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FABRICATION OF COPPER OXIDE NANOPARTICLES DOPED PCL/PVP NANOFIBROUS MATS BY ELECTROSPINNING AND EVALUATION OF THEIR ANTIBACTERIAL ACTIVITIES

BAKIR OKSİT NANOPARTİKÜL KATKILI PCL/PVP NANOLİFLİ MATLARIN ELEKTROÇEKİM İLE ÜRETİMİ VE ANTİBAKTERİYEL AKTİVİTELERİNİN DEĞERLENDİRİLMESİ

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ABSTRACT

In this study, PCL/PVP/CuO nanofibrous mats for antibacterial applications were fabricated by electrospinning technique using PCL/PVP as a biopolymer matrix and copper oxide nanoparticles (CuONPs) as antimicrobial agents. PCL/PVP/CuO nanofibrous mats were successfully produced by doping different ratios of CuONPs (0.5%, 1%, and 2% wt) into the PCL/PVP solution. The chemical, morphological, and wetting properties of the prepared composite nanofibrous mats were evaluated by FT-IR, FE-SEM analysis, and water contact angle measurements. The morphological investigation indicated that the fiber diameters of the resulting nanofibers decreased as the CuONP content added to the PCL/PVP matrix increased, and the mean fiber diameter value measured for the PCL/PVP/2%CuONPs nanofibrous sample was 186.73. Moreover, wetting behavior of nanofiber surfaces displayed that the incorporation of PVP significantly enhanced the surface wettability of PCL with hydrophobic properties, but the addition of CuONPs to the obtained PCL/PVP matrix decreased it. An in vitro bactericidal assay was performed to investigate the efficacy of PCL/PVP/CuO nanofibrous samples against *Staphylococcus aureus*. The addition of CuONPs to the fibers led to antibacterial activity, which was found to increase with higher doping ratios. The results showed the potential of PCL/PVP/CuO nanofibrous mats to serve as an effective biomaterial for antibacterial applications.

Keywords: Antibacterial nanofibrous mats, copper oxide nanoparticle, electrospinning technique, polycaprolactone, polyvinylpyrrolidone

ÖZET

Bu çalışmada, biyopolimer matris olarak PCL/PVP ve antimikrobiyal ajan olarak bakır oksit nanopartikülleri (CuONPs) kullanılarak elektroçekim tekniği ile antibakteriyel uygulamalar için PCL/PVP/CuO nanolifli matlar üretildi. PCL/PVP/CuO nanolifli matlar, PCL/PVP çözeltisine farklı oranlarda CuONPs'in (ağırlıkça %0,5, %1 ve %2) katkılanmasıyla başarılı bir şekilde üretildi. Hazırlanan kompozit nanolifli matların kimyasal, morfolojik ve ıslanma özellikleri FT-IR, FE-SEM analizleri ve temas açısı ölçümü ile değerlendirildi. Morfolojik inceleme, PCL/PVP matrisine eklenen CuONPs içeriği arttıkça sonuçlanan nanoliflerin lif çaplarının azaldığını ve PCL/PVP/2%CuONPs nanolifli örneği için ölçülen ortalama lif çapı değerinin 186.73º olduğunu gösterdi. Dahası, nanolif yüzeylerinin ıslanma davranışı, PVP'nin dahil edilmesinin, hidrofobik özelliklere sahip PCL'ın yüzey ıslanabilirliğini önemli ölçüde arttırırken ancak elde edilen PCL/PVP matrise CuONPs ilavesinin ise azalttığını

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gösterdi. PCL/PVP/CuO nanoliflli örneklerinin *Staphylococcus aureus*'a karşı etkinliğini araştırmak için bir in vitro antibakteriyel aktivite testi gerçekleştirildi. CuONPs'ın liflere eklenmesi antibakteriyel aktiviteye yol açtı ve bu aktivitenin daha yüksek nanopartikül katkılanma oranlarıyla arttığı bulundu. Sonuçlar, PCL/PVP/CuO nanolifli matların antibakteriyel uygulamalar için etkili bir biyomalzeme olarak işlev görebilme potansiyelini gösterdi.

Anahtar Kelimeler: Antibakteriyel nanolifli matlar, bakır oksit nanopartikül, elektroçekim tekniği, polikaprolakton, polivinilpirolidon

INTRODUCTION

The interdisciplinary field of nanotechnology is rapidly expanding and holds enormous potential to transform a variety of industries, such as agriculture, electronics, medicine, and textiles. Particles that fit within the range of 1 to 100 nanometers in at least one dimension are known as nanoparticles. These materials have piqued the interest of researchers due to their notable chemical and physical properties that differentiate them from their bulk forms. These attributes include but are not limited to, strength and stiffness, electrical conductivity, chemical reactivity, and diverse biological activities (Khan, Saeed, & Khan, 2019; Sahooli, Sabbaghi, & Saboori, 2012). Inorganic nanoparticles, particularly metal-based ones, have gained popularity as a potential solution for bacteria resistant to traditional antibiotics. These nanoparticles have unique mechanisms of action that differ from traditional antibiotics, making them effective against resistant strains and even preventing the development of resistance. In addition, they can target multiple biomolecules involved in the development of resistant strains (Slavin, Asnis, Häfeli, & Bach, 2017). Compared to organic antimicrobial agents, CuO nanoparticles are a more robust and stable material with a longer shelf life. As efficient materials for the treatment of water, textiles, and other items, copper and its complexes are being employed more frequently. Microorganisms are extremely sensitive to copper and its complexes even at low concentrations, whereas human tissues barely react to them. Due to their tendency for rapid oxidation in the presence of air, metallic copper nanoparticles, particularly those formed when Cu^{2+}, can become unstable chemically and physically (Das, Nath, Phukon, & Dolui, 2013; Raffi et al., 2010).

