



## RESEARCH ARTICLE

## Coating of Orthodontic Archwires by Zein Based MgO Nanofibers Using Electrospinning System

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**ABSTRACT**

Fixed orthodontic appliances might negatively influence oral health by promoting the formation of demineralizing lesions and instigating gingival issues. This research is designed to coat orthodontic archwires with Zein-based magnesium oxide nanoparticles (Zein-MgO NPs), and evaluate the mechanical, physical, and antibacterial properties of the coated wires. A novel coating process, Electrospinning Deposition, was employed to coat rectangular stainless steel and nickel titanium orthodontic arch wires (n=72) with Zein-based MgO nanoparticles. The wires were first cleaned in an ultrasonic bath before being coated. The properties of the coated wires were characterized using Field Emission Scanning Electron Microscopy (FeSEM) with Energy Dispersive X-ray Spectroscopy (EDS), X-ray diffraction (XRD), and Atomic Force Microscopy (AFM). The FeSEM, EDS, and AFM analyses of the coated wires validated the presence of Zein-based MgO nanoparticles on the coated surface, potentially suggesting their antibacterial potential. Further, the Electro Spray Deposition method showed to be effective, with optimized parameters: distance 5 cm, voltage 10-12 kV, needle gauge 21 cm, and a flow rate of 1.5 ml/hour. It was demonstrated that stainless steel and nickel titanium orthodontic wires can be successfully coated with Zein-MgO Nfs using the Electro spray Deposition method. This coating could potentially enhance the physical and mechanical properties of the wires, as well as their antibacterial properties. Further research is required to evaluate the long-term effects and benefits of this novel coating.

**INTRODUCTION**

The main goal of orthodontic therapy is to improve the masticatory system's performance while also considerably enhancing the patient's smile's aesthetic appeal[1]. However, orthodontics has possible risks, much like many medical procedures. Enamel demineralization, tooth decay, gingivitis, recession, periodontal disease, and root resorption are examples of serious adverse effects[2]. Making every effort to prevent these negative situations is essential. Modern orthodontic treatments often employ a fixed appliance, which consists of brackets that stay in the patient's mouth during the whole procedure and archwires that are changed out at follow-up appointments. Unfortunately, the prevalence of these components results in the expansion of plaque accumulation sites, which in turn increases the surface area available for the development of bacterial biofilm. The adherence of bacterial biofilm is influenced by features including the surface topology and chemical characteristics of orthodontic components[3]. Due to their capacity to produce reactive oxygen species, which interfere with bacterial metabolism and lessen the possibility of bacterial resistance, metal oxide nanoparticles like ZnO, AgO, and TiO have found extensive usage in the

mechanical and biomedical fields[4]. Their employment has been constrained, nonetheless, by worries about biosafety and deteriorating material qualities. A powerful antibacterial agent known as nanoparticulate MgO has recently been added to dental materials to increase their antibacterial effectiveness[5]. However, MgO nanoparticles have a propensity to aggregate and form clusters, which may limit their use in dental settings. Zein polymer can be added to the formulation of MgO nanoparticles as a possible means of preventing MgO particle agglomeration. Zein is a naturally occurring protein polymer that has a wide range of medical uses[6]. By lowering their hydrophobic characteristics, this polymer can stabilise particles to prevent clumping. One technique to improve orthodontic components' qualities, such as the reduction of bacterial biofilm development, is to modify their surface. The inclusion of layers containing substances that may have bactericidal or bacteriostatic properties can be a part of such modifications[7]. Accordingly, parameters such surface roughness, mechanical and frictional qualities, thickness, corrosiveness, coating stability, and bacterial adherence can be influenced by the coatings used on orthodontic wires[8–10]. The correct covering technique must be used if a good coating is to be obtained. There are several coating methods, including thermal evaporation, radiofrequency magnetron sputtering, physical vapour deposition, and the less popular sol-gel thin film dip-coating technique[9]. In this investigation, we used the Electro Spray Deposition Method, a brand-new coating technique[11–12]. This process has several benefits, including high purity of the starting material, great structural uniformity, and control over the conductivity of the final product[13–14]. In contrast to conventional spray drying, the Electro spray Deposition process employs an electrostatic charge to dry the feed solution at a lower temperature[15–17]. As a result of the polar solvent collecting more electrons than the solute when an electronic charge is applied, larger solvent concentrations are present at the surface during droplet formation, which improves drying efficiency at lower temperatures[18–20]. In this pilot work, antimicrobial nanoparticles (Zein-based magnesium oxide nanoparticles and magnesium oxide nanoparticles[21–22]) were coated on orthodontic arch wires to assess their physical, mechanical, and antibacterial capabilities. Some of the negative effects of orthodontic treatment may be reduced or even completely eliminated by creating a coating that adheres flawlessly to the archwire and has antibacterial qualities[22–23].

## **STUDY MATERIALS AND PROCEDURES**

### **Materials Used**

For the investigation, stainless steel samples measuring 0.019 by 0.025 inches and NiTi orthodontic upper archwires were used (provided by Dentaureum GmbH & Co.KG, Ispringen, Germany). Additionally, magnesium oxide powder from Qingdao Hesiway Industrial in China and zein polymer powder from Glentham Life Sciences in the UK were used.

