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Structural and Morphological Characteristics of PPY-PANI Composite Thin Films by Solution Mixing Method

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Abstract
In this work, the structural and morphological characteristics of pure polypyrrole (PPY), polyaniline (PANI), and their blends in varying ratios were studied. The X-ray diffraction (XRD) analysis revealed distinct diffraction peaks corresponding to the characteristic structures of PPY, PANI, and their blends (PPy-PANI), indicating successful composite formation and phase interactions. Field-emission scanning electron microscopy (FE-SEM) results showed porous, interconnected morphologies in composites, which enhance surface area and gas adsorption sites compared to pure polymers. Such enhancement is very useful for gas sensing applications of PPY/PANI thin film composites at elevated temperatures.

Keywords: Polyaniline; Polypyrrole; Thin films; Composites; Crystalline structure; Morphology
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1. Introduction

Polyaniline (PANI) stands out as one of the most promising and extensively studied material from the family of intrinsically conducting polymers (ICPs) due to its unique properties, environmental stability, and relatively straightforward synthesis. Often described as an organic metal, PANI can transition from an insulating state to a highly conductive one through a process known as doping, making it a versatile material for a wide range of advanced applications. PANI's molecular structure consists of reduced (benzoid) and oxidized (quinoid) repeating units [1,2]. Its most remarkable characteristic is its tunable electrical conductivity, which can span over 15 orders of magnitude (from ~10^{-10} S/cm to over 10^{5} S/cm) depending on its oxidation state and protonation level. This behavior is governed by its unique doping mechanism, which involves protonic acid doping rather than the redox doping common in other polymers [3,4]. The emeraldine base form of PANI is insulating, but when treated with acids like hydrochloric acid, it converts to the conductive emeraldine salt. Beyond its electrical properties, PANI exhibits good environmental stability, retaining its conductivity under ambient conditions for extended periods. It is generally insoluble in common solvents, which can be a processing challenge, though this can be overcome using functionalized protonic acids or specific solvents like N-methyl-2-pyrrolidone (NMP) [5]. PANI films and coatings are also known for their electrochromic properties, changing color from light yellow (leucoemeraldine base) to blue (emeraldine salt) to violet (pernigraniline base) as the oxidation state changes [6,7].

Polypyrrole (PPY) belonging to the pantheon family of conducting polymers, has emerged as a workhorse material, prized particularly for its high electrical conductivity and exceptional environmental stability [8]. Alongside polyaniline, it is one of the most extensively researched organic conductors, bridging the gap between traditional polymers and metals for a host of advanced technological applications [9,10]. PPY is characterized by a backbone of interconnected five-membered pyrrole rings. In its neutral state, it is only semi-conductive. However, upon oxidative doping—which removes electrons from the polymer chain—it becomes highly conductive, achieving values typically in the range of 10 to 100 S/cm, with some reports of even higher conductivity under optimized conditions. This doped state involves the formation of charge carriers (polarons and bipolarons) along the conjugated chain, which are responsible for charge transport [11,12]. A key advantage of PPY over some other conducting polymers is its outstanding stability in air and water, allowing it to retain its conductivity for long periods [13]. PPY is typically obtained as an intractable, insoluble black powder or film, presenting a significant challenge for processing. To overcome this, researchers often synthesize PPY as a colloidal dispersion or incorporate dopant anions that improve solubility, or combine it with other processable polymers to form composites. PPY is also biocompatible, a property that underpins its significant use in biomedical applications [14,15].

The combination of polyaniline (PANI) and polypyrrole (PPY) into blends and composites represents a powerful strategy in materials science, aiming to synergize the advantageous properties of both conducting polymers. While individually impressive, PANI and PPY have their own limitations, such as PANI's need for protonic acid doping and PPY's processability challenges. By creating PANI/PPY systems, researchers can develop materials with enhanced electrical, electrochemical, and mechanical properties for more demanding applications [16-18].

