

RESEARCH PAPER

Fabrication of Electrospinning Nanofibers Scaffold of Grape seed Extract Loaded on Biopolymer: Physiochemical Characterization by Mechanical Strength/Tensile Strength

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ABSTRACT

In this investigation fabricate nanofibers scaffold Electro spun from PVA/Chitosan loaded with grape seed extract with valium ratio 7:3(V/V) followed by 0.5% (W/V). Was examined by FTIR, XRD and SEM, Tensile strength and contact angle. The results showed that PVA/CS nanofibers loaded with grape seed extract were successfully fabricated, exhibiting a uniform fibrous structure with a small number of beads and fine pores. The nanofibers showed good wettability, with a water contact angle of 64.21°. SEM analysis revealed that the fabricated fibers had an average diameter of approximately 243 nm, confirming the formation of nanometer-scale fibers.

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INTRODUCTION

Electrospinning is one of the electro hydrodynamic techniques that produces a precipitated liquid droplet which is elongated and extended into a continuous jet through the application of high voltage to a polymer melt or solution. The solvent evaporates and deposits ultra-fine fibers whose diameter ranges between a few nanometers and some micrometers on a collector. The first advantage of electrospinning is that it is a flexible technology that allows one to generate fibers of various morphology, high surface-area-

volume ratios and structural properties that can be varied. Due to these properties, nanofibers produced through electrospinning are highly useful in biomedical need particularly in tissue engineering and drug delivery systems [1]. The electrospinning process relies on the interaction of the surface tension and electrostatic forces on a polymer melt or solution. The electric field becomes high on the tip of the spinneret when the droplet is subjected to a high-voltage electric field, due to the fact that the electrostatic force is stronger than the surface tension. As it moves in the direction of a grounded collector, a charged jet

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is expelled from its apex, stretched, and thinned. Continuous nanofibers in the form of a nonwoven mat are deposited during this procedure as a result of solidification brought on by solvent evaporation or cooling [2]. This study used grape seeds extract as a natural bioactive and incorporate it electro spun nanofibers, because of its strong antioxidant properties which include phenolic compound which can scavenge free radical generated from radiation exposure and block formation of reactive oxygen species (ROS) and hydrogen peroxide and reduce stress corrosion, it works as cellular component damage protection. In addition, evaluate the biocompatibility of nanofibers as a wound dressing [3]. Scanning electron microscopy (SEM) has become an advanced tool for observing the three-dimensional ultrastructural properties of biological tissues, enabling in-depth observation of surface morphology and spatial relationships, which conventional light and transmission electron microscopy are not capable of observing [4].

MATERIALS AND METHODS

CS (2000-3500cps) very high molecular weight was purchased from Glentham LIFE SCIENCES Ltd and PVA (molecular weight 14000 g/mol) from THOMAS BAKER (India). Glacial acetic acid (99.7%) was purchased from LOBA CHEMIE PVT.LTD

Meanwhile, local grape seeds were collected from local markets, washed, dried, and ground into powder. Subsequently, grape seed extract (GSE) was prepared and converted into nanoparticles using solvent evaporation and ultrasonication techniques.

Preparation electrospinning grape seed nanofibers

As previously mentioned, polymer solutions were made. A 0.5% (w/v) concentration of dried grape seed extract was added to the polymeric matrix. To attain molecular-level dispersion, reduce particle aggregation, and create a homogenous solution appropriate for electrospinning, the resultant polymer–extract mixture was continuously swirled for 24 hours at 10ml polypropylene syringes with 21-gauge stainless steel needles were filled with the prepared solution. To start the electrospinning process, a copper electrode was used to link the needle to a high-voltage power source that delivered 20 kV. Flow rate 1 ml/h, As the grounded collector, the nanofibers were gathered on a flat plate covered with aluminum foil and placed 10 cm away from the needle tip. To eliminate any remaining solvent and create homogeneous Nano fibrous scaffolds appropriate for further characterization and biological uses, the Electro spun mats were