Electrospinning technology can produce nanofibers with controllable size and structure, high porosity, and a large specific surface area. Electrospinning nanofibers exhibit a significant increase in specific surface area, at least 100 times more than conventional microfibers, due to their significantly smaller fiber diameter than microfibers made using traditional spinning techniques including melt spinning, wet spinning, dry spinning, and dry-wet spinning(Qi et al., 2021). These properties make them highly desirable for use in a variety of research and development fields, particularly in biological materials (Li et al., 2018, 2019a). The electrospinning technique has attracted a lot of attention in recent years for producing biodegradable polymer fibers due to its ease, cost-effectiveness, and reproducibility. Biodegradable polymers such as polyglycolic acid, polylactic acid, polylactic-co-glycolic acid, fibrin, gelatin, PHB, and poly (ε-caprolactone) (PCL) are commonly used in this process (Ajalloueian et al., 2014; Maleki, Azimi, Ismaeilimoghadam, & Danti, 2022; Mayilswamy, Jaya Prakash, & Kandasubramanian, 2023; Sarıipek, Özaytekin, & Erci, 2023).

Polycaprolactone (PCL) is a synthetic polymer that is semi-crystalline in nature. With a glass transition temperature of -62°C, PCL has a relatively low melting point, typically ranging from 55-60°C, depending on the degree of crystallinity (Fadaie, Mirzaei, Geramizadeh, & Asvar, 2018). PCL has been approved for use in the biomedical field due to its biocompatibility and low cost. PCL is a highly compatible and soluble material that can be easily processed at room temperature. In addition, PCL nanocomposites provide an ideal matrix to promote cell growth and ensure sustained release of antimicrobial agents (Díez-Pascual & Luceño-Sánchez, 2021). Its approval by the FDA makes it an attractive option for use in drug delivery systems, especially as electrospinning nanofibers (Saracino et al., 2021). While PCL has the potential for use in the medical field, its hydrophobic nature and slow degradation rate are limiting factors. To overcome this, blending hydrophilic polymers with PCL could improve water diffusion near PCL chains, leading to faster hydrolytic cleavage and potentially overcoming these biological disadvantages (Y. Wang et al., 2022). The characteristic properties of PCL, coupled with its disadvantages, have opened avenues for the development of novel functional composite materials. PCL's performance can be enhanced by combining it with various substances for a range of biomedical applications (Raina, Pahwa, Khosla, Gupta, & Gupta, 2022).

Polyvinylpyrrolidone (PVP) is a favorable option when it comes to blending with PCL because of its biocompatibility, ability to dissolve efficiently in various solvents, and potential to interact with materials that are both hydrophobic and hydrophilic (Chaudhuri, Mondal, Ray, & Sarkar, 2016). There is preliminary evidence that

adding PVP can make PCL nanofiber membranes more hydrophilic(Li et al., 2019b). Without incorporating antibacterial properties, PCL and PVP utilization may result in severe infections that necessitate further medical interventions. Thus, to prevent this outcome, it is critical to introduce strong antibacterial agents, for instance, metal and metal oxide nanoparticles, into PCL/PVP nanofibers, thereby conferring them with exceptional antibacterial characteristics (Liu et al., 2022).

The primary aim of this research was to fabricate nanofibrous mats composed of a blend of polycaprolactone (PCL) and polyvinylpyrrolidone (PVP) doped with varying concentrations of copper oxide nanoparticles using the electrospinning technique. The nanofibrous samples were characterized using scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FT-IR). Also, the wetting behavior of electrospinning nanofibrous mats was analyzed via water contact angle tests. Furthermore, the prepared nanofibers were tested for their antimicrobial activity against *S. aureus* to evaluate their potential use in medical applications.

MATERIAL AND METHODS

Materials

Poly (ε-caprolactone) (PCL, molecular weight 45,000 g mol^{-1}) granules and polyvinylprolidone (PVP, molecular weight 40,000 g mol^{-1}) in powder form were procured from Sigma-Aldrich (USA) to serve as a biopolymer matrix. The solvents used were dichloromethane (DCM, 99,8%, Merck) and ethanol (Eth, %99.8, Merck). Copper oxide nanopowder with 30 nm average particle size to be used as additive material was obtained from Nanografi Nano Teknoloji, Turkiye. The chemicals utilized in the work were of analytical grade and were employed without any additional purification.

