Evaluation of Titanium Alloy Surface Roughness After Phosphoric Acid Etching

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ABSTRACT

Background: *Tooth loss can be caused by periodontal disease, caries, and other factors. Dental implants made of titanium are a common solution to replace tooth loss. Titanium grade 5 (Ti-6Al-4V) is often chosen for an implant. Modern commercial implant surface roughness (Ra) ranges from 1 to 2 μm. Surface roughness has an important role in osseointegration. The acid etching method can be used to improve surface roughness. This study evaluates titanium alloy surface roughness after etching with varying concentrations of H_3PO_4 acid.*

Method: *A laboratory experiment with a pre-test and post-test group design. 25 titanium alloy samples were divided into 5 groups (n=5) and treated with saline, HCl 37%, and H_3PO_4 acid 38%, 48%, and 58%. Surface roughness was measured using a surface roughness tester, while EDS and SEM tests analyzed titanium composition and surface characteristics.*

Result: *The titanium used in this study is a titanium alloy. Surface roughness modification using acid had a higher roughness compare to saline with significant different ($p<0.05$). Surface roughness in the group H_3PO_4 48% and 58% had a lower roughness compared to HCl 37% but no significant different ($p>0.05$). Referring to the SEM results, each acid showed morphological changes on the surface and for saline there were no morphological changes.*

Conclusion: *Varying concentrations of H_3PO_4 acid can effectively improve the surface roughness of titanium alloys, with solutions at 48% and 58% demonstrating similar effectiveness to 37% HCl acid.*

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INTRODUCTION

The periodontal tissue is a supporting structure surrounding the tooth and attaching to the alveolar bone to maintain teeth in their sockets.[1,2] Periodontal disease encompasses various inflammatory conditions affecting the tooth-supporting structures, leading to tooth loss and contributing to systemic inflammation. It can be classified into gingivitis and periodontitis.[3] According to Riskesdas 2018, the prevalence of periodontitis in Indonesia remains high, with a 74.1% case percentage, being a significant cause of tooth loss.[4]

Loss of teeth in the jaw arch can result in alveolar bone loss, disrupted masticatory function, orofacial structural disturbances, tooth migration, and affect supporting tissues.[5,6,7] Tooth loss can be treated with removable partial dentures (RPDs), fixed partial dentures (FPDs), or implants.[8,9] Dental implant treatment has become a prevalent option for replacing tooth loss. Typically, titanium materials are used for dental implants, with titanium grade 5 (Ti-6Al-4V) being favored for its superior physical and mechanical properties compared to pure titanium (Cp-Ti).[10] The formation of bone around the implant, known as osseointegration, is crucial for the success of dental implants.[11]

Despite its advantages, implant treatment can fail due to various reasons, including inadequate osseointegration.[12] Achieving optimal osseointegration can be facilitated through techniques like etching, commonly employed to create porous surfaces for bone growth to adhere to titanium implants.[13] Osteoblast activity can increase on micro-roughness surfaces ranging from 1 to 100 micrometers (μm) compared to untreated or smooth surfaces.[14,15] Some commercial implants have surface roughness (Ra) variations from 1 to 2 μm.[15] Surface modifications on implants can enhance roughness using various acids. Phosphoric acid (H_3PO_4), a weak acid, is one such immersion solution known for its non-carcinogenic properties, although high concentrations can be highly acidic and corrosive.[16]

Diab Al-Radha AS (2016) investigated the effects of acid etching on titanium implant surfaces using different acids, including hydrochloric acid, sulfuric acid, and nitric acid, with varying concentrations and etching times. Each acid demonstrated a distinct influence on the surface roughness of titanium implant surfaces.[13] Surface roughness modification plays a significant role in supporting the osseointegration process.[17] Therefore, researchers are interested in studying the surface roughness of titanium alloy implant materials through acid etching using weak acids, such as phosphoric acid, aiming to induce roughness on titanium alloy surfaces.