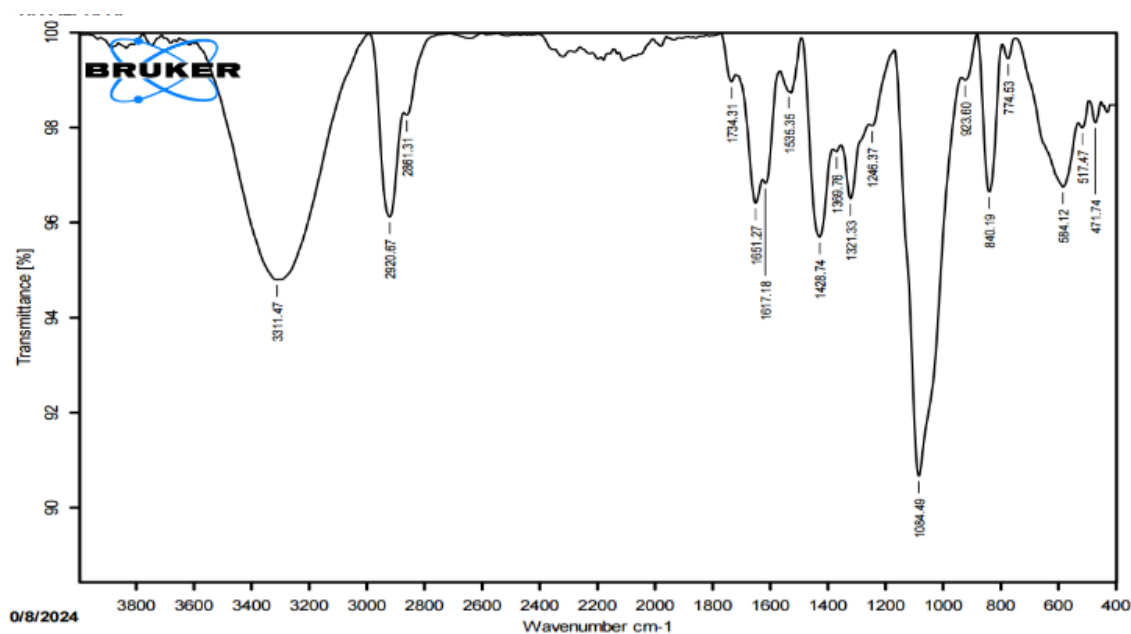


Fig. 1. FTIR spectrum of PVA/CS/GS NF.

allowed to dry at room temperature.

Fourier transform infrared (FTIR) spectroscopy

Fourier transform infrared (FTIR) spectroscopy was used to study the structural and chemical properties of loaded polymer nanofiber structures containing grape seed extract. The basis of FTIR spectroscopy is based on the vibrational transitions of molecular bonds, in which molecules absorb certain frequencies of infrared radiation that correspond to their distinct vibrational modes. Because each component has a distinct absorption spectrum, FTIR is a trustworthy instrument for both qualitative and quantitative identification of chemical structures. All of the spectra in this investigation were vector-normalized over the 400–4000 cm^{-1} wavenumber range to guarantee precise spectral data comparison and interpretation [5]. An FTIR-8400S spectrometer (Shimadzu, Japan).

X-ray Diffraction (XRD)

PVA/CS/GS nanofibers' crystal structures were characterized using X-ray diffraction (XRD). The XRD analysis was performed using the usual

methodologies described in the literature [6]. Generally, when an X-ray beam is directed onto a specimen at specific angles, diffraction occurs as a result of constructive interference from parallel crystalline planes according to Bragg's law ($n\lambda = 2d \sin \theta$), where λ is the wavelength of the incident X-rays, θ is the diffraction angle, d is the interplanar spacing, and n represents the order of diffraction. In amorphous materials, the lack of long-range ordering causes destructive interference, resulting in broad halos rather than precise peaks.

