

# Morphological and Mechanical Characterization of Crosslinked Electrospun PVA and PEO Nanofibrous Mats Incorporating Gelatin and TiO<sub>2</sub>

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## Research Article

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# **Morphological and Mechanical Characterization of Crosslinked Electrospun PVA and PEO Nanofibrous Mats Incorporating Gelatin and TiO<sub>2</sub>**

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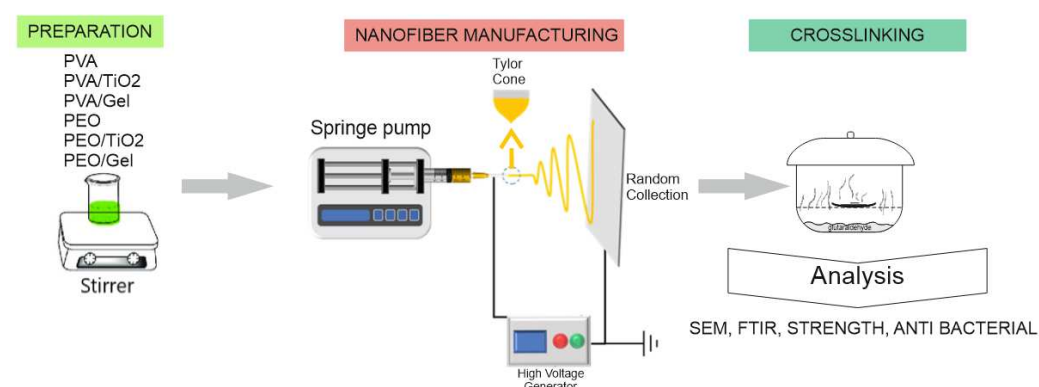
## **Abstract**

Research on the use of nanofiber surfaces produced by electrospinning in medical textiles has been a noteworthy field in recent years. Biodegradable mats are particularly used in the medical field for applications such as wound dressings, selective separation, immobilization of biologically or pharmacologically active agents and molecules, and as scaffolds for tissue engineering. In this study, the antibacterial and mechanical properties of two different polymer types (PVA and PEO) obtained with and without the use of TiO<sub>2</sub> and Gelatin were examined. Six different nanofiber surfaces/mats were obtained and these surfaces; SEM, FTIR, strength and antibacterial analyzes were performed.

It was determined that the nanofiber thicknesses of the obtained surfaces were homogeneous and on average the thickest fibers were measured in the PVA/TiO<sub>2</sub> sample. Chemical analyses with FT-IR showed characteristic bonds of polymers and the presence of gelatin. Crucially, while glutaraldehyde crosslinking proved ineffective for PEO polymers due to the absence of reactive functional groups, it demonstrated a profound synergistic effect in PVA/TiO<sub>2</sub> nanocomposites, resulting in an exceptional 221% increase in tensile strength.

**Keywords** Nanofibers · Electrospinning · Crosslinking · Nanopowders

## Graphical Abstract



## 1 Introduction

Nanofiber surfaces are structures consisting of very fine fibers (usually 1–1000 nm in diameter), have a high surface area and exhibit special physical/chemical properties. Thanks to these features, they are used in many different areas such as filtration-purification [1], biomedical applications [2-4], textile and wearable technologies [5, 6].

It has been noted that nanofibers are useful in many fields due to their high surface areas. The versatile performance of nanofiber surfaces is determined by the selection, control, and optimization of nanofiber properties during the manufacturing process [7].

The most used method, which is one of the production methods of nanofiber surfaces, is the electrospinning method. Electrospinning mainly consisting of a syringe, a high voltage supply, and a collector. To date, hundreds of materials have been converted into nanosurfaces/nanoproducts by electrospinning. The source of the great power is high voltage and, surface tension and electrostatic force are believed to be the two main factors that maintain the shape of the yarn [8]. In 1964, Geoffrey Taylor was the first to describe the dynamics of an electrically charged jet flow. He discovered that as the electrical voltage increased, separate droplets merged to form a long filament [9]. This discovery has facilitated the development of the electrospinning method.

Polyvinyl alcohol (PVA) is a type of polymer commonly used in the production of nanosurfaces. It can also be modified using natural materials for the production of nanofibers that can be used in the biomedical field. PVA's solution can be prepared with water and is an environmentally friendly polymer type [10]. PVA is soluble in polar solvents and this solubility depends largely on the degree of hydrolysis,

that is, the mole percentage of OH<sup>-</sup> groups [11]. In studies, the antibacterial activities of nanofiber surfaces obtained using PVA polymers and active ingredients have been tested [12-18]. The antibacterial properties of nanofibres produced using PVA/Ag nanowires (PVA/Ag NWs) have been investigated. It was observed that the resulting Ag NWs/PVA nanofibers exhibited superior antimicrobial activities against both gram-negative (*E. coli*) and gram-positive (*S. aureus*) bacteria, especially being more sensitive to *S. aureus*. It was concluded that these hybrid nanofibers hold potential for biomaterial applications such as wound dressing [14]. In the study conducted by Khaleel et al., ZnO/PVA-PVP nanofibers were obtained and the characterization and antimicrobial effects of these nanofibrous surfaces were examined. One of the key results of the research is that the addition of zinc oxide (ZnO) nanoparticles enhances antimicrobial efficacy, particularly against the bacterium gram-negative *Escherichia coli* [17].

Polyethylene oxide (PEO) is one of the few biodegradable synthetic polymers permitted for use as a raw material in the production of food, cosmetics, personal care products, and pharmaceuticals [19]. Therefore, water-soluble PEO polymers have an important place in the production of biodegradable nanofibers [20]. Studies have been conducted evaluating the antimicrobial efficacy of PEO polymers and in general, positive results have been obtained against antimicrobial effects as a result of the studies [19, 21-25]. Lin et al. modified the PEO solution by adding carboxymethylated and natural dandelion polysaccharide and evaluated its antimicrobial effects. As a result of the study, it was observed that PEO nanofibers containing dandelion were effective against *L. monocytogenes* bacteria, but the highest antimicrobial effect was in PEO nanofibers containing carboxymethylated dandelion. It was concluded that the use of these mats in food packaging has potential [21]. In a study, Amiri et al., investigated the development of teicoplanin-loaded chitosan-PEO nanofibres. The researchers observed that the resulting nanofibers were able to sustain teicoplanin for up to 12 days and increase antibacterial activity against *Staphylococcus aureus* by 1.5 to 2 times [23].

