



Article

Preparation of Nano Zinc Oxide with Polycaprolactone and Evaluation of Electrospun Scaffold Composite

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Abstract: The creation and development of medical polymer polycaprolactone (PCL) and zinc oxide nanofibers (ZnO NPs) and their catalytic activity against dangerous bacteria and germs are the most important electrospinning techniques in this work. 2% and 3% were applied in addition to the electrospinning process. Pure polymer divided by the weight of zinc oxide nanoparticles (PCL). Scanning electron microscopy (SEM), AFM surface roughness, X-ray diffraction (XRD), material crystallization, FT-IR spectroscopy, and the nature of the interaction between nanoparticles (ZnO) and pure polymer were used to determine the fiber diameter. This reaction shows catalytic properties and mechanical stability through mechanical property testing. In addition to how viscosity increases with increasing concentration. High porosity is provided by water angle testing and good adhesion by these scaffolds. These concentrations can be used as an antibacterial and antibacterial agent.

Keywords: Nanofibers, ZnO nanoparticles, Electrospinning, Scaffolds, Polycaprolactone.

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1. Introduction

This time, nanofibers are utilised in air filtration and many other applications and are characterised by high porosity and permeability as well as by specialised surfaces thanks to electrospinning methods, which are significant and widely used [1],[2],[3]. Due to its high porosity and effectiveness against bacteria and pathogens, it can be employed in protective masks [4],[5]. A thermoplastic, semi-crystalline, biocompatible polymer has undergone thorough analysis [6], [7]. This Polycaprolactone polymer is frequently utilized in tissue engineering [8]. is an effective means of medication delivery [9]. The mechanical and antibacterial qualities of polycaprolactone are excellent [10]. When nanoparticles are added to PCL fibres, a hybrid PCL/ZnO nanocomposite is created [11]. It possesses the same qualities as (ZnO) particles, which are used to treat bacteria because they can prevent their growth [12]. Human biocompatibility of this PCL/ZnO hybrid fibre is good [13]. Electrospinning is a sophisticated biological technique used to create nanofibers [14]. High porosity and a huge surface area characterize Elect Trepan [15],[16]. Using the results of this investigation, ZnO nanoparticles were mixed with PCL polymer. nanoparticles produced by electrospinning, with consideration for the device's parameters, the feed rate, the voltage being used, and the distance between the needle and the collector. The size, shape, and surface morphology of the nanofibers were investigated. After adding quantities of nanoparticles, this hybrid PCL/ZnO compound

demonstrated that it has strong corrosion resistance and is used in the field of medicine and its applications.

2. Materials and Methods

2.1. Materials

The following items were purchased in Baghdad: polycaprolactone ($M_w = 85\,000$), nanoparticles (120 nm particle size), ZnO (100 nm particle size) from Sigma Aldrich, as well as methanol and chloroform, methanol, dimethylformamide (DMF), and 12 ml plastic syringes. The materials that were used but not specifically mentioned were created by the labs of the University of Baghdad.

2.2. Nanofibers produced by Electrospinning

The strong solvents chloroform, methanol, and DMF (75%:25%:15%) should be used to dissolve the pure polymer PCL (12% w/v) over the course of 10 hours while stirring continuously. Different amounts of ZnO nanoparticles—2% and 3% by weight of the whole polymer—are dissolved in the polymer. The optimum homogeneous mixture that may be used for electrospinning is created by appropriately combining these amounts with PCL and stirring continuously [17]. Calculate the 10 cm distance between the collection and the needle and the flow rate of 0.4 ml per hour to regulate the quantity of pumping. The solution is subjected to a 14 kV voltage. the collector with aluminum plates.

2.3. Fiber morphological

The samples were examined utilising the morphological membrane characteristics, the PCL/ZnO hybrid nanofiber films, the JEOL JFC 1600 Autofine paint, and an electronic scanner JSM 6390 operating at 10 Kv. The JSM 6390 electronic scanner was then utilised at 10 kV to analyse each sample. We utilise ImageJ software to analyse SEM pictures and determine the typical diameter of nanofibers.

2.4. Applying Atomic Force to Microscopy

AFM (Ambios Universal, CA, USA) atomic force microscope equipment was used to confirm the images. surface irregularities. With a resolution of 510×510 pixels, images were captured using high resolution AFM [18].

2.5 Spectroscopy of X-rays

We used X-ray diffraction to gather information on the PCL/ZnO hybrid nanofiber using EDX analysis using a Swift ED attached to a JEOL JSM 6390 SEM. This information included the X-ray signals, intensity, and energy distribution produced when the radiated electron hit the sample's surface.

