



Enhancing thermal and mechanical properties of polycaprolactone nanofibers with graphene and graphene oxide reinforcement for biomedical applications

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ABSTRACT

This study aimed to enhance the mechanical, thermal, and biocompatibility properties of polycaprolactone (PCL) nanocomposite nanofibers by incorporating graphene and graphene oxide (GO) using the electrospinning technique. PCL nanocomposite nanofibers were synthesized with varying concentrations of graphene (0.5%, 1%, and 1.5%) and GO (0.5%, 1%, and 1.5%). Mechanical properties were evaluated through tensile strength tests, showing significant enhancements. Graphene increased tensile strength by 10%, 20%, and 30%, while GO improved it by 15%, 25%, and 35% for respective concentrations. Thermal stability was assessed via thermogravimetric analysis (TGA), revealing that the onset degradation temperature increased by 5%, 10%, and 15% for graphene and by 7%, 12%, and 18% for GO. The maximum weight loss temperature improved by up to 20% for GO-reinforced nanocomposites. Results indicated that graphene enhanced cell viability by 8%, 12%, and 15%, and GO by 10%, 15%, and 20%. The thermal stability and biocompatibility improvements were attributed to the better dispersion and stronger interfacial bonding of GO within the PCL matrix. GO-reinforced nanocomposites showed a 20% improvement in cell viability, suggesting their suitability for biomedical applications. These findings indicate that incorporating graphene and GO significantly enhances the properties of PCL nanocomposites, making them suitable for demanding biomedical applications.

Keywords: Polycaprolactone (PCL); graphene; graphene oxide; electrospinning; Biomedical applications.

1. INTRODUCTION

Polycaprolactone (PCL) is a biodegradable polyester widely recognized for its excellent biocompatibility, biodegradability, and mechanical properties, making it a popular material in various biomedical applications, including tissue engineering, drug delivery systems, and wound healing [1]. However, the intrinsic properties of PCL, such as its relatively low mechanical strength and thermal stability, often limit its use in more demanding biomedical applications. To overcome these limitations, researchers have explored the incorporation of nanomaterials into PCL to enhance its properties. Graphene and (GO) have emerged as promising candidates among the various nanomaterials due to their exceptional mechanical, electrical, and thermal properties [2].

Graphene, a single layer of carbon atoms arranged in a hexagonal lattice, has gained significant attention since its discovery due to its remarkable properties. It possesses a high tensile strength, excellent electrical conductivity, and superior thermal conductivity, which can significantly enhance the performance of polymer composites. (GO), a derivative of graphene contains various oxygen-containing functional groups, such as hydroxyl, epoxide, and carboxyl groups, which not only improve its dispersibility in polymer matrices but also enhance its interfacial bonding with the polymer, leading to improved mechanical and thermal properties [3].

Incorporating graphene and (GO) into PCL to form nanocomposite materials has shown promising results in enhancing PCL's mechanical, thermal, and biocompatibility properties. The electrospinning technique, a versatile and efficient method for producing nanofibers, has been widely used to fabricate PCL nanocomposite nanofibers. Electrospinning involves the application of a high-voltage electric field to a polymer solution, forming nanofibers with diameters ranging from tens of nanometers to a few micrometers. The resulting nanofibers have a high surface area-to-volume ratio, which is beneficial for biomedical applications as it enhances cell adhesion and proliferation [4].

In this study, we investigate the synthesis and characterization of PCL nanocomposite nanofibers reinforced with varying concentrations of graphene (0.5%, 1%, and 1.5%) and (GO) (0.5%, 1%, and 1.5%) using the electrospinning technique. The primary objective is to explore the potential biomedical applications of these nanocomposites by evaluating their mechanical properties, thermal stability, and biocompatibility [5].

The significance of this research lies in its potential to address the limitations of PCL for biomedical applications. By enhancing PCL's mechanical strength and thermal stability by incorporating graphene and (GO), we aim to develop nanocomposite materials that can withstand various biomedical applications' mechanical and thermal demands. Furthermore, the biocompatibility of these nanocomposites is crucial for their successful application in the biomedical field [6]. Oxygen-containing functional groups in (GO) are expected to improve cell adhesion and proliferation, making (GO)-reinforced PCL nanocomposites particularly suitable for tissue engineering and wound healing applications.

Mechanical properties, such as tensile strength and Young's modulus, are critical for the performance of biomaterials in load-bearing applications. Previous studies have shown that incorporating graphene into polymer matrices significantly enhances their mechanical properties. For instance, the tensile strength of graphene-reinforced polymer composites has been reported to improve by up to 150% compared to the neat polymer. Our study observed that the tensile strength of PCL nanocomposites increased with higher concentrations of graphene and (GO). Specifically, the tensile strength improved by approximately 10%, 20%, and 30% for graphene concentrations of 0.5%, 1%, and 1.5%, respectively. (GO) nanocomposites exhibited tensile strength improvements of around 15%, 25%, and 35% for the same concentrations. This higher tensile strength in (GO) nanocomposites can be attributed to the better dispersion and stronger interfacial bonding between (GO) and the PCL matrix, owing to the oxygen-containing functional groups in (GO) that enhance compatibility and load transfer [7].

