

SODIUM CHLORIDE SOLUTION TEST OF Ti₆Al₄V IMPLANT SPECIMENS MANUFACTURED BY ADDITIVE TECHNOLOGY

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ABSTRACT

Additive technology is a valuable process for medical implant manufacturing. The most common metallic material in this area is the Ti₆Al₄V alloy. The research aimed to learn the behaviour of the implant material in saltwater environments. During the sodium chloride test, the weight loss of the test sample and the solute ion concentration are evaluated. The corrosion resistance tests were performed with a 3% concentration of NaCl solution at 36°C, with a duration of 4, 8, 12, and 16 days. The weight loss quality is determined by the test sample weight measured before and after the tests. The NaCl solution was also tested to characterise the most relevant quality element, which was retrieved from the implant material during the test. The solute metal ion concentration in the NaCl solution is determined by an inductively coupled plasma–optical emission spectroscope (ICP). Based on the experimental results, it can be declared that the weight loss increased as a function of time, and the measurable element was the aluminium in the NaCl solution. The chemical component of aluminium can cause many diseases and health problems. Aluminium can have various harmful effects on the human body, including neurological problems, lung disease, anaemia, bone and muscle damage, and skin inflammation. Some studies have also linked it to Alzheimer's disease and breast cancer. Untreated 3D-printed Ti₆Al₄V alloys show significant aluminium leaching, highlighting the need for surface modification before clinical application..

Keywords: corrosion, additive manufacturing, Ti₆Al₄V, ICP test, implant.

INTRODUCTION

Titanium alloys, in particular Ti₆Al₄V, are important alloys for dental and orthopaedic implants due to their excellent mechanical and biological properties (Haydar, et al., 2021; Wang, et al., 2020). In recent years, there has been increasing interest in the use of 3D powder bed metal deposition technology (selective laser melting, SLM) for the fabrication of Ti₆Al₄V medical implants instead of powder metallurgy, forging and casting processes. The 3D printing process increased the use of titanium alloy applications in several areas, such as aerospace and medical applications. The lightweight metal alloys have excellent mechanical properties such as strength and ductility.

Titanium and its alloys exhibit biocompatibility, which is a key factor in their importance in medical technology, such as implants (Naghavi, et al., 2022). It is well understood and tested that different chemical elements in the functioning of the human body can be beneficial, harmful, or toxic to healthy functioning. In the case of the Ti₆Al₄V alloy, aluminium is the most dangerous element and is responsible for the health disease (Jomova, et al., 2022).

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Titanium alloy, Ti6Al4V, is widely used for dental implants due to its lightweight, high strength, and biocompatibility (Nicholson, 2020). However, there is concern that over time, the implant will corrode and may release aluminium and vanadium ions into the body (Noumbissi, et al., 2019; Tyczyńska, et al., 2024; Qureshi, 2021).

Few studies show that the release rate of the ions depends on several factors, such as the acidity level of the mouth environment and the mechanical pressure applied to the implants (Delgado-Ruiz, & Romanos, 2018; Saha, & Roy, 2022). Arregui et al. reported that the number of aluminium ions released may be small, but long-term exposure still raises questions about the safety of the implants, as elevated levels of aluminium can be toxic and may lead to renal failure (Arregui, et al., 2021). It is noted that the acceptable range of aluminium in the blood is typically less than 16 µg/L (Sjögren, et al., 2015). Arregui et al. also concluded that while the number of aluminium ions released increased over time, the amount of vanadium ions released was negligible in the case of the Ti6Al4V alloy experimented with (Arregui, et al., 2021; Laupheimer, et al., 2024).

The researchers tested the Ti6Al4V alloy corrosion resistance by several methods to determine its corrosion behaviour (Sharp, et al., 2022; Chen, et al., 2022). In the case of this alloy, a wear damage phenomenon was detected. The wear process causes weight loss (Nsiah-Baafi, et al., 2022). The porosity level of the 3D printed materials can be the reason for the weight loss. The electrolyte during the corrosion tests can penetrate the pores, and in this way, the contact surface is much greater than in the case of a metal without porosity (Morris, et al., 2021). The surface roughness and the porosity level can increase the osseointegration of the implant (Kia, et al., 2022). It can be found that the surface of the 3D-printed metal is a cardinal point in the implants. The corrosion resistance of titanium alloys is due to the passive layer formed on their surface. The surface preparation of the 3D-printed sample depends on the function of the implant (Kónya, et al., 2024). In our experiments, we wanted to quantitatively and qualitatively determine the weight loss of the 3D-printed titanium alloy.

