Enhancing biocompatibility and osseointegration of medical implants using Ti based nanocomposite coatings

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e Abstract The surface of the replacement medical implant can be coated, which is one of the most basic and effective ways to develop and strengthen bones and increase their compatibility for the human body to solve problems with bone defects, such as the problem of releasing ions from some elements of the body. Medical alloy implants are one of the major drawbacks in bone fracture surgery, and biocompatible implants are new ways to ensure that these implants work well. These implants play a major role in accelerating osseointegration processes, but at the same time they suffer from the problem of releasing niobium and zirconium ions, which may cause serious diseases to human health. To stop these toxic ions from releasing, a bioceramic nanolayer coating was put on the alloy’s surface. This made it more biocompatible and stopped the toxic ions from escaping. Among several types of coatings, the best nanocomposite coating was selected. Three criteria and levels were used: voltage, time, and coentartation. A Takeuchi statistical program was used to collect data on all the tests (thickness, adhesion, contact angle, and roughness) in order to find the best coated sample. The study then used atomic force microscopy (AFM) and field emission scanning electron microscopy (FE-SEM) methods.

Keywords: titanium alloys, nanocomposite coating, electrophoretic deposition technique, replacement implants

1. Introduction

Biomedical titanium alloys are crucial biomedical alloys used in bone replacement prosthetic implants because they have excellent mechanical properties, high corrosion resistance, and biocompatibility (Zhang & Chen, 2019). This alloy has no adverse effects on the human body (Tibau et al., 2019). However, despite these unique qualities, they are placed in the human body, which makes them potentially harmful. The time frame is thought to be between 15 and 20 years (Van Noort, 1987). Some of the components of this alloy are harmed by the release of ions (Sarraf et al., 2021). These ions can cause numerous skin, bone, or blood problems or may be carcinogenic (Markowska-Szczupak et al., 2020). To prevent the release of these ions, scientists have worked to isolate this alloy from various fluids of the human body by coating the surface of the compensatory alloy with biologically compatible medical nanomaterials such as hydroxyapatite, magnesium oxide, and yttria-stabilized zirconia. By enhancing the adhesion strength between the coating material and the substrate, the surface roughness of the titanium alloy surface significantly improved the mechanical characteristics of the nanocoating according to numerous investigations (Jemat et al., 2015). The surface roughness augmentation of titanium implant alloys can enhance their performance. Several studies have shown that achieving rougher surfaces on titanium implants can improve osseointegration, which is the process of the implant fusing with the surrounding bone (Wang et al., 2020). Surface roughness provides greater adaptability of the implant to the environment and creates more bonding locations for cell amalgamation (Bazaka et al., 2021). Additionally, surface roughness has been found to affect cell attachment, proliferation, and protein adsorption, all of which are important for the success of an implant (Jahani & Wang, 2021; bin Anwar Fadzil et al., 2022). Metal-on-metal bearings in total hip replacement systems can release wear and corrosion particles. Therefore, coiled wire-based permanent birth control implants made of Ti alloys, which is an example of a bad thing that can occur with metallic implants, can be used. The presence of metals is a reaction to these events (Jasim et al., 2020). Since immunological or inflammatory responses frequently become clinically significant only in specific predisposed populations for which micromechanical and chemical bonding should be used to create perfect metal and ceramic joining interfaces, it can be challenging to predict such adverse outcomes (Abbass et al., 2021; Lambiase et al., 2021). Chitosan, a polymeric substance with high biological compatibility and efficacy, can be utilized to increase the strength of adhesion to the surface of medical alloys. This substance is frequently employed even though there are alternative substances, such as polymethyl alcohol (PVA), that are also effective and compatible with living things, except that the former dissolves in alcohol and the latter...
dissolves in water. The two materials were utilized independently and in combination in this study, and a comparison between them was performed to determine which of the two materials was superior (Heydariyan et al., 2023; Abraham & Venkatesan, 2023; Festas et al., 2020). Electrophoretic deposition, also known as EPD, is a fascinating electrochemical technique that combines the synergistic forces of electrophoresis and deposition. This innovative method harnesses the power of electricity to manipulate and deposit particles onto a desired surface, opening up a world of possibilities for various applications. EPD is a new and effective way to make materials and change their surfaces. Electrophoresis, which is the movement of charged particles in an electric field, and deposition, which is the small buildup of these particles on a substrate, are used. By carefully controlling electric fields and selecting suitable suspensions, EPD enables precise and controlled deposition of particles, allowing for the creation of intricate structures and coatings with tailored properties. This remarkable technique has applications in diverse fields, such as electronics, energy storage, and biomedicine (Jasim et al., 2020). Electrophoretic deposition, also known as EPD, is a fascinating and innovative technique that harnesses the power of electrochemistry to bring together two fundamental processes: electrophoretic and deposition. EPD seamlessly combines these processes and offers many exciting possibilities and applications in various fields. Let us delve deeper into the intricacies of this technique and explore the wonders it can unveil. During the initial stage of this fascinating process, a captivating phenomenon occurs. An enchanting electric field is applied between two meticulously positioned electrodes. The suspended particles, gracefully suspended in a carefully chosen solution, begin their mesmerizing dance as if under a spell. With an air of anticipation, these particles, charged with energy, elegantly glide toward the electrode with the opposite charge. It is a captivating spectacle where science and beauty intertwine, leaving us in awe of the intricate forces at play. During the second fascinating stage of this mesmerizing process, the enchanting particles gracefully gather and accumulate at the deposition electrode, where they embark on a captivating journey of transformation. These particles harmoniously come together as if guided by an invisible hand, gradually forming a remarkably uniform and exquisitely homogeneous film. This captivating phenomenon is a testament to the intricate dance of nature’s elements, as they effortlessly create a visually stunning masterpiece that will captivate the senses. (Corni et al., 2008; Loghmani et al., 2013; Fukada et al., 2004). Electrophoretic deposition (EPD) has emerged as a viral and widely employed coating method in various industries. This technique has gained significant traction due to its remarkable ease of use and cost-effectiveness. With its simple yet efficient process, EPD offers many advantages that make it an attractive choice for coating applications (Jasim et al., 2020). Covering metallic implants with calcium phosphate (Cap) reduces the discharge of potentially harmful metal ions and boosts bioactivity, two factors that might promote the uptake of EPD (Combes & Rey, 2010; Sorkhi et al., 2019). The utilization of hydroxyapatite (HAP) in dentistry and medical procedures has gained immense popularity owing to its remarkable biocompatibility and striking chemical resemblance to authentic bone tissue. Its exceptional properties have made it a good material for various applications in the field of healthcare (Hussein et al., 2017). Some of the elements used to make medical alloys release ions after ten to fifteen years, which causes chronic inflammation; this is the main problem with these alloys. The alloy is treated by covering it with a nanoceramic material (Losiewicz et al., 2022a). In general, the Ti-13Nb-13Zr alloy is very resistant to corrosion and works well with living organisms. However, there are some risks associated with placing these materials inside a person’s body, such as bone tissue damage, wear problems, mechanical problems, and possible foreign body reactions. Therefore, researchers must improve the performance of this vital alloy to achieve complete integration (Losiewicz et al., 2022a).

