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DEGRADATION BEHAVIOR OF DIFFERENT MAGNESIUM ALLOYS FOR TEMPORARY IMPLANTS: ROLE OF ALLOYING ELEMENTS, MICROSTRUCTURE, AND TESTING MEDIA

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Abstract: *Biodegradable Mg-based alloys seem to be the future raw materials used for the fabrication of temporary implants used in orthopedic, cardiovascular, dental, and general surgery specializations. The main problem of Mg-based alloys is their faster degradation rate in the human body, which will compromise the biomechanical stability of the implants before the end of the healing process and implicitly increase the tissue healing time. In this study, we investigated how different test media influence the corrosion behavior of magnesium-based alloys. The results obtained indicate that the choice of media significantly influences the corrosion patterns; in particular, simulated body fluid (SBF) is a more corrosive medium. This is due to the high concentration of Cl^- ions and the absence of organic compounds in the composition.*

Keywords: *Magnesium-based alloy, degradation behavior, Dulbecco's Modified Eagle's Medium, SBF*

1. INTRODUCTION

Biodegradable alloys based on magnesium, iron, and zinc look to be the future raw materials used for the fabrication of different temporary implants for many medical specializations, like orthopedic, cardiovascular, dental, and general surgery [1–9]. One reason is the failure of the classical medical devices used in different surgical specializations due to the materials and their complications [10–14]. Also, the bifunctional properties of the magnesium-based alloys look very promising: better mechanical properties than other biodegradable materials in use, biodegradability, easy to be metabolized, and positive effects on the cells and tissues [1,2,4]. The main problem of magnesium alloys for surgical implants is their faster degradation rate in the human body, which will compromise the biomechanical stability of the implants before the end of the healing process, and implicitly increase the tissue healing time [15,16]. In this context, in order to optimize the implant's bifunctional properties due to the control of the degradation process of magnesium-based alloys, several methods were developed: new alloying elements for magnesium-based alloys [17–22], new processing methods based mainly on plastic deformation and heat treatments [23–26], and new surface modification methods [27–37]. The corrosion behavior of magnesium-based materials for medical implants is still a very challenging topic, with different results being obtained according to the *in vivo* and *in vitro* environments [17,38,39]. Also, research groups obtain various results after *in vitro* testing because they use different testing media and different testing conditions [40,41]. In order to increase the similarity of *in vitro* testing media with *in vivo* conditions, researchers include some organic components to increase the complexity of the medium, following some comparative studies between static and dynamic corrosion phenomena, as well as the effect of the hydrogen diffusion coefficient [42].

The development of standardized *in vitro* test methods remains a challenging aspect, as they fail to accurately predict how magnesium-based materials will interact with living tissues, especially when the tissues have different water content [17,43]. However, before performing *in vivo* tests on animals, it is necessary to perform as many *in vitro* tests as possible. In this context, different types of test media with different chemical compositions

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have been developed. The test media usually contain inorganic ions, or inorganic ions together with organic compounds. The degradation behavior is different in the case of media containing only inorganic ions compared to those containing also the organic components, like glucose, proteins, and vitamins [44–47]. Experimental results from the literature confirm that the corrosion rate increases with increasing chloride levels in the solution. As an example, SBF (simulated body fluid) contains chlorine ions at concentrations of 147.8 mM, whereas DMEM (Dulbecco's Modified Eagle's Medium) has a concentration of 121 mM, which means a higher corrosion rate is expected in the caseof using SBF as a testing medium [48,49]. A selection of the most used testing media for biodegradable magnesium-based alloys is shown in Figure 1, with different complexities in terms of composition. As the chemical complexity of the media increases, its composition becomes more comparable to that of human body fluids (serum/plasma and interstitial fluid) [50].

The objective of the current experimental research was to determine the degradation behavior of different magnesium-based alloys, potentially used for medical implants, in 2 simulated media. The use of corrosive media with a composition similar to that of the internal biological environment for *in vitro* testing enables a more reliable prediction of *in vivo* behavior, resulting in a more accurate assessment of corrosion rate and mechanisms. The corrosive media commonly used in current research include simple isotonic solutions, simulated body fluids, complex saline solutions, cell culture media, and protein-containing media.