Preparation of Electrospun PCL/PVP/CuONPs Nanofibrous Mats

Pure PCL and PCL/PVP solutions were prepared in DCM-Eth (V/V, 3:1) binary solvent by adjusting the weight of each component. To produce PCL nanofibers, pure PCL granules were dissolved in CF-DMF and the resulting spinning solution, containing 17% (w/v) PCL, was continuously stirred for 4 h at room temperature. For PCL/PVP nanofibers, PCL and PVP (w/w, 7:3) granules were dissolved sequentially in DCM-Eth solvent. First, PVP granules were mechanically stirred for 4 h, and then PVP granules were added and stirred continuously overnight for complete dissolution. To produce PCL/PVP/CuONPs nanocomposite fibers, CuONP content was added to the PCL/PVP spinning solution as 0.5, 1.0, 2.0 wt% of the total polymer mass and mechanically stirred for 60 min to achieve homogenization. All working solutions were let to stabilize for 1h at ambient temperature before electrospinning. The spinning solutions were placed into a plastic syringe and subsequently, electrospinning was carried out at a flow rate of 0.5 mL/h, a high voltage of 12 kV, a distance between the needle tip and the collector of 12 cm, 35 °C and 40% relative humidity. Then, the resulting nanofibrous mats were kept in an oven at 37 °C for one day to remove solvent residues. The production processes of PCL/PVP/CuONPs nanofibrous composite mats are given in Figure 1.

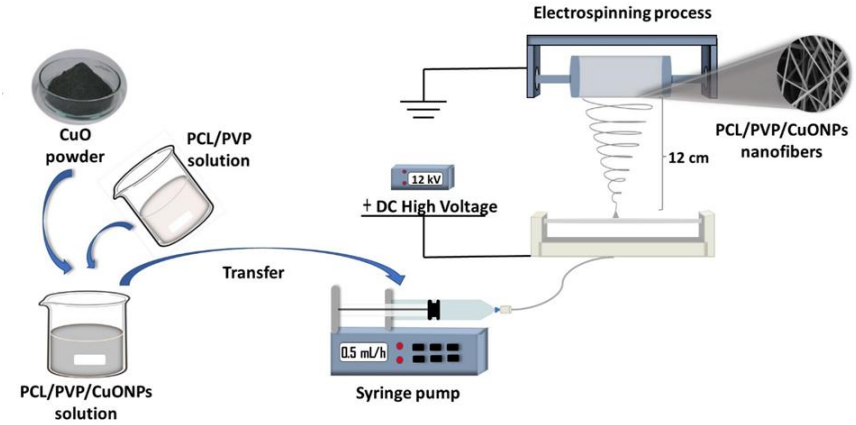

Figure 1. Schematic Representation of Production Processes of PCL/PVP/CuONPs Nanofibrous Composite Mats

Characterization of Fabricated Nanofibrous Mats

The chemical structures of the produced nanofibrous mats were evaluated employing Fourier transform infrared spectroscopy (FT-IR) Bruker Vertex 70, attenuated total reflection mode in the range 400-4000 cm^{-1} with a resolution

of 4 cm^{-1}. The morphology of the prepared nanofiber mat surfaces was observed by using a field emission scanning electron microscope (FE-SEM,Zeiss GeminiSEM 500)in BITAM laboratory, Turkey. ImageJ software was utilized to determine the average diameter (AD) of the fibers (n = ∼100) and given as standard deviation ± mean diameter. Wetting properties of the nanofiber mat surfaces were detected at room temperature using a digital water contact angle meter (Biolin Scientific Attension, Theta Lite). Each nanofiber sample was cut to 20 × 20 mm and placed on the setup. 5 µL volume of water droplets at room temperature was dripped onto all surfaces to measure the contact angle. Six measurements were averaged for each sample.

Antibacterial Test

The plate count assay was used to evaluate the antibacterial efficacy of the nanofibrous mats. The bacterial suspensions of *Staphylococcus aureus* (*S. aureus*) were prepared at a concentration of 1.5×10^8 CFU/mL in 10 mL of liquid Nutrient Broth (NB) medium and then added to the surface of a circular sheet mat (20 mg: 20.00 mm in diameter) and incubated at 37°C for 24 h. Following the incubation period, the nanofibrous samples underwent a washing process using 1 mL of NB medium. Then, 100 µL of the washed solution was collected and placed onto Nutrient agar plates to facilitate inoculation. Subsequently, the plates were incubated at 37°C for 24 h, and then the colonies present in the plates were counted for analysis.

RESULT AND DISCUSSION

The chemical structures of all prepared nanofibrous mats were revealed by FT-IR spectroscopy. The FT-IR spectra of the nanofibrous PCL, PCL/PVP, and PCL/PVP/CuO mats are displayed in Figure 2. The FT-IR spectra of PCL mats exhibited characteristic bands at 1724 cm^{-1} (C=O stretching vibrations); 1471 cm^{-1}, 1453 cm^{-1} and 1367 cm^{-1} (-CH_2 bending vibrations); 1240 cm^{-1} and 1177 cm^{-1} (C-O-C stretching vibrations); and 1106 cm-1 and 1048 cm^{-1} (C-O stretching vibrations) (Chakrapani, Gnanamani, Giridev, Madhusoothanan, & Sekaran, 2012). FT-IR spectra of PCL/PVP nanofibers showed bands at 1724 cm^{-1} (C=O of PCL), 1667 cm^{-1} (C=O of PVP), 2944 cm^{-1} (C-H asymmetric stretching), 2867 cm^{-1} (C-H symmetric stretching), 1421 cm^{-1} (C-N vibration), 1460 cm^{-1} (C-H deformation), 1366 cm^{-1} (CO symmetric stretching) and 1240 cm^{-1} (C-O-C) asymmetric stretching). This demonstrates the inclusion of these two polymers in the PCL/PVP nanofiber composite composition (Jia, Huang, Dong, Liu, & Nie, 2016). The spectrum of PCL/PVP/CuONPs nanocomposites displayed nearly no change in the peak positions of the absorption peaks indicating the molecular dispersion of CuONPs in PCL/PVP nanofibers. The unchanged characteristic absorption bands indicate that PCL/PVP nanofibers retain the chemical feature of PCL/PVP nanofibers and the antimicrobial feature of CuONPs. Furthermore, their combination confirms the absence of any chemical interactions during the electrospinning process of PCL/PVP/CuONPs nanofibers and the successful physical doping of CuONPs into PCL/PVP nanofibers by Vander Waals forces. Moreover, a rise in the intensity of these characteristic peaks was observed with increasing CuONPs addition in nanofibers with different CuONPs content. This can be attributed to the constitution of new atoms or bonds within the environment in the presence of CuONPs.

