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Morphological and Elemental Evolution of *Moringa oleifera* Leaf Extract-Doped Polyaniline Thin Films Fabricated by Spray Pyrolysis for Light-Harvesting Applications

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ABSTRACT

This study investigates the morphological and elemental characteristics of green-sensitized polyaniline (PANI) nanocomposite thin films, fabricated using a spray pyrolysis technique with *Moringa oleifera* leaf extract as a natural dopant. Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) were employed to analyze the surface structure, aggregation patterns, and elemental composition of thin films doped with varying volumes (0, 5, 10, 15, 20 mL) of the extract. Results indicate that the introduction of *Moringa oleifera* dye significantly alters the surface morphology of pure PANI, transforming it from a smooth, homogeneous film to a rougher, clustered texture. EDS confirmed successful dye integration, showing a progressive increase in oxygen content from approximately 2% in pure PANI to 25% in composites doped with 20 mL of the extract. This oxygen enrichment indicates functionalization with oxygenated phytochemical groups from the natural extract. These morphological changes enhanced surface area, porosity, and cluster formation that are directly contribute to improved light-trapping capabilities. In conclusion, *Moringa oleifera* leaf extract serves as an effective, eco-friendly natural dopant that modifies the phytochemical properties of PANI nanocomposite thin films, enabling their optimization for enhanced light-harvesting applications in dye-sensitized solar cells and photocatalysis.

Keywords:
Polyaniline,
Moringa oleifera,
Green Doping,
SEM,
EDS,
Morphology,
Thin Films,
Spray Pyrolysis.

INTRODUCTION

The pursuit of sustainable and efficient materials for optoelectronic devices has driven significant interest in conducting polymers like polyaniline (PANI). Its properties, including tunable conductivity and environmental stability, can be enhanced through doping and composite formation. Traditional doping of PANI typically relies on synthetic acids or metal salts to protonate the imine nitrogen atoms, transitioning the polymer from its insulating emeraldine base to the conductive emeraldine salt form (Tanweer *et al.,* 2024; Tewari *et al.,* 2023; Choudhary et al., 2023). However, many conventional dopants involve harsh, corrosive, or toxic chemicals, raising environmental and health concerns during synthesis, processing, and disposal. This has driven exploration of green alternatives, particularly natural plant derived extracts rich in bioactive compounds such as polyphenols, flavonoids, alkaloids, and other phytochemicals (Vinth *et al.,* 2022; Subramanian *et al.,* 2023; Maleji *et al.,* 2023). These biomolecules can act as reducing agents, stabilizers, dopants, or modifiers, enabling milder synthesis conditions while potentially introducing additional functional groups that influence electronic structure, charge transport, and interfacial properties (Nassar *et al.,* 2024; Sanches *et al.,* 2014; Botsa *et al.,* 2020). *Moringa oleifera* leaf extract, rich in polyphenols, chlorophyll, and other phytochemicals, presents a promising, renewable candidate for modifying PANI's electronic and physical structure (Prasad et al., 2016; Pant *et al.,* 2018; Tanweer et al., 2024; Beljin *et al.,* 2024).

The performance of thin films in applications such as dye-sensitized solar cells (DSSCs) and photocatalysis is critically dependent on their morphological attributes. A high surface area, porous structure, and optimal surface roughness improve light absorption, charge separation, and reactant accessibility (Bousalem *et al.,* 2020; Ahamad *et al.,* 2024); Andreas *et al.,* 2019; Azmayesh *et al.,* 2024; Tabrani *et al.,* 2025; Tesfaye *et al.,* 2025). While extensive literature documents the optical, electrical, and photocatalytic properties of acid-doped or metal-composited PANI thin films, studies specifically addressing *Moringa oleifera* mediated doping or compositing of PANI particularly with detailed correlations between synthesis parameters, morphological transformations using scanning electron microscopy (SEM) /Transmission Electron Microscopy(TEM)/ Atomic Force Microscopy (AFM) and elemental profiling via XPS/EDS and optoelectronic/light-harvesting performance remain relatively scarce.