72 orthodontic archwires were used in the experiment, which had a four-group design. Two test groups had SSW and NiTi coated with Zein-based magnesium oxide nanofibers, while the other two groups had no coating at all. Phase I of the investigation involved the analysis of the coating material (MgO and Zein polymer) utilising an X-ray diffractometer (XRD) and Fourier transform infrared spectroscopy (FTIR) (FLUOROMATEFS-SPECTROMETER, USA). Phase II prepare suspensions of materials and evaluate them. Orthodontic archwires were cleaned, coated, and then characterised by FeSEM with EDX.

### **Phase I: Powder Analysis**

#### **Using an XRD-6000**

X-ray powder diffractometer and Cu K radiation, powder were examined. Each measurement was made between 10° and 80° using a complete 2-scan at 5° min<sup>-1</sup>.

#### **FTIR spectra**

FTIR spectra of zein polymer and MgO nanoparticles were obtained. Between 4000 and 400 cm<sup>-1</sup>, the spectra were captured.

### Phase II: Creation and evaluation of material suspensions

Specific amounts of Zein and MgO powders were dissolved in a mixture of ethanol and distilled water, which was then magnetically agitated and sonicated to create the suspensions of Zein and Zein/MgO. The suspensions were analysed using FTIR.

### Phase III: Sample Preparation

Orthodontic stainless steel and NiTi archwire straight segments were cut into 50mm lengths and separated into four groups: two uncoated control groups and two experimental groups coated with MgO and Zein-based MgO. All archwires underwent ultrasonic cleaning in deionized water, followed by air drying.

**Electrospinning Coating:** For roughly an hour, each nanosuspension that had been previously created was electrospun onto the sample. The samples were then kept until the testing day in a sealed petri plate.

## RESULTS

### Powder Analysis:

#### XRD Assessments:

The MgO nanoparticles were investigated utilising an XRD analysis at 20–80° (2 $\theta$ ) using CuK radiation. The (1 1 1), (0 0 2), (2 0 2), (1 1 3), and (2 2 2) planes were represented by the peaks at angles 18.57°, 36.96°, 38.02°, 42.98°, 62.36°, and 74.71°. (JCPDS No. 87-0653). This shows that MgO nanoparticles have formed a polycrystalline cubic structure. No further impurity phases could be seen in the XRD pattern. The material's strong crystallinity was suggested by a prominent (0 0 2) orientation peak. The (0 0 2) reflection was used to calculate the average crystallite size, which was found to be 21 nm.n as shown in Fig( 1).

The zein polymer's XRD patterns showed two distinct peaks at around 20 angles, at around 9 and 21, respectively. These angles were used to calculate the typical d-spacing values, which came out to 9.8 °Å and 4.2 °Å. These exact d-spacing values have a clear explanation based on proven research. The backbone distance within the alpha-helix determines the lower d-spacing (4.2 °Å), but the side-by-side positioning of the alpha-helices determines the longer d-spacing (9.8 °Å). Insight into the degree of alpha-helix packing is provided by the ratio between these two peaks (29,30), as shown in Fig ( 2).

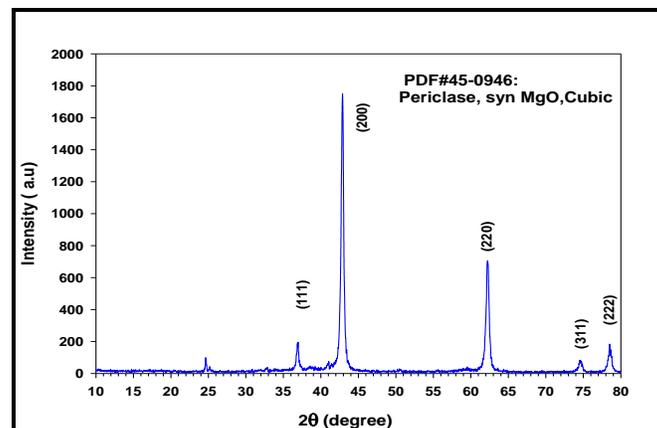
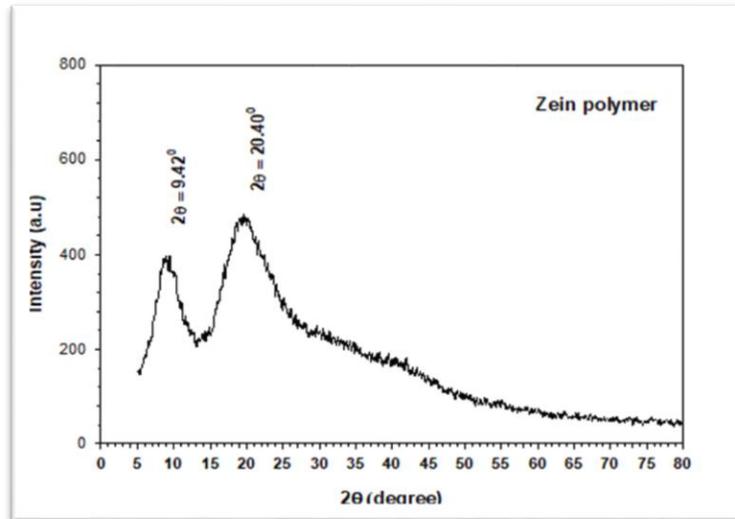