The conversion of gelatin and gelatin-blended polymers into nanofibers by electrospinning has led to revolutionary developments in the biomedical subject, especially in the production of advanced scaffolds in drug delivery and regenerative medicine. Gelatin nanofiber membranes (GNF) exhibit high

porosity, large surface area, and biocompatibility properties [26]. Huang et al. (2024) produced gelatin (GA)-based nanofiber membranes by electrospinning; developed a multifunctional food packaging material by loading purple potato anthocyanin (PPA) and syringic acid (SA) into these nanofibers. FTIR analyses revealed that hydrogen bonding and electrostatic interactions occur between SA and gelatin chains. The prepared nanofibers exhibited strong antimicrobial action against *E. coli* and *S. aureus* bacteria [27]. Wang et al. (2019) produced polysaccharide composite nanofibers with gelatin and pullulan protein by electrospinning. In the study, it was determined that solution viscosity, surface tension and electrical conductivity increased with the increase in gelatin content. SEM analyses revealed that fiber diameter is primarily dependent on solution concentration. With the increase in concentration, the fiber diameter also increased. However, with the increase in gelatin, which has polyelectrolyte properties, conductivity increased and a decreasing trend was observed in fiber diameters. Tensile strength increased in proportion to gelatin content, while elongation at break decreased. Electro-spun gelatin/pullulan nanofiber membranes have been considered as a promising candidate for tissue engineering scaffold applications because they can imitate the extra cellular matrix (ECM) both structurally and compositionally [28].

TiO<sub>2</sub> consists of three different phases: anatase, rutile, and brookite. While these different structures of TiO<sub>2</sub> affect its physicochemical properties, they also change its application areas [29]. TiO<sub>2</sub> nanoparticles are also frequently used in the production of nanofibers. With the use of these particles, the properties of nanosurfaces such as photocatalytic activities [29], antimicrobial effects [30, 31], and UV protection [32] effects change. In their study, Yerli Soylu et al. succeeded in significantly increasing the photocatalytic performance and antibacterial effect of the material by supporting the titanium dioxide structure with five different metal oxides. It was observed that the obtained surfaces exhibited a high disinfection capacity against microorganisms such as *E. coli* and *S. Aureus* [31]. Ryu et al. examined the effects of the position of silver-doped titanium dioxide (Ag-TiO<sub>2</sub>) nanoparticles on polymeric nanofibers on photocatalytic and antimicrobial performance. The researchers prepared two different composite structures in which particles are embedded in fibers or placed on their surface by electrospinning and electrospraying methods. According to the results obtained, it has been proven that

nanoparticles placed on the surface give much superior results in self-cleaning and preventing bacterial growth under visible light [33]. TiO<sub>2</sub> nanoparticles were selected due to their well-established antibacterial properties and their ability to improve the functionality of electrospun nanofibrous structures. Previous studies have demonstrated that TiO<sub>2</sub>-incorporated electrospun PVA nanofibers exhibit significant antibacterial activity against both *Escherichia coli* and *Staphylococcus aureus* [34]. These studies with gelatin and TiO<sub>2</sub> show us the antibacterial properties of these two products. Therefore, the use of these two agents was preferred in this study.

Rather than broadly comparing conventional formulations, this study focuses on how the selected polymer matrices, functional additives, and crosslinking agent work together. Specifically, the present work reveals the profound reinforcing effect of glutaraldehyde crosslinking on the mechanical strength of PVA/TiO<sub>2</sub> nanocomposites—yielding an exceptional 221% increase in tensile strength compared to uncrosslinked samples. Additionally, it systematically evaluates how solution-specific properties and electrospinning parameters act synergistically to govern the uniformity of fiber diameter distributions (CV values) across different structural formulations.

The aim of this study is to fabricate nanofibrous surfaces based on polyvinyl alcohol (PVA) and polyethylene oxide (PEO) via the electrospinning method, and to comprehensively investigate the structural, mechanical, and biological properties of these surfaces resulting from the incorporation of gelatin as a biopolymer additive and TiO<sub>2</sub> nanoparticles as an inorganic additive. In this study, the morphological characteristics (SEM), chemical structures (FTIR), mechanical properties, and antimicrobial activities of nanofibrous mats prepared using PVA, PEO, PVA–gelatin, PVA–TiO<sub>2</sub>, PEO–gelatin, and PEO–TiO<sub>2</sub> were evaluated, with the aim of identifying the optimal formulation suitable for biomedical and protective textile applications. The originality of this study lies in the systematic comparison of different polymer–biopolymer–nanoparticle combinations prepared using the same methodology, with particular emphasis on the simultaneous evaluation of mechanical performance and antimicrobial activity. This study; It comprehensively reveals the morphological, structural, mechanical and biological effects of combinations of common synthetic polymers such as PVA and PEO with gelatin

biopolymer and TiO<sub>2</sub> nanoparticles. Furthermore, to better understand the role of hydroxyl and amine groups during the glutaraldehyde vapor crosslinking process, PEO-based nanofibers—which lack reactive functional groups for this specific crosslinker—were intentionally subjected to the same crosslinking procedure. In this study, PEO serves as a structural negative control matrix to comparatively elucidate how the crosslinking mechanism strictly depends on the polymer chemistry and how it dictates the final mechanical performance of the mats.

## **2 Experimental**

### **2.1 Materials**

PVA, PEO polymers, glutaraldehyde, gelatin and TiO<sub>2</sub> were supplied from Sigma Aldrich. The molecular weight of PVA (Cas no: 9002-89-5) is in the range of 89,000-98,000, and the molecular weight of PEO (Cas no: 25322-68-3) is 100.000 g/mol. Gelatin (Cas no: 9000-70-8) is obtained from swine skin and has a bloom value of ~175. Glacial acetic acid (99.85%) is used and supplied from Apcpure company. TiO<sub>2</sub> powders (CAS 13463-67-7) have an average particle size of 21 nm. Glutaraldehyde solution (25% in water, Grade II) was obtained from Sigma-Aldrich (Cat. No. G6257) and used as a crosslinking agent without further purification.