MATERIAL AND METHODS

Test specimen

The test samples were made by a fiber laser SLM device (Sisma MYSINT 100). They used parameters of 3D printing on the base of the recommendation by the Sisma company: 125 W laser power, 55 µm focus, 1000 mm/s scanning speed and 20 µm layer thickness. During the printing process, Argon shielding gas was used (35 L/min). The powder material used was plasma-spraying spherical powder (Grade 5) made by LPW Technology (Runcorn UK). The powder size distribution was between 15 µm and 45 µm, and the density was 4.43 g/cm³. The chemical composition of the powder is shown in Table 1.

Table 1. Chemical composition of the used Ti6Al4V (wt. %)

Alloy	Al	V	Fe	O	N	C	H	Ti
wt. %	6.11	4.02	0.17	0.09	0.023	0.01	0.003	Balance

The specimens were cleaned after 3D printing, without heat treatment and any surface treatments were applied. The prismatic test sample sizes were 10x7x2 mm (0.6200 g). It was used for all experiments, a new test sample of the same size and made by the same process.

The printed test sample is shown in Figure 1. The untreated surface of the printed specimen can be seen.

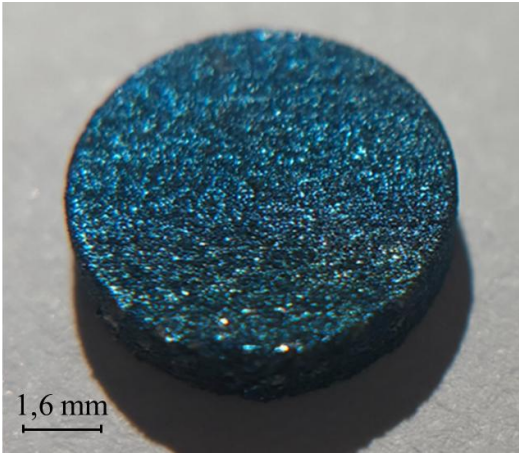


Figure 1. The used test sample geometry and size (illustration).

Weight loss corrosion test

The weight loss experiments were carried out in an aqueous sodium chloride solution (3%) at 36°C and used different durations: 4 days, 8 days, 12 days, and 16 days (on three identical samples for all time periods). After the experimental time ran out, the metal specimens were removed from the solution, washed with distilled water, and dried by airflow (Haraszi, et al.. 2022)

The weight loss was determined by Kern, AIJ 220-4MN analytical balance, with a resolution of 0.0001g.

Determination of the dissolved metal ions

An inductively coupled plasma–optical emission spectrometry (ICP-OES) was used to determine the dissolved ions in the NaCl solution in a corrosive environment. The instrument used in the investigated experiments was a Spectro Genesis ICP-OES spectrometer (with axial plasma observation). The plasma is very stable due to the unique operating frequency and broad bandwidth (wavelength range of 175–775 nm), which is equipped with a CCD detector; in this case, this method is suitable to determine about 30 elements at the same time (ranging from g/L up to percentage level).

RESULTS AND DISCUSSION

The corrosion resistance test results were evaluated using two different measurement methods. The weight loss is determined by measuring the weight (g) and identifying the dissolved ions.

Corroded material quantity determination

The corrosion level is determined by quantity measurement. The results of the weight loss measurements are shown in Table 2. It can be clearly seen that the weight loss increased during the test period. The corrosion caused a continuous weight decrease in the test samples.

Table 2. Weight loss test results in NaCl solution 3% at 36°C.

Duration (day)	Weight loss Δm (g)
4	0.0001 g
8	0.0003 g
12	0.0006 g
16	0.0013 g

The corrosion process can be described by a good approximation of the points of the obtained values. The corrosion process is characterised by the following function (1):

$$y = 6 \cdot 10^{-6} \cdot x^3 - 6 \cdot 10^{-4} \cdot x^2 + 0.0002 \quad (1)$$

Figure 2 shows the weight loss tendency as a function of the treatment time. The weight loss increases due to the NaCl solution treatment time at 36°C. The used NaCl solution was 5 mL in all tests.