Contact angle (CA)

measurements were utilized to evaluate the wettability of both grape seed extract-loaded nanofiber scaffolds and Electro spun polymer nanofiber scaffolds. This method involves measuring the angle formed between a water droplet and the sample surface, which is commonly used to determine whether a surface is hydrophilic or hydrophobic [7].

Field Emission Scanning Electron Microscopy (FESEM)

Field Emission Scanning Electron Microscopy

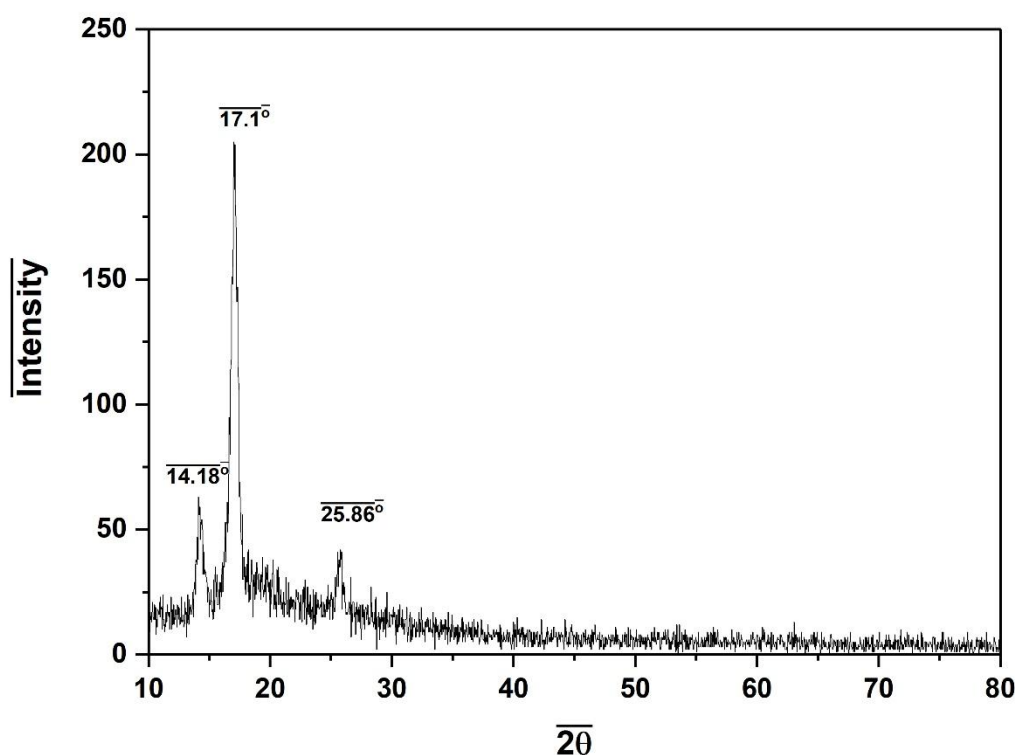


Fig. 2. PVA/CS/GS nanofibers.

(FESEM) was used to analyze the surface morphology of the grape seed extract-loaded nanofiber scaffolds and the Electro spun polymer nanofiber scaffolds. Fiber morphology, diameter distribution, and surface architecture at the micro- and nanoscale can be seen in detail because to this method's high-resolution two-dimensional pictures [8].

RESULTS AND DISCUSSION

FTIR of PVA/CS/GS NF

Fig. 1 shows the FTIR spectrum of PVA/CS/GS, the absorption bands of the three components in their blends. This indicates homogeneity of the prepared blends. New absorption bands appear here at 1734 cm^{-1} due to the carbonyl group presented in Grape seeds, and a band at 1617 cm^{-1} in the chemical structure of Grape seeds.