### **2.2 Preparation of Solutions, Nanofiber Production and Crosslinking**

PVA and PEO are two types of water-soluble polymers, and in this study, the solutions were prepared with water. PVA solution was prepared at 10% and stirred at 80 °C for 4 hours. PEO solution 10% was prepared and stirred for 24 h at room temperature. The TiO<sub>2</sub> ratio was set at 1% in both polymers. In previous studies, these concentrations have been preferred to enhance the antibacterial functionality of nanofiber formulations [35, 36]. After homogenizing 1% TiO<sub>2</sub> with distilled water in the sonicator bath for half an hour, it was added to PVA and PEO solutions. These solutions to which TiO<sub>2</sub> was added were mixed in a magnetic stirrer for 2 h and homogenized in a sonicator bath for 30 min. Gelatin solution was prepared by stirring 12% (1.2 g gelatin) at a ratio of 1:9 water:glacial acetic acid for 30 minutes. The prepared gelatin solution was mixed with PVA and PEO solutions in a ratio of 8:2 (PVA or PEO:Gelatin).

Once prepared, the solutions were loaded into syringes with a capacity of 5 ml and Spellman brand electrospinning device was used in the production of nanofiber mats. In Table 1, the working conditions of the samples in electrospinning are given.

In a bath containing glutaraldehyde/acetic acid (5/0,25 ml), the samples were kept in a desiccator for 24 hours and cross-linking with steam was performed.

**Table 1.** Working conditions of samples

| Sample Name          | Voltage Rating (kV) | Flow Rate (ml/h) | Collector Distance (cm) |
|----------------------|---------------------|------------------|-------------------------|
| PVA                  | 16.7                | 0.3              | 28                      |
| PVA-TiO <sub>2</sub> | 15.5                | 0.3              | 28                      |
| PVA-Gel              | 20                  | 2                | 15                      |
| PEO                  | 20                  | 2                | 13                      |
| PEO-TiO <sub>2</sub> | 15.5                | 0.3              | 20                      |
| PEO-Gel              | 16                  | 0.5              | 18                      |

## 2.3 Tests and Analyses

SEM and FTIR analyses were carried out at the University of Greater Manchester. SEM images were obtained with Hitachi S-3400N device. SEM analyses were performed to examine the obtained nanosurfaces, visualize the particles, and measure the fineness values of the nanofibers and the average. Cross-sectional measurements of the nanofibers were carried out via the Image J program as 50 of each sample.

All samples were chemically characterized by Fourier-transform infrared (FTIR) spectroscopy on a Thermo Nicolet iS10 model device.

Strength tests were conducted at the Textile Engineering Physical Testing Laboratory of Istanbul Marmara University. Strength measurements were taken using an Instron 4411 model testing machine, while the thickness values of the samples were measured using a Mitutoyo testing machine. Before the tensile tests, each sample was prepared by cutting it into a rectangular shape with a length of 50 mm and a width of 10 mm.

Antibacterial tests were performed using *S. aureus* (gram +, ATCC 25923) and *E. coli* (gram -, ATCC 35218) bacteria at University of Greater Manchester. The samples were cut in an area of 1x1 cm<sup>2</sup> and used in the tests and each sample was analyzed three times.

### 3. Results and Discussion

#### 3.1 SEM Views

SEM images were obtained at magnifications of 5  $\mu\text{m}$  and 50  $\mu\text{m}$  to evaluate the general fiber network structure and local morphological features (Figure 1). Table 2 shows the fiber diameter, coefficient of variation and interpretation of the samples. When the table was examined, it was observed that the most homogeneous fiber diameter distributions were in nanofibers obtained with PEO polymers. This is not only related to the polymer type, but also to the synergistic effect of polymer solution properties and electrospin parameters. Previous studies have shown that viscosity, surface tension, electrical conductivity, voltage, flow rate, and collector distance strongly influence jet stability and fiber diameter uniformity [8, 28].

Among the samples, PVA-TiO<sub>2</sub> exhibited the highest average fiber diameter, whereas pure PVA showed the lowest average diameter. Upon examining Figures 1.b and 1.e, although TiO<sub>2</sub> particles are visible on the PEO/TiO<sub>2</sub> nanosurfaces (Figure 1.e), the presence of particles was not observed on the PVA/TiO<sub>2</sub> nanosurfaces (Figure 1.b). Because PVA polymers can form both polar and hydrogen bonds, TiO<sub>2</sub> particles are thought to be embedded in the nanofiber [37]. For this reason, it was observed that the mean diameter of PVA/TiO<sub>2</sub> nanofibers (mean=509.79 nm) (Table 2) was higher than the others. On the other hand, since PEO, although hydrophilic, does not present as strong hydrogen bonding as PVA [38, 39], it can be interpreted that the particles collect on the fiber surface. In hydrophilic polymer matrices, TiO<sub>2</sub> and similar inorganic nanoparticles are well distributed in the fiber, while in less polar matrices, there is particle aggregation [40]. Furthermore, the apparent increase in average fiber diameter observed for PVA and PVA-TiO<sub>2</sub> produced under nearly identical process conditions (0.3 ml/h flow rate, 28 cm collector distance, and narrow voltage range of 15.5–16.7 kV) can be mainly attributed to the incorporation of TiO<sub>2</sub> nanoparticles.

In contrast, PVA-Gel was electrospun under substantially different conditions (20 kV, 2 ml/h, 15 cm) than pure PVA, involving a markedly higher flow rate and a shorter collector distance. According to previous reports, an increase in flow rate delivers a greater volume of solution to the Taylor cone per unit time, reducing the residence time available for solvent evaporation and polymer chain stretching,

which favors the formation of thicker and less uniform fibers [41, 42]. Likewise, a shorter tip-to-collector distance shortens the whipping region in which the jet undergoes bending instability and elongation, limiting the degree of fiber thinning before deposition [42]. The combination of these two effects would be expected to broaden the fiber diameter distribution independently of any compositional influence of gelatin, which is consistent with the considerably higher coefficient of variation obtained for PVA-Gel (CV = 51.7%) relative to neat PVA (CV = 37.37%). Accordingly, the broad size distribution of PVA-Gel nanofibers should not be attributed to gelatin incorporation alone, but rather to the combined effect of the higher flow rate and shorter collector distance employed for this formulation.

