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Chitosan/glycerol gel films for the accurate in vitro evaluation of the corrosion of biodegradable medical magnesium alloys

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INTRODUCTION

Medical magnesium-based metals are widely used in clinical applications, such as bone fixation devices, dental implants, and cardiovascular stents, thanks to their excellent biocompatibility and absorbability, as well as their ability to promote bone regeneration. However, when their degradation rate in the body exceeds expectations, excessive accumulation of magnesium ions may lead to hemolysis or osteolysis, while if the rate is too slow, there is a risk of inflammation from residual material. Therefore, it is essential to accurately quantify the degradation process in order to prevent these complications.

Biodegradable implants interact not only with bodily fluids and blood, but also with tissues and organs such as bones, muscles, and the extracellular matrix. Therefore, traditional electrochemical corrosion evaluation methods that rely on liquid electrolytes fail to accurately assess the degradation of these materials.

To address this issue, we used a chitosan hydrogel, which simulates the elemental composition and microstructure of biological tissues, as the background electrolyte. For example, the 3D porous network of the hydrogel mimics the extracellular matrix and intramuscular connective tissue, allowing for a more accurate assessment of the degradation process.

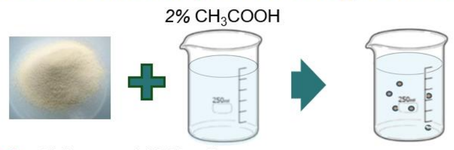
In this study, we compared the degradation of the magnesium alloy AZ91D in liquid and gel-based electrolytes through electrochemical analysis and observation of changes in the alloy's microstructure. Our results show that, unlike in solution, where the degradation is rapid and uniform, the alloy degrades more slowly when in contact with gel, accompanied by pitting corrosion on the surface and the formation of a mixed layer between the gel and alloy corrosion products.

We also investigated the influence of chitosan deacetylation degree, and electrolyte composition on the corrosion properties of the alloy. These findings can provide a basis for customizing corrosion tests to specific implant sites or individual patient variations in the future.