The most common method for synthesizing PANI is the chemical oxidative polymerization of aniline monomer. This is typically carried out in an acidic aqueous medium (e.g., 1M HCl) using ammonium persulfate ($(NH_4)_2S_2O_8$) as the oxidant. The reaction is exothermic and proceeds at low temperatures (0-5°C) to control the molecular weight and polydispersity. The resulting powder, the emeraldine salt form, is then often de-doped with a base like ammonium hydroxide to yield the processable emeraldine base, which can later be re-doped as needed [19,20]. Electrochemical polymerization is another important method, particularly for creating thin films directly onto electrode surfaces. By applying a constant potential or cycling the potential of an electrode immersed in an acidic aniline solution, a adherent and conductive PANI film is deposited. This method allows for precise control over film thickness and morphology [21]. Similarly, to prepare PPy polymers, the most common synthesis route is the chemical oxidative polymerization of pyrrole monomer. This is typically performed in aqueous solution using oxidants such as iron(III) chloride ($FeCl_3$) or ammonium persulfate. The reaction is relatively straightforward and can be carried out at room temperature, yielding a black precipitate of doped PPY. A significant advantage of this method is the ability to polymerize pyrrole directly onto insulating substrates or within porous matrices, allowing for the creation of composite materials [22,23]. Electrochemical polymerization is the other primary method, especially for creating uniform, adherent thin films. It involves applying an anodic potential to a working electrode immersed in a solution containing the pyrrole monomer and a supporting electrolyte salt. The electrolyte's anions (e.g., Cl^-, NO_3^-, ClO_4^-, or larger polymeric anions) are incorporated into the growing film as dopants to balance the charge. This method offers excellent control over film thickness, morphology, and doping level [24,25].

On the other hand, the preparation of PPy-PANI materials can be achieved through several methods, each yielding a distinct morphology. Physical blending is a straightforward method that involves mechanically mixing pre-synthesized PANI and PPY powders. However, due to the intrinsic insolubility of both polymers, this approach often results in poor interfacial contact and inhomogeneous mixtures, leading to suboptimal electrical and mechanical properties. It is less common than chemical or electrochemical methods [26,27]. The *in-situ* chemical polymerization is a highly effective and widely used technique. It typically involves a two-step process: first, one polymer (e.g., PANI) is synthesized. Then, the second monomer (e.g., pyrrole) is polymerized within the matrix of the first polymer. For instance, a PANI network can be created, and PPY can be grown on its surface and within its pores. This method promotes intimate contact at the molecular level, creating a composite with a cohesive, interpenetrating network that facilitates charge transport. The order of polymerization (PANI-then-PPY or PPY-then-PANI) can influence the final composite's morphology and properties. As well, electrochemical co-deposition method allows for the direct synthesis of a composite film on an electrode surface [28,29]. The electrolyte solution contains both aniline and pyrrole monomers. By applying a suitable potential, both monomers oxidize and polymerize simultaneously, forming a homogeneous, blended film. The ratio of the two polymers in the final film can be controlled by adjusting the monomer concentration ratio in the solution. This technique is excellent for producing high-quality, adherent thin films for sensors and electrodes [30]. PANI and PPY are often combined together within a third, host matrix to create ternary composites. A common example is embedding both polymers into an inorganic framework like graphene oxide, carbon nanotubes, or metal oxides. The conductive carbon or metal oxide provides a high-surface-area scaffold, while the combination of the two polymers can lead to improved stability and electrochemical performance compared to single-polymer composites [31,32].

The unique combination of properties makes PANI, Ppy, and their blends and composites suitable for diverse applications. PANI-based coatings provide exceptional corrosion protection for metals like steel. They act as a physical barrier and, more importantly, induce the formation of a passive metal oxide layer through anodic protection, significantly slowing the corrosion rate [33,34]. Similar to PANI, PPY can be used in smart anticorrosion coatings for metals like steel and aluminum. The polymer acts as a physical barrier and can provide anodic protection, shifting the metal's potential into its passive region and drastically reducing the corrosion rate [35]. The combination of PANI's excellent anodic protection capability and PPY's high stability can result in coatings that offer more robust and long-lasting corrosion inhibition for metals [36]. The composite coating can provide a more effective physical

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barrier and a more stable passivating layer than single-polymer coatings [37].

