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RESEARCH ARTICLE

Influence of sulfuric acid concentration on the surface roughness of titanium alloy plates

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ABSTRACT

The surface roughness of an implant material (titanium alloy) is an important factor in optimizing osseointegration. Various efforts have been made to optimize the roughness of implant materials, such as acid etching. Previous research showed etching with sulfuric acid (H_2SO_4) at a temperature of 60 °C for 60 minutes could increase roughness. However, further research is needed regarding the concentration of H_2SO_4 that can optimize surface roughness. This research aimed to determine the concentration of H_2SO_4 that can be used to obtain optimal roughness. This study used a pre-test and post-test group design by immersing titanium alloy plates in a solution of saline water (negative control), 12.17 M HCl (positive control), and H_2SO_4 at various concentrations, namely 6.94 M, 9 M, and 11.06 M for 60 minutes at 60 °C. Next, a surface roughness test and Scanning Electron Microscopy (SEM) were carried out. The research results were analyzed statistically using the Shapiro-Wilk, Wilcoxon, Kruskal-Wallis, and Mann-Whitney tests. The results showed that in the H_2SO_4 group, there were significant differences before and after treatment ($p < 0.05$). The higher the concentration of H_2SO_4 used, the higher the surface roughness value. The SEM test showed that the group with a higher H_2SO_4 concentration had a rougher topography and more visible grooves. In conclusion, etching with H_2SO_4 can increase the surface roughness of titanium alloys, and H_2SO_4 at a concentration of 11.06 M led to the highest roughness value in this study.

Keywords: acid etching; dental implant; sulfuric acid; surface roughness; titanium alloy

INTRODUCTION

Tooth loss due to caries, periodontal disease, or trauma can cause problems such as psychological, social, and physical disorders, thereby declining the quality of life.[1] There are several treatments to replace missing teeth, such as removable dentures, fixed dentures, and dental implants.[2]

Removable dentures offer the quickest and most cost-effective treatment option but may lack comfort and ease of cleaning. On the other hand, fixed dentures provide comfort but require preparation for neighboring teeth and can be challenging to clean.[2] Dental implants offer comfort without affecting neighboring teeth and simplify maintenance compared to fixed and removable dentures, making them a favorable treatment choice.[3,4]

Successful use of implants is characterized by osseointegration.[4] A critical factor in the osseointegration process is the surface roughness of the implant material.[5,6] Most commercial implant systems have surface roughness ranging from 1 to 2 μm.[7] The implant material widely used in dentistry is titanium alloy (Ti-6AL-4V) because it has basic properties such as biocompatibility, biomechanics, and good strength to withstand occlusal pressure.[5,8] Various efforts have been made to develop materials that can optimize osseointegration, one of which is by modifying the surface of titanium alloy using the acid etching method.[5]

Researchers use acid etching to modify the surface of dental implants using a strong acid treatment. Acid etching can cause microporosity to form on the surface of the implant material and increase roughness on the surface of the implant material.[6] This causes activation of osteoblast transcription factors by cells around the implant.[9] This method is also known to be able to remove the oxide layer and contamination.[10,11] Etching with sulfuric acid (H_2SO_4) causes the surface of titanium to be rougher than other acid types (HCl, H_3PO_4, HF, and HNO_3).[12] Apart from that, based on research by Chauhana P et al and Al-Radha, etching with sulfuric acid at 60 °C for 60 minutes can increase the average value of the surface roughness of the implant material.[5,6] However, it is necessary to know more about the concentration of sulfuric acid that can be used to obtain optimal roughness. Therefore, this study aimed to investigate the influence of sulfuric acid (H_2SO_4) with three concentrations at a temperature of 60 °C for 60 minutes on the surface roughness of titanium alloy plates.

MATERIALS AND METHODS

This was a laboratory experiment with a pre-test and post-test group design. This research obtained ethical approval from the Health Research Ethics Commission, Faculty of Dentistry, Universitas Trisakti, with number 670/S1/KEPK/FKG/7/2023. The research was conducted from August to December 2023.