Table 1

The chemical composition of the Mg-based alloys

Alloy code	Composition (wt.%)						
	Zn	Ag	Zr	Y	Ca	Mn	Mg
Alloy1	7.4	1.5	1.0	0.20	-	-	89.90
Alloy2	6.4	2.5	1.0	0.20	-	-	89.90
Alloy3	4.3	-	-	-	0.3	0.62	94.78
Alloy4	-	-	-	-	0.9	-	99.10

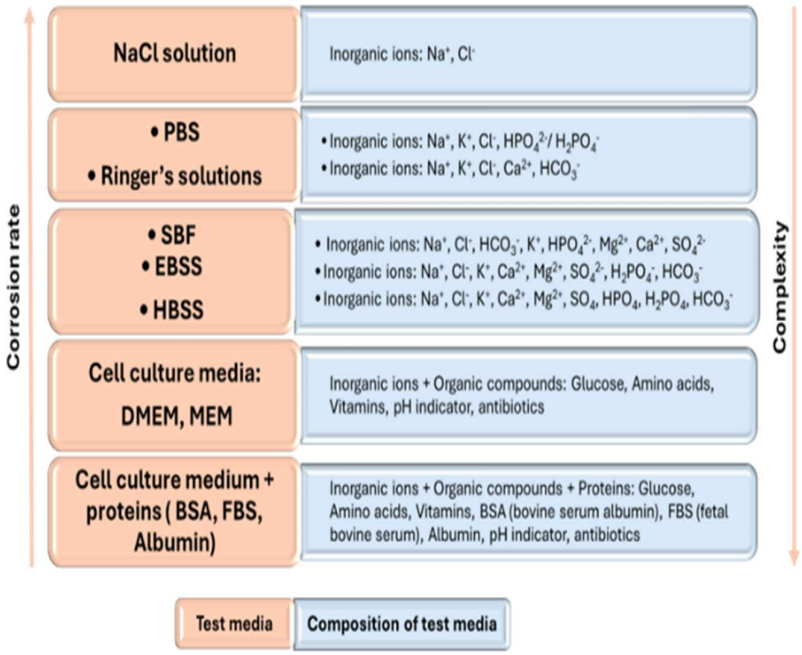


Fig. 1. The current testing media for corrosion evaluation of the biodegradable magnesium-based alloys for potential medical implants

2. MATERIALS AND METHODS

The chemical composition of the studied Mg-based alloys is summarized in Table 1. All investigated magnesium-based alloys were produced by the melt casting method. The purpose behind the use of different alloying elements was to follow their influence on the magnesium-based alloys' microstructure, which is essential for understanding their corrosion behavior. Accordingly, a complex microstructural analysis of the experimental magnesium-based alloys was carried out using optical microscopy (Nikon Metrology Europe, Leuven, Belgium) and scanning electron microscopy

(SEM - ESEM Quattro S Microscope, Thermo Fisher Scientific, Oregon, USA). During the SEM investigations, elemental chemical composition was measured using energy-dispersive X-ray spectroscopy (EDS; Thermo Fisher Scientific, Oregon, USA).

For the evaluation of the corrosion behavior of Mg-based alloys, immersion tests were carried out in two different simulated media to determine both the amount of hydrogen released and the mass loss of the samples.

The assessment of hydrogen evolution consists of measuring the volume of hydrogen generated during the immersion of the alloy, which is directly proportional to the quantity of magnesium dissolved from the alloy. The hydrogen release rate (ml/cm²) represents the volume of hydrogen released per unit area of the investigated sample surface subjected to the corrosion process and was determined at 24, 48, 72, 96, 120, 144, 168, and 192 hours on five samples of each investigated alloy.

