Fabrication of polypyrrole/nylon composite with high radar transmission loss for electromagnetic interference shielding application

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ABSTRACT

This study investigates the fabrication of conductive polypyrrole (PPy)/nylon composites via a controlled interfacial polymerization technique. The composite was synthesized by positioning a nylon fabric at the interface of an aqueous phase containing oxidant and p-toluene sulfonic acid (p-TSA, 0.1 M) as the dopant, and an organic phase consisting of 3% (v/v) pyrrole in hexane. The influence of oxidant types and reaction temperatures on the formation of the PPy layer was systematically evaluated. Morphological and structural characterization revealed that the PPy was successfully integrated into the nylon matrix, increasing the fabric thickness from 0.09 mm (raw) to 0.1 mm. Secondary electrochemical deposition was performed on the PPy/nylon composite in an aqueous solution containing 0.1 M pyrrole to achieve a greater PPy thickness. Furthermore, electromagnetic interference (EMI) shielding measurements in the X-band frequency range (8–12 GHz) showed a shielding effectiveness (SE) exceeding 20 dB. These results suggest that the as-prepared PPy/nylon composites are promising candidates for lightweight, flexible radar-absorbing materials in advanced electronic applications.

Keywords: Conductive polymer; Interfacial polymerization; EMI.

1. INTRODUCTION

Electromagnetic Interference (EMI) shielding materials have become a paramount necessity in both civilian and defense sectors, particularly in the X-band frequency range (8–12 GHz) typically used for radar applications. Historically, traditional EMI shielding materials have relied heavily on metals such as copper, aluminum, and stainless steel. While these materials exhibit high shielding effectiveness due to their improved electrical conductivity, they suffer from several inherent drawbacks, including high density, susceptibility to corrosion, and poor mechanical flexibility.

In recent years, the research focus has shifted toward lightweight, flexible, and absorption-dominant materials. Conductive polymer composites (CPCs) have emerged as a revolutionary class of materials, offering a unique combination of tunable electrical properties, ease of processing, and low weight [1-3]. Among various intrinsically conducting polymers (ICPs), polypyrrole (PPy) has garnered significant attention due to its high electrical conductivity, environmental stability, and strong dielectric loss characteristics [4-6]. However, PPy in its pure form—typically as a synthesized powder or a standalone film—exhibits poor mechanical properties. To overcome these limitations, PPy is frequently integrated with flexible substrates to form composites. Nylon 6, a synthetic thermoplastic with excellent tensile strength, elongation at break, and chemical resistance, serves as an ideal scaffold. By combining the conductivity of PPy with the mechanical robustness of nylon cloth, it is possible to fabricate a composite that is both electrically active and mechanically durable [7].

The performance of PPy/nylon composites is heavily dictated by the synthesis methodology and the resulting polymer morphology. While chemical in-situ polymerization is a widely adopted technique, the electrochemical deposition plays a decisive role in enhancing the thickness and overall conductivity and reflection loss of the composite. In this study, a PPy/nylon composite was

fabricated via an interfacial polymerization method using a hexane/water biphasic system. The thickness of the PPy layer was further enhanced by the electrochemical polymerization of the PPy/nylon composite in a pyrrole solution. The resulting composites were characterized for their thickness, and EMI shielding effectiveness in the 8 - 12 GHz range, providing a comprehensive assessment of their potential as lightweight radar-absorbing materials.

2. EXPERIMENTAL

2.1. Materials

The 1000-mesh nylon 6 fabric was sourced from China. Pyrrole (C_4H_5N 99.95%) was purchased from Macklin (China). Ferric chloride hexahydrate ($FeCl_3.6H_2O$ 99.95%), ammonium persulfate $(NH_4)_2S_2O_8$ 99% (Xilong), and n-hexane (n-C_6H_{14}, 99.95%) were obtained from Xilong (China).

2.2. Preparation of PPy/nylon composite

2.2.1. Interfacial polymerization of PPy/nylon composites

The PPy/nylon composite was synthesized via interfacial polymerization. A 1000-mesh nylon 6 fabric (initial thickness of 0.09 mm) served as the substrate, which was pre-cleaned with ethanol to reduce surface contaminants.