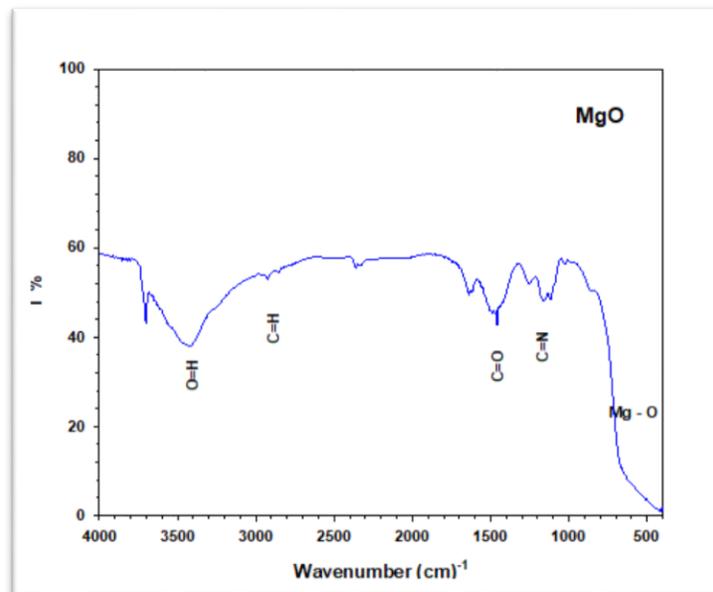
Fig.1. XRD pattern of MgO nanoparticles



**Fig. 2. XRD of Zein polymer**

#### Fourier Transform Infrared Spectroscopy (FTIR) of the Powder:

The MgO nanoparticles' FTIR examination revealed a noticeable band at 3495  $\text{cm}^{-1}$ , which is associated with the (O-H) stretching mode of hydroxyl groups. Because of dampness, these groupings are visible on the surface. The bending vibration of water molecules is the cause of the 1423  $\text{cm}^{-1}$  peak that was noticed. The major signal at 424  $\text{cm}^{-1}$  is an indication that Mg-O vibrations have formed [31, 32]. As shown in Fig (3)



**Fig. 3. FTIR spectrum of MgO nanoparticle**

There are four distinct bands in the FTIR spectrum of -zein (Figure 4) that are typical of proteins. A band known as amide A is seen between 2800 and 3500  $\text{cm}^{-1}$ , and it is caused by the stretching of the N-H and O-H bonds from the protein's amino acids. The stretching of the carbonyl (C=O) of amide groups inside the peptide groups, commonly known as amide I, is responsible for a band that can be seen at 1650  $\text{cm}^{-1}$ . The N-H bond's angular deformation vibrations are represented by the

band designated as amide II, which is situated at 1540  $\text{cm}^{-1}$ . Last but not least, the band at 1230  $\text{cm}^{-1}$  is connected to the C-N bond's axial deformation vibrations [33,34].

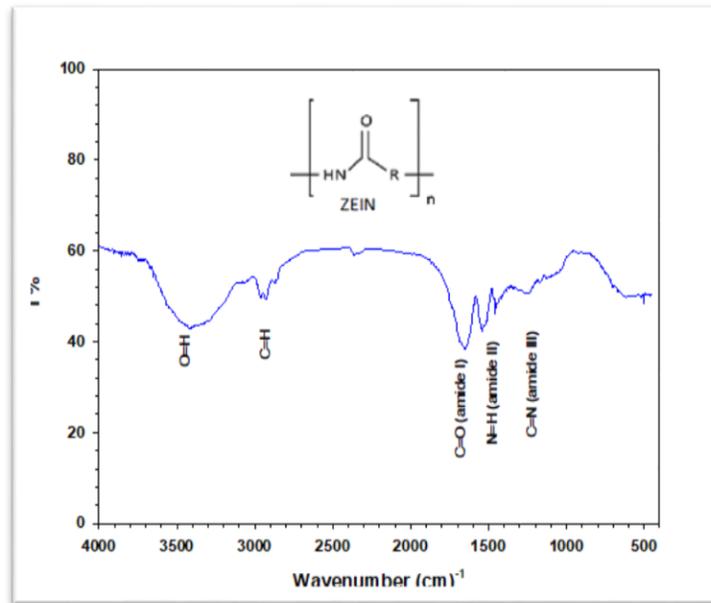


Fig.4. FTIR of Zein polymer

#### Suspension Analysis

**FTIR of Suspension:** The Zein-based MgO nanofiber's spectral data (Figure 4), showed a band at 1652  $\text{cm}^{-1}$  and 2674  $\text{cm}^{-1}$ , which corresponds to the typical asymmetric stretching of C=C and CH<sub>2</sub> groups, respectively. The hydrogen connection between the hydroxyl groups in Zein and MgO was also linked to a band at 3488  $\text{cm}^{-1}$ .