A similar parameter-dependent pattern is evident among the PEO-based samples, which were also produced under different voltage, flow rate, and collector distance combinations (Table 1). Notably, PEO nanofibers exhibited the lowest CV (26.4%) among all samples despite being electrospun at the highest flow rate (2 ml/h) within the PEO group, a condition that would normally be expected to favor a wider diameter distribution. This discrepancy suggests that, beyond the individually applied process parameters, the intrinsic solution properties of PEO interact with the spinning conditions in a way that can offset the diameter-broadening effect typically associated with a higher flow rate [41]. Taken together, the differences in fiber diameter and CV across the six formulations cannot be attributed to polymer or additive type alone. They more likely reflect the combined influence of solution-specific properties and the electrospinning parameters applied for each formulation, in agreement with previous studies emphasizing the multi-parametric nature of fiber morphology control in electrospinning [41, 42].

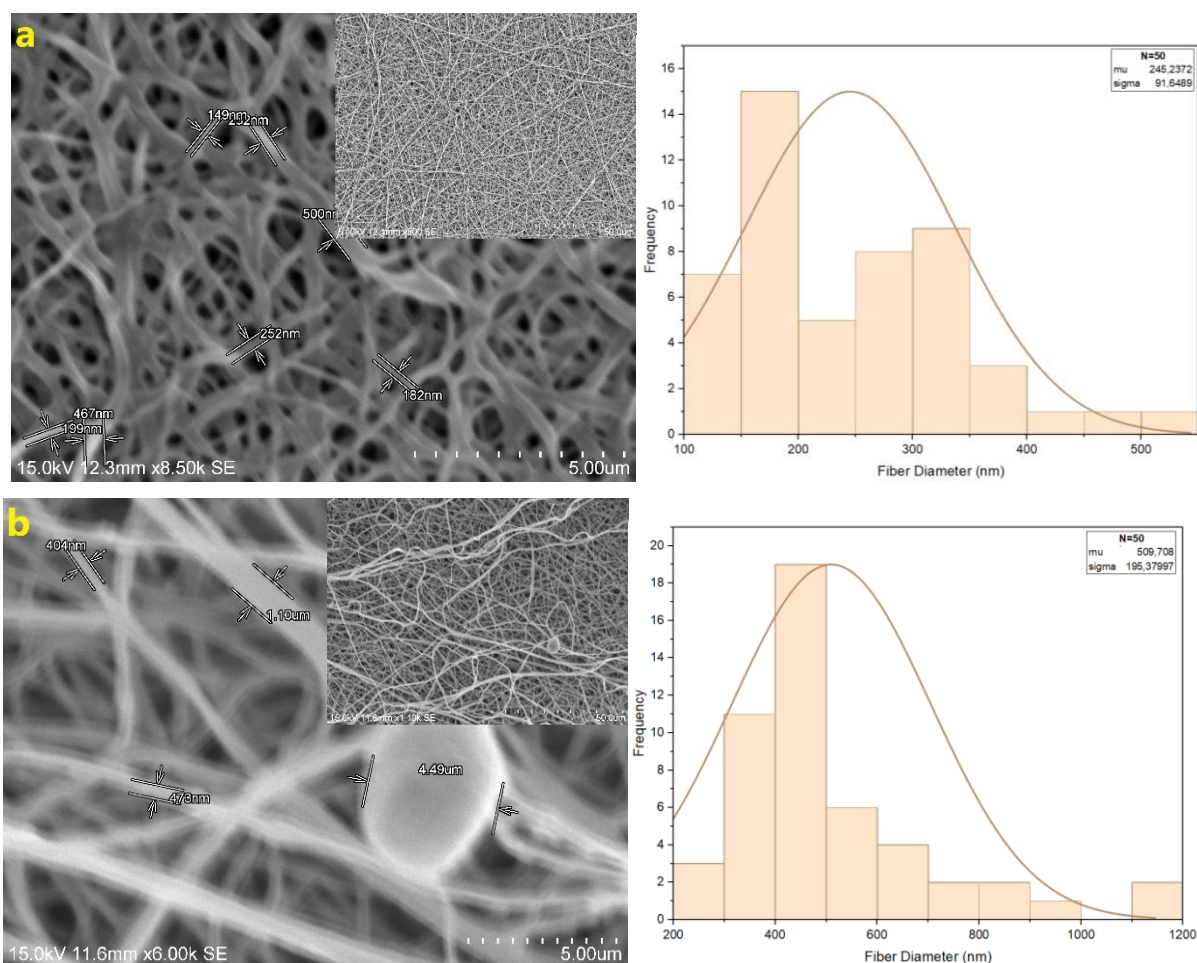
When the SEM images of the gelatin-containing nanofibrous mats (Figures 1c and 1f) were examined, no bead-like defects or gelatin-rich aggregates were observed on the fiber surfaces. This relatively uniform morphology may be attributed to the good compatibility of gelatin with the polymer matrices and possible hydrogen-bond interactions, as previously reported for electrospun gelatin-based composite nanofibers [28].

