Brazilian Journal of Clinical Medicine and Review

PUBLICAÇÕES CIENTÍFICAS DE ACESSO ABERTO

Original Research

Electrochemical Analysis of the Influence of Brushing on the Corrosion Resistance of CP Ti Alloy

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Citation: Azenha MR, Kato RB, Souza DR, Oliveira ES, Dias EB, Ferreira PM, Col G, Bezzon OL. Electrochemical Analysis of the Influence of Brushing on the Corrosion Resistance of CP Ti Alloy. Brazilian Journal of Clinical Medicine and Review. 2025:Jan-Dec;03(1):bjcmr6.

https://doi.org/10.52600/2763-583X.bjcmr.2025.3.1.bjcmr6

Received: 13 June 2024
Accept: 11 July 2024
Published: 15 July 2024

Abstract: The use of metal alloys in dentistry continues to be applied in a large number of clinical situations involving conventional prostheses or those on implants. The objective of this study was to evaluate the effect of brushing and corrosion on the surface of CP Ti. Specimens with a diameter of 15 mm and a thickness of 2.5 mm were prepared using the lost-wax casting method. They were then divided into 2 groups (n=6) and subjected to different tests, as described in the materials and methods section. The results obtained showed no statistically significant change in the surface roughness of the specimens between the tested groups. However, images obtained through 3D Digital Interferometry and Scanning Electron Microscopy suggested the formation of surfaces with fewer irregularities in group 2, which was subjected to mechanical brushing before the electrochemical tests. The oxide resistance (Roxide) values observed after EIS at -250 mV were statistically different from each other ($p \leq 0.05$), with group 1, subjected only to the electrochemical test, showing lower oxide resistance compared to group 2, which was brushed mechanically before the electrochemical test. It was concluded that brushing positively influences the corrosion resistance of CP Ti.

Keywords: Corrosion; Abrasion; Brushing; Titanium.

1. Introduction

Modern dentistry presents a great variety of metallic materials developed through in vitro and in vivo tests, with metal alloys being applied in numerous clinical situations such as conventional prostheses and those on implants. The behavior of metallic materials in oral rehabilitations depends on biological aspects such as interaction with oral structures, making the structural conditions to which they will be subjected of utmost importance for achieving long-term clinical success [1,2].

One of the biggest concerns for professionals working with dental prostheses is the occurrence of failures that can compromise the results of rehabilitations, the most common being biocorrosion, fatigue, material fractures, wear, and metal allergies. Corrosion resistance is considered the main factor to be analyzed in an alloy, as it derives from its fracture resistance and the maintenance of the aesthetic/functional quality of oral rehabilitation. All dental metallic materials are subject to corrosion, especially when present in the unstable environment of the oral cavity. Factors such as temperature fluctuations, moisture presence, significant pH changes due to diet, material alterations in the presence of oxygen, and decomposition greatly contribute to the onset of corrosion and the degradation of materials [2-4].

With the increasing demand for oral rehabilitations with high aesthetic and functional requirements, titanium dental implants are increasingly recommended as the best rehabilitative option. These implants are routinely exposed to various adverse factors, such as temperature and oxygen level changes (due to food and saliva changes), masticatory forces, and chemical components that contribute to the metal degradation process. Therefore, it is essential to study the alloys involved in the process of replacing lost teeth. The aim of this study was to verify through electrochemical analysis the influence of the abrasive process that occurs during brushing on the corrosion resistance of CP Ti alloy, helping professionals maintain the longevity of restorative dental materials.

2. Material and Methods

To carry out the experimental phase of this research, commercially pure titanium alloy was used, as described in Table 1, with its chemical composition explicitly stated in Table 2.

2.1 Sample Preparation

2.1.1 Obtaining the Specimens

The specimens were prepared in the form of pellets, with a diameter of 15.0 mm and a thickness of 2.5 mm, using the lost-wax casting method from patterns obtained through a Teflon mold made in the precision workshop of the Department of Dental Materials and Prosthesis at the School of Dentistry of Ribeirão Preto – USP (Figures 1A, 1B, and 1C).