FE-SEM images of the produced PCL, PCL/PVP, and PCL/PVP/CuO nanofiber samples are shown in Figure 3. For the determination of the average diameter of the obtained fibers, the diameter of fibers was measured at 100 various points on the FE-SEM image. It was observed that the prepared nanofibers generally exhibited beadles and continuous uniformly distributed nanostructures of varying diameters. The average fiber diameters of PCL and PCL/PVP nanofibers were 81.80 and 261.98 nm, respectively (Table 1). The distinction can be attributed to the PCL solution's stronger conductivity and lower viscosity when compared to PVP, which raises the fibers' elongation stress. One of the most crucial electrospinning variables impacting fiber diameter is solution viscosity. When compared to a solution with a lower viscosity, a higher viscosity solution produces fibers with a larger diameter. Since the addition of PVP to PCL led to a more viscous solution, it resulted in nanofibers with higher average fiber diameter than PCL nanofibers. Moreover, the fiber diameter distribution plot shows that PCL nanofibers have a diameter distribution ranging from 40-300 nm but mostly fibers between 50-100 nm were obtained. It was determined that approximately 48% of PCL/PVP fiber diameters were in the range of 200-300 nm. With the addition of 0.5% CuONP to PCL/PVP nanofibers, the average fiber diameter declined to 210.30 and it was observed that approximately 41% of PCL/PVP/CuONPs nanofibers comprised of fibers with a diameter of 100-200 nm and 42% of fibers with a diameter of 200-300 nm. The enhanced solution conductivity, which has an impact on the Taylor cone size, can be used to explain the decrease in the diameter size of PCL/PVP nanofibers. It was found that the diameter distribution of PCL/PVP fibers was wide and that this structure was not particularly homogeneous in the diameter distribution

histogram plots of the nanofibers in Figure 4. In the case of PCL/PVP/CuONPs fibers, together with a decrease in diameter, and a rise in the homogeneity of the fiber structures was also seen.

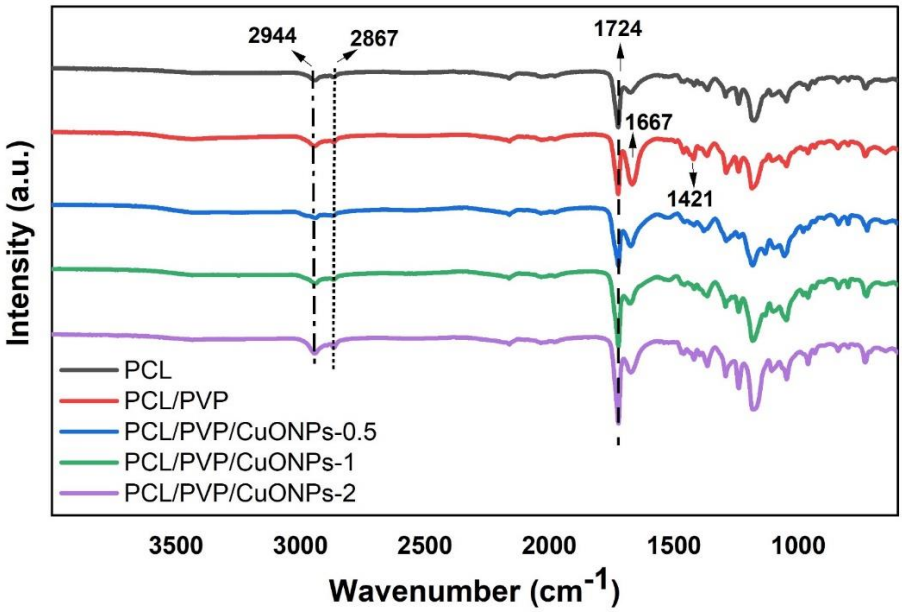

Figure 2. The FT-IR Spectra of PCL, PVP and PCL/PVP/CuO (with 0.5%, 1% and 2% CuO content) Nanofibrous Mats