X-ray diffraction (XRD)

Fig. 2 show the broad peak around $19\text{--}20^\circ$ (2θ) corresponds to amorphous cellulose or lignocellulosic components, representing disordered arrangements of carbon, oxygen, and

hydrogen in the grape seed matrix. The peak at $\sim 24^\circ$ (2θ) reflects semi-crystalline regions, possibly due to polyphenols or proanthocyanidin aggregates (which are partially ordered aromatic structures). The absence of sharp, narrow peaks suggests mostly amorphous material, confirming that grape seed powder lacks significant inorganic crystallinity (such as metal oxides or salts). Thus, the XRD pattern of grape seed typically shows a semi-amorphous nature, consistent with organic plant matrices rich in polyphenols and carbohydrates [9].

SEM of PVA/CS/GS Nanofibers

The mean particles of grape seed were about 642 nm . Fig. 3 show the surface morphology of the GSE powder, The fiber bundles embedded within the GS matrix. The diameter of the enlarged fibers ranged between $321\text{--}607\text{ nm}$.

Hydrophilicity

the Fig. 4 showed the hydrophilicity of nanofibers scaffold by measured propensity of material to attract and interact with water The

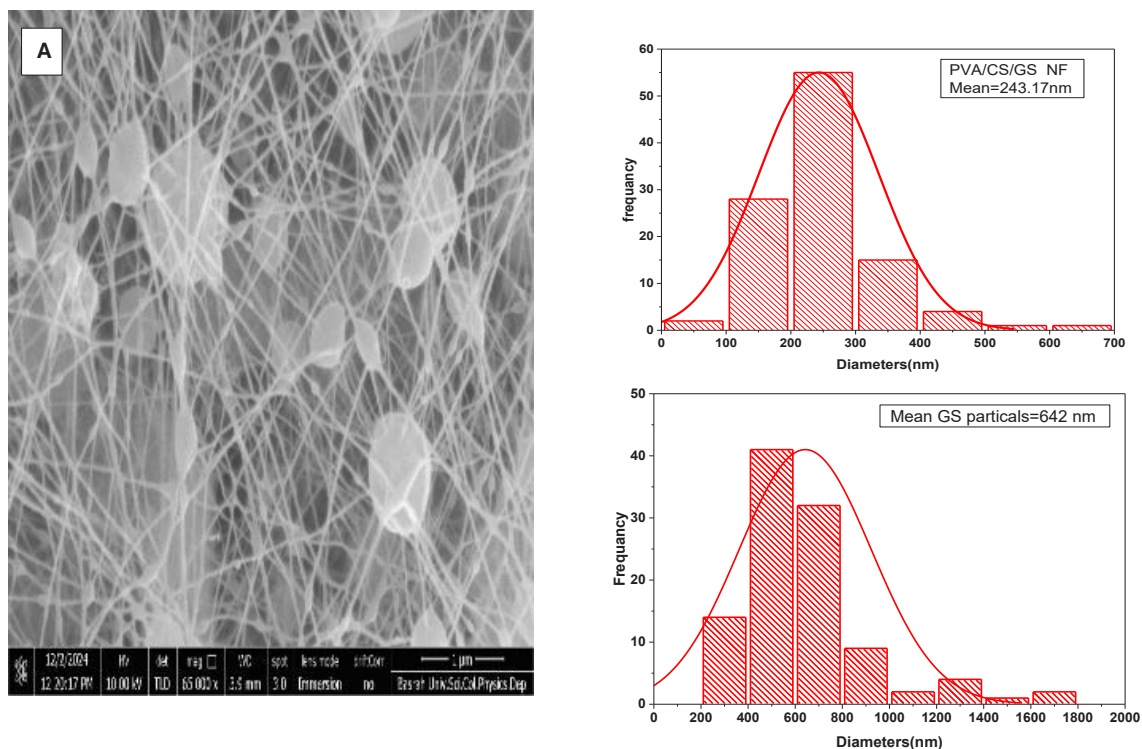


Fig. 3. SEM of PVA/CS/GSE NF (A) showing fine morphology with occasional small beads of fiber histogram (B) illustrating the frequency of fiber diameters (C) The frequency of GS particles.

primary determinants of a surface's wettability are surface energy and roughness. The measured contact angle of the PVA/CS/GS nanofibers blend was found to be about 56.36 °, 87.90 °, 66.72°, and 64.21° respectively, indicating the hydrophilic nature of the materials. The CA of CS is higher than PVA related to compact polymer chain, while PVA have small CA value show higher hydrophilicity, which can be attributed to the presence (-OH) functional groups in the backbone molecular structure.

