

# Synthesis and Characterizations of MnO<sub>2</sub> – Polyaniline Nanocomposite

<sup>1</sup>J. B. Bhaiswar , <sup>2</sup>D.P.Meghe

<sup>1</sup>Department of Physics, Nagpur Institute of Technology, Nagpur

<sup>2</sup>Department of Physics, Nagpur Institute of Technology, Nagpu

<sup>1</sup>jitendrabhaiswar@gmail.com

<sup>2</sup>dhira941983@gmail.com

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## Abstract:

The Polyaniline and Its Nanocomposite with MnO<sub>2</sub> were synthesized in aqueous sulphuric acid solutions by chemical oxidation at room temperature. The Prepared nanocomposite products were characterized by Fourier Transform Infra red spectroscopy (FT-IR), Transmission electron Microscopy (TEM) and Thermo gravimetric analysis (TGA). Fourier transform absorption peak at 443 cm<sup>-1</sup> indicate the presence MnO<sub>2</sub> nanoparticle, in the the surface of bulk Polyaniline matrix. Transmission Electron Microscopy clearly indicated the particle size and their morphology. TGA peak of Bulk PANI and Nanocomposites shoes that Nanocomposite has better thermal stability as compare to Bulk Polyaniline

**Keywords-** Polyaniline, synthesis, Morphology, TEM, TGA

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## 1. INTRODUCTION:

The synthesis and different characterizations of pure Polyaniline and Nanocomposite with MnO<sub>2</sub> nanomaterials which has predictable a immense contract of consideration in the last two decades [1]. Polyaniline (PANI) exists in a variety of chemical and physical properties [2–5]. Polyaniline is one for the most part of capable conducting polymers due to its high conductivity, easy preparation, good environmental stability and large variety of applications [6]. The filling or encapsulation of nanoparticles in Polyaniline, may lead to termination of the growth of the nanoparticles by controlling of their nucleation by the Polyaniline. Polyaniline has a simple and reversible acid/ base doping/dedoping chemistry, enabling control over properties such as solubility, thermal stability, electrical conductivity and optical activity [7]. Synthesis of Polyaniline based nanocomposites has led to a number of potential applications in electronic and optical devices, catalysis and analytical sensors etc. Polyaniline has twisted nanocomposite with dissimilar inorganic materials likes PANI/PbS, PANI/CdS, PANI/Poly (vinyl chloride) etc. [8–12]. The Synthesis and characterization of PANI nanocomposites has been the subject of considerable interest recently [13]. It has been bring into being that the original materials show signs of improved Thermal, electrical and mechanical properties due to the synergistic effect of the organic and inorganic components. . MnO<sub>2</sub> is an attractive material from a number of points. It show the quantum imprisonment effects in an experimentally available size range [14]. Moreover, the broad range of structural variety in the

nano-system this material became a promising candidate in the field of nanotechnology and opened up new possibilities for the fabrication of high concert devices based on these nanostructures [15].

A numeral of investigate works encompass published in recent times regarding the synthesis and morphological study of PANI/MnO<sub>2</sub> nanocomposites using sol-gel methods [9]. On the other hand, to the best of our knowledge, no works have been reported on preparing PANI/MnO<sub>2</sub> nanocomposites via chemical oxidation technique with (0.4N) H<sub>2</sub>SO<sub>4</sub> and APS as oxidant. Under these circumstances, it is worthwhile to conduct an explorative investigation on preparing the nanocomposites by oxidation technique. In this paper, we describe our initial work, which has been utilized for synthesis and characterizations study of PANI/MnO<sub>2</sub> nanocomposite with thermal stability.

## 2. MATERIALS AND METHODS:

### 2.1. Production of Polyaniline via chemical route:

Production of Polyaniline was conceded by the chemical route of aniline in the presence of sulphuric acid with APS (Ammonium per-sulphate) as oxidant. Both (0.4 mol) APS and 0.4mol H<sub>2</sub>SO<sub>4</sub> is added in 100 ml distilled for 2 hours in a four-neck round bottom reaction flask. The resolution of APS in H<sub>2</sub>SO<sub>4</sub> was further added drop-wise in the mixture of aniline with a magnetic stirrer for 3 hours to initiate polymerization. The product was presently agreed at room temperature for 6-7 hours. A precipitation of PANI with green color obtained. The synthesized Polyaniline were recovered from the polymerization mixture and washed with deionized water repeatedly until the washing liquid was completely colorless.