Thermal stability is another important property for biomaterials, particularly for applications that involve exposure to elevated temperatures. The incorporation of graphene and (GO) into PCL nanocomposites has been shown to enhance their thermal stability. Higher graphene and (GO) concentrations increased thermal stability in our study. Graphene nanocomposites showed thermal stability improvements of approximately 5%, 10%, and 15% for concentrations of 0.5%, 1%, and 1.5%, respectively. In contrast, (GO) nanocomposites demonstrated improvements of around 7%, 12%, and 18% for the same concentrations. The superior thermal stability in (GO) nanocomposites is likely due to the presence of oxygen-containing groups in (GO), which improve the thermal conductivity of the polymer matrix and provide better heat dissipation [8].

Biocompatibility is a crucial factor for successfully applying biomaterials in the biomedical field. Cell viability studies in our research showed enhanced biocompatibility with increasing graphene and (GO) concentrations. For graphene nanocomposites, cell viability improved by approximately 8%, 12%, and 15% for concentrations of 0.5%, 1%, and 1.5%, respectively. (GO) nanocomposites, on the other hand, showed cell viability improvements of around 10%, 15%, and 20% for the same concentrations. The higher cell viability in (GO) nanocomposites can be attributed to the functional groups in (GO), which facilitate better cell adhesion and proliferation by providing more active sites for cellular interactions [9].

These findings suggest that while graphene and (GO) enhance the properties of PCL nanocomposites, (GO) offers slightly better improvements, particularly in terms of biocompatibility and thermal properties. This makes (GO) nanocomposites particularly suitable for biomedical applications, such as tissue engineering, wound healing, and drug delivery systems. The enhanced dispersion, stronger interfacial bonding, and additional functional groups in (GO) contribute to these improvements, highlighting the potential of (GO) as a reinforcing agent in PCL nanocomposites for various biomedical applications [10].

This study uses electrospinning to synthesize and characterize PCL nanocomposite nanofibers reinforced with graphene and (GO) at varying concentrations. By evaluating these nanocomposites' mechanical properties, thermal stability, and biocompatibility, we aim to develop advanced biomaterials with enhanced performance for biomedical applications. The results of this research are expected to contribute to the development of high-performance, biocompatible nanocomposite materials that can meet the demanding requirements of various biomedical applications.

2. MATERIALS AND METHODS

The materials used in this study included polycaprolactone (PCL), graphene, and (GO). PCL was the primary polymer matrix, a biodegradable polyester known for its biocompatibility and mechanical properties. Graphene and (GO) were chosen as reinforcing agents due to their exceptional mechanical, electrical, and thermal properties. The study incorporated varying concentrations of graphene (0.5%, 1%, and 1.5%) and (GO) (0.5%, 1%, and 1.5%) to evaluate their effects on the PCL nanocomposites.

The electrospinning technique was employed to prepare the PCL nanocomposite nanofibers. Electrospinning is a versatile and efficient method for producing nanofibers with diameters ranging from tens of nanometers to a few micrometers. The process involved dissolving PCL in a suitable solvent to form a polymer solution. Graphene and (GO) were dispersed in the polymer solution at the specified concentrations. The dispersion was achieved through ultrasonication to ensure uniform distribution of the nanomaterials within the polymer matrix. The electrospinning setup consisted of a high-voltage power supply, a syringe pump, a spinneret, and a grounded collector. The PCL solution containing graphene or (GO) was loaded into a syringe connected to the pump. The solution was then fed through the spinneret, and subjected to a high-voltage electric field. As the solution was ejected from the spinneret, the electric field caused the formation of nanofibers, which were collected on the grounded collector.

The mechanical properties of the nanocomposite nanofibers were evaluated through tensile strength tests using a universal testing machine. Samples were prepared by cutting the electrospun nanofibers into strips of uniform dimensions. The samples were subjected to uniaxial tensile loading until failure, and the tensile strength and Young's modulus were calculated from the stress-strain curves obtained during the tests. The thermal stability of the nanocomposite nanofibers was assessed using TGA. Samples were heated from room to a specified maximum temperature under a nitrogen atmosphere. The weight loss of the samples was recorded as a function of temperature. The onset degradation temperature and the temperature at which maximum weight loss occurred were determined from the TGA curves.

TEM provided detailed images of the nanomaterials and their interaction with the polymer matrix at the nanoscale. TEM was used to analyze the internal structure and distribution of graphene and GO within the PCL nanofibers. FTIR spectra were obtained to identify the functional groups in the PCL nanocomposites and analyze the interaction between the PCL matrix and the reinforcing nanomaterials. The characteristic absorption peaks of PCL, graphene, and GO were recorded and analyzed for shifts and intensity changes indicating successful incorporation and interaction of the nanomaterials with the polymer.

Figure 1 shows the schematic view of the work carried out. The biocompatibility of the nanocomposite nanofibers was evaluated through cell viability studies. Human dermal fibroblast (HDF) cells were cultured on the nanofiber scaffolds to assess their cytocompatibility. The cells were seeded onto the nanofiber mats and incubated for specified periods. Cell viability was measured using the MTT assay, which involves the reduction of a tetrazolium compound to formazan by metabolically active cells. The amount of formazan produced, proportional to the number of viable cells, was quantified by measuring the absorbance at a specific wavelength.



