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Structural and Morphological Analysis of Conducting Polyaniline Prepared Via Interfacial Polymerization

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***Abstract* - Low cost, high conductivity, stability, and favorable physicochemical properties render polyaniline (PANI) a widely used conducting polymer in various industrial applications. Interfacial polymerization (IP) has emerged as the preferred method for the synthesis of PANI. The study aimed to analyze PANI samples synthesized via interfacial polymerization using X-ray diffraction (XRD), ultraviolet–visible (UV–Vis) spectroscopy, Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM). XRD revealed the crystalline nature of PANI, while FTIR and UV–Vis analyses confirmed the formation of conducting PANI. Furthermore, SEM demonstrated significant morphological variations in PANI emeraldine salt.**

Keywords - polyaniline; interfacial polymerization; x-ray diffraction; scanning electron microscopy; ultraviolet–visible spectroscopy; Fourier transform infrared spectroscopy

I. INTRODUCTION

Conductive polymers constitute an important class of polymers that exhibit a range of unique properties, including optical activity, electrochemical behavior, mechanical strength, electrical conductivity, corrosion resistance, and stability, and have been extensively studied over the past few decades. These materials have attracted considerable attention due to their wide range of applications, such as biosensors [1,2], electrochemical displays [2,3], corrosion protection [2,4], rechargeable batteries [2,5], solar cells [6,7], diodes [7,8], solar energy conversion, electrochemical sensors, and capacitors [6,7,9–11].

Due to low cost, high electrical conductivity, environmental stability [2,12], good redox and thermal properties [13,14], reversible acid–base chemistry in aqueous solutions, and ease of synthesis [2,15], polyaniline (PANI) is among the widely used conducting polymers in various industrial applications today. Although there are several methods for the synthesis of PANI, including electrochemical synthesis, solution polymerization, electropolymerization, and UV polymerization, interfacial polymerization (IP) has emerged as a novel, reliable, and effective technique for the chemical synthesis of bulk, high-quality, water-soluble conducting PANI due to its ease of preparation, potential for large-scale production, and rapid reaction. Furthermore, the usage of an anionic polymer in IP results in a bundled structure that exhibits a strong affinity toward cationic polymers [2].

IP involves the step-growth polymerization of two reactive monomers or agents that are dissolved separately in two immiscible phases, with the reaction occurring at the interface of the two liquids [16,17]. This method facilitates the synthesis of polymers at low temperatures with minimal side reactions and eliminates the need for catalysts or phase transfer agents [17,18]. IP is a nontemplated approach that facilitates the formation of monomer–anion (oligomer–anion) aggregates due to the high local concentrations of monomers and dopant anions at the liquid–liquid interface [19]. These aggregates function as initiation centersduring polymerization, leadingto the formation of powders characterized by a fibrous structure [20].

PANI salts have been synthesized using various dopants that improve their solubility in common organic solvents, thereby enhancing their processability. MacDiarmid and Epstein reported several experimental observations supporting the concept of “secondary dopant” of *m*-cresol and many other phenol derivatives, which expand PANI molecules from initial coil-like conformations, modifying the crystalline structure of PANI emeraldine salt (ES) [21]. However, relatively few studies have investigated the influence of secondary dopant acids on the electronic transition, morphology, size, and crystalline structure of PANI ES.

In this work, a simple experimental method is employed to synthesize a crystalline form of PANI ES and to investigate the influence of a secondary dopant, acidic *m*-cresol, at different concentrations on the morphology, size, and crystalline structure of PANI ES. X-ray diffraction (XRD), ultraviolet–visible (UV–vis) spectroscopy, Fourier-transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM) are used to analyze PANI and *m*-cresol-doped PANI.

II. MATERIALS AND METHODS

A. Materials

Ammonium peroxydisulfate, aniline monomer, *m*-cresol, toluene, and hydrochloric acid were purchased from a chemical manufacturer.