METHOD

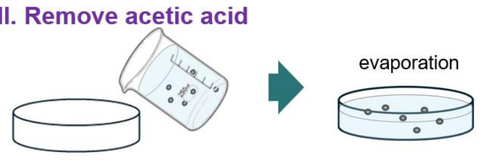
Synthesis of gels

I. Dissolve chitosan powder using acetic acid

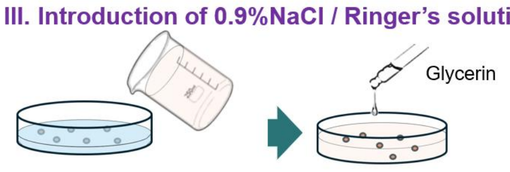


Cs1 - Chitosan *"100ing"*
Cs2 - Chitosan *"Protein"*

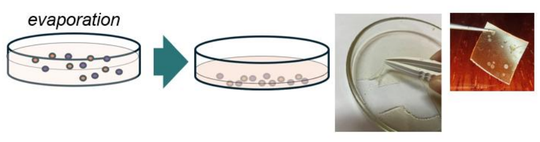
II. Remove acetic acid



III. Introduction of 0.9%NaCl / Ringer's solution

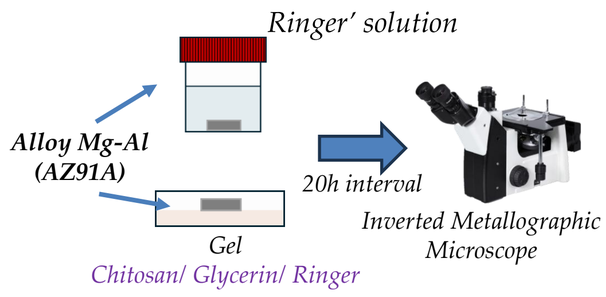


Elastic film



Experimental methods

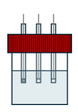
Contact test



Electrochemical assessment

PDP and EIS

Traditional liquid electrolysis cell



***RE, CE**: AgCl*
***WE**: AZ91A*
***Electrolyte**-Ringer's solution*

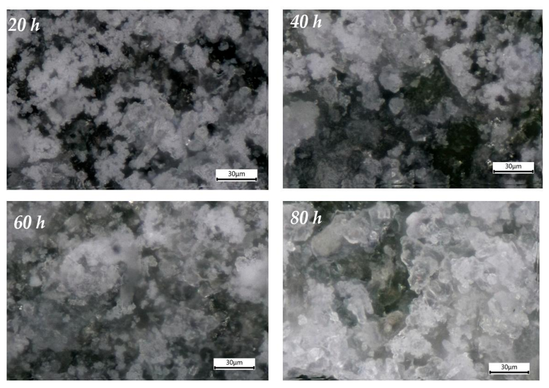
Electrochemical workstation

***RE**: Ni*
***CE**: Graphite*
***WE**: AZ91A*
***Electrolyte**-Chitosan/ Glycerin/ Ringer*

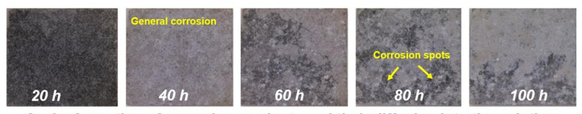
RESULTS & DISCUSSION

THE EVOLUTION OF THE ALLOY SURFACE DURING THE DEGRADATION PROCESS

Mg-Al alloy - Ringer's solution

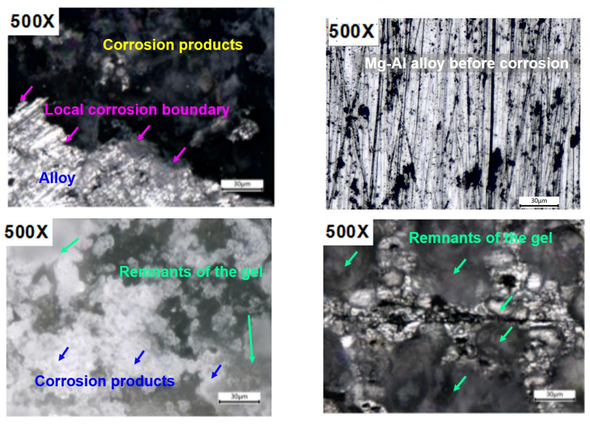


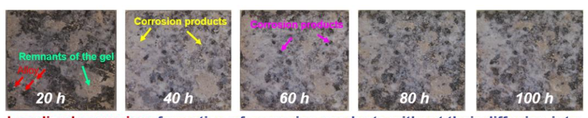
Number of corrosion products increases over time



Cycle: formation of corrosion products and their diffusion into the solution

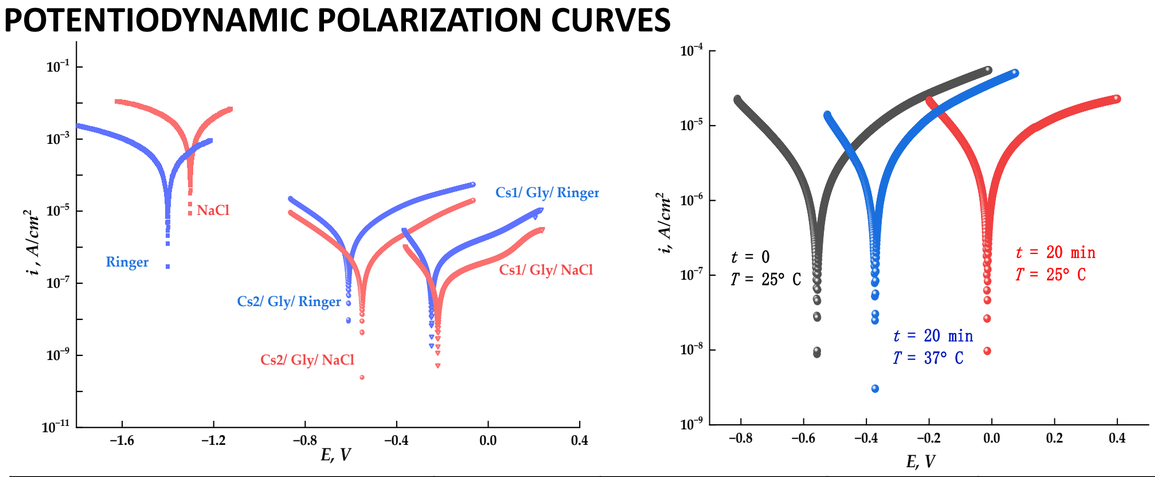
Mg-Al alloy - gel Cs2/ Gly/ Ringer





Localized corrosion: formation of corrosion products without their diffusion into the solution

