

Mechanical Performance of Biodegradable Magnesium Alloy after Immersion in Simulated Body Fluid

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Abstract: Magnesium (Mg) alloys, with a light weight, favorable strength, biodegradability, and biocompatibility, have the potential to be employed as biomedical implants; however, rapid degradation prevents their usage because it lowers their mechanical properties. In this study, ZE41A Mg alloy was investigated for mechanical properties after processed with powder mixed wire electric discharge machining (PMWEDM) and hydroxyapatite (HA) coating. Zinc metal powder is mixed with dielectric medium for PMWEDM process and electrochemical deposition (ECD) technique is used for preparing HA coating. Field emission scanning electron microscopy (FE-SEM) was employed to study surface morphologies. Bending/flexural and compression testing of polished, PMWEDMed, and HA-coated samples were performed before and post immersion in simulated body fluid (SBF). Results reveal a loss of bending (σ_b) and compressive (σ_c) strengths when immersed samples were compared with non-immersed ones. Immersed samples showed a decline in mechanical strengths as a result of SBF degradation. PMWEDMed samples exhibited the maximum loss of strength among all three.

Keywords: PMWEDM; Hydroxyapatite; Bending Strength; Compressive Strength; FE-SEM; Simulated Body Fluid.

1. Introduction

Currently, biodegradable materials are gaining popularity in the field of biomedical implants as temporary implants since they degrade in physiological conditions and allow injured tissue to regain its strength. These biodegradable materials are biodegradable iron alloys, cobalt-chromium alloys, magnesium (Mg) alloys, and polymers such as polyglycolic acid and polylactic acid (PLA) [1, 2]. Magnesium (Mg) has a density of 1.75-1.84 g/cm³ and an elastic modulus of 45 GPa, which is similar to that of human bone (1.75 g/cm³, 5-30 GPa); this similarity helps decrease the stress-shielding effect. Furthermore, Mg is nonmagnetic, has good mechanical strength, excellent casting and cutting capabilities with suitable thermal conductivity [3]. Mg alloys may help avoid the need for a second operation after complete tissue healing by degrading in human bodily fluids and absorbing or excreting the degraded byproducts [4]. Mg-Zn-RE-Zr alloy is a unique set of magnesium alloys with excellent performance characteristics owing to alloying additions. The alloying of Zinc (Zn) in Mg improves both mechanical characteristics and corrosion resistance [5]. A few studies indicated that up to 6 wt% of Zn concentration increases yield strength [6], whereas up to 4 wt% of Zn content improves tensile strength and elongation [7]. The solid solution of Zn in Mg can improve corrosion resistance by increasing the electrode potential of the α -Mg matrix, which is higher than pure Mg [8]. Rare earth metals exhibit superior castability, fine grain structure, increased strength, and improved anti-corrosion properties [9]. Zirconium (Zr), specifically, is added to magnesium because it has the highest effect on grain refinement in magnesium alloys [10].

In recent years, more attention has been paid to the ZE41A alloy, which belongs to the aforementioned group, because the cast alloy has low microporosity, good machinability, and corrosion resistance [11]. As a result, it finds use in commercial and aerospace sand castings, as well as biomedical applications [9]. However, the application of this alloy is limited by its low mechanical properties at room temperature, particularly poor ductility caused by the production of a brittle ternary phase on the grain boundaries [12]. A mechanical research found that the Mg-2Zn-1Ca (ZX21) alloy has good mechanical properties, including an ultimate tensile strength of 283 MPa and a failure elongation of 29% due to recrystallization and grain refining by Ca [13]. Another study of rolled and annealed Mg-2Zn-0.2Ca Mg alloy showed an ultimate tensile strength of 285 MPa, a tensile yield strength of 204 MPa, and a fracture elongation of 24% due to an improved solid-solution strengthening process [14]. Gu et al. [15] found that an Mg-2Sr alloy outperformed pure Mg in terms of corrosion resistance and mechanical characteristics while exhibiting no cytotoxicity and a positive host response. Many other researchers conduct studies to evaluate the effect of surface modifications and hot working on mechanical and corrosion properties of magnesium alloys [16-19].