### 2.2. Synthesis of PANI-MnO<sub>2</sub> Nanocomposites:

The synthesis method of PANI/MnO<sub>2</sub> nanocomposite is same to that of bulk PANI. Different amount of MnO<sub>2</sub> was dispersed into the APS solution and stirred for 1 hour prior to the addition of aniline. Aniline (0.4 mol) stirred with 0.4mol H<sub>2</sub>SO<sub>4</sub> in 100 ml of distilled water were added drop-wised using burette into the APS-MnO<sub>2</sub> solution and stirred vigorously to form homogeneous dispersion.

### Characterizations;

Fourier Transform Infra Red (FTIR) spectroscopy (Model: Perkin Elmer 120) ° pure f PANI and nanocomposite was studied in the frequency range of 400–4000 cm<sup>-1</sup>. TGA thermograms of all samples were recorded on Perkin-Elmer Diamond TGA/DTA in argon atmosphere at a heating rate of 10°C/ min. TGA profile were taken over the temperature range of 30-800°C. TEM micrographs of were taken on Transmission Electron Microscope PHILIPS model-CM200 with resolution 2.4Å.

## 3. RESULT AND DISCUSSION;

### 3.1. FT-IR SPECTRA:

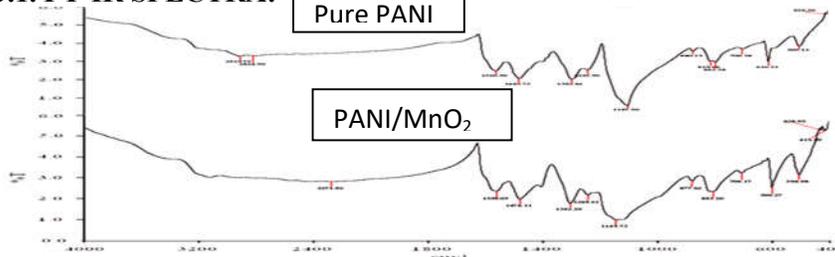


Fig I. FT-IR spectra pure PANI, & PANI/MnO<sub>2</sub> nanocomposite.

Polyaniline samples observed specific stretching vibrations for the different structural forms. The precise groups of PANI and their IR vibration frequencies from the literature are given below:

1. The frequency band at  $2900\text{ cm}^{-1}$  is assigned to aliphatic C–H stretching mode.
2. The Band at  $1587\text{ cm}^{-1}$  Quinonoid ring at Benzenoid ring -  $1510\text{ cm}^{-1}$
3. C-N stretching -  $\sim 1380\text{ cm}^{-1}$
4. The absorption band at  $1150\text{ cm}^{-1}$  for benzenoid and quinonoid unit
5. The absorption band at  $1100\text{ cm}^{-1}$  for stretching mode.
6. Fragile absorption band at  $425\text{ cm}^{-1}$  for  $\text{MnO}_2$  in pure PANI surface.

IR study undoubtedly indicates that  $\text{MnO}_2$  are definitely included into the bulk polymer environment.