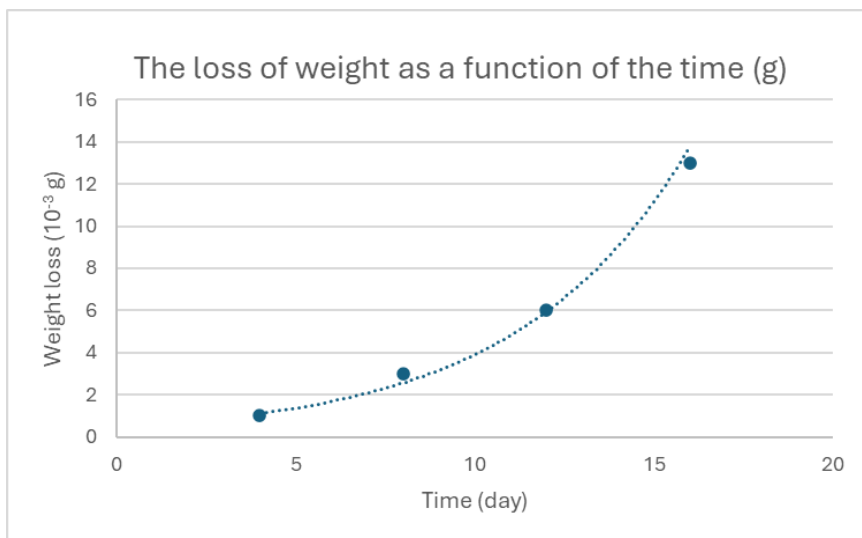


Figure 2. The weight loss as a function of time in 3% NaCl solution.

The weight loss data of the titanium alloy investigated unequivocally show that the corrosive dissolution increases over a longer time.

Corroded material quality determination

By the plasma–optical emission spectrometry method, it was determined that the dissolved elements. Only the dissolved aluminium was detected in the main elements of the alloy. Table 3 shows the dissolved aluminium quantity.

Table 3. The plasma–optical emission spectrometry results from the dissolved ions in 3% NaCl solution at 36°C.

Duration (day)	Al ions mg/L (ppm)
4	0.003
8	0.004
12	0.013
16	0.014

Figure 3 shows the dissolved aluminium ion quantity (ppm). It can be seen that the dissolution rate of the aluminium is different from the corrosion rate as a function of the weight loss (Figure 3). In the case of the aluminium dissolution, it is difficult to determine any function over time. Even the vanadium and titanium dissolution detection was impossible because of the measurement limit, but it can be supposed that the dissolution rate of the different elements of the alloy is different. It can find some research results which determine the dissolution rate of vanadium and suggest that the dissolution of vanadium begins rapidly, but after, as a function of

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time, the dissolution rate decreases (U.S. Department of Health and Human Services, 2025; Ghisheer, et al., 2024).

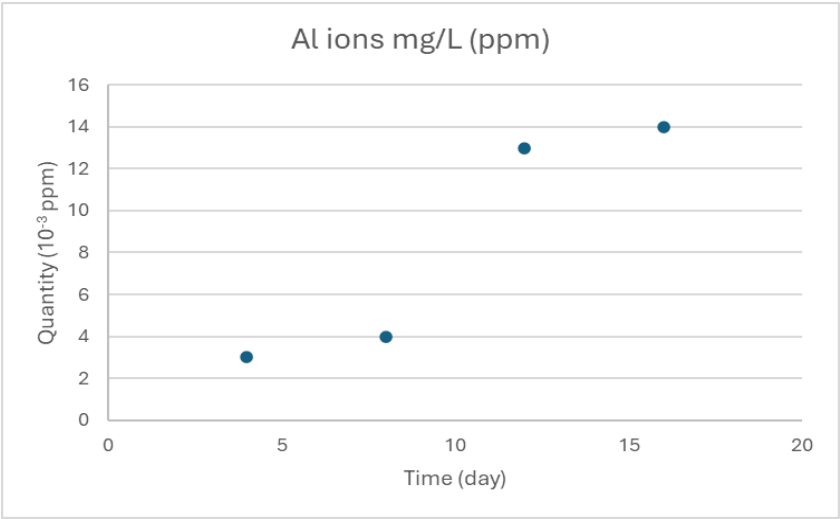


Figure 3. The weight loss as a function of time in 3% NaCl solution.

No results on aluminium leaching have been found in the literature, which was determined based on the examination of Ti6Al4V 3D printed implant materials. The results suggest that further, longer-term experiments are needed to determine the other elements that leach from the alloy and to understand the kinetics of corrosion. It should be noted that from the weight loss, we can only infer general corrosion, but from the amount of ion leaching, we have determined that it is the harmful aluminium that is typically present in the brine.

CONCLUSIONS

The amount and quality of material loss were determined for the alloy under investigation. Based on this, it can be concluded that the use of 3D-printed Ti6Al4V alloy after printing is not recommended due to the leaching of aluminium, which causes health damage. However, the roughness of the printed surface would be acceptable in terms of osseointegration. However, it needs to be investigated which of the different surface treatment methods can help prevent aluminium leaching from the 3D printed sample surface and make the implants suitable for the patients. Our aim is to continue the research by investigating the effect of different surface treatment processes on corrosion resistance.

DECLARATIONS OF INTEREST STATEMENT

The authors affirm that there are no conflicts of interest to declare in relation to the research presented in this paper.

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