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Efficacy of Electropolishing on the Surface of Ti-6Al-4V ELI Alloy to Minimize Bacterial Adhesion on Implants

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Titanium and its alloys are the most commonly used materials for biomedical applications such as implants. The Ti-6Al-4V ELI (Extra Low Interstitial) alloy is particularly desired for its excellent mechanical performance and remarkable biocompatibility. However, infection is a limiting factor after implantation, making it necessary to adopt surface treatment to minimize this process. Bacterial adhesion to the substrate is strongly influenced by surface roughness and wettability. Therefore, the present study aims to evaluate these parameters after dry electropolishing of the Ti-6Al-4V ELI alloy. The Ti-6Al-4V ELI samples were sectioned with Ø = 14 mm and 3 mm thick, followed by sanding with silicon carbide #600, #1200 and #1600. Subsequently, they were sent for dry electropolishing for 30 min with a voltage potential of 13 to 18 V and characterized. Data were processed statistically using ANOVA. The Ra roughness values were statistically significant: 143±24 nm for 17 V compared to 193±18 nm for the control. The wettability test confirmed that all surfaces obtained are considered hydrophilic, favorable for lower bacterial adhesion, with 16 V being the best condition. Contact angle was found to be 77±6° for 16 V and 59±4° for the control. Dry electropolishing is essential for surface enhancement and biomedical applications.

Keywords*: Roughness, wettability, Ti-6Al4V-ELI alloy, dry electropolishing, bacterial adhesion.*

1. Introduction

Innovation in biomaterials for implantation in surface treatments that contribute to osseointegration and antibacterial properties is of pertinent clinical interest. The Ti-6Al-4V ELI (Extra Low Interstitial) alloy is the most widely used for biomedical applications, as it has excellent mechanical properties, biocompatibility and corrosion resistance. However, some limitations need to be addressed, such as long-term corrosion, wear between joints, bacterial infection and insufficient osseointegration[1-4].

To control bacterial infection in implants, it is necessary to control the roughness and wettability of the metal surface, since these parameters directly influence bacterial adhesion to the substrate. The commonly evaluated roughness parameters are: mean roughness (Ra), root mean square roughness (Rq) and maximum height (Rz). Wettability from the contact angle allows the characterization of the surface as hydrophilic $10° < \Theta < 90°$, hydrophobic $90° < \Theta < 150°$, superhydrophilic $\Theta < 10°$ and superhydrophobic $\Theta > 150°$. Hydrophilic surfaces are promising for controlling infections in implants, since they minimize bacterial adhesion to the substrate[2,5-8].

One strategy to control roughness and inhibit bacterial adhesion on the surface of a material is electropolishing. The principle of electropolishing is the removal of material from a metal or alloy through the process of anodic dissolution, based on the fundamentals of electrolysis (Faraday's Law). The method is characterized by the elimination of roughness, absence of crystallographic and grain boundary attack, resulting in smooth and shiny surfaces[9,10].

Electropolishing (EP), or electrolytic polishing, is a process widely applied in the metal finishing industry, biomedical and semiconductor applications, pharmaceutical installations, and superconducting niobium cavities. Factors such as electrolyte, operating parameters, voltage (V), temperature, and current density directly determine the surface finish. However, during the EP process, a passive layer composed of oxides is formed on the metal surface, which interferes with the uniform surface finish[9-11].

To improve the limitations of EP, dry electropolishing (DEP) technology has emerged, combining the EP process with gentle mechanical abrasion, which is effective in reducing the formation of the passive layer and increasing the removal rate. In DEP, liquid electrolytes are replaced by porous ion-exchange particles composed of polystyrene

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divinylbenzene that absorb the active liquid electrolyte, allowing the system to remain “dry” while achieving polishing results. Waste generation is therefore reduced, as the electrolytic liquid is encapsulated within the active particles, reducing the environmental impact of the process and increasing operational safety. Several materials, such as titanium and titanium alloys, have been studied using the DEP process. The authors describe a reduction in preliminary defects in the manufacturing process and a reduction in the average roughness Ra, obtaining a smooth surface finish that partially preserves the compressive residual stresses[12-18].