This work focuses exclusively on the structural and compositional changes induced by *Moringa oleifera* dye in PANI thin films. Using SEM and EDS, we systematically analyze how varying the dye concentration influences film morphology, aggregation behavior, and elemental makeup, providing crucial insights for engineering green-sensitized materials for advanced light-harvesting technologies.

MATERIALS AND METHODS

Materials

Ammonium peroxide sulphate (APS) [$(NH_4)_2S_2O_8$], Hydrochloric acid (HCl), and Aniline [$C_6H_5NH_2$] as the reactants, *Moringa oleifera* leaf extract, glass substrates, electrical oven, distilled water, beakers, magnetic stirrer. All chemicals were analytical grade and used as received.

Synthesis of Polyaniline (PANI)

Polyaniline (PANI) was synthesized via chemical oxidative polymerization of aniline in an acidic medium, using ammonium persulfate (APS) as the oxidant. 0.3 M of aniline was dissolved in 0.5 M hydrochloric acid (HCl) solution under constant magnetic stirring at room temperature for 4 h to ensure complete protonation and homogeneous mixing. Subsequently, an equimolar amount of 0.5 M APS was added dropwise to the reaction mixture over a period of time while maintaining vigorous stirring. The polymerization reaction was continued with stirring for an additional 1 h. The resulting dark green reaction mixture was then allowed to stand undisturbed at room temperature for 24 h to complete the polymerization process.

The obtained PANI precipitate was filtered, washed repeatedly with distilled water until the filtrate became colorless, and subsequently used for thin film deposition via spray pyrolysis. All reactions were conducted under ambient laboratory conditions without additional temperature control beyond room temperature.

Thin Film Deposition via Spray Pyrolysis

Precursor solutions were prepared by mixing synthesized PANI with different volumes of Moringa dye extract (0, 5, 10, 15, and 20 mL). Films were deposited onto ultrasonically cleaned glass substrates using a custom spray pyrolysis system. The substrate temperature was maintained at 300°C, nozzle pressure was kept constant, and multiple passes ensured uniform thickness. The deposited films were annealed at 150°C for 1 hour to improve adhesion and crystallinity.

Characterization: SEM and EDS

The surface morphology and elemental composition of the PANI composites were analysed using SEM-EDX (Axia ChemiSEM). The samples were sputter-coated with a thin layer of gold to enhance conductivity prior to imaging. SEM micrographs were taken at various magnifications to observe surface texture, particle distribution, and aggregation patterns. All micrographs presented in this study were recorded at a uniform magnification of ×500 to enable consistent comparison of surface features across different dopant concentrations. EDS spectra were collected at multiple points on each sample to determine the average atomic percentage of constituent elements (Carbon, Nitrogen, Oxygen, Zinc, etc.) and ensure homogeneity. All experiments and characterizations were conducted in triplicate, and the presented results correspond to the average values obtained from these repeated measurements.

RESULTS AND DISCUSSION

Scanning Electron Microscopy (SEM) Analysis

Morphology of the PANI and PANI/*Moringa oleifera* dye nanocomposites with different volumes (5, 10, 15 and 20 mL) are shown in Figure1. Figure 1a exhibits a relatively smooth surface with minimal aggregation, indicative of a well-dispersed polymer matrix without substantial external modification. This morphology is advantageous for electrochemical applications such as supercapacitors but limits its utility in light-harvesting technologies such as dye-sensitized solar cells, where enhanced surface roughness is required to improve light trapping, absorption, and charge generation (Nassa *et al.,* 2024).