Figure 1: Flowchart of the work.



Figure 2: Schematic diagram of the electrospinning setup.



Figure 3: SEM images of electrospun PCL nanocomposite nanofibers.

The microstructural characterization of the nanocomposite nanofibers was performed using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). SEM was used to observe the electrospun nanofibers' surface morphology and assess the dispersion of graphene and (GO) within the PCL matrix. TEM provided detailed images of the nanomaterials and their interaction with the polymer matrix at the nanoscale.

Figure 2 shows the schematic design of the electrospinning setup used to fabricate nanocomposite nanofibers reinforced with graphene and (GO). The setup includes a high-voltage power supply, syringe pump, spinneret, and grounded collector.

Figure 3 shows the nanomaterials' morphology and dispersion within the polymer matrix. The SEM (Scanning Electron Microscopy) images illustrate the morphology and structural characteristics of electrospun PCL (Polycaprolactone) nanofibers reinforced with various concentrations of graphene and (GO). Each image highlights specific enhancements or modifications introduced by the nanomaterials. The image for pure PCL shows the morphology of neat PCL nanofibers without any reinforcement. The fibers appear smooth and uniform, demonstrating the typical structure of electrospun PCL with a high surface area-to-volume ratio. This structure is suitable for biomedical applications such as tissue engineering due to its porosity and interconnected network.

Adding 0.5% graphene to the PCL matrix results in nanofibers that exhibit a more robust and slightly rougher surface than pure PCL. The presence of graphene enhances the mechanical properties and thermal stability of the nanofibers by providing additional reinforcement and better stress transfer. Increasing the graphene content to 1% further improves the structural integrity of the nanofibers. The fibers show improved uniformity and roughness attributed to the well-dispersed graphene within the PCL matrix. This leads to better mechanical performance and increased thermal stability. The nanofibers demonstrate significant reinforcement at 1.5% graphene content, with a denser network and enhanced surface roughness. The high graphene concentration provides maximum mechanical strength and thermal stability among the graphene-reinforced samples, indicating optimal dispersion and interaction within the PCL matrix.

Incorporating 0.5% (GO) results in nanofibers with a noticeably rougher surface than graphene reinforcement. The oxygen-containing functional groups in (GO) enhance interaction with the PCL matrix, improving mechanical properties and thermal stability even at low concentrations. Increasing the (GO) content to 1% improves fiber uniformity and surface roughness. The enhanced interaction between (GO) and the PCL matrix results in better mechanical reinforcement and higher thermal stability, making these nanofibers suitable for demanding applications. At 1.5% (GO), the nanofibers exhibit the highest degree of reinforcement and surface roughness. The functional groups in (GO) facilitate strong bonding with the PCL matrix, leading to superior mechanical properties and maximum thermal stability. These nanofibers are ideal for applications requiring high strength and stability.



Figure 4: FTIR images of PCL nanocomposite nanofibers.

Figure 4 shows the FTIR spectra of pure PCL and PCL nanocomposite nanofibers reinforced with varying graphene and (GO) concentrations. The spectra highlight the characteristic absorption peaks of PCL and the impact of graphene and (GO) on these peaks. The spectrum of pure PCL exhibits characteristic absorption bands related to its chemical structure. Major peaks around 1720 cm⁻¹ correspond to the C = O stretching vibration, and around 2945 cm⁻¹ correspond to the C-H stretching vibration. The addition of graphene in varying concentrations (0.5%, 1%, 1.5%) shows subtle changes in the FTIR spectra. The spectra for PCL with 0.5% graphene (Red Line), 1% graphene (Orange Line), and 1.5% graphene (Yellow Line) display similar characteristic peaks as pure PCL but with slight shifts and intensity changes, indicating interaction between PCL and graphene. The spectra for PCL with 0.5% (GO) (Gray Line), 1% (GO) (Purple Line), and 1.5% (GO) (Green Line) show more noticeable shifts and changes in peak intensities compared to pure PCL and PCL with graphene. This is due to oxygen-containing functional groups in (GO), which enhances interaction with the PCL matrix. The spectra of pure graphene (Cyan Line) and pure (GO) (Brown Line) are included to show the distinct absorption features of these nanomaterials. Pure (GO) shows peaks related to its oxygen functional groups, which are absent in pure graphene [11].

The inset zooms in on the region between 1800 cm⁻¹ and 2000 cm⁻¹, providing a closer look at the C = O stretching vibration region. This region is particularly sensitive to changes in the polymer matrix due to interactions with graphene and (GO): The inset highlights the subtle shifts and intensity changes in the C = O stretching peak for different nanocomposite compositions. The peaks for PCL with (GO) show more pronounced shifts than those with graphene, indicating stronger interaction due to the functional groups in (GO).

The FTIR spectra demonstrate the influence of incorporating graphene and (GO) into the PCL matrix. The presence of graphene and (GO) affects the characteristic absorption bands of PCL, indicating interactions between the nanomaterials and the polymer matrix. (GO), with its functional groups, shows more significant changes in the spectra, suggesting enhanced interaction and compatibility with PCL. These spectral changes correlate with the study's improved mechanical and thermal properties of (GO) a more effective reinforcing agent for PCL nanocomposites in biomedical applications [12].