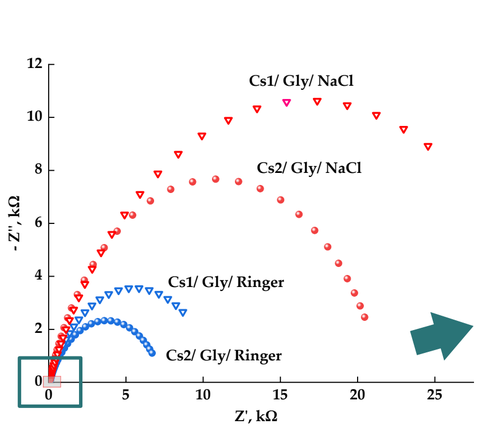
POTENTIODYNAMIC POLARIZATION CURVES

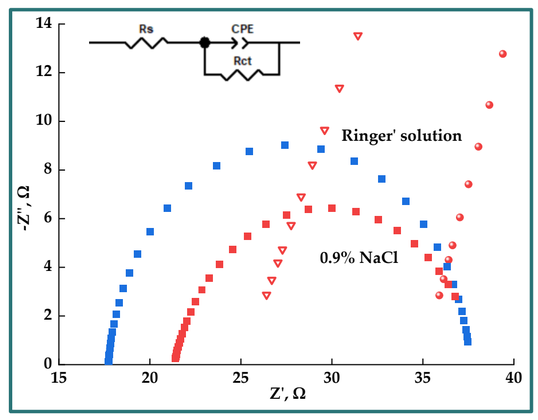


gel		*Chitosan*	i_{corr}, μA/cm²	E_{corr}, V	v_{corr}, mm/a
Chitosan/ glycerin/ Ringer		*100stoing*	*0.455*	*-0.247*	*0.010*
		protein	*2.290*	*-0.557*	*0.048*
Chitosan/ glycerin/ NaCl		*100stoing*	*0.172*	*-0.220*	*0.004*
		protein	*0.518*	*-0.551*	*0.011*
Ringer's solution			*425.74*	*-1.401*	*9.009*
0.9% NaCl			*11357*	*-1.303*	*240.341*
Chitosan/ glycerin/ Ringer (protein)	*Contact - 0min; 25 ℃.*		*2.290*	*-0.557*	*0.048*
	Contact - 20min; 25 ℃.		*7.337*	*-0.419*	***0.155***
	Contact - 20min; 37 ℃.		*3.063*	*-0.372*	*0.065*
[1] AZ31; Subcutaneous area on the back of mice; 21d					***0.223***
[2] AZ91; Subcutaneous area on the back of mice; 60d					***0.56***

ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

EIS





gel	*Chitosan*	R_s, Ω	R_{ct}, Ω	C_{dl}, $Ω^{-1}S^n$	*n*
Chitosan/ glycerin/ Ringer	*100stoing*	*66.71*	*10924*	*5.01E-5*	*0.7*
	protein	*50.49*	*7424*	*5.27E-7*	*0.7*
Chitosan/ glycerin/ NaCl	*100stoing*	*25.06*	*33517*	*2.01E-5*	*0.7*
	protein	*34.91*	*21632*	*8.84E-6*	*0.8*
solution		R_s, Ω	R_{ct}, Ω	C_{dl}, $Ω^{-1}S^n$	*n*
Ringer's solution		*17.71*	*19.89*	*70.80*	*0.9*
0.9% NaCl		*21.43*	*15.41*	*31.80*	*0.9*

CONCLUSION

1. The addition of glycerol enhances the elasticity of chitosan gel films, likely due to its molecules insert themselves into the chitosan structure, disrupting its rigid intermolecular hydrogen bonds to increase molecular mobility and thus make the chitosan film more flexible. .
2. In contact corrosion tests, the surface condition of the alloy after interaction with the gel more closely resembles the in vivo scenario.
3. Compared to conventional simulated body fluids, test results using the gel as the background electrolyte align more closely with in vivo data; furthermore, after 20 minutes of contact at room temperature, the assessment results show improved agreement.
4. Both the type of chitosan and the composition of the electrolyte solution influence the performance of the gel film in degradation evaluation.

FUTURE WORK / REFERENCES

FUTURE WORK

- The composition of corrosion products was analyzed using XRD and EDS.
- The degradation mechanism of magnesium alloy in gel medium was studied.
- KBr-Chitosan windows were prepared, and the degree of deacetylation of different brands of chitosan was determined using infrared spectroscopy.

REFERENCES

1. Walker, J.; Shadanbaz, S.; Kirkland, N. T.; Stace, E.; Woodfield, T.; Staiger, M. P.; & Dias, G. J. Magnesium alloys: predicting in vivo corrosion with in vitro immersion testing. *Journal of Biomedical Materials Research Part B: Applied Biomaterials*, **2012**, *100*(4), 1134-1141. https://doi.org/10.1002/jbm.b.32680
2. Sanchez, A. H. M.; Luthringer, B. J.; Feyerabend, F.; & Willumeit, R. Mg and Mg alloys: how comparable are in vitro and in vivo corrosion rates? A review. *Acta biomaterialia*, **2015**, *13*, 16-31. https://doi.org/10.1016/j.actbio.2014.11.048