



Synthesis of Polyaniline/Polyeugenol Composites as Conductive Polymers

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ABSTRACT: This study synthesised polyaniline/polyeugenol (PANI/PE) composites via a solution mixing approach at ambient temperature, using dichloromethane (DCM) as solvent. The composites were prepared with PE mass fractions of 5%, 10% and 15% relative to PANI weight. The fabrication involved consecutive stages of PANI and PE polymerisation, followed by composite blending. Structural analysis was conducted using Fourier Transform Infrared (FTIR) spectroscopy. Morphology, elemental composition, thermal behaviour and electrical performance were characterised by Thermogravimetric Analysis (TGA), Scanning Electron Microscopy with Energy-Dispersive X-ray Spectroscopy (SEM-EDX) and four-point probe conductivity measurements, respectively. The PANI/PE composites were obtained as greyish-green solids with yields of 91.14% (5% PANI/PE), 93.00% (10% PANI/PE) and 81.21% (15% PANI/PE). FTIR spectra confirmed successful composite formation, displaying characteristic absorptions corresponding to both PANI backbone (--NH-- stretching at $3,446\text{ cm}^{-1}$, $3,425\text{ cm}^{-1}$ and $3,444\text{ cm}^{-1}$) and PE functional groups (--OH and methoxy vibrations at 856 cm^{-1} – 867 cm^{-1}). SEM micrographs revealed reduced interparticle voids and uniform filling of surface gaps, indicating good dispersion of PE within the PANI matrix. TGA results demonstrated enhanced thermal stability with increasing PE content, suggesting improved composite resistance to heat-induced degradation. Electrical analysis showed higher conductivity in PANI/PE composites than in pristine PANI, with maximum performance at 10% PANI/PE, indicating this composition as the optimal formulation. Overall, these findings suggest that PE incorporation enhances the thermal and electrical properties of PANI by improving homogeneous composite structuring.

Keywords: polyaniline, polyeugenol, composite, PANI/PE, conducting polymer

1. INTRODUCTION

Conductive polymers have attracted considerable research interest in recent years owing to their ability to integrate favourable electrical properties with the intrinsic advantages of polymeric materials, including mechanical flexibility, low density and facile processability. Among this class of materials, polyaniline (PANI) has emerged as one of the most extensively investigated conductive polymers due to its intriguing features and broad range of potential applications. It possesses excellent environmental stability, good physicochemical properties and tunable electrical conductivity achieved through doping or structural modification.^{1–3}

PANI is typically synthesised from aniline monomers ($C_6H_5NH_2$), which are benzene-derived compounds.^{4,5} The electrical conductivity of PANI varies depending on the synthesis method, catalysts, reaction conditions and the type and characteristics of dopants employed. In addition, PANI has been reported to be readily and cost-effectively synthesised, and to undergo reversible changes in its electrical properties through protonation–deprotonation processes. These characteristics make PANI highly promising for the development of advanced conductive materials, including polymer composites designed to enhance electrical, thermal and dielectric performance.^{6,7}

PANI can be synthesised by chemical oxidative polymerisation, one of the most widely used techniques.^{8,9} According to Namsheer and Rout, this method is considered one of the simplest, as aniline monomers are directly mixed with oxidising agents to form PANI.² However, compared to pure PANI, its composites often display noticeable differences, including slight structural variations, reduced durability and decreased mechanical strength. These changes are commonly associated with modifications involving thermoplastic polymers such as polypropylene (PP), polymethyl methacrylate (PMMA) and polyvinyl chloride (PVC), which are incorporated to develop materials with improved electrical conductivity while still maintaining favourable mechanical properties.¹⁰ The development of PANI-based materials continues to be crucial as modern electronic devices increasingly require functional materials for dielectric applications, microwave absorption and electromagnetic interference (EMI) shielding.^{11–13}

In particular, polyeugenol (PE) is a polymer synthesised through the polymerisation of eugenol.^{14,15} Eugenol is widely used in industry, contributing to its high economic value. This compound can serve as a raw material in the manufacture of food products, such as vanilla flavouring. Eugenol contains hydroxyl, allyl and methoxy functional groups, which enable various chemical modifications and facilitate the formation of new monomers and polymers. PE has been widely applied as both a catalyst and an adsorbent, offering several advantages, including thermal stability, good mechanical strength, and high performance.¹⁶

PE possesses an aromatic structure and phenolic groups that enable π – π interactions with PANI chains, potentially enhancing the thermal stability and dielectric properties of the composite. The combination of PANI/PE is therefore expected to yield a conductive material with improved electrical and thermal performance while maintaining the environmentally friendly characteristics of the bio-based polymer component.^{17–19}

Based on these developments, studies on the synthesis and characterisation of PANI/PE composites are essential for the advancement of conductive polymer materials with enhanced dielectric performance and thermal stability derived from renewable resources.