The mass loss was evaluated through the immersion of the experimental alloys in the test medium for various time intervals (24, 48, 72, 96, 120, 144, 168, 192, 216, and 240 hours). Five samples of each investigated alloy type were used for every time point. A thermostatic bath, Julabo Model (DONAU LAB Kft, Budapest, Hungary), was used to maintain the samples at a constant temperature of 37 ± 1 °C. Monitoring the pH of the testing medium was also essential, as the degradation of magnesium alloys leads to the accumulation of HO^- ions, which shift the solution's pH toward alkaline values, unless stable corrosion products form on the sample surface, inhibiting this process. pH measurements were performed using a HI2210 pH meter (Hanna Instruments, Cluj-Napoca, Romania) at 24, 48, 72, 120, 168, and 92 hours. Mass loss was determined using the following formula:

$$M.L. = \left(\frac{m_0 - m_t}{m_0 - m_t}\right) \times 100$$

where:

m_0– mass of the sample before immersion;

m_t– mass of the sample at different time intervals.

Immersion tests were carried out in Simulated Body Fluid (SBF) and Dulbecco's Modified Eagle's Medium (DMEM), because both of them simulate the composition of human body fluids. Followed Kokubo protocol, we obtained SBF medium using as reagents: potassium chloride (KCl), sodium chloride (NaCl), magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$), sodium bicarbonate ($NaHCO_3$), sodium sulfate (Na_2SO_4), potassium phosphate dibasic trihydrate ($K_2HPO_4 \cdot 3H_2O$), calcium chloride ($CaCl_2$), and 1M hydrochloric acid (HCl) for pH adjustment to 7.4. Dulbecco's medium (pH = 7.0 - 7.6), purchased from Merck (Darmstadt, Germany), contains, in addition to inorganic salts, amino acids, vitamins, and glucose. The Simulated Body Fluid (SBF) uses tris(hydroxymethyl)aminomethane as a buffering agent, while Dulbecco's medium uses 4-(2-hydroxyethyl)piperazine-1-ethane-sulfonic acid (HEPES).

The experimental tests required parallelepiped-shaped samples measuring 15 mm × 15 mm × 5 mm (length × width × height) for precise comparison of the analyzed specimens.

3. RESULTS & DISCUSSION

3.1. Microstructural characterization of the experimental magnesium alloys

Optical and SEM-EDS microscopy were used to investigate the microstructural characteristics of four magnesium-based alloys from the Mg-Zn (Alloy 1, Alloy 2, Alloy 3) and Mg-Ca (Alloy 4) systems, with different other alloying elements.

The results obtained are presented in Figures 2 and 3. The microstructure of the investigated samples revealed characteristic features associated with Mg-based alloys casting.

All microstructures exhibit polyhedral α-Mg grains of various sizes, within which globular compounds can be observed. Precipitates or compounds with different morphologies were identified along the grain boundaries for samples from the Mg-Zn system (Alloy 1, Alloy 2, Alloy 3). In the case of the magnesium alloys from the Mg-Zn system alloyed with different Ag ratios (Alloy 1 and Alloy 2), a decrease in grain size and more pronounced grain boundaries were observed with increasing Ag concentration. In the case of Alloy 4, from the Mg-Ca system, a eutectic structure (α-Mg + Mg_2Ca) was identified at the grain boundary.

