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Morphology and Mechanical Properties of Polyvinyl Alcohol Nanofibres Incorporated with Graphene Oxide

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Abstract: *GO from waste tyres offers a promising route for eco-friendly nanocomposites. However, research on its incorporation into electrospun PVA nanofibres is rather limited. The study aimed to evaluate the morphological structures and mechanical properties of PVA/GO nanofibre membranes at three GO concentrations: 0.025 wt%, 0.050 wt%, and 0.075 wt%. SEM and EDX analyzed morphology and composition, while tensile testing assessed the mechanical properties of the membranes. The SEM results revealed that incorporating GO into PVA produced more uniform and densely packed fibres, reducing the average PVA fibre diameter from 543.1 nm to 299.7 nm. The EDX confirmed GO incorporation, with increasing carbon and oxygen peaks. Mechanical testing revealed that PVA had the highest tensile stress (6.55 MPa) and elongation at break (80.55 %), while PVA/GO membranes showed reduced values, likely due to poor GO dispersion. These findings demonstrate the feasibility of using waste tyre-derived GO in PVA nanofibres, highlighting their potential for several applications.*

Keywords: Graphene Oxide, Polyvinyl Alcohol, electrospinning, FE-SEM, Mechanical properties.

1. INTRODUCTION

The development of nanofibre membranes has gained significant attention due to their unique properties, including a high surface area-to-volume ratio, high porosity, and low density, making them suitable for various applications in healthcare, materials science, bioengineering, catalysis, energy, environmental remediation and agriculture [1-5]. Electrospinning is a well-established technique for producing nanofibres, valued for its straightforward equipment and process, high yield, cost-effectiveness, and versatility with a wide range of polymer types [6-10]. It allows for precise control over fibre size uniformity and orientation, as well as porosity, which can be adjusted through various experimental parameters [11]. Among the polymers used, polyvinyl alcohol (PVA) stands out as a versatile option, known for its ease of processing, ability to cross-link, and compatibility with various additives [12]. Recent advancements in nanocomposites have demonstrated that incorporating nanomaterials, such as carbon nanotube (CNT), zinc oxide (ZnO), and silica nanoparticles (SiO_2) into PVA can significantly enhance its mechanical, thermal, and electrical properties [13-14].

As a form of carbon nanotube, graphene, renowned for its exceptional mechanical strength and conductivity, has been incorporated into polymer matrices for various advanced applications [15]. However, conventional sources of GO rely on graphite, a carbon-based material are obtained from natural sources. In contrast, recent efforts have focused on sustainable methods of synthesizing GO from waste materials, such as used tyres, to reduce environmental impact [16]. GO derived from waste tyres offers the potential to create high-performance products, eco-friendly nanocomposites [17]. Yet, limited research has been conducted on the integration of tyre-derived GO into PVA electrospun nanofibres, especially concerning their morphology, and mechanical properties.

Despite the promising characteristics of GO, its incorporation into electrospun nanofibres, particularly those derived from waste tyres, remains underexplored. Previous research has focused on the use of chemically synthesized GO in polymer matrices [18], highlighting the need to evaluate the morphological structure, and mechanical properties of the GO incorporated with PVA nanofibres.

The objectives of this research were to evaluate the morphological structure and elemental composition of electrospun PVA nanofibres incorporated with synthesized graphene oxide (GO) derived from waste tyres through SEM and EDX analysis. Additionally, the study aimed to investigate the mechanical properties, such as tensile strength and elongation at break, at different GO concentrations.

In this study, GO had been synthesized from waste tyres using a modified Hummer's method and then incorporated into PVA solutions at different concentrations (0.025, 0.050, and 0.075 wt%). The nanofibres were produced through electrospinning, and the resulting materials underwent the SEM-EDX analysis for morphological characterization, and mechanical testing for tensile properties. The TGA was used to support findings related to mechanical properties of the samples.

This research focuses on creating environmentally sustainable nanofibres with improved mechanical properties by incorporating graphene oxide (GO) derived from waste tyres into PVA. By employing the FESEM and EDX analyses, important information regarding the morphological structure and elemental composition of electrospun GO/PVA nanofibre can be discovered. These methods are essential for comprehending the behavior of nanofibres and maximizing performance for a range of applications, building on earlier research [19-21]. The outcomes will offer valuable insights into potential applications in industries like filtration, biomedical devices, and energy

storage, while simultaneously promoting sustainable practices in material science by utilizing waste materials in nanotechnology.