2.6. Infrared Spectrum FTIR

The 500 FTIR spectrum from Perkin Elmer was used to obtain infrared (FT-IR) spectra with good range. Both the pure polymer PCL as well as the ZnO nanoparticles and the range of spectra 500–4000 were treated.

2.7. Mechanical properties

We install the handles in the tensile testing machine. After that, we place the sample between the handles. The length of the gauge should be five times the thickness of the sample. After that, we start the machine and break the sample until it reaches the deformation stage (usually up to 20-30% strain). We record the load force and displacement [19].

2.8. Angle of Contact with Water

Through the use of the Wilhelm plate method, the hydration angle of the nanofibers was determined [20]. Weighing the sample and soaking it in PBS for 24 hours allowed us to calculate the fibre porosity after the produced nanofibers were electrospun and evaluated for hydrophobicity and hydrophilicity using water with a surface tension of 72 dyne/cm.

3. Results

3.1.SEM analysis

The hybrid composite of ZnO nanoparticles and pure PCL polymer is indicated by its surface morphology. We discovered using SEM that the nanofiber membrane exhibits

good permeability and random structure. Fibers (Fig. 2a) The nanofibers are 393 ± 64 with 2 wt% ZnO in average diameter. In addition, we observe that pure PCL and 3 wt% ZnO fibers contain nanoparticles on their surface (Fig.2b). Nanoparticles release a lot of energy. This composite contains 400 ± 53 nanofibers on average. Likewise, the pure polymer has an average fiber diameter of 300 ± 80 , as in (Fig.2c)[21].

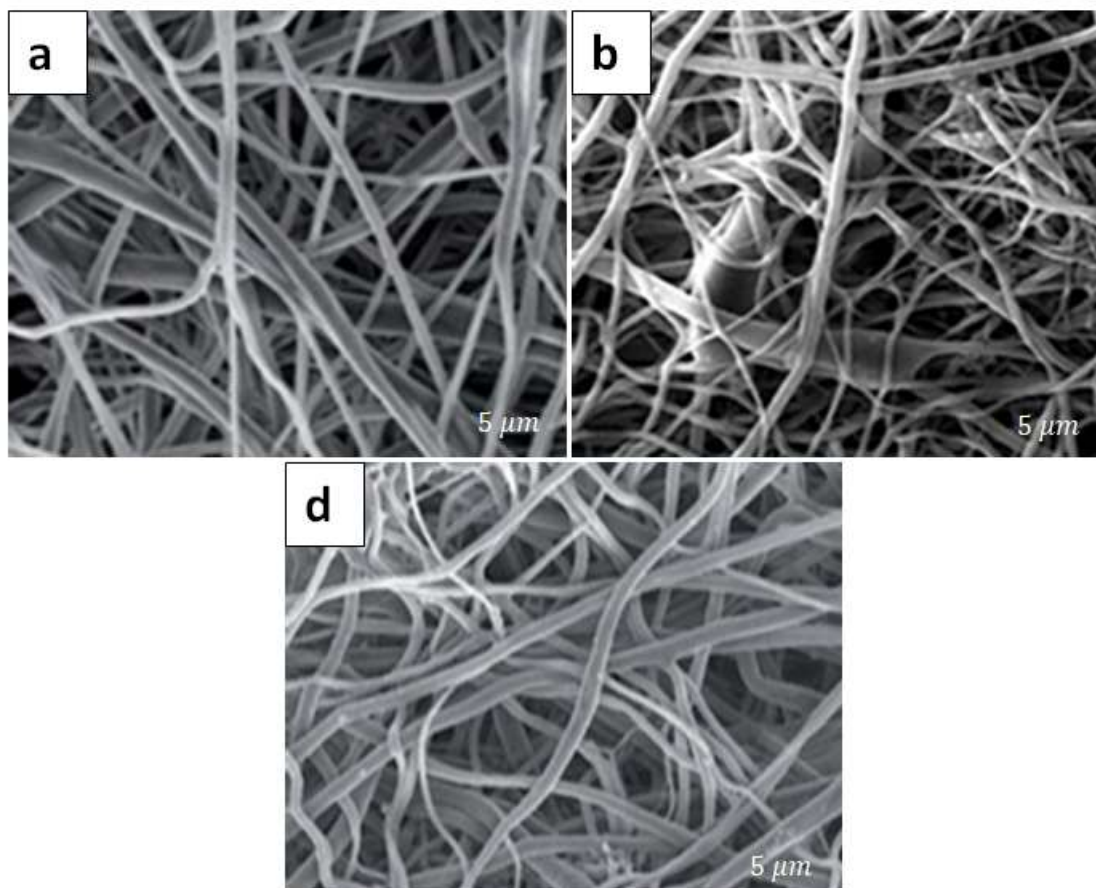


Figure 1. SEM images of an electrospun PCL membrane with distribution of fibre diameter and 2 wt% and 3 wt% ZnO nanoparticles