With the addition of 5 mL *Moringa oleifera* leaf extract, the film exhibited a markedly rougher texture accompanied by the emergence of small aggregates and clustered structures (Fig. 1b), evidencing effective interaction between the phytochemical dopant and PANI chains. The increased roughness and surface area enhance light absorption and likely introduce oxygenated functional groups that may facilitated improved electron transfer and charge separation. These features make the composite promising for dye-sensitized solar cells (DSSCs) and photocatalytic processes, including pollutant degradation under illumination (Nassar *et al.,* 2024). At 10 mL dye loading, larger clusters develop, producing a more heterogeneous and aggregated surface compared to the lower concentration (Figure 1c).

Although this morphology increases light absorption by increasing the photoactive sites, excessive aggregation may impair the mobility of the charge and encourage the recombination of electrons, thereby potentially decreasing the efficiency of the PV. Then, but also remains of great value for photocatalytic applications where surface area and trapping of light are of the highest importance (Tahoun *et al.,* 2023).

Further increase to 15 mL dye leads to even larger aggregates and pronounced clustering, yielding greater surface heterogeneity (Figure 1d). The rougher structure boosts light absorption capacity but heightens the risk of charge recombination and reduced mobility in photovoltaic devices. Conversely, the enlarged surface area strongly supports photocatalytic reactions such as water splitting and environmental pollutant degradation (Satyam & Patra, 2025).

The highest dye concentration (20 mL) produces the most aggregated morphology, characterized by prominent large clusters that create substantial light-trapping sites (Figure 1e). This configuration maximizes surface area for light interaction and absorption, rendering the material particularly suitable for photocatalytic applications, whereas charge transport limitations from recombination make it less ideal for solar cell performance.