Tensile Strength test

Tensile tests on composite fibers are performed to determine their tensile properties during material development and qualification processes. These tests are also essential for the design and structural layout of composite materials, as well as for quality assurance. The PVA/CS/GS NF stress-strain relationship is depicted in Fig. 5. The Young's Modulus was 0.013MPa, and the tensile strength was 1.8N, and the elongation for PVA/CS/GS NF was 14.9%. The decrease in Young's modulus and Tensile strength after adding GSE is related to modifying the internal structure of the nanofiber and weakens the intermolecular interaction between PVA and CS.

The FTIR spectrum of chitosan/PVA nanocomposite has distinct spectral patterns due to the interactions between the two polymers.

The migration of the hydroxyl absorption band to a position of 3288 cm^{-1} , plus the enhancement of band width is an indication of the development of a strong hydrogen bond between chitosan and polyphenols. The alterations in the amide bands at 1647 and 1538 cm^{-1} evidence the variation of the chemical environment of the amine and amide groups, indicating the interaction between chitosan and polyphenol molecules and creation of the hydrogen bonds or molecular complexes between chitosan and polyvinyl alcohol (PVA).

In addition, the fact that the chitosan carbon-oxygen (C-O) stretching bands have been integrated into a strong absorption band at 1082 cm^{-1} implies that there has been the development of an all-encompassing polymer network. Evidence of grape seed adsorption into the polymer matrix is also clear in the chitosan-polyphenol composite loaded with grape seed nanoparticles. The existence of absorption bands at 1734 cm^{-1} , which indicates carbonyl groups, and the 1617 cm^{-1} , which indicates aromatic carbon-carbon (C=C) bonds, shows the presence of grape seed polyphenols as [10].

Moreover, the changes in the bands of chitosan amide at 1647 and 1538 cm^{-1} and the expansion of the O-H stretching region are additional indicators of the existence of hydrogen-bond interaction between chitosan and the phenolic compounds of grape seeds. The X-ray diffraction (XRD) of