### 3.2. TEM MICROGRAPH:

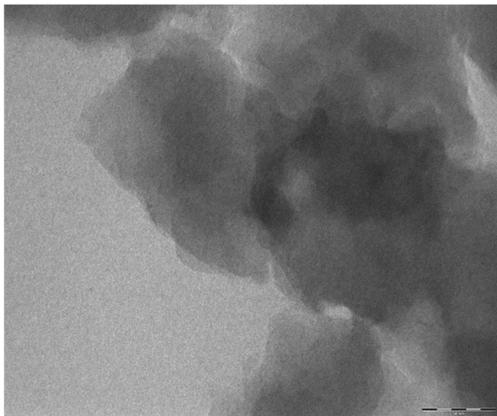


Fig.2.A. TEM OF PURE PANI

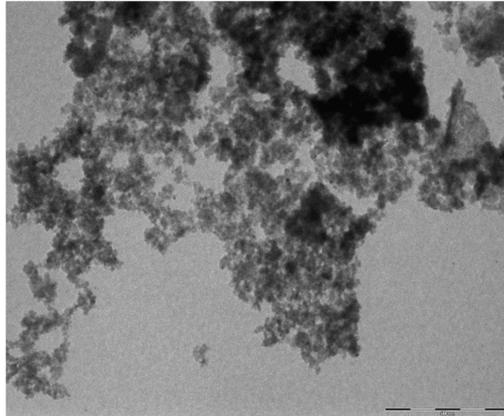
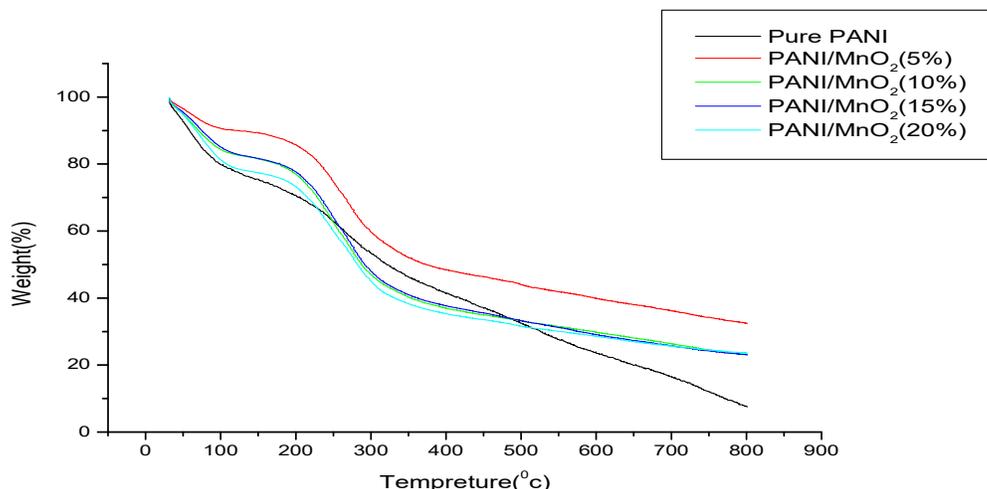


Fig.2.B. TEM OF PANI/ $\text{MnO}_2$  Nanocomposite.

The fig 2. A and B. indicated that PANI/  $\text{MnO}_2$  nanocomposite is in nanoscale with particle size 15 nm. The Photograph of bulk PANI in the 100nm scale and PANI/ $\text{MnO}_2$  nanocomposites is in 55nm scale indicated  $\text{MnO}_2$  nanoparticle well dispersed in Nanocomposite.

### TGA THERMOGRAPH:

The TGA measurements of Bulk Polyaniline and their nanocomposites show that  $\text{MnO}_2$  nanoparticles with different wt percentages get good thermal stability as compare to pure Polyaniline. Fig. 3. Indicate the TGA curve of pure PANI and Its nanocomposites with different wt percentages. TGA curve of complimentary PANI demonstrate around 94 % in weight loss when heated from room-temperature up to  $800\text{ C}^\circ$ . The disintegration thermograms of pure PANI exhibit two stage weight-loss behaviors. The first step at  $40$  to  $130\text{ C}^\circ$  which is due to ejection of water molecules and the evaporation of residual solvents. The weight loses in the first stages around stage is about 12%. The second step wt loss occurred at  $200$  - $800\text{ C}^\circ$  shows disintegration of the polymer due to impurities, and it is upto 94%. The thermograms of PANI/  $\text{MnO}_2$  nanocomposites also demonstrate two stage weight-losses. The first step is due to removal of water molecules, while the second step is a corrosion step. The weight lost at all stages for nanocomposites is less than bulk PANI indicated Polyaniline nanocomposite is thermally more stable as bulk Polyaniline .A exceptional characteristic of PANI nanocomposites which is absent in bulk PANI salt is that they depart at the back a considerable amount of scum even after  $800^\circ\text{C}$  which may be the respective metal oxide which are undegradable below  $800^\circ\text{C}$ , while PANI shows almost 94% weight loss at  $800^\circ\text{C}$ .



**Fig.3.TGA thermograph of PANI and PANI/MnO<sub>2</sub>**

#### 4. CONCLUSIONS:

Polyaniline-coated MnO<sub>2</sub> nanocomposites have been synthesized via chemical oxidation technique. The concentration of the MnO<sub>2</sub> in nanocomposites was low as a very weak vibration absorption peak of Mn–O bonding at 420 cm<sup>-1</sup> is observed in the FTIR spectrum. The average size of the nanocomposites is estimated to be 55 nm as observed in the TEM micrograph. The thermal stability for the nanocomposites has been better as major mass losses occurred at higher decomposition temperature as compared to the pure polymer in the TGA profile.

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