The aqueous phase consisted of a 0.5 M oxidant (ferric chloride hexahydrate or ammonium persulfate) and 0.1 M p-TSA as the dopant. The organic phase was prepared by dissolving 3% (v/v) pyrrole in n-hexane. During polymerization, the nylon fabric was placed on the surface of the aqueous solution. The organic phase was then added dropwise to establish the reaction at the liquid-liquid interface.

As the monomers diffused and contacted the oxidant, a PPy layer was deposited onto the nylon fibers. The reaction proceeded for 30 minutes, during which the fabric color changed from white to black. Subsequently, the resulting composite was rinsed with distilled water and dried at room temperature. The dropwise addition of the organic phase was controlled to maintain interface stability and minimize mixing, aiming for a consistent polymer deposition. This reaction duration was chosen to achieve the PPy loading required for subsequent electromagnetic interference (EMI) shielding measurements.

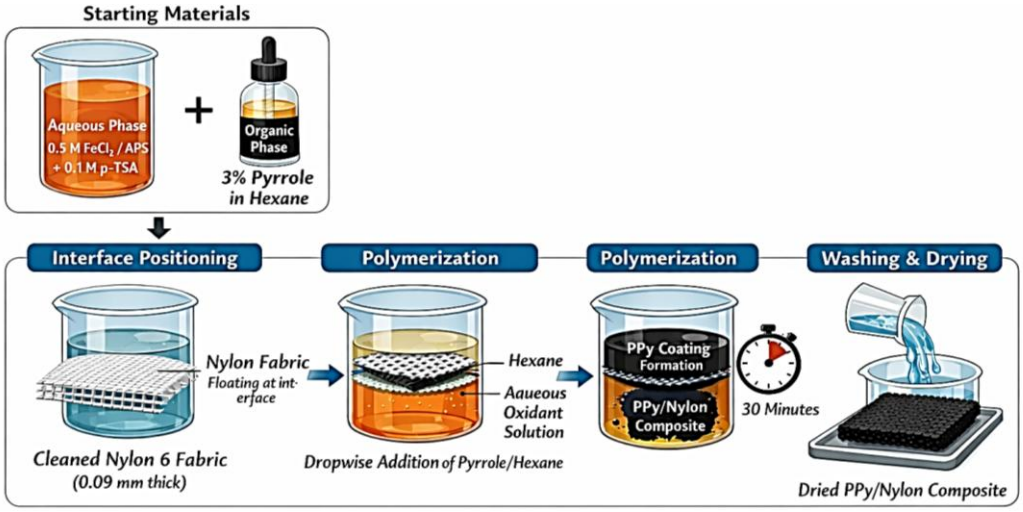

Figure 1. *Synthesis of PPy/nylon composite via interfacial polymerization.*

2.2.2. Electrochemical deposition of PPy

To further increase the PPy loading, an electrochemical deposition was performed in a standard three-electrode cell containing an aqueous electrolyte of 0.1 M pyrrole and 0.1 M p-TSA. In this

setup, the chemically-coated PPy/nylon fabric served as the working electrode, where the pre-existing PPy layer acted as a conductive surface for additional polymer growth. A platinum mesh was utilized as the counter electrode. By applying a constant current density of 2 mA/cm², the PPy layer was systematically thickened as the pyrrole was oxidized and grafted onto the nylon fibers. The deposition was maintained for 10 minutes to achieve the targeted polymer density.