RESEARCH METHOD

This was an experimental laboratory with a pre-test and post-test group design. This research was conducted in the Dental Material and Testing Center of Research and Education Faculty of Dentistry, Universitas Trisakti, and Integrated Research and Testing Laboratory, Gajah Mada University. Research permits and ethics approval were obtained from the Ethics Committee for Health Research Faculty of Dentistry, Universitas Trisakti, with ethical clearance Number: 673/S1/KEPK/FKG/7/2023.

In this study, titanium alloy plates measuring 10x10x2mm were used for each treatment group, so the total number of subjects was 25 (n=5). The subjects were divided into 5 groups with different treatments. Group 1 was immersed in saline acid solution as a negative control group. Group 2 was immersed in an HCl 37% acid solution as the positive control group. Group 3 was immersed in a phosphoric 38% acid solution. Group 4 was immersed in a phosphoric 48% acid solution. Group 5 was immersed in a phosphoric 58% acid solution.

The titanium samples underwent surface roughness testing before etching treatment using a surface roughness tester (Tester Surtonic S-100 Series) and observing the surface morphology of the samples before etching treatment using a scanning electron microscope (SEM) to observe changes before and after treatment. For sample immersion, saline solution, 37% HCl, and H_3PO_4 acid with concentrations of 38%, 48%, and 58% can be prepared. For phosphoric acid, dilution can be performed from 85% phosphoric acid to 38%, 48%, and 58% (M1 x V1=M2 x V2), and pH checking is carried out for each solution.

Each solution is heated using a SH-2 Digital Lab Thermostatic Hot Plate Magnetic Stirrer Mixer until the temperature reaches 60°C. Immersed the samples in each container according to the solution group, ensuring each sample was immersed, and place them in an Ov-30 oven at 60°C for 60 minutes. After 1 hour, rinse each sample with distilled water for 5 minutes and dry the samples using an incubator at 37°C for 24 hours. Then, measure the surface roughness after etching treatment using a surface roughness tester (Surtonic S-100 Series) and observe the surface morphology of the samples after etching treatment using a scanning electron microscope (SEM).

The data collected from the immersion of Ti-6Al-4V into each solution underwent a normality test. The normality test was conducted using the Shapiro-Wilk method, as the sample size was < 50. If the data is normally distributed ($p>0.05$), the homogeneity of the data is then tested using the Levene test. If the data is normally distributed and homogeneous, a paired T-test is conducted to observe the differences before and after the treatment, and a one-way ANOVA test is performed to observe the differences between each group. If the test results show significant differences, a Post Hoc test is conducted.

RESULTS

Prior to conducting this study, to ensure the composition of the titanium alloy used in this research, an Energy-Dispersive X-ray Spectroscopy (EDS) test was performed with the results in Table 1 and Figure 1, The results of the EDS test on the titanium alloy (Ti-6Al-4V) showed that the sample used in this study has the highest titanium content, it is observed that Titanium constitutes 98.96%, Aluminium 0.53%, Vanadium 0.45%, and Iron 0.06%.

Table 1. EDS test percentage (%) result

Element	Mass (%)	Atom (%)
Al	0,30	0,53
Ti	99,15	98,96
V	0,48	0,45
Fe	0,07	0,06
Total	100,00	100,00

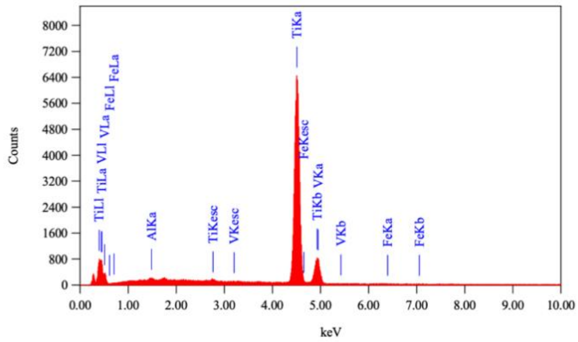

Figure 1. Energy Dispersive X-ray Spectrocopy test result

This study was conducted on 25 samples divided into 5 groups. Each group consists of 5 samples. Based on the roughness test results, it can be observed that saline water did not induce any changes in the roughness of Ti-6Al-4V after immersion. However, both 37% HCl acid and H_3PO_4 acid solutions caused changes in roughness following immersion. The results are presented in Table 2 and Figure 2.