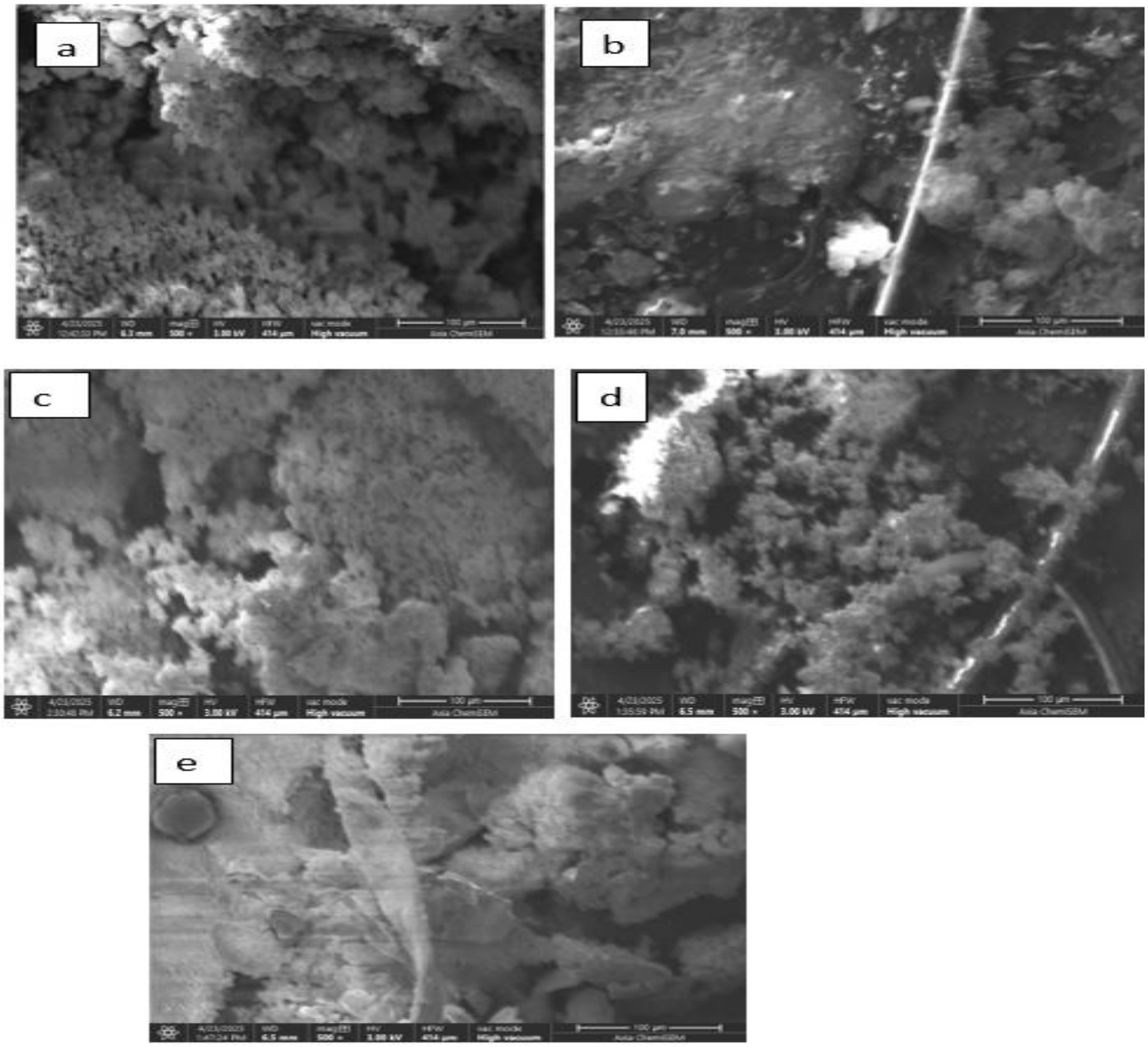

Figure 1: SEM Images at×500 Magnification (a) PANI, (b) PANI (5 mL of *Moringa oleifera* dye, (c) PANI (10 mL of *Moringa oleifera* dye), (d) PANI (15 mL of *Moringa oleifera* dye), (e) PANI (20 mL of *Moringa oleifera* dye)

Energy Dispersive X-Ray Analysis (EDX)

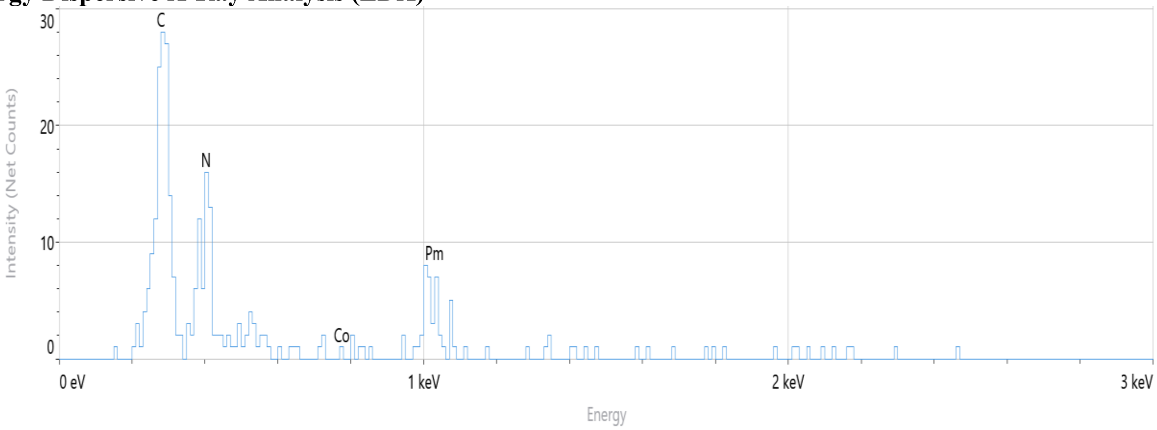

Figure 2: Intensity (counts) Versus Energy (keV) of EDS Analysis of the Spray-coated PANI Nanocomposite thin Film without *Moringa oleifera* dye