2. Materials and Methods

The Ti13Zr13Nb alloy, a material manufactured as a plate for this study, was examined using analytical tools with the model numbers XEPOS and SPECTRO. The chemical composition of the alloy (Ti13Zr13Nb) is shown in Table 1.

![Figure 1 (a) EPD system and (b) representation of the EPD cell configuration.](https://www.malque.pub/qjs/index.php/msj)
The thickness and adhesion as shown in Table 4 and Figure 4. The findings that are referenced above were a primary focus. As a result, higher V, t, and C values were selected from the signal.

Since the preparation of HA, Taguchi was used to choose the optimal conditions for the HA coating pattern, which is in agreement with JCPDS Card No. (09-325) and indicates the presence of the HA phase, 3.2.

Because of the addition, altered the angle of the peaks for each phase of the titanium alloy (beta and alpha). (Łosiewicz et al., 2022b). The following figure shows the HA coating pattern, which is in agreement with JCPDS Card No. (09-432) and indicates the presence of the HA phase, particularly at 2θ=26.55°, 63.41° and 32.31°. There is a phase of MgO that corresponds to the JCPDS card number (450946), especially at an angle of 2θ= 63.54°, and a phase of YSZ that corresponds to the card JCPDS card number (46-1224), at an angle of 2θ=28.66°.