Table 2. Results of the roughness test pre-test and post-test

	Ra Pre-test (µm)	Ra Post-test (µm)
Saline	0,446 ± 0,044	0,446 ± 0,044
HCl 37%	0,480 ± 0,050	0,620 ± 0,055
H_3PO_4 38% acid solution	0,446 ± 0,044	0,526 ± 0,027
H_3PO_4 48% acid solution	0,440 ± 0,059	0,546 ± 0,038
H_3PO_4 58% acid solution	0,460 ± 0,054	0,560 ± 0,027

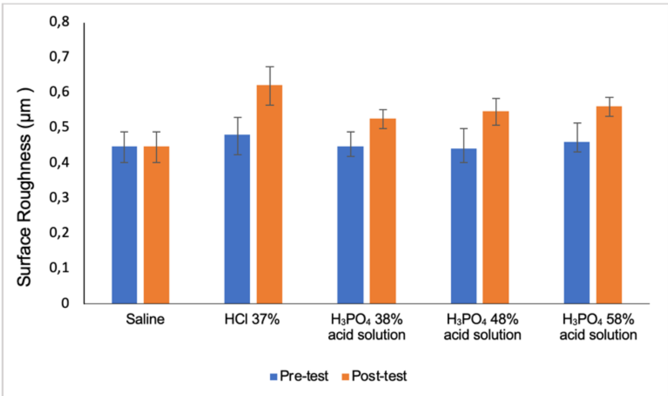

Figure 2. Surface roughness test result

Before immersing the sampels, the solutions used tested for pH to determine the pH level contained in each solution (Table 3).

Table 3. Results of pH test

Solution	pH
Saline	6,14
HCl 37%	-
H_3PO_4 38% acid solution	0,48
H_3PO_4 48% acid solution	0,35
H_3PO_4 58% acid solution	0,30

After collecting the data, statistical analysis was conducted using the SPSS software. The data analysis began with a normality test using the Shapiro-Wilk method due to the sample size being < 50. Based on the analysis results, the data were normally distributed with a $p>0.05$ (Table 4). Since the data were normally distributed, the homogeneity test was conducted using the Levene method. The analysis indicated that the data percentages were homogenous, with a $p>0.05$ (Table 5).

Table 4. Normality test result

Shapiro-Wilk	Pre-test			Post-test		
	Statistic	df	Sig.	Statistic	df	Sig.
Saline	0,871	5	**0,272**	0,871	5	**0,272**
HCl 37%	0,915	5	**0,501**	0,881	5	**0,314**
H_3PO_4 38% acid solution	0,871	5	**0,272**	0,881	5	**0,314**
H_3PO_4 48% acid solution	0,899	5	**0,403**	0,950	5	**0,735**
H_3PO_4 58% acid solution	0,898	5	**0,400**	0,871	5	**0,269**

Table 5. Homogenity test result

Uji Levene	Pre-test dan Post-test			
	Levene Statistic	df1	df2	Sig.
Based on Mean	1,124	9	40	**0,369**
Based on Median	0,429	9	40	0,911
Based on Median and with adjusted df	0,429	9	32,215	0,909
Based on trimmed mean	1,132	9	40	0,364

To observe the differences before and after the test, paired T-tests were conducted. The results showed no difference between the pre-test and post-test for saline water ($p>0.05$). However, differences were observed between the pre-test and post-test for 37% HCl and H_3PO_4 acid solutions, with a $p<0.05$ (Table 6).

Table 6. Paired T-test result

Solution	t	df	Sig. (2-tailed)
Pre-test dan Post-test HCl 37%	-4,722	4	**0,009**
Pre-test dan Post-test H_3PO_4 38% acid solution	-2,961	4	**0,042**
Pre-test dan Post-test H_3PO_4 48% acid solution	-4,760	4	**0,009**
Pre-test dan Post-test H_3PO_4 58% acid solution	-3,426	4	**0,027**

This was followed by a one-way ANOVA test, which revealed that all groups had significantly different data if the p<0.05 (Table 7). Since the one-way ANOVA test showed significant differences, further testing was conducted to identify specific differences between the groups.