The sample was titanium alloy (Ti-6Al-4V) in the form of a plate with dimensions of 10 x 10 x 2 mm. The samples were first measured for initial roughness using a surface roughness test (S-100 series, Taylor Hobson, United Kingdom) at the Dental Material and Testing Center of Research and Education (DMT-CORE), Faculty of Dentistry, Universitas Trisakti.[12]

The roughness measurements were carried out by fixing the sample on a glass plate and then placing the sample under the stylus of the S-100 series surface roughness tester. Once the start button is hit, the stylus will move automatically, and the Ra results will be displayed in micrometers (μm) on the monitor. The measurements were carried out in triplicate on different surface areas to obtain Ra_1, Ra_2, and Ra_3 values from one sample. The surface roughness value was obtained from the average of the three roughness values. The measurements were carried out on all the samples from each group, and the values obtained were averaged to get the total average value from one group.[13,14] Then, the samples were sent to the Integrated Research and Testing Laboratory, Universitas Gajah Mada, for scanning electron microscopy testing (SEM Jeol JSM-6510, Akishima, Tokyo, Jepang) to see the morphology of the pre-test samples.

Next, the samples were divided into five groups, with each containing five samples. The samples were immersed in saline water (negative control), 12.17 M HCl (positive control), 6.94 M H_2SO_4, 9 M H_2SO_4, and 11.06 M H_2SO_4 in labeled containers. The immersion was done in an oven (Ov-30, *PT. Prioritas Bangun Nusantara,* Tangerang City, Banten) for 60 minutes at 60 °C.[12] Then, the samples were rinsed with distilled water and dried at 37 °C in an incubator (LIB-080M, Labtech, Korea). The final roughness measurement was carried out on the samples using a surface roughness tester, and scanning electron microscopy testing was carried out again with magnifications of 500x, 1000x, 3000x, and 5000x.[6,12]

SEM testing was done by testing the sample using scanning electron microscopy (SEM) by placing it in a specimen holder and coating it with a layer of gold and palladium (Au/Pd).[15] Then, the specimen holder was placed on the SEM tool; the computer screen then showed the morphology of the sample.[16]

The normality of the data was tested using the Shapiro-Wilk method because the samples used in this study were < 50. If the data results are not normally distributed ($p > 0.05$), the Wilcoxon test is then carried out to determine the differences before and after treatment for each group and the Kruskal-Wallis test to determine the differences between groups. If the test results show a significant difference, the Mann-Whitney test is carried out. Apart from that, the SEM test results were presented descriptively.

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RESULTS

The sample (Ti-6Al-4V) was tested using Energy-Dispersive X-ray Spectroscopy (EDS) at the Integrated Research and Testing Laboratory, Universitas Gajah Mada, to determine the composition of the sample to be studied prior to treatment (Figure 1). The results of the EDS analysis showed that there were elements such as Al, Ti, V, and Fe, which are components that form titanium alloy (Ti-6Al-4V). This analysis also showed that Ti was the largest element that has a role in the formation of Ti-6Al-4V, with an atomic number of 98.96%.

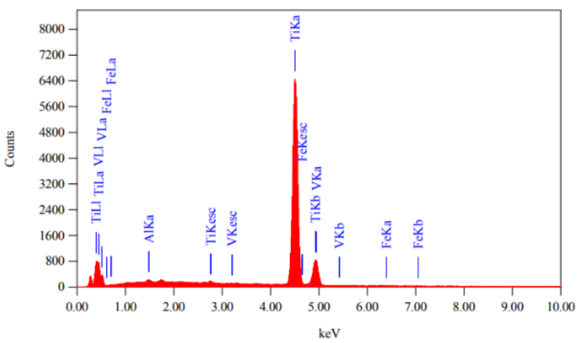
The results of the surface roughness measurements showed that there was an increase in roughness values in the strong acid treatment groups (HCl and H_2SO_4). However, there was no increase in roughness when treated with saline water. The roughness value in the 12.17 M HCl group was lower than that in the 6.94 M H_2SO_4, 9 M H_2SO_4, and 11.06 M H_2SO_4 groups. The 11.06 M H_2SO_4 treatment group was found to have the highest average roughness value, which is 0.66 µm (Figure 2).