B. Synthesis

A total of 9 ml of aniline was dissolved in 10 ml of toluene and subsequently combined with 100 ml of water to form the organic phase. Meanwhile, 11.8 g of ammonium peroxydisulfate was added to a 250 ml beaker containing an aqueous solution prepared by mixing 10 ml of hydrochloric acid with 100 ml of water. The two solutions were then carefully transferred to a beaker to generate a clear interface

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between the aqueous and organic phases. Green PANI formed at the interface after 3–5 min and diffused into the aqueous phase, which was homogeneously filled with dark-green PANI after 24 hr. The ionic liquid (organic salt and liquid) appeared colored due to the formation of aniline oligomers. The mixture was then filtered, and the solid residue was collected and repeatedly washed with acetone and water to remove unreacted chemicals and aniline oligomers until the washings were colorless.

The obtained solid was subsequently doped with *m*-cresol to increase its conductivity. Three different quantities (5 ml, 10 ml, and 15 ml) of *m*-cresol were mixed with equal quantities (8 gm) of PANI samples. The resulting solid samples were vacuum dried in an oven at 80°C for 10 hr to obtain acid-doped PANI.

C. *Characterization Techniques*

The synthesized PANI and *m*-cresol–doped PANI were characterized using UV–Vis absorption spectroscopy with a Varian Cary 5000 UV-Vis-NIR spectrophotometer in the range 234–766 nm. FTIR spectra were recorded using a Thermo Nicolet Avatar 370 FT-IR spectrometer by the KBr pellet method in the range 400–4000 cm^{-1}, with a resolution of 4 cm^{-1}. X-ray diffraction patterns were obtained using a Bruker AXS D8 Advance diffractometer with Cu (wavelength = 1.5406 Å). The diffractometer was operated at 40 kV and 35 mA. A scanning step of 0.02° in 2θ with a dwell time of 59.7 s per step was used. Surface imaging was examined using SEM with a JEOL JSM-6390LV equipped with a tungsten filament.

III. RESULTS AND DISCUSSION

A. *Reaction and Mechanism*

IP was performed to obtain green PANI at the interface (Figure 1). As the reaction progressed, the organic phase developed a darker color, indicating the completion of the reaction. Figures 2(a) and (b) show pure PANI and PANI doped with different concentrations of *m*-cresol (5 ml, 10 ml, and 15 ml).

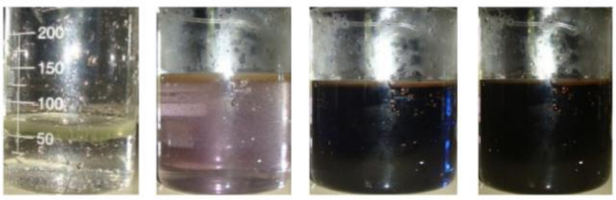

Fig. 1. Interfacial polymerization of PANI ES.

Fig. 2. (a) Pure PANI. (b). PANI doped with different concentrations of *m*-cresol (5 ml, 10 ml, and 15 ml).

B. *XRD Analysis*

Figures 3(a)–(d) show the XRD patterns of Samples 1–4, respectively. Normally, the ES form of conducting PANI exhibits an amorphous nature. However, the XRD pattern obtained for all the prepared samples exhibited well-ordered peaks, which indicate the semicrystalline nature of PANI. This crystalline nature is associated with the conducting behavior of PANI and arises due to the presence of benzenoid and quinoid rings. The Debye–Scherrer equation was used to calculate the crystallite size of the samples:

$$D = [0.89\ \lambda]/[\beta\ \mathrm{Cos}\ \theta],$$

where β is the full width at half maximum and λ is the wavelength of X-ray radiation.

The calculated crystalline sizes were 26.91, 27.27, 26.91, and 25.91 nm for Samples 1–4, respectively. All the samples exhibited a sharp peak at 11.6°, which corresponds to the d-spacing value of 7.6 Å, characteristic of the conducting form of aniline. Additionally, the peaks observed in the range of 29–35° for the four samples correspond to a d-spacing value of approximately 2 Å, which indicates that portions of the samples are rigid and well-ordered, indicating the doped state of the PANI.