Table 7. One-way ANOVA test result

Uji Anova One- way	Pre-test dan Post-test				
	Sum of Squares	**df**	**Mean square**	**F**	**Sig.**
Between Groups	1715,620	9	190,624	8,962	**<0,001**
Within Groups	850,800	40	21,270		
Total	2566,420	49			

Post-hoc Tukey tests were performed for more specific analysis. Based on the data, the pre-test and post-test for 37% HCl, 48%, and 58% H_3PO_4 acid solutions showed significant differences with $p < 0.05$ (Figure 3). Additionally, based on the post-hoc test for the post-test of all groups, saline water with all solutions, and 37% HCl with 38% H_3PO_4 acid solutions showed significant differences with $p < 0.05$ (Figure 4).

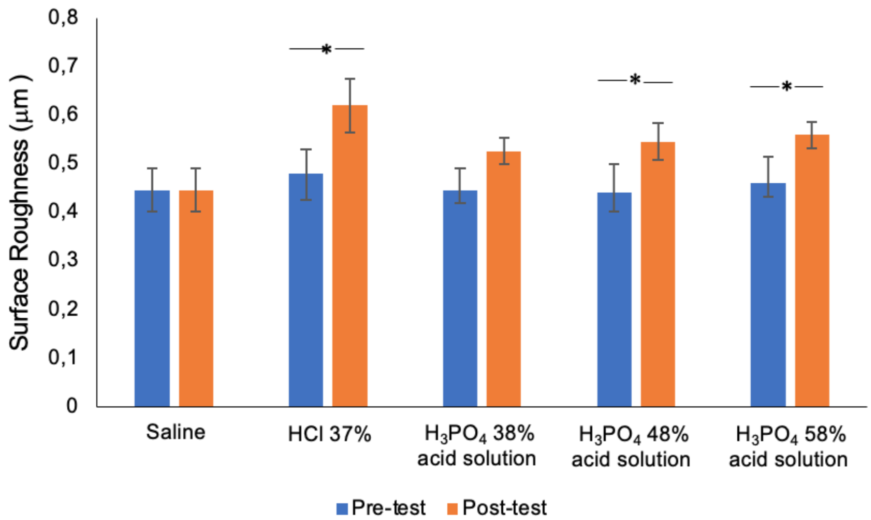

Figure 3. Post-hoc tukey pre-test and pos-test test result

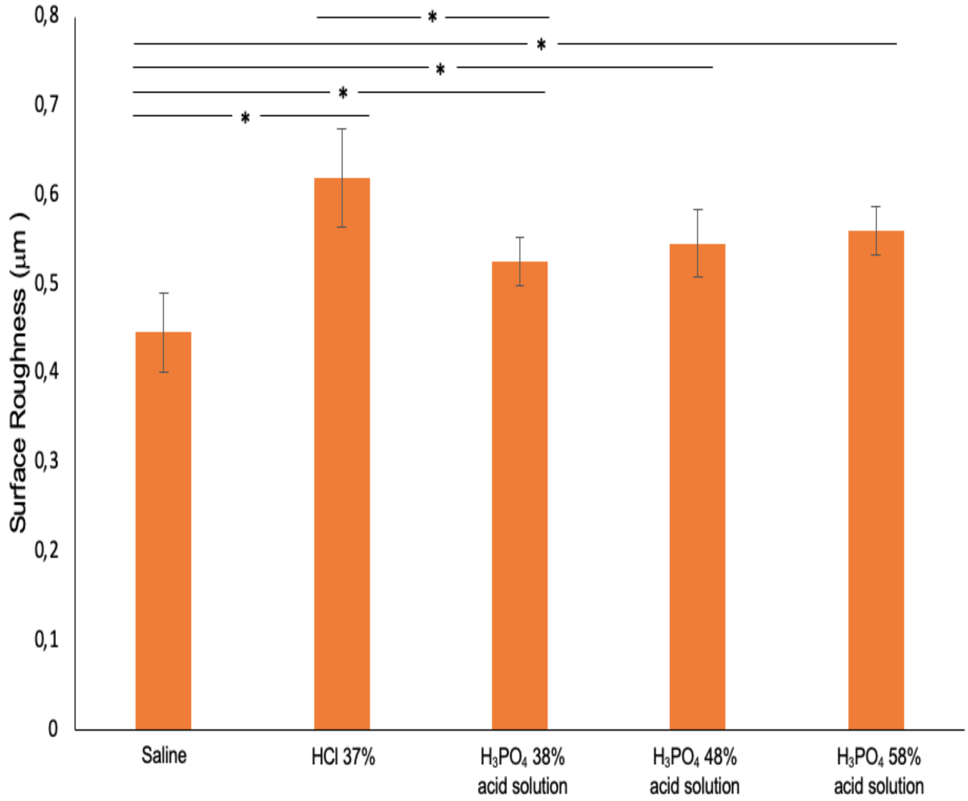

Figure 4. Post-hoc tukey post-test test result

The morphology of the samples can be observed using SEM analysis, as shown in the results (Figure 5), depicting SEM morphology at magnifications of 500x, 1000x, 3000x, and 5000x.