2. MATERIALS AND METHODS

2.1 Materials

The primary materials used in this study include Polyvinyl Alcohol (PVA, Mw 125,000) sourced from Sigma-Aldrich and graphene oxide (GO) synthesized from waste tyres using the modified Hummers method as detailed in a previous study [16]. Distilled water was used as the solvent. Four different nanofibre membranes were produced using electrospinning (Fig. 1), which were controlled PVA membrane and three GO/PVA electrospun membranes with different GO concentrations namely 0.025 wt%, 0.050 wt%, and 0.075 wt%, respectively.

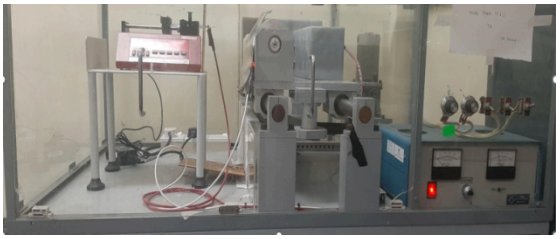


Fig. 1. Electrospinning setup used in the study.

2.2 Solution Preparation

For the preparation of the solutions, 12 wt% of PVA solution was prepared by dissolving the PVA into distilled water at a temperature of 80 - 90 °C for 3 - 4 hours. Once the homogeneous PVA solution was obtained, the solution was cooled down to a room temperature. GO was added into the three PVA solutions in varying concentrations which were 0.025 wt%, 0.050 wt%, 0.075 wt%. The mixtures were further stirred at room temperature for 4 - 5 hours to ensure homogeneity, after which the PVA/GO solutions were ready for electrospinning.

2.3 Electrospinning Process

The prepared PVA and PVA/GO solutions were loaded into a syringe with a stainless-steel needle. The syringe was set in a syringe pumping machine. A high-voltage supply ranging from 10 kV - 13 kV was applied during the electrospinning process with a feed rate of 0.2 mL/min. The nanofibres were collected on an aluminum foil placed 12 cm away from the needle. The collection process was carried out for 3 hours for each solution to form a nanofibre membrane.

2.4 Morphological and elemental composition analysis

The morphology of the nanofibre membranes was observed using a Scanning Electron Microscope (SEM-EDX) (Hitachi TM3000). The samples were gold-coated to enhance conductivity, and images were captured at 5000x and 10000x magnifications. Meanwhile, the Energy Dispersive X-ray Spectroscopy (EDX) was used to analyze the elemental composition of the fibres.

2.5 Mechanical Testing

Tensile strength and elongation at break were measured using the Tenso-Lab 4 machine with a loading rate of 5 mm/min and a 100 Newton load cell. Twelve dog bone-shaped samples were cut for each type of the nanofibre membranes, and their mechanical properties were evaluated.

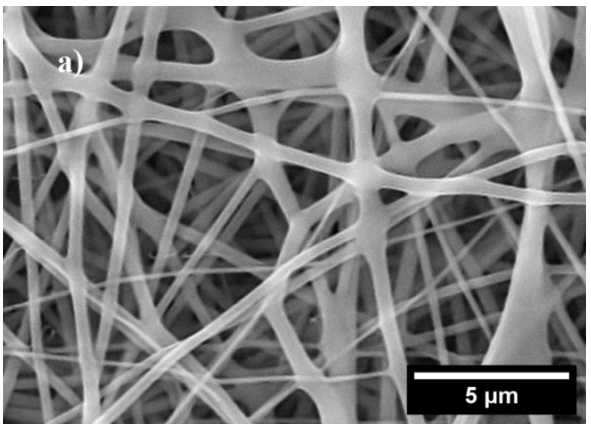
2.6 Thermal Gravimetric Analysis

The thermal degradation of the PVA and PVA/GO nanofibre membranes were tested using thermal gravimetric analysis (TGA). The experimental works were carried out using an SDT Q600 from TA Instrument under a nitrogen atmosphere in the range of 20 to 500 °C with a heating rate of 5 °C/min.