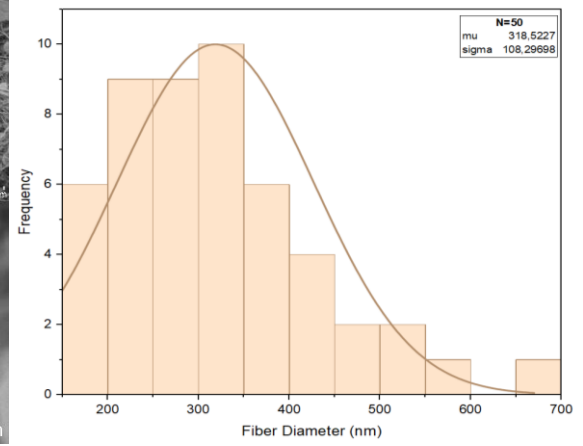
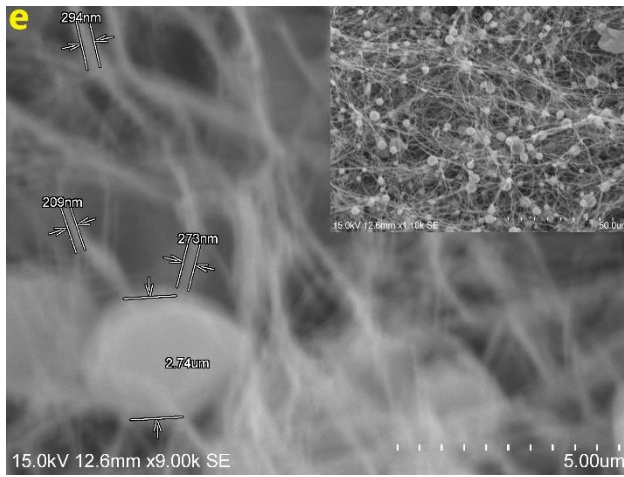
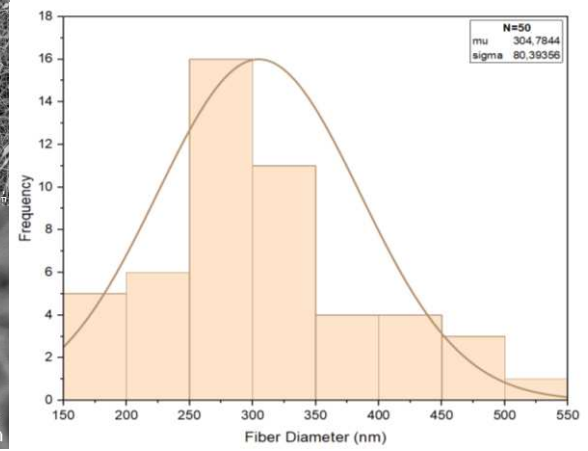
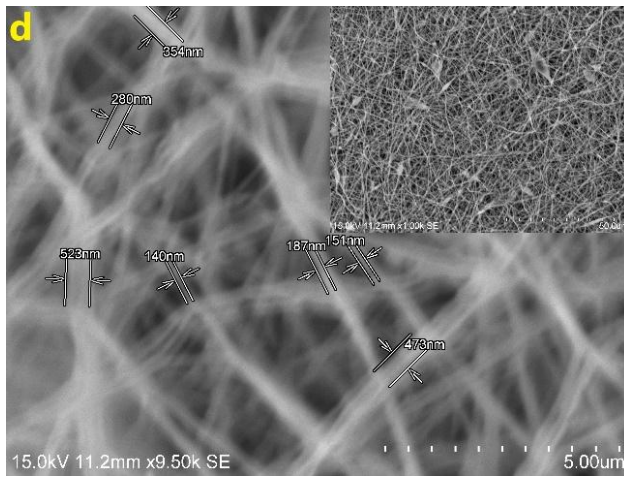
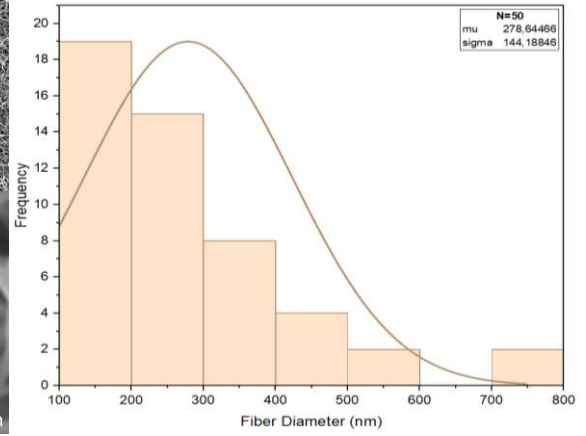
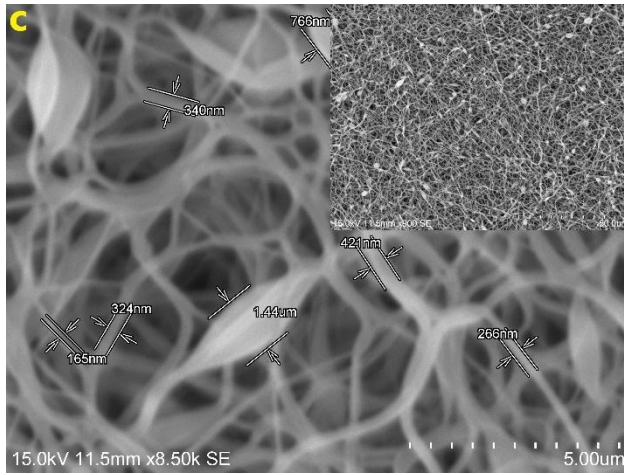
With the exception of PVA-Gel nanofibers, the average fiber diameters were generally measured consistently. This indicates that there was a good balance between solution viscosity and surface tension during production [28].

**Table 2.** Values of fiber diameters, CV % and Interpretation of the samples measured with

Image J.

| Sample               | Mean fiber diameter $\pm$ SD<br>(nm) | CV (%) | Interpretation            |
|----------------------|--------------------------------------|--------|---------------------------|
| PVA                  | 245.24 $\pm$ 91.65                   | 37.37  | Lowest mean diameter      |
| PVA-TiO <sub>2</sub> | 509.79 $\pm$ 195.38                  | 38.3   | Thickest fibers           |
| PVA-Gel              | 278.65 $\pm$ 144.19                  | 51.7   | Broadest distribution     |
| PEO                  | 304.78 $\pm$ 80.36                   | 26.4   | Most homogeneous          |
| PEO-TiO <sub>2</sub> | 318.62 $\pm$ 106.27                  | 33.4   | Moderate variation        |
| PEO-Gel              | 339.03 $\pm$ 123.37                  | 36.4   | Slightly thicker than PEO |





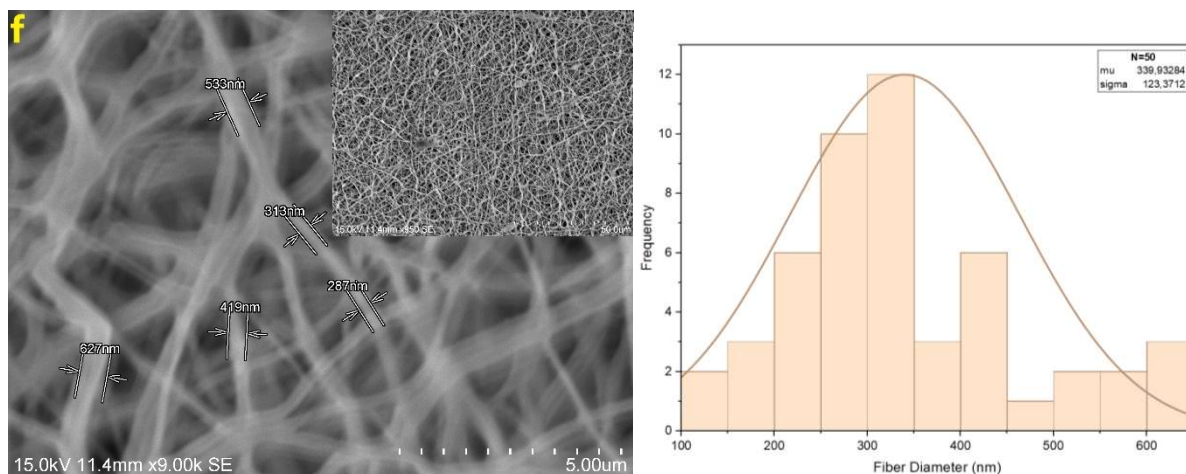


**Figure 1.** SEM images of the produced surfaces and distribution of fiber fineness. a: PVA, b: PVA-TiO<sub>2</sub>, c. PVA-Gel, d. PEO, e. PEO-TiO<sub>2</sub>, f. PEO-Gel