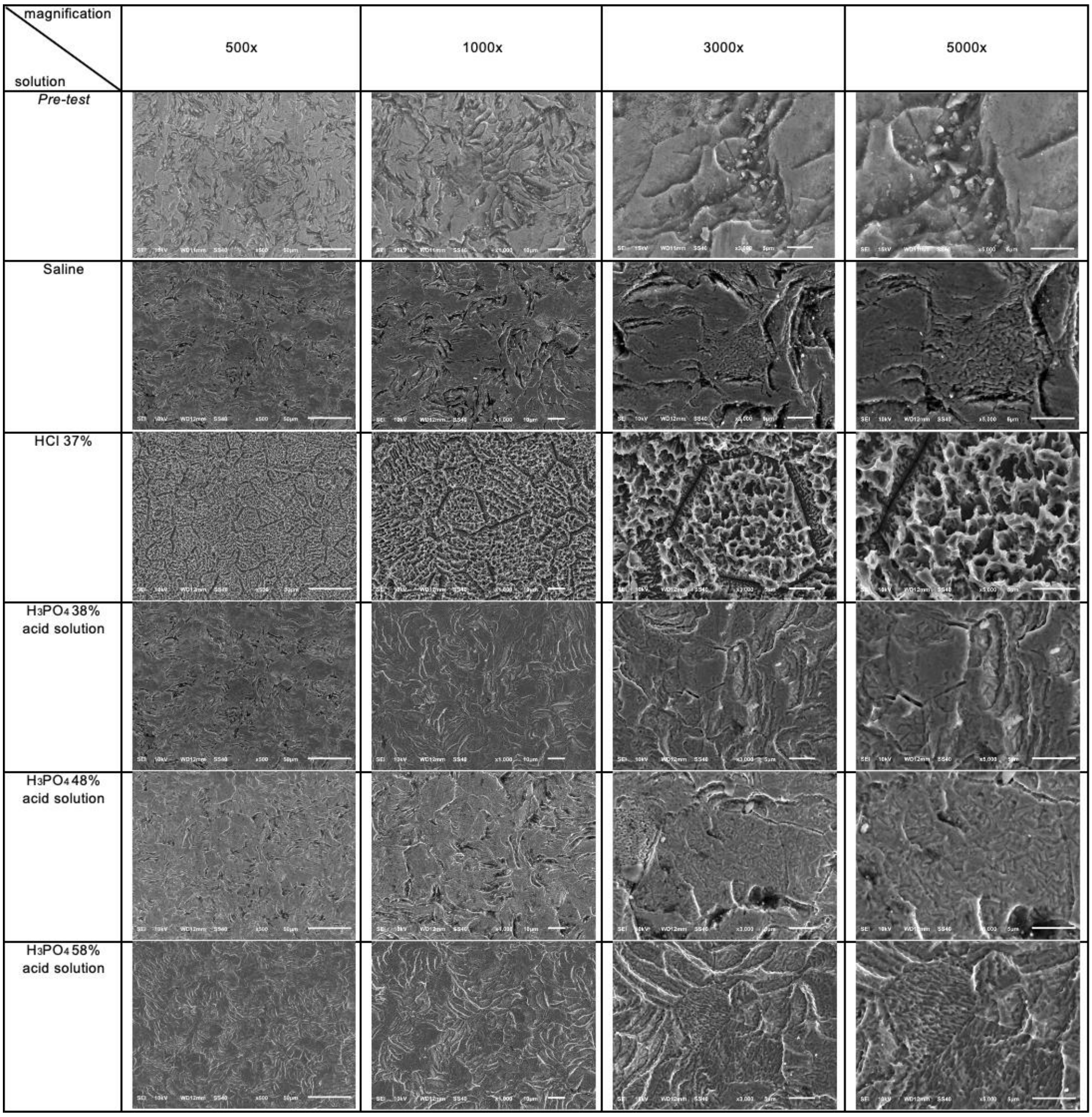

Figure 5. Scanning Electron Microscope (SEM) result with 500x, 1000x, 3000x and 5000x magnification

Based on the SEM results shown at magnifications of 500x and 1000x, irregular scratches were observed in both the pre-test and saline water groups, with no pores visible, indicating no difference between the two images. At magnifications of 3000x and 5000x, it was observed that titanium immersed in saline water had better results, but this result could be biased as the images taken may appear rough to the naked eye, thus not aligning with the measured points. Also, SEM results showed in magnifications of 500x, 1000x, 3000x, and 5000x, the 37% HCl group appeared rough, porous, and had depressions.

Based on the SEM results of the 38% phosphoric acid group at magnifications of 500x and 1000x, irregular white scratches were observed, and the scratches on the surface were spaced apart, with no pores visible, indicating a smooth surface. At magnification of 3000x, shallow depressions and scratches were observed, and at a magnification of 5000x, there were some very shallow pores in one area. For the 48% phosphoric acid group, irregular and spaced scratches were observed at magnifications of 500x and 1000x, with no pores visible. At a magnification of 3000x, deeper pores were observed compared to the 38% phosphoric acid group, and at a magnification of 5000x, deeper and more spaced