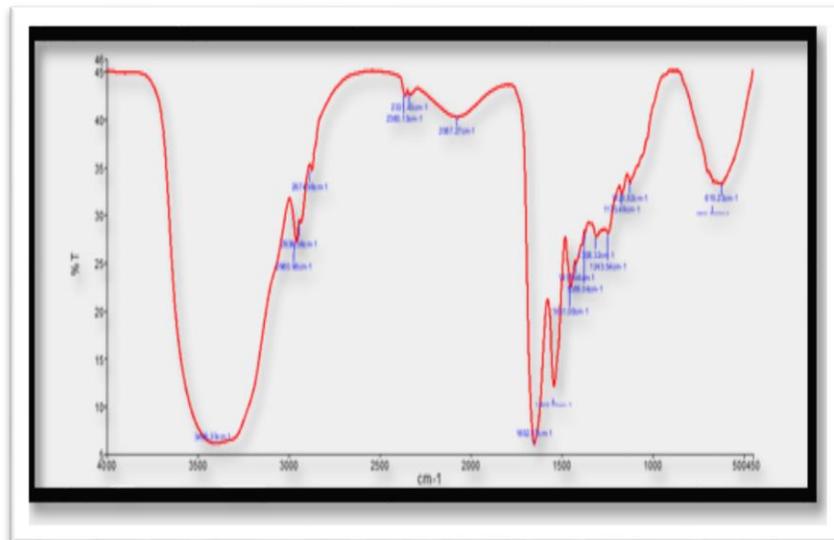


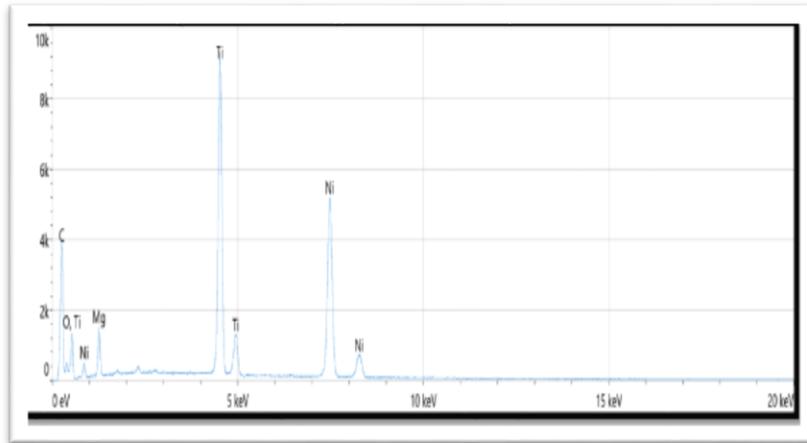
Fig. 5. FTIR OF Zein based mgo suspension

#### Examination of Orthodontic Archwires

#### EDX Spectroscopy Analysis

For all sample groups, including the control and those coated with MgO nanofibers for both SSW and NiTiW, EDX micrographs demonstrated the chemical elemental analysis and ion distribution. In contrast to the control groups, the EDX examination demonstrated the presence of magnesium (Mg) and oxygen (O) atoms on the coated segments of orthodontic archwires for both the SSW and NiTiW groups as shown in figures (6,7). Both materials' concentrations of the **necessary** components decreased as well. This unequivocally proves that magnesium oxide nanofibers.

(a)



(b)

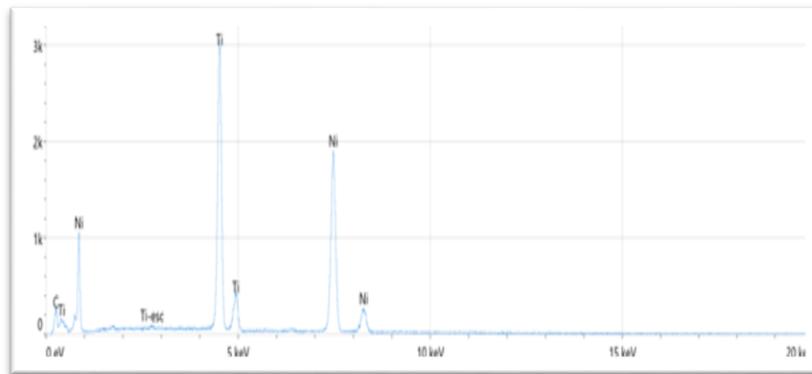
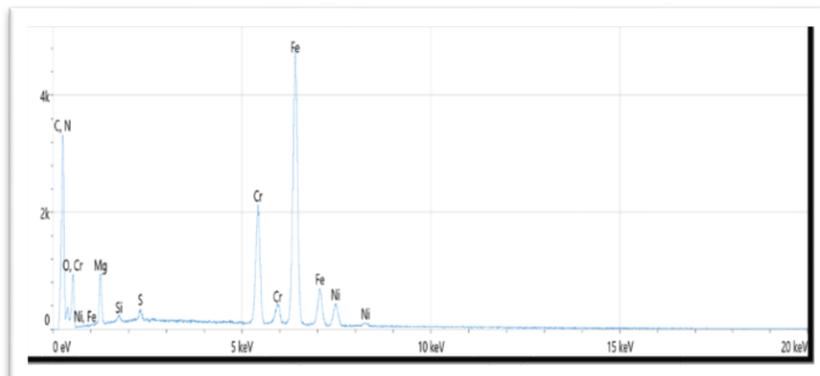