2. EXPERIMENTAL

2.1 Materials

Prior to polymerisation, aniline (E. Merck) was mixed with 1 M hydrochloric acid (HCl) and ammonium peroxydisulfate. Eugenol was obtained from Indesso Aroma Indonesia, while boron trifluoride etherate ($\text{BF}_3 \cdot \text{O}(\text{C}_2\text{H}_5)_2$), diethyl ether, dichloromethane, dimethyl sulfoxide (DMSO), methanol, acetone, universal pH paper and ammonium peroxydisulfate were sourced from Merck. Furthermore, nitrogen was supplied by Samator industry.

2.2 PANI Synthesis

PANI was synthesised via oxidative polymerisation. Aniline (9.3 mL) was dissolved in 1 M HCl (100 mL) and stirred at 0°C–5°C. Ammonium peroxydisulfate (28.25 g) was dissolved in distilled water (50 mL), and the solution was added dropwise to aniline-HCl mixture over 8 hours. The resulting PANI precipitate was filtered using a Buchner funnel, washed with 1 M HCl, and dried in an oven at 50°C for 5 h.

2.3 PE Synthesis

Polymerisation of eugenol (5 g) was conducted at room temperature (28°C–30°C) under a nitrogen atmosphere for 4 h. An aliquot of 0.25 mL boron trifluoride etherate initiator was added every hour. The reaction was terminated by the addition of methanol (1 mL), followed by extraction with chloroform and pH adjustment to neutral.

2.4 PANI/PE Composite Synthesis

PANI/PE composites were prepared with PE contents of 5%, 10% and 15% by weight relative to aniline. The PANI-PE mixture was stirred for 2 h at room temperature, washed with distilled water and subsequently dried in an oven.

2.5 Fourier Transform Infrared (FTIR) Analysis

FTIR analysis was performed using a Shimadzu FTIR Prestige-21 spectrophotometer (Shimadzu Corporation, Kyoto, Japan) in the range of $4,000\text{ cm}^{-1}$ – 400 cm^{-1} with a resolution of 4 cm^{-1} . Samples were prepared as KBr pellets, and spectra were recorded at room temperature with the samples placed directly in the holder.

2.6 Viscosity Measurement

The intrinsic viscosities of PANI and PE were determined using an Ostwald viscometer. PANI solutions with DMSO solvent were prepared at concentrations of 0.001 g/mL, 0.0015 g/mL, 0.002 g/mL and 0.0025 g/mL. Meanwhile, PE solutions were prepared with chloroform solvent at concentrations of 0.005 g/mL, 0.01 g/mL, 0.015 g/mL and 0.02 g/mL. Flow times were measured at room temperature. Relative viscosity (η_r) and specific viscosity (η_{sp}) were calculated, and a plot of η_{sp}/C versus concentration (C) was constructed to determine the intrinsic viscosity.

2.7 Characterisation of PANI/PE Composites

Thermal properties of the PANI/PE composites were determined by Thermogravimetric/Differential Thermal Analysis (TG/DTA) with a Simultaneous Thermal Analysis (STA) PT 1600 LINSEIS instrument (LINSEIS Messgeräte GmbH, Selb, Germany). Membrane morphology was analysed using a JEOL Benchtop JCM 7000 Scanning Electron Microscope equipped with Energy Dispersive X-ray (SEM-EDX) (JEOL Ltd., Tokyo, Japan).