3. RESULTS AND DISCUSSION

3.1 Morphology and elemental composition analysis of controlled PVA and PVA/GO nanofibre membranes using SEM-EDX

The SEM images of controlled PVA nanofibres (Fig. 2a) show irregularities with occasional gaps and less uniformity than GO samples, suggesting a pure, filler-free PVA composition. This aligns with Wang et al. [22], who reported irregularity of pure PVA fibres. When 0.025 wt% GO was added, the PVA/GO fibres appeared thinner, smoother, and more interconnected (Fig. 2b). GO inclusion enhances fibre uniformity and smoothness. At 0.050 wt% GO, the fibres formed a denser, more twisted network with GO agglomerates, improving interconnectivity (Fig. 2c). At 0.075 wt% GO (Fig.2d), the fibres were thinner with visible GO agglomerates. Zhang et al. [15] also reported bead formation in PVA/GO composites with increased GO, affecting fibre uniformity. From the observation, it shows that as the concentration of GO increases, it reduces the resultant diameter of PVA nanofibres. It is expected that the GO enhances the PVA solution conductivity, leading to a greater stretching of polymer jets during electrospinning, which can result in thinner fibres. Similar observation was also reported by several studies [22-26].



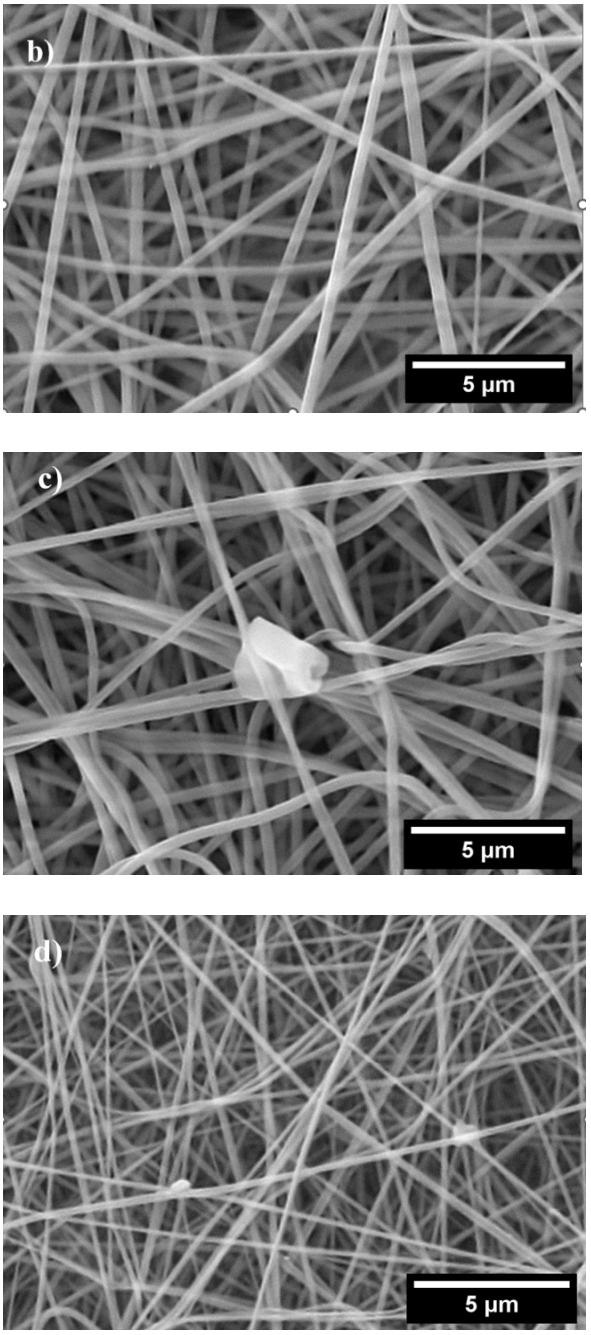


Fig. 2. SEM Image of Nanofibre Membrane at 10000x Magnification a) Controlled PVA b) PVA/0.025 wt% GO, c) PVA/0.050 wt% GO, and d) PVA/0.075 wt% GO.

In addition, the fibre diameter measurements reveal a significant reduction in diameter with GO incorporation (Fig. 3). Controlled PVA fibres have an average diameter of 543.1 ± 188.9 nm, showing high variability. Adding 0.025 wt% GO reduced the diameter to 403.1 ± 115.7 nm with improved uniformity, likely due to the presence of GO, which enhanced the solution conductivity of PVA during electrospinning. At 0.050 wt% GO, the diameter slightly increased to 446.4 ± 95.5 nm but remained more uniform than the controlled PVA. With 0.075 wt% GO, the smallest diameter (299.7 ± 56.5 nm) was achieved. This trend matches Abdullah et al. [23], who observed thinner and more uniform fibres with GO.

