Correlation between immersion testing methods for evaluating corrosion rate in magnesium alloys

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***Abstract*—A challenge when predicting physiological corrosion behaviour of biomaterials is the lack of established correlation between the results from in vitro and in vivo studies. This project investigates how the selection of immersion testing method affects the degradation of magnesium based alloys by comparing corrosion rates obtained from three methods: bioreactor, incubator and isothermal calorimetry. The methods produced significant differences in corrosion rates despite material samples and testing medium being identical. The bioreactor produced the highest corrosion rates, likely due to the flow of testing medium as well as active pH regulation. The incubator and calorimeter results were more similar, with calorimetric corrosion rates derived from pressure evolution being consistently lower than the corresponding corrosion rates calculated from mass loss. The findings of the report emphasise the importance of choosing parameters that simulate the physiological environment in order to achieve reliable corrosion rates in-vitro. Although the dataset was too small to determine a definitive correlation to traditional immersion methods, the in-house built isothermal calorimeter produced reasonable measurements. This suggests that further optimization of the instrument could result in a reliable immersion method for obtaining continuous corrosion rates, along with measurements of hydrogen gas evolution. This method is relevant for further studies, as it has the potential to reduce the reliance on animal trials in early stages of biodegradable material development.**

I. INTRODUCTION

MAGNESIUM (Mg) is a non-toxic, naturally occurring element in the human body, with high degradability in a physiological environment. This makes Mg a suitable material for biodegradable implants. The biodegradable properties of Mg have been studied for over a century. The first notation of its use was in blood vessel ligatures by Huse in 1878, and Mg based materials have since then been used in several wound closure and cardiovascular applications [1]. Due to Mg having an elastic modulus and density similar to that of cortical bone, it may also be suitable for biodegradable orthopaedic implants [1]. Biodegradability eliminates the need of implant removal surgery, as well as avoiding some of the risks associated with permanent implants such as encapsulation, peri-implantitis and stress shielding. Pure Mg is too malleable and degrades too quickly to be used as internal fixations of larger bone fractures [1]. However, Mg based alloys show promising results in terms of mechanical properties, although they generally degrade even faster than pure magnesium [2]. The challenge therefore lies in finding a Mg alloy that degrades slowly enough to provide support throughout the healing process.

How quickly a material degrades, referred to as its corrosion rate, is measured in millimetres per year [mm/y]. The most common methods for acquiring the corrosion rate include mass loss (ML), evolved hydrogen gas and electrochemical methods [1]. When determining the corrosion rate of a material, the test environment should be as similar as possible to the environment where the material is to be used [3]. In the application of orthopaedic implants, the material degrades enclosed in a stable, aqueous environment. Therefore, the most suitable experimental method for biomedical applications is immersion testing in simulated blood plasma with temperature and pH maintained at normal physiological conditions. In this project, both ML and evolved hydrogen based corrosion rates are derived from immersion testing. Electrochemical corrosion rate methods are beyond the scope of this project.

A. Immersion testing for evaluating Mg alloys degradation

Immersion testing consists of placing a specimen of the sample material in a testing medium and measuring the resulting degradation. The corrosion rate can then be calculated from the ML according to equation 1, from [4].

$$CR = \frac{K \cdot \Delta m}{A \cdot t \cdot \rho}, \tag{1}$$

where CR is the corrosion rate [mm/y], K=8.763104 (a constant used to convert corrosion rate unit to [mm/y] [4]), t is the time of exposure [hours], A is the surface area [cm^2], Δm is the mass loss [g], and ρ is the density of material [kg/m^3].

"American Society for Testing and Materials" (ASTM) develops standards for material testing.

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Korrelation mellan immersionsmetoder för korrosionstest av magnesiumlegeringar

The most relevant to this project were ASTM-G1-03(2017)e1 [4] and ASTM-G31-21 [3]. More specifically, ASTM-G1-03(2017)e1 describes the critical standpoints of sample preparation when it comes to the chosen material, how to properly clean a specimen from corrosion products and how to determine the corrosion rate based on ML. Whereas ASTM-G31-21 describes critical parameters for immersion testing.

1) Parameters affecting the corrosion rate: ASTM-G31-21 [3] is used to understand affecting parameters and factors when it comes to immersive degradation testing of Mg. It refers to details such as flow of the testing medium, pH level and regulation, temperature, and recommended duration of degradation test. The standard also recommends that the minimum testing medium volume to specimen area ratio being 0.2 ml/mm^2, as well as replenishing the solution if necessary due to exhausted constituents. These recommendations are based on the general degradation behaviour of metals. For magnesium in particular, the testing medium's composition, pH, and flow are especially important to take into account.

