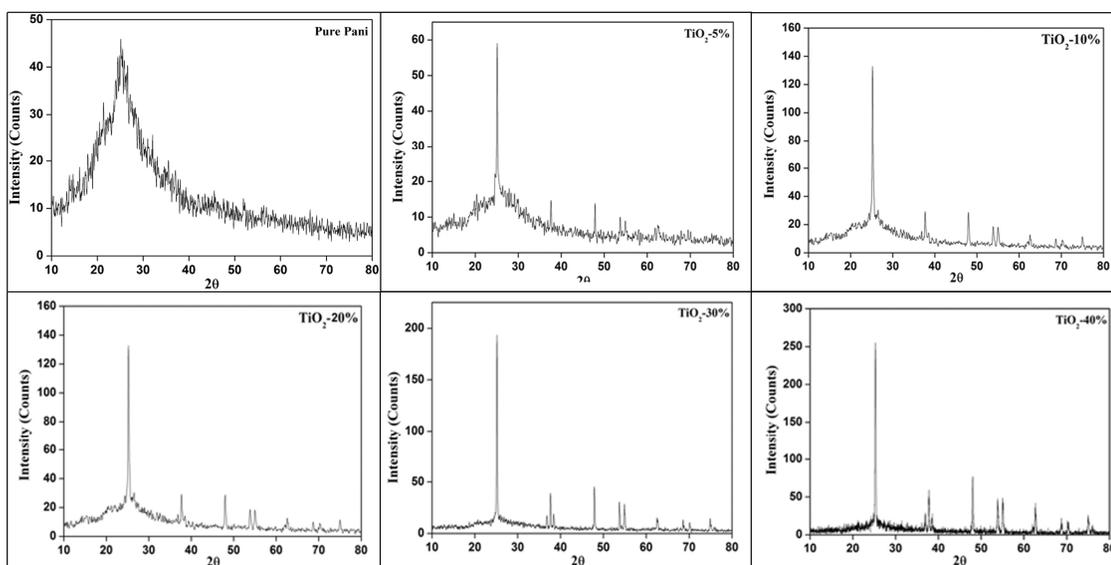


Fig. 1. X-ray diffraction patterns of Pure Polyaniline and TiO₂ / PANI (5%, 10%, 20%, 30%, 40%).

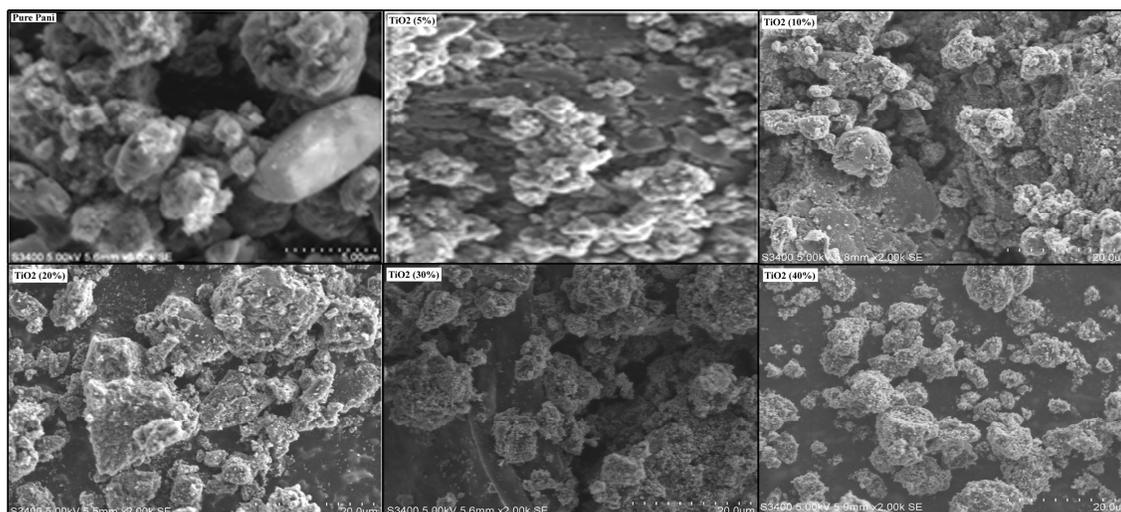


Fig. 2. SEM micrographs of Pure Polyaniline and TiO₂ doped with 5%, 10%, 20%, 30%, 40%.