Hydroxyapatite (HA) coatings on the surfaces of Mg and Mg alloys help reduce degradation rates by enhancing surface and structural properties. HA is the most abundant mineral in bone tissue, with excellent biocompatibility and biological activity. HA is unable to bear significant loads due to its intrinsic brittleness and low fracture toughness, but it can be used as a coating for other load-bearing biomaterials [20, 21]. In a degradation study of the Mg-1Zn-1Gd (ZG11) alloy, Li et al. [22] discovered that the formed HA layer effectively covered the microholes and microcracks on the implant surface. Unconventional machining processes like EDM/WEDM are useful in enhancing the surface properties of Mg alloys at optimum parametric settings, which further helps to control corrosion rate and improve mechanical properties. The alteration of surface and subsurface characteristics during WEDM processing affects Mg alloy mechanical and corrosion behaviour [23]. Thermal damage is reduced and bio-functionality is improved when process parameters such as pulse on time (T_{on}), servo voltage (SV), peak current (I_p), pulse off time (T_{off}), and cutting speed, etc. are optimized during the machining of Mg alloys. PMWEDM is a hybrid method that uses powder in the dielectric to enhance surface finish, which can further help to regulate degradation rate.

Researchers have investigated the impacts of powder-mixed EDM and WEDM techniques on a wide range of materials, including die steels, titanium alloys, tungsten-cobalt alloys, and various magnesium alloys, but there has been little research into the effect of zinc powder-mixed WEDM on mechanical properties of ZE41A Mg alloys. Furthermore, the effect of HA coating on the mechanical characteristics of ZE41A alloy is unknown. As a result, this work conducts tests to explore the effects of PMWEDM and HA coating on the mechanical properties of Mg alloy samples following immersion in SBF.

2. Methodology

2.1 Work Material

Experiments have been conducted on as cast ZE41A Mg alloy samples of size $70 \times 12 \times 4$ mm for 3 point bending test and $\varnothing 12.5 \times 25$ mm for compression test. A fine zinc metal powder has been used for the purpose of PM-WEDM. Zinc metal powder with 99% purity and mesh size 300 has been mixed with de-ionized water. Table 1 represents the physical, mechanical and thermal properties of ZE41A Mg alloy. The elemental composition of the ZE41A magnesium alloy is detailed in table 2. The sample surface was characterized by using a field emission scanning electron microscope (FESEM) (JEOL model JSM-7610F Plus).

2.2 Experimental Set-up for PMWEDM and preparation of HA coating

Samples of ZE41A Mg alloy in the present study were machined by using Electronica ELPULS 40A DLX Sprint-cut CNC WEDM, as shown in figure 1, using a soft brass wire electrode with a 0.25 mm diameter. The de-ionized (DI) water and Zn metal powder mixture is used as a dielectric fluid. The HA coating was prepared using electrochemical deposition (ECD) and alkali heat post treatment, and figure 2 shows the experimental ECD set-up for HA coating process. To prepare for the coating process the samples were mechanically ground with abrasive papers of grit size 200, 400, 600, 1000, 1500 and 2000. The samples were then cleaned with deionized water, ultrasonically cleaned with Chino Scientific apparatus in acetone at room temperature for 10

minutes, and finally dried in air. During ECD coating, the samples were taken as cathode (+ve) and platinum strip was used as anode (-ve), the current of 0.2 A value is passing at a constant voltage of 3.5 V while the stirring speed for electrolyte was maintained at 200 rpm. This layer of coating obtained was in the form of di-calcium phosphate di-hydrate or brushite ($\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$) which is a weak and unstable form of calcium phosphate (CaP) coating [24]. To obtain more stable phase of HA [$\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$] alkaline treatment (acid-base reaction) is required which converts brushite into hydroxyapatite. For this the as deposited samples were submerged in a 1 M NaOH solution at 80 °C for 2 hours [25, 26].

Table 1. Properties of ZE41A Mg alloy

Properties	Value in metric
Ultimate Tensile Strength (UTS)	205-210 MPa
Yield Strength	140-145 MPa
Compressive Strength	340-350 MPa
Brinell Micro-hardness (as cast)	62-65 HB
Vickers Micro-hardness (as cast)	65-72 HV
Elongation	3.50 %
Modulus of Elasticity	44.12 GPa
Density	1.84 g/cm ³
Thermal Expansion Coefficient (α)	27 $\mu\text{m}/\text{m}^\circ\text{C}$
Grain Size (as cast)	78-82 μm

Table 2. Elemental composition of ZE41A Mg alloy

Element	Zinc (Zn)	Zirconium (Zr)	Rare Earth Materials (REEs)	Manganese (Mn)	Copper (Cu)	Nickel (Ni)	Magnesium (Mg)
% content	3.5-5	0.4-1	0.75-1.8	0.15	0.10	0.010	91.5-95