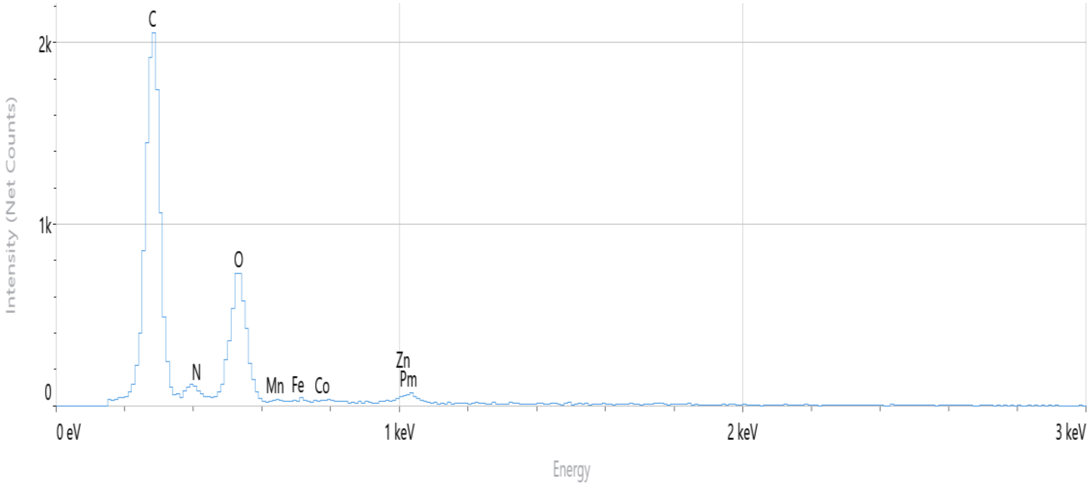

Figure 3: Intensity (counts) Versus Energy (keV) of EDS Analysis of the Spray-coated PANI Nanocomposite thin Film with 5mL of *Moringa oleifera* dye

Figure 4: Intensity (counts) VERSUS energy (keV) of EDS Analysis of the Spray-coated PANI Nanocomposite thin Film with 10 mL of *Moringa oleifera* dye

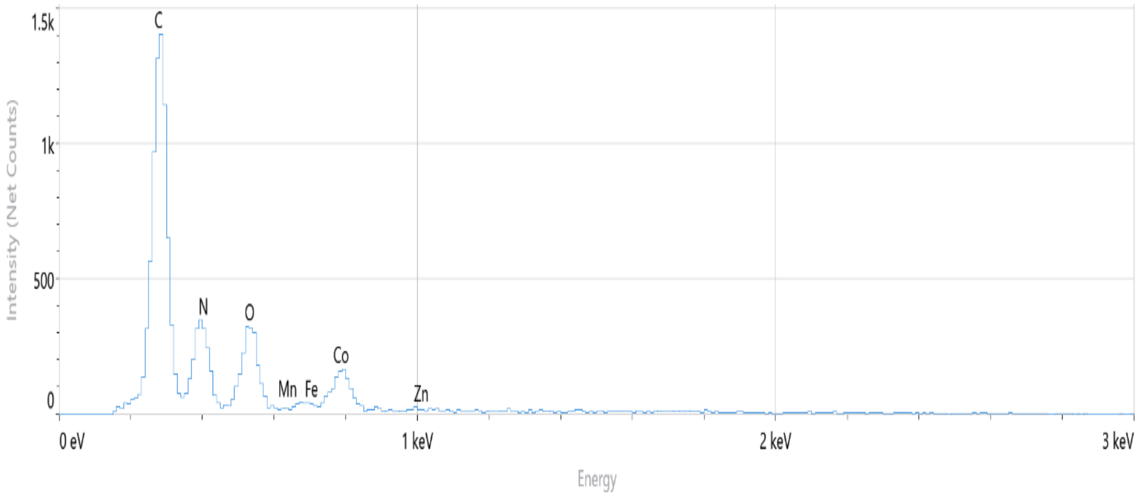

Figure 5: Intensity (counts) Versus Energy (keV) of EDS Analysis of the Spray-coated PANI Nanocomposite thin Film with 15 mL of *Moringa oleifera* dye