Fig. 6. EDX of NiTi substrate (a) coated (b) uncoated

(a)



(b)

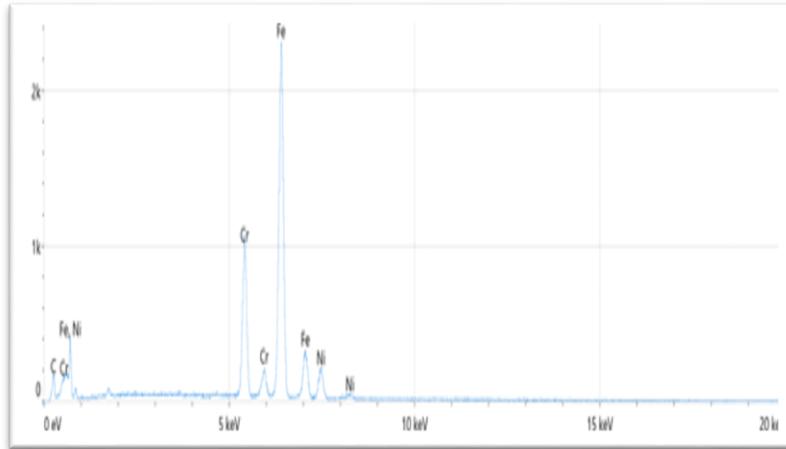
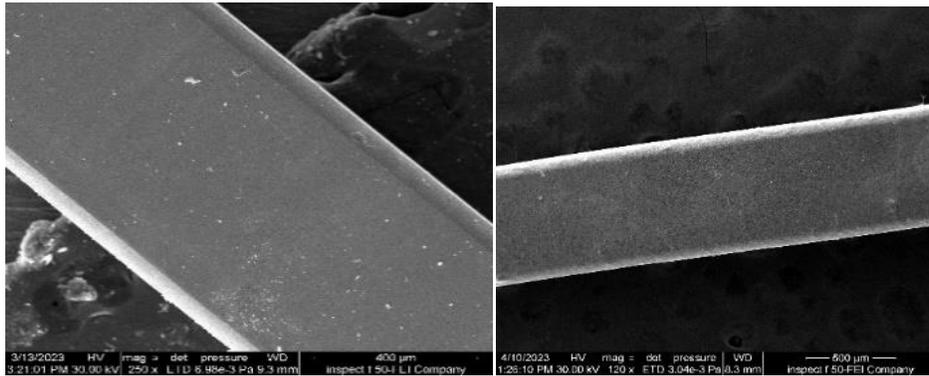


Fig. 7. EDX of SS substrate (a) coated (b) uncoated

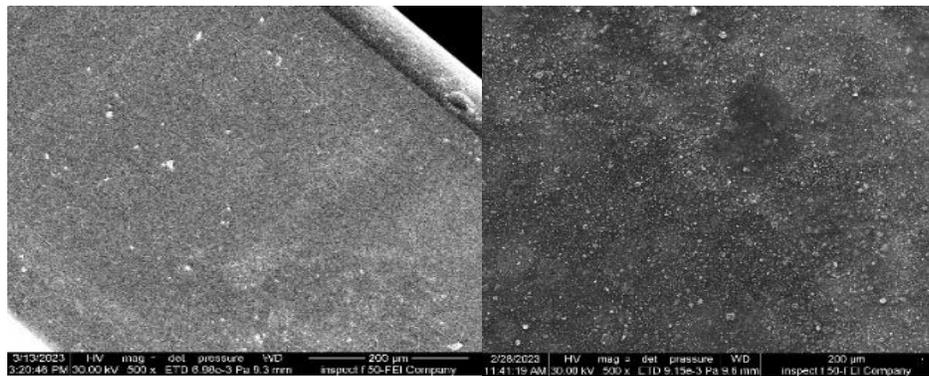
**Characterization by FeSEM**

FeSEM images at various magnifications indicated the difference between the surface topography of the uncoated control archwire segment in comparison to coated ones with Zein\MgO for both SSW and NiTiW groups, as shown in figures ( 8 and 9, respectively)

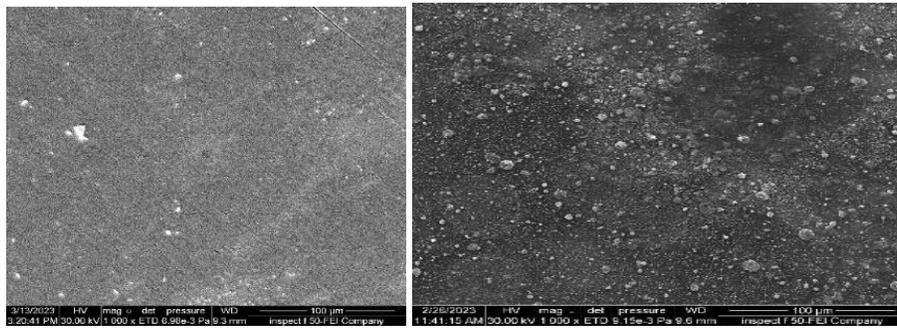
(a)