PANI is highly sensitive to its chemical environment. Changes in gas concentration, pH, or the presence of specific biological molecules can alter its conductivity. This makes it ideal for chemical sensors (e.g., for detecting ammonia, humidity) and biosensors (for glucose, DNA, etc.). The conductivity of PPY is highly sensitive to its chemical environment. This makes it an effective transducing material for chemical and biological sensors. It can detect gases (like ammonia), vapors, and specific biomolecules through measurable changes in its electrical resistance. PANI/PPY composites can be engineered for enhanced sensor sensitivity and selectivity. The blended material can possess a richer diversity of interaction sites for analyte molecules. For instance, in gas sensing, the composite can show a superior response to ammonia or humidity compared to its individual components. The tunable surface chemistry also makes these composites promising for biosensing applications [38-40].

PANI is a key material in supercapacitors and advanced batteries. Its fast and reversible redox reactions enable high charge-discharge rates and high specific capacitance, making it an excellent candidate for pseudocapacitive energy storage. PPY's rapid redox switching makes it a suitable electrode material for supercapacitors (electrochemical capacitors), where it provides high power density and good cycle life through pseudocapacitive charge storage. High-Performance supercapacitors is a primary application of the combination of PANI and PPY, which can create an electrode material that leverages the high pseudocapacitance of both polymers. The composite often exhibits a broader working potential window, higher specific capacitance, and better cycling stability than electrodes made from either polymer alone, due to improved mechanical integrity and more efficient charge distribution [41-43].

The ability of PANI to be switched electrochemically allows for the controlled release of drugs. Its biocompatibility and electrical activity also make it a candidate for neural interfaces and tissue engineering scaffolds. PPY's biocompatibility and electrical activity make it an ideal material for neural interfaces. It is used in neural probes and nerve guidance conduits to stimulate nerve regeneration or record neural signals. It is also researched for controlled drug delivery systems, where an electrical stimulus triggers the release of therapeutic agents stored within the polymer matrix [44-46].

Additionally, PANI can be processed into conductive inks and paints for printing flexible circuits, RFID tags, and antistatic coatings for packaging materials and display screens. As an electroactive polymer, PPY can change volume upon oxidation and reduction. This property is harnessed in soft actuators, often called "artificial muscles," which can contract and expand in response to small electrical voltages. These are promising for robotics, microfluidics, and biomedical implants [47,48].

The synergistic effects in PPy-PANI blends and composites open doors to superior performance in electromagnetic interference (EMI) shielding. The high electrical conductivity achievable with PANI/PPY composites makes them effective lightweight absorbers of electromagnetic waves. The multiple interfaces within the composite promote the absorption and dissipation of microwave radiation, which is crucial for protecting electronic devices from interference [49,50].

This study aims to synthesize and characterize PPy-PANI composites with different composition ratios and to investigate how their structural, morphological, and crystalline features. X-ray diffraction (XRD) and field-emission scanning electron microscopy (FE-SEM) were used to examine crystallinity and surface morphology. The goal is to correlate specific microstructural changes such as surface roughness, lamellar features, and crystallite size.

2. Experimental Part

Sigma Aldrich supplied polypyrrole (PPY) powder with a purity of 98%, polyaniline (PANI) powder with a purity of 99.9%, and dimethyl sulfoxide (DMSO) with a purity of 99.6%.To prepare a homogenous solution, 50 mg of PPy was dissolved in 10 ml of DMSO, and 50 mg of PANI was dissolved in 10 ml of DMSO, left at room temperature for 2 days while continuously stirred by a magnetic stirrer at 200-400 rpm. Used a 0.45 µm filter to filter it. A uniform black solution was produced. The PPY and PANI prepared solutions were mixed with a ratio of (1:1, 2:1,1:2) ml. The solutions were magnetically stirred for 2 hours at 200-400 rpm at 50°c. And in an ultrasonic bath for 15 min to form a uniform solution.