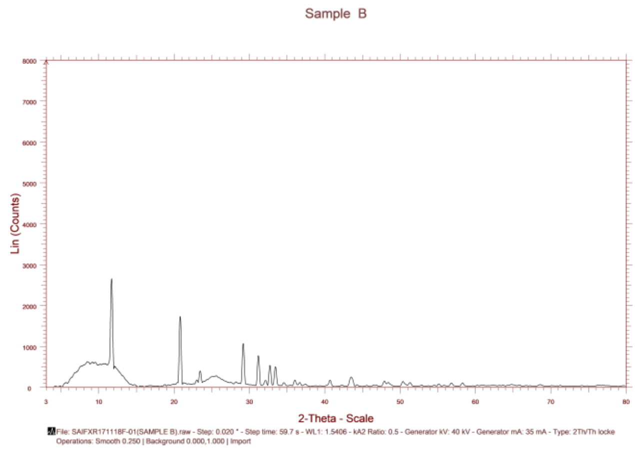

Fig. 3(a). XRD spectrum of Sample 1 (Pure PANI).

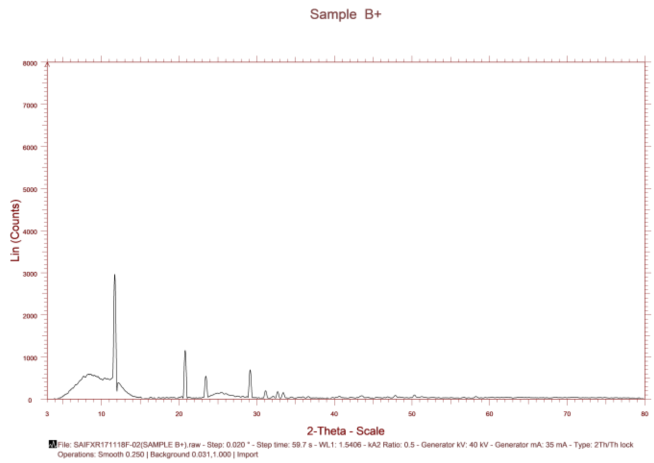

Fig. 3(b). XRD Spectrum of Sample 2 (8 gm of PANI doped with 5 ml of *m*-cresol).

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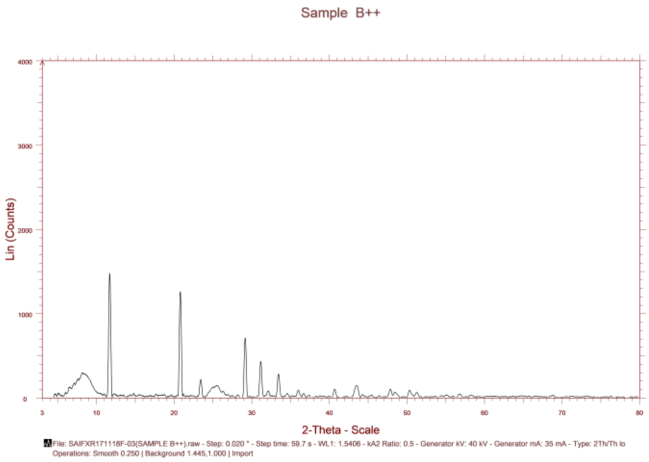

Fig. 3(c). XRD Spectrum of Sample 3 (8 gm of PANI doped with 10 ml of *m*-cresol).