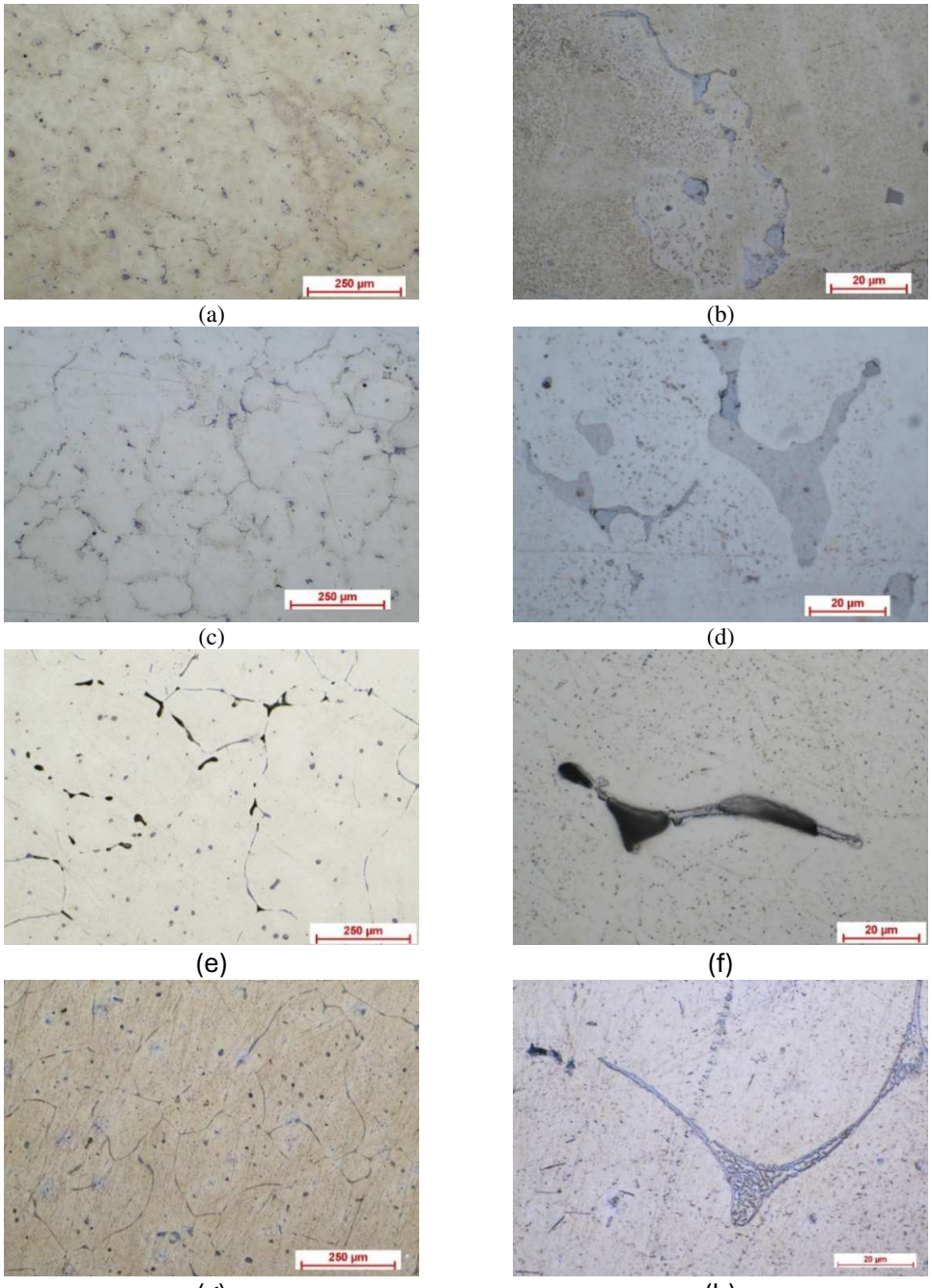
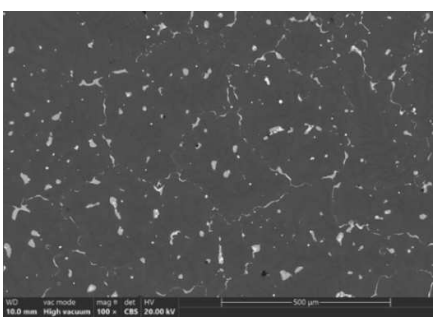
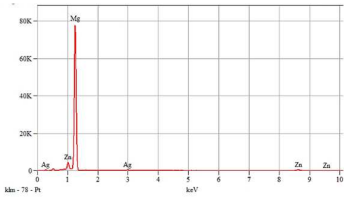


