

Changes in compressive strength of Ti-6Al-4V during immersion in artificial saliva at pH 6.5 and dynamic treatment

Harly Prabowo *, Adinda Putri Salsabila and Nadia Ella Veradilla

Department of Prosthodontic, Faculty of Dental Medicine, Airlangga University, Surabaya, Indonesia.

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Abstract

Background: Dental implant materials have been developed over time and become an alternative to dental treatment. One of the materials, Ti-6Al-4V, is widely used. The pH saliva in the oral cavity can change due to various factors, thereby affecting the implants durability. This research was conducted to determine the durability of Ti-6Al-4V implants based on their effect on the oral environment.

Objective: To analyze change of compressive strength of Ti-6Al-4V in artificial saliva pH 6.5 and dynamic stress treatment.

Material and Methods: Laboratory experimental and analytic research was conducted with 27 Ti-6Al-4V implant samples, which were classified into 3 groups, each consisting of 9 samples, including those with no treatment, aquades solution, and artificial saliva with pH of 6.5. In the negative control group, the samples were treated dynamically, while the samples in the positive control group were not treated. Samples from the treatment group were subjected to a dynamic test in accordance with ISO 14801 standards while being immersed in artificial saliva with pH of 6.5. Thereafter, a compression test was conducted on each group. A one-way ANOVA test was used to analyze the comparison of the results.

Results: The results of data analysis using One-Way ANOVA found a significance value of less than 0.05 (Sig<0.05). This shows that there is no significant difference between each group and the average maximum compression value was $T1 < T3 < T2$.

Conclusion: There were no significant changes in compressive strength of Ti-6Al-4V after immersion test with artificial saliva pH 6.5 and dynamic treatment.

Keywords: Ti-6Al-4V; Artificial saliva; Immersion test; Compressive strength; Dynamic pressure treatment

1 Introduction

Implants are a treatment option for tooth loss. The ideal implant material should have high biocompatibility, low corrosion rates, and adequate fracture resistance. Titanium (Ti) dental implants are considered the "gold standard" biomaterial for prosthetic treatment [1]. According to the American Society for Testing and Materials (ASTM), there are six types of titanium available in the market: four grades of pure titanium (CpTi) (grade I - grade IV) and two titanium alloys (grade V Ti-6Al-4V and Ti-6Al-4V-ELI - Extra Low Interstitial Alloys). The mechanical and physical properties of CpTi depend on the oxygen residue present in titanium. Pure titanium contains trace elements such as carbon, oxygen, nitrogen, and iron, which can enhance the mechanical properties of pure titanium. Ti-6Al-4V is a widely used titanium grade V in the field of dentistry because of its good mechanical and physical properties, making it suitable for use as an implant material [2].

* Corresponding author: Harly Prabowo

Titanium implants have a high survival rate, ranging from 97-99% [3]. Despite the high survival rate, titanium implants are not immune to the risk of failure, which can be caused by biological and mechanical factors. The most common biological factor is related to inadequate osseointegration due to bacterial infection. Untreated infection can spread to the bone tissue around the implant, leading to peri-implantitis [4]. Implant failure due to mechanical factors includes loose screws and fractures of implant components or prosthetic teeth [5].

In the oral cavity, saliva serves as a protector to maintain the health of teeth and the oral environment. It also plays a crucial role in balancing the pH to keep it optimal, typically ranging from 6.5 to 7.5, with an average of 6.7, this balance helps inhibit the process of demineralization, contributing to overall oral health [6].

Changes in saliva pH in the oral cavity can be caused by the influence of bacteria, enzymes, and various other factors. A low saliva pH can lead to tooth roughness, contributing to the development of cavities, while a basic pH can result in the formation of dental plaque. The variation in pH within the oral cavity can impact the durability of metal implants. Implants in the human body may undergo degradation when exposed to environments with fluctuating temperatures, pH variations, high humidity, and other microorganism-related factors [7].

To test the durability of a metal, this research utilizes an immersion test of implants in artificial saliva with an optimum pH of 6.5. Artificial saliva is employed because it serves as a substitute for saliva in the oral cavity, acting as a buffer solution, moisturizing oral tissues, and possessing chemical and physical properties similar to human saliva [8]. Furthermore, the selection of an optimum pH of 6.5 is made because it is assumed to represent the oral cavity environment under optimal conditions that can inhibit demineralization. However, according to some literature, it is mentioned that bacterial growth still occurs at this optimum pH of 6.5 [6].