Figure 1. Electronica ELPULS 40A DLX Sprint-cut CNC WEDM machine

2.3 In vitro immersion and mechanical properties

The samples were immersed in simulated body fluid (SBF) prepared according to Kokubo et al. [27], while maintaining a pH of 7.4 (equal to human blood pH of 7.40 ± 0.05) [28], and ion concentrations taken were comparable to human blood plasma. Degradation process of Mg alloys is time dependent and maximum corrosion or degradation of Mg-alloys occurs during the first week of immersion in SBF [29], because after one week a passive layer grows and slows down the degradation rate. In this study, the polished, PMWEDMed and HA-coated samples were immersed for 14 days in SBF. Samples were immersed in SBF using polystyrene bottles stored inside the BOD incubator in 95% air, 5% CO_2 at $37 \pm 1.5^\circ\text{C}$ for 14 days. After every 72 hours SBF was replaced to maintain an average pH value of 7.4. The quantity of SBF for different Mg samples was calculated by using the SBF to surface area ratio as 0.20 mL/mm^2 [30]. Mechanical characteristics of Mg samples were determined using 3-point bending/flexural and compression tests on a Heico Micro UTM at Punjab Engineering College, Chandigarh, as shown in figure 3 (a) and (b) with a strain rate of 0.1. Bending and compression tests were conducted using samples manufactured in accordance with ASTM E290 and E9, respectively. Bending and compression tests were performed on samples before and after being immersed for 14 days..

3. Results and Discussion

3.1 Surface morphology

The FESEM examination shows the effects of powder-mixed machining and coating on surface morphologies such as microholes, crack density, and crater development. These surface properties are especially important for forecasting the corrosion behavior of magnesium alloys, as surface degradation is closely related to mechanical performance. During the WEDM process, high-intensity spark energy is generated between the sample and wire electrodes, heating and evaporating the work sample. Because of the tremendous discharge energy, massive vapour bubbles explode, leaving large craters in the electric field [31]. Heating and cooling molten metal causes thermal stresses, which eventually result in micro holes and cracks. These surface imperfections cause rapid degradation due to the formation of hydrogen gas during immersion experiments, resulting in pitting corrosion [32].



Figure 2. ECD set-up for HA coating

FE-SEM images of surface morphologies of PMWEDMed, polished and coated samples are shown in figure 4 (a), (b) and (c), respectively. Due to the high discharge energy and flushing rates, craters form on the surface of the PMWEDMed sample, as well as micro-cracks and microholes, as seen in figure 4 (a). This leads to increased surface roughness, which promotes deterioration. On the other hand, there were no significant craters, microcracks, or microholes on the polished sample's surface, as shown in figures 4 (b). However, lines generated by abrasive paper during polishing can be detected, as well as alpha Mg with Mg-Zn-Zr T-phase [33]. HA-coated samples create surfaces with small craters, but no microcracks or holes as shown in figure 4 (c). CaP particles were found on the surface and a CaP layer or HA coating covered the cracks and holes caused by

machining. It may be concluded that HA coating might aid in the formation of smooth surfaces with less irregular geometries, thereby improving the mechanical performance of Mg alloys by keeping degradation at an optimal level.