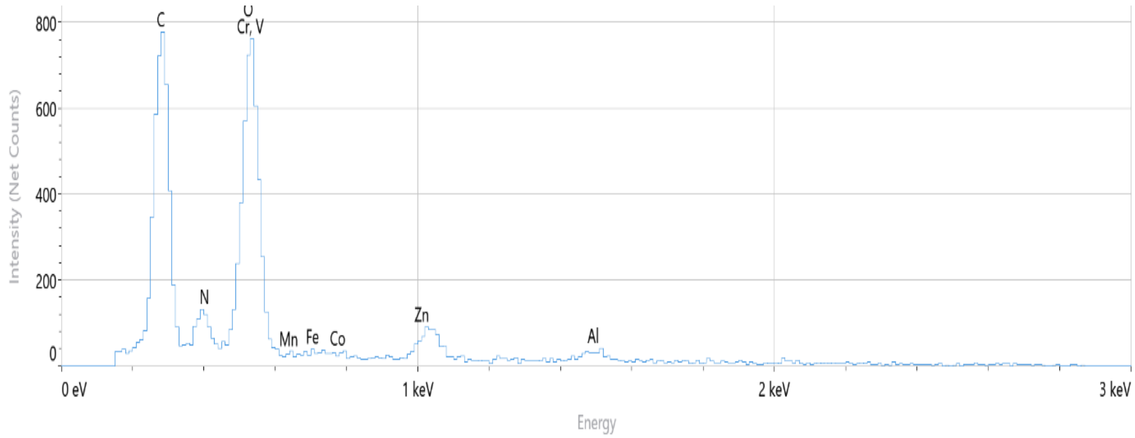

Figure 6: Intensity (counts) Versus Energy (keV) of EDS Analysis of the Spray-coated PANI Nanocomposite thin Film with 20 mL of *Moringa oleifera* dye

Energy-dispersive X-ray Spectroscopy Analysis (EDS)

The elemental characteristics of PANI and PANI/MO (*Moringa oleifera)* dye are presented in Figures 2 to 6. The EDS spectrum in Figure 2 reveals that the predominant elements in the pure PANI nanocomposite thin film are carbon (C) and nitrogen (N), with a minimal oxygen (O) content of approximately 2%. This confirms the absence of any functional groups that could promote light absorption or enhance charge separation in the material.

The low oxygen content indicates that the film is relatively simple and non-functionalized, limiting its ability to enhance charge transport and light harvesting in devices such as solar cells and photocatalytic reactors. While PANI is a conducting polymer, its performance in light-harvesting applications can be significantly improved by incorporating oxygenated groups that facilitate electron injection and charge mobility. The absence of these groups in the pure PANI matrix suggests that further modification with functional dyes, such as *Moringa oleifera,* is necessary to improve efficiency in these applications (Tahoun *et al.,* 2023; Maleji *et al.,* 2023).

The EDS spectrum of PANI with 5 mL of volume Moringa oleifera dye incorporatio (Figure 3) reveals an increase in oxygen content to ~10%. The elevated oxygen indicates successful introduction of oxygenated functional groups from the dye, enhancing charge separation, electron transfer, and light-harvesting properties. These improvements make the composite more effective for DSSCs and photocatalytic applications, including pollutant degradation (Nassar et al., 2024; Chomkotichai *et al.,* 2024).

The EDS spectrum showing further increase in oxygen content to ~15% at higher dye concentration (Figure 4) depicts progressive functionalization that boosts charge transfer, electron mobility, and light absorption, benefiting photovoltaic performance in DSSCs and photocatalytic efficiency for processes such as hydrogen production and environmental remediation (Kumar *et al.,* 2018; Bousalem *et al.,* 2020; Beljin *et al.,* 2024).