Fig. 2. The optical microscopy images (10x and 100x) for: (a) and (b) Alloy 1, (c) and (d) Alloy 2, (e) and (f) Alloy 3, (g) and (h) Alloy 4

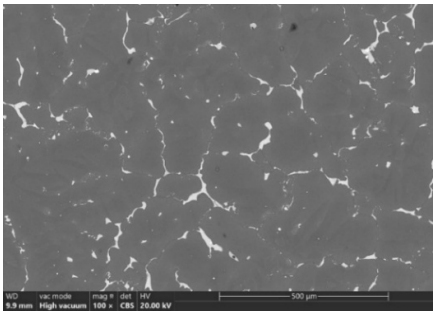
As illustrated by the SEM images in Figure 3, the microstructure of the studied magnesium alloys clearly highlights the intermetallic compounds and grain boundaries present in the Mg-based alloys' microstructure.

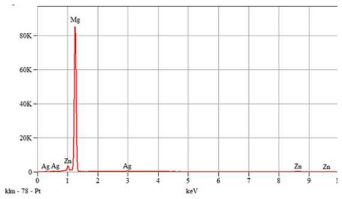




Element	Weight, %
Mg	89.83±0.35
Zn	7.45±0.24
Ag	1.49±0.12
Zr	1.04±0.05
Y	0.19±0.03
Total	100.00

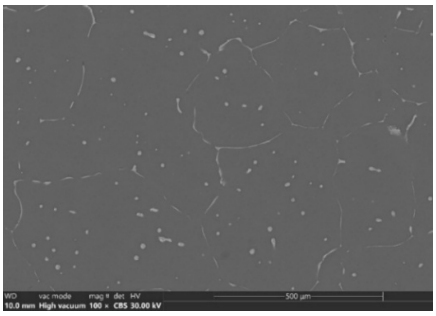
(a)

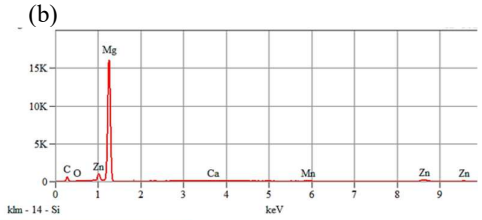




Element	Weight, %
Mg	90.09± 0.32
Zn	6.32± 0.22
Ag	2.52± 0.12
Zr	0.92± 0.08
Y	0.15±0.03
Total	100.00

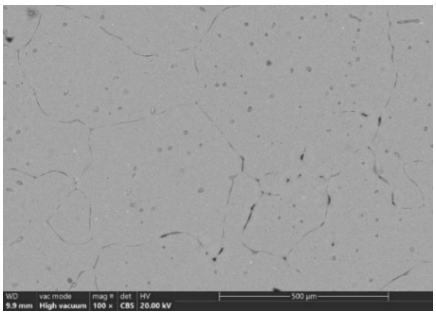
(b)

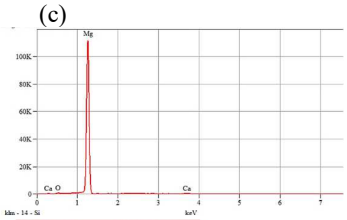




Element	Weight, %
O	2.17± 0.07
Mg	92.51± 0.34
Zn	4.38±0.20
Ca	0.38± 0.02
Mn	0.56±0.05
Total	100.00

(c)





Element	Weight, %
O	2.11± 0.07
Mg	97.05± 0.34
Ca	0.84± 0.04
Total	100.00

(d)

Fig. 3. The scanning electron microscopy images, EDS spectra, and chemical composition for: (a) Alloy 1, (b) Alloy 2, (c) Alloy 3, (d) Alloy 4

The SEM microscopy results confirm the information highlighted by optical microscopy, but more clearly. In addition, EDS analysis revealed the presence of alloying elements.