To prevent the specimens from overheating during cutting, a wire-cutting machine was used to divide the specimens into sections at a moderate cutting speed and flow rate. According to the required evaluation, the thickness of the samples is determined by their 2 mm and 10 mm radii. The one face of each sample was ground utilizing SiC Emerald sheets with grit levels of 220, 400, 600, and 1000. They were then cleaned for 15 minutes in an ultrasonic bath of acetone and distilled water. Magnesium oxide nanoparticles (30-60 nm, purity of 99%, white color, 3.140 g/cm3 density) were used. To prepare the EPD solution, hydroxyapatite (HA) (30–55 nm, 99.9% purity, white color, and 4.176 g/cm3 density) was used. HA particles were used as a chitosan substance and as an adhesive on the surface of the specimen (Ti13Nb13Zr). One of the essential stages in the experimental process of EPD is the preparation of the suspension, which begins by making a solution containing 0.5 g/L chitosan by dissolving it in a 1% acetic acid solution that functions as a binding agent for powdered materials such as HA, MgO, and YSZ. Preparing suspensions is a crucial stage in the experimental process of EPD. Once the suspension solution was prepared, which included a mixture of ethanol and chitosan dissolved in acetic acid (a solvent) and 5% distilled water, the nanopowder of the HA70MgO15YSZ15 material was added. A magnetic stirrer was used to deagglomerate all the suspensions. A powerful sonicator immediately followed it. An ultrasonic processor (MIXSONIX Inc., New York, USA) was used for 30 min to verify that all the solutions were stable. As shown in the design for the EPD system employed in this work, it consists of a beaker with two electrodes submerged in the suspension and a digital power supply. The cathode and anode were made of Ti13Zr13Nb alloy and 316 L stainless steel, respectively. After coating, the coating layer on the sample surface was allowed to dry normally at room temperature.

3. Results and discussion

3.1. X-ray diffraction

X-ray diffraction (XRD) was performed for the uncoated and nanoceramic coatings (XRD type PW1730 Philips, USA), and the purity of the phase present in the coating was tested. Diffraction patterns were obtained using an X-ray diffractometer. The copper-coated XRD generator was operated at 45 kV and 30 mA, and a scanning speed of 2°C/min was used. Scanning. The average was 10-80. (Łosiewicz et al., 2022b). The XRD patterns of both the uncoated and coated substrates are shown in Figure 2.b. Figure 2.a shows the base alloy pattern, which corresponds to the beta and alpha phases and the JCPDS card number (044-1294), since no impurity phase was detected in this alloy. The addition of nanoceramic materials to the base alloy resulted in the appearance of new peaks and, depending on the effect of the addition, altered the angle of the peaks for each phase of the titanium alloy (beta and alpha). (Łosiewicz et al., 2022b). The following figure shows the HA coating pattern, which is in agreement with JCPDS Card No. (09-432) and indicates the presence of the HA phase, particularly at 2θ=26.55°, 63.41° and 32.31°. There is a phase of MgO that corresponds to the JCPDS card number (450946), especially at an angle of 2θ= 63.54°, and a phase of YSZ that corresponds to the card JCPDS card number (46-1224), at an angle of 2θ=28.66°.

3.2. Results of the Taguchi experiment

Following the creation of the suspension for the HA70MgO15YSZ15 nanocoating repeat, the statistical method of Taguchi was used to choose the optimal conditions for the HA70MgO15YSZ15 coating preparation on the Ti13Zr13Nb substrate. Since the preparation of HA70MgO15YSZ15 aimed to achieve the maximum coating thickness and adhesion, this was the primary focus. As a result, higher V, t, and C values were selected from the signal-to-noise ratio (S/N) as the optimal choice, as shown in Table 4 and Figure 4. The findings that are referenced above were obtained following the Taguchi design (L9) for the thickness and adhesion measurements of the nano-HA70MgO15YSZ15 coating utilizing DC.
3.2. Results of the Taguchi experiment

Following the creation of the suspension for the HA$_{70}$MgO$_{15}$YSZ$_{15}$ nanocoating repeat, the statistical method of Taguchi was used to choose the optimal conditions for the HA$_{70}$MgO$_{15}$YSZ$_{15}$ coating preparation on the Ti13Zr13Nb substrate. Since the preparation of HA$_{70}$MgO$_{15}$YSZ$_{15}$ aimed to achieve the maximum coating thickness and adhesion, this was the primary focus. As a result, higher V, t, and C values were selected from the signal-to-noise ratio (S/N) as the optimal choice, as shown in Table 4 and Figure 4. The findings that are referenced above were obtained following the Taguchi design (L9) for the thickness and adhesion measurements of the nano-HA$_{70}$MgO$_{15}$YSZ$_{15}$ coating utilizing DC.