2.8 Conductivity Measurement

For conductivity measurements, 200 g of PANI/PE composite (for each variation) was pressed into pellets with a thickness of 0.5 cm. The pellet surfaces were coated with two layers of silver paste, and two platinum wires were attached as electrodes. The measurement circuit was connected to an LCR impedance metre (U2826 Eucol, China) operating at 1 volt. The frequency range applied was 20 Hz–5 MHz. Impedance data were recorded as real (Z) and imaginary (Z'') components and analysed using

Zview software. The resistance (R) used to determine conductivity was obtained from the subtraction between the charge-transfer resistance and the solution resistance, as described in Equation 1. In which, σ , l , R and A represent conductivity (S/m), sample thickness (cm), resistance (Ω) and active area of silver electrode (cm^2), respectively.

$$\sigma = \frac{l}{RA}$$

(1)

3. RESULT AND DISCUSSION

3.1 PANI Synthesis

PANI was synthesised via chemical oxidative polymerisation using HCl as a dopant, which enhances electrical conductivity, solubility, polymerisation capability and stability. During the initiation stage, the solution colour changed from transparent yellow to brownish-red and subsequently to light green, indicating the coupling of aniline monomers to form PANI. In the propagation step, the colour further changed from light green to bluish-green and a solid product formed within the reaction medium.⁶ The synthesised PANI was obtained predominantly in the emeraldine salt form, which exhibits higher conductivity than other oxidation states of PANI, such as pernigraniline and leucoemeraldine.

Table 1: Characteristics of PANI synthesis results

Characteristics		Study Results	References
Shape	Solid		Solid ²⁰
Colour	Dark green		Dark green ⁶
Melting Point	310°C		300°C–330°C ²¹
Solubility	Insoluble in water, solvents and organic solvents		Hard to dissolve ²²
Yield percentage	97.96%		–

The characteristics of the synthesised PANI are shown in Table 1. The product appeared as a dark green solid with a melting point of approximately 310°C and was insoluble in water and common organic solvents. Although PANI was difficult to dissolve in DMSO, dissolution could be facilitated by sonication combined with stirring.

3.2 PE Synthesis

PE was synthesised via cationic addition polymerisation under a nitrogen atmosphere to prevent hydrolysis, which could deactivate the catalyst.²³ The synthesis comprised three stages, including initiation, propagation and termination. During initiation, the catalyst (boron trifluoride etherate) attacked the allyl group of eugenol, promoting double-bond cleavage. This reaction was indicated by a colour change to red-purple, consistent with Markovnikov’s rule, whereby carbocation stability determines the reactivity of subsequent monomer incorporation.²⁴

During the propagation stage, covalent bonds formed between carbocation chains and eugenol monomers, resulting in elongated polymer chains. The initially liquid mixture gradually thickened into a gel, and the colour turned purplish-black. Termination was achieved by adding methanol, which halted polymerisation and produced methoxy end groups.²⁰

PE polymerisation reaction is displayed in Figure 1, and the results of the PE synthesis are presented in Table 2. The synthesised PE appeared as a light brown solid with a melting point of approximately 79°C and was soluble in chloroform, dichloromethane (DCM) and DMSO, consistent with previous reports.

Table 2: Characteristics of PANI synthesis results

Characteristics		Study Results	References
Shape	Solid		Solid ²⁵
Colour	Light brown		Light brown ²⁶
Melting Point	79°C		75°C–80°C
Solubility	Soluble in chloroform, DCM dan DMSO		Soluble in organic solvents ¹⁶
Yield percentage	63.30%		83% ²⁵

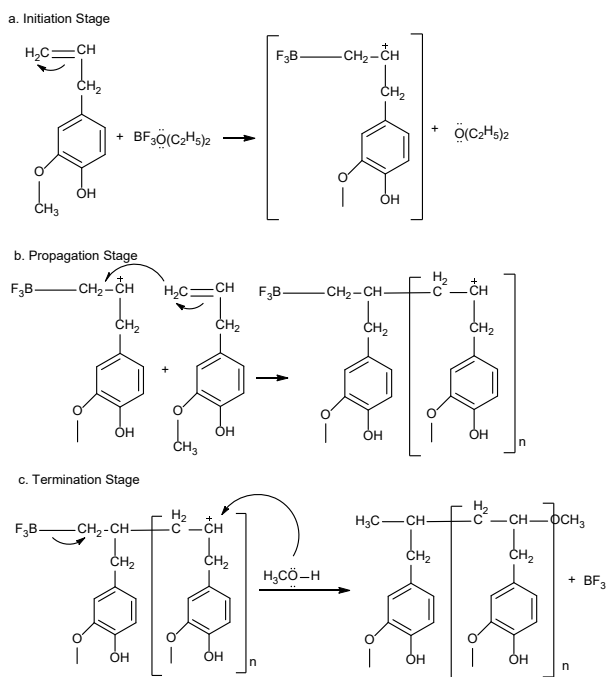


Figure 1: Polymerisation process of eugenol.