As magnesium degrades, the pH increases which leads to alkalization of the testing medium unless sufficiently buffered [1]. In alkaline conditions, a passive layer of magnesium hydroxide forms on the specimen surface, lowering the corrosion rate [1]. By increasing the testing medium volume, the effects of alkalization can be minimized. Consequently, the corrosion rate of magnesium varies depending on the medium volume to specimen surface area-ratio according to an article by Kirkland [5]. Therefore, Kirkland suggests a minimum volume ratio of 0.5 ml/mm^2, as opposed to the 0.2 ml/mm^2 recommended by ASTM [3].

It is important to note that even without alkaline conditions, corrosion products may cling to the specimen surface. This layer is to some extent dislodged naturally as an effect of H_2 bubbling, but the removal can be increased by applying flow of the test medium [1]. The composition of the testing medium also directly affects the formation and adhesion of the protective layer [6]. As a result, more complex testing medium compositions typically decrease the corrosion rate. The testing medium therefore must be chosen to simulate the physiological conditions in order to achieve a relevant prediction of in-vivo behaviour.

B. *Instruments for immersion testing*

In this project, three different immersion testing instruments were used: a bioreactor, an incubator and an in-house developed isothermal calorimeter. They were all adjusted to be as compliant as possible with the ASTM standards.

1) Incubator: An incubator is a chamber designed to keep environmental conditions constant, allowing for controlled studies of a sample. Additional to temperature regulation, parameters such as humidity, pressure and atmospheric gas composition may also be controlled or measured, depending on the instrument design. For immersion degradation testing, one or more vials containing testing medium and immersed specimens are placed inside the incubator for the duration of the test. Unless the incubator is designed specifically for immersion degradation testing, there are usually no automatic features available for regulating or measuring the pH levels of the testing medium.

2) Bioreactor: A bioreactor is a general term describing devices that are designed to enable biological processes, such as cell cultures or biochemical reactions. While the components may vary depending on the specific application, bioreactors for degradation testing typically consist of a single glass or plastic container holding several specimens immersed in testing medium. The temperature is regulated by an electric heating blanket wrapped around the test container, and a temperature probe submerged in the testing medium. Additional features include pH probe and gas sparging. In the application of Mg alloy degradation, alkalization is prevented by sparging CO_2 into the testing medium. A built in rotor, also known as an agitator, creates continuos flow in the testing medium in order to distribute the heat and CO_2 gas.

3) Isothermal Calorimetry: An isothermal calorimeter is designed to detect small changes in heat resulting from chemical reactions. Similar to the incubator, the construction consists of an insulated, temperature controlled chamber containing one or several channels. Each channel contains one sample cell (i.e. a test vial with one specimen immersed in testing medium) along with a reference cell without any specimens. Temperature sensors measure the thermal power in each channel, from which the enthalpy of the chemical reaction can be studied. While the thermal power resulting from the degradation is not included in the scope of this project, additional enthalpy calculations could be performed in order to derive the corrosion rate from these measurements. Instead, the isothermal conditions were used to derive corrosion rates from the pressure evolution (PE) in the cell.

Equation 2 shows the degradation reaction of magnesium in water, which produces magnesium oxide and hydrogen gas.

$$Mg(s) + 2H_2O(l) \rightarrow Mg(OH)_2(s) + H_2(g) \quad (2)$$

The ratio of degraded magnesium to hydrogen gas is 1:1. In a closed environment such as the calorimeter cell, H_2 builds up over time and increases the pressure. The isothermal conditions of the calorimeter then allows application of the ideal gas law to calculate the ML from the PE. This way, a continuos corrosion rate is obtained as a complement to the average corrosion rate derived from the ML measurement.

C. Focus of the project

This study aims to carry out comparable degradation experiments on Mg alloys using isothermal calorimetry, bioreactor- and incubator-based immersion testing. By comparing the corrosion rates resulting from the different methods, we hope to gain a better understanding of how the choice of immersion testing method affects the corrosion rate. This research could facilitate more accurate predictions of in vivo corrosion behaviour, as the present variation within in vitro results impedes the establishment of a correlation to in vivo corrosion [1]. This project also evaluates some advantages and disadvantages of isothermal calorimetry. Unlike traditional ML-based immersion testing methods, isothermal calorimetry provides continuous insight into the degradation process. PE-based measurements also provide insight into the production of hydrogen gas, which has been proven clinically relevant for in-vivo trials [1].

II. MATERIALS AND METHODS

A. Materials and testing medium

Immersion testing using different methodological approaches should be ASTM standard compliant, and an overview of the parameters and compliance for each analysed method can be found in table I.

1) Materials for testing and specimen preparation: The material samples consisted of magnesium alloyed with zinc, calcium and zirconium. The zinc concentrations varied, referred to as low, medium and high Zn, while the concentration of the other alloying elements remained constant. All samples were homogenised, while some were additionally extruded or aged by heat treatment, or both. These treatments were also done to analyse differences between the material treatment methods in other projects. The specimens were cut into discs, with approximately 1mm in height and 10mm in diameter. They were thereafter ground to smooth surfaces with SiC paper up to P4000 (1200 Grit) to allow maximum coverage of the specimen surface on both circular sides. The specimens were lastly cleaned with ethanol and carefully weighed. After weighing, specimens had to be used as soon as possible for the experiments, with maximum 1 hour passing (to avoid oxidization of the samples) before the specimen would have to be re-ground at P4000.