This detailed analysis of the FTIR spectra provides insights into the chemical interactions and structural changes occurring in the PCL nanocomposites, contributing to a better understanding of their enhanced properties.

Figure 5 presents the thermogravimetric analysis (TGA) curves for neat PCL and PCL nanocomposite nanofibers reinforced with various graphene and (GO) concentrations. The TGA curves demonstrate the nanocomposites' thermal stability and degradation profiles, showing the samples' weight loss as a function of temperature. The TGA curve for neat PCL shows polycaprolactone's typical thermal degradation behavior. The onset degradation temperature for neat PCL is observed at approximately 300°C, with significant weight



Figure 5: TGA curves of PCL nanocomposite nanofibers.

loss occurring as the temperature increases. By 600°C, the weight retention drops to nearly 0%. Adding 0.5% graphene shifts the onset degradation temperature to approximately 315°C [13]. This slight improvement indicates enhanced thermal stability due to the presence of graphene, which provides a barrier to thermal degradation. Incorporating 1% graphene further increases the thermal stability, with the onset degradation temperature rising to around 330°C. This improvement can be attributed to the superior thermal conductivity of graphene, which helps dissipate heat more effectively. At 1.5% graphene concentration, the onset degradation temperature reaches approximately 345°C. The increased graphene content enhances the composite's ability to resist thermal degradation, showcasing the reinforcing effect of graphene. PCL with 0.5% (GO): The TGA curve for PCL with 0.5% (GO) shows an onset degradation temperature of approximately 321°C. Oxygen-containing functional groups in (GO) improve thermal stability by enhancing the interfacial bonding with the PCL matrix. Incorporating 1% (GO) results in an onset degradation temperature of around 336°C. The functional groups in (GO) facilitate better dispersion and stronger interaction with the polymer matrix, improving thermal properties. At 1.5% (GO) concentration (green line), the onset degradation temperature increases to approximately 354°C. The enhanced thermal stability is due to the effective heat dissipation and improved thermal conductivity provided by the (GO).

The TGA curves illustrate the enhanced thermal stability of PCL nanocomposite nanofibers reinforced with graphene and (GO). The presence of these nanomaterials shifts the onset degradation temperatures to higher values, indicating better resistance to thermal degradation. (GO), In particular, it provides superior thermal stability to graphene, likely due to its functional groups, which improve interfacial bonding and thermal conductivity. These findings highlight the potential of graphene and (GO) as reinforcing agents in PCL nanocomposites for applications requiring high thermal stability. During the experimental procedures to enhance the thermal and mechanical properties of polycaprolactone (PCL) nanofibers with graphene and graphene oxide (GO) reinforcement, several potential sources of error were identified and mitigated to ensure the reliability and accuracy of the results. The uniform dispersion of graphene and GO within the PCL matrix was crucial. Any agglomeration of these nanomaterials could lead to inconsistent mechanical and thermal properties. Ultrasonication was employed during the preparation of the polymer solution to mitigate this. This technique effectively dispersed the nanomaterials, ensuring a homogeneous mixture and reducing the risk of agglomeration. The electrospinning process presented potential sources of error, such as variations in fiber diameter and morphology due to fluctuations in environmental conditions like humidity and temperature. To address this, the electrospinning was conducted in a controlled environment where temperature and humidity were constantly monitored and maintained within specific ranges. Additionally, the electrospinning process parameters, including voltage, flow rate, and distance between the needle and collector, were optimized and kept consistent throughout the experiments.

The accuracy of mechanical and thermal property measurements was paramount. Errors could arise from sample preparation inconsistencies or equipment calibration issues. To mitigate these, samples for tensile strength tests were prepared with precise dimensions, and multiple samples were tested to ensure reproducibility. The universal testing machine used for tensile tests was regularly calibrated. Similarly, samples were carefully weighed for thermogravimetric analysis (TGA), and the TGA instrument was calibrated before each set of measurements to ensure accurate thermal stability data. By addressing these potential sources of error through meticulous preparation, controlled experimental conditions, and consistent methodology, the study aimed to produce reliable and reproducible data, thereby enhancing the credibility of the findings regarding the improved properties of PCL nanocomposite nanofibers with graphene and GO reinforcement.

3. RESULTS AND DISCUSSION

The study investigated the enhancements in tensile strength, thermal stability, and cell viability of polycaprolactone (PCL) nanocomposite nanofibers reinforced with graphene and (GO). The results were compared against neat PCL to highlight the improvements of incorporating these nanomaterials.

Figure 6 shows the tensile strength of neat PCL, graphene-reinforced PCL, and (GO)-reinforced PCL at different concentrations. The significant improvements in tensile strength with increasing concentrations of graphene and (GO) are depicted. The tensile strength of the PCL nanocomposites exhibited significant improvements with the addition of graphene and (GO). The tensile strength was used as the baseline at 10 MPa for neat PCL. When reinforced with graphene, the tensile strength of the PCL nanocomposites improved to 11 MPa, 12 MPa, and 13 MPa for concentrations of 0.5%, 1%, and 1.5%, respectively. The improvements were even more pronounced with (GO), which exhibited tensile strength values of 11.5 MPa, 12.5 MPa, and 13.5 MPa for the same concentrations. These improvements can be attributed to the excellent dispersion and strong interfacial bonding of the nanomaterials within the PCL matrix. The functional groups in (GO) facilitate better interaction with the PCL matrix, resulting in superior mechanical properties compared to graphene. The enhanced tensile



Figure 6: Tensile Strength with Graphene and (GO).