3.2. Investigation Using Atomic Force Microscopy

Using the AFM test to assess the roughness of surface forms. Additionally, we measured the rough surface R_{rms} at three concentrations and took three pictures, which are displayed in Fig.2. Pure PCL and the degree of surface roughness after varying ZnO concentrations of 2% and 3% were added. This leads us to the conclusion that surface roughness rises with concentration [22]. Table 1 illustrates how surface roughness is impacted by the nanofiber. Roughness increases with fibre diameter [23].

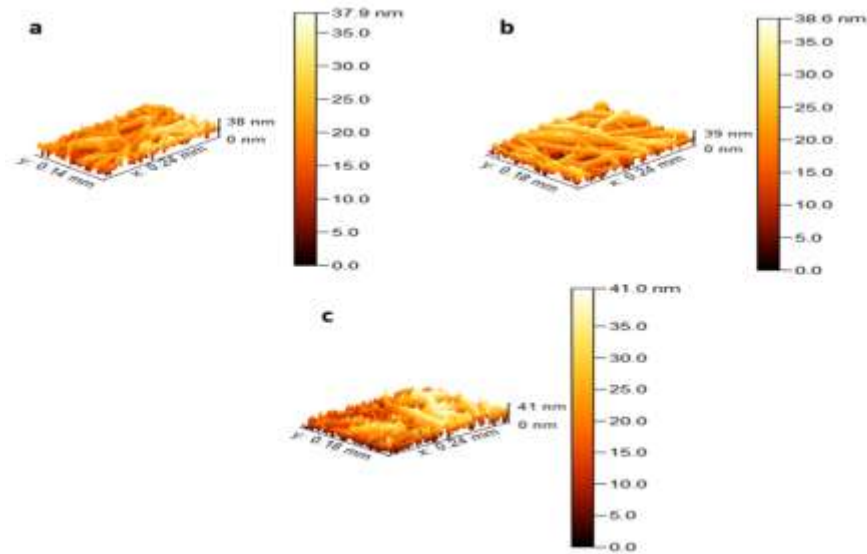


Figure.2. AFM, PCL and PCL/ZnO scaffolds: Surface Roughness, a = PCL, b = PCL/ZnO 2wt.% , c = PCL/ZnO 3wt.%,

Table 1. This table includes the surface roughness parameters of PCL with the addition of different concentrations of ZnO. Parameters include average roughness (S_a), root mean square (S_q), maximum height (S_z),

Sample	Roughness average S_a (nm)	Root mean square S_q (nm)	Ten point height S_z (nm)	Average diameter (nm)
PCL Neat	5.38846	6.59038	37.9451	16.6413
2wt.% ZnO	6.25131	7.61650	38.5882	17.4853
3wt.% ZnO	8.19555	9.95029	41.0000	19.7152

3.3.FT-IR analysis

The carbonyl ester group ($-C=O$) PCL has a peak at 1724 cm^{-1} , as determined by FTIR studies, according to Fig.2 [24], [25], [26]. Additionally, a peak representing the crystallization phase is present at 1294 cm^{-1} ($C-C$). Peaks at 1239 cm^{-1} and 1168 cm^{-1} , which suggest $C-O-C$ representation and non-symmetry, are present. There is also a carbonate group peak at 877 cm^{-1} and another peak at 961 cm^{-1} , which we can see. This shows that CO_2 , $-\text{OH}$, and PO_4 have been mixed.

