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SYNTHESIS OF CHITOSAN-POLYANILINE-TIO_2 POLYMER BASED NANO PARTICLES AND THEIR CHARACTERIZATION

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ABSTRACT

The TiO_2 nanoparticles doped chitosan-polyaniline was synthesised by wet chemical method. The growth of the self-assembly of polymer nanocomposite was characterised by XRD, FT-IR and SEM. The XRD, FTIR and SEM images were also studied in the project to give the perfectness in structure of titanium Oxide Particles interact in chitosan-PANI. These results indicate that chitosan-polyaniline-TiO_2 were possible to use in many research applications

KEYWORDS: PANI, FT-IR, XRD, SEM.

INTRODUCTION

Nanosized polymers has been found to be effective due to a huge specific surface area and higher reduction reactivity. To solve this issue, many polymer materials have been proposed to remove dyes such as PANI, PVP and chitosan.[1]

Chitosan is one of the abundant natural biopolymers with superior characteristics such as biocompatibility, high mechanical strength, low cost, chemically inertness, biodegradability and excellent film forming ability. Over recent years, hybrid composites material based on chitosan have been developed metal oxides, conducting polymers and metal nanoparticle due to the excellent properties of individual components and outstanding synergistic effect simultaneously. Chitosan has shown the possibility to adsorb large amounts of metal ions. This, in turn, has resulted in much interest in ascertaining its potential to remove metal ions over a large range of sewage systems and types.[2-3]

However, the usages of acid or base for the regeneration of chitosan is not always technically advantageous. The huge volume of wastewater produced from its regeneration process needs further treatment, making the process non-environmental-friendly and non-sustainable.[4-5]

PANI is also effective for adsorption of the dyes through chemical interaction, amid the several conducting polymers such as polyaniline (PANI), polypyrrole (PPy) and polythiophene (PTh) have been used for sensing of toxic gases. PANI has attracted significant attention for their due to the easy preparation, low operating temperature, low cost than other conducting polymers, redox behaviour, high environmental stability, good controllable electrical and optical properties. The potential applications of PANI have organic light weight batteries, microelectronic devices, sensors, anticorrosive coatings and excellent pseudo capacitors.[6-8] The present study is aimed at synthesis of chitosan-PANI-TiO_2 nano particles using simple wet chemical method and reagents and the characterization using FT-IR, X-ray diffraction (XRD), and Scanning electron microscopy (SEM).

EXPERIMENTAL SECTION

Materials

Chitosan (average molecular weight of 180 kDa and 90% deacetylation) was purchased from M/s South India Sea foods, Kochi, Kerala, India. Aniline monomer was distillation to remove impurities and stored below 0°C and all other chemicals likes potassium peroxodisulfate (PDS) were purchased from Merck Chemic Ltd., Mumbai, India, Titanium tri chloride (98%) was purchased LOBA Chemie (P) Ltd., Acetic acid (99.7%), Sodium hydroxide (98%) and sulphuric acid (95%) were purchased from Fischer Chemic Ltd., Chennai, were of an analytical reagent grade and used without any further purification. Millipore water was used for all experiments

PREPARATION Of MATERIALS

Preparation of PANI

5 mM CTAB solution was prepared in 1 M H_2SO_4. Then, 10 ml of 50 mM aniline solution was added to the CTAB solution and the mixture was stirred for 60 min using a magnetic stirrer while the temperature was maintained below 0 °C. Afterwards, a pre-cooled solution of 50 ml of 60 mM PDS was added dropwise to the aniline solution and stirred for 60 min. The precipitate formed was filtered, washed several times with water and acetone, and then dried.