3.3 PANI/PE Composite Synthesis

The PANI/PE composites were obtained as greyish-green solids, with yields of 91.14%, 93% and 81.21%, for 5%, 10% and 15% PANI/PE, respectively. In this study, the proposed reaction mechanism involves the release of H atoms by the O atoms, as shown in Figure 2, because PE was acidic. The phenolic O atoms attack the benzene C atoms, forming a bond. Furthermore, the bond allows electrons to move farther, increasing the composite material's conductivity.

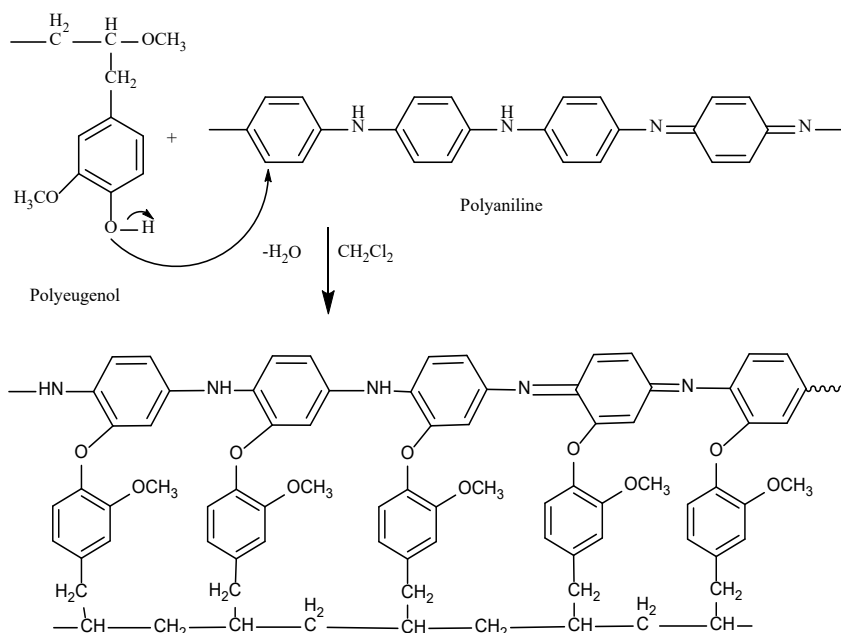


Figure 2: Approximation of one of the PANI/PE composite structures.

3.4 FTIR Analysis

The FTIR spectra of PANI/PE composites (see Figure 3) confirmed the emergence of characteristic absorption bands from both PANI and PE. For the 5%, 10% and 15% PANI/PE composites, typical PANI absorptions were observed at $1,580\text{ cm}^{-1}$ – $1,573\text{ cm}^{-1}$ for the $\text{C}=\text{C}$ stretching group of the quinoid ring. The absorptions at $1,487\text{ cm}^{-1}$ – $1,476\text{ cm}^{-1}$ were associated with the $\text{C}=\text{C}$ stretching group of the benzenoid ring. The bands at $1,290.43\text{ cm}^{-1}$, $1,294.29\text{ cm}^{-1}$ and $1,289.47\text{ cm}^{-1}$ were attributed to the $\text{C}-\text{N}$ groups. Additionally, the vibrations at $1,118.76\text{ cm}^{-1}$, $1,113.94\text{ cm}^{-1}$ and $1,101.40\text{ cm}^{-1}$ were related to the $-\text{NH}=$ group.

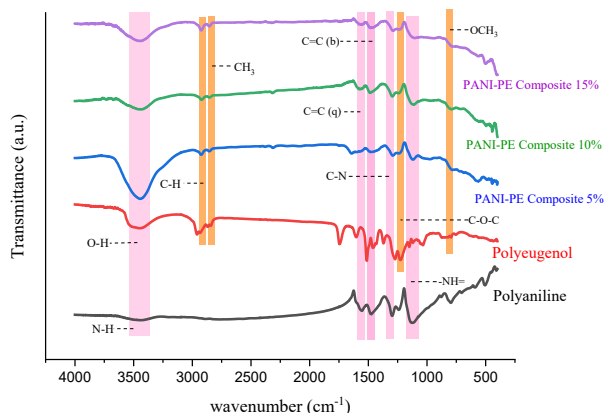


Figure 3: Comparison of FTIR spectra of PANI, PE and PANI/PE composites.