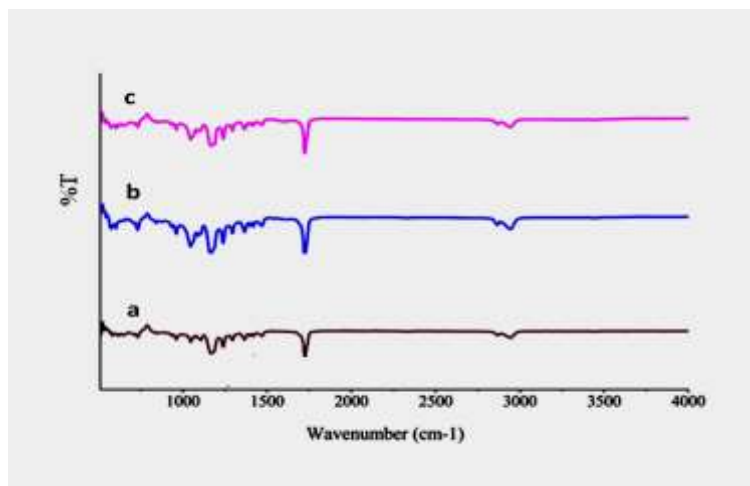


Figure 2. FT-IR analysis a = PCL , b = PCL/ZnO 2wt.% , c = PCL/ZnO 3wt. %

3.4. X-ray diffraction analysis

The pure PCL nanofiber's spectrum is represented by the X-ray diffraction of the XRD nanofibers in Fig.3. When ZnO nanoparticles are added, we observe three distinct peaks at $2\theta = 21.4^\circ$, 22.0° , and 23.7° degrees, respectively, corresponding to (110), (111), and (200) the level of the crystal structure [27]. There is a change in crystal behavior. At $2\theta = 21.2^\circ$, 22.0° , and 23.7° , the sharpness of the pure polymer exhibits diffraction. We observe that the semi-crystallinity of this PCL polymer is what distinguishes it from pure PCL, as shown by the fact that the peaks are identical when ZnO 2wt. % is added. We see a change in the nature of the pure PCL polymer when the concentration of ZnO is raised because adding a concentration of ZnO 3wt.% to the nanofibers of the polymer modifies the crystalline structure. We observe that the nanofibers with a tiny diameter have a high degree of crystallinity and are more solid.

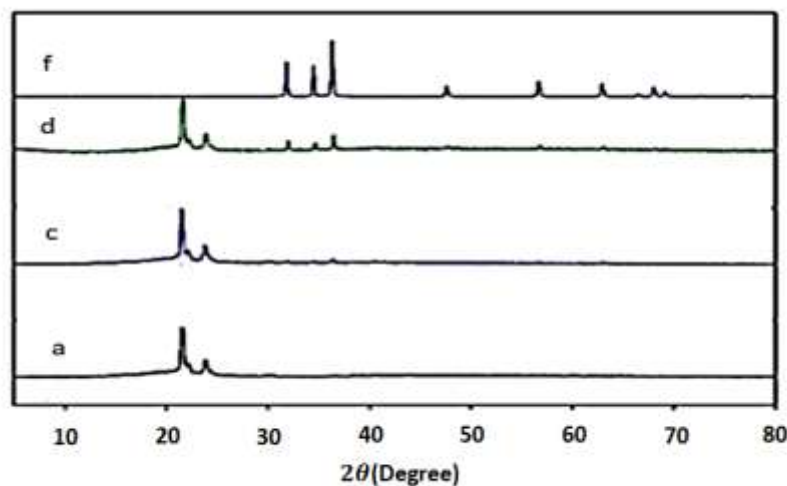


Figure 3. XRD analysis a = PCL , c = PCL/ZnO 2wt.% , d = PCL/ZnO 3wt.% , f = ZnO

3.5. Strength of Tensile

The PCL/ZnO strain and tension curves were displayed. The more nanoparticles there are, the higher the tensile strength. Along with the increase in nanoparticles, the tensile modulus also rises. Tensile strength of PCL polymer fibre is higher than that of plain PCL, even with 3% ZnO particles present. Because of their high surface energy and rapid agglomeration, nanoparticles provide the explanation. Consequently, these particles coalesce and accumulate within the polymer matrix, impeding the transmission of stress among the concentration centres. By increasing the interstitial gaps and shifting

the stress from the PCL polymer fibre to the filler, ZnO nanoparticles help. Table.1 [28] displays the findings for mechanical and tensile properties. It demonstrates that the 3% sample has equal fracture stress and modulus, as well as elongation (%). When combined with ZnO nanoparticles, the pure PCL polymer, which has an elongation of 252 ± 9 MPa, exhibits great flexibility. Both the tensile strength modulus and elasticity increased by 326 ± 13 MPa as a result. We draw the conclusion that PCL/ZnO fibres with greater nanoparticle content have good mechanical qualities.