### 3.2 FT-IR Analysis

FT-IR analyses of the obtained nanosurfaces are given in Figures 2 and 3. When Figure 2 (PVA) is examined, the asymmetric peaks at approximately  $3300\text{ cm}^{-1}$  show the polymeric association of the free hydroxyl groups and the bound -OH stress vibration [43, 44]. We can comment peaks that in the range of  $2910$  and  $2940\text{ cm}^{-1}$  to existing CH bands in PVA [45]. The  $1426\text{ cm}^{-1}$  band also refers to the CH<sub>2</sub> bond [46]. PVA/gelatin nanofibers, distinct peaks observed at  $1653\text{ cm}^{-1}$  and  $1550\text{ cm}^{-1}$  correspond to the Amide I (C=O stretching) and Amide II (N-H bending and C-N stretching) vibrations, respectively [47]. These peaks confirm the presence of gelatin's peptide bonds, which are absent in pure PVA and PVA/TiO<sub>2</sub> nanofibers.

When the FT-IR spectrum of PEO was examined (Figure 3), a peak was observed at  $2883\text{ cm}^{-1}$  (CH<sub>2</sub> stretching) in all three samples [48]. The peak around  $1467\text{ cm}^{-1}$  corresponds to the bending vibration of CH<sub>2</sub> groups. The strong band observed at around  $1096\text{ cm}^{-1}$  corresponds to the C-O-C stretching vibration of PEO [49]. Together with the other characteristic bands in the  $840\text{--}1340\text{ cm}^{-1}$  region, this band ( $1096\text{ cm}^{-1}$ ) supports the semicrystalline helical conformation of PEO [50]. In the PEO-Gel nanofibers, new bands appear in the range of  $1650\text{--}1550\text{ cm}^{-1}$ , compared to PEO and PEO-TiO<sub>2</sub>. These bands originate from the amide I and amide II vibrations of gelatin, suggesting the formation of hydrogen bonds between PEO and gelatin [47]. However, this band is weaker than PVA-Gel nanofibers.

This weaker band is consistent with the comparatively lower hydrogen-bonding capacity of PEO relative to PVA proposed in the SEM discussion (Section 3.1), where TiO<sub>2</sub> particles were similarly observed to reside on the PEO fiber surface rather than becoming embedded.

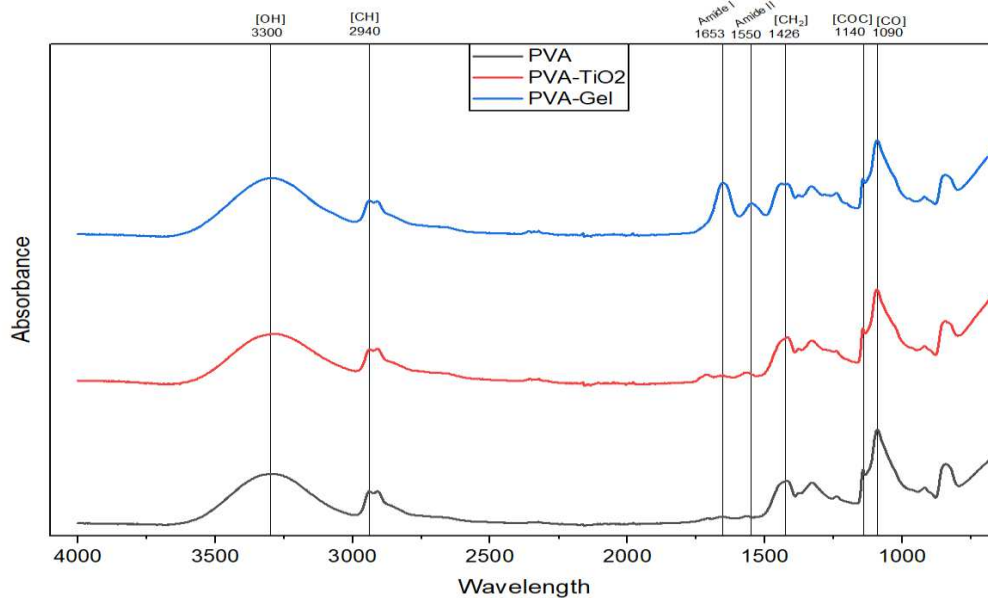


**Figure 2.** FT-IR graph of PVA surfaces

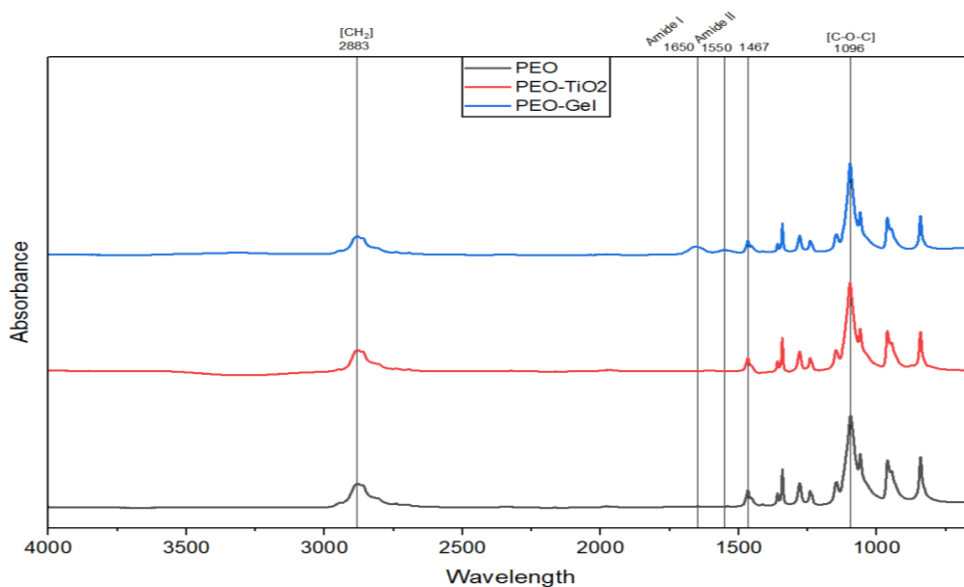


**Figure 3.** FT-IR graph of PEO surfaces

### 3.3 Strength Tests

In Figure 4, strength tests of the cross-linked and non-cross-linked values of the nanosurfaces are given. It was observed that the tensile strength increased after cross-linking of PVA nanofibers. The increase in pure PVA samples was 5%, PVA-TiO<sub>2</sub> samples increased by 221% and PVA-Gel samples

