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Electrospun PA12 Nanofibers: Effect of Solution Parameters on Fiber Morphology

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ABSTRACT

Carbon fiber-reinforced polymers (CFRPs) are widely used in different industries due to their exceptional mechanical properties and low density. However, delamination and poor vibration damping capacity pose significant challenges to their application. This study focuses on the production of nanofibrous polyamide 12 (Nylon 12) mats obtained through electrospinning to improve the structural properties of epoxy CFRP laminates. Three different Nylon 12 solutions have been prepared using various solvents, and the process parameters for electrospinning have been optimized. The nanofibers produced by the solution with a mixture of formic acid and anisole solvent showed different morphologies depending on the solution concentration. An increase in Nylon 12 concentration leads to thicker, porous fibers. Thermal analysis has demonstrated that the electrospinning process does not alter the thermal properties of the polyamide. The results suggest that the use of PA12 nanofibrous membranes could represent a promising strategy for improving interlaminar fracture toughness in laminated CFRPs.

1 | Introduction

Carbon fiber reinforced polymers (CFRPs) are renowned for their exceptional mechanical properties, often surpassing those of traditional metals while maintaining a lower density. These advantageous characteristics have led to an increasing adoption in critical sectors such as aerospace, automotive, and sports, where there is a pressing need to combine outstanding mechanical performance with lightweight materials [1, 2]. Laminated CFRPs, which are multilayered composites created by stacking several layers, face certain challenges that may hinder their broader application, notably delamination and limited vibration damping capabilities [3]. To enhance the quality and safety of laminated composites and extend their service life, the scientific community is actively exploring various strategies to mitigate delamination and improve damping properties. Increasing the durability of these materials could reduce resource consumption and waste, thus decreasing the environmental impact and enabling higher sustainability of the components produced. The integration of membranes based on rubbery nanofibers between CFRP layers is highly effective in hindering delamination and improving damping capacity [4, 5] however, it may result in a reduction of other thermomechanical properties due to the low thermal properties of the integrated elastomer. The use of polyamides, especially Nylon 6 and 66, is common for reinforcing composites [6]. In this case, thanks to their melting temperature being higher than the glass transition temperature (Tg) of common matrices, the other laminate's thermomechanical properties

TABLE 1 | Solutions details and electrospinning parameters.

Polymeric solution	Flow rate (*mL/h*)	Electric potential (*kV*)	Needle-collector distance (*cm*)	Needle diameter (*mm*)
Nylon 12 (8% wt.) in Formic acid/DCM (1:1 wt.)	0.75	15	20	0.51
Nylon 12 (8% wt.) in Formic acid/Anisole (6:4 wt.)	0.5	15	10	0.51
Nylon 12 (15% wt.) in Formic acid/Anisole (6:4 wt.)	0.8	17.5	10	0.84

remain unaffected. In this context, the use of Nylon 12 nanofibers has not yet been investigated against delamination. The aim of this experimental work is to produce nanofibrous membranes composed of polyamide 12 via the electrospinning process to enhance the interlaminar properties of laminated CFRPs.

2 | Materials and Methods

Nylon 12 pellets and solvents, all Sigma–Aldrich, were utilized without any preliminary treatment or purification. To prepare the Nylon 12 solutions, the polymer was introduced at a concentration of 8 %wt. into vials containing either a single solvent (anisole) or a solvent mixture (formic acid/DCM and formic acid/anisole). The vials were subjected to continuous magnetic stirring and controlled heating at 80°C for a minimum of 3 h to ensure complete dissolution of the polymer. The electrospinning apparatus utilized for membrane production comprised a high-voltage generator, a syringe infusion pump with an adjustable flow rate, a syringe serving as a reservoir for the polymer solution, a needle, and a grounded collector covered with a polyethylene-coated sheet for the collection of the nanofibrous membrane. The electrospinning parameters applied to the prepared solutions are summarized in Table 1.

The thermal properties of the membranes were evaluated using Differential Scanning Calorimetry (DSC, Q2000 from TA Instruments). Scanning Electron Microscopy (SEM) analyses were conducted using a ThermoFisher Phenom ProX benchtop SEM, after gold sputter coating.