In order to prepare PPY-PANI thin films, glass substrates were thoroughly cleaned by sequential rinsing with running water, ultrasonic treatment in distilled water for 15 minutes, and immersion in pure ethanol (99.9%) followed by an additional ultrasonic cleaning. After drying and brief sunlight exposure, the substrates were gently wiped with a soft paper cleaner to ensure complete removal of contaminants and disinfection. Following dissolution, the prepared solution was deposited onto pre-cleaned glass substrates using the drop-casting technique. The solutions of PPY, PANI, and their ratios were dropped onto the pre-cleaned substrates at a concentration of 75 µL/cm^3 and placed in an electric oven at a

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temperature of 80°C for 15 minutes.

Film thickness measurements were carried out using the optical interferometry method. The average film thickness was approximately 290 nm for all samples. This film thickness is suitable for structural characterization such as XRD and FE-SEM.

3. Results and Discussion

The XRD patterns presented in Fig. (1) offer a fascinating insight into the structural evolution from pure PPy and PANI to their composite materials, specifically focusing on the influence of varying PPy-PANI weight ratios. The bottom-most pattern, corresponding to pure PPy, exhibits a broad, featureless hump centered on 2θ≈20° to 30°. This characteristic feature is typical of amorphous or highly disordered polymeric materials, indicating a lack of long-range crystalline order in the pure PPy sample. Conversely, the top-most pattern, representing pure PANI, displays a sharp, intense reflection at 25.0°, which is clearly indexed as the (200) plane of PANI. A diffraction broad peak at 25.0° is characterized by weak intensity and broadness, which is diagnostic of amorphous PPy [51,52]. Emeraldine salt of PANI displays a sharp peak at 2θ = 25.115°, indicating (200) means the Polymer is semi-crystalline in nature, as the patterns show a sharp peak because of the presence of benzenoid and quinonoid groups in the polyaniline [53]. Moving to the composite samples, the patterns, from bottom to top, correspond to PPy-PANI ratios of 2:1, 1:1, and 1:2. When combining PPy and PANI in various molar ratios, the XRD patterns reveal changes in crystallinity. The pattern of (1:1) sample indicates the effect of the semi-crystalline nature of PANI on the amorphous nature of PPy; the peak was seen around 2θ=24.951°, and the pattern of (2:1) sample suggests the impact of PPy amorphous nature on the crystalline PANI; the small peak was seen around 24.907°. At least, the pattern of sample (1:2) appears a sharp peak at 2θ=25.038°, which is attributed to the effect of PANI on PPY. This sharp peak confirms that the synthesized PANI possesses a significant degree of crystallinity, often attributed to the ordered stacking of polymer chains. The presence of this intense reflection in PANI serves as a crucial reference point for analyzing the composite structures. A careful examination reveals a progressive trend in the structural characteristics as the PANI content increases. The pattern for the 2:1 PPy-PANI composite, which is rich in the amorphous PPy component, shows a broad reflection, though a faint, broadened peak is discernible near the 25.0° position, suggesting the initial incorporation of PANI and a slight reduction in overall disorder compared to pure PPy. As the ratio shifts to 1:1 PPy-PANI, the reflection around 25.0° becomes more pronounced, though still broader than in pure PANI, confirming the co-existence of both the amorphous PPy phase and the emerging crystalline PANI phase within the composite. The pattern for the 1:2 PPy-PANI composite, containing the highest amount of PANI among the three composites, exhibits a peak at 25.0° that is significantly sharper and more intense than the other two composite patterns, closely resembling the characteristic PANI peak but with some degree of broadening still evident. This suggests that as the PANI content increases, the material's overall crystallinity is enhanced, and the influence of the PANI's ordered structure becomes dominant. The inset of the graph provides a zoomed-in view of the 2θ range from 23° to 27°, focusing specifically on the primary reflection. This magnified view clearly illustrates the systematic sharpening and intensification of the peak as the weight ratio of PANI increases, further supporting the conclusion that the crystallinity of the composite is predominantly dictated by the PANI component. Furthermore, a slight shift in the peak position can be observed in the composites relative to pure PANI, which could be indicative of interactions or mixing between the PPy and PANI chains, potentially suggesting the formation of an interpenetrating network or co-crystal structures rather than a simple physical mixture. This shift, while subtle, is a critical observation, as it hints at the successful formation of a true composite structure where the PPy chains subtly influence the lattice parameters or ordering of the PANI component, resulting in a slightly modified crystal structure. The absence of any new, distinct peaks in the composite patterns suggests that no new crystalline phases have been formed, and the materials are best described as an interplay between the inherent amorphous nature of PPy and the semicrystalline nature of PANI. In summary, the XRD data compellingly demonstrates a structure where the PPy-PANI composites transition from an amorphous-dominated structure to one with progressively enhanced PANI-like crystallinity as the PANI content increases, with evidence suggesting molecular-level interactions between the two polymers rather than simple phase separation.