increased by 3%. Notably, the reinforcing effect of cross-linking was substantially more pronounced in PVA-TiO<sub>2</sub> (an increase of approximately 221%, from 1.28 to 4.12 MPa) than in pure PVA (~5%). This synergistic effect can be attributed to TiO<sub>2</sub> nanoparticles providing additional interaction sites with PVA hydroxyl groups, which restrict polymer chain mobility and increase interfacial bonding force within the crosslinked network, consistent with previous reports on PVA/TiO<sub>2</sub> nanocomposites [51]. In previous PVA-Gelatin nanofiber studies, it was observed that when cross-linking with glutaraldehyde, it increased the strength significantly [52, 53]. However, this effect was not seen in this study. This limited rise may be related to the considerably broader fiber diameter distribution of PVA-Gel (CV = 51.7%, Section 3.1), where structural heterogeneity and morphological defects arising from the electrospinning parameters may decrease the mechanical benefit of chemical cross-linking. At the same time, the cross-linking results obtained by Sengor et al. for PVA/gelatin nanosurfaces using glutaraldehyde vapor yielded values close to those reported in the present study [53]. Therefore, the observed strength values (~3%) for PVA-Gel in this study are not unprecedented.

On the other hand, the tensile strength of PEO nano surfaces decreased slightly after cross-linking. Glutaraldehyde is a chemical that has the ability to cross-link with amine (-NH<sub>2</sub>) and hydroxyl (-OH) groups [54] however, since PEO polymers do not contain these groups (see Figure 3), it can be said that cross-linking does not occur between them. Since PVA polymers contain hydroxyl (-OH) groups, it can be said that the tensile strength of cross-linked surfaces increase. Briefly, it can be said that the tensile strength of PEO are reduced or not affected due to the incompatibility between the crosslinker and the polymer.

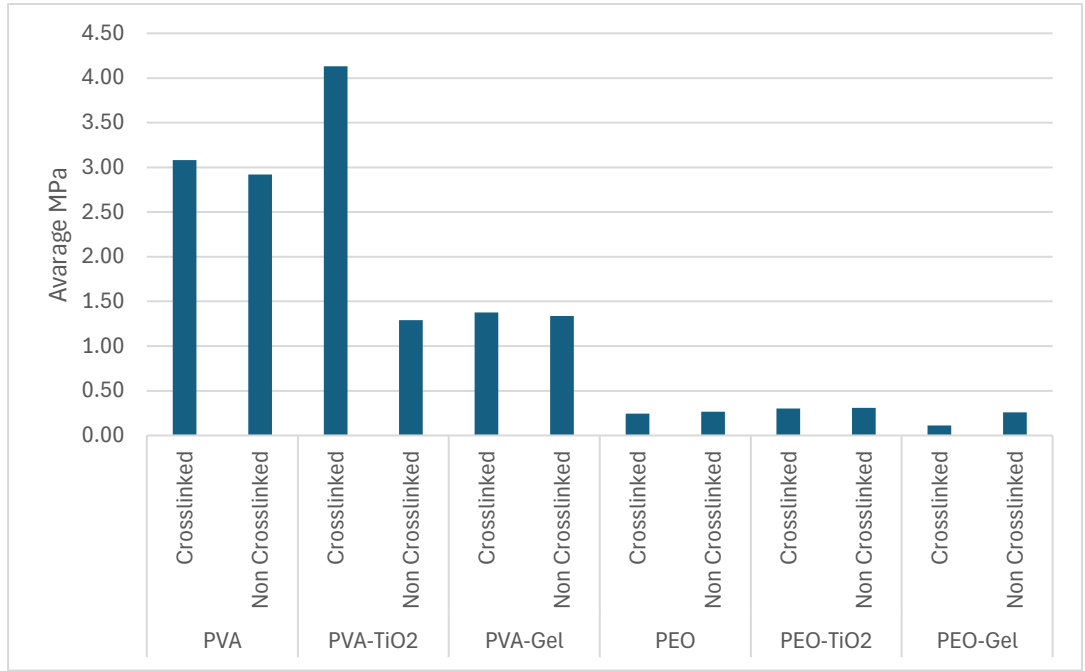


**Figure 4.** Strength bar graph of cross-linked and unlinked samples.

### 3.4 Evaluation of Diffusion-Based Antibacterial Release

The antimicrobial effects of the samples are shown in Figure 5. As a result of the observation, no inhibition zone was observed in any of the petri dishes. This result indicates that no antibacterial agent capable of diffusing from the polymer matrix into the surrounding agar medium is released. No diffusion-based antibacterial activity was observed under the applied test conditions.

The absence of inhibition zones suggests that any potential antibacterial effect of TiO<sub>2</sub> or gelatin was likely suppressed due to cross-link-induced surface passivation. Therefore, the antibacterial behavior of the nanofibers, if present, is expected to be contact-dependent rather than diffusion-based [55]. Since the high cross-linking density can slow down the diffusion of agents and reduce the release [56], it can be said that the antibacterial effects of TiO<sub>2</sub> and gelatin are restricted.

The findings obtained in this study differ from previous studies reporting antibacterial activity in electrospun nanofibers containing TiO<sub>2</sub>. Considering that TiO<sub>2</sub> particles are activated under appropriate photocatalytic conditions [30, 31, 33, 34], the absence of antibacterial activity observed in this study may be attributed to the lack of such activation conditions. In the present study, the lack of inhibition zones may therefore be associated with the immobilization of TiO<sub>2</sub> and gelatin within the crosslinked

nanofibrous structure, insufficient diffusion of active components into the agar medium, and the possibility that any antibacterial effect, if present, is contact-dependent rather than release-based.