3 | Results and Discussion

The electrospinning process for producing polymeric nanofibers requires a homogeneous solution. For this reason, at first, it was necessary to identify an appropriate solvent or solvent system to effectively solubilize the polymer. Indeed, the sole use of formic acid, a common solvent for polyamides [7], is insufficient for dissolving Nylon 12. According to Behler et al. [8], a solvent mixture composed of formic acid and dichloromethane (DCM) in a 1:1 wt ratio effectively dissolves both Nylon 11 and Nylon 12. Conversely, Meireman et al. tested the dissolution of PA11 using a mixture of formic acid combined with 30 other solvents; the optimal result was achieved with an 8 %wt. polymer concentration in a formic acid/anisole 6:4 mixture, which allowed for complete dissolution of the polymer at room temperature [9]. In the present work, such solvent system was tentatively used for PA12 dissolution. Nylon 12 dissolution in pure anisole was incomplete, with some pellets remaining intact even after 48 h at 80°C. Therefore, the formic acid/DCM (FA/DCM) and formic acid/anisole (FA/An) solvent systems were used to produce the polymeric solutions. In particular, a 1:1 wt FA/DCM mixture and a 6:4 wt FA/An mixture were selected, as listed in Table 1. The FA/DCM mixture allowed for the complete dissolution of PA12 at 80°C after 3 h. The FA/An mixture required 24 h of stirring at 80°C to achieve complete solubilization and obtain a homogeneous solution suitable for the electrospinning process. Then, appropriate electrospinning process parameters (flow rate, voltage, needle-to-collector distance, needle diameter) were found. As a result, macroscopically uniform nanofibrous mats were obtained. SEM analysis (Figure 1A) revealed significant inhomogeneities in the fibers' morphology obtained by electrospinning the FA/DCM solution at 8 %wt. concentration, with some exhibiting a "ribbon-shaped" morphology. In contrast, the fibers produced from the FA/An solution at 8 %wt. concentration (Figure 1B) displayed a regular morphology free from defects or beads, with an average fiber diameter of 166 ± 30 nm.

Since the reinforcing action of the membrane toward the laminate's interlaminar fracture toughness may be affected by nanofibers' morphology (e.g., fiber diameter and fiber surface), a different solution was produced to obtain higher diameters. The concentration of Nylon 12 was increased from 8 to 15 %wt., using the same solvent system (FA/An). The resulting fibers, shown in Figure 2, exhibited an average diameter approximately doubled (341 ± 90 nm) compared to the one deriving from the 8 %wt. solution, with a porous surface morphology.

The thermal properties of the two distinct membranes derived from FA/An solutions (8 and 15 %wt. concentration) were evaluated. DSC analysis (Figure 3) indicates that the thermal characteristics of both membranes remain consistent, despite being produced from solutions with different concentrations and exhibiting significantly different morphologies. This finding suggests that the electrospinning technique does not influence the fundamental thermal properties of the membranes.

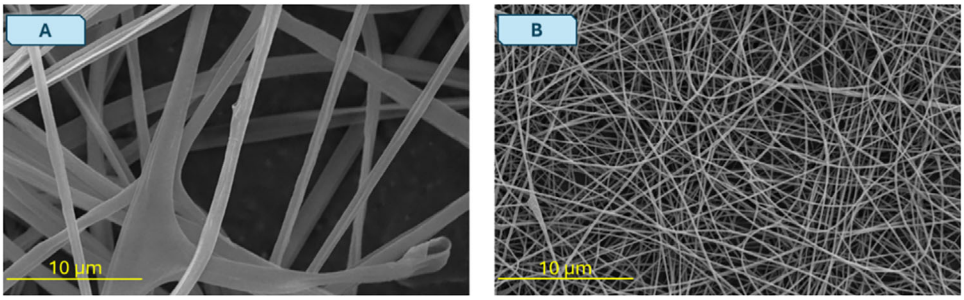

FIGURE 1 | (A) fibers produced from the FA/DCM solution at 8 %wt. concentration; (B) nanofibers obtained from the FA/An solution at 8 %wt. concentration.

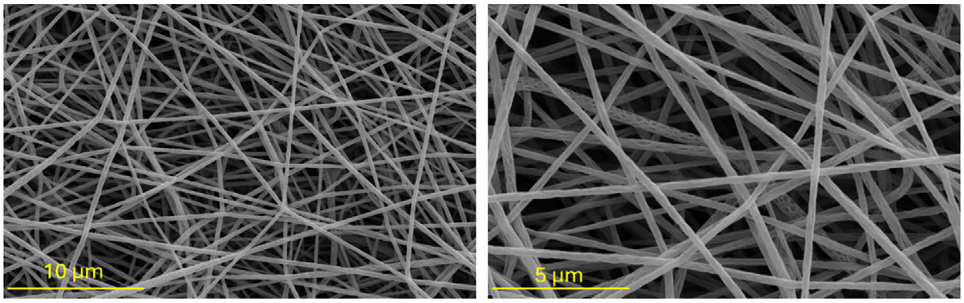

FIGURE 2 | SEM of nanofibers obtained from electrospinning of 15 %wt. solution in FA/An.