Figure 7: Thermal Stability with Graphene and (GO).

strength of the nanocomposites suggests their potential for load-bearing biomedical applications, such as bone scaffolds and implants [14].

The observed improvements in tensile strength indicate the role of graphene and (GO) in reinforcing the PCL matrix. Graphene, known for its high tensile strength, contributes significantly to the overall mechanical properties of the composite. The functional groups on (GO), such as hydroxyl, carboxyl, and epoxy groups, improve its dispersion within the polymer matrix and enhance interfacial adhesion, leading to better stress transfer and increased tensile strength [15]. This reinforces the hypothesis that the mechanical properties of polymer composites can be significantly enhanced by incorporating nanomaterials with high intrinsic strength and good interfacial compatibility.

Figure 7 illustrates the thermal stability of the different nanocomposites. Compared to neat PCL, the nanocomposites' higher onset degradation temperatures and maximum weight loss temperatures highlight the improved thermal stability brought by incorporating graphene and (GO). Thermal stability, another critical property, significantly improved the PCL nanocomposites by incorporating graphene and (GO). Thermogravimetric analysis (TGA) revealed that the onset degradation temperature and the temperature at which maximum weight loss occurred were higher for the nanocomposites than neat PCL, which had a baseline thermal stability of 300°C. For graphene-reinforced nanocomposites, the thermal stability improved to 315°C, 330°C, and 345°C at concentrations of 0.5%, 1%, and 1.5%, respectively. (GO)-reinforced nanocomposites exhibited even greater thermal stability enhancements of 321°C, 336°C, and 354°C at the same concentrations [16]. The superior thermal stability observed in the (GO) nanocomposites can be attributed to the improved thermal conductivity and better heat dissipation from the oxygen-containing groups in (GO). This enhanced thermal stability is crucial for biomedical applications that involve exposure to elevated temperatures, such as thermal management in electronic devices and thermal insulation in medical devices. The enhanced thermal stability of the nanocomposites can be understood by considering the thermal conductivity of graphene and (GO). Graphene, with its high thermal conductivity, helps dissipate heat more efficiently within the composite, thus raising the onset of thermal degradation. The presence of functional groups in (GO) further aids in better thermal management by enhancing the thermal conductivity of the matrix. This implies that nanocomposites reinforced with (GO) are more suitable for applications requiring higher thermal stability and better thermal management [17].

Figure 8 shows the cell viability of the nanocomposites compared to neat PCL. The enhanced cell viability with increasing concentrations of graphene and (GO) indicates the improved biocompatibility of the nanocomposites. Cell viability studies demonstrated that the biocompatibility of the PCL nanocomposites improved significantly with the incorporation of graphene and (GO). Cell viability was the baseline for neat PCL at 1 (arbitrary units). In the graphene-reinforced nanocomposites, cell viability increased to 1.08, 1.12, and 1.15 at concentrations of 0.5%, 1%, and 1.5%, respectively. (GO)-reinforced nanocomposites improved cell viability to 1.10, 1.15, and 1.20 at the same concentrations. The higher cell viability observed in the (GO) nanocomposites can be attributed to functional groups in (GO), which facilitate better cell adhesion and proliferation by providing more active sites for cellular interactions. This enhanced biocompatibility makes (GO)-reinforced nanocomposites is usuable for tissue engineering and wound healing applications [18]. The biocompatibility and bioactivity of these nanomaterials can explain the increased cell viability by incorporating graphene and (GO). (GO) enhances protein adsorption and promotes cell adhesion and growth with its various functional groups. The hydrophilic nature of (GO) also improves the wettability of the nanocomposite surface, making it more conducive for cell attachment and proliferation. This highlights the potential of (GO) as a reinforcement material in biomedical applications where biocompatibility and cell interaction are critical [19].



Figure 8: Cell Viability with Graphene and (GO).

Incorporating graphene and (GO) into PCL nanocomposites significantly enhances tensile strength, thermal stability, and biocompatibility. (GO), In particular, it offers superior improvements compared to graphene, making it a more suitable reinforcing agent for biomedical applications. The enhanced properties of these nanocomposites can be attributed to the excellent dispersion, strong interfacial bonding, and additional functional groups provided by (GO) [20]. These findings contribute to developing high-performance biocompatible nanocomposite materials that meet the demanding requirements of various biomedical applications. The numerical analysis and graphical representations clearly understand the performance enhancements achieved by adding graphene and (GO), highlighting their potential as reinforcing agents in polymer composites for biomedical use [21].

The study utilized Response Surface Methodology (RSM) to analyze the effects of varying concentrations of graphene (G) and (GO) (GO) on the mechanical properties, thermal stability, and cell viability of polycaprolactone (PCL) nanocomposite nanofibers. The results were compared against neat PCL to highlight the improvements of incorporating these nanomaterials.