Based on the normality test results, the research data were not normally distributed ($p < 0.05$) (Table 1), so the Wilcoxon test was conducted. The results of the Wilcoxon test showed that the saline water group had no significant difference in the average before and after treatment ($p > 0.05$). Meanwhile, the HCl 12.17 M, H_2SO_4 6.94 M, H_2SO_4 9 M, and H_2SO_4 11.06 M groups had significant differences before and after treatment ($p < 0.05$). In other words, the treatment influenced the final roughness measurement results (Table 2).

The Kruskal-Wallis test was carried out to determine differences between groups. The results of this test showed no significant difference between the pre-test groups ($p > 0.05$), but showed a significant difference between the post-test groups ($p < 0.05$) (Table 3). After significant differences were identified in the post-test groups, Mann-Whitney test was carried out. Based on the results of the Mann-Whitney test, there was a significant difference in the average roughness ($p < 0.05$) between the saline water group and the other groups, namely HCl 12.17 M, H_2SO_4 6.94 M, H_2SO_4 9 M, and H_2SO_4 11.06 M. However, there was no significant difference in the average roughness ($p > 0.05$) between HCl 12.17 M and H_2SO_4 6.94 M, H_2SO 9 M, and H_2SO_4 11.06 M, H_2SO 6.94 M, and H_2SO_4 9 M, and H_2SO 11.06 M, and H_2SO_4 9 M with H_2SO 11.06 M (Table 4).

The molarity and type of acid are also known to influence the topographic character of surface roughness. Based on the SEM test results with magnifications of 500x, 1000x, 3000x, and 5000x (Figure 3), the pre-test group showed that the surface of the titanium alloy was minimally porous, not grainy, irregular, smooth, but had shallow scratches. The saline water post-test group had a similar image to the pre-test group.

The SEM test results of the 12.17 M HCl post-test group with magnifications of 500x, 1000x, 3000x, and 5000x showed a rougher surface image than the pre-test and the saline water post-test groups. The image of the 12.17 M HCl group



Elements	Mass%	Atoms%
Al	0.30	0.53
Ti	99.15	98.96
V	0.48	0.45
Fe	0.07	0.06
Total	**100.00**	

Figure 1. SEM-EDS test results of pre-test samples

showed that the surface was porous, grainy, and had apparent grooves. The 6.94 M H_2SO_4 post-test group also had a quite similar topography to the 12.17 M HCl group, but in this group, the pores were larger, and the grooves looked more profound than the topography of the 12.17 HCl group.

Apart from that, the SEM test results in the 9 M H_2SO_4 post-test group with magnifications of 500x, 1000x, 3000x, and 5000x had a rougher surface image compared to the 6.94 M H_2SO post-test group. The image of the surface roughness of the 9 M H_2SO_4 post-test group was shown by the presence of a white image, namely pores, in the SEM test results. The image characteristics of the H_2SO_4 11.06 M post-test group were different from those of the H_2SO_4 9 M post-test group, as indicated by deeper grooves and a more prominent surface than the previous group.

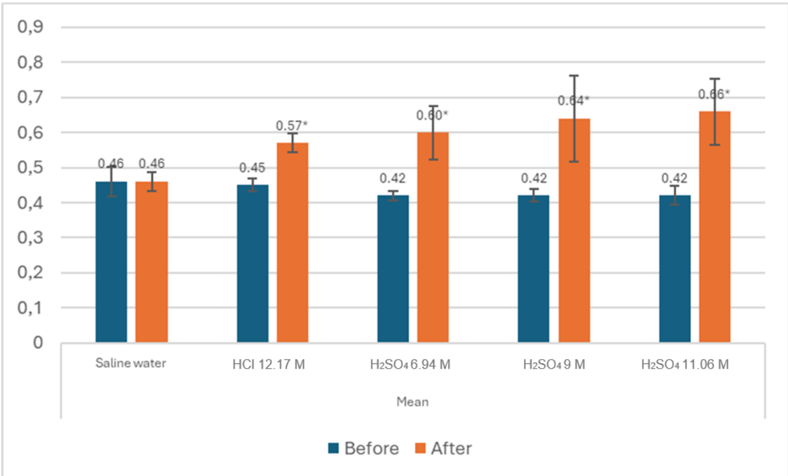


Figure 2. Graph of average surface roughness before and after treatment (n = 5) (*significant difference (p > 0.05))