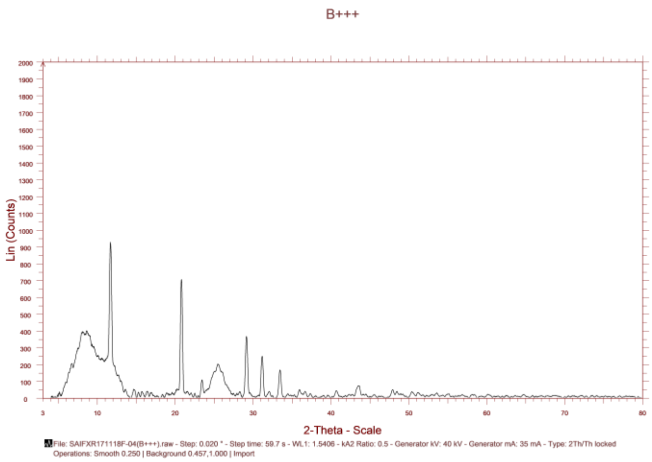

Fig. 3(d). XRD Spectrum of Sample 4 (8 gm of PANI doped with 15 ml of *m*-cresol).

C. *FTIR Analysis*

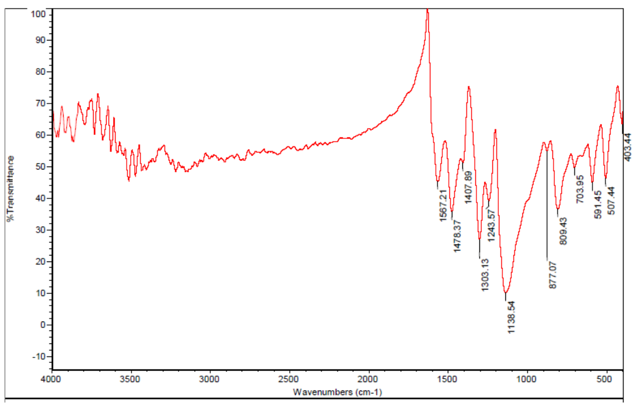

Fig. 4(a). FTIR Spectrum of Sample 1 (Pure PANI).

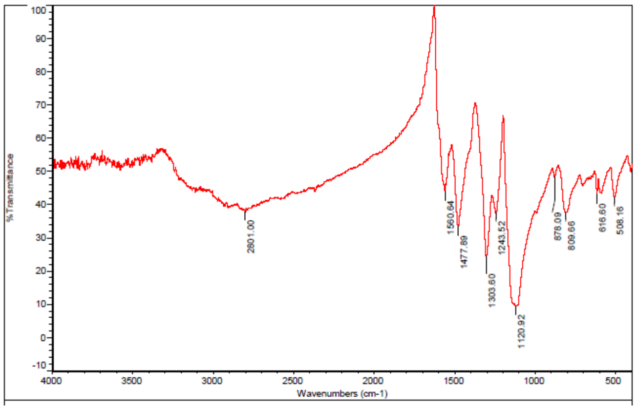

Fig. 4(b). FTIR Spectrum of Sample 2 (8 gm of PANI doped with 5 ml of *m*-cresol).

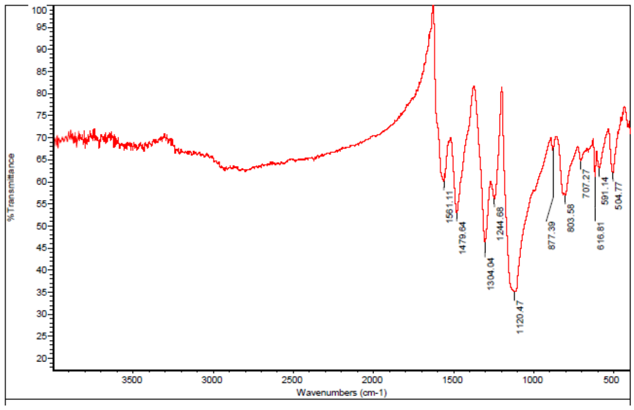

Fig. 4(c). FTIR Spectrum of Sample 3 (8 gm of PANI doped with 10 ml of *m*-cresol).