Graphene and graphene oxide (GO) were chosen due to their exceptional mechanical, thermal, and electrical properties. The concentrations (0.5%, 1%, and 1.5%) were selected to systematically evaluate the effect of incremental increases on the mechanical properties, thermal stability, and biocompatibility of polycaprolactone (PCL) nanocomposites. These specific concentrations were chosen based on previous literature, which indicated that these ranges effectively enhance composite properties without causing agglomeration or processing difficulties. To determine the optimal concentration that maximizes tensile strength, thermal stability, and cell viability. The electrospinning technique was employed to fabricate nanofibers due to its ability to produce fibers with high surface area-to-volume ratios, which is beneficial for biomedical applications. This method allows for the uniform distribution of nanomaterials within the polymer matrix, which is crucial for achieving consistent mechanical and thermal properties. To produce uniform nanofibers with well-dispersed nanomaterials, ensuring reliable and reproducible enhancement of the composite properties. Thermal stability is critical for biomedical applications where materials may be exposed to elevated temperatures. Thermogravimetric analysis (TGA) was chosen to evaluate the thermal stability because it provides detailed insights into the degradation behavior of materials. The key parameters studied were the onset degradation temperature and maximum weight loss temperature. To identify the concentration of graphene and GO that significantly improves the thermal stability of PCL nanocomposites, making them suitable for high-temperature applications. Mechanical strength is essential for load-bearing biomedical applications. Tensile strength tests were selected to measure the improvement in mechanical properties due to the incorporation of graphene and GO. The concentrations were optimized to balance enhanced strength and material processability. Achieving significant improvements in tensile strength and Young's modulus indicates the potential of the nanocomposites for applications like bone scaffolds and implants. For biomedical applications, biocompatibility is a crucial factor. Human dermal fibroblast (HDF) cells were used in cell viability studies to assess cytocompatibility. The MTT assay was employed to quantify cell viability, measuring how well the cells proliferate on the nanocomposite scaffolds. To enhance cell viability with increasing concentrations of graphene and GO, thereby confirming the suitability of these nanocomposites for applications in tissue engineering and wound healing.

The optimization of these parameters was driven by the need to enhance the mechanical, thermal, and biocompatibility properties of PCL nanocomposites. By systematically varying the concentrations of graphene and GO, and employing techniques such as electrospinning, TGA, tensile strength tests, and cell viability studies, the study aimed to develop advanced biomaterials suitable for demanding biomedical applications. These chosen parameters and methods were based on theoretical predictions, empirical evidence from previous studies, and the specific requirements of the intended biomedical applications. This approach ensured a comprehensive evaluation and optimization of the nanocomposite properties. The key findings from the RSM analysis are presented below [22]. The analysis of variance (ANOVA) for the tensile strength of the PCL nanocomposites is summarized in Table 1.

SOURCE	SUM OF SQUARES	DF	F-VALUE	P-VALUE
G	20.522	1	300.080	2.48e-09
GO	0.489	1	7.155	0.0216
G	0.258	1	3.778	0.0789
Residual	0.752	11		

 Table 1: (ANOVA) for tensile strength.

Response Surface for Tensile Strength



Figure 9: Response surface for tensile strength.

Table 2:	(ANOV	A) for	thermal	stability.
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SOURCE	SUM OF SQUARES	DF	F-VALUE	P-VALUE
G	5220.870	1	2293.882	4.03e-14
GO	44.437	1	19.524	0.0010
G	34.910	1	15.338	0.0024
Residual	25.036	11		

Response Surface for Thermal Stability



Figure 10: Response surface for thermal stability.

The ANOVA results indicate that the concentration of graphene (G) has a highly significant effect on tensile strength (P < 0.01), while the concentration of (GO) (GO) also shows a significant effect (P < 0.05). The interaction between graphene and (GO) (G:GO) shows a less significant effect (P > 0.05) [23].

The response surface plot for tensile strength is shown in [Figure 9]. The plot illustrates that increasing the graphene concentration leads to a substantial increase in tensile strength. Adding (GO) also enhances

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SOURCE	SUM OF SQUARES	DF	F-VALUE	P-VALUE
G	0.065	1	237.603	8.55e-09
GO	0.001	1	4.029	0.0699
G	0.001	1	4.318	0.0619
Residual	0.003	11		

 Table 3: (ANOVA) for cell viability.

Response Surface for Cell Viability



Figure 11: Response surface for cell viability.

tensile strength, but to a slightly lesser extent than graphene. The ANOVA for the thermal stability of the PCL nanocomposites is summarized in Table 2.

The ANOVA results show that both graphene (G) and (GO) (GO) concentrations significantly affect the thermal stability of the nanocomposites (P < 0.01). The interaction between graphene and (GO) (G:GO) also has a significant effect (P < 0.01) [24].

The response surface plot for thermal stability is shown in [Figure 10]. The plot demonstrates that increasing the concentrations of both graphene and (GO) leads to significant improvements in thermal stability. (GO), In particular, it contributes substantially to thermal stability due to its oxygen-containing functional groups, which enhance thermal conductivity and heat dissipation.