Furthermore, for the typical absorptions of PE for all PANI/PE composites, the bands at $3,446.94\text{ cm}^{-1}$, $3,425.72\text{ cm}^{-1}$ and $3,444.05\text{ cm}^{-1}$ revealed the presence of O-H groups. Furthermore, the absorptions at $2,924.21\text{ cm}^{-1}$, $2,921.32\text{ cm}^{-1}$ and $2,923.25\text{ cm}^{-1}$ were attributed to aromatic C-H groups. The bands at $2,855.73\text{ cm}^{-1}$, $2,853.81\text{ cm}^{-1}$ and $2,855.73\text{ cm}^{-1}$ were correlated with CH_3 groups. Additionally, the absorptions at $1,240.28\text{ cm}^{-1}$, $1,241.25\text{ cm}^{-1}$ and $1,232.57\text{ cm}^{-1}$ were assigned to C-O-C groups. The vibrations observed at 859.32 cm^{-1} , 867.04 cm^{-1} and 867.04 cm^{-1} , were attributed to $-\text{OCH}_3$ groups.

These results confirm successful interaction between PANI and PE in the composite structure. Variations in O–H absorption intensity among different compositions suggest differences in PE distribution within the PANI matrix, potentially due to local compositional inhomogeneity during blending.

Table 3: Comparison of functional group absorption in PANI, PE, and PANI/PE composites

Functional Group	Wavenumber (cm^{-1})				
	PANI	PE	5% PANI/PE Composite	10% PANI/PE Composite	15% PANI/PE Composite
N-H stretch	3,433	–	3,446.94	3,425.72	3,444.05
C=H stretch	–	2,968.58	2,924.00	2,921.00	2,923.00
C=C quinoid	1,554	–	1,580.36	1,573.02	1,581.70
$-\text{CH}_3$	–	1,601	1,562.00	1,573.00	1,581.00

(continue on next page)

Table 3: (continued)

Functional Group	Wavenumber (cm ⁻¹)				
	PANI	PE	5% PANI/PE Composite	10% PANI/PE Composite	15% PANI/PE Composite
C=C <i>benzonoid</i>	1,476	–	1,482.36	1,487.18	1,476.57
C-O-C	–	1,267.29	1,482.00	1,487.00	1,476.00
-C-N <i>stretch</i>	1,297.18	–	1,290.43	1,294.29	1,289.47
N-H=	1,145	–	1,118.76	1,113.94	1,101.40
Aromatic -OCH ₃	–	820.75	856.00	867.00	867.00

3.5 Viscosity Measurement

Intrinsic viscosity was determined using an Ostwald viscometer. Since intrinsic viscosity is directly proportional to molecular mass, a higher intrinsic viscosity indicates a greater polymer molecular weight.²²

Table 4: Intrinsic viscosity values of PANI

Concentration (g/mL)	Relative viscosity (η_r)	Specific viscosity (η_{sp})	Intrinsic viscosity (η_i)
0.001	1.18	0.181	189.51
0.0015	1.19	0.191	
0.002	1.20	0.199	
0.0025	1.20	0.204	

Note: DMSO comparison solvent with $\eta_r = 1.000$ and $\eta_{sp} = 0$

Table 5: Intrinsic viscosity values of PE

Concentration (g/mL)	Relative viscosity (η_r)	Specific viscosity (η_{sp})	Intrinsic viscosity (η_i)
0.005	1.14	0.143	138.16
0.01	1.23	0.226	
0.015	1.34	0.344	
0.02	1.43	0.429	

Note: Chloroform comparison solvent with $\eta_r = 1.000$ and $\eta_{sp} = 0$

Based on Tables 4 and 5, the intrinsic viscosity of PANI was greater than that of PE, indicating a relatively larger molecular mass. The incorporation of PE into PANI contributed to increased overall molecular interactions within the composite, which may enhance thermal resistance due to the combined macromolecular structure of both polymers.²⁷

3.6 Characterisation of Thermal Properties

The thermal properties of PANI, PE and the composites were analysed by TGA. This analysis was carried out to determine the mass loss of the polymer and the thermal resistance of in constituent materials over a temperature range of 25°C to 900°C. The thermograms of PANI, PE and PANI/PE composites are presented in Figure 4.