The inter-planar distance and crystallite size were calculated by Bragg's law and Debye-Scherrer equation as [54,55]:

$$D = \frac{k\lambda}{\beta cos\theta} \quad (1)$$

$$n\lambda = 2dsin\theta \quad (2)$$

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where n is the order of diffraction, λ is the wavelength (nm), d is the inter-planar spacing, θ is the Bragg's angle**,** and k is the Bragg's constant (0.9), λ is the wavelength (Cu Kα = 0.15405nm), β is the full-width at half maximum of the most intense peak (in radians), and θ is the peak position [56]. The XRD pattern revealed that polyaniline's crystal size was 32.3 nm. Pure PPy is predominantly amorphous, while pure PANI exhibits semi-crystalline characteristics. Forming composites of PPy and PANI alters their structural properties, with the degree of crystallinity and amorphousness dependent on the specific ratios used.

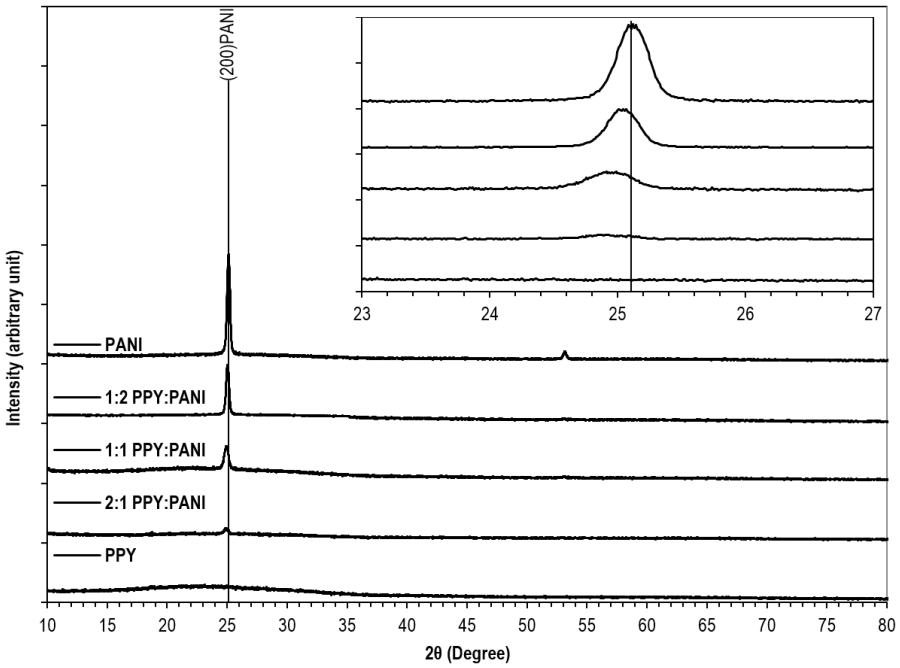
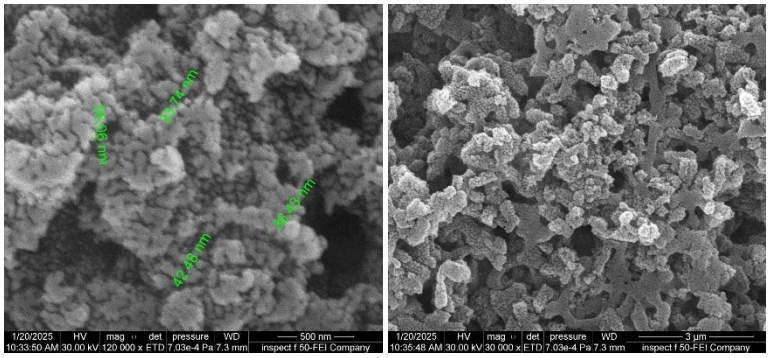