The present study adopts the innovative dry electropolishing technology to evaluate the roughness and wettability parameters of Ti-6Al-4V ELI alloy in order to minimize bacterial adhesion to implants.

2. Materials and Methods

Samples of the Ti-6Al-4V ELI alloy (Table 1 shows the chemical composition of the alloy) were sectioned (STRUERS cutter) with a diameter of 14 mm and a thickness of 3 mm, cleaned in an ultrasonic bath with acetone, distilled water (for 5 minutes in each solution), 95% ethyl alcohol, and then cold dried. Characterization was carried out by optical microscopy (OM), sanding with silicon carbide # 600, # 1200, # 1600, followed by electropolishing.

Dry electropolishing was performed using equipment (Dlyte 100 Pro), under voltage (V) and time (min) control. Initially, the samples in triplicate were subjected to gentle mechanical abrasion, using porous ion exchange particles of polystyrene divinylbenzene for 15 minutes, with a voltage threshold of 13 to 18 V. Then, they were cleaned and immersed in Ti 06 Dlyte 01- MAS G L130 and Ti 01 Dlyte Mix MAS Plus for another 15 minutes, with the same voltage threshold. After electropolishing, the samples were cleaned again and characterized using optical profilometry and wettability testing.

Optical microscopy was performed using an Olympus BX 53 M microscope. Surface roughness and topography values were evaluated using an optical profilometer, WYKO NT1100. The center of the samples was the reference for the measurements of Ra, Rz and Rq.

The wettability test was based on the contact angle, using an SEO Phoenix drop shape analysis system, under ambient conditions of 23º C. Deionized water with a volume of 3 µL and a needle with an internal diameter of 0.30 mm supplied with a syringe, was used for the procedure. ANOVA was adopted for statistical treatment of the data.

Table 1. Chemical composition of the Ti-6Al-4V ELI alloy.

Element	Composition (wt%)
Ti	Balance
N	0.05
C	0.01
Fe	0.3
H	0.015
Al	6.2
V	4.5
O	0.2

3. Results and Discussion

The experimental results will be presented in the form of figures and graphs. Figure 1 shows the surface of the Ti-6Al-4V ELI alloy used in this study. Control samples and two of the best electropolishing conditions, 16 and 17 V for 30 minutes, are shown in Figure 2.

It can be observed in Figure 2 that the sample’s surface improved after electropolishing, due to the applied voltage potential. Sample “c”, with a voltage potential of 17 V, was smoother and shinier when compared to sample “b”, with a voltage potential of 16 V.

From dry electropolishing, the roughness values Ra, Rq and Rz were obtained, represented in Figure 3a and b.

It can be observed in both graphs that the voltage potential (V) influenced the roughness of the samples. After applying a voltage potential of 17 V, there was a significant reduction in Ra to 49.49 nm, Rq to 39.86 nm and Rz to 1.967 µm, compared to the control sample. A study with Zn-Ni-Alkaline samples, after dry electropolishing, significantly reduced roughness. The average Ra value, measured in three random zones of the sample before and after post-processing, was approximately 151.4 ± 10.1 nm and 12.2 ± 1.8 nm, respectively, and after processing it was 12.2 ± 1.8 nm. The values obtained are in line with the findings of other authors, indicating that a surface with roughness below 0.2 µm significantly reduces bacterial accumulation and adhesion[19-21].

It is worth mentioning the use of dry electropolishing for the preparation of cutting edges of WC-Co (cemented carbide) tools. This technique allowed the reduction of defects that precede the grinding process and the parameters of the cutting-edge radius increased as a function of the electropolishing time. The growth rate was higher during the initial stages, due to the sharp geometry of the cutting edge, which was also observed in the reduction of roughness. The surface finish by this technique allows the workpiece tolerance to be maintained, together with minimum levels of surface roughness, helping to preserve a significant amount of the compressive stress state introduced during grinding[22,23].