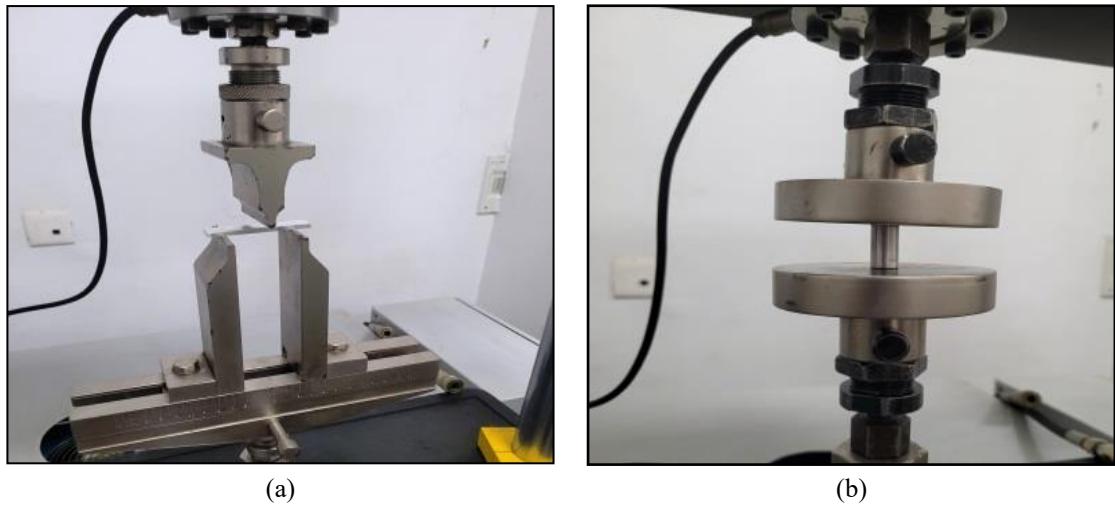


Figure 3. Mechanical test set-up for (a) 3-point bending and (b) compression testing