The EDS spectrum indicating significant oxygen enrichment to ~20% at 15 mL dye loading (Figure 5) shows that the heightened oxygenated groups markedly improve charge separation and light absorption, enhancing suitability for light-harvesting devices and photocatalysis (Tahoun *et al.,* 2023).

The EDS spectrum of the composite with 20 mL dye, exhibiting the highest oxygen content (~25%) (Figure 6), demonstrates substantial functionalization that maximizes light absorption and charge separation, favoring photocatalytic applications. However, combined with excessive aggregation (as noted in prior morphological analysis), it may compromise charge transport in photovoltaic devices like DSSCs (Serouti *et al.,* 2023; Bousalem *et al.,* 2020).

The elemental composition of the undoped polyaniline (PANI) and PANI/MO nanocomposite thin films was quantitatively evaluated by energy-dispersive X-ray spectroscopy (EDS). The average weight percentages of the detected elements, obtained from triplicate measurements on different regions of each sample, are summarized in Table 1.

Table 1: Average Weight (%) of Elements in EDS Analysis

Element	Polyaniline	PANI/MO at 5 mL	PANI/MO at 10 mL	PANI/MO at 15 mL	PANI/MO at 20 mL
C	20.9	27.8	15.0	27.6	10.6
N	77.3	3.8	10.0	9.8	3.2
S	8.0	4.8	8.7	38.1	10.7
Cl	1.0	2.4	11.0	14.0	9.3
O	12.8	26.1	19.0	17.4	22.5
Fe	0.0	0.8	0.0	0.0	0.0

As shown in Table 1, the pure PANI film exhibited a composition dominated by nitrogen (77.3 wt%) and carbon (20.9 wt%), with sulfur (8.0 wt%) and chlorine (1.0 wt%) originating from the ammonium peroxodisulfate oxidant and hydrochloric acid dopant used during oxidative polymerization, respectively. A baseline oxygen content of 12.8 wt% was also detected in the undoped film.

Upon incorporation of *Moringa oleifera* leaf extract, a clear increase in oxygen content was observed across all doped samples, rising to 26.1 wt% (5 mL), 19.0 wt% (10 mL), 17.4 wt% (15 mL), and 22.5 wt% (20 mL). This oxygen enrichment is attributed to the successful functionalization of the PANI matrix with oxygenated phytochemical groups (e.g., hydroxyl, carbonyl, and carboxyl moieties) present in the natural extract. Carbon and nitrogen contents showed moderate fluctuations, while sulfur and chlorine levels varied with dopant volume, reflecting interactions between the plant extract and the polymer backbone. A minor iron impurity (0.8 wt%) was detected only in the 5 mL doped sample, most likely originating from trace elements in the *Moringa oleifera* leaves.

These EDS findings align with prior studies on natural extract-mediated modifications of conductive polymers, where oxygenated phytochemicals from plant sources improve interfacial properties and functionality in nanocomposites (Tanweer *et al.,* 2024). The enhanced oxygen content and associated morphological changes directly support improved light-trapping efficiency, positioning these eco-friendly PANI/MO films as promising materials for applications in dye-sensitized solar cells and photocatalysis, building on related uses of *Moringa oleifera* extracts in photovoltaic and photocatalytic systems (Lorenzo, 2019).

CONCLUSION

The morphological and elemental analyses conducted in this study revealed that the inclusion of *Moringa oleifera* dye nanoparticles substantially modified the surface structure and elemental composition of PANI thin films. The incorporation of the dye enhanced surface roughness, increased oxygen content, and introduced functional groups that promote charge separation and electron transfer properties essential for efficient light harvesting systems. These results highlight the value of natural dye sensitization for tailoring PANI-based nanocomposites toward sustainable energy and environmental applications. Future work will include full device fabrication and optoelectronic testing to quantify efficiency gains.

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