Table 1. Pre-test and post-test surface roughness normality test results (n = 5)

Shapiro-Wilk	Pre-test		
	Statistics	df	Sig.
Saline water	0.722	5	0.016
HCl 12.17 M	0.684	5	0.006
H_2SO_4 6.94 M	0.552	5	0.000
H_2SO_4 9 M	0.684	5	0.006
H_2SO_4 11.06 M	0.881	5	0.314
Shapiro-Wilk	**Post-test**		
	Statistics	df	Sig.
Saline water	0.871	5	0.269
HCl 12.17 M	0.877	5	0.295
H_2SO_4 6.94 M	0.872	5	0.274
H_2SO_4 9 M	0.943	5	0.689
H_2SO_4 11.06 M	0.665	5	0.018

df : degrees of freedom
Sig. : significance (probability)

Table 2. Mean, standard deviation of surface roughness pre-test and post-test, and Wilcoxon test results (n = 5)

Group	P-value
Saline water	1.000
HCl 12.17 M	0.043*
H_2SO_4 6.94 M	0.043*
H_2SO_4 9 M	0.043*
H_2SO_4 11.06 M	0.043*

Wilcoxon test, *Significantly difference (p < 0.05)

Table 3. Kruskal-Wallis test results for pre-test and post-test groups

Group	df	Sig.
Pre-test	4	0.083
Post-test	4	0.016

(*) : sig. (p < 0.05)
df : degrees of freedom
Sig . : significance (probability)

DISCUSSION

This research focused on determining the surface roughness value of titanium alloy plates (Ti-6Al-4V) before and after treatment. Titanium alloy is the metal material most widely used for dental implants because of the superiority of the material composition, namely Ti, Al, V, and Fe.[17,18] The material used in this research had been confirmed to have this composition based on the results of the EDS test. Thus, the material used in this research, namely titanium alloy (Ti-6Al-4V), has advantages such as corrosion resistance, bioinert, and suitable biocompatibility.[19]

The results of this study showed that saline water can be a medium for storing dental implants because there was no change in roughness values from the pre-test to the post-test ($p < 0.05$). Saline water has the same solute concentration as human body fluids or is isotonic. Therefore, there is no difference in osmotic concentration between saline water and body fluids, so it does not cause significant changes in the osmotic pressure of cells or surrounding tissues.[20] The use of saline water as an implant storage medium has also been known in research conducted by Kohler et al; they reported that the disadvantage of etching in an open environment is that it causes a hydrophobic surface. A hydrophilization method can be applied to reduce hydrophobicity by storing the sample in a salt solution after acid etching.[12]

This research also showed that the group etched with 12.17 M HCl experienced an increase in the surface roughness value from before to after treatment ($p < 0.05$). The roughness value of HCl was higher than that of the saline water group but lower than that of the sulfuric acid group. Based on this research, 60 minutes was not the optimal time to increase roughness in the HCl group.

The result of this research is in line with research conducted by Al-Radha, showing that etching titanium with 37% HCl produces the highest surface roughness value, namely 0.59 µm in 30 minutes. The roughness value is significantly different from this research, 0.57 µm after etching with 12.17 M HCl for 60 minutes. The results of this research are in line with Al-Radha's research because, in his research, 30 minutes is the optimal

Table 4. Mann-Whitney Test Results

Group	Saline water	HCl 12.17 M	H_2SO_4 6.94 M	H_2SO_4 9 M	H_2SO_4 11.06 M	ΔRa (µm)
Saline water	-					0
HCl 12.17 M	0.008*	-				0.12
H_2SO_4 6.94 M	0.008*	0.511	-			0.18
H_2SO_4 9 M	0.011*	0.454	0.750	-		0.22
H_2SO_4 11.06 M	0.010*	0.314	0.738	0.914	-	0.24