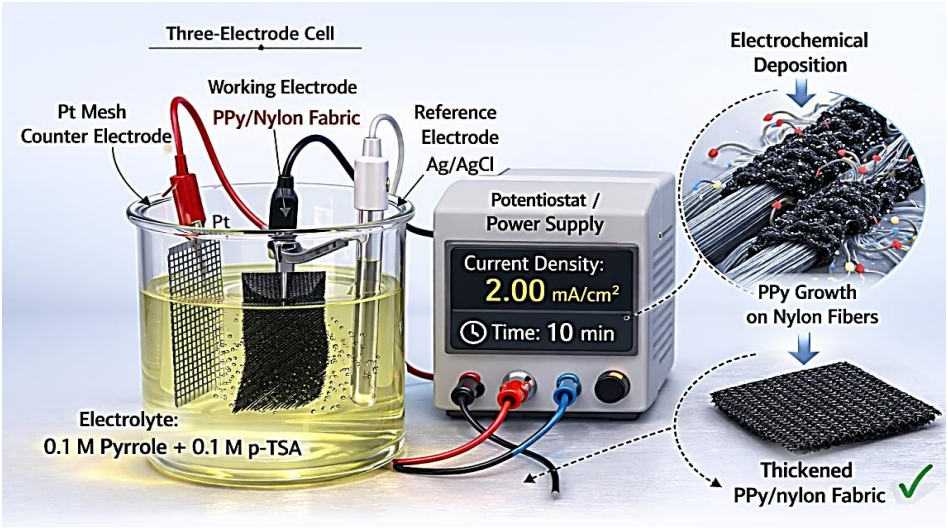

Figure 2. *Schematic of electrochemical deposition to increase PPy loading on PPy/Nylon fabric.*

2.2.3. Characterization

The PPy deposition on the nylon fabric was evaluated by determining the weight percentage of PPy and the increase in thickness relative to the pristine fabric. The morphology of the PPy layer was investigated using a Hitachi S-4800 field-emission scanning electron microscope (FE-SEM).

The electromagnetic interference (EMI) shielding effectiveness of the composites was evaluated by measuring the transmission loss within the X-band frequency range (8–12 GHz). The measurement setup consisted of an Agilent E8362C Vector Network Analyzer (VNA), an 85054D calibration kit, and a pair of X-band horn antennas. The PPy content remaining on the fabric after the rinsing process was used as a key metric to characterize the composite fabrication. The proposed mechanism for the formation of the PPy layer is illustrated in Figure 2, showing the interaction between the pyrrole and the ferric chloride hexahydrate oxidant at the interface.

3. RESULTS AND DISCUSSION

3.1. PPy/nylon composite using chemical method

During the experiment, the initial white nylon fabric changed to a black color after a reaction period of 30 minutes, indicating the presence of PPy (Figure 3). A portion of the formed PPy precipitated as a free powder in the solution. The PPy content remaining on the fabric after the cleaning process was measured to evaluate the PPy loading on the nylon substrate. The amounts of PPy deposited on the fabric using ferric chloride hexahydrate and ammonium persulfate (APS) as oxidants are summarized in Table 1.

The weight percentage of PPy varied depending on the type of oxidant used, with loading levels of 13.81% for ferric chloride hexahydrate and 9.41% for APS. This difference in loading can be associated with the different oxidation potentials of the two initiators. The higher standard reduction potential of APS (E^0 = +2.01 V compared to Fe^{3+} E^0 = +0.77) may influence the polymerization path, potentially leading to more PPy forming as a separate precipitate rather than adhering to the fiber surface [8]. Conversely, the lower potential of $FeCl_3$ may support a more

moderate reaction, resulting in a higher proportion of polymer deposition on the nylon fibers [9]. These surface characteristics were further investigated using SEM.

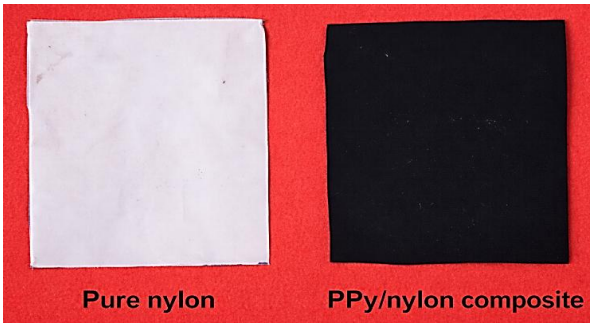

Figure 3. *Images of pure nylon 6 (left) and PPy/nylon 6 composite (right).*

Table 1. *Amount of polypyrrole deposited onto the nylon 6 using different oxidants.*

Oxidant	Sample 1		Sample 2		Sample 3		Average PPy deposition (%)
	Pure nylon (g)	PPy/nylon (g)	Pure nylon (g)	PPy/nylon (g)	Pure nylon (g)	PPy/nylon (g)	
$FeCl_3$	0.35	0.41	0.36	0.42	0.35	0.40	13.81%
$(NH_4)_2S_2O_8$	0.34	0.38	0.36	0.40	0.36	0.39	9.41%