The spacer placed between the plunger and the cylinder ensures the thickness of the wax pattern. With the mold assembled, excess liquefied Picodip wax (Renfert, Hilzingen, Germany) was dripped into the Teflon mold, previously isolated with petroleum jelly, using a dropper. The wax was liquefied in the Hotty Led digital wax heater (Renfert GmbH, Hilzingen, Germany), which allowed the wax to be controlled at a temperature of 75 ± 5°C, as specified by the American Dental Association specification number 4.

Table 1. Dental Alloy.

Material	Commercial Name	Origin	Abbreviation
Titanium – CP	Tritan	Dentaurum, Inc, Pforzheim, Germany	Ti-cp

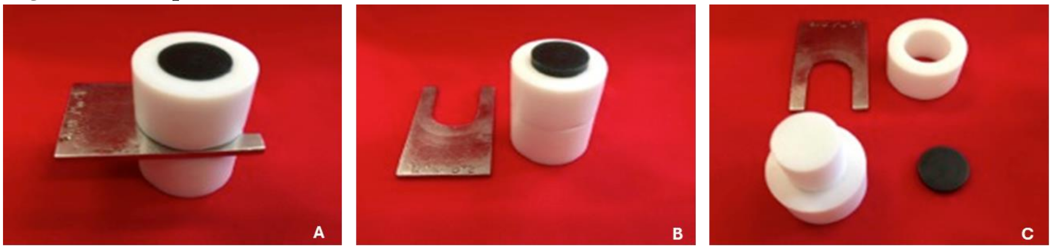
Table 2. Chemical Composition of the Dental Alloy (wt.%).

Alloy	Ti (%)	Others (%)
Ti-CP	99.5	0.5

A waiting time of 10 minutes was adopted to allow the wax to solidify completely at room temperature. After this period, the excess wax was removed, and the surface was smoothed with the aid of a flat spatula. Then, the wax pattern thus prepared was carefully removed from the Teflon mold by removing the spacer and activating the plunger (Figure 2).

Figure 1. Dental alloy. Mold for the fabrication of sample discs. A. Disassembled mold. B. Assembled mold, anterior view. C. Assembled mold, top view.

Figure 2. Complete solidification of the wax in the mold.



2.1.2 Embedding and Casting of Wax Patterns

For embedding the wax patterns, a specific investment material for the alloy used was employed, as described in Table 3. The wax patterns were embedded in Rematitam Plus investment (Dentaurum, Ispringen, Germany) manipulated under vacuum, following the manufacturer's instructions. Five specimens were included in the same silicone ring (Figure 3).

Table 3. Investment Material Used.

Material	Commercial Name	Origin
Investment	Rematitam Plus	Dentaurum, Ispringen, Germany

Figure 3. Specimens attached to the sprue for embedding in the investment material.

Next, the investment rings were placed in the Edgcon 5P oven (EDG, São Carlos, Brazil). The heating cycle of the rings was carried out according to the manufacturer's recommendations: heating at a rate of 5°C/min from room temperature to 150°C, holding at this temperature for ninety minutes; heating at a rate of 5°C/min to 250°C, holding at this temperature for ninety minutes; heating at a rate of 5°C/min to 1000°C, holding at this temperature for sixty minutes; cooling at a rate of 5°C/min to 400°C. After being removed from the oven, the rings were placed in the Rematitan machine (Dentaurum, Pforzheim, Germany), and the castings were performed by electric arc under vacuum and argon atmosphere, with metal injection under vacuum-pressure.

2.1.3. Sandblasting and Preparation of Specimens

After cooling in water, the rings were de-embedded, and the specimens obtained were sandblasted (Multijet III – EDG Equipamentos e Controle Ltda., São Carlos, Brazil) to remove adhered investment material. This sandblasting was performed with aluminum oxide particles of approximately 110 μm in size, with a pressure of 80 psi (5.62 kgf/cm²) to remove residual investment and oxides deposited on the surface.

Next, the specimens had their ends separated from the sprue. After being separated with a carborundum disc, the specimens were prepared with carbide and aluminum oxide burs. The face analyzed for corrosive behavior underwent metallographic polishing using a polishing machine (Motorized Grinder – Polipan-U – Pantec), starting with 180-grit sandpaper and progressing to 240, 360, 400, 600, 800, 1200, 2000, and finishing with felt discs and a