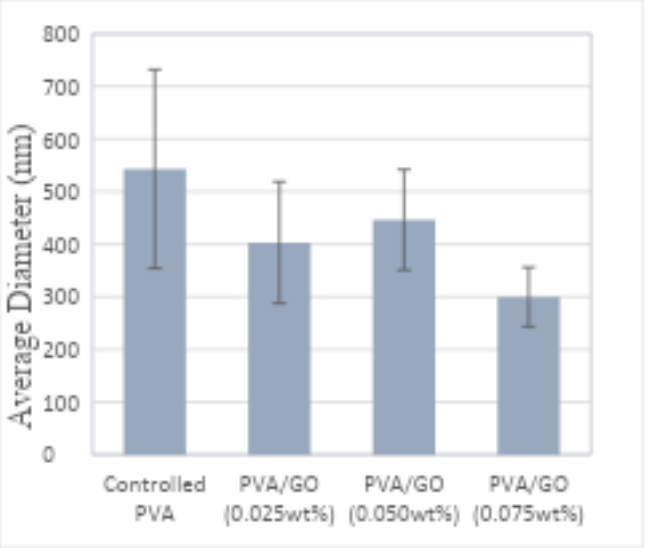
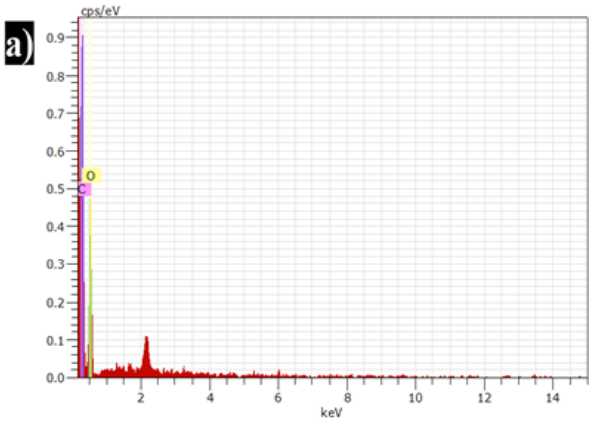


Fig. 3. Average fibre diameter for controlled PVA and PVA incorporated with 0.025 wt%, 0.050 wt%, and 0.075 wt% GO.

3.2 Elemental Composition Analysis

An elemental composition analysis was conducted using Energy Dispersive X-ray Spectroscopy (EDX). As shown in Fig. 4 (a, b, c, d), the GO has successfully incorporated into the PVA nanofibres. The controlled PVA sample served as a reference for comparison, and the spectra revealed progressively higher carbon peak intensities with increasing GO concentrations from 0.025 wt% to 0.075 wt%. The proportional increase in oxygen intensity alongside carbon indicated the presence of oxygen-rich functional groups in GO. The GO used in this study was produced via the modified Hummers method, which is known to introduce a significant amount of oxygen-containing functional groups to the graphene sheets. In Table 1, the controlled PVA exhibits the lowest carbon and oxygen peaks, while the PVA/GO composite with 0.075 wt% GO displays the highest peaks for both elements. This confirms that the amount of GO incorporated into the PVA matrix increased proportionally with the GO concentration, demonstrating the effectiveness of the fabrication process.



