Iron-based biodegradable alloys: importance of the testing parameters for corrosion evaluation via immersion tests

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INTRODUCTION: Cardiovascular diseases such as arteriosclerosis are widespread nowadays. Implanting a stent in the injured blood vessel is a common solution. Currently, permanent stents are used and could lead to long-term complications, such as restenosis. To avoid these issues, biodegradable stents are developed. They have to offer the required mechanical support to the blood vessel during the healing period (about one year), be biocompatible, and corrode at a suitable rate, i.e. in 1-2 years [1]. This work deals with the evaluation of the corrosion properties of iron-based alloys by immersion tests. It highlights the importance to follow a well established protocol to obtain reliable and reproducible results.

METHODS: The protocol has been defined for pure iron. Sheets were produced by melting pure iron pieces (Alfa Aesar, 99.99%) in an arc furnace. After a heat treatment of 10 minutes at 1000°C, the cast ingots were hot rolled to a thickness lower than 2mm. Since edges of square samples are favourable to corrosion, the chosen sample shape was a disk. Disks of 11mm in diameter were machined by electrical discharge machining. Both sides of the samples were polished and then cleaned in an ultrasonic bath of ethanol.

The samples were immersed in a pseudo-physiological solution (simulated body fluid, SBF), with a composition mimicking the ionic composition of blood plasma. The pH was in the range 7.3-7.5. Samples were placed in an oven at 37°C. The test duration was 7 days. The ratio of the volume of solution to the sample surface was kept constant and close to 20ml/cm^2. Samples were fixed in the flask, to avoid shocks and prolonged contact of a face with the flask. This prevents any disturbance on the stability of layers deposited/formed during the corrosion process.

The corrosion rate was estimated by released ion concentration, measured by ICP and by mass loss after removal of the corrosion products. The surface after corrosion was characterized by SEM+EDS. The influence of different parameters on corrosion was investigated. (a) The effect of the solution stirring was observed by comparing the immersion in static and stirred conditions. (b) Samples have been polished down to different levels: down to #1200 SiC paper, down to 1µm and down to 1µm followed by 1minute OPS. (c) The buffer has been modified: samples have been immersed in SBF alone (pH at 7.3-7.5 by adding HCl), SBF+50mM HEPES, and SBF+50mM TRIS.

RESULTS: We show that the considered parameters influence the corrosion rate. Corrosion is faster in a stirred solution than in static conditions. The polishing performed on the sample changes its roughness. It seems that samples polished down to 1200 grit are corroded more slowly than the ones polished down to 1µm or OPS. The buffering system has an important impact on corrosion. Indeed, adding 50mM of TRIS to SBF greatly increases the corrosion rate. No noticeable difference is observed between SBF alone and SBF+50mM HEPES in terms of corrosion rate.

DISCUSSION & CONCLUSIONS: Based on these results, the following protocol has been validated. Since blood circulates in the body, the solution is stirred, preventing the accumulation of corrosion products close to the sample surface. The stent surface being smooth, polishing is carried out down to 1µm. A solution buffered with 50mM HEPES is used to maintain a constant pH. A further improvement would be to test the physiological buffer, namely the HCO_3^-/CO_2 system.

By imposing specific parameters, we ensure the reliability and reproducibility of the experiments, enabling a comparison of the corrosion behaviour of different materials.

REFERENCES: [1] H.Hermawan, et al. (2010) *Acta Materialia* **6**:1852-1860.

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