In the patterns of composites PANI, TiO₂ anatase diffraction peak at 25.28⁰, 37.8⁰, 48.0⁰, 53.88⁰, 62.28⁰ and 68.78⁰ are due to (110), (101), (111) and (211) are present in PANI/TiO₂ composite which indicates the presence of TiO₂ in PANI [JCPDS file no. 21-1276]. Peaks show the crystalline structure of composite and obtained results are in good agreement with the literature. The intensity of composite is lower than pure TiO₂ because non crystalline polyaniline reduces mass-volume percentage of TiO₂. As concentration of TiO₂ increases then crystal size also increases.

B. Scanning Electron Microscope (SEM)

As evident from figure 2 SEM image of polymer samples of pure polyaniline and TiO₂ doped polyaniline synthesized by oxidation polymerization method. Pure PANI has granular agglomerates without inter space is seen in the image whereas TiO₂ have platelet type structure, as composition increases there is an increment in the particle size. TiO₂ doped in PANI is more crystalline because grain size is increasing as polyaniline is shifting to PANI-TiO₂.

C. Fourier Transform Infrared spectroscopy (FTIR)

Figure 3 showing the fourier transform infrared spectroscopy of pure PANI and TiO₂ doped polyaniline (TiO₂/PANI).

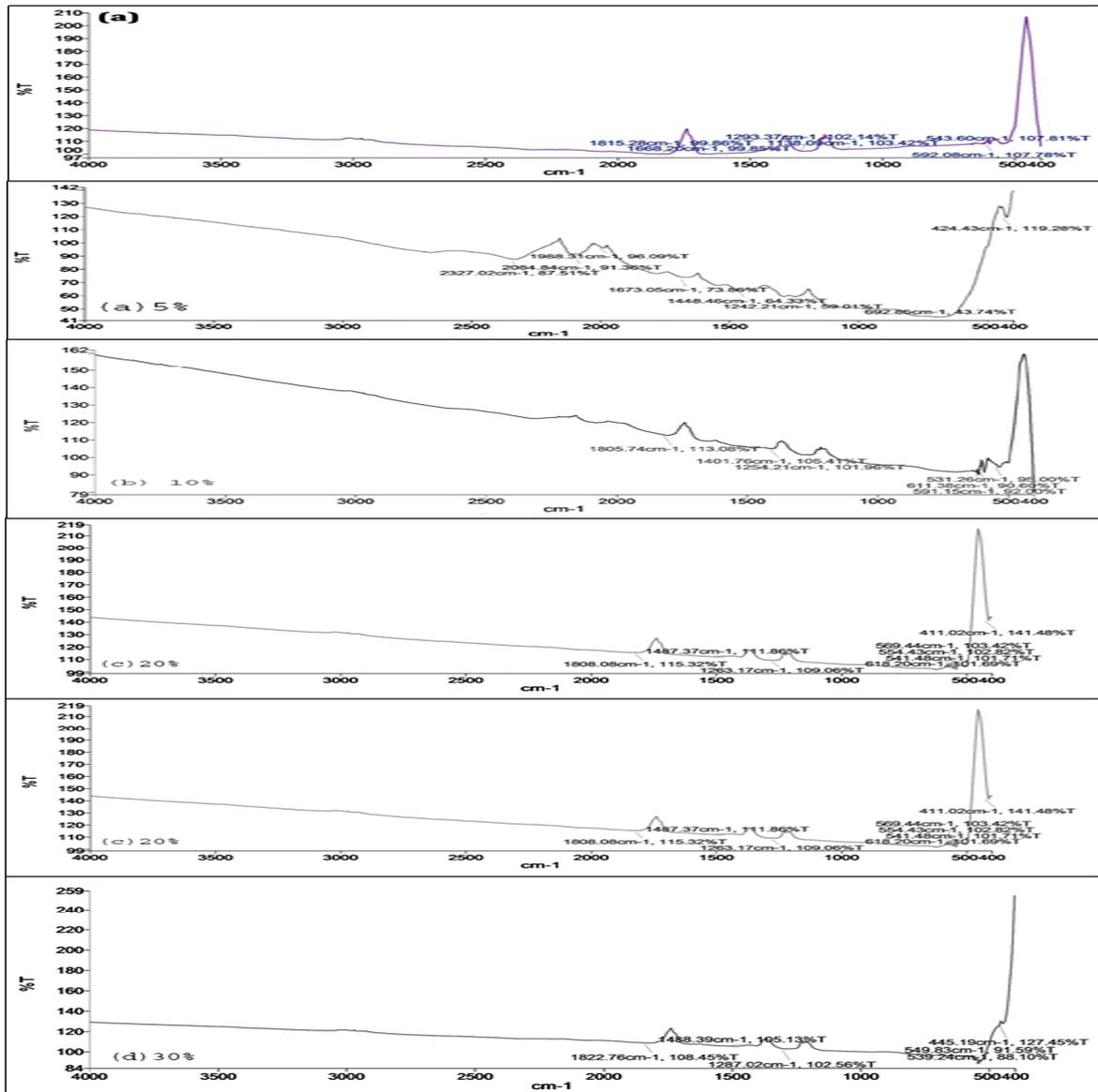


Fig. 3. FTIR spectroscopy of Pure Polyaniline and TiO₂/PANI (5%, 10%, 20%, 30%, 40%).

This is useful technique for characterizing materials and providing the information on molecular structure, vibrations and bonding stretches in the compound. When irradiated with infrared light (photons), sample can transmit, scattered or absorb the incident radiation. Absorbed infrared radiation usually excites molecules into higher energy vibrational states this can occurs when the energy of the light matches the energy difference between two energy states. Infrared