For Alloy 3 (which contains 4.3 wt.% Zn) and Alloy 4 (which contains 0.9 wt.% Ca), the presence of elements existing in the composition, as well as the development of a surface oxide layer, denotes a greater susceptibility to oxidation. Also, in the case of Alloy 1 and Alloy 2, the elements Zn, Ag, Zr, and Y were detected in their compositions.

3.2. Determination of degradation behavior by immersion testing

Figure 4 shows the evolution of hydrogen release rates for all the investigated Mg-based alloys in two testing media: Simulated Body Fluid (SBF) and Dulbecco's Modified Eagle's Medium (DMEM). The cumulative hydrogen release (ml/cm²) and the daily hydrogen release rate (ml/cm²/day) over time provide essential information about the corrosion behavior of the alloys.

The best degradation behavior in the SBF environment was observed for Alloy 2 (with 6.4 wt.% Zn and 2.5 wt.% Ag), which had the lowest hydrogen release rate during the test period (up to 192 hours). This was followed by Alloy 1 (with 7.4 wt.% Zn and 1.5 wt.% Ag), Alloy 4 (with 0.9 wt.% Ca), and finally Alloy 3 (with 4.3 wt.% Zn), which showed the highest hydrogen release rate. These results suggest that silver, especially at higher concentrations, reduces the corrosion rate. This is observed even when the zinc content in the alloy exceeds 4-5 wt.%, a threshold above which zinc is known to negatively influence the corrosion resistance of the alloy [51–53]. At concentrations higher than 4-5 wt.%, Zn tends to form secondary Mg-Zn intermetallic phases, often distributed along grain boundaries, which can act as cathodic sites towards the α-Mg matrix. This microgalvanic coupling accelerates localized corrosion, especially in chloride-rich environments such as SBF.

Calculation of the daily hydrogen release rate supports the presented observations. Alloy 2 recorded values close to the physiological tolerance limit of the human body (2.25 ml/cm²/day), reaching a maximum of 2.90 ml/cm²/day. For Alloy 3, it is observed that it significantly exceeded the limit tolerated by the human body, reaching values exceeding 12 ml/cm²/day. Alloys 1 and 4 showed intermediate behavior compared to alloys 2 and 3, with values that also exceeded the tolerable limits for the human body.