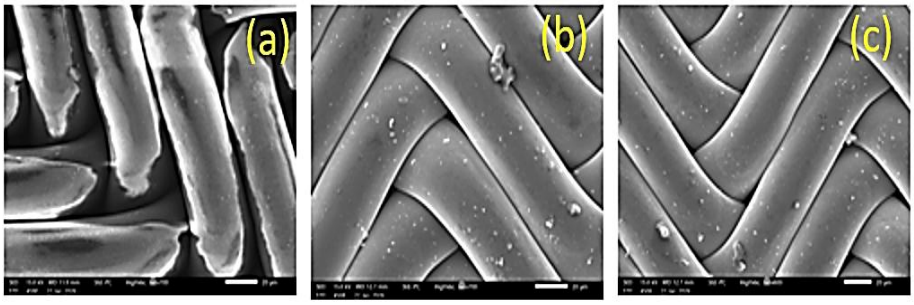

Figure4. *SEM images of fabric of a) pure nylon, b) PPy/nylon from APS and c) PPy/nylon from* $FeCl_3$.

SEM images of PPy/nylon composites (Figure 5) show the surface morphology of the samples. The pure nylon sample (a) exhibits a smooth surface, typical of insulating polymers, which often results in surface charging during imaging. Upon PPy deposition, samples (b) and (c) display a granular texture and increased surface roughness. The observed reduction in surface gloss in the composites is consistent with the presence of a PPy coating on the fibers, which promotes diffuse light scattering.

The choice of $FeCl_3.6H_2O$ as the primary oxidant in this study is also supported by the nature of the counter-ions. The chloride (Cl^-) ions generated from $FeCl_3$ are smaller than the tetrahedral sulfate (SO_4^{2-}) anions produced by APS. This difference in ionic size can affect the intercalation efficiency into the polymer chains and the resulting doping levels, which is a key factor in achieving the desired electrical properties for EMI shielding applications [10]

The characteristic glossy appearance of the pure nylon sample (a) is attributed to its smooth surface morphology and significant surface charging typical of insulating polymers. Upon the oxidative deposition of PPy, the samples (b) and (c) exhibit a matte, granular texture. This transition is due to increased surface roughness, which promotes diffuse scattering, and enhanced electrical conductivity, which eliminates charging effects. The lack of gloss in the composites serves as qualitative evidence of a successful and uniform coating of PPy on the nylon fibers.

In contrast, the milder potential of Fe^{3+} aligns more closely with the oxidation threshold of the pyrrole monomer, facilitating a controlled polymerization that preserves chain integrity. Furthermore, the chloride (Cl^-) counter-ions generated by $FeCl_3$ are significantly less bulky than the large, tetrahedral sulfate (SO_4^{2-}) anions produced by APS. This reduced steric hindrance allows Cl^- to intercalate more efficiently into the polymer chains, promoting higher doping levels and a more ordered, compact morphology that enhances charge carrier mobility. Consequently, $FeCl_3$ was chosen as the most effective oxidant for the chemical synthesis of PPy.

To confirm the chemical identity of this coating, EDX analysis was performed at the regions of polymer deposition. While the pristine nylon spectrum only exhibits peaks for carbon, nitrogen, and oxygen, the composite spectra show distinct signals for chlorine (from the $FeCl_3$ oxidant). The detection of these dopant-related elements provides quantitative evidence that the observed granular layer is functionalized, doped PPy, which is essential for establishing the conductive network required for EMI shielding.

The chemical structure was further characterized using FTIR spectroscopy. Absorption bands at 1545 cm^{-1} and 1462 cm^{-1} are observed, corresponding to the C=C and C–N stretching vibrations of the pyrrole ring, respectively, confirming the presence of the conjugated PPy backbone. Notably, the peak at 1175 cm^{-1} is associated with the doped state of PPy, explaining the material's interaction with electromagnetic waves. Compared to the pristine nylon spectrum, a slight shift in the amide I (C=O) and amide II (N–H) bands of the nylon matrix was recorded. This shift suggests the existence of interfacial hydrogen bonding between the nylon amide groups and the N–H units of the PPy chains. The synergy between morphological observations from SEM and the chemical fingerprints from EDX and FTIR confirms that the interfacial polymerization technique produces a structurally stable and chemically doped conductive coating on the nylon substrate.