Fig. (3) XRD patterns of pure PANI, PPY, and PPY/PANI blends

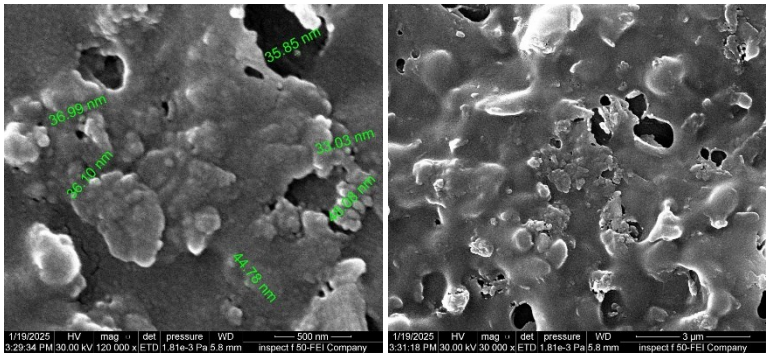
Table (1) XRD parameters and inter-planar spacing of PPY, PANI, and PPY/PANI blends

Sample	2θ (Deg.)	FWHM (Deg.)	d_{hkl} (Å)	D (nm)	(hkl)	Phase
PPY	Amorphous					
2:1 PPY:PANI	24.907	0.386	3.5721	21.1	(200)	PANI
1:1 PPY:PANI	24.951	0.351	3.5659	23.2	(200)	PANI
1:2 PPY:PANI	25.038	0.273	3.5536	29.8	(200)	PANI
PANI	25.115	0.252	3.5429	32.3	(200)	PANI