Figure 3 S/N ratio and means for the multiresponse performance index.
Taguchi’s statistical method was used to determine the best conditions for preparing a HA$_{70}$MgO$_{15}$YSZ$_{15}$ layer on a Ti13Zr13Nb substrate in suspension for HA$_{70}$MgO$_{15}$YSZ$_{15}$ stabilized preparation. Optimizing the coating thickness was a primary concern throughout the HA70MgO15YSZ15 layer preparation. Table 3 and Figure 2 show that the optimal voltage, duration, and concentration were determined by maximizing the signal-to-noise ratio (S/N). Taguchi design (L9) thickness measurements for the HA$_{70}$MgO$_{15}$YSZ$_{15}$ layer produced the above results, whereas the electrophoretic deposition technique yielded the optimal HAP coating thickness (Table 3).

Table 3 Thickness, adhesion, roughness, contact angle, and multireaction and performance indices of the different coated samples.

<table>
<thead>
<tr>
<th>No</th>
<th>Voltage (V)</th>
<th>Time (sec)</th>
<th>C%</th>
<th>Thickness (μm)</th>
<th>Adhesion (N)</th>
<th>Roughness (μm)</th>
<th>Contact (angle °)</th>
<th>MRPI</th>
</tr>
</thead>
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<tr>
<td>1</td>
<td>25</td>
<td>90</td>
<td>2</td>
<td>47</td>
<td>35.301</td>
<td>0.704</td>
<td>12.901</td>
<td>7.224607341</td>
</tr>
<tr>
<td>2</td>
<td>25</td>
<td>120</td>
<td>4</td>
<td>58</td>
<td>39.008</td>
<td>0.621</td>
<td>14.701</td>
<td>10.00493061</td>
</tr>
<tr>
<td>3</td>
<td>25</td>
<td>150</td>
<td>6</td>
<td>60</td>
<td>47.034</td>
<td>0.542</td>
<td>15.21333</td>
<td>12.1888127</td>
</tr>
<tr>
<td>4</td>
<td>35</td>
<td>90</td>
<td>4</td>
<td>73</td>
<td>42.067</td>
<td>0.627</td>
<td>11.14333</td>
<td>14.17304102</td>
</tr>
<tr>
<td>5</td>
<td>35</td>
<td>120</td>
<td>6</td>
<td>69</td>
<td>40.102</td>
<td>0.491</td>
<td>2.556333</td>
<td>12.7395256</td>
</tr>
<tr>
<td>6</td>
<td>35</td>
<td>150</td>
<td>2</td>
<td>51</td>
<td>43.023</td>
<td>0.245</td>
<td>8.346667</td>
<td>9.470784438</td>
</tr>
<tr>
<td>7</td>
<td>45</td>
<td>90</td>
<td>6</td>
<td>67</td>
<td>44.019</td>
<td>0.496</td>
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<tr>
<td>8</td>
<td>45</td>
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<td>2</td>
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<td>49.120</td>
<td>0.591</td>
<td>9.680667</td>
<td>11.79639594</td>
</tr>
<tr>
<td>9</td>
<td>45</td>
<td>150</td>
<td>4</td>
<td>75</td>
<td>52.884</td>
<td>0.327</td>
<td>7.828333</td>
<td>17.32213123</td>
</tr>
</tbody>
</table>

The coating thickness is closely related to the biobased coating material. The strength of the adhesion of this layer usually characterizes this ability. The table above shows the measurements of the thickness of the coating layer composed of the nanocomposite materials mentioned above. When the voltage, deposition time, and concentration of the composite coating solution change, the thickness of the films changes, as does the adhesion of the material. The nanoparticles brightened the sediment basis. This is because changing the voltage leads to a change in the deposition rate. Nanoparticles are deposited on a sedimentation basis, i.e., the higher the voltage is, the faster the nanoparticles are deposited. We also noticed that the change in the voltage also affected the contact angle and the other two parameters, namely, the concentration of the suspension, which was three concentrations. Increasing the concentration led to an increase in thickness. As the particle velocity decreased, the deposition time also increased the thickness of the nanocoating on the deposition base, which also led to an increase in the adhesion of the biobased nanocoating to the deposition base. Of these nine models shown in the table above, through the influence of the three factors together and the use of the Taguchi statistical program, the thickness and adhesion of the nanocoating to the deposition base at the ninth value are the greatest, as shown in Table 4, Figure 4, and Figure 5. The nanocoating also has the greatest thickness among the nine coated models in the table above. The following images below show the cross-sections of the nanocomposite coatings measured in micrometres, which were calculated using an optical microscope.