scratches than 38% phosphoric acid. And for the 58% phosphoric acid group, irregular scratches were observed, and there were more scratches and no spaces visible. However, at magnifications of 3000x and 5000x, some pores were visible, resulting in a rough appearance at these magnifications.

DISCUSSION

This study is a laboratory experimental research with a pre-test and post-test group design method using titanium alloy (Ti-6Al-4V). This study was tested using a surface roughness tester and SEM equipment with the aim of evaluating the surface roughness of Ti-6Al-4V obtained before immersion and after immersion in several solutions, namely saline water, 37% HCl acid, and H_3PO_4 acid solutions with various concentrations. EDS testing was also conducted to observe the composition of the titanium alloy used in this study.

Based on the EDS test results, the samples used in this study contained 98.96% titanium, 0.53% aluminium, 0.45% vanadium, and 0.06% iron. This research is consistent with Fitriani CY and Wibawa A (2019) who stated that titanium alloy contains 90% titanium, 6% aluminium, 4% vanadium, and 0.25% iron[18] and according to Damisih et al (2018), the content of titanium alloy is 90.07% titanium, 5.93% aluminium, 3.80% vanadium, and 0.07% iron.[19] Although the titanium alloy used in this study does not resemble the actual composition of Ti-6Al-4V, it still contains titanium, aluminium, vanadium, and iron.