Table 2. Tensile properties of electrolytic PCL fibers containing different concentrations of ZnO particles

Sample	Stress at break (MPa)	Elongation at break (%)	Modulus (MPa)
PCL	1.23 ± 0.22	252 ± 9	2.83 ± 0.88
PCL/ZnO 2wt.%	1.72 ± 0.19	326 ± 13	3.39 ± 0.73
PCL/ZnO 3wt.%	2.77 ± 0.19	426 ± 17	4.27 ± 1.29

3.6. Porosity and Water Contact Angle

Wettability influences both the adherence of cells to the fibre and their proliferation, making it a crucial function in the testing of wound dressings. Initially, when we select the PCL matting, we find that this polymer is hydrophobic [29]. This test is used to assess the phases and behaviour of the nanofibers composited in PCL/ZnO, as well as the wettability of the surface. Once the first 2% concentration of ZnO nanoparticles is added, the hydration angle is high Fig.5. The hydration angle reduced, which we noticed, indicating that this polymer is now hydrophilic instead of hydrophobic. It is also evident from Table 2 that the angle dropped by more than 3% upon adding the second concentration of ZnO. As such, Testing is necessary to determine whether the produced substance is safe and able to enter cells.

Table 2. Water contact angle and hydrophobicity by electrospinning of nanofibers

Sample	Contact Angle (°) (Hydrophilicity)	FR (mL/h)	TCD (cm)	Voltage (kV)
PCL	106.2 ± 4.4	0.4	10	14
PCL/ZnO 2wt.%	95.6 ± 4.9	0.4	10	14
PCL/ZnO 3wt.%	87.6 ± 4.3	0.4	10	14

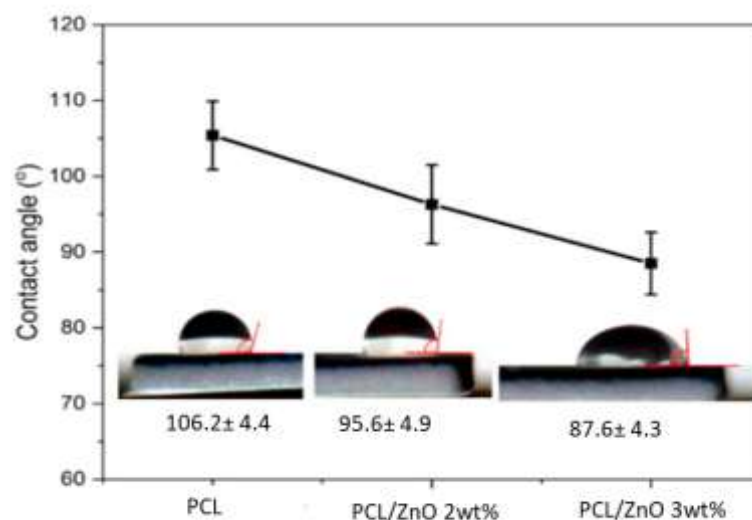


Figure 5. Angle of contact between PCL/ZnO nanofibrous scaffolds

4. Discussion

Nanotechnology is a modern technology used in materials processing and has distinct biological applications. The main goals of tissue engineering in various fields include gene transfer, wound healing, and cancer prevention. Furthermore, it is used to replace damaged cells with biomaterials made from nanofibers [30]. Target characterization: Two concentrations of nanofiber were used, achieving good results for modern applications [31]. Nanofibers are a good product and have good conditions for vascular regeneration. Electrospinning can be used to form nano-scaffolds. Pure nanofibers were produced from PCL. Figure 1 shows that they are finely grained, and the added concentrations of ZnO indicate that this scaffold has good high porosity and can be used for cell adhesion. This study has good properties that transform the synthetic polymer from hydrophilic to hydrophobic [32].

5. Conclusion

Our research leads us to the conclusion that pure PCL, an electrically insulating polymer, has been used to create nanofibers. Nanoparticles were added to this scaffold to graft different concentrations of ZnO 2wt.% and 3wt.%. We see that the surface shape of the fibers and their diameters differ. The PCL reaction was also explained, and this was also supported by FTIR analysis. Nanoparticles are used. The X-ray spectra of the PCL/ZnO hybrid nanofibers were also investigated, which revealed low crystalline phases of the polymer and soft nature after mixing ZnO. Through testing the mechanical properties and tensile strength, we found that a 3% concentration of nanoparticles has a high surface roughness, high tensile strength, and good wettability by testing the water contact angle. This leads us to the conclusion that nanofibers can be used in tissue engineering and treatment of wounds and damage, and that they are particularly strong against bacteria. And its reproduction.

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