2) Testing medium: All experiments were run with the same mixed testing medium solution for the immersion itself, this being Biowest's "Hanks' Balanced Salts Solution L0607-500" (HBSS) with additional salts as buffers, these being calcium chloride dihydrate (0.265mg/ml $CaCl_2.2H_2O$) and sodium bicarbonate (1.6mg/ml $NaHCO_3$). The testing medium was kept at the same temperature and pH of 37°C and 7.4 for all tests.

The volume of the test solution per specimen surface area was kept at a recommended level of minimum 0.2ml/mm^2 or higher, with the exception of one incubator test (section II-B2).

3) Cleaning medium: Cleaning the specimens after immersion was carried out chemically using a chromium trioxide based cleaning solution (H_2O + CrO_3+$Ba(NO_3)2$+$AgNO_3$) and ethanol together with careful agitation and air drying with the goal of only removing corrosion products that had been accumulated during immersion. Chemical cleaning procedure was applied to the experiments, as mentioned in ASTM-G1-03(2017)e1. Thereafter the specimens were weighed, and this process was repeated until negligible change in mass could be noticed in between washes to avoid bias and achieve stable and reliable results.

B. Instruments for immersion testing

According to ASTM-G31-21 [3], any material in contact with the immersion test, i.e. the container or other materials to support the specimens should not be able to cause any contamination of the test solution. All specimens were supported by provided custom designed holders, to efficiently give stable support and maximise exposed surface area. The specimen holders were 3D printed out of nylon 66.

The length of each test was determined to be at least 7 days (168 hours) based on preliminary results and suggested duration formula in [3] (Duration of test in hours = 50/corrosion rate [mm/y]). The duration should be long enough to cover cases where degradation might be initially high but stabilising in later stages due to protective film being formed on specimen surface.

1) Bioreactor: A commercial bioreactor instrument (Applikon model: m02.2) was used for bioreactor immersion testing. The bioreactor method involved 15 samples in one container with 1200ml testing medium, with 5 replicate specimens for each material condition (sample). The immersion continued for 7 days, which allowed to keep the following conditions: pH within 7.4±0.2, adjusted automatically by CO_2 sparging and agitation with a rotor at the bottom of the container at 10 RPM, and temperature within 37 ± 1°C.

2) Incubator: A commercial incubator instrument (Haier Biomedical model: HCP-168) was used for incubator immersion testing. The incubator method involved placing a specimen in the middle of a beaker with testing medium, immersed freely with a minimum of 5mm from the surface. This experiment also varied in minimum testing medium volume per sample to test Kirklands [5] conclusion, where minimum volumes of 0.1, 0.2, 0.3, 0.4, 0.5 ml per mm^2 was measured. The actual volume per sample were varying due to the manual pH measurements of the liquid every day, as the measurement itself required an approximate of 1.5ml aliquot extraction (enough to cover the pH probe) that later had to be disposed of due to the risk of contamination from the pH probe (Orion 911600 & Orion 720A). The experiment was run for a total

Table I
METHOD COMPARISON OF INCUBATOR, CALORIMETER, AND BIOREACTOR.
**Some references have been able to measure H_2 evolution with incubator setup, this project's did not allow for this.*

Parameters	**Incubator**	**Calorimeter**	**Bioreactor**
Environment controls			
(G31: 9.5) Temperature (37°C)	✓	✓	✓
(G31: 9.1) pH level (7.4)	✓ CO_2 atmosphere	X	✓ CO_2 sparging
(G31: 9.8) Volume of testing medium	0.1–0.5 ml/mm^2	0.11 ml/mm^2 (17 ml/vial)	0.42 ml/mm^2 (1200 ml, 15 specimens) **Note:** Specimens were placed in the same container
(G31: 9.7) Testing medium flow	X	X	✓ Rotor (10 rpm)
(G31: 9.10) Test Duration	192 hours (8 days)	168 hours (7 days)	168 hours (7 days)
Evaluation of degradation			
(G1) Mass loss [µg] at the end of immersion	✓	✓	✓
H_2 evolution	X*	✓ (Pressure-evolution *continuous in situ*)	X
Report / Presentation of corrosion rate			
Corrosion rate recalculated to [mm/y]	From mass loss	From mass loss (and pressure)	From mass loss

of 8 days (192 hours). The temperature and pH were controlled by the incubator itself and set to 37 ± 0.2°C and 4% CO_2 atmosphere respectively.

To avoid testing medium evaporation in the environment, parafilm (PM-996) with holes was used to cover the beakers.