*Significantly difference (p < 0.05)

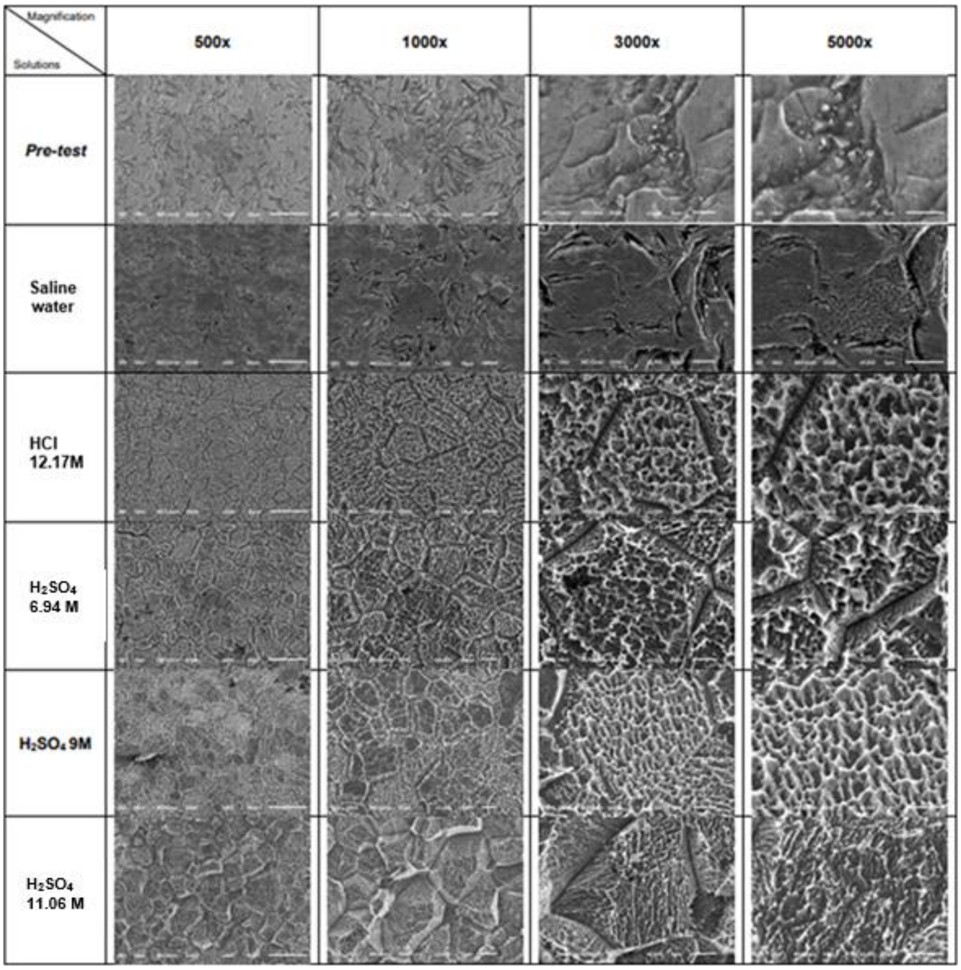


Figure 3. SEM test results with magnifications of 500x, 1000x, 3000x, and 5000x in the pre-test group, saline water post-test group, 12.17 M HCl post-test group, 6.94 M H_2SO_4 post-test group, 6.94 M post-test group H_2SO_4 test 9 M, post-test group H_2SO_4 11.06 M

time for etching with 37% HCl, which is equivalent to 12.17 M in this research, but there will be a decrease in surface roughness values at times 45 and 60 minutes. Conversely, it was reported that etching with H_2SO_4 can optimally increase the surface roughness for 60 minutes.[6] According to the research results, the group treated with sulfuric acid (H_2SO_4) had a higher roughness value than those treated with HCl and saline water and those not treated.[6,12]