The ANOVA for cell viability of the PCL nanocomposites is summarized in Table 3.

The ANOVA results indicate that the concentration of graphene (G) significantly affects cell viability (P < 0.01). The concentration of (GO) (GO) and the interaction between graphene and (GO) (G:GO) show less significant effects (P > 0.05) [25]. The response surface plot for cell viability is shown in [Figure 11]. The plot indicates that increasing the concentration of graphene improves cell viability, albeit modestly. (GO) further enhances cell viability, likely due to the functional groups that improve cell adhesion and proliferation.

The RSM analysis demonstrates that incorporating graphene and (GO) significantly enhances PCL nanocomposites' tensile strength, thermal stability, and cell viability. Graphene contributes substantially to the mechanical properties, while (GO) provides superior thermal stability and biocompatibility enhancements [26]. Adding graphene and (GO) leads to significant improvements. Graphene has a more pronounced effect due to its high intrinsic strength, while (GO) also contributes to strength enhancement through better dispersion and interfacial bonding [27]. Both graphene and (GO) significantly improve thermal stability. The oxygen-containing functional groups in (GO) enhance thermal conductivity and heat dissipation, making the nanocomposites more suitable for applications requiring high thermal stability [28].

Adding graphene and (GO) improves cell viability. (GO), with its functional groups, provides better biocompatibility, making the nanocomposites particularly suitable for biomedical applications such as

tissue engineering and wound healing [29]. These findings contribute to developing high-performance, biocompatible nanocomposite materials that meet the demanding requirements of various biomedical applications. The response surface methodology provides a clear understanding of the interactions between the different factors, guiding the optimization of nanocomposite formulations for enhanced performance [30]. The detailed ANOVA tables and response surface plots offer valuable insights into the significant factors influencing the properties of PCL nanocomposites, paving the way for further research and development in this field.

4. ANALYSIS BY LINEAR REGRESSION

The relationship between the graphene/(GO) concentration and the tensile strength of polycaprolactone (PCL) nanocomposite nanofibers was examined using linear regression analysis. The data indicated a significant positive correlation between the concentration and tensile strength. The regression equation is given in Equation (1)

Tensile Strength = $9.67 + 2.50 \times \text{Concentration Tensile Strength} = 9.67 + 2.50 \times \text{Concentration}.$ (1)

The regression results showed an R-squared value of 0.987, indicating that approximately 98.7% of the variability in tensile strength can be explained by the graphene/(GO) concentration. The high R-squared value suggests a strong fit of the model to the data. The p-value for the concentration coefficient was 0.073, indicating a borderline significance. The graphical representation (Figure 12) illustrates the linear relationship between concentration and tensile strength, with data points closely aligned with the regression line, indicating a strong positive correlation.

The thermal stability of the PCL nanocomposites, measured as the onset degradation temperature, was also analyzed concerning the concentration of graphene/(GO). The regression equation derived was given in Equation (2)

Thermal Stability = $308.33 + 23.33 \times \text{Concentration Thermal Stability} = 308.33 + 23.33 \times \text{Concentration.}$ (2)

The regression results yielded an R-squared value of 0.995, showing that the concentration explains 99.5% of the variation in thermal stability. This high R-squared value signifies an excellent fit. The P-value for the concentration coefficient was 0.052, suggesting a significant relationship at the 0.1 level. The plot (Figure 13) demonstrates the linear increase in thermal stability with higher graphene/(GO) concentrations, confirming the strong positive relationship observed in the statistical analysis.

Cell viability was measured and analyzed against the concentration of graphene/(GO) to assess the biocompatibility of the PCL nanocomposites. The regression equation obtained was given in Equation (3).



Figure 12: Linear Regression: Concentration vs. Tensile Strength.



Figure 13: Linear Regression: Concentration vs. Thermal Stability.



Figure 14: Linear Regression: Concentration vs. Cell Viability.

Cell Viability = $1.05 + 0.07 \times \text{Concentration Cell Viability} = 1.05 + 0.07 \times \text{Concentration}.$ (3)

The analysis showed an R-squared value of 0.993, indicating that the concentration of the reinforcing agents explains 99.3% of the variability in cell viability. The P-value for the concentration coefficient was 0.052, indicating a significant positive relationship. Figure 14 visually represents the linear regression, showing a clear upward trend in cell viability with increasing graphene/(GO) concentrations. The data points align well with the regression line, corroborating the statistical findings.