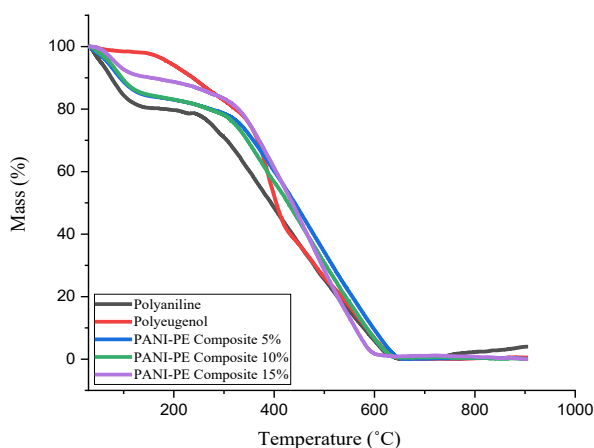


Figure 4: Termogram TGA of PANI, PE, and PANI/PE composites.

PANI experienced two stages of mass loss during heating under an oxygen atmosphere. The first mass loss stage occurred at temperatures of 50°C–150°C, due to water dehydration. Subsequently, the second mass loss was observed between 150°C and 400°C. PE exhibited a primary loss stage beginning at approximately 165°C, indicating excellent thermal resistance.

All PANI/PE composites in this study displayed two mass loss stages. The first stage, at 25°C–150°C, corresponded to the moisture loss or water dehydration. The second stage involved chain-breaking between PANI and PE, followed by the release of monomers. The mass reduction temperatures for 5%, 10% and 15% PANI/PE composites were 311°C, 297°C and 320°C, respectively.

Based on the TGA profile, increasing PE content improved the thermal resistance of the composite materials. However, the 10% PANI/PE composite showed slightly reduced stability compared with the 5% PANI/PE sample, attributed to non-uniform PE dispersion within the PANI matrix. This created weak interfacial regions, allowing heat to propagate more readily and thereby reducing the thermal stability.

3.7 Morphological Characterisation of PANI and 5% PANI/PE Composite

SEM-EDX analysis was performed to examine surface morphology, and the results are presented in Figure 5. Pure PANI exhibited granular morphology with large spherical particles and rough surfaces.

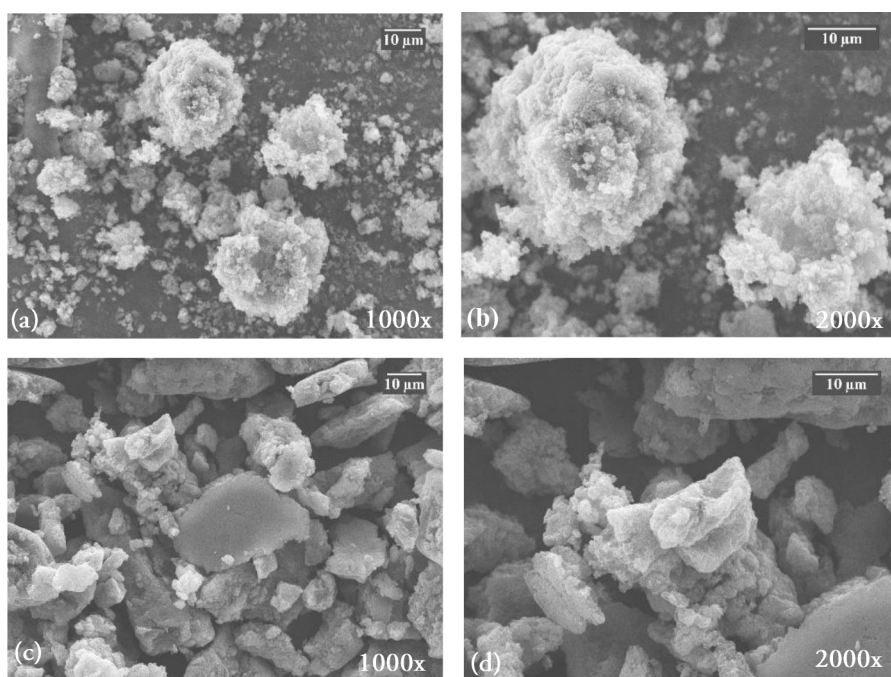


Figure 5: Morphology of (a) PANI 1000 $\times$, (b) PANI 2000 $\times$, (c) PANI/PE composite 1000 $\times$ and (d) PANI/PE composite 2000 $\times$.