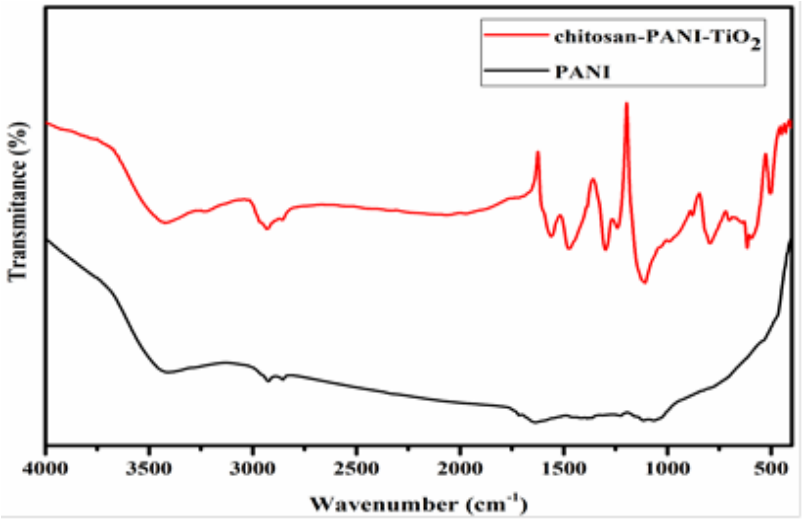
Synthesis of chitosan-polyaniline-TiO_2 (CS-PANI-TiO_2) composite

0.50 g of CS in 25 ml acetic acid (10%) was drop wise added to 30 minutes and then addition of 25 ml of titanium trichloride solution and continuously stirred for 1h. Subsequently, 10 ml of aniline was dissolved in 25 ml of 1.0 M H_2SO_4 the reaction vessel was maintain at ice bath condition (0-5°C) for 30 minutes stirring with using magnetic stirrer and followed by 50 ml PDS was quickly added into the above solution. During the addition, solution slowly turns to the emerald green colour. These were the confirmation of the polymerization from the aniline monomer. The reaction vessel was continuously stirred for 4 h by maintaining below 5 °C to ascertain the completion of reaction. Then allowed settling for 24 h at room temperature and the supernatant solution was discarded. The residue was washed several times with distilled water and filtered and dried in an oven at 80^0C for 12 h, which is designed as CS-PANI-TiO_2. Further the composite was Characterised and done adsorption studies.

CHARACTERIZATION TECHNIQUES

Powder X-ray diffraction patterns of all the freshly prepared PANI, and CS-PANI-TiO_2 nanocomposite materials were recorded in the 2θ range from 10 to 80° using a PANalytical X-pert PRO diffractometer with Cu-Kα radiation (λ = 0.154060 nm, 40 kV and 30 mA) with a step size of 0.07° s^{-1}. Fourier Transform Infrared (FTIR) spectra of all the samples were recorded in the 4000 to 400 cm−1 range using a 'Perkin-Elmer RX1' spectrophotometer with 4 cm^{-1} resolution for 20 scans. The surface morphological images were recorded by means of a field-emission scanning electron microscope (Field Emission Scanning Electron Microscopes SIGMA HV – Carl Zeiss with Bruker Quantax 200 – Z10 EDS Detector. operated at an accelerating voltage of 15 kV.

FT-IR spectral analysis



The TiO_2 is well identified by two main absorbance peaks in the (OH) stretching region (3100–4000 cm^{-1}). The broad band at 3432 cm^{-1} is associated with weakly bonded hydroxyl groups, the other band with a maximum at 850 cm^{-1} is associated to vibration of bulk TiO_2 skeletal frequency region. The 1268 cm^{-1} band is a fingerprint associated with the C–N stretching typical of PANI. The band at 1518 cm^{-1} attributed to C=N stretching of quinoid diimine unit (the oxidised form of PANI). C–C aromatic ring stretching of the benzenoid diamine unit (the reduced form of PANI) appears at 1353 cm^{-1}.[8-9]