After conducting the immersion test, a dynamic fatigue test for dental implants was carried out using the ISO 14801 standard, with a total of 2 million loading cycles assumed to represent two years of dental implant usage. Following this, a compression test will be performed to determine the mechanical properties value of the implant [9].

This research is based on cases involving healing abutments, where the surface of these abutments often undergoes changes in different oral environments. Thus, this study aims to identify the types of oral environments that can cause surface changes in the neck portion of Ti-6Al-4V implants.

2 Material and methods

This research is considered experimental laboratory research with post-test only group designs. Sample in the form of a titanium Grade V (Ti-6Al-4V) implant, screw type, produced for IKG Prof. R. Hartono by PT. Marthys Orthopedic Indonesia with a diameter of 3.25 mm and a length of 26 mm.

The samples are divided into three groups: the first group undergoes no immersion testing and dynamic treatment, the second group undergoes immersion testing with distilled water (aquades) and dynamic treatment, and the third group undergoes immersion with artificial saliva at pH 6.5 and dynamic treatment. Subsequently, all three groups will undergo compression testing to determine the changes in mechanical properties (compressive strength).

The entire implant specimen was provided with an implant holder using a cylindrical Brass Rod Holder measuring 12mm x 20mm with a hole drilled to accommodate the Ti-6Al-4V implants. The implants were affixed to the Brass Rod Holder using DEVCON® Plastic Steel Putty (A) as the embedding material. The next step involved crafting the specimen head made of VERABOND® Nickel Chromium (Vera-bond, Aalba Dent, Inc., Fairfield, CA) in a hemisphere shape.

Each sample will be placed in a container filled with artificial saliva with a pH of 6.5 for immersion testing. Each specimen is filled with a solution according to the ASTM G31-72 standards, which is at least (0.2 to 0.4) times the sample surface area. In this study, approximately 1 liter of artificial saliva and distilled water (aquades) will be used. To determine the pH conditions of artificial saliva before and after treatment, measurements will also be conducted using a pH meter.

The Dynamic Fatigue Testing Machine used in the research is the Hung Ta Load Cell (Type HT-9711T5, Hung-Ta Instrument Co.,Ltd, Taipei, Taiwan; Li et al., 2021). The Dynamic Fatigue Testing Machine is set according to ISO 14801 by positioning the implant along the longitudinal axis at a 30° angle, with a hemispherical loading member attached to the implant. The applied pressure on the research specimen is 50 N. The frequency set on the Dynamic Fatigue Testing Machine during testing is 12 Hz following ISO 14801 standards. The number of loading cycles administered is 2 million, assuming dental implant usage for 2 years.

The assembled samples are then mounted on the jig of the Dynamic Fatigue Testing Machine, and dynamic pressure treatments are applied. Each sample requires approximately 2 days for the completion of dynamic pressure treatment, resulting in a total time of around 54 days for the dynamic pressure application. After subjecting the specimens to dynamic pressure treatment, compressive strength testing is conducted using the Hung Ta Type HT-9501 universal testing machine to determine the mechanical properties of the Ti-6Al-4V implant specimens.

2.1 Statistical Analysis

The data from this research were analyzed using normality testing with the Shapiro-Wilk Test, homogeneity testing with the Levene test, and One-Way ANOVA to assess differences between the control group and the treatment group. The data were then statistically processed using IBM SPSS Statistics 25.

3 Results

3.1 Research Data

This research is conducted through an analytical experimental approach in the laboratory, starting with the fabrication of Ti-6Al-4V implant specimens as shown in Figure 1. The main objective of this study is to observe the changes in the compressive strength of Ti-6Al-4V implants using the Hung Ta Type HT-9501 universal testing machine after immersion in artificial saliva with a pH of 6.5 and dynamic treatment following ISO 14801 standards.



Figure 1 The assembled implant with brass rod and hemisphere before undergoing further testing



Figure 2 The immersion process with artificial saliva at pH 6.5 and dynamic treatment following ISO 14801 standards with a total of 2 million load cycles

Ti-6Al-4V implants were then subjected to compression testing to obtain the maximum compression values from the specimens. From this treatment, average results and standard deviations of the sample groups were obtained as shown in the following table.



