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Dynamic degradation of porous magnesium under a simulated environment of human cancellous bone

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ABSTRACT

This study shows the effect of dynamic flow on the degradation behaviour and mechanical integrity of porous magnesium. A test rig that mimics the environment surrounding a cancellous bone was developed and a dynamic immersion test was performed to assess the degradation rate of the material for bone scaffold application. Three different percentages of porous magnesium (30%, 41%, and 55%) were immersed in simulated body fluid. The results show that mass loss and mechanical integrity of the specimens deteriorated linearly with an increase in porosity and degradation time, correlating to a drop of 41% and 89%, respectively, within 3 days.

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1. Introduction

Augment bone repair and regeneration requires a bone graft or scaffold. Annually, about 2.2 million bone scaffolds are used in orthopaedic procedures worldwide, mainly for stimulating new bone formation to replace and regenerate lost bone as a result of trauma, infection, or disease [1]. Among other materials for bone scaffolds, metallic biomaterials such as stainless steel, cobalt-chromium alloys, and titanium alloys are the ones most used when a mechanical load is present. However, despite their high mechanical strength and fracture toughness, they also come with disadvantages such as the possible release of toxic metallic ions

and poor stimulation of new bone growth due to elastic moduli mismatch [2,3]. These disadvantages have given rise to a new area of interest—the utilization of biodegradable metals. This type of metal is expected to fulfill its mechanical function and then degrade in vivo without causing any toxicological problems [4].

Compared to iron-based and newly introduced zinc alloys, magnesium and its alloys are the most investigated biodegradable metals for their potential application as biomedical implants [4,5]. This metal and its alloys possess interesting mechanical properties similar to that of human bone; the Young's modulus (41–45 GPa) of these metals is close to that of cortical bone (3–23 GPa) [6,7]. Besides that, magnesium has a low density and adequate strength-to-weight ratio [8,9]. From a bioactivity point of view, magnesium has a stimulatory effect on bone growth due to the formation of bone-apatite like hydroxyapatite crystals, which are quite favorable for bone strength [10–12]. The mechanical property of magnesium could be further manipulated to achieve the low Young's modulus of cancellous bone (0.01–2.0 GPa), thus turning it into a porous structure to match that of cancellous bone [13]. Porous magnesium has been shown to induce early vascularization

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Table 1

The chemical composition (in wt. ppm) of the commercially pure magnesium 99.9% purity.

| Element | Al | Zn | Fe | Cu | Mn | Ni | Si |
|---------|----|-----|-----|----|-----|-----|----|
| wt. ppm | 70 | <20 | 280 | 20 | 170 | <10 | 50 |

leading to good integration with tissue regeneration after gradual degradation [14,15]. Ideally, this porous structure will have 25–90% porosity and a 10–1000 μm pore size to provide the ideal condition for infiltration of essential nutrients, oxygen, and progenitor cells for cell survivability [1,16].

Once implanted, the porous magnesium scaffolds will be in contact with cancellous bone and will biomechanically adapt to the mechanical loading from the perpetual motion of physiological activities through the mechanobiological signalling of osteocytes [17,18]. The bone marrow, which is the home for progenitor cells of osteoblasts and osteoclasts, will move as a fluid medium with a flowrate range of 0.012–1.67 ml/min [19–21]. The interaction between the bone marrow movement and the cancellous bone structure induces mechanical stresses that stimulate the mechanobiological response to the bone quality and bone healing process [18]. The movement of bone marrow through the porous structure of cancellous bone due to pressure difference is generated by continuous cycles of mechanical loading from physiological activities [22]. This bone marrow movement in the cancellous bone must be considered as an actual boundary for testing the material of bone scaffolds. Unfortunately, all studies conducted on porous biodegradable metals for potential bone scaffold applications have been done under static immersion tests only [14,23–25]. Therefore, to address this gap, in this study, we integrated a biomechanical condition of cancellous bone for testing porous pure magnesium specimens under a dynamic immersion condition. The tests were performed with variations in time of immersion under a constant flow rate of Simulated Body Fluid (SBF), which represents daily physiological activities. The influence of the dynamic immersion condition on the degradation behaviour and mechanical property of the specimens will be the focus of our assessments. We believe that the results from this study will provide insight into the degradation behaviour of porous biodegradable metals when used as scaffolds, as they are tested in a more realistic, in vivo-simulating condition.