Figure 4 Shows the HA$_{70}$MgO$_{15}$%YSZ$_{15}$% coating cross-section layer.

3.3. Contact angle measurements:

A steady drop of distilled water was used to test the uncoated Ti13Zr13Nb substrate and coated samples to determine how they reacted to wet. For this purpose, CAM 110-O4W optical contact angle equipment is connected to a CCD camera. The contact angle of the uncoated substrate was 73.19. With the addition of the nanocomposite material, the contact angle became 7.82833, according to the multiresponse and performance indices, indicating that the contact angle decreased significantly, which indicates that this nanocoating transformed the substrate from a hydrophobic substance into a hydrophilic substance, which means that the coated substrates have very high biocompatibility. When the contact angle and hygrosopicity of an alloy surface are both enhanced, it becomes much easier for important tissues to grow on the surface, leading to better osseointegration. Figure 6 shows the measurements of the contact angles for the coated and uncoated samples.
Figure 5 Water contact angle measurements of (a). Uncoated and (b). HA70%MgO15%-ZrO215%-coated titanium samples.

3.4. Measurement of the roughness of the coating by atomic force microscopy:

Roughness is a crucial element in the electrophoretic deposition (EPD) process in bone replacement applications. Table 4 displays the average roughness values for the coated and uncoated substrates. In contrast, Figure 6 shows the nanoscale roughness as assessed by AFM. The roughness was evaluated with a micrometer. Nanocomposites deposited on the surface of the Ti13Zr13Nb substrate and their roughness. Strong attachment between the implant and the host bone is achieved due to this quality, which influences adhesion, differentiation, and proliferation at the implant surface. Differences in the EPD parameters cause distinct variations in the shape of the deposited nanomaterials on the surface of the Ti13Zr13Nb alloy.

Table 4 Mean roughness values for two samples coated with the nanocomposite HA70%MgO15% YSZ15% and uncoated substrates.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Roughness nm</th>
<th>Particle size(mean diameter) nm</th>
</tr>
</thead>
<tbody>
<tr>
<td>HA70%MgO15%YSZ15%</td>
<td>57.15</td>
<td>51.21</td>
</tr>
</tbody>
</table>

3.5. Field emission scanning electron microscopy

Field emission scanning electron microscopy images revealed that the nanomaterials used in the coating were distributed homogeneously on the surface of the sample. This suggests that the coating technique is very successful because this technique was used to coat nanoceramic materials that are biocompatible at room temperature and without any phase shift. This finding is consistent with previous research (Ceramic et al., 2021).
4. Conclusions

The results of the present study showed that the addition of nanomaterials (hydroxyapatite, magnesium oxide, and YSZ) to a single nanocomposite in the presence of chitosan resulted in the production of alternative bioalloy surface treatment coatings that improved the adhesion strength and added bioactivity. Titanium alloy medical implant surfaces can be coated with a nanocomposite material that provides superior performance. A nanobioceramic coating on a titanium alloy increases its biocompatibility. This stops toxic ions from releasing, which extends the alloy’s useful life and decreases the risk of problems. Additionally, it was thought to be extremely hydrophilic and had an improved contact angle. This coating thus stimulates osseointegration. It greatly affects the surface properties of paint, such as its thickness, coverage, roughness, and adhesion strength. These findings are promising since extremely hydrophilic surfaces with high biocompatibility that promote tissue growth on the titanium alloy surface are essential in the early stages.

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Ethical considerations

The study was conducted in accordance with the code of research ethics at the University of Diyala. Based on the ethical guidelines for research involving human participants, all informants provided informed consent for participation in this study.

Conflict of interest

The authors certify that they have no affiliations with or involvement in any organization or entity with any financial or nonfinancial interest in the subject matter or materials discussed in this manuscript.

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