Figure 3 The compressive strength testing process on Ti-6Al-4V implants using the Hung Ta Type HT-9501 universal testing machine

Table 1 Research Data

Specimen	Maximum	Compression Value	
	Without Treatment	Aquades	pH 6.5
1	175,2900	179,2900	182,9800
2	172,8467	17,8874	182,6956
3	180,3007	178,5799	175,9449
4	182,5383	182,8945	179,6656
5	170,7430	179,8877	176,0682
6	173,1990	177,0476	178,0128
7	170,5039	180,0341	175,9952
8	181,6027	178,7512	180,7078
9	170,7064	178,2615	174,7381
Average	175,3034	179,2926	178,5343
Standar Deviasi	4,9044	1,6181	3,1024

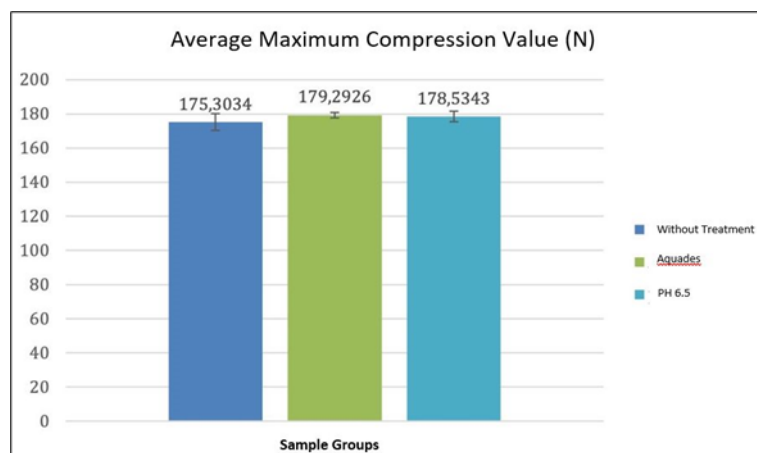


Figure 4 Graph of the average maximum compression values and standard deviation

In the Untreated group, the maximum value is 182.5 N, the minimum is 170.1 N, and the average is 175.3 N. In the Aquades group, the maximum value is 182.8 N, the minimum is 177.04 N, and the average is 179.2 N. In the pH 6.5 group, the maximum value is 182.9 N, the minimum is 174.7 N, and the average is 178.5 N. It can be observed that the average maximum strength of Ti-6Al-4V implants, after immersion in artificial saliva with a pH of 6.5 and dynamic treatment to withstand loads, yields higher results compared to the control group or those without treatment. However, it is lower than the group subjected to immersion in distilled water (aquades).

After obtaining the maximum compression values, the data from this research can be subjected to descriptive analysis, as shown in Table 2.

Table 2 Descriptive Analysis

	N	Mean	Std. Dev	Std. Error	95% Confidence Interval for Mean		Min	Max
					Lower Bound	Upper Bound		
Without Treatment	9	175.3034	4.9044	1.6348	171.533	179.073	170.50	182.54
Aquades	9	179.2926	1.6180	0.5393	178.048	180.536	177.05	182.89
pH 6.5	9	178.5342	3.1024	1.0341	176.149	180.919	174.74	182.98
Total	27	177.7101	3.7782	0.7271	176.215	179.204	170.50	182.98

3.2 Analysis of Research Findings

Data from the compression testing, once obtained, underwent assumption testing in this subsection. The assumption tests that can be used in One-Way ANOVA statistical analysis are normality and homogeneity of variance tests. These assumption tests aim to determine the presence of differences in the measurement results among the untreated sample group, aquades, and pH 6.5. In Table 3, the results of the normality assumption test using the Shapiro-Wilk test are presented.

The results of the normality test using Shapiro-Wilk are as follows:

Table 3 Normality Test

	Treatment	Shapiro-Wilk		
		Statistik	df	Sig.
Maximum Compression Value	Without Treatment	0.844	9	0.064
	Aquades	0.894	9	0.219
	pH 6,5	0.898	9	0.242

Normality testing was conducted using the Shapiro-Wilk test, and the results for the Untreated group $p=0.064$, Aquades $p=0.219$, and for pH 6.5 $p=0.242$. These results indicate that the research data for all groups follow a normal distribution.

After conducting the normality test, the next step is to perform the homogeneity of variance test using the Levene test. The results of the homogeneity test using Levene are as follows:

Based on Table 4, the data obtained from the measurements generally have significance values greater than 0.05, which resulted in a p-value of 0.065. This indicates that the variance data is homogeneous.