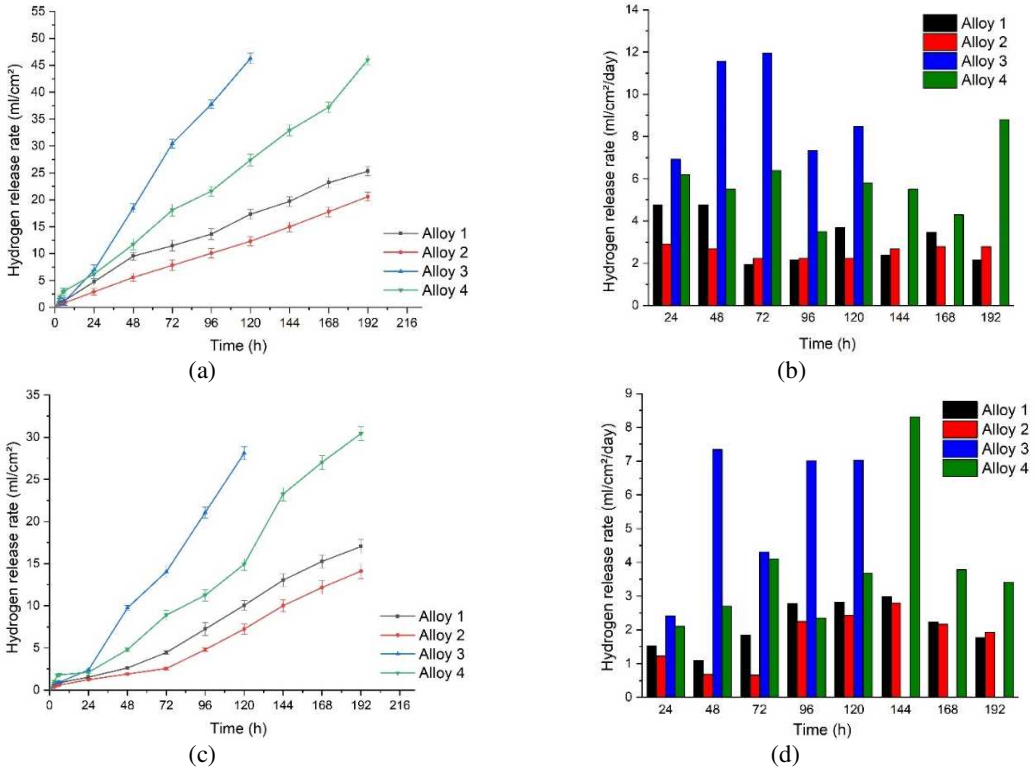


Fig. 4. Hydrogen release rate of the experimental Mg-based alloys in: (a) and (b) SBF Medium; (c) and (d) DMEM

When testing the investigated Mg-based alloys in DMEM medium, lower hydrogen release rates were observed than in SBF medium, indicating that DMEM is less aggressive during the degradation process. Again, Alloy 2 (with 6.4 wt.% Zn and 2.5 wt.% Ag) showed the best evolution, with the lowest cumulative and daily hydrogen release.

The differences between the behavior of the investigated Mg-based alloys in the two media, SBF and DMEM, may be due to their different chemical compositions and the buffer systems they contain. According to the data in the specialized literature, if the concentration of chloride ions, Cl^-, in the test medium exceeds a concentration of 30 mM, the magnesium hydroxide layer ($Mg(OH)_2$) formed on the alloy surface becomes unstable. This is converted into magnesium chloride ($MgCl_2$), a salt soluble in the medium [42,54]. Thus, the alloy surface becomes susceptible to corrosion again. As the content of Cl^- ions in the SBF medium is 147.8 mM, this explains the obtaining of a higher hydrogen release rate. In addition, the buffer system used in the preparation of the SBF medium, tris(hydroxymethyl)aminomethane, amplifies the degradation by

consuming OH^- ions from the medium [55]. In contrast, the slower degradation behavior observed in the DMEM medium can be mainly attributed to the lower Cl^- concentration (121 mM), which reduces the decomposition rate of $Mg(OH)_2$.

The evolution of mass loss and pH for the investigated Mg-based alloys in the two testing media is presented in Figure 5.