Statistically, the saline water group had a significantly different average roughness than those treated with strong acid ($p < 0.05$). This result aligns with research that titanium alloys etched with strong acid have a rougher topography compared to those etched with weak acid. Immersion in acidic solutions is also known to remove or damage the oxide layer formed by titanium to protect its surface.[12] The acid works by penetrating the oxide layer and forming a more soluble compound than water, thereby creating porosity on the titanium surface. This process can create a rough or porous structure. Therefore, the surface of titanium that has gone through the acid etching processes may exhibit a rougher and more complex structure compared to the surface of titanium that has not been treated.[10,11]

There was a significant difference in the roughness values of the HCl and H_2SO_4 groups before and after treatment. Apart from that, there were significant differences between each group after treatment. The difference in roughness values can also be seen from the description of the SEM test results, showing that the 11.06 M H_2SO_4 group had a rougher topography compared to the other groups. The results of this research showed that the etching rate of titanium alloy depended on the type of acid and its concentration. According to previous research, titanium alloys etched with sulfuric acid (H_2SO_4) cause higher surface roughness values compared to HCl, H_3PO_4, HF, and HNO_3.[12]

Research conducted by Kohler et al. (2020) reported changes in surface roughness on commercially pure titanium (CpTi) after etching with sulfuric acid (H_2SO_4). They reported that etching with different concentrations of H_2SO_4 cause the Titanium to have very different surface roughness, even with the same treatment. Kohler et al. also reported that surface etching with 9M H_2SO_4 had a higher roughness value than 18 M H_2SO_4. This shows that a higher acid concentration is not always followed by a higher surface roughness. At an H_2SO_4 concentration of 9 M, the solubility of the titanium oxide layer increases, but as the concentration of H_2SO_4 becomes more concentrated, the solubility of the titanium oxide layer begins to decrease.[12]

The decrease is due to the formation of insoluble titanium salts on the surface of the specimen. These salts are insoluble, so they cannot dissolve the titanium oxide layer, even at higher acid concentrations. Based on the research results, the higher the solution concentration in the treatment group, the higher the roughness value. This result showed that etching with 11.06 M H_2SO_4 had yet to reach the maximum limit where a very concentrated concentration can reduce the solubility level of the oxide layer.[12,21]

Thus, this study showed that immersing titanium alloy plates in 11.06 M H_2SO_4 had the highest average surface roughness, 0.66 μm, with the roughness category being slightly rough (0.5 – 1.0 μm). However, it is necessary to investigate further regarding the maximum limit of H_2SO_4 concentration that can dissolve the oxide layer to obtain the optimal roughness aacording to the most current commercial dental implant systems (1.0 – 2.0 μm).[7]

According to Pierre C et al, an implant material surface roughness with an average of 1.0 – 2.0 μm is effective in increasing implant removal torque compared to smoother surfaces. In addition, surface roughness with an average of > 2.0 μm is known to cause poorer osseointegration due to a higher risk of bacterial colonization.[22] To optimize this roughness, the sulfuric acid etching method can be combined with the sandblasting method. The combination of these two methods is called the SA (sandblasting and acid etching) method.[23]

Based on the results of this research, the acid etching method can form micropores, but it is difficult to achieve very high roughness. Acid etching, according to Sasikumar Y et al, is also known to not produce contamination particles.[11] The advantages of this etching method can cover the disadvantages of the sandblasting method, where the risk of contamination of spraying particles with abrasive materials on the surface is high and this method only produces macro-sized roughness. Therefore, further research can be carried out regarding surface modification using the SA method to obtain optimal roughness in the future.[11,22,23]

CONCLUSION

Based on the results, it can be concluded that the type and concentration of acid influence surface roughness. Etching with a sulfuric acid solution affects the surface roughness value of the titanium alloy plate, where the value is higher than etching with a saline water solution and hydrochloric acid solution. In this study, etching with sulfuric acid at 11.06M showed the highest increase in surface roughness.

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CONFLICT OF INTEREST

The authors declare no competing interests.

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