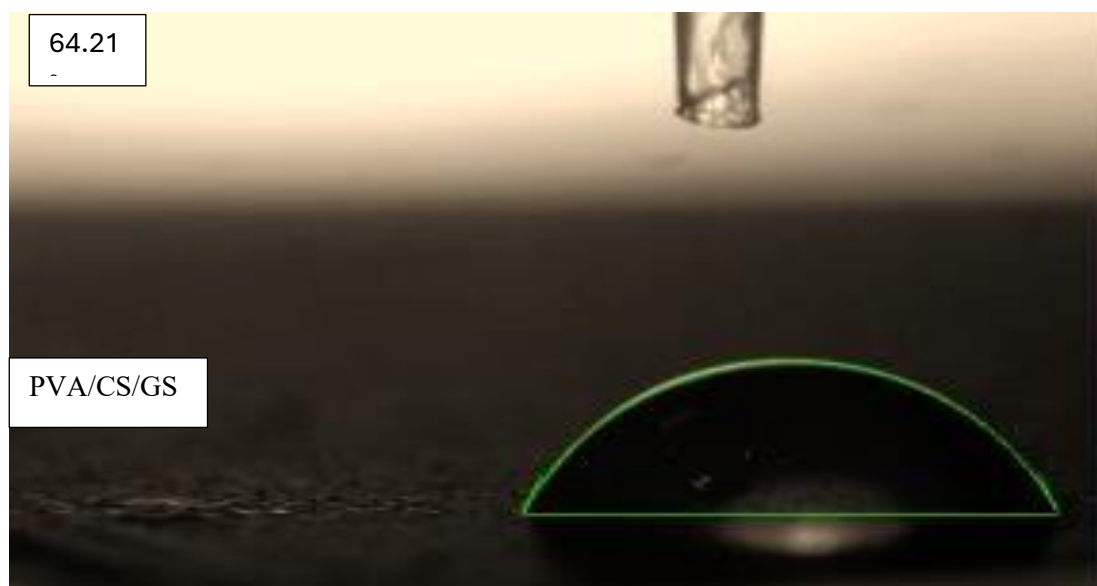


Fig. 4. Contact angle (CA) measurements PVA/CS/GS.

chitosan polymers reveals that chitosan has a crystalline structure because of the occurrence of hydroxyl and amine groups and the development of strong hydrogen bonds which provide an ordered arrangement of molecules. This is in agreement with the earlier structural research on chitosan [11]. Conversely, polyvinyl alcohol (PVA) XRD analysis depicts that the polymer film is quasi-crystalline, characteristic of partially hydrolyzed polyvinyl alcohol polymer [12]. It is certainly evident that the typical crystallinity peak of PVA indicates its quasi-crystalline structure, whereby polymer chains are bound together by the hydrogen bond [13].

The XRD data of grape seed powder indicate a semi-crystalline structure, which is congruent with reports of natural plant matrices being mostly amorphous because of their heterogeneous polyphenol and polysaccharide composition. The occurrence of secondary crystalline peaks is explained by the existence of microcrystalline cellulose areas in the plant matter [14,15].

Moreover, the chitosan/PVA blend diffraction peaks demonstrate a significant drop in intensity, which can be attributed to partial destruction of the initial crystalline structures and formation of

new hydrogen bonds among the molecules of polymers. The behavior is consistent with past results, which have shown that the combination of both chitosan and PVA creates hybrid semi-crystalline structures due to the interaction of the chain of polymer [16].

Grape nanoparticles incorporated in the CS/PVA nanofibers led to a decrease in the surface roughness and the maintenance of fibers. The distribution of grape seed nanoparticles using scanning electron microscopy showed a uniform distribution of particles with an average diameter of about 243.17 nm, which showed great compatibility between the nanoparticles and the polymer blend of CS/PVA. The finding is in agreement with more recent reports on the incorporation of natural antioxidants into nanofibers, which also indicate that chitosan-based nanofibers promote structural stabilization and uniformity when natural antioxidant nanoparticles are loaded through hydrogen bonding and electrostatic interactions [17].

The analysis of the mechanical properties of CS/PVA nanofibers coupled with the addition of grape seed nanoparticles has indicated a decrease in the elastic modulus as well as tensile strength of

