Coating of Bio-mimetic Minerals-Substituted Hydroxyapatite on Surgical Grade Stainless Steel 316L by Electrophoretic Deposition for Hard tissue Applications

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Abstract. Third-era bio-implant materials intend to empower particular live cell reactions at the atomic level, these materials represented with a resorbable and biocompatibility that bodies recuperate once they have been embedded. Necessitate to decrease expenses in public health services has required the utilization of surgical grade stainless steel (SS 316L) as the most inexpensive choice for orthodontic and orthopaedic implants. 316L SS is one of the broadly used implant biomaterials in orthodontic and orthopaedic surgeries. Yet, frequently those discharge for toxic metal ions is confirm from the implants and hence a second surgery is required will remove those implant material. One approach to managing the discharge of toxic metal ions is to coat the implant substance with bio-mimetic minerals in hydroxyapatite (HA). Bio-mimetic minerals such as magnesium (Mg), strontium (Sr), also zinc (Zn) were revealed with animate bone growth furthermore restrain bone resorption both in vitro and in vivo. The present work deals with the electrophoretic deposition (EPD) for multi minerals substituted hydroxyapatite (M-HA) on the surface treated 316L SS under distinctive temperatures (27°C, (room temperature), 60 and 80°C). The resultant coatings were characterized by FT-IR, XRD, SEM-EDX, adhesion strength and leach out analysis.

1. Introduction

Around Different bio-implant materials, 316L SS is generally utilized within orthodontic and orthopedic implants applications due to its cheap, beneficial biocompatibility, phenomenal mechanical properties, and facile fabrication [1]. To this reason, it is significant in creating strategies on enhancing that bioactivity and corrosion safety for these implants. Yet the formation of chromium oxide layer on the 316L SS surface might have been unabated will endure for that physiological condition and consequently those discharges of metallic ions, for example, chromium, iron also nickel are indicated in the human body. So, the surface treatment for the 316L SS implant is essential to stop those discharges about metallic ions in physiological medium [2]. Additionally, the 316L SS are bio-inert, keeping in mind the end goal to guarantee the bioactivity of these sorts of materials, the surface of the 316L SS bioimplants are typically coated with biomimetic materials, for example, apatite. Apatite [HA] is the generally utilized bioceramic for coating metallic implants and is most researched amid the earlier decade [3-4].
The biological properties of the apatite-based biomaterials might be improved by the inclusion of divalent ions that have invigorated various studies on ion-substituted coatings to encourage positive biological effects. In particular, the beneficial impact of $\text{Mg}^{2+}$, $\text{Sr}^{2+}$, $\text{Zn}^{2+}$ is an attractive and relevant divalent minerals ions for substitution into HA and is directly linked with the mineralization of calcified tissues, specifically amid the early phases of osteogenesis where its lessening causes bone loss and bone weakness, and encourage osteoblast propagation. With respect to the above focuses, Mg, Sr, Zn substituted HA is relied upon to have preferable bioactivity over pristine HA [4, 5].

The strong chemical adhesion of pristine/substituted HA makes its bio-implant coating to support the fresh osteoblast cell development [6]. Though owing to the constant interface with the harsh environment, the apatite coating degrades throughout long-term implantation also brings about the toxic metal ions leach out of underlying 316L SS bio-implants. Therefore, surface treatment of implant previous to the progress of bio-ceramic coating is vital in the hindrance of implant toxic metal ions leach out and to survive through long-term implant situation [2]. In the current study, the inclination of sort 316L SS in $\text{H}_2\text{SO}_4$ to frame passive layer is investigated and the M-HA electrophoretic deposition of the surface treated 316L SS is achieved.

2. Experimental Details

Type 316LSS electrodes of 10 mm x 10 mm x 2 mm size (brought from Sigma-Aldrich) were used as the metal substrate for the electrophoretic deposition (EPD). The 316L SS electrodes were mechanically polished with 120-1000 silica carbide sheets until all apparent marks were detached. Following smoothing, the electrode was washed with acetone and sonicated before exterior treatment. The whole electrode surface was subjected to acid etched of entire immersion in an acid with $\text{H}_2\text{SO}_4$ 60 min at 27°C (room temperature. The EPD process was carried out at room temperature, 60 and 80°C from 2.0 g of M-HA nanoparticles (M-HA synthesis procedure adapted from our earlier reports [4,5]) suspension in 100 ml isopropanol. A flag shape platinum sheet was used as the anode and the working electrode (316L SS) was used as the cathode. The gap between the anode and cathode was set aside to be 1 cm. The voltage used was between 90 for a fixed time interval of 5 min. The as-coated samples were subjected to different characterization methods such as FT-IR (Nicolet 380, Perkin Elmer, ), XRD (Bruker D8 ), SEM (SEM-JEOL JSM-6400), adhesion (Model 5569, Intron) and metal ions leach out tests (ICP-AES analysis).
3. Results and Discussion

3.1. Functional group analysis

Figure 1 represents the FT-IR spectra of M-HA coated on 316LSS implants under various temperatures. From the FT-IR, all M-HA coated samples were observed similar patterns of the spectrum. The bands for phosphate at 935 (ν2), 532 (ν4), 606 (ν1), and 1091 (ν3) cm⁻¹ correspond to bending stretching vibration modes respectively [7]. The bands at 3459 and 630 cm⁻¹ suggests the existence of stretching modes of the OH- group of M-HA [4-5, 7]. All these acquired bands confirmed the coating of M-HA on 316LSS implant.