The surface topography of the present study after dry electropolishing was evidenced by optical profilometry, as shown in Figure 4.

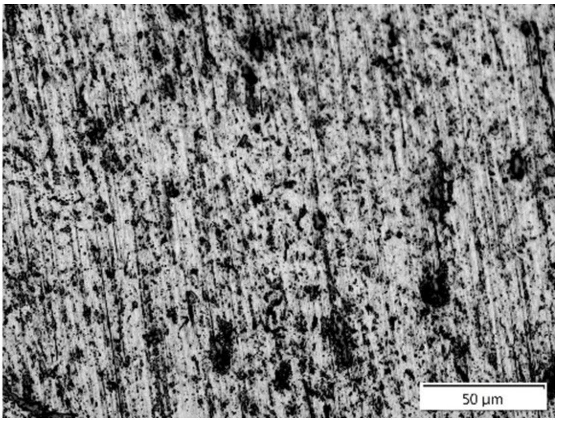

Figure 1. Grinded Ti-6Al-4V alloy.

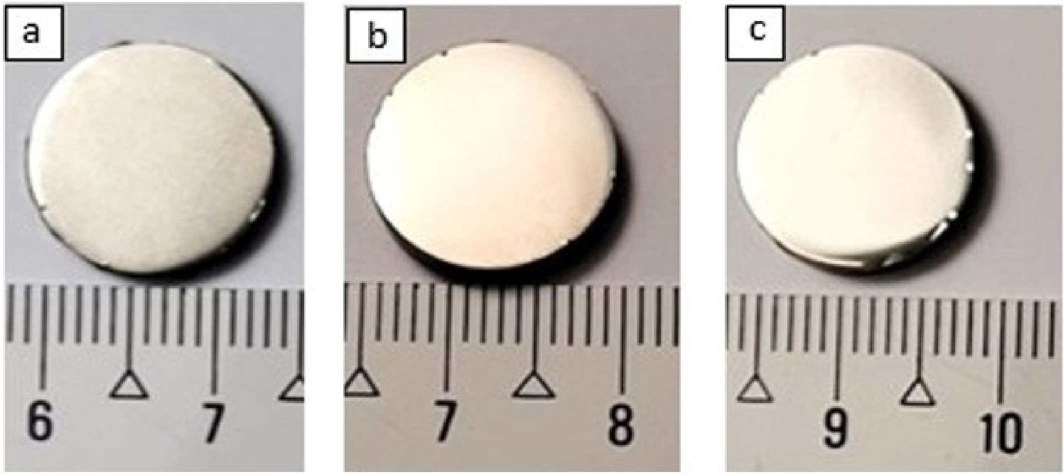

Figure 2. (a) Control sample; (b) sample electropolished at 16V; (c) sample electropolished at 17V (both for 30 minutes).