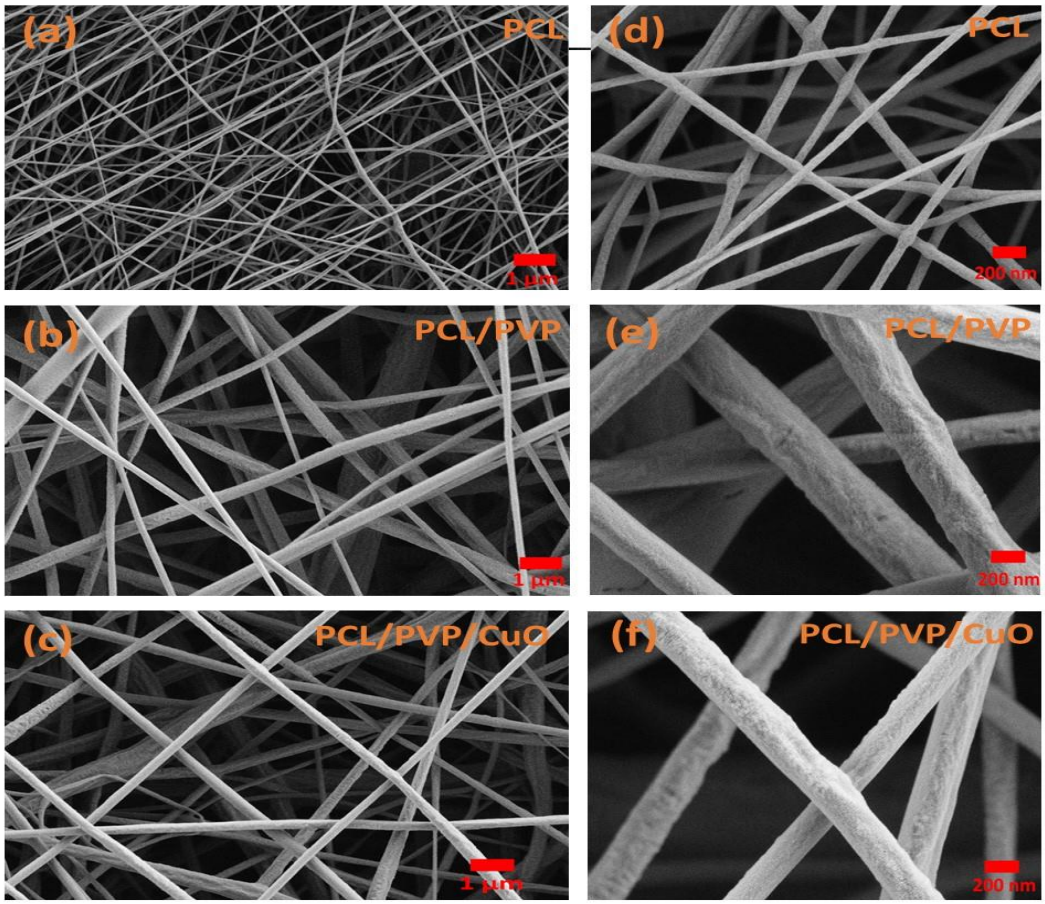

Figure 3. FE-SEM images of (a) PCL, (b) PCL/PVP, and (c) PCL/PVP/2%CuONPs, and high magnification images of (d) PCL, (e) PCL/PVP, and (f) PCL/PVP/0.5%CuONPs

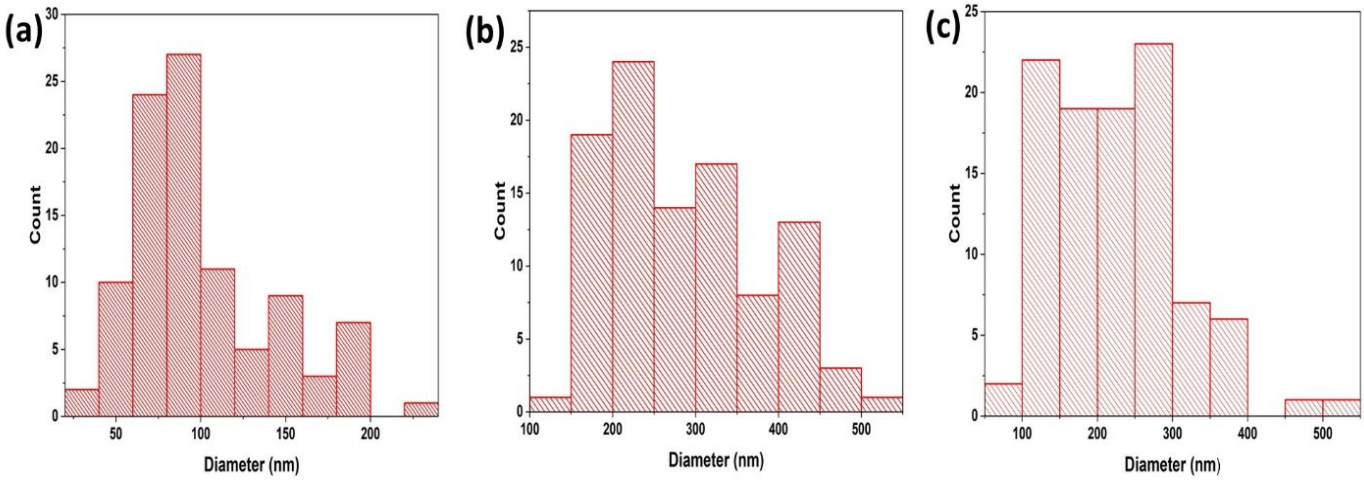

Figure 4. Fiber size distribution histograms of (a) PCL, (b) PCL/PVP and (c) PCL/PVP/0.5%CuONPs nanofibrous mats