3) Isothermal calorimetry: Utilizing the calorimeter setup, an in-house developed isothermal calorimeter with the capacity of 4 channels was used. Each channel contained an individual sample with testing medium. The temperature was constantly kept at 37°C. The vials used for this method only allowed a volume of 17ml per channel. No pH measurements were performed.

Before measurements, a manual calibration had to be performed to adjust the sensors data readings in post. The pressure sensors were calibrated using a syringe with known air volume change in each sealed vial's environment and equation 3.

$$\alpha = \frac{\Delta U}{\Delta p}, \tag{3}$$

where α is the resulting calibration coefficient for pressure, ΔU is the voltage output change [V] and Δp is the injected pressure change [Pa].

For the heat sensor calibration, a suggested steady-state calibration by Wadsö [7] was used. Using a power supply unit (BK precision model: 9201B) with known voltage and current supply and confirming the voltage output with a multimeter (Kyoritsu model: KEW 1051). A circuitry was set up in the calorimeter, connecting vials filled with 17ml deionized water in their cells with 5kΩ resistances each. These vials were all coupled in series with the power supply unit and multimeter. Thereafter the calibration coefficients can be retrieved by applying an output voltage of 30V, and a current of 1A over 12 hours, to equation 4.

$$\epsilon = \frac{P}{U_{mean}} = \frac{I^2 \cdot R}{U_{mean}}, \tag{4}$$

where ϵ is the calibration coefficient for heat, P is the generated power [W], U is the output voltage [V], I is the supplied current [I], R is the resistance [Ω] for an individual cell.

C. Evaluation of corrosion rate

1) Measurement of specimen mass and dimensions: Specimen characteristics such as mass, diameter and thickness were determined by a µg scale (Mettler MT5) and micrometer of µm scale (Mitutoyo 293-100 micrometer). Due to small specimen size, the measurements required accuracies of these scales. When measuring mass, the specimen was simply put in the centre of the scale and let stabilise. As for geometric characteristics, the specimens were lightly pinched between the anvil and spindle with attention to the applied force, to avoid any indentation to the soft material.

2) Measurements of corrosion rate based on mass loss: The corrosion rate in [mm/y], based on ML can be determined using equation 1. The change in mass was determined by mass of the specimens before immersion and mass after cleaning procedure.

3) Measurements of corrosion rate based on the evolution of gas: ML was obtained from the H_2 gas generation rate by measuring the pressure change of the system and inserting it in equation 5, which was derived from the ideal gas law.

$$\Delta m = M \cdot n = M \cdot \frac{\Delta p \cdot V}{R \cdot T}, \tag{5}$$

where Δm is the mass loss [g], M is the molar mass of the sample [g/mol], n is the amount of substance [mol], Δp is pressure change [Pa], V is volume of the system [m^3], the gas constant R = 8.31446261815324 [J/(kg·mol)] and T is the temperature in the system [K]. The corrosion rate was then calculated using equation 1.

III. RESULTS

The experimental results using the same material samples and testing medium can be found in figure 1. whereas results from other references with similar alloy compositions are presented in figure 2.

A. Corrosion rates based on incubator testing

The incubator degradation test results that are presented in figure 3 show corrosion rates ranging from 0.3136mm/y to 0.3929mm/y. Only one specimen was tested for each volume ratio, and the specimen with the 0.2mL/mm^2 volume ratio was contaminated, likely causing the spiked corrosion rate. The contaminated specimen was therefore excluded from the calculations of average corrosion rate and standard deviation. Manual pH measurement data for the experiment can be found in table II.

B. Corrosion rates based on isothermal calorimetry testing

1) Calibration of pressure and heat measurement: Resulting calibration coefficients for the calorimeter's individual channels pressure and heat sensors can be seen in table III.

2) Corrosion rates in collaboration, isothermal calorimeter: The results from isothermal calorimetry was done by Msaebes & Zaghmout [15]. The results from the calorimeter experiments can be found in figure 1, both corrosion rates from traditional ML and PE (reflecting H_2 evolution). The corrosion rates from the PE was rendered from a provided python script developed for analysis of Mg degradation in the specific calorimeter setup. The different corrosion rate data calculations (ML and PE) stem from the same specimens, but are calculated in different ways.

C. Results from immersion testing in bioreactor (by Blidberg)

The results from the bioreactor immersion tests done by Blidberg [16] and literature survey was used as benchmark tests. The average corrosion rates of differently treated materials tested in the bioreactor are presented in table 1. These were used as comparison values for the analysis of the incubator and calorimeter results, as seen in figure 4 when comparing isothermal calorimetric and bioreactor corrosion rate results.

All corrosion rate results can be found in appendix A in table IV, V, VI. Exclusions were made in regards to some results that gave highly deviating corrosion rates in comparison to other tests of other specimens from the same sample.