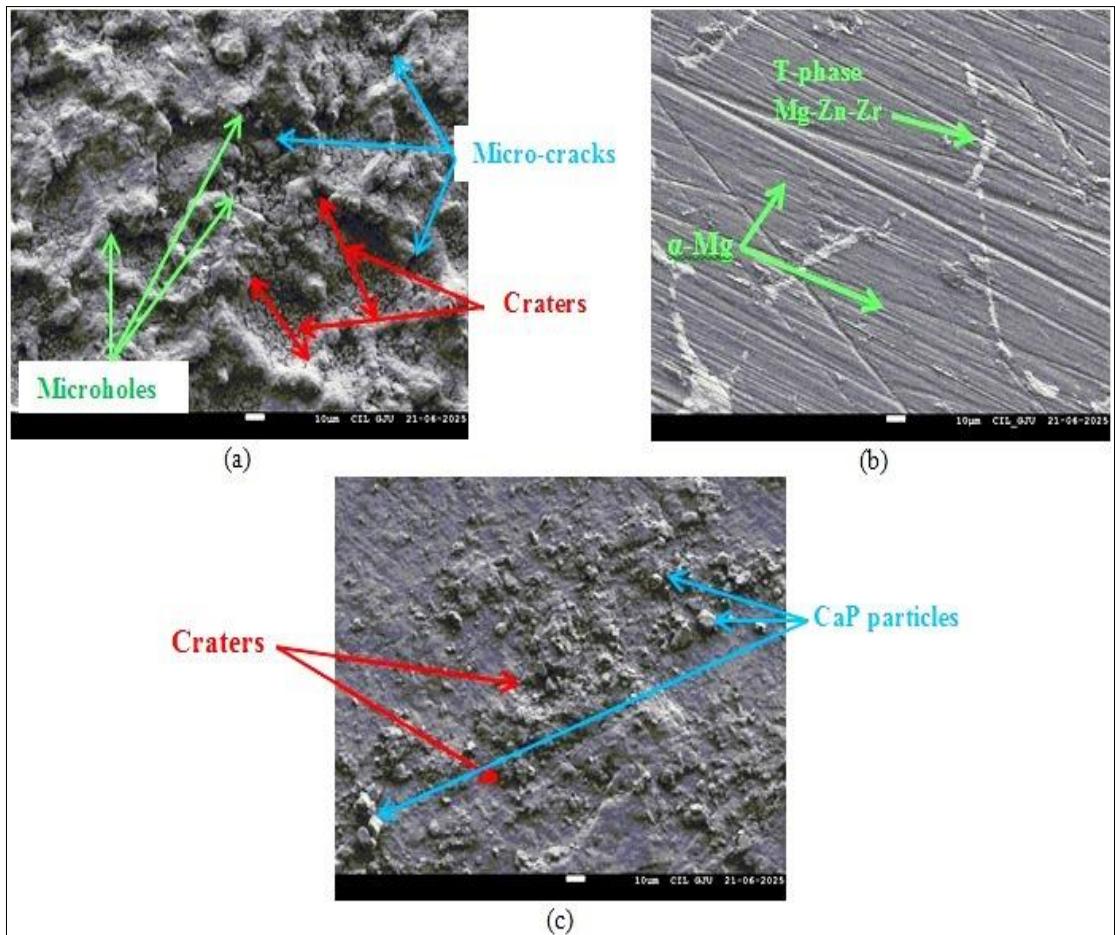


Figure 4. FE-SEM images of (a) PMWEDMed, (b) polished and (c) coated samples

3.2 Mechanical Properties

The mechanical properties of the samples were assessed at room temperature, and samples were tested for bending and compressive strengths before and after immersion, as illustrated in Figure 5 (a)-(l). In figure 5, polished Mg samples for bending and compression were shown as (a) & (c) before and (b) & (d) after 14 days of immersion in SBF, respectively. Similarly, figure 5 (e)-(h) and (i)-(l) were shown for PMWEDMed and HA coated samples. The crack initiation on the surface of the Mg samples can be seen in figure 5; immersed samples of PMWEDMed and coated samples subjected to compression tests fell apart, which shows the loss in compressive strength of immersed samples. A loss of bending/flexural strength was also noted by AbdelGawad et al. [34], in their comparative study of EZ33 and ZE41 Mg alloy. They reported a loss of 18% flexural strength for ZE41 and 13% for EZ33 alloy after 7 days immersion in HBSS. Song et al. assessed the mechanical integrity of a Mg-2Zn-Mn-Ca-Ce alloy after it underwent ECAP and water annealing [35]. Hou et al. investigated the biodegradability and mechanical properties of rolled and annealed ZX11 Mg alloys after different immersion periods [36]. They reported that extended immersion times resulted in lower strength values due to increased degradation.

In this investigation, a significant decrease in mechanical properties was also observed, as indicated in figures 6 (a) and (b). Table 3 shows that after 14 days of immersion in SBF, polished, PMWEDMed, and coated samples decreased 31.05%, 34.59%, and 32.85% of their compressive strength (σ_c), respectively. The percentage loss in compression measurements was almost equal for all three alloy samples but maximum for PMWEDMed samples. As shown in table 4, bending results showed a loss of 35.72%, 39.15%, and 36.88% of bending strength (σ_b) for polished, PMWEDMed, and coated samples, respectively. From these results it is clear that biodegradation of magnesium alloy causes considerable loss in mechanical properties. Powder-mixed WEDM and HA coating have no considerable effect on mechanical properties, owing to the fact that they only modify surface properties of the Mg alloy. They have no significant effect on the microstructure of Mg alloy, which is a crucial factor for mechanical performances. But different coating techniques and varying coating thicknesses (especially thicker layers of coatings) can help in controlling degradation and ultimately enhancing the mechanical performance of Mg alloys.

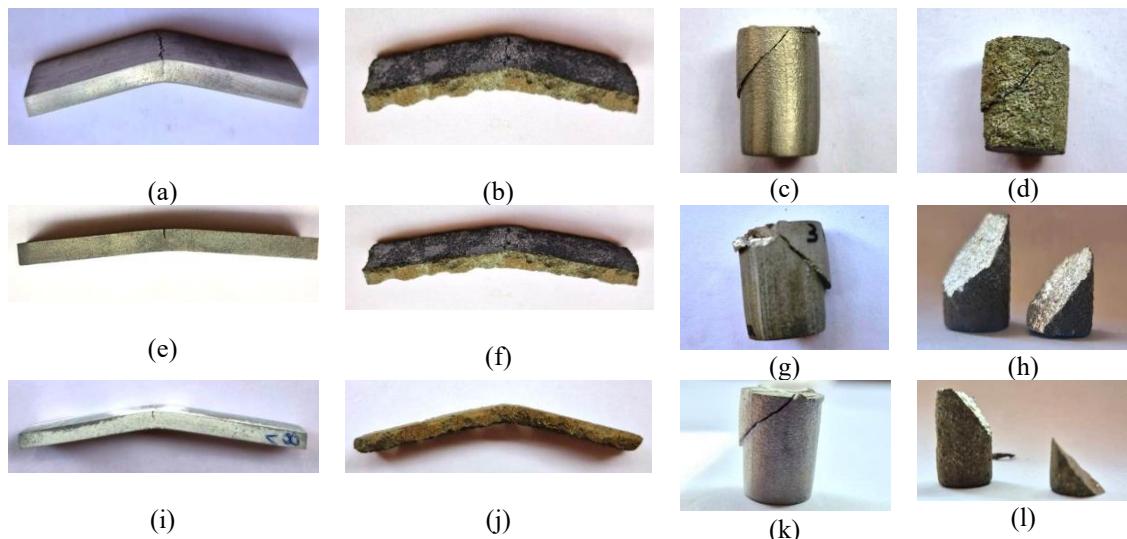


Figure 5. Bending and compression sample before and after immersion in SBF, Polished samples from (a) to (d), PMWEDMed samples from (e) to (h) and HA coated samples from (i) to (l)