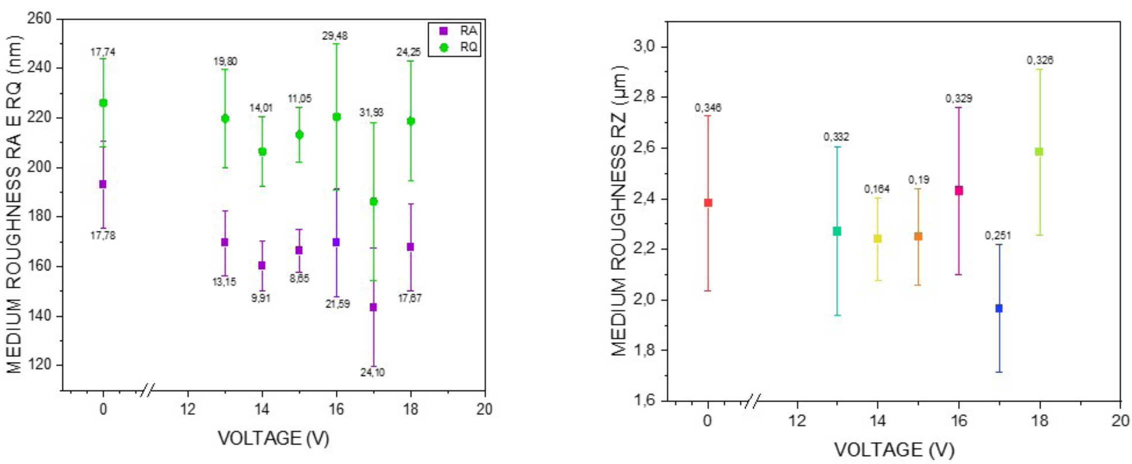

Figure 3. Roughness results: (a) Ra and Rq; (b) Rz.

The main results are highlighted as the variation in surface topography, evidenced by the color scale and reduction of roughness Ra, Rq and Rz after the applied voltage potential, when compared to the control sample. The highest rate of roughness reduction occurred at a voltage potential of 17 V. The amount of bacterial adhesion can be affected by surface topography[24].

After analysis by optical profilometry, the data on the effect of voltage (V) on Ra, Rq and Rz were statistically treated by the ANOVA test, the main results are highlighted:

1. Ra (Mean Roughness): The p-value for Ra was 0.077 and 0.102 in separate analyses, suggesting that voltage may have a moderate but not highly significant effect;
2. Rq (Root Mean Square Roughness): The p-value was 0.428, indicating no statistically significant effect of voltage;
3. Rz (Height from peak to valley): The p-value was 0.295, also indicating no significant effect;
4. Confidence interval (CI) for Ra: The interval for 0 V (193.1 µm) does not overlap with 17 V (143.6 µm), indicating a significant reduction in roughness. The confidence intervals for 13 V, 14 V, 15 V, 16 V, and 18 V overlap, suggesting that there are no statistically significant differences between these voltages

The Tukey test for the effect of voltage (V) on Ra, Rq and Rz obtained by ANOVA analysis of variance is shown in Figure 5.

The three graphs presented in Figure 5a, b and c indicate that almost all of the intervals for pairwise comparisons of the conditions studied in this work include zero. The exception is the Ra interval for the comparison between the control sample and the 17 V potential. This suggests that for most of the comparisons there is no significant difference between the conditions, as zero falls within the confidence intervals. However, the comparison between the control sample and the 17 V potential shows a significant difference in the Ra