Table 2. *Relative wt.% of the elements present in the sample PPy/nylon.*

Elemental	PPy/nylon	
	Mass (%)	Atom (%)
C	60.55	67.46
N	18.61	17.78
O	15.03	12.57
Cl	5.81	2.19
Total	100	100

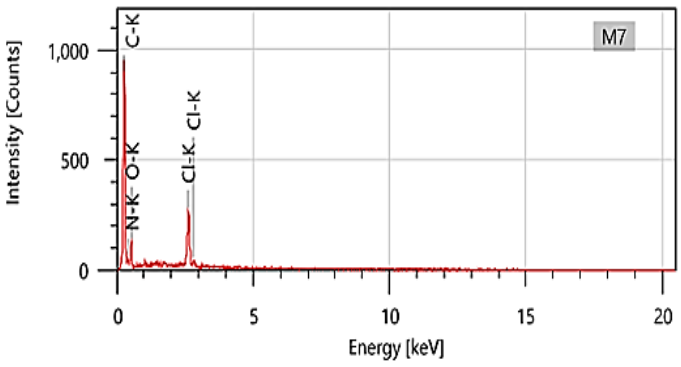

Figure 5. *EDX spectrum of PPy/nylon.*

3.2. PPy thickening using the electrochemical method

The thickness of the PPy layer was further enhanced by applying a constant current of 50 mA to the chemically-pre-seeded $5x5$ cm^2 PPy/nylon fabric (corresponding to a current density of 2 mA/cm^2). After a deposition period of 10 minutes, a measurable increase in the PPy layer was

achieved. This enhancement was quantitatively confirmed by the substantial increase in the PPy loading content on the nylon fibers, as well as a measurable increment in the overall coating thickness compared to the initial chemically-synthesized samples (Table 3).

Table 3 presents the evolution of the physical properties of the nylon fabric throughout the multi-stage synthesis process. The data reveal a progressive and controlled increase in both fabric thickness and weight, moving from the pristine state (0.09 mm, 0.35 g) to the final electrochemical composite (0.11 mm, 0.45 g). Specifically, the initial chemical polymerization resulted in a 17% mass increase, while the subsequent electrochemical step contributed an additional 11.4%, confirming the effectiveness of the hybrid synthesis approach.

Table 3. *Fabric thickness and weight of pure nylon and PPy/nylon composite.*

Properties	Pure nylon	Chemical PPy/nylon	Electrochemical PPy/nylon
Fabric thickness (mm)	0.09	0.1	0.11
Fabric weight (g)	0.35	0.41	0.45

Notably, the incremental increase in thickness—approximately 10 % per stage—suggests that the PPy layer does not merely form a superficial coating but instead wraps uniformly around the individual fibers of the 1000-mesh structure. This conformal growth is critical, as it ensures a dense conductive network for high electromagnetic interference (EMI) shielding while preventing the formation of a brittle bulk layer.

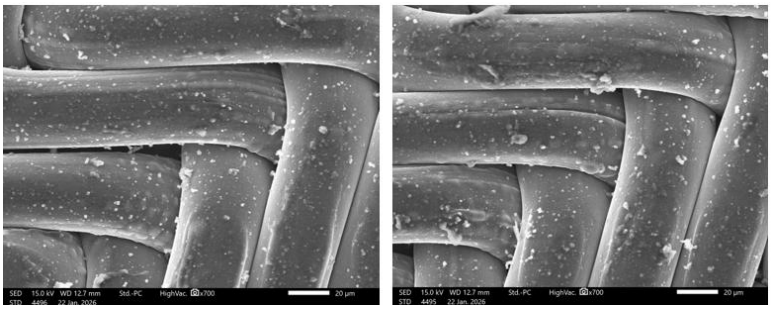

Figure 6. *SEM images of PPy/nylon composite thickened using electrochemical method.*