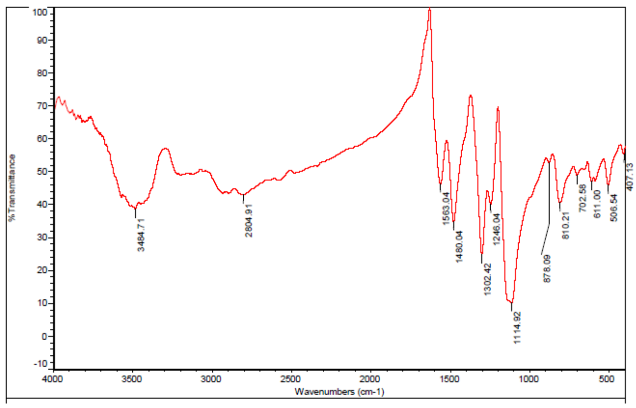

Fig. 4(d). FTIR Spectrum of Sample 4 (8 gm of PANI doped with 15 ml of *m*-cresol).

Figures 4(a)–(d) show the FTIR spectra of Samples 1–4, respectively. The various peaks observed in all four samples are consistent with the ES form of doped PANI. The peaks at 1567.21, 1560.64, 1561.11, and 1563.04 cm^{-1} indicate C–N stretching of the quinoid ring. The prominent peaks at 1478.37, 1477.89, 1479.64, and 1480.04 cm^{-1} correspond to the C–C stretching of the benzenoid ring. The peaks observed at 1303.13, 1303.60, 1304.04, and 1302.42 cm^{-1} confirm the doped state of PANI. Furthermore, the peaks observed at 1243.57, 1243.52, 1244.68, and 1264.04 cm^{-1} are attributed to the C–N stretching, while those at 809.43, 809.66, 803.58, and 810.21 cm^{-1} indicate the out-of-plane C–H bending.

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D. UV Analysis

Figures 5(a)–(d) show the absorption spectra of Samples 1–4, respectively. The peaks observed in all four samples confirmed the conducting form of PANI. UV–Vis spectra of the prepared samples exhibited peaks in the ranges 327–330 nm and 667–679 nm. The first absorption band is attributed to π–π* electron transition. The second absorption band arises due to the transition of π to the polaron in the conducting salt form of PANI. The bands in the range 667–679 nm indicate the doping level of the prepared samples. The observed peaks indicate the formation of a polaron lattice, which reflects the level of protonation in the PANI samples.

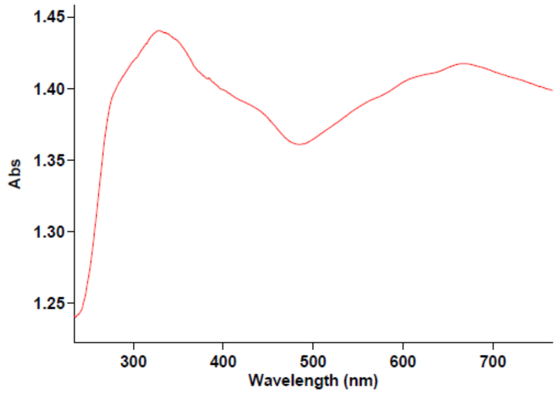

Fig. 5(a). UV Spectrum of Sample 1 (Pure PANI).

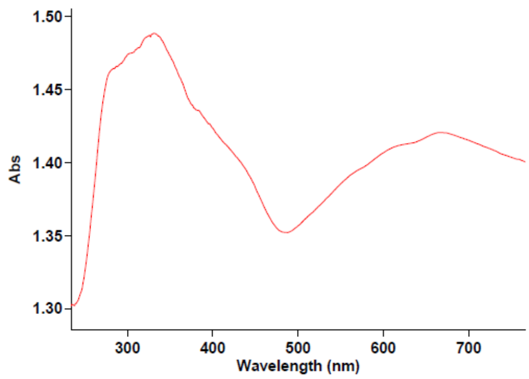

Fig. 5(b). UV Spectrum of Sample 2 (8 gm of PANI doped with 5 ml of *m*-cresol).