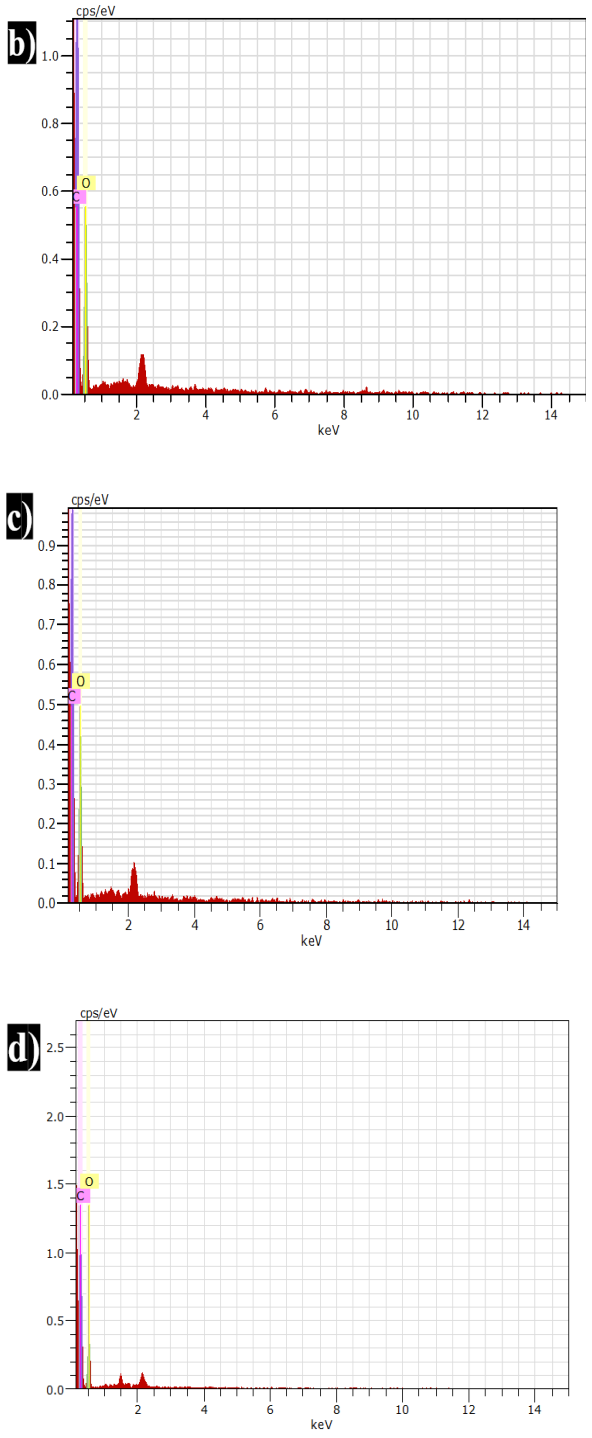


Fig. 4: EDX spectra of a) controlled PVA b) PVA/GO with 0.025 wt%g c) PVA/GO with 0.050 wt% d) PVA/GO with 0.075 wt%

Table 1: Elemental compositions of controlled PVA, PVA/0.025 wt% GO, PVA/0.050 wt% GO, and PVA/0.075 wt%GO, respectively.

Sample	Atomic weight (%)	
	C K	O K
Controlled PVA	71.61	25.95
PVA/GO (0.025 wt%)	72.53	25.67
PVA/GO (0.050 wt%)	73.44	23.16
PVA/GO (0.075 wt%)	74.18	23.34

3.3 Mechanical properties of controlled PVA and PVA/GO nanofibre membranes

The mechanical properties of the nanofibre membranes show that GO incorporation influences both tensile stress (σ) and elongation at break (Fig.5). The controlled PVA exhibits the highest tensile strength and elongation at break compared to PVA with GO. The tensile strength of the controlled PVA is approximately 6.55 MPa and the elongation at break is about 80.55 %. The controlled PVA provides continuous polymer network, allowing better load distribution during stretching. Furthermore, the addition of GO can disrupt the regular arrangement of PVA chains. If GO is not well-dispersed, it weakens the material and reduces its ability to elongate before breaking. GO is expected to restrict the mobility of PVA chains, making the material more brittle and reduce its elongation at break. According to the study of Botlhoko et al., the primary reason the elongation at break reduces with GO loading is because the membranes become more brittle as the GO content rises [27]. As shown in Fig. 5, the addition of 0.075 wt% GO reduces the tensile stress by 12.8 % compared to the controlled PVA, while the elongation at break significantly decreases by 48.93 %.

As the GO concentration increased to 0.050 wt%, the tensile stress of the membrane was slightly reduced by 2.28 % compared to the 0.075 wt% GO sample. The elongation at break remained relatively high, indicating a balance between strength and flexibility. However, with a lower GO content of 0.025 wt%, the tensile stress decreased further by 36.38 % relative to the 0.050 wt% GO membrane, accompanied by a 14.25 % reduction in elongation at break. From this analysis, it shows that the presence of GO in the samples may have a weak interfacial bonding with PVA. To further investigate, a TGA analysis was conducted.