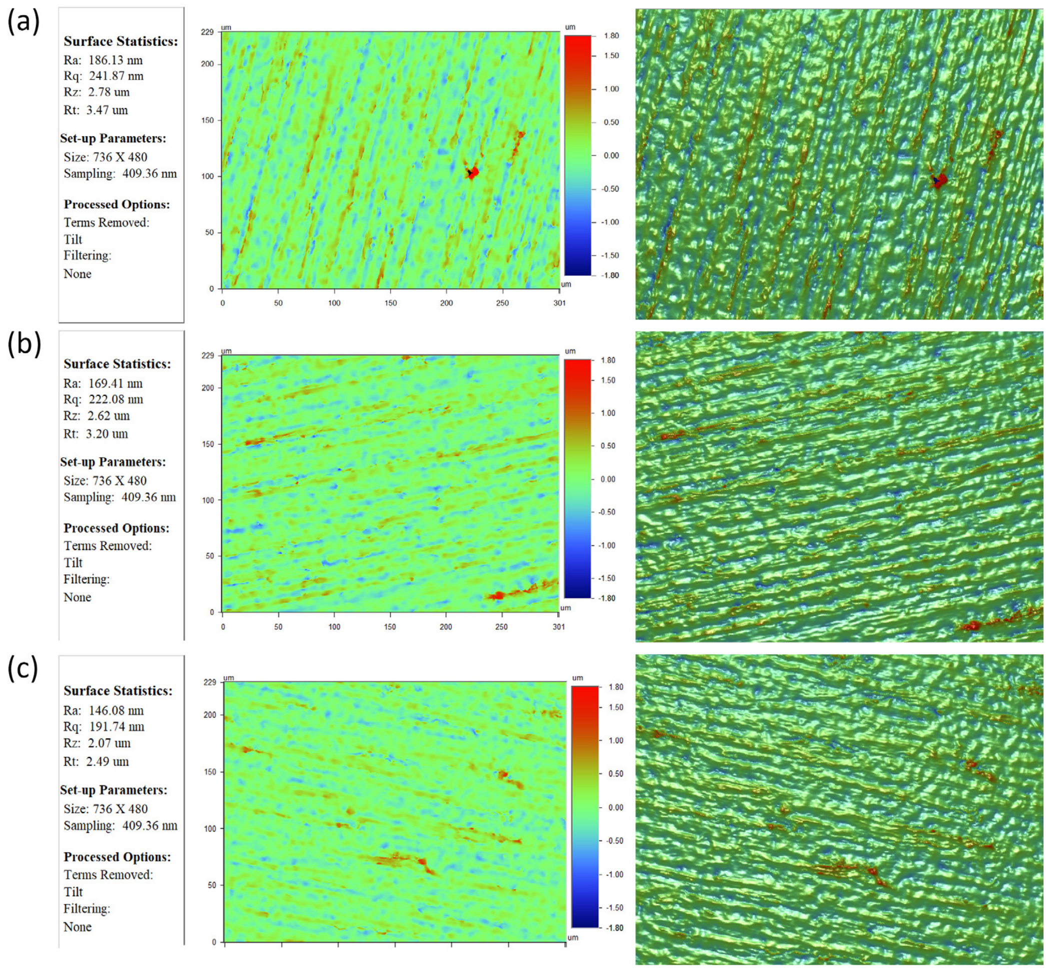

Figure 4. Surface topography: (a) control sample; (b) sample electropolished at 16 V for 30 min; (c) sample electropolished at 17V for 30 min.

value, indicating that this specific condition had a distinct effect on surface roughness compared to the others.

The wettability test of these samples can be observed through the contact angle, as shown in Figure 6. There is a variation in the contact angle depending on the applied voltage potential. Compared to the control sample, all presented surfaces are considered hydrophilic, with a contact angle variation of $10° < \Theta < 90°$, capable of minimizing bacterial adhesion to the substrate. As the contact angle increases, bacterial adhesion decreases[2,24]. Considering the limits of the standard deviation bars, it is evident that the conditions of 16 V and 17 V stand out in the graph, indicating statistically better results compared to the other conditions.

Analysis of variance was also applied to the wettability test, showing that the p-value (0.001 with the control sample and 0.002 without the control sample) indicates that voltage has a significant effect on the contact angle in both cases. The F-value is also high, suggesting a strong relationship.

The statistical data from Tukey's test for the effect of voltage (V) on contact angle are shown in Figure 7. Unlike the roughness data, significant differences are observed in this case. Regarding the control sample, which is the main focus of this analysis, the confidence interval for the comparison between the control and the 16 V sample does not include zero. This indicates that the 16 V electropolishing condition significantly alters the surface wettability compared to the control, suggesting improved hydrophilicity. The interval between the control and the 17 V sample, which showed a subtle difference in roughness, includes zero but is very close to the extreme.

The comparison of Ra, Rq, and Rz results did not reveal significant differences among them. Although the 17 V sample showed a slightly more pronounced decrease in average roughness, the differences were not statistically significant, indicating the need for a larger number of replicates.

Regarding the contact angle, taking the control sample as a reference, significant differences were observed only when the electropolished 16 V sample was considered. The association of roughness and wettability results through the contact angle suggests that the 16 V sample may be the most promising in the study, with the highest contact angle of 77°. Since a higher contact angle corresponds to lower bacterial adhesion, this result is particularly important.