XRD pattern analysis

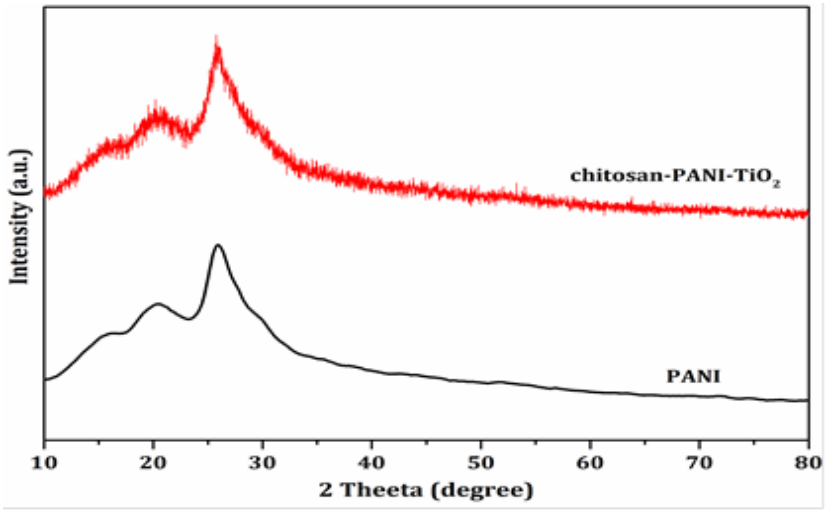


Figure 3: a show the XRD patterns of different samples. The diffraction peaks of TiO_2 fit well with JCPDS: 21-1272, corresponding to the rutile and anatase titania. Diffraction peaks at 25.78° are the (101) face of PANI, which is attributed to the periodicity parallel to the polymer chains of PANI. As the peak intensity of TiO_2 was much higher than that of PANI, the diffraction peaks of PANI could not be observed in PANI/TiO_2 composite.[10-11]

The average crystallite size of the resultant nanocomposite PANI/TiO_2 was calculated using the Scherrer formula,

$$D= 0.9\ \lambda/\ \beta^* \cos\theta$$

Where, D is the average crystallite size, λ the wavelength of the X-ray source, β the full-width at half maximum and θ is the angle of the diffracted peak position and it was found to be ≈3 nm.

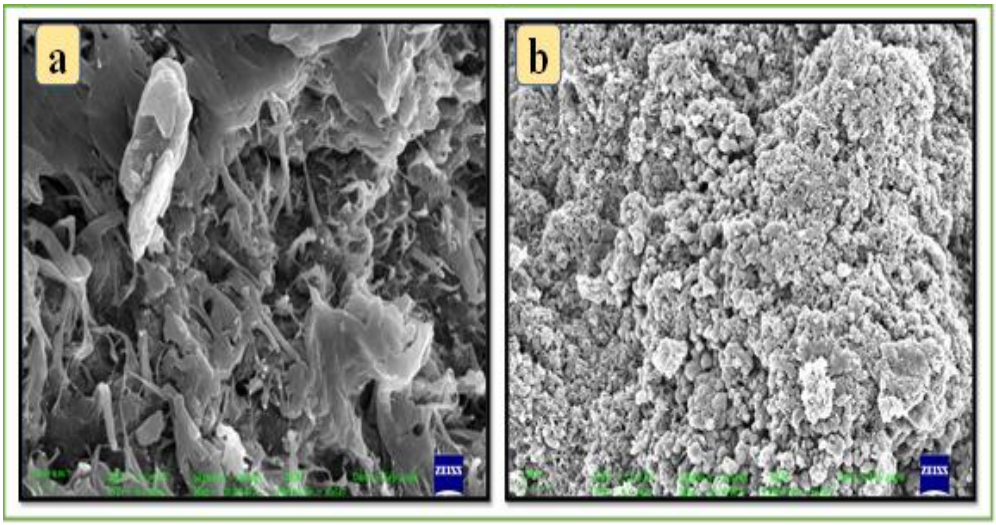


Fig. (a) SEM image of polyaniline (PANI) and (b) modified chitosan-PANI-TiO2.

The morphology of the PANI and chitosan-PANI-TiO_2 composites was analyzed by FE-SEM and the micrographs are shown in Figure 4. The morphology and structure of the products are shown in Fig. 1. From Fig. 1a, it is observed that the pure PANI shows flakes shape and the TiO2 spherical shape attached with PANI nano flakes was shown in Fig. 1 b).[12-13]

CONCLUSION

Development of synthesis of Nano materials over a range of sizes, shapes and chemical composition is an important aspect of nanotechnology. The size-dependent physical-chemical properties of nanoparticles have fascinated and inspired research activities. The present work describes some aspects of synthesis of chitosan-PANI-TiO_2 nanoparticles and characterization by FT-IR, XRD and SEM. The study can be further preceded by studying the application of the as-prepared polymer composite nanoparticles.

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