The morphological evolution of the PPy/nylon composites was further elucidated through SEM analysis (Figure 6). To compare, the chemically synthesized sample (Figure 4c) exhibits a relatively thin and uniform PPy layer adhering to the nylon fibers. In contrast, the electrochemically-thickened sample displays a significantly more robust and dense PPy coating. The individual fiber diameters appear thickened, indicating that the electrochemical process promoted the secondary growth of PPy atop the initial chemical seed layer. This observation is consistent with the physical data, where the fabric thickness increased to 0.11 mm and the total weight reached 0.45 g. The SEM images confirm that this additional PPy loading forms a continuous and highly conductive network, which is essential for achieving improved EMI Shielding while maintaining the architecture of the 1000-mesh fabric.

The current–voltage (I–V) (Figure7) characteristics clearly demonstrate the difference in electrical conductivity between the blank nylon fabric and the PPy/Nylon6 composite. The blank nylon sample exhibits extremely low current values in the order of 10^{-9} A over the applied voltage range from −5 to 5 V, indicating very high electrical resistance and confirming the insulating nature of nylon. In addition, the I–V curve shows noticeable fluctuations and non-linear behavior, which is typical for insulating materials with negligible charge transport. In contrast, the PPy/Nylon sample displays a significantly higher current in the milliampere range (approximately 10^{-2} A) and a nearly linear I–V relationship that passes through the origin. This linear behavior indicates ohmic conduction and suggests the formation of effective conductive pathways within the material. The

dramatic increase in current, by several orders of magnitude compared to the blank nylon, confirms that the deposition of polypyrrole (PPy) onto the nylon fibers significantly enhances the electrical conductivity of the fabric.

Figure 8 illustrates the EMI Shielding Effectiveness (EMI SE) of the PPy/nylon composites in the X-band frequency range (8–12 GHz). It is evident that the electrochemically-thickened sample exhibits improved electromagnetic interference (EMI) shielding performance compared to the chemically-synthesized counterpart. While the chemical PPy/nylon sample achieves a maximum TL of approximately -24 dB, the secondary electrochemical deposition effectively pushes the attenuation further, reaching a peak of nearly -27 dB. This enhancement is attributed to the increased PPy loading and coating thickness (0.11 mm compared to 0.10 mm), which facilitates a more continuous and dense conductive network on the nylon fibers. The consistent 4–6 dB improvement across the entire bandwidth demonstrates that the electrochemical step is a vital and effective method for optimizing the Shielding Effectiveness of the composite.

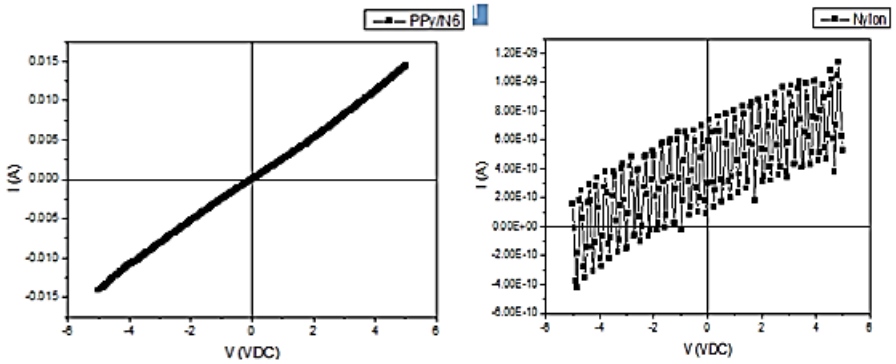

Figure 7. *I–V curves of PPy/Nylon6 composite fabric and pristine nylon fabric measured from −5 to 5 V.*

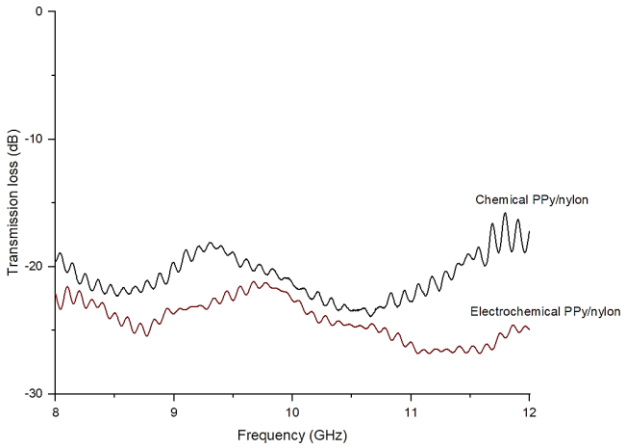

Figure 8. *Transmission loss of chemically synthesized PPy/nylon and electrochemically thickened PPy/nylon.*