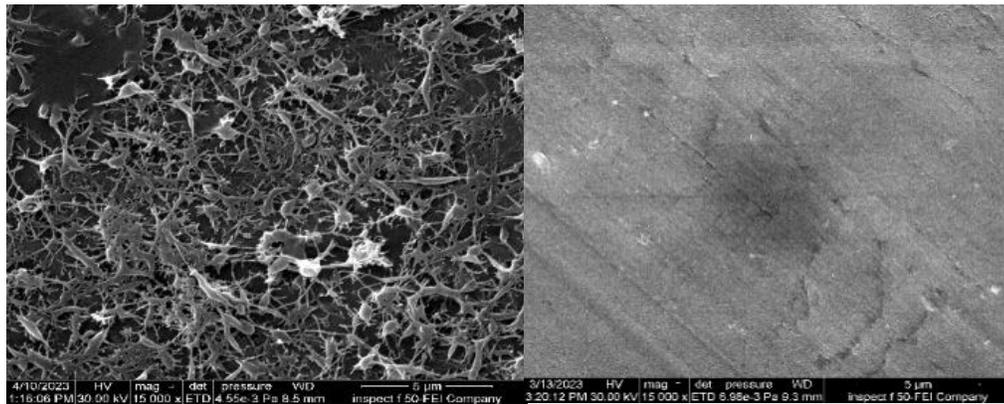
(b)



(c)

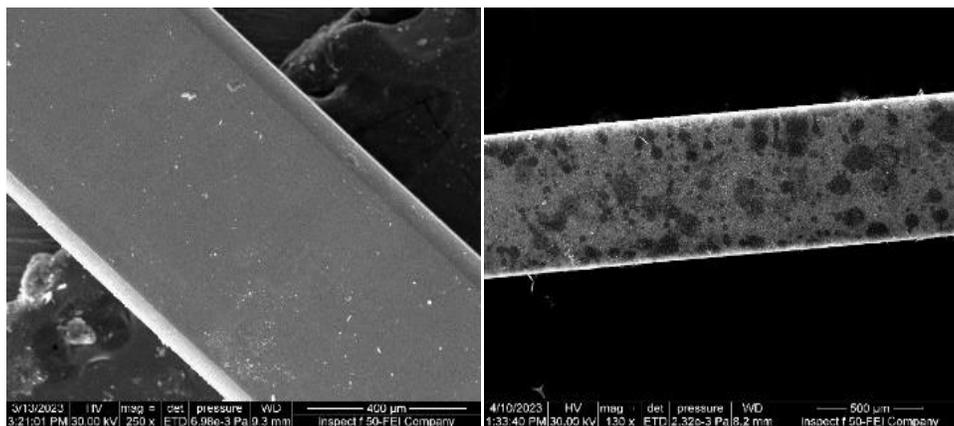


(d)