Table 1. Average Fiber Diameters and Water Contact Angle Measurements of PCL, PCL/PVP and PCL/PVP/CuONPs Nanofibrous Mats

Sample	Mass ratios of PCL/PVP (wt%)	Content of CuONPs (wt%)	WCA values (°)	Average fiber diameters (nm)
PCL	1/0	0	117.46	81.80
PCL/PVP	7/3	0	61.53	261.98
PCL/PVP/CuO-0.5	7/3	0.5	75.56	210.30
PCL/PVP/CuO-1	7/3	1	90. 46	198.04
PCL/PVP/CuO-2	7/3	2	102.92	186.73

Figure 5 displays the elemental mapping for all the nanofibers produced. The EDS spectra of PCL, PCL/PVP, PCL/PVP/2%CuONPs nanofibers revealed the presence of C, O, N and Cu elements, along with their corresponding peak intensities and mass percentages. Electron mapping imaging was employed to verify the uniform dispersion of CuONPs in the fibers during the production of CuONPs-doped PCL/PVP nanofibers. The mapping outcomes for the FE-SEM image of PCL nanofibers in Figure 6a are presented in Figure 6b, which clearly indicates the successful production of PCL nanofibers and the presence of C and O in the medium. Figure 6d displays the mapping results for the FE-SEM image in Figure 6c. The successful production of PCL/PVP nanofibers was verified by the presence of C, O, and N elements in the medium, as indicated by the research results. The mapping outcomes obtained on the FE-SEM image in Figure 5e are finally displayed in Figure 5f. The findings demonstrated that Cu was present in the PCL/PVP fibers. Additionally, it was found that the PCL/PVP/CuO medium had more oxygen than PCL/PVP did. The presence of Cu in the medium and the increase in the amount of O is evidence of the successful doping of CuO NPs into PCL/PVP nanofibers. In addition, it was determined that all elements were predominantly homogeneously distributed, but Cu was also observed to accumulate in very small spots.