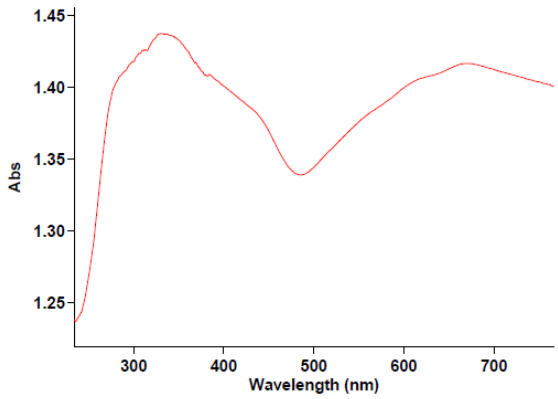

Fig. 5(c). UV Spectrum of Sample 3 (8 gm of PANI doped with 10 ml of *m*-cresol).

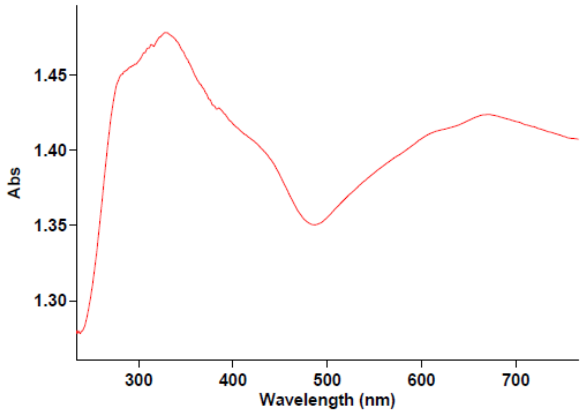

Fig. 5(d). UV Spectrum of Sample 4 (8 gm of PANI doped with 15 ml of *m*-cresol).

Figures 6(a)–(d) show the SEM images of Samples 1–4, respectively. Significant variations in surface morphology of the sample are evident from the SEM images. The images show high crystallinity, indicating the conducting state of the prepared samples. The presence of highly ordered and rigid regions in the SEM images further indicates the crystalline nature of the samples.

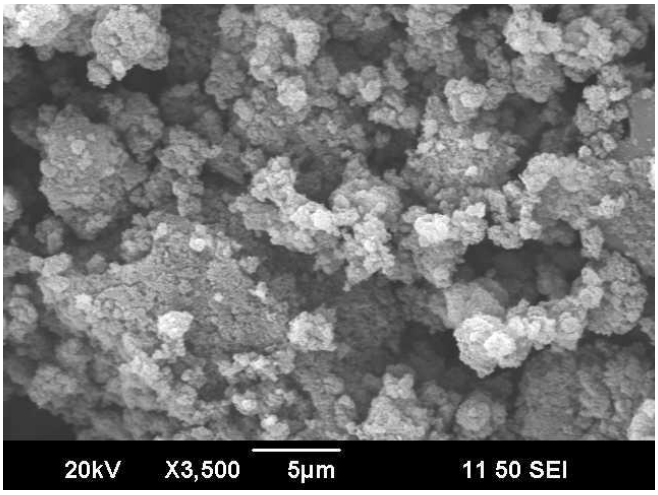

Fig. 6(a). SEM of Sample 1(Pure PANI).