The assumptions in the One-Way ANOVA testing have been met, allowing the One-Way ANOVA test to be conducted. The results of this test are presented in Table 5.

Table 4 Homogeneity of Variance Test

		Levene Statistic	df1	df2	Sig.
Maximum Compression Value	Based on Mean	7.720	2	24	0.003
	Based on Median	3.379	2	24	0.051
	Based on Median and with adjusted df	3.379	2	13.336	0.065
	Based on trimmedmean	7.450	2	24	0.003

Table 5 One-Way ANOVA Test

Maximum Compression Value					
	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	80.782	2	40.391	3.338	0.053
Within Groups	290.374	24	12.099		
Total	371.156	26			

Based on Table 5. the data obtained p-value is 0.053. Therefore, it is concluded that there is no significant difference in the average maximum compression values among the various combinations of treatments given.

Based on the earlier One-Way ANOVA test, it was found that there is no significant influence between the independent variable and the dependent variable. This is indicated by the non-significant test results. The same can be observed from the post-hoc test using Tukey-HSD. The results of the Tukey-HSD test are presented in the following table.

Table 6 Multiple Comparisons of Tukey-HSD Test

(I) Treatment	(J) Treatment	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
Without Treatment	Aquades	-3.98924	1.63971	0.057	-8.0841	0.1056
	pH 6.5	-3.23083	1.63971	0.141	-7.3257	0.8640
Aquades	Without Treatment	3.98924	1.63971	0.057	-0.1056	8.0841
	pH 6.5	0.75840	1.63971	0.889	-3.3364	4.8532
pH 6.5	Without Treatment	3.23083	1.63971	0.141	-0.8640	7.3257
	Aquades	-0.75840	1.63971	0.889	-4.8532	3.3364

The mean difference values in Table 6. represent the average difference between two compared treatment groups. To determine whether the values obtained from two treatment groups differ significantly, the Sig value can be used. If the Sig value < 0.05, it indicates a significant difference. In the pH 6.5 treatment group, when comparing the maximum compression values with the untreated group, the values are not significantly different because Sig. = 0.141, which means Sig. > 0.05. If the pH 6.5 group is compared with the aquades group, the values are not significantly different because Sig. = 0.889. The same applies to the others. Additionally, there are mean difference values for each result. In the pH 6.5 group compared to the untreated group, the positive mean difference value is 3.23083. This means that the maximum compression value in the pH 6.5 group is higher than the value obtained in the untreated group by 3.23083.

Conversely, if the mean difference value is negative, it means that the maximum compression in the first group is lower than the maximum compression in the second group.

To facilitate comparison between treatment groups, this Tukey-HSD test can be summarized and presented in the form of homogeneous subsets, as shown in Table 7.

Table 7 Homogenous Subsets on Tukey-HSD Test

Treatment	N	Subset for alpha = 0.05		
		1	2	3
Without Treatment	9	175.3034		
Aquades	9	178.5342		
pH 6,5	9	179.2926		
Sig.		0.057		

Based on Table 3.7, it is found that 1 subset or group of effects is formed. Treatments in the same subset indicate that the values are not significantly different. Meanwhile, treatments located in different subsets indicate that the values are significantly different. In other words, statistically, it can be concluded that there is one common effect, so among the treatments in this study, they either have the same effect or are not significantly different.

4 Discussion

Dental implants made from Titanium Grade V Ti-6Al-4V are the preferred choice for implant materials due to various advantages such as excellent mechanical properties, good corrosion resistance, high fatigue strength, and a low Young's modulus. These characteristics contribute to the success and long-term sustainability of dental implant usage [10]. In the oral cavity, various factors can influence changes in saliva pH levels, including dietary factors, oral hygiene products, smoking habits, systemic diseases, and radiation to the salivary glands [7].