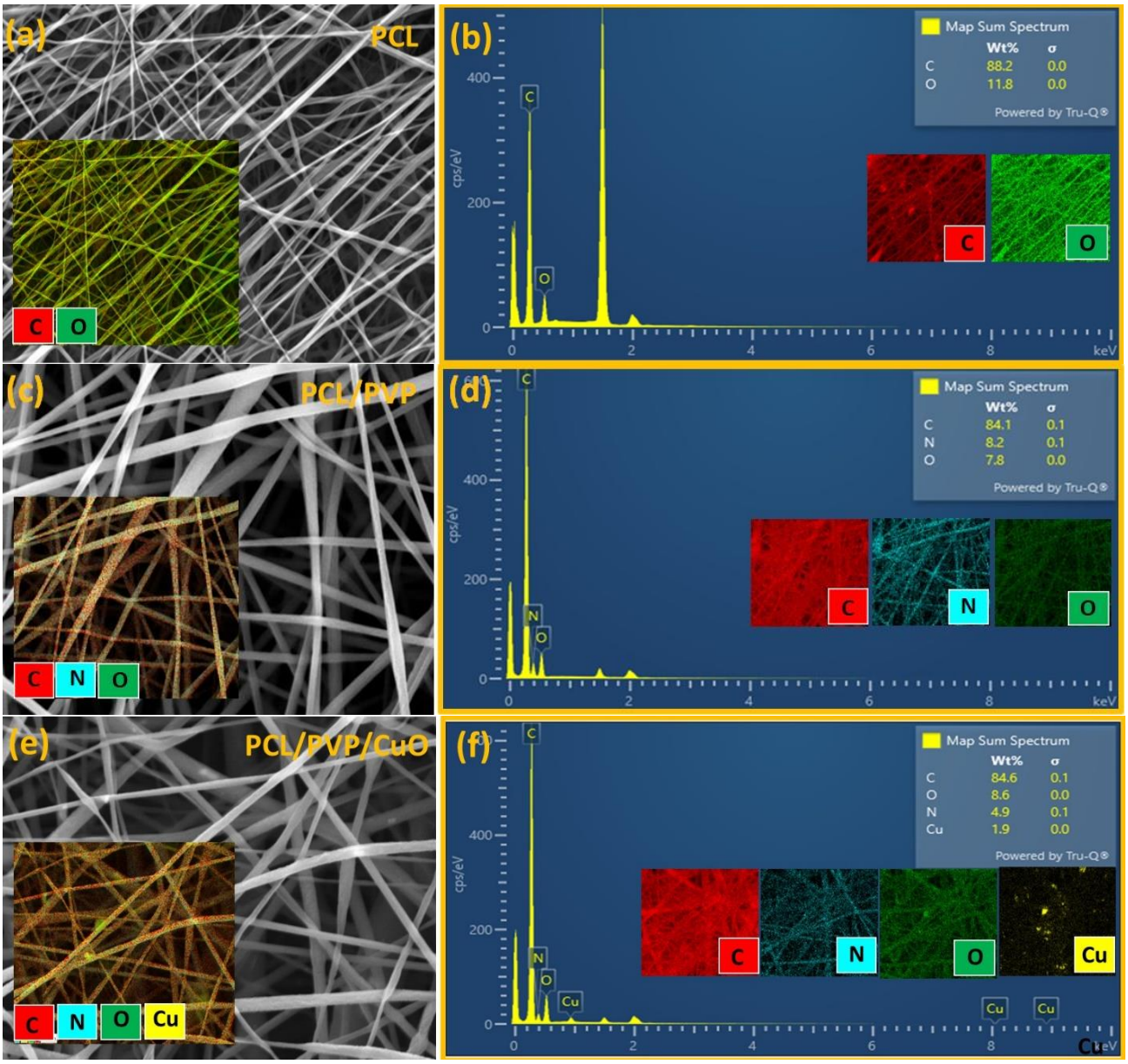

Figure 5. FE-SEM EDS Analysis of (a) PCL, (c) PCL/PVP, and (e) PCL/PVP/2.0%CuONPs, and FE-SEM EDS Mapping Images of (b) PCL, (d) PCL/PVP, and (f) PCL/PVP/2.0%CuONPs

Water contact angle (WCA) tests were conducted, as depicted in Figure 6, to examine the wetting characteristics of nanofibrous mats. The measured WCA values of each nanofibrous surface are given in Table 1. The contact angles of water droplets on the surfaces of the PCL, PCL/PVP, and PCL/PVP/CuONPs nanofiber were measured to determine their wettability. The PCL nanofibrous mat surface's WCA value, which has a hydrophobic nature due to the crystal regions in its structure, was measured as 117.46°. The WCA value of PCL/PVP composite nanofibers obtained by blending PCL with PVP was measured as 61.53°, which was significantly lower than that of PCL. The hydrophilic nature of PVP is mainly due to its hydrophilic amino and carboxyl groups and hence, when blended with PCL, it leads to the improvement of the wettability behavior of PCL fibers. Different contents of CuONPs were dropped to the PCL/PVP matrix and as displayed in Figure 6, the WCA value of PCL/PVP fibers increased with increasing CuO*f*NPs (0.5, 1 and 2wt%). The addition of CuONPs to PCL/PVP nanofibrous mats resulted in a decrease in their hydrophilic nature, as evidenced by the increase in contact angles which was measured for

PCL/PVP/0.5%CuONPs, PCL/PVP/1%CuONPs, and PCL/PVP/2%CuONPs nanofibrous mats as 75.56°, 90.46°, and 102.92°, respectively (Mallakpour & Mansourzadeh, 2017; L. Wang et al., 2023)

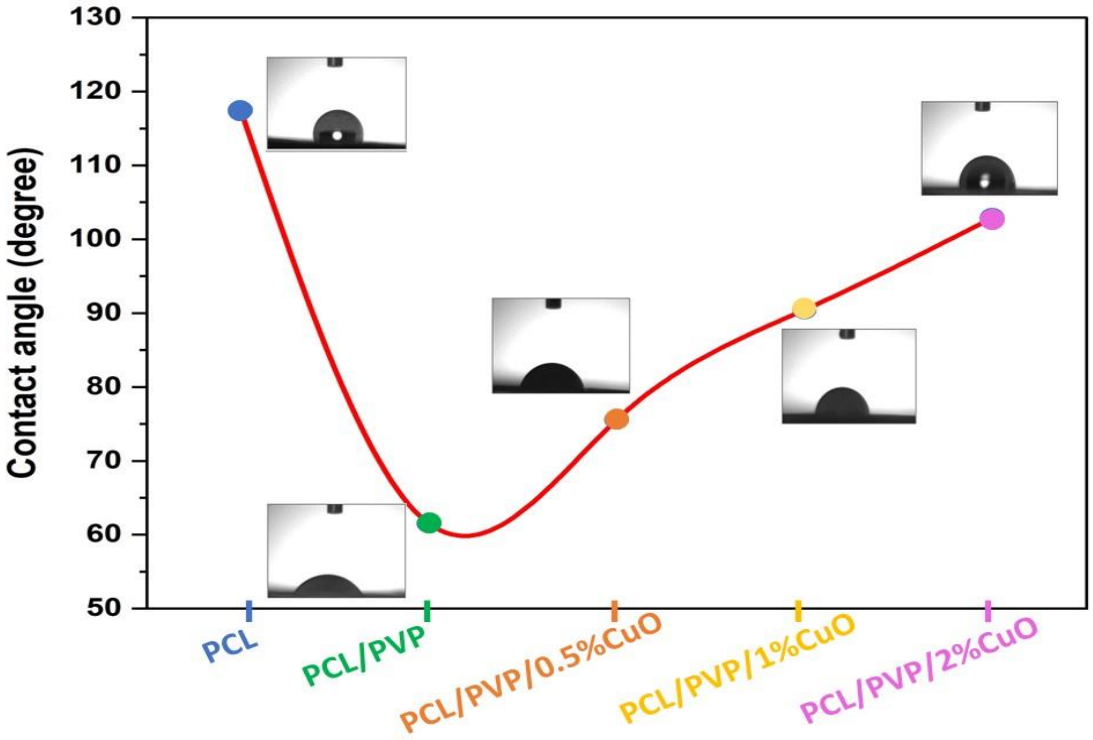

Figure 6. Water Contact Angle Measurements of PCL, PCL/PVP, and PCL/PVP/CuONPs Nanofibrous Mats

Staphylococcus aureus was selected to assess the antibacterial activity of the nanofibrous samples. As shown in Figure 7, the nanofibers containing CuONPs showed antibacterial activities. In comparison with PVP/PCL, CuONPs/PVP/PCL nanofibers enhanced the antibacterial efficacy due to the incorporation of CuONPs. The efficiency also increased with the amount of CuONPs loaded into the nanofibrous mats.