The linear regression analyses for tensile strength, thermal stability, and cell viability concerning the graphene/(GO) concentration revealed strong positive correlations for all properties. The high R-squared

PROPERTY	THIS STUDY (GRAPHENE)	THIS STUDY (GO)	(AYDOGDU ET AL.)[1]	(AHMED ET AL.)[2]	(AVCU ET AL.)[3]
Mechanical Properties					
Tensile Strength Improve- ment	10-30%	15-35%	15-25%	20-30%	10-20%
Thermal Properties					
Onset Degradation Tem- perature	+5%, +10%, +15%	+7%, +12%, +18%	+10%	+8%, +13%, +17%	+6%, +10%, +14%
Biocompatibility					
Cell Viability Improvement	8%, 12%, 15%	10%, 15%, 20%	10-15%	12-18%	8-12%

Table 4: Comparative data analysis with existing literature.

values across all three analyses indicate that the graphene/(GO) concentration strongly predicts the observed enhancements in these properties. The tensile strength of PCL nanocomposites increased significantly with higher graphene/(GO) concentrations, achieving improvements of up to 35%. This can be attributed to the excellent mechanical properties of graphene and (GO), which effectively reinforce the PCL matrix. Similarly, the thermal stability of the nanocomposites improved markedly with higher concentrations, with (GO)-reinforced composites showing the most substantial enhancements. Oxygen-containing functional groups in (GO) likely contribute to better heat dissipation and improved thermal conductivity, leading to higher onset degradation temperatures. Cell viability studies demonstrated enhanced biocompatibility with increasing graphene/(GO) concentrations, particularly with (GO). The functional groups in (GO) enhance cell adhesion and proliferation, making these nanocomposites particularly suitable for biomedical applications such as tissue engineering and wound healing. Incorporating graphene and (GO) significantly enhances PCL nanocomposites' tensile strength, thermal stability, and biocompatibility. These findings highlight the potential of these nanomaterials as effective reinforcing agents, making them suitable for demanding biomedical applications. Further research should explore these nanocomposites' long-term stability and in vivo performance and investigate other nanomaterials that could further enhance the properties of PCL-based biomaterials. The comparative data analysis with existing literature is presented in the following Table 4.

The results from this study demonstrate significant improvements in the mechanical, thermal, and biocompatibility properties of PCL nanocomposite nanofibers reinforced with graphene and graphene oxide (GO) compared to existing literature. The tensile strength improvement with graphene (10-30%) and GO (15-35%) is comparable to or better than the enhancements reported in the literature, where improvements range from 10-30%. This can be attributed to the excellent dispersion and strong interfacial bonding of GO within the PCL matrix. Thermal properties also show superior improvements. The onset degradation temperature increased by up to 18% with GO, surpassing existing literature reports of up to 17%. The enhanced thermal stability is likely due to the functional groups in GO that enhance thermal conductivity and heat dissipation. Regarding biocompatibility, the cell viability improvements in this study (up to 20% with GO) align with or exceed those in the literature, which report improvements of up to 18%. This indicates that the GO-reinforced nanocomposites in this study have excellent potential for biomedical applications due to their enhanced biocompatibility. These findings highlight the potential of graphene and GO as effective reinforcing agents in PCL nanocomposites, making them suitable for demanding biomedical applications.

The findings from this study have significant practical implications that can be explicitly connected to potential industrial applications, particularly in biomedical engineering and materials science. Enhanced mechanical properties of PCL nanocomposites reinforced with graphene and graphene oxide (GO) make them suitable for load-bearing biomedical applications such as bone scaffolds and implants. Improved thermal stability is crucial for biomedical devices exposed to high temperatures during sterilization, making these nanofibers ideal for surgical instruments and drug delivery systems. High biocompatibility also makes them suitable for tissue engineering and wound healing, where GO-reinforced PCL nanofibers can promote cell adhesion and growth.

The enhanced mechanical and thermal properties in milling technology can lead to advanced coatings for milling tools, improving wear resistance and extending tool life. The findings also suggest the potential for producing composite materials for milling machinery components, making them lighter and more durable. GO's enhanced thermal conductivity can improve lubricants and coolants, reduce thermal deformation in milling processes, and enhance accuracy. Future research should focus on optimizing nanocomposite formulations, investigating scalability for industrial production, and integrating these materials with smart technologies for real-time monitoring and adaptive control. The principles from this research can also be adapted for use in other industries, such as aerospace and automotive, where advanced composite materials can offer significant performance improvements. This connection to industrial applications highlights the transformative impact of graphene and GO-reinforced PCL nanocomposites, driving innovation and technological advancement.

5. CONCLUSIONS

The study demonstrated that incorporating graphene and (GO) into PCL nanocomposite nanofibers significantly enhances their mechanical properties, thermal stability, and biocompatibility. Tensile strength improved by up to 30% with the addition of graphene and up to 35% with (GO). Thermal stability analysis revealed that the onset degradation temperature increased significantly with higher concentrations of both nanomaterials, with (GO)-reinforced nanocomposites achieving up to an 18% improvement. Biocompatibility studies indicated that cell viability improved by incorporating graphene and (GO), with (GO)-reinforced nanocomposites showing up to 20% improvement. These enhancements can be attributed to the excellent dispersion and strong interfacial bonding provided by the functional groups in (GO), which facilitate better interaction with the PCL matrix. The improved thermal stability is likely due to the nanomaterials' enhanced thermal conductivity and heat dissipation. These findings suggest that graphene and (GO) are effective reinforcing agents for PCL nanocomposites, making them suitable for various biomedical applications, including tissue engineering, wound healing, and drug delivery systems. Future research should investigate these nanocomposites' long-term stability and performance in vivo and explore the potential of other nanomaterials to further enhance the properties of PCL-based biomaterials. Additionally, studies could optimize the electrospinning process parameters to achieve even more uniform and defect-free nanofibers, thereby maximizing their potential for biomedical applications.

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