Table 3. Compressive strength of ZE41A Mg alloy

Sample name	Maximum load of samples before immersion (in N)	Maximum load of samples after immersion (in N)	Compressive strength (σ_c) before immersion (N/mm ²)	Compressive strength (σ_c) after immersion (N/mm ²)	% loss of compressive strength (σ_c) after immersion
Polished	44160	25780	337.88	232.96	31.05
PMWEDM	43260	24170	329.45	215.50	34.59
HA Coated	43790	25140	336.08	225.66	32.85

Table 4. Bending strength of ZE41A Mg alloy

Sample name	Fracture Load (F) before immersion (in N)	Fracture Load (F) after immersion (in N)	Bending Strength (σ_b), before immersion in (N/mm ²)	Bending Strength (σ_b), after immersion in (N/mm ²)	% loss of Bending strength (σ_b) after immersion
Polished	709	341	265.73	170.79	35.72
PMWEDM	712	335	258.68	157.41	39.15
HA Coated	686	355	263.14	166.08	36.88

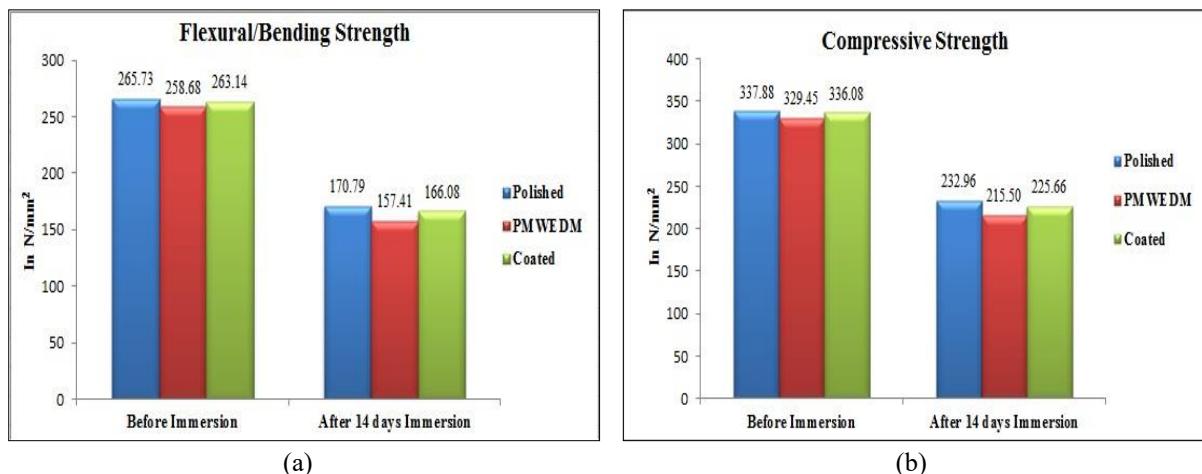


Figure 6. Bending (a) and compression (b) results before and after 14 days immersion

4. Conclusions

In this study, ZE41A Mg alloy was investigated for mechanical properties after processed with powder mixed wire electric discharge machining (PMWEDM) and hydroxyapatite (HA) coating. FE-SEM was employed to study surface morphologies and mechanical testing of polished, PMWEDMed, and HA-coated samples were performed before and after immersion in SBF for 14 days. FE-SEM images of polished, PMWEDMed and HA coated samples were compared. HA-coated and polished samples had smoother surfaces than PMWEDMed; it was due to uneven surface geometries produced by high spark energy created during machining in the form of craters, microcracks, and microholes. Mechanical tests of Mg alloy showed a decline in bending and compressive strengths after immersion in SBF. PMWEDMed samples exhibited the lowest bending and compressive strengths, owing to the highest degradation during immersion. Loss of mechanical strength was also highest for PMWEDMed samples as compared to polished and HA-coated samples. A loss of 31.05%, 34.59%, and 32.85% was found for polished, PMWEDMed, and coated samples, respectively, for compressive strength after 14 days of immersion in SBF. From bending/flexural tests, a loss of 35.72%, 39.15%, and 36.88% of bending strength was found for polished, PMWEDMed, and coated samples, respectively.

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Conflict of interest

The authors declare that there are no conflicts of interest.

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