The loading of some metal nanoparticles and substances derived from plants on PCL/PVP nanofibrous mats and their antibacterial properties have been the subject of numerous studies in the literature. Hu et al. (2018) employed electrospinning technology to create nanofibers made of polyvinylpyrrolidone (PVP) and polycaprolactone (PCL) that were loaded with zinc oxide/silver bimetallic nanoparticles. They tested the antibacterial activity of these nanofibers against *Staphylococcus aureus* and *Escherichia coli*. The research discovered that adding metal nanoparticles to PVP/PCL nanofibers produced fibers with improved antibacterial activity and good biocompatibility(Hu et al., 2018). In another research, Suganya et al. (2011) suggested using nanofibers constructed of PCL/PVP that included crude bark extracts of the medicinal plant *Tecomella undulata* to treat skin infections. In their study, they evaluated the antibacterial activity of the nanofibrous samples against *Pseudomonas aeruginosa*, *Staphylococcus aureus*, and *Escherichia coli* and obtained inhibition zones of 30, 24, and 28 mm in diameter, respectively. They concluded that the loaded PCL/PVP nanofibrous mats have strong antibacterial activity against these bacteria and can be used in the treatment of skin infections (Suganya, Senthil Ram, Lakshmi, & Giridev, 2011). Additionally, recent studies have shown that CuONPs have strong antibacterial and antibiofilm properties. In a recent study, Erci et al. (2020) synthesized and evaluated the antibacterial and antibiofilm characteristics of CuONPs on *Staphylococcus aureus*. The results established that CuONPs demonstrated significant potential in preventing the growth of *Staphylococcus aureus* (Erci, Cakir-Koc, Yontem, & Torlak, 2020).

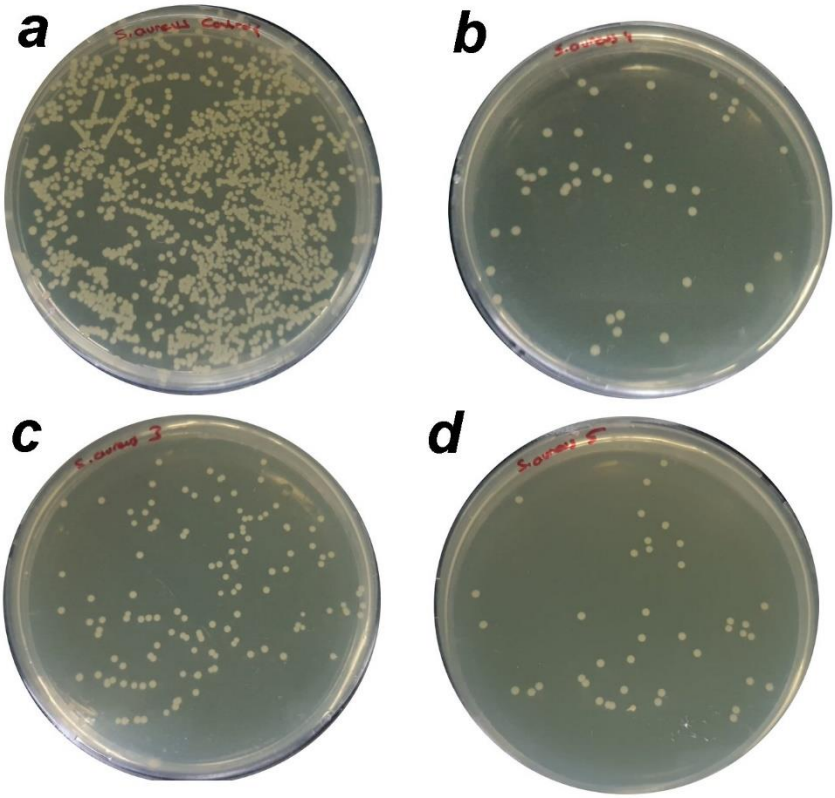

Figure 7. Antibacterial Activity Nanofibrous Mats Against *S. aureus*. (a) PCL/PVP, (b) PCL/PVP/0.5%CuO, (b) PCL/PVP/1.0%CuO, and (c) PCL/PVP/2.0%CuO

CONCLUSION

CuONPs were incorporated into PCL/PVP nanofibrous mats using electrospinning technology, and their antibacterial properties were evaluated. This is the first study to demonstrate the loading of CuONPs into PCL/PVP nanofibrous mats and to evaluate their antibacterial potentials. The morphologies of the prepared nanofibrous mats were observed to be uniform and continuous. The interactions between PCL and PVP were weak, according to FT-IR spectra. The resulting nanofibers were found to have both hydrophobic and hydrophilic properties and were confirmed to contain CuONPs through FE-SEM, EDX, and FT-IR analyses. The nanofibers also demonstrated good bacteriostatic properties against *S. aureus*. The results suggest that increasing the addition of CuONPs decreased the fiber diameters of nanofiber samples while increasing their antibacterial properties. In conclusion, this work has demonstrated the potential of PCL/PVP/CuONPs nanofibrous mats for use in a variety of antibacterial applications, including materials for wound healing.

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