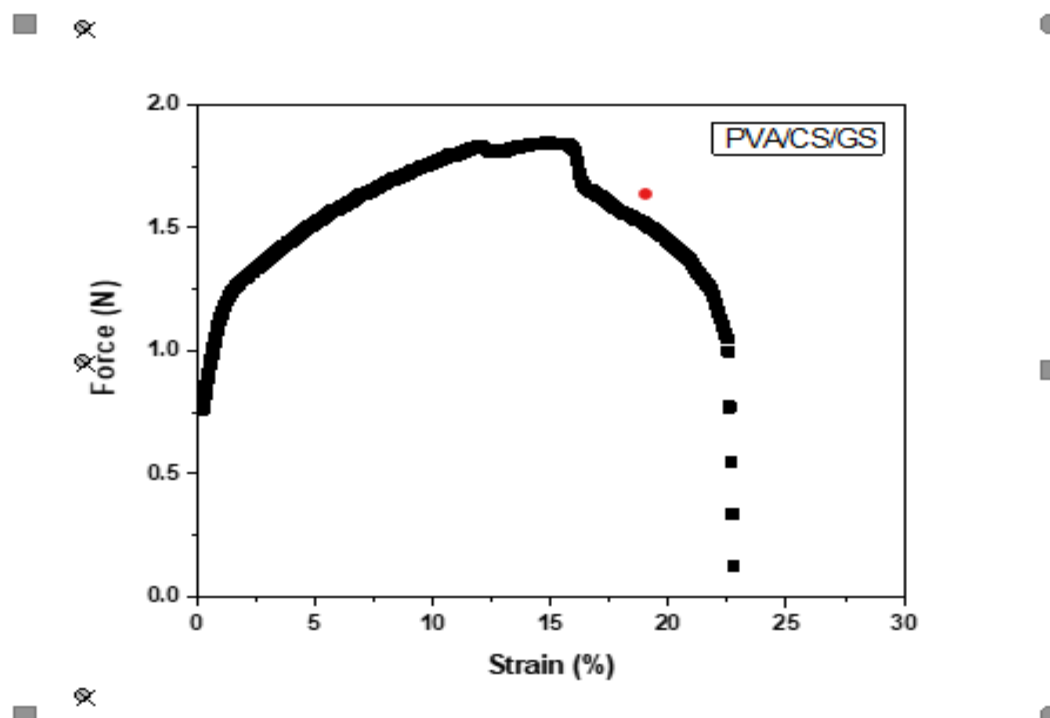


Fig. 5. Stress-strain curves and tensile strength of PVA/CS/GS NF.

the matrix after the addition of the nanoparticles. This decrease is explained by the fact that more structural anisotropy of fibers and a partial break of the hydrogen bond between polymer chains occur, which agrees with the prior research [18].

PVA/CS/GS nanofibers, on the contrary, had a greater elongation at the end of break (37%), which implies greater flexibility. This is in line with the findings that natural extracts and polyphenol additives have been reported to behave as a structural modifier in polymer matrices, enhance chain mobility, heighten flexibility, and lower stiffness [19,20]. On the whole, the mechanical profile of the PVA/CS/GS nanofibers is more flexible, and they are more suitable in the context of applications that involve compliant biomaterials.

CONCLUSION

In this study, multifunctional bioactive nanofibrous scaffolds composed of polyvinyl alcohol (PVA), chitosan (CS), and grape seed extract (GSE) nanoparticles were successfully fabricated via the electrospinning technique for potential biomedical and wound dressing applications. FTIR spectroscopic analysis confirmed the homogeneous dispersion and successful incorporation of grape seed polyphenols into the PVA/CS polymeric matrix, evidenced by characteristic absorption bands at 1734 cm^{-1} (C=O stretching) and 1617 cm^{-1} (aromatic C=C vibrations), as well as significant shifts and broadening in the -OH and amide regions that demonstrate strong intermolecular hydrogen bonding. XRD patterns corroborated the semi-amorphous nature of the GSE-loaded scaffolds, showing that the addition of polyphenolic compounds partially disrupts the intrinsic crystallinity of PVA and CS, facilitating the formation of a flexible hybrid polymer network. FESEM and SEM micrographs revealed a continuous, defect-free, and uniform nanofibrous architecture with GSE nanoparticles (average diameter 243-642 nm) homogeneously entrapped within the electrospun fibers (321-607 nm), demonstrating excellent interfacial compatibility. Water contact angle measurements validated the pronounced hydrophilic character of the composite nanofibers (ranging from 56.36° to 87.90° , which is essential for cell adhesion, fluid management, and exudate absorption in wound healing. Mechanical evaluation demonstrated that the incorporation of GSE acted as a structural modifier; while it led to a moderate decrease in tensile strength and Young's

modulus (0.013 MPa), it substantially enhanced fiber ductility and flexibility (elongation at break reaching up to (37%)), yielding a compliant scaffold tailored for dynamic tissue movement. Overall, the synergism among the biocompatibility of chitosan, the film-forming and hydrophilic nature of PVA, and the antioxidant functionality of grape seed extract establishes the electrospun PVA/CS/GS nanofibrous mat as a highly promising, green-derived candidate for advanced wound dressing and tissue regeneration scaffolds.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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