Based on the surface roughness measurements using a surface roughness tester, the results for the saline water group showed no changes in the pre-test and post-test results. Based on the SEM results shown in Figure 5, at magnifications of 500x and 1000x, irregular scratches were observed in both the pre-test and saline water groups, with no pores visible, indicating no difference between the two images. At magnifications of 3000x and 5000x, it was observed that titanium immersed in saline water had better results, but this result could be biased as the images taken may appear rough to the naked eye, thus not aligning with the measured points. Saline water served as the negative control in this study because it is commonly used as a medium for storing dental implants, and its salt concentration is similar to that of the human body, thus causing no significant difference in osmotic pressure between saline water and body fluids, which does not lead to significant changes in cell or tissue osmotic pressure.[20]

Based on the surface roughness measurements using a surface roughness tester, the results for the 37% HCl group were higher than those for H_3PO_4. SEM results showed that at magnifications of 500x, 1000x, 3000x, and 5000x, the 37% HCl group appeared rough, porous, and had depressions. This is also supported by the study conducted by Diab et al (2016) which stated that 37% HCl is effective on titanium surfaces in increasing surface roughness[13], and according to Speight J G (2017), HCl is a colourless, strong, and corrosive acid. Therefore, it was used as a positive control in this study.[21]

Based on the results of the research using a surface roughness tester on the Ti-6Al-4V surface to observe surface roughness before and after treatment, it was found that each phosphoric acid (H_3PO_4) had an effect on increasing surface roughness on Ti-6Al-4V. If measured from the surface topography classification of implants, groups using 38%, 48%, and 58% phosphoric acid belong to the slightly rough category (Ra 0.5 - 1.0 µm) because the results of Ti-6Al-4V immersed in H_3PO_4 acid in this study ranged from 0.52 to 0.56 µm. From the Ra surface roughness tester results after treatment, the higher the concentration used for Ti-6Al-4V immersion, the better the roughness produced. However, the results obtained did not show optimal roughness (Ra 1.0 - 2.0 µm).[15] The roughness occurring on Ti-6Al-4V is due to oxidation. This oxidation can be caused by chemical treatment using acid as one of the surface oxidation methods.[22]

In comparing all samples based on roughness and SEM results, 37% HCl acid was superior in increasing roughness. This is also evident from the pH test results; 37% HCl acid is a strong acid, and therefore, it cannot be detected using a pH test kit, indicating that the pH of 37% HCl acid is below 0. The higher roughness produced by 37% HCl acid may also be due to the pH affecting corrosion rates. The corrosion rate of Ti-6Al-4V can increase faster if the acid used has a low pH. This is also in line with the research conducted by El-Sayed et al (2022), which stated that the corrosion rate of HCl is higher than that of H_3PO_4.[23]

Based on the post-hoc test results for each solution used saline water compared to other acids showed significant differences, and the comparison of 37% HCl acid with 38% H_3PO_4 acid showed significant differences. If all H_3PO_4 acids are compared, there are no significant differences in the results. However, 37% HCl acid compared to 48% and 58% H_3PO_4 acids showed no significant differences. Based on the pH test, these phosphoric acids are weak acids, but the pH measurement results showed 0.35 for 48% H_3PO_4 acid and 0.30 for 58% H_3PO_4 acid. According to the book Chemical Principles, 8th edition, H_3PO_4 is not completely dissociated, so it is considered a weak acid, but the pH measurement results classify it as a strong acid because the H+ ions in H_3PO_4 can interact with water molecules, and H_3PO_4 has a high H+ ion with a total of 3, resulting in a strong acid pH measurement. This research is also in line with the research conducted by Franca et al (2018), which stated that phosphoric acid can increase micro-roughness, clean surfaces, and increase surface porosity.[12] From this study, it can be seen that the effectiveness of 48% and 58% phosphoric acid in roughening the Ti-6Al-4V surface is equivalent to the effectiveness of 37% HCl acid.

CONCLUSION

Based on the findings of this study, it is concluded that phosphoric acid (H_3PO_4) has an effect on increasing roughness on the surface of titanium alloy. In this research, both 48% and 58% concentrations of phosphoric acid demonstrate effectiveness comparable to 37% hydrochloric acid (HCl), and the highest effectiveness in enhancing roughness observed in the study is with 58% phosphoric acid.

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