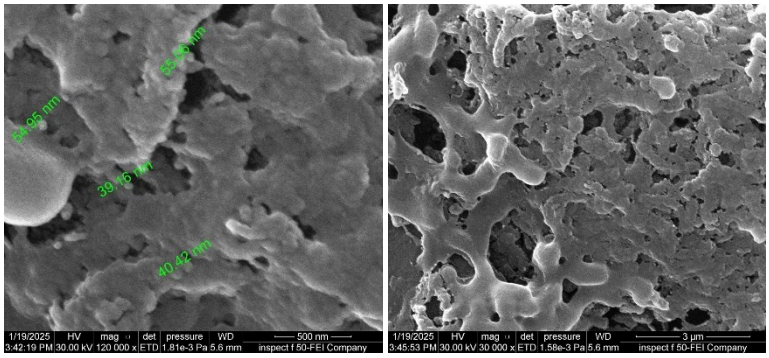
When studying PPY and PANI dissolved in DMSO and deposited by the drop-casting technique, the FE-SEM helps to determine the changes in the morphological structure of these polymer blends and the extent to which the solvent affects their shape and homogeneity. Figure (2) shows the FE-SEM images of the pure PPY, pure PANI, and PPy-PANI composite thin films with various concentrations of PPY-PANI (1:1, 1:2, and 2:1). As shown in Fig. (2a), the PPY thin film showed a highly uniform cauliflower-like coral hemisphere with grain sizes of about 42.17 nm, uniformly distributed over a large surface area [57]. However, as the polymer concentration is increased, a sudden precipitate occurs, and PPy nanoparticles start agglomerating and stacking on one another. In Fig. (2b), the polyaniline shows how the drop-casting technique affects the surface's composition and particle distribution across the thin layers. Due to the particles' inhomogeneous distribution, the distribution of nanoparticles shows some agglomerations or surface defects [58,59]. The particle diameter of polyaniline is around 39.13 nm in both polymers. Particle diameters are almost similar. Figure (2c), for ratio (1:1), shows an irregular structure with some areas showing a lamellar shape and roughness on the surface without large defects and some clumping. Figure (2d) also shows disorganized particles, a lamellar properties mixture, a less agglomerated distribution, and obvious roughness. Finally, figure (2e) also shows disorganized shapes with needle structures; the surface roughness is more pronounced than in the previous samples due to the concentration of PPY.



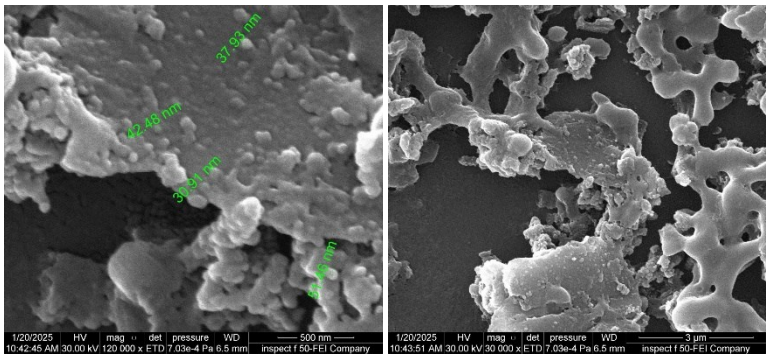
(a)



(b)



(c)



(d)

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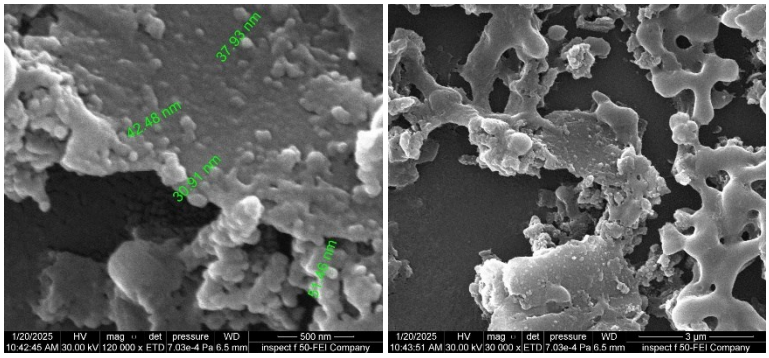

(e)

Fig. (4) FE-SEM images of (a) pure PPY, (b) pure PANI, (c) PANI/PPY (1:1), (d) PANI/PPY (2:1), and (e) PANI/PPY (1:2)

4. Conclusion

In this work, PPY–PANI composites with different composition ratios were successfully synthesized and characterized to improve NO_2 gas sensing. XRD results revealed slight peak shifts and reduced crystallite size, indicating structural interaction and lattice distortion upon blending PPY with PANI. FE-SEM images showed that the 1:1 composite developed an irregular, rough surface with lamellar features, providing higher surface area and effective gas diffusion pathways.

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