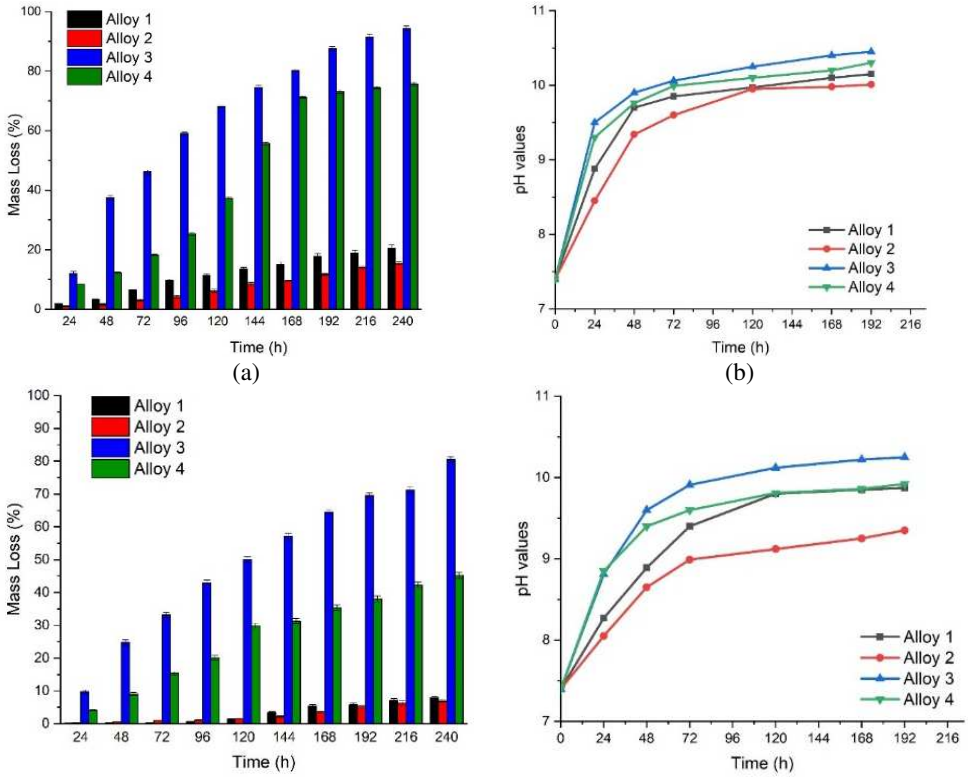


Fig. 5. Mass loss and pH evolution of the experimental magnesium-based alloys in: (a) and (b) Simulated Body Fluid Medium; (c) and (d) Dulbecco's Modified Eagle's Medium

The evolution of pH values in both test media confirms the results observed in the mass-loss test. Alloy 3, which showed the highest mass loss, caused the most significant increases in pH in both DMEM and SBF media, exceeding 10.5 in SBF and around 10.2 in DMEM.

This sharp alkalization results from the continuous formation of $Mg(OH)_2$ and the subsequent release of OH^- ions, a typical by-product of rapid magnesium corrosion.

4. CONCLUSION

In this study, the corrosion process of four magnesium-based alloys was evaluated: 3 from the Mg-Zn system (Alloy 1, Alloy 2, and Alloy 3) and 1 from the Mg-Ca system (Alloy 4). The microstructure of the investigated samples revealed characteristic features associated with Mg-based alloys casting. Even all alloys exhibit polyhedral α-Mg grains, the grain size and the precipitates or compounds along the grain boundaries are influenced by alloying elements and their concentration. The corrosion mechanism was similar for all experimental alloys. The best degradation behavior was observed for Alloy 2 (with 6.4 wt.% Zn and 2.5 wt.% Ag), which presented the lowest hydrogen release rate, the lowest mass loss, and the most stable pH values in both testing media. The results confirm that the testing media play a critical role in determining corrosion rates. Specifically, the high concentration of Cl^- ions and the absence of organic compounds make SBF a significantly more corrosive medium for the magnesium alloys studied compared to other media. However, the daily hydrogen release rate and values for mass loss demonstrate that it is necessary to optimize the chemical composition of the studied Mg-based alloys or apply appropriate surface treatments to improve their degradation behavior before being used as raw material for temporary implants.

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Comportamentul la degradare a diferitelor aliaje de magneziu pentru implanturi temporare: rolul elementelor de aliere, microstructurii și mediilor de testare

Aliajele biodegradabile pe bază de Mg par a fi viitoarele materii prime utilizate pentru fabricarea implanturilor temporare utilizate în specializările ortopedice, cardiovasculare, stomatologice și chirurgie generală. Principala problemă a aliajelor pe bază de Mg este rata de degradare rapidă în corpul uman, ceea ce va compromite stabilitatea biomecanică a implanturilor înainte de sfârșitul procesului de vindecare și implicit va crește timpul de vindecare a țesuturilor. Obiectivul cercetării experimentale actuale a fost de a determina efectul mediilor de testare asupra comportamentului la coroziune al diferitelor aliaje pe bază de Mg. Investigațiile privind comportamentul la coroziune demonstrează că mediile de testare au o influență puternică, fluidul corporal simulat (SBF) fiind un mediu mai coroziv datorită prezenței unei concentrații mai mari de ioni de Cl^- și absenței compușilor organici în compoziția sa.
Cuvinte cheie: aliaj de magneziu, degradare, fluidul corporal simulat, Mediul Eagle modificat de Dulbecco (DMEM)

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