Therefore, further quantitative antibacterial tests, such as bacterial reduction assays, colony-counting methods, or contact-killing tests, are recommended to better evaluate the antibacterial potential of these nanofibrous mats.

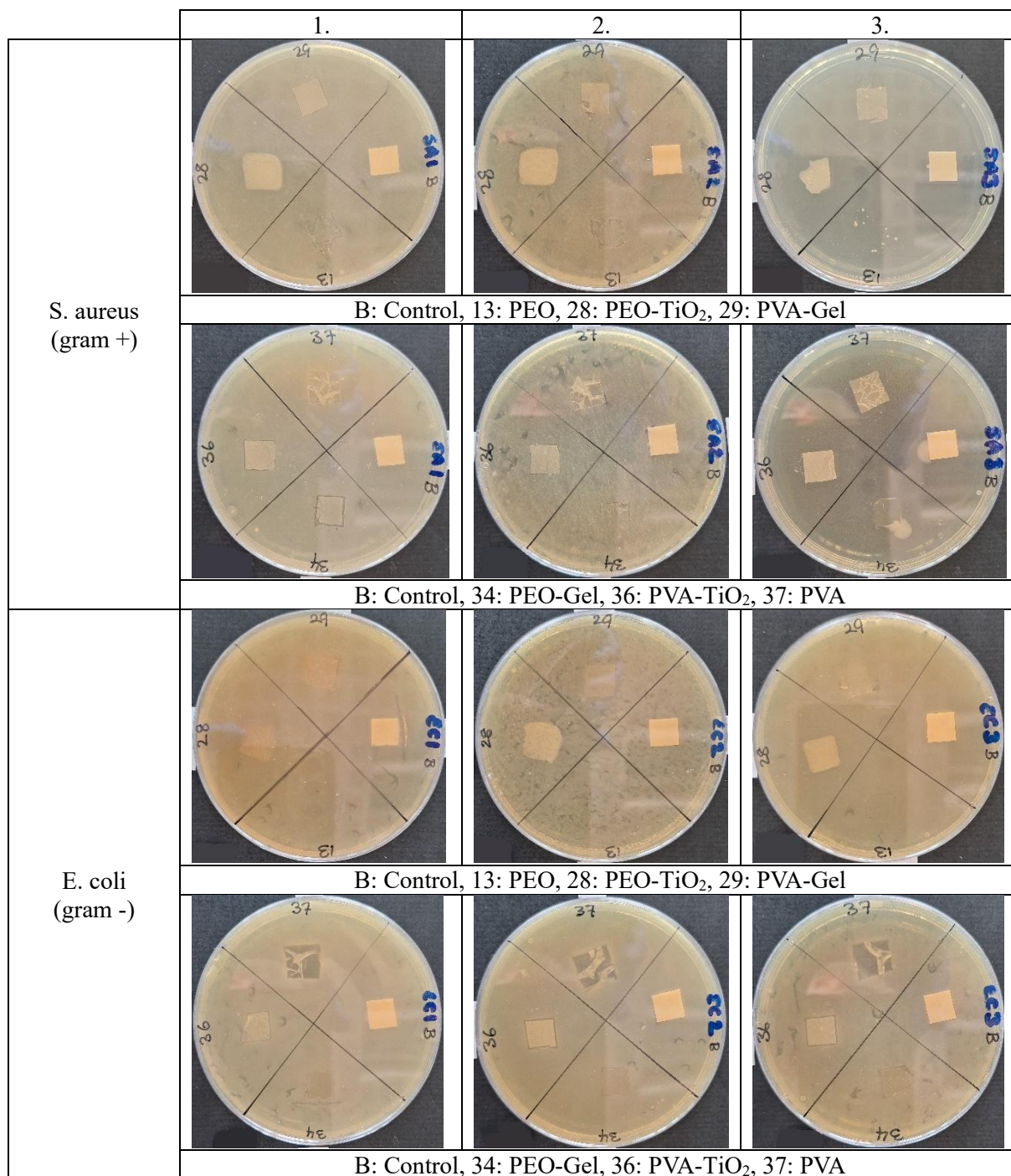


**Figure 5.** Antibacterial test images of crosslinked PVA- and PEO-based nanofibrous mats against *E. coli* and *S. aureus*.

### 3 Conclusion

In this study, the physical and antibacterial effects of nanofibrous surfaces obtained by using TiO<sub>2</sub> and gelatin agents and two different polymers were examined. The results of the analysis are listed below:

- When SEM images were examined, it was observed that TiO<sub>2</sub> particles were embedded in PVA nanofibers and adhered to the surface on surfaces obtained with PEO polymers. It has been observed that gelatin is embedded in both polymers. The measured fiber diameters are consistent across the surface. In general, it can be said that the fiber diameters of PEO surfaces are more homogeneous than PVA surfaces.

- FT-IR analysis confirmed successful incorporation of gelatin into both PVA and PEO nanofibers, evidenced by the appearance of characteristic Amide I and Amide II bands that are absent in the neat polymers. These peptide-bond signals, together with shifts in the polymer functional groups, indicate that gelatin is not aggregated but molecularly integrated within the fibre matrices through hydrogen-bond interactions.

- After 24 hours of exposure between glutaraldehyde vapor and polymers, whilst the strength of PVA has increased, that of PEO has decreased. It can be concluded that the reason for this is that glutaraldehyde vapour is not suitable for PEO polymers.

- In antibacterial analyses, no inhibition zone was formed in any of the samples. Due to the surface passivation caused by the cross-linking process, it was concluded that the antibacterial effect of TiO<sub>2</sub> and gelatin was suppressed. It is also concluded that there must be a photocatalytic effect to improve the antibacterial effect of nanofibers containing TiO<sub>2</sub> nanoparticles.

Based on the data obtained from this study, it can be concluded that antimicrobial activity can be achieved for both polymers (PVA and PEO) by selecting appropriate cross-linking agents or methods.

### Conflict of Interest

The authors declare that they have no conflicts of interest regarding this publication.

## Data Availability Statement

The data supporting the findings of this study, including SEM images, fiber diameter measurements, FTIR spectra, tensile test results, and antibacterial assay records, are available from the corresponding author upon reasonable request.

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