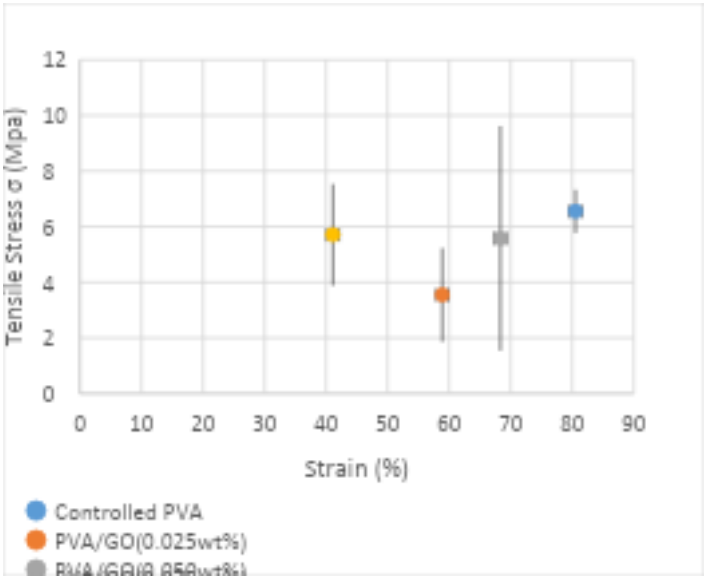


Fig. 5. Tensile stress–Elongation at break of nanofibre membranes with various GO loading. (Each dot represents the average results of 3 specimens)

Fig. 6 depicts that the thermal stability of PVA nanofibres decreased with increasing GO concentration.

The degradation temperature of controlled PVA nanofibre membrane is approximately 310 °C. With the addition of GO in the PVA, the degradation temperature decreased by up to 16 %, suggesting a poor dispersion or weak interfacial bonding between GO and PVA. The 0.025 wt% GO sample exhibits the weakest interfacial bonding, resulting in the lowest degradation temperature, around 260 °C. Meanwhile, the 0.050 wt% and 0.075 wt% GO membranes demonstrate better thermal stability, indicating improved dispersion and bonding compared to the 0.025 wt% GO sample. In addition, the 0.075 wt% GO sample shows a slightly higher thermal stability than the 0.050 wt% sample, but may experience GO aggregation, which can reduce elongation at break as shown in Fig. 5.

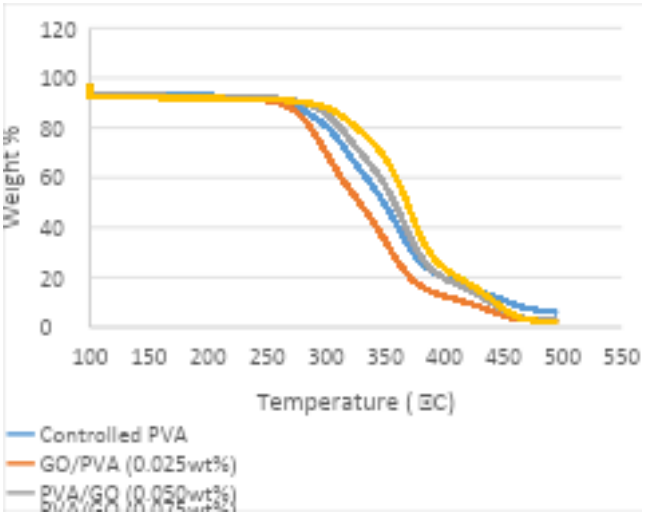


Fig. 6. TGA Curves of controlled PVA and GO/PVA nanofibre membranes.

4. CONCLUSIONS

The study demonstrates the feasibility to incorporate tyre-derived graphene oxide (GO) at three different concentrations into polyvinyl alcohol (PVA) nanofibres. The SEM analysis revealed that pure PVA nanofibres exhibited irregular structures, while GO incorporation improved uniformity and reduced fibre diameter. The addition of 0.025 wt% GO decreased the diameter from 543.1 nm to 403.1 nm, while 0.075 wt% GO resulted in the smallest diameter of 299.7 nm. The EDX confirmed the incorporation of GO in PVA, showing progressively higher carbon and oxygen peaks with increasing GO concentrations. The mechanical testing indicates that the control sample PVA had the highest tensile stress (6.55 MPa) and elongation at break (80.55 %). GO incorporation reduced these properties due to poor dispersion and restricted PVA chain mobility. The 0.025 wt% GO sample exhibited the weakest interfacial bonding, significantly affecting the sample mechanical properties. These findings highlight the potential of GO/PVA nanofibres for various applications but emphasize the need for improved dispersion strategies.

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