Meanwhile, the 5% PANI/PE composite showed a similar morphology, with increased surface coverage and reduced interparticle voids. The presence of PE between PANI particles reduced particle spacing and improved interfacial contact, facilitating more efficient electron transport.

The results of EDX analysis for PANI and the 5% PANI/PE composite are presented in Table 6. It reveals the presence of C, O, N and Cl atoms in PANI. The emergence of O and Cl was due to their presence in the oxidant and dopants during synthesis. The 5% PANI/PE composite showed lower C content and higher O content than PANI. The decrease in C atoms could be caused by the distribution of atoms and the filling of PANI cavities. Furthermore, the increase in O atoms was due to the addition of PE, which contains hydroxyl groups (O-H). Cl was also found in the composite because these atoms were sourced from HCl in PANI.

Table 6: PANI EDX analysis results

Elements	%Atom	
	PANI (%)	5% PANI/PE Composite
Carbon (C)	60.25 ± 1.24	58.31 ± 0.83
Oxygen (O)	25.41 ± 2.71	26.64 ± 2.22
Nitrogen (N)	11.08 ± 1.15	13.65 ± 1.06
Chloride (Cl)	3.27 ± 0.16	1.40 ± 0.09

3.8 Conductivity Measurement

Conductivity is an important property of the PANI/PE composite material, as it is being considered for use as a conductive polymer. Electrical conductivity was measured using an LCR metre. The data were converted to a specific electrical resistivity for the PANI/PE composite, and the conductivity was then calculated.

Table 7: Conductivity of PANI, PANI/PE composites, and selected PANI-based composites

Material	Conductivity/ (S/cm)	
	Study Result	References
PANI	1.957×10^{-4}	1.1×10^{-4} . ²⁸
5% PANI/PE Composite	30.81×10^{-4}	(This study)
10% PANI/PE Composite	66.91×10^{-4}	(This study)
15% PANI/PE Composite	15.44×10^{-4}	(This study)
5% PANI/PS Composite	–	3.96×10^{-4} . ²⁹
10% PANI/PS Composite	–	2.24×10^{-4} . ²⁹
15% PANI/PS Composite	–	2.30×10^{-4} . ²⁹
PANI/PVDF composites		1.0×10^{-6} . ³⁰
HDPE/LLDPE/PANI blends		3.7×10^{-6} . ³¹
Epoxy-PANI composites		1×10^{-10} . ³²

Based on Table 7, the PANI/PE composites exhibited significantly enhanced conductivity compared with pure PANI. This concluded that the addition of PE to composite materials could increase conductivity due to its conduction bands. The bands were denser, resulting in the excitation process or the passage of electrons becoming faster. The highest conductivity was observed at 10% PANI/PE composite. This enhancement is attributed to improved interfacial interaction, homogeneous dispersion, and the formation of continuous conductive pathways.³³

At lower PE content (5% PANI/PE), dispersion was less optimal, limiting charge transport. Conversely, excessive PE (15% PANI/PE) introduced a relatively insulating phase, disrupting the continuity of conductive PANI domains and thereby reducing conductivity.

The synthesised PANI/PE composite demonstrated a trend similar to that reported by Narayanasamy et al., where an increase in composite composition led to enhanced electrical conductivity.¹¹ Overall, the 10% PANI/PE composite demonstrated the optimum balance between structural integration and electrical performance.

4. CONCLUSION

In conclusion, the PANI/PE composite was successfully synthesised by preparing PANI and PE separately, followed by subsequent blending. FTIR analysis confirmed the presence of characteristic functional groups originating from both PANI and PE, indicating successful composite formation. Morphological analysis showed that PE filled the interparticle voids of PANI and formed agglomerates attached to the PANI surface. TGA and electrical conductivity measurements showed that PANI/PE composites containing 5%, 10% and 15% PE exhibited better thermal stability and conductivity than pristine PANI. The thermal stability temperatures were 311°C (5% PANI/PE), 297°C (10% PANI/PE) and 320°C (15% PANI/PE), while the corresponding conductivity values were 30.81×10^{-4} S/cm, 66.91×10^{-4} S/cm and 15.44×10^{-4} S/cm, respectively.

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