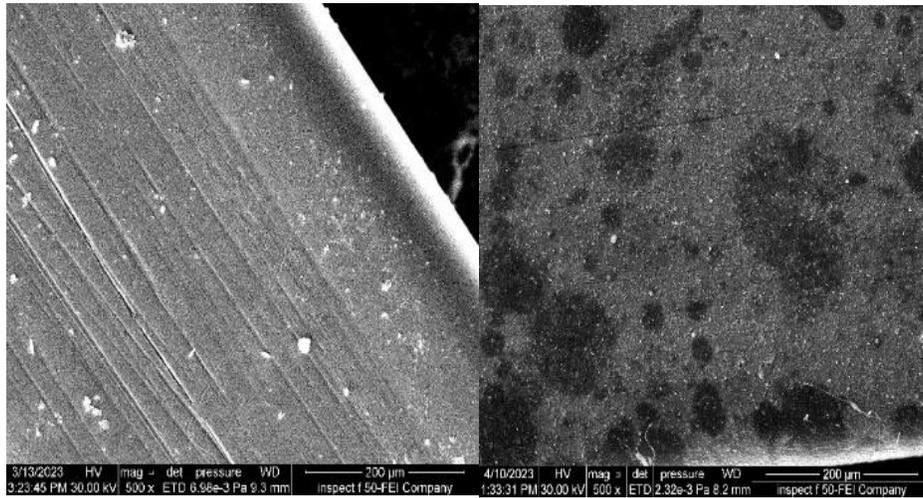


**Fig. 8: FeSEM images for the examined SSW at different magnification power for uncoated and coated**

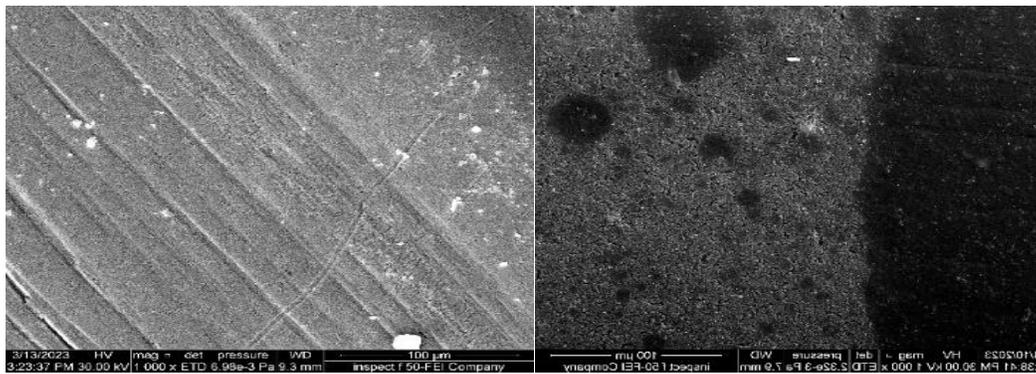
(a)



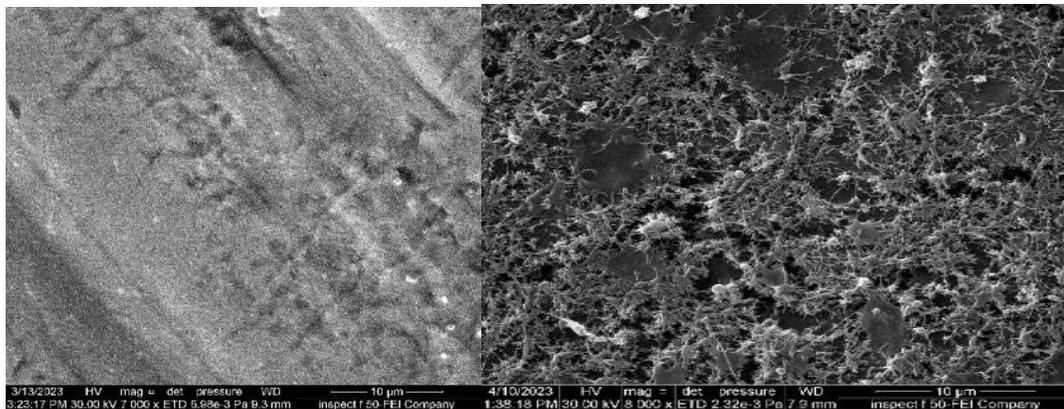
(b)



(c)



(d)



**Fig. 9.** FeSEM images for the examined NiTiW at different magnification powers for uncoated and coated.

## DISCUSSION

The main goal of this work is to develop a novel method for coating orthodontic archwires with nanoparticles. Following articles will examine the physical, mechanical, and microbiological characteristics of these nano-coated wires as well as their potential advantages for therapeutic applications after the wire has successfully undergone this novel method of surface treatment.

Orthodontists work to deliver the best care possible, resulting in stable, aesthetically pleasing, and functional outcomes. The danger of periodontal infections and the development of white spot lesions surrounding the appliances might jeopardise the outcome of treatment, despite advancements in orthodontic materials and techniques. Fixed orthodontic therapy has been linked to greater oral bacterial carriage and considerable modifications, according to prior research<sup>35,36,37</sup>. This is most likely because the appliance alters the oral environment. The use of nanoparticles as antimicrobial agents is growing in importance in dentistry and may be an effective method for both treating and preventing tooth infections<sup>[38]</sup>. Antimicrobial nanoparticles can be placed as coatings on surfaces<sup>[41]</sup> or combined directly with substances like composites, glass ionomers, or topically applied agents<sup>[39, 40]</sup>. These are the two main ways to use them into orthodontics. Orthodontic stainless steel and NiTi archwires, which are often employed during treatment with fixed appliances, are a popular location for the formation of microbial plaque<sup>[42, 43]</sup>. According to several research that evaluated microbial adhesion to various orthodontic archwires, biofilm adherence increases with time and is positively connected with surface roughness of archwires<sup>[44–45]</sup>. Although the idea of administering antibiotics locally is not new, clinical and laboratory experiments have yielded inconsistent outcomes, perhaps as a result of variations in delivery methods<sup>[46]</sup>. Numerous research have attempted to decrease oral microbiomes by coating orthodontic archwires with various antimicrobial agents, such as photocatalytic TiO<sub>2</sub>, silver material, silver nanoparticles, and N-doped TiO<sub>2</sub>. Orthodontic archwires are a useful delivery system for antibacterial agents because of their close contact to the enamel surface. The archwires can continually supply a new source of antimicrobial compounds since they are periodically changed throughout treatment<sup>[51]</sup>. Zein-based magnesium oxide nanocomposites were selected for coating in this investigation since prior research had shown that they were simple to adsorb to specimen surfaces, stick to them, and release MgO over a lengthy period of time<sup>[52]</sup>. As typical examples of various archwire diameters and cross-sections, the research used stainless steel and NiTi orthodontic upper archwires that were 0.019 to 0.025 inches in diameter. By aiming X-ray beams at the sample's surface and measuring the angles and intensities of the deflected X-rays, the XRD equipment can determine a material's crystalline phases from single or complicated composites (Khan et al., 2020). The strong crystallinity of the material was shown by a high-intensity (0 0 2) orientation peak in the XRD pattern. The "fingerprint," of a material may be seen in the infrared (IR) spectrum, which has absorption peaks that match the frequencies of atomic bond vibrations <sup>[53]</sup>. No two compounds create the same IR spectra since every substance has a different atomic structure and composition. Because peak magnitude is directly proportional to the quantity of the material, IR spectroscopy can therefore positively identify any type of material. Infrared (IR) is a useful instrument for quantitative analysis with contemporary software techniques<sup>[54]</sup>. Therefore, FTIR spectroscopy was employed to ascertain the type of chemical link that existed between the zein polymer and MgO nanoparticles in the resulting colloidal solution. The zein-coated MgO nanowires' FTIR spectra revealed distinct bands that corresponded to the bridging hydrogen-b. The "fingerprint," of a material may be seen in the infrared (IR) spectrum, which has absorption peaks that match the frequencies of atomic bond vibrations<sup>[53]</sup>. Since every substance has a different atomic structure and content, no two compounds have the same IR spectrum. Because the size of a spectrum's peak is closely correlated with the amount of material present, IR spectroscopy can thus positively identify every type of substance. Infrared (IR) acts as a crucial instrument for quantitative analysis<sup>[54]</sup> with the help of contemporary software techniques.