In the oral cavity, various factors can influence changes in the pH level of saliva, including food factors, oral hygiene products, smoking habits, systemic diseases, and radiation to the salivary glands [7]. This can be affected by the rate of saliva flow, saliva buffer capacity, and microorganisms in the oral cavity [11]. Changes in the pH level in the oral cavity can affect the durability of applied implant metals. Changes in the pH level in the oral cavity may indirectly relate to the applied implant. Due to the interaction between the oral cavity pH environment and implant metals, this study aims to test the resistance of a metal based on its influence on the oral cavity environment. The use of artificial saliva serves as a substitute for saliva function in the oral cavity, playing a role as a buffer solution and moisturizing the oral tissue [8]. Additionally, pH 6.5 can be assumed to represent the optimum oral cavity environment that inhibits the demineralization process. However, within the pH range of 6.5 – 7.5, bacterial growth in saliva is also present. Therefore, this study employs immersion and dynamic treatment tests on implants with artificial saliva at the optimum pH of 6.5 to determine if there are changes in the compressive strength of Ti-6Al-4V [6].

Based on the research results for each treatment group after dynamic testing using ISO 14801 standards with a load of 50N, 2 million loading cycles, and a frequency of 12 Hz, further compression testing was conducted on the samples using the Universal Testing Machine Hung Ta Type HT-9501. The average maximum compression values obtained were 175.3034 N for the untreated control group, 179.2926 N for the aquades group, and 178.5343 N for the sample using artificial saliva pH 6.5 [9].

In the statistical results of the One-Way ANOVA in Table 5.5, it is observed that there is no significant influence in the testing of the untreated group, aquades, and artificial saliva with pH 6.5. The non-significant data in the table could potentially be attributed to various factors, such as minimal differences in the pH between artificial saliva with pH 6.5 and aquades, where aquades has a neutral pH of 7 [12]. Variations in pH within the oral cavity can impact the durability of metal implants. Environmental factors such as fluctuating temperatures, changes in pH levels, high humidity, and the presence of other microorganisms can lead to unstable metal changes and increased degradation [7].

In its application in the oral cavity, the mechanical strength of an implant can be influenced by several factors, including the load it experiences during chewing and physiological conditions such as osseointegration and alveolar bone resorption. In this context, the alveolar bone plays a crucial role in providing support to the teeth and achieving optimal esthetic and functional outcomes in prosthetic reconstruction [13].

In this study, a one-piece implant type was utilized, where the abutment is integrated with the implant, forming a single unit. In its application, this implant is inserted into the bone with the abutment part positioned above the gingiva (non-submerged) [14]. The implant specimens are mounted on a holder with a 30-degree off-axis inclination, following the standards for fatigue testing on implants and abutments (ISO 14801) to simulate the worst-case scenario of implant placement, including the method used in "All-on-four" implant placement. In the "All-on-four" system, two anterior implants are placed perpendicular and parallel to each other, while two posterior implants, especially in the mandible, are inserted with an inclination of 30-45 degrees to reduce the risk of pressure accumulation, bone resorption at the implant-bone interface, and to achieve good bone anchorage [15]. The magnitude of the inclination angle and the applied load cycles on the implant is implemented until the specimen experiences fatigue leading to subsequent fracture [9]. Most mechanical failures in implant restorations are attributed to this fatigue. The failures can impact the abutment area, implant body, or the implant screw, depending on the implant diameter, load case, and implant position [16].

In the case of one-piece implants, the 10-year period of usage does not lead to structural damage or failure. This is evidenced by a survival rate of 96.83% [17]. This indicates that the average load cycles tolerated by humans using the implant do not exceed 6-10 years, depending on the implant type. This duration correlates with the number of chewing load cycles that the implant can endure, which is 6-10 million cycles. Excessive accumulation of chewing cycles may lead to deformation. If the number of load cycles in this study is increased from 2 million to more than 10 million cycles, there is a possibility that the implant strength could decrease, potentially resulting in fatigue cracks and fractures in the implant.

The ISO 14801 standard provides clearer guidelines regarding the frequency and loading cycles used. For testing in dry conditions, it recommends applying 5 million load cycles with a maximum frequency of 15 Hz. However, in wet conditions, the suggested number of load cycles is 2 million, and the frequency is only 2 Hz. Therefore, in this research, there is no significant change observed in the implants because the testing has some limitations that need to be adjusted accordingly.

5 Conclusion

From the results of the conducted research, Ti-6Al-4V implants under the conditions of immersion in artificial saliva with a pH of 6.5, dynamic loading of 50N, 2 million load cycles, and a frequency of 12 Hz do not exhibit significant trends in changes in compressive strength compared to the aquades group or the untreated control specimens.

Compliance with ethical standards

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Disclosure of conflict of interest

All authors declare that they have no conflicts of interest.

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