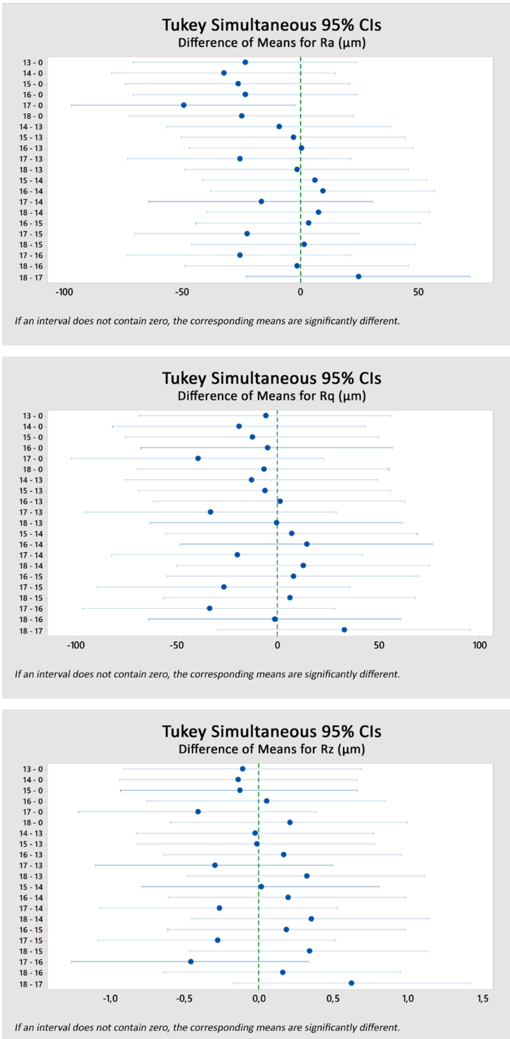

Figure 5. Tukey test for Ra, Rq and Rz.

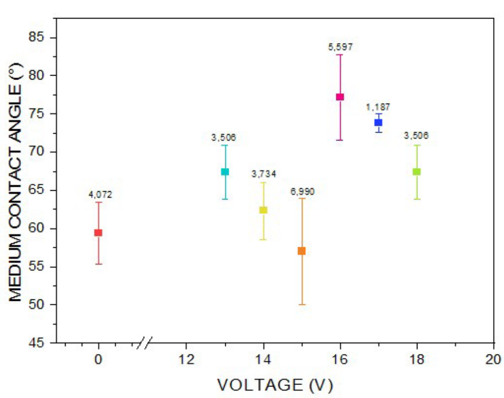

Figure 6. Representation of the contact angle.

Overall, the findings highlight the critical relationship between Ra roughness and surface wettability in achieving hydrophilic surfaces.

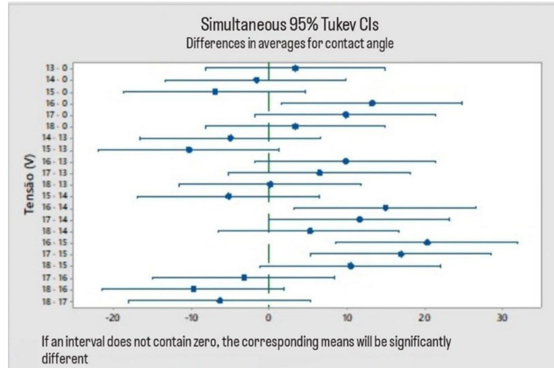

Figure 7. Tukey test for contact angle.

4. Conclusion

Dry electropolishing yielded favorable results for polishing the Ti-6Al-4V ELI alloy within the electropolishing range of 13 to 18 V. The main conclusions are:

1. The sample electropolished at 16 V for 30 minutes exhibited promising polishing parameters.
2. Surface topography showed a reduction in Ra, Rq, and Rz roughness as a function of voltage potential, as confirmed by optical profilometry analysis.
3. Hydrophilic surfaces were achieved using the innovative dry electropolishing technique, as evidenced by the wettability test. This significantly contributes to minimizing bacterial adhesion to the substrate.

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