spectroscopy is particularly useful for determining the functional groups present in a molecule. Many functional groups vibrate at nearly the same frequencies independent of their molecular environment. Further many subtle structural details can be gleaned from frequency shifts and intensity changes arising from the coupling of vibrations of different chemical bond functional groups. The band at 1805 cm^{-1} corresponded to quinoid and/or benzenoid structure of PANI. Also the band at 1254 cm^{-1} assigned to C-N stretching, 611 cm^{-1} is associated with C-C and C-H of benzoid unit.

IV. CONCLUSIONS

In this present work we are synthesized pure polyaniline and TiO_2 doped polyaniline with different concentration (5%, 10%, 20%, 30% and 40%) and are characterized by many techniques such as x-ray diffraction, scanning electron microscopy and fourier transform infrared spectroscopy to understand about structural properties. More literature are reviewed and finally selected TiO_2 for the doping in a polyaniline, Stoichiometric amount of aniline, hydrochloric acid, ammonium persulphate, TiO_2 are weighed and mixed in a proper proportion like 0.2M aniline, 0.2M ammonium persulphate, 1N hydrochloric acid, 5%, 10%, 20%, 30% and 40% of TiO_2 . Structural analysis of pure PANI and TiO_2 doped PANI were carried out by x-ray diffraction, scanning electron microscopy and fourier transform infrared spectroscopy. XRD reveals the crystallinity of PANI after doping TiO_2 and is confirmed by JCPDS file. Pure PANI has granular agglomerates and TiO_2

have platelet type structure, TiO_2 doped in PANI is more crystalline because grain size is increasing as polyaniline is shifting to PANI- TiO_2 . FTIR study showed the bonding and vibrations and confirms the composite of TiO_2 in PANI.

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