![FT-IR spectra](image)

Figure 1. FT-IR spectra acquire for the M-HA coating on 316L SS at different coating temperature for (a) RT (b) 60 min and (c) 80°C.

3.2. Phase analysis

Figure 2 reveals the usual XRD patterns of the M-HA coating on the acid etched 316L SS at RT, 60 and 80°C. The major diffraction peaks of M-HA are apparent between 25° and 55° interval. Moreover the EPD temperature raises the intensity of M-HAP coating rise (Figure 2.
a-c) [8]. XRD patterns clearly affirm that the coating temperature plays an important part in the control of crystallinity of the films.

![XRD spectra](image)

**Figure 2.** XRD spectra acquire for the M-HA coating on 316L SS at different coating temperature for (a) RT (b) 60 min and (c) 80°C.

### 3.3. Surface Morphology

The surface morphology of acid etched 316L SS, M-HA-RT, M-HA-60, and M-HA-80 coatings on acid etched 316L SS are represented as Figure 3a–h. Figure 3a demonstrate acid etched implant and the morphology shows the existence of a great amount of excellent pores on the surface, which would ensure strong adherent coating on implant substrate. SEM images obviously confirm that the coating temperature shows a vital role in the influence of morphology of the films. It is furthermore held that excessive temperature can support the coating of M-HA on the exterior owing to significant dispersion movement [9]. From on this, the EPD deposition of M-HA on 316L SS was carried out at RT and raising the temperature from 60 and 80 °C. The FESEM morphology as illustrated in Figure 3b for the M-HA coating on the acid etched implant at RT consists of nano-flaks of size varying between 55 and 82 nm. When,
the temperature was increased to 60 & 80°C for the M-HA coating on the acid etched implant, the coating morphology became very much agglomerated as revealed in Figure 3(c-d). Figure 3(e-g) indicates the cross-sectional appearance of M-HA-RT, M-HA-60, M-HA-80 coating, and EDX analysis of M-HA-80. Additionally, there are no gap between the coating and implant substrate. This recommends that the adhesion between the coating and implant is great enough to maintain the forces during in-vivo surgery.

Figure 3h demonstrate the EDX analysis of the coated samples which authenticate the existence of Mg, Sr, Zn, Ca, P and O in the M-HA-80 coating on specimens. The Ca: P proportion of M-HA-80 coating is 1.68 [10]. The strong peaks of Mg, Sr, Zn, Ca, P and O represent the existence of a definite M-HA-80 coating over the metal exterior. Adhesion strengths of the M-HA-RT and M-HA-60 coatings are predictable as 7.9 and 8.3 MPa correspondingly; whereas that of the M-HA-80 coating on acid etched 316L SS sample is found to be 11.5 MPa.

Figure 3. SEM images of the (a) acid etched 316L SS (b) M-HA-RT (c) M-HA-60 (d) M-HA-80 (e) Cross sectional view of M-HA-RT coating (f) Cross sectional view of M-HA-60 (g) M-HA-80 coating (h) EDX Analysis of M-HA-80.

3.4. Leach out analysis of ICP-AES analysis

The stimulated leach out the investigation of HA-RT, HA-60 and HA-80 nanoparticle films on acid etched 316L SS samples in simulated body fluid solution after incubated for 1 hr at a marked potential of 455 mV vs. SCE was carried out employing inductively coupled plasma atomic emission spectrometry (ICP-AES) and is demonstrated in Figure 4. A considerable
quantity of metal ions such as Cr, Fe, Ni, and Mo were released out from the pristine 316L SS. There is no restriction layer on the 316L SS implant exterior to anticipate the attack of chloride ions existing in the SBF solution. While the 316L SS implant substrate is coated with M-HA nanoparticles, a number of toxic ions that release out are found to be lesser than that of the pristine 316L SS implant and also comprise the release out of the significant amount of Ca and P ions from the M-HA-RT, M-HA-60 and M-HA-80 coatings. Though, the release of Ca ions could not be anticipated from the particular coating of the M-HA-RT, M-HA-60, and M-HA-80. In the M-HA-80 coated 316L SS substrate, the leach out of toxic metal ions is considerably reduced contrasted to that of the acid etched 316L SS and individual coatings. This consideration serves to the affirmation of the construction of chromium-enriched inert oxide layer by the M-HA-80 coating on acid etched 316L SS surface which reduces a number of toxic metal ions release.
Figure 4. Metal leach out study of acid etched 316L SS, M-HA-RT, M-HA-60 and M-HA-80 coating on acid etched 316L SS implants.

4. Conclusion

Minerals substituted hydroxyapatite coating was effectively produced on acid etched 316L SS surface by the EPD process. The FTIR, XRD and SEM results validated the development of M-HA-coated on 316L SS implant. Phase and morphologies for the M-HA films were influenced by the coating temperature. The leach out investigation revealed that the M-HA-80 coating on 316L SS implant surface was controlled the rate of dissolution of poisonous metal ions. The as-coated M-HA on the acid etched surface of implants showed enhanced adhesion strength of 11.5 MPa. In conclusion, the M-HA-80 coating on acid etched 316L SS improves its performance in hard tissue applications.

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6. References

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