2. Materials and methods

2.1. Dynamic degradation test rig

Fig. 1a shows a schematic view of the test rig in this study that was designed to mimic the condition of daily physiological activities of bone marrow flow through a cancellous bone structure, which follows a laminar flow [18]. The design was inspired by the setup used in previous works on dynamic immersion tests of magnesium alloy for coronary stent applications including one

on the permeability study of cancellous bone [26,27]. The test rig created a fully laminar flow in a channel 41 mm in length (L) and provided a flow rate of 0.025 ml/min with a Reynolds number (Re) of 5.44 by regulating a pump with a flowrate capacity of 0.015–32 ml/min. The pump was connected to a 250 ml tank immersed in a water bath using a 2 mm inner diameter silicone tube. A specimen chamber with a 2-mm diameter (D) was placed in the channel to clamp and hold the specimen in place during testing. The specimen and the chamber had a gap of 2 mm, to ensure that the whole surface area of the specimen would be exposed to the fluid medium. Two pressure gauges (EMA, China) were placed before and after the specimen chamber to measure the pressure difference between suction and discharge, and connected to a data acquisition (DAQ—National Instruments, USA) system. A modified tee-connector was used to trap hydrogen gas coming out of the channel with the assistance of a manometer containing lubricant oil (SAE 5W-30, density of 860 kg/m³). As shown in Fig. 1d, the evolving hydrogen gas displaced the oil upward by Δh depending on the testing duration.

2.2. Preparation of porous magnesium specimens

A commercially pure magnesium rod with a diameter of 25.4 mm and 99.9% purity (Goodfellow Inc, Cambridge, UK) was cut into cuboid-shaped specimens measuring 5 × 5 × 3 mm. The chemical composition of the tested material as shown in Table 1. Inter-connected holes with varying porosity, as shown in Fig. 1d, were drilled into the cuboids using an 800 μm diameter drill bit of a CNC machine (HAAS, USA). The morphological indices of the specimens, including porosity and surface area, were determined using CAD models [28] and are presented in Table 2. Prior to outer surface grinding, any excess materials and chemicals were removed using air jets after which the specimens were immersed in acetone for 15 min. Interdental brushes (Tepe, USA), 0.6 mm and 0.8 mm in size, were then used to clean the internal surface of the specimens before their final immersion in acetone for another 15 min. The outer surface of the specimens was polished using an abrasive paper grit #1200, and then ultrasonically cleaned in acetone for 15 min, rinsed with acetone, and dried in a vacuum chamber for 1 h before being subjected to immersion tests.

2.3. Dynamic immersion test

The porous magnesium specimens were subjected to dynamic immersion tests using the test rig system shown in Fig. 1 using an SBF as per Kokubo et al. [29] that has a composition per litre as shown in Table 3. The pH of the SBF was adjusted to 7.4 using drops of 1.0 M HCl of up to 5 ml, and its temperature was maintained at 37 °C ± 1 °C; the flowrate was kept constant at 0.025 ml/min. The specimens were then subjected to dynamic immersion tests for periods of 24, 48, and 72 h. The SBF volume used in this study

Table 2

The morphologic details of the porous pure magnesium specimens.

| Type | Porosity | Surface area | Mass | Volume | Surface area per volume | Mass per Surface area |
|------|----------|------------------------|---------|-----------------------|-------------------------|------------------------|
| A | 30% | 189.30 mm ² | 82.8 mg | 52.87 mm ³ | 3580.48 m ⁻¹ | 0.44 kg/m ² |
| B | 41% | 209.81 mm ² | 70.3 mg | 44.57 mm ³ | 4707.43 m ⁻¹ | 0.34 kg/m ² |
| C | 55% | 225.75 mm ² | 53.3 mg | 33.83 mm ³ | 6673.07 m ⁻¹ | 0.24 kg/m ² |

Table 3

Composition for preparing 1 litre SBF solution.

| Reagents | NaCl | NaHCO ₃ | KCl | K ₂ HPO ₄ ·3H ₂ O | MgCl ₂ ·6H ₂ O | HCl 1.0M | CaCl ₂ | Na ₂ SO ₄ | Tris buffer |
|----------|---------|--------------------|---------|--|--------------------------------------|----------|-------------------|---------------------------------|-------------|
| Quantity | 8.035 g | 0.355 g | 0.225 g | 0.231 g | 0.311 g | 39 ml | 0.292 g | 0.072 g | 6.118 g |

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