Substratre	Method	Thickness (mm)	EMI SE (dB)	Ref
Nylon 66	Interfacial	0.15	21	[11]
Polyester	Insitute chemical	0.25	25	[12]
Carbon fiber/PPy	Electrochemical	0.4	42	[13]
This research	Interfacial+ electrochemical deposition	0.11	27	

While some studies utilizing carbon fiber or significantly thicker coatings (above 0.3 mm) report higher SE values up to 40 dB [13], the current PPy/nylon composite achieves a competitive attenuation of 27 dB at a minimal thickness of only 0.11 mm. According to electromagnetic shielding theory, SE is directly proportional to both electrical conductivity and material thickness (d). In this study, although the thickness is kept very low to maintain fabric flexibility, the high PPy loading density achieved through the hybrid method ensures a sufficient conductive network to reach the 20-30 dB range required for most commercial and military shielding applications. The consistent improvement across the 8–12 GHz band indicates that the secondary electrochemical deposition is an effective method for tuning the Shielding Effectiveness of lightweight, flexible textile composites.

4. CONCLUSIONS

The successful fabrication of a high-performance PPy/nylon composite was achieved through a synergistic two-step process, where an initial chemical interfacial polymerization was enhanced by a secondary electrochemical thickening step. Physical and morphological analyses, including SEM imaging, confirmed that applying a 2 mA/cm² current density for 10 minutes effectively increased the fabric thickness to 0.11 mm and the mass to 0.45 g by promoting a dense, conformal secondary growth of PPy around the individual fibers of the 1000-mesh nylon. This structural optimization resulted in a significant improvement in electromagnetic interference (EMI) shielding performance, as evidenced by a 4–6 dB increase in transmission loss across the 8–12 GHz X-band, ultimately reaching a peak attenuation of nearly -27 dB while preserving the material's inherent mechanical flexibility.

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TÓM TẮT

Chế tạo vật liệu composite polypyrrole/nylon có độ tổn hao truyền qua cao cho ứng dụng chống nhiễu điện từ

Nghiên cứu này khảo sát quá trình chế tạo vật liệu composite polypyrrole (PPy)/nylon dẫn điện thông qua kỹ thuật polime hóa tại mặt phân cách. Vật liệu composite được tổng hợp bằng cách đặt vải nylon tại bề mặt phân cách giữa pha nước chứa chất oxy hóa cùng chất pha tạp acid p-toluenesulfonic (p-TSA, 0.1 M), và pha hữu cơ gồm 3% (v/v) pyrrole trong hexane. Ảnh hưởng của các loại chất oxy hóa và nhiệt độ phản ứng lên sự hình thành lớp PPy đã được đánh giá một cách hệ thống. Kết quả đặc trưng hình thái và cấu trúc cho thấy PPy đã được tích hợp thành công vào nền vải nylon, làm tăng độ dày của vải từ 0,09 mm (vải thô) lên 0,1 mm. Quá trình lắng đọng điện hóa thứ cấp đã được thực hiện trên composite PPy/nylon trong dung dịch nước chứa 0,1 M pyrrole để đạt được độ dày PPy lớn hơn. Thêm vào đó, các phép đo hiệu quả che chắn nhiễu điện từ (EMI SE) trong dải tần băng X (8–12 GHz) cho thấy hiệu quả che chắn vượt quá 20 dB. Những kết quả này gợi mở rằng vật liệu composite PPy/nylon được chế tạo là ứng viên đầy triển vọng cho các loại vật liệu hấp thụ radar nhẹ và linh hoạt trong các ứng dụng điện tử tiên tiến.

Từ khoá: Polime dẫn điện; Polime hóa bề mặt; EMI.