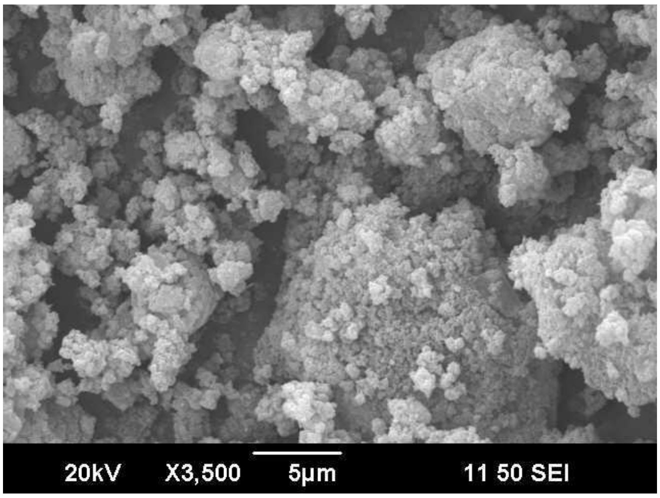

Fig. 6(b). SEM of Sample 2 (8 gm of PANI doped with 5 ml of *m*-cresol).

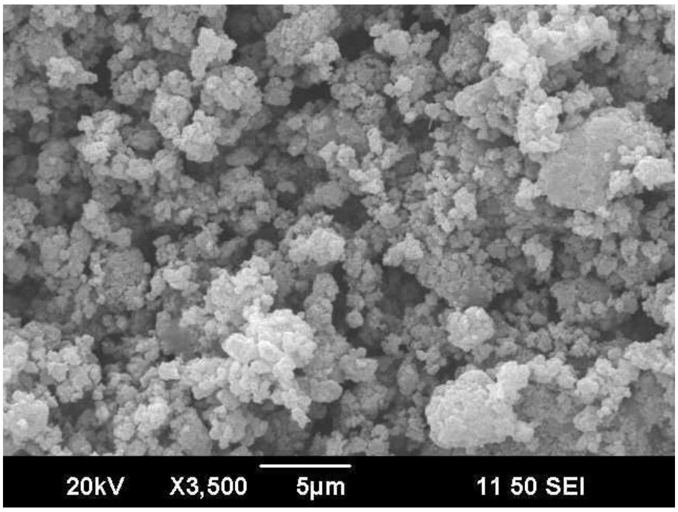

Fig. 6(c). SEM of Sample 3 (8 gm of PANI doped with 10 ml of *m*-cresol).

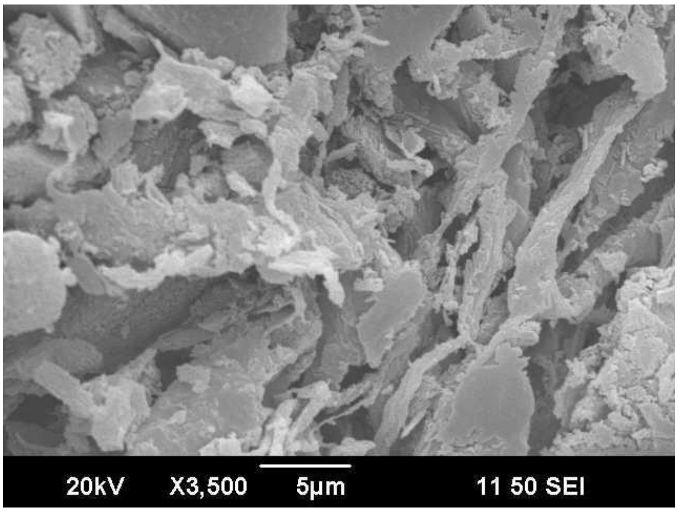

Fig. 6(d). SEM of Sample 4 (8 gm of PANI doped with 15 ml of *m*-cresol).

IV. CONCLUSION

In this work, the conducting form of PANI was successfully synthesized via IP method. The synthesized polymers were characterized using XRD, FTIR, UV–Vis, and SEM techniques. The XRD patterns obtained in this study reveal that the prepared samples are crystalline in nature, which is characteristic of conducting polymers. The characteristic peaks obtained in FTIR analysis confirm the chemical structure of PANI. Furthermore, the UV–Vis spectra confirm the formation of conducting PANI. SEM images reveal significant variations in the morphology among the samples, which are attributed to the variations in *m*-cresol content in different samples.

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