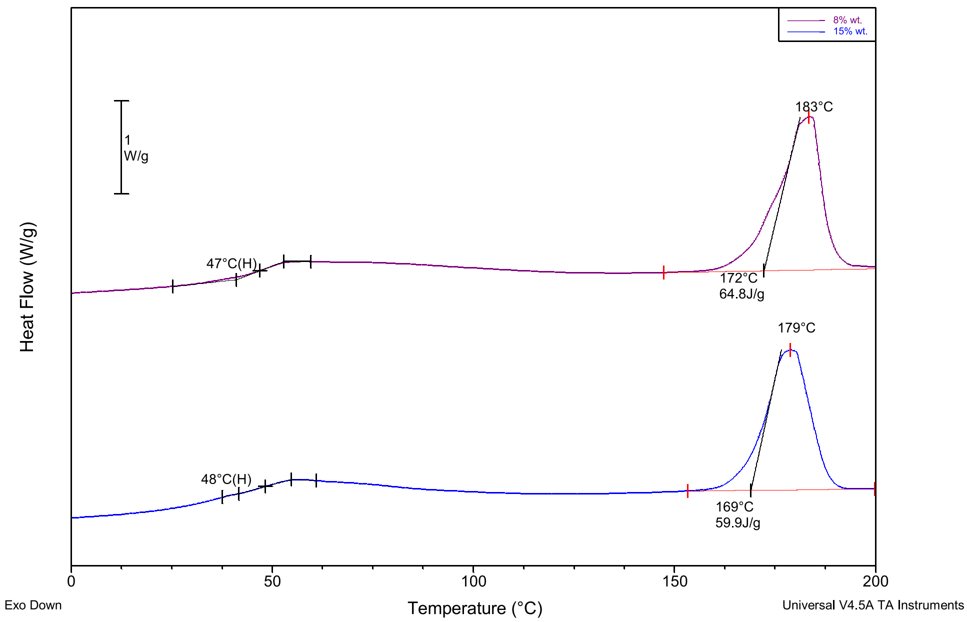

FIGURE 3 | DSC thermograms of electrospun Nylon 12 membranes from 8 %wt. (purple) and 15 %wt. (blue) solutions in FA/An solvent system.

4 | Conclusions

This study successfully demonstrated the feasibility of producing nanofibrous polyamide 12 membranes suitable for integration into CFRP laminates, aimed at enhancing their interlaminar properties. The electrospun membranes were characterized through SEM, revealing a defect-free morphology. By varying the solution concentration, i.e., 8 and 15 %wt., distinct membrane morphologies were obtained, which may differently affect the interlaminar fracture toughness of the nanomodified laminates.

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Conflicts of Interest

The authors declare no conflicts of interest.

References

1. E. Arussi, J. Reiland, M. Ierides, and L. Borghero, Composite Materials: A Hidden Opportunity for the Circular Economy Authors.

2. C. Zhang, X. Zhang, Y. Ling, et al., "A Novel Eco-friendly Strategy on the Interfacial Modification of a Carbon-fiber-reinforced Polymer Composite via Chitosan Encapsulation†," *Materials Chemistry Frontiers* 6 (2022): 765–774.

3. Z. Zhang, P. Myler, E. Zhou, and R. Zhou, "Strength and Deformation Characteristics of Carbon Fibre Reinforced Composite Wrapped Aluminium Foam Beams," *Journal of Composites Science* 6 (2022): 288, https://doi.org/10.3390/jcs6100288.

4. H. Saghafi, I. Ahmadi, R. Khamedi, et al., "The Thickness Effect of Rubbery Nanofibrous Mat on Modes I–II Fracture Mechanism of Composite Laminates," *Thin-Walled Structures* 196 (2024): 111556, https://doi.org/10.1016/j.tws.2024.111556.

5. M. P. Falaschetti, F. Rondina, E. Maccaferri, et al., "Improving the Crashworthiness of CFRP Structures by Rubbery Nanofibrous Interlayers," *Composite Structures* 311 (2023): 116845, https://doi.org/10.1016/j.compstruct.2023.116845.

6. T. Brugo and R. Palazzetti, "The Effect of Thickness of Nylon 6,6 Nanofibrous Mat on Modes I–II Fracture Mechanics of UD and Woven Composite Laminates," *Composite Structures* 154 (2016): 172–178, https://doi.org/10.1016/j.compstruct.2016.07.034.

7. E. Maccaferri, L. Mazzocchetti, T. Benelli, et al., "Is Graphene Always Effective in Reinforcing Composites? The Case of Highly Graphene-Modified Thermoplastic Nanofibers and Their Unfortunate Application in CFRP Laminates," *Polymers* 14 (2022): 5565, https://doi.org/10.3390/polym14245565.

8. K. Behler, M. Havel, and Y. Gogotsi, "New Solvent for Polyamides and Its Application to the Electrospinning of Polyamides 11 and 12," *Polymer* 48 (2007): 6617–6621, https://doi.org/10.1016/j.polymer.2007.08.058.

9. T. Meireman, L. Daelemans, S. Rijckaert, H. Rahier, W. Van Paepegem, and K. De Clerck, "Delamination Resistant Composites by Interleaving Bio-based Long-chain Polyamide Nanofibers Through Optimal Control of fiber Diameter and fiber Morphology," *Composites Science and Technology* 193 (2020): 108126, https://doi.org/10.1016/j.compscitech.2020.108126.