Therefore, FTIR spectroscopy was used to clarify the nature of the chemical link between the zein polymer and MgO nanoparticles in the resulting colloidal solution. The unique bands in the FTIR spectra of the zein-coated MgO nanowires align with the bridging hydrogen-bonded hydroxyl group of the zein and MgO. The durability of the zein coatings around the metal oxide nanofibers depends on this hydrogen bond. The best-certified equipment for providing direct images of coated and uncoated surfaces is FeSEM that are used to examine nanoparticles' topography and the shape of the sample (Wadhwa et al., 2022). As observed in FESEM images, the surfaces of the nanofibers-coated archwires of both groups showed that the nanofibers were uniformly distributed. The rapid attachment of the NFs to the stainless steel and NiTi surfaces is thought to be the result of electrostatic attraction caused by the particles' highly charged nature; like the successful deposition of the other nanoparticles on diverse surfaces, like dental implants (Wood et al., 2015), elastomeric chains (Subramani et al., 2020), ligature elastics (Kamarudin et al., 2020) and mini-screws (Hasan, 2022). To discover the essential elemental composition of a substance can utilized from EDX (Wadhwa et al., 2022). Chemical analysis and elemental mapping throughout the magnification range with EDX spectroscopy can significantly improve imaging in the SEM (Tran et al., 2018; Lu et al, 2019). When compared to control groups, the elemental examination revealed the presence of Mg atoms on the coated orthodontic archwire segments of both SSW and NiTiW. This obviously demonstrates the specimens being covered with the NFs. This was consistent with the findings of Subramanian et al. (2019) , who reported that presence of Inorganic fullerene-like tungsten (IF-WS<sub>2</sub>) nanoparticles and Wood et al. (2015), Garner et al. (2021), and Hasan (2022), who reported that the presence of Cl and P peaks in the EDX spectra identified NPs coating.

## CONCLUSION

The study demonstrates that commercially available orthodontic wires can be significantly enhanced using a unique electrospray coating technique. This innovative approach to nanoparticle coating markedly improves the surface properties of stainless steel (SS) and nickel-titanium (NiTi) wires, offering notable benefits in orthodontic applications. Although the research is based on in vitro experiments, which may not entirely replicate the complex conditions of the oral environment despite efforts to simulate these conditions using artificial saliva and an orbital shaker, the findings remain highly significant. One of the standout aspects of this study is its pioneering application of zein/MgO nanofibers to coat orthodontic archwires. This is the first time such a technique has been explored, marking a significant advancement in the field. The relatively straightforward and cost-effective nature of the coating process makes it highly practical for widespread use. Additionally, the choice of magnesium oxide as a coating material is particularly promising due to its well-documented antibacterial properties. This could play a crucial role in reducing bacterial biofilm formation on orthodontic appliances, thereby improving oral hygiene and reducing the risk of infection during orthodontic treatment. The study's strengths lie in its innovative approach and the potential implications for future orthodontic treatments. By demonstrating the feasibility and benefits of coating orthodontic wires with zein/MgO nanofibers, this research opens up new possibilities for enhancing the performance and safety of orthodontic materials. The findings suggest that incorporating nanotechnology into orthodontic treatments can lead to significant improvements in the properties of orthodontic wires, making them more effective and reliable. Overall, the study highlights the potential of nanoparticle coatings to revolutionize orthodontic materials and improve patient outcomes.

## Data Availability

The data used to support the conclusions of this study are available on this page.

## Conflicts of Interest

The authors declare that they have no competing interests.

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