IV. ANALYSIS AND DISCUSSION

A. Reliability and accuracy of measurements

Overall, our results come from small datasets consisting of 2-5 specimens per material sample. Taking that into account, we do believe that the conclusions of this project should be considered indicators for future research.

B. Correlation between degradation measurements by different methods in this project

The bioreactor consistently resulted in higher corrosion rates than both the incubator and the calorimeter, as visualised in figure 1. Since all specimens were prepared and measured following the same protocol, as well as being immersed in the same testing medium, this deviation was most likely caused by the varying environmental controls of the testing methods. The main differences were found in the testing medium volumes, means of pH regulation and agitation of the testing medium. We anticipated that the difference in testing medium volume would affect the corrosion rates since the calorimeter did not fulfil the minimum testing medium volume recommended by ASTM [3] nor Kirkland [5]. The incubator results in figure 3 support this theory, considering that neither the lowest volume ratio nor the highest volume ratio yielded corrosion rates within the standard deviation. However, the results did not indicate a large enough effect to fully explain the difference in corrosion rates between the calorimeter and bioreactor displayed in figure 4.

The calorimeter's lack of pH regulation could have slowed down the degradation process since we were unaware if alkalization of the testing medium occurred. However, the daily pH measurements of the incubator (see table II) confirmed stable pH levels of 7.4 $\pm$0.3, which nonetheless resulted in significantly lower corrosion rates than the bioreactor. That leaves us with the only possible explanation being that the agitation of testing medium, which was only present in the bioreactor, must increase the corrosion rate. Both the agitator itself and gas sparging contribute to flow in the testing medium. This likely increased the removal of corrosion products from the specimen surface, thus reducing the effect of the protective layer that normally forms through reactions with the testing medium.

As seen in figure 4, the corrosion rates derived from PE seem to correlate well with the ML, although consistently lower. A reason for the lower corrosion rates derived from pressure compared to ML could be leakage of H_2 in the calorimeter's cells. Another aspect could be incorrect calibration coefficients, as these were initially recommended to be tested with air filled vials, whilst it would be more reasonable

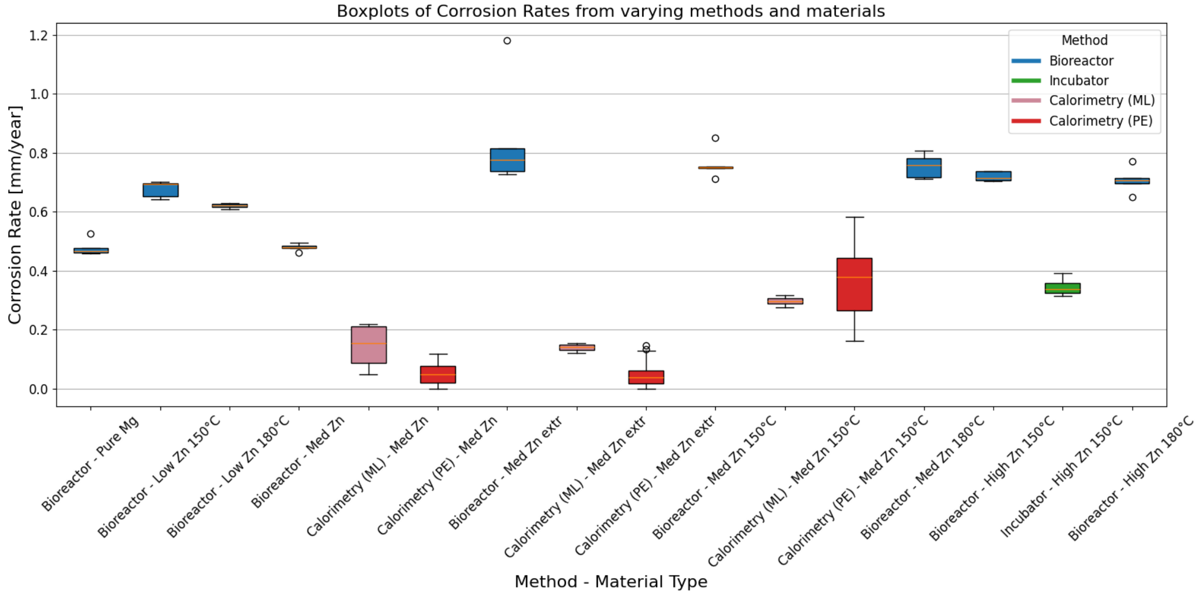

Figure 1. Box-plots of corrosion rates using varying materials and different methods of both testing and calculation. The corrosion rates from calorimeter data utilising mass loss (ML) based and pressure evolution (PE) based calculations were generated from the same specimens. All specimens used the same testing medium.

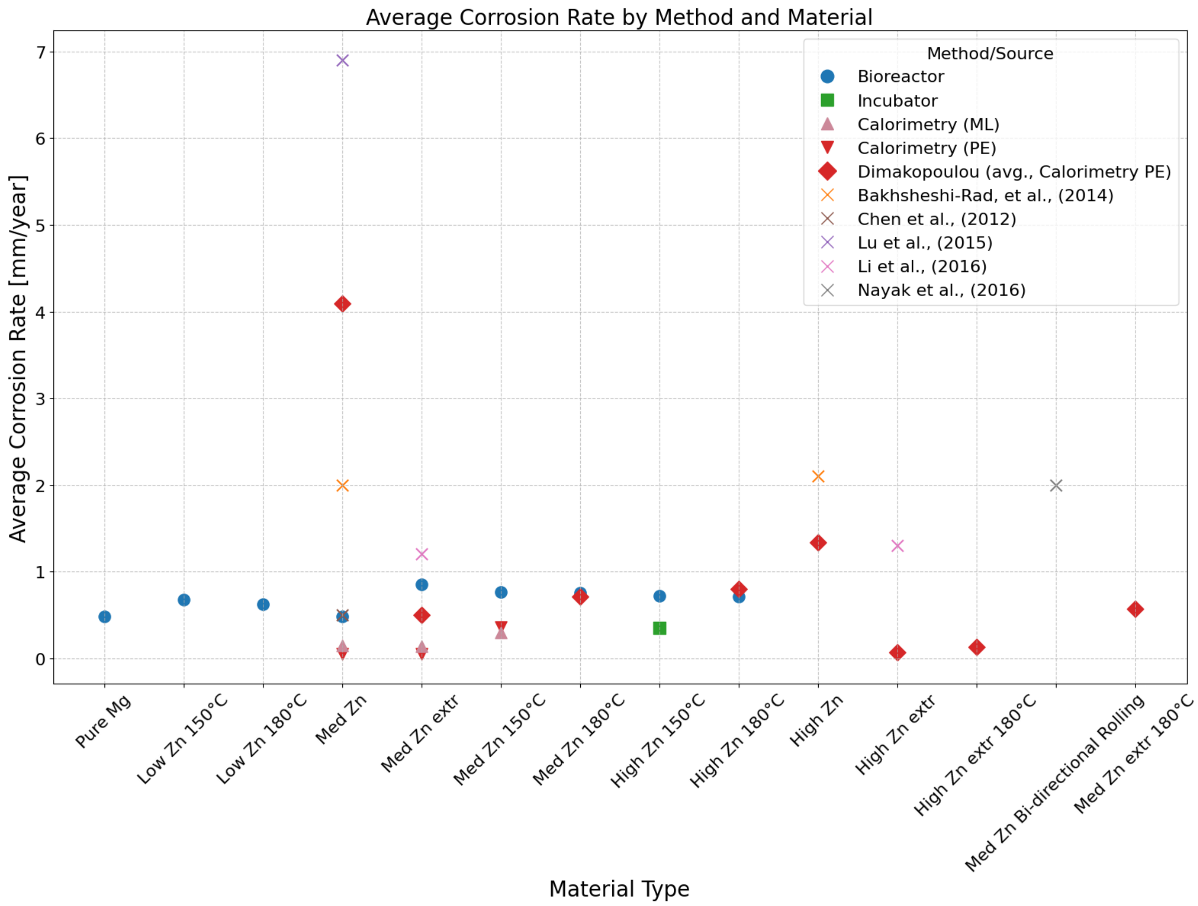

Figure 2. Comparison of collected corrosion rate results with values from literature. The reference values are sorted into medium and high Zn concentrations, defined as 3 percent and 4 percent weight content, respectively.
Data extracted from [8], [9], [10], [11], [12], [13] and Agathi Dimakopoulou [14].

to try a calibration with liquid present, e.g. deionized water or testing medium. The cleaning protocol of the specimens before weight measurement could be too abrasive. While the cause of the deviation between PE

Table II
TABLE OF pH MEASUREMENTS OF ALIQUOT FOR INCUBATOR IMMERSION TESTING WITH HIGH ZN 150°C AT VARYING MINIMUM ELECTROLYTE VOLUMES. NOTE THAT NO MEASUREMENTS WERE DONE OVER THE WEEKEND.

Vol. [ml/mm^2]	Day 0	Day 1	Day 2	Day 3	Day 6	Day 7	Day 8
0.1	8.04	7.44	7.41	7.46	7.41	7.61	7.48
0.2	8.04	7.37	7.34	7.38	7.55	7.62	7.47
0.3	8.04	7.24	7.29	7.40	7.45	7.47	7.35
0.4	8.04	7.35	7.31	7.40	7.52	7.75	7.61
0.5	8.04	7.35	7.32	7.40	7.56	7.55	7.48

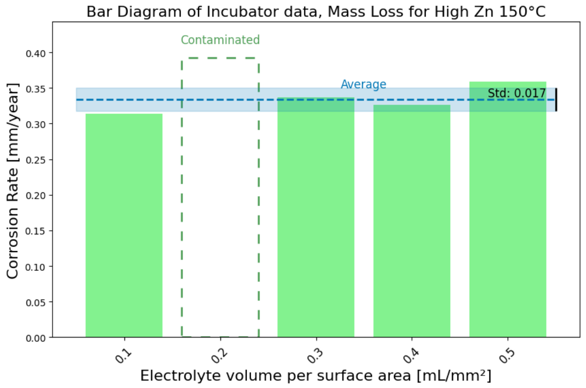

Figure 3. Plot showing the data from immersion testing using incubator and different testing medium minimum volumes. The corrosion rates in [mm/y] from left to right were: 0.3136, 0.3929, 0.3371, 0.3259 and 0.3591. The contaminated specimen was excluded in the calculation of average CR and standard deviation.

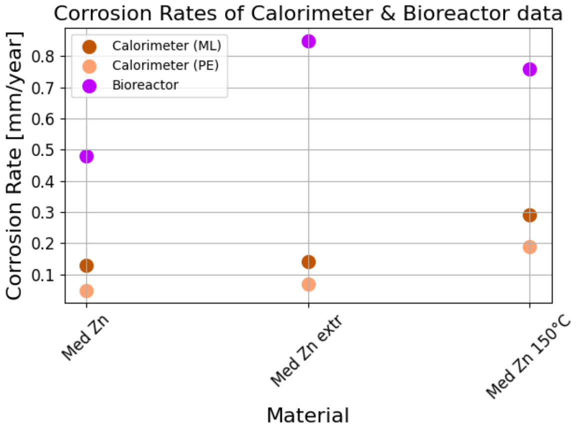

Figure 4. Scatter plot of collected data corrosion rate averages from calorimetric and bioreactor degradation testing. ML = Mass Loss, PE = Pressure Evolution.

Table III
TABLE OF RESULTING CALIBRATION COEFFICIENTS. α IS THE PRESSURE COEFFICIENT AND ϵ IS THE HEAT COEFFICIENT.

Channel	α [Pa/mV]	ϵ [mW/mV]
1	213.01	8.62
2	209.45	8.49
3	213.32	8.54
4	216.48	8.82

and ML was not determined, the correlation indicates that the calorimeter not only can act as an alternative to traditional ML-based immersion testing, but also provide further insight into the degradation process. For example, to see how the corrosion rate varies over time without having to repeat the experiment with different immersion durations.

C. Comparison to literature results

The comparison in figure 2 shows that even when testing similar materials with similar testing media, the results will often vary. The variations in our results therefore seem reasonable compared to the corrosion rates from previous studies. This further proves the need of taking method parameters into account when conducting degradation studies of magnesium alloys.

D. Sustainability and Ethics

These results can contribute to the research and development of biodegradable implants, by indicating how to improve in vitro degradation testing in order to achieve reliable predictions of in vivo corrosion. This could reduce the reliability on animal testing and expedite the process of reaching human clinical trials. The PE measurements from isothermal calorimetry are also valuable when exploring the consequences of hydrogen evolution in vivo. From a sustainability viewpoint, isothermal calorimetry can reduce the amount of resource consuming experiments, since continuous monitoring allows for more data to be gained from each specimen.

E. Projections and recommendations for future work

In future research, it would be valuable to collect a larger dataset by conducting more experiments. In the calorimetric setup, there seemed to be leakage of H_2 gas, indicated by the PE over time for some cells. These specimen's PE data therefore had to be discarded. Leakage would need to be prevented to allow more data collection from a singular experiment. The pH levels of the testing medium should be measured before and after immersion in order to evaluate its effect on the corrosion rate. An increased amount of testing medium could be explored to comply with the recommended ASTM minimum volume of 0.2ml/mm^2, especially when considering the effects of alkalization and replenishment of testing medium due to exhausted constituents mentioned in [3].

The effect of the flow from the sparging in the bioreactor setup should also be studied, and what implications it might have on the protective film that forms on the surface of the specimens in the testing medium.

CONCLUSIONS

Isothermal calorimetry shows promising results for deriving Mg-alloy corrosion rates from PE, which were consistently lower than those from ML. Corrosion rates obtained from the bioreactor were significantly higher compared to those from the incubator and calorimeter. Agitation of testing medium likely increases the corrosion rate even at low rotational speeds, by preventing the protective surface film to form.

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Both authors have contributed equally to the report writing, some sections/subsections were divided in the writing process. Sample preparation was done contributed equally, as well as calibration efforts. Post processing and diagram generation of the collected data was done by Fredrik Giang in Python and conceptual presentation and analysation was done mainly by Hanna Bohman. Internal research group presentations were co-constructed and incubator pH measurements was carried out by both authors equally.

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APPENDIX

Table IV
TABLE WITH CORROSION RATES FROM SARA BLIDBERG [16], USING BIOREACTOR AND VARYING MATERIALS USED FOR ANALYTICS IN THIS PROJECT. RED MARKED CELLS SHOW DATA THAT WERE EXCLUDED.

Material	**CR**
Pure Mg	0.5259
Pure Mg	0.4675
Pure Mg	0.4578
Pure Mg	0.4776
Pure Mg	0.4616
Med Zn	0.4844
Med Zn	0.4765
Med Zn	0.4953
Med Zn	0.4605
Med Zn	0.4808
Med Zn extr	1.1809
Med Zn extr	0.7263
Med Zn extr	0.7367
Med Zn extr	0.8149
Med Zn extr	0.7772
Low Zn 180°C	0.6227
Low Zn 180°C	0.6304
Low Zn 180°C	0.6078
Low Zn 180°C	0.3282
Low Zn 180°C	0.9201
Med Zn 180°C	0.7173
Med Zn 180°C	0.7109
Med Zn 180°C	0.7591
Med Zn 180°C	0.7828
Med Zn 180°C	0.8081
High Zn 180°C	0.7723
High Zn 180°C	0.7077
High Zn 180°C	0.6497
High Zn 180°C	0.6971
High Zn 180°C	0.7132
Low Zn 150°C	0.6941
Low Zn 150°C	0.6975
Low Zn 150°C	0.7005
Low Zn 150°C	0.6425
Low Zn 150°C	0.6513
Med Zn 150°C	0.8505
Med Zn 150°C	0.7502
Med Zn 150°C	0.7117
Med Zn 150°C	0.7521
Med Zn 150°C	0.747
High Zn 150°C	0.7075
High Zn 150°C	0.7051
High Zn 150°C	0.7371
High Zn 150°C	0.7134
High Zn 150°C	0.7381

Table V
TABLE WITH CORROSION RATES FROM ISOTHERMAL CALORIMETRY PERFORMED BY MENA MSAEBES AND YARA ZAGHMOUT [15], WITH VARYING MATERIALS AND CORROSION RATE CALCULATION METHODS USED FOR ANALYTICS IN THIS PROJECT. RED MARKED CELLS SHOW DATA THAT WERE EXCLUDED.

Calc. Method	**Material**	**CR**
ML	Med Zn	0.05
ML	Med Zn	0.2191
ML	Med Zn	0.2081
ML	Med Zn	0.0987
ML	Med Zn extr	0.1354
ML	Med Zn extr	0.1538
ML	Med Zn extr	0.1492
ML	Med Zn extr	0.122
ML	Med Zn 150°C	0.2767
ML	Med Zn 150°C	0.3171
ML	Med Zn 150°C	0.2911
ML	Med Zn 150°C	0.3041
ML	Med Zn 180°C	1.7357
ML	Med Zn 180°C	1.7858
ML	Med Zn 180°C	1.7569
PE	Med Zn	0.02
PE	Med Zn	0.03
PE	Med Zn	0.2081
PE	Med Zn	0.0987
PE	Med Zn extr	0.05
PE	Med Zn extr	0.05
PE	Med Zn 150°C	0.6
PE	Med Zn 150°C	0.48
PE	Med Zn 150°C	0.23
PE	Med Zn 180°C	2.16
PE	Med Zn 180°C	1.72

Table VI
TABLE WITH CORROSION RATES FROM VARYING LITERATURE SOURCES USED FOR ANALYTICS IN THIS PROJECT. [8], [9], [10], [11], [12], [13] AND [14]

Source, Method	**Material**	**CR**
Bakhsheshi-Rad et al., (2014)	Mg-3Zn	2
Bakhsheshi-Rad, et al., (2014)	Mg-4Zn	2.1
Chen et al., (2012)	Mg-3Zn	0.5
Nayak et al., (2016)	Mg-3Zn Bi-dir Rolling	2
Lu et al., (2015)	Mg-3Zn-0.3Ca	6.9
Li et al., (2016)	Mg-3Zn-0.2Ca extr	1.2
Li et al., (2016)	Mg-4Zn-0.2Ca extr	1.3
Dimakopoulou, iCal (PE)	Med Zn extr peak aged	0.572738
Dimakopoulou, iCal (PE)	Med Zn extr	0.5339
Dimakopoulou, iCal (PE)	Med Zn extr	0.3705
Dimakopoulou, iCal (PE)	Med Zn peak aged	0.7027
Dimakopoulou, iCal (PE)	Med Zn peak aged	0.7843
Dimakopoulou, iCal (PE)	Med Zn	1.9061
Dimakopoulou, iCal (PE)	Med Zn	1.3887
Dimakopoulou, iCal (PE)	High Zn	1.3279
Dimakopoulou, iCal (PE)	High Zn	1.3917
Dimakopoulou, iCal (PE)	High Zn peak aged	0.6317
Dimakopoulou, iCal (PE)	High Zn peak aged	0.5391