Indian Journal of Advances in Chemical Science

Article

Preparation of Poly(ε-caprolactone)/Polyethylene Glycol (PCL/PEG) Blends Nanofibers by Electrospinning for Biomedical Applications

B. Sowmya[1,2], Athira John[1], P. K. Panda[1,2]*

[1]Materials Science Division, CSIR-National Aerospace Laboratories, Old Airport Road, Kodihalli, Bengaluru, India, [2]Academy of Scientific and Innovative Research, Ghaziabad, Uttar Pradesh, India

ABSTRACT

Scaffolds are 3D template for regenerating the damaged tissues. Polymer scaffolds offer flexibility, mechanical strength, and specific surface interaction sites for cell adhesion. The increasing rate of accidents in recent years has led to a rise in the demand for artificial substrates like scaffolds. In this work, the effort has been made to prepare electrospun polymer nanofibers for biomedical applications. Nanofibers are of more interest in this field because the architecture of the fibers at the nanoscale mimics the extracellular matrix (ECM). Poly(ε-caprolactone) (PCL) is a semi-crystalline, hydrophobic polyester while polyethylene glycol (PEG) is a hydrophilic polyether. The PCL/PEG blend nanofibers of various ratios were successfully prepared by electrospinning. The pure and blend nanofibers were characterized by Fourier-transform infrared (FTIR) spectroscopy, differential scanning calorimetry (DSC), and scanning electron microscopy (SEM). It was observed from the FTIR and DSC studies that both PCL and PEG phases coexist. The SEM results displayed the changes in the morphology with the addition of PEG.

Key words: Scaffold, Electrospinning, Nanofibers, Polycaprolactone, Polyethylene glycol, hydrophobic, Hydrophilic.

1. INTRODUCTION

Nanofibers are of interest due to its unique properties such as lightweight, higher aspect ratio, larger surface area, and porous structure which directly contribute to numerous applications in diversified fields such as sensors, filtration, biomedical protective clothing, and energy devices. Above all, the nanofibers have a crucial cellular activity and distinct surface properties which mimic extracellular matrix (ECM) which, in turn, makes it a desirable candidate exclusively for biomedical application. The properties of nanofibers are further tailor-made to enhance their functionality by incorporating two or more polymers. The resultant nanofiber blends should possess better cell adhesion and cellular functions as compared with their monophasic fibers [1]. Electrospinning is one of the simple and straight-forward techniques used for forming nanofibers from polymer solutions.

Biopolymers are of natural or synthetic origin and can either be biodegradable or non-biodegradable. Biopolymers are sustainable and hence are of importance in many applications. Poly(ε-caprolactone) (PCL) is a synthetic biopolymer synthesized from ring-opening polymerization of ε-caprolactone, which is semi-crystalline, aliphatic polyester, biodegradable, and hydrophobic [2]. PCL scaffolds offer a suitable environment for cell growth [3]. Polyethylene glycol (PEG) is a crystalline thermoplastic polymer which is water soluble and is hydrophilic in nature. For tissue engineering applications, hydrophilicity and surface roughness of the scaffolds are a peculiar property for cell activity, and hence, the PEG is blended with PCL to impart good wettability to the substrate for proper cell contact. The properties such as biocompatibility, biodegradability, non-toxic nature, porous structure, and mechanical strength are pertinent for any material to be used in biomedical application. Cell attachment, proliferation, and surface morphology property of a scaffold are exclusive for tissue engineering applications [4].

In the present study, the effort has been made to prepare pure PCL and PCL/PEG-based blend nanofibers for biomedical applications. The structure, morphology, and biocompatibility of the nanofibers are studied.

2. EXPERIMENTAL

2.1. Materials

PCL and PEG in pellets form, acetone and ethanol, were procured from Sigma-Aldrich, India.

2.2. Sample preparation

2.2.1. Preparation electrospun nanofibers

The individual polymer solutions, PCL in acetone and PEG in ethanol were prepared at 12 wt% and 20 wt% using a magnetic stirrer at 500–750 rpm. Acetone was used to prepare blend solutions at different ratios, namely, PCL/PEG: 90/10, 80/20, and 70/30, respectively, and stirred for 4 h. The prepared solution was filled in a 10 ml syringe with a fine needle and was electrospun. The electrospinning conditions were as follows: Tip to collector distance was 8–10 cm, relative humidity was 60–75%, high voltage was 15–19 kV, and the flow rate varied from 0.5 to 1 ml/h.

***Corresponding author:**
E-mail: pkpanda@nal.res.in

ISSN NO: 2320-0898 (p); 2320-0928 (e)
DOI: 10.22607/IJACS.2020.801001

Received: 08th November 2019;
Revised: 04th December 2019;
Accepted: 22nd December 2019

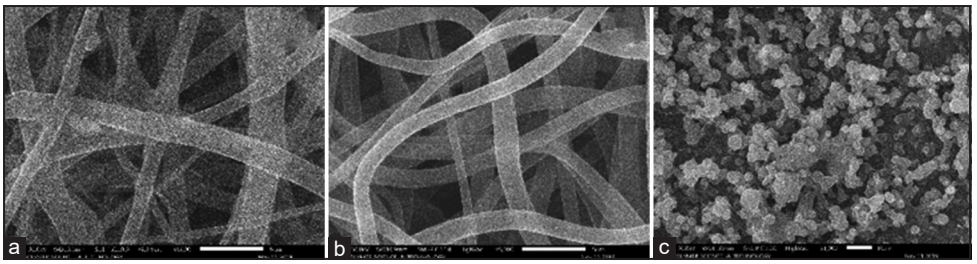

Figure 1: Scanning electron microscopy micrographs of electrospun PCL and its blend nanofiber (a) PCL, (b) 90/10 blend, (c) PEG.

2.3. Characterization

2.3.1. Fourier-transform infrared (FTIR) spectroscopy

The infrared spectroscopy analysis was performed using PerkinElmer FTIR spectrometer. FTIR measurements of pure and blend specimens were collected in the range of 400–4000 cm^{-1} with a resolution of 4 cm^{-1}. The functional groups of the respective samples were identified using their characteristic peak values.

2.3.2. Scanning electron microscope (SEM)

The surface morphology and diameter of the electrospun nanofibers were examined on JEOL JSM-IT300 SEM at 30 kV.

2.3.3. Differential scanning calorimetry (DSC)

The DSC was carried out using DSC Q200. DSC analysis was carried out in the range of −80°C–150°C at a heating rate of 10°C/min in a nitrogen atmosphere and the T_m and ΔH_m were recorded.

3. RESULTS AND DISCUSSION

3.1. DSC

The DSC thermogram of homopolymers PCL and PEG has sharp endothermic peak at 59°C and 67°C, respectively. Similarly, the DSC thermograms for PCL/PEG blend samples show the endotherm in the range of 57–63°C. For blend samples, a double melting endotherm corresponding to PCL and PEG was found on the DSC heating thermograms for PEG greater than 20 wt%. From the DSC results, it is noticeable that with the addition of PEG to PCL, the onset of melting increases with the increase in PEG content. The higher enthalpy of fusion was observed for 80/20 sample, but for pure PCL and pure PEG, it was 63 J/g and 203 J/g. The values are analogous to the literature [5,6].

It was noticeable that after the addition of PEG, extra peaks occurred in the thermogram with respect to the PEG moiety. In spite of the literature reports that PCL-PEG blending depends on the volume of each component, it was observable that immiscibility and phase separation can take place among the two components resulting in extra peaks during heating and cooling cycles [7].

3.2. FTIR

In the FTIR spectra of PEG, the peak at 1058 cm^{-1} attributes to the stretching vibration of the functional group C–O while the peak at 3399 cm^{-1} represents the stretching vibration of functional group of O–H. The peak at 2877 cm^{-1} is associated with the C–H stretching vibration of $–CH_2$ of PEG. The carbonyl stretching of pure PCL nanofibers was confirmed by the peak at 1722 cm^{-1}. The peaks at 1240 cm^{-1} and 1045 cm^{-1} represent asymmetric C–O–C stretching and symmetric C–O–C stretching, respectively. Since there are no new peaks displayed in the FTIR spectra of blend nanofibers, it was evident that there was no chemical interaction between PCL and PEG. The results were identical with the earlier reports by authors [8,9].

3.3. SEM

The morphological results of electrospun mats of PCL and their blend systems examined using SEM are shown in Figure 1. The PEG solution produced particles of average diameter of 2–3 μm. The PCL and PCL/PEG systems showed the ease of electrospinning with continuous fiber formation. From the SEM micrographs of blends, it was noticed that the electrospun mats comprised branched irregular fibers with coarse and flat surface. This was due to the incorporation of PEG which is difficult for electrospinning. There were no significant changes in fiber diameters of the blends as compared with pure PCL. The surface roughness of the nanofibers varies with the composition of their constituent polymers. The 90/10 system exhibited moderate roughness which promotes the cell adhesion and its growth.

4. CONCLUSION

The PCL and PCL/PEG blend nanofibers were successfully prepared by electrospinning technique while PEG solution was not spinnable. From FTIR studies, it was noticed that there was no chemical bond formation between the two polymers and the spectra revealed the characteristic peaks of respective polymers in the blend samples. However, PCL-PEG blends were spun at different concentrations, among which 90/10 sample indicated the desired surface roughness which was crucial for cell adherence. This sample will be analyzed for biocompatibility and tissue engineering studies.

5. ACKNOWLEDGMENT

The authors would like to thank CSIR for providing fellowship and also acknowledge Materials Science Division, CSIR-NAL, Bengaluru, and the Department of PST, JSS S&T University, Mysuru, for extending the facilities for carrying out our research work.

6. REFERENCES

1. M. Ramalingam, S. Ramakrishna, (2017) Introduction to nanofiber composites. In: ***Nanofiber Composites for Biomedical Applications***, Illinois: Woodhead Publishing, p3-29.
2. A. Bahareh, N. Parviz, R. Mohammad, A. Shahram, (2014) Poly (ε-caprolactone) fiber: An overview, ***Journal of Engineered Fabrics and Fibers***, **9:** 74-90.
3. P. K. Panda, B. Sahoo, (1990) Synthesis and applications of electrospun nanofibers-a review, ***Nanotechnology***, **1:** 399-416.
4. Q. Chen, J. A. Roether, A. R. Boccaccini, (2008) Tissue engineering scaffolds from bioactive glass and composite materials, ***Topics in Tissue Engineering***, **4:** 1-27.
5. C. Lu, G. Sheng-rong, Z. Yaqiong, Y. Ming, (2006) Synthesis and aggregation behavior of four types of different shaped PCL-PEG block copolymers, ***Polymer International***, **55:** 694-700.
6. S. Jia, D. Yu, Y. Zhu, Z. Wang, L. Chen, L. Fu, (2017) Morphology, crystallization and thermal behaviors of PLA-based composites:

Wonderful effects of hybrid GO/PEG via dynamic impregnating, ***Polymers***, **9:** 528.

7. A. O. Lobo, S. Afewerki, M. M. M. de Paula, P. Ghannadian, F. R. Marciano, Y. S. Zhang, T. J. Webster, A. Khademhosseini, (2018) Electrospun nanofiber blend with improved mechanical and biological performance, ***International Journal of Nanomedicine***, **13:** 7891-7903.
8. Y. Fang, H. Kang, W. Wang, H. Liu, X. Gao, (2010) Study on polyethylene glycol/epoxy resin composite as a form-stable phase change material, ***Energy Conversion and Management***, **51:** 2757-2761.
9. F. Ahmed, S. Saleemi, Z. Khatri, M. I. Abro, I. S. Kim, (2015) Co-electrospun poly (ε-caprolactone)/cellulose nanofibers-fabrication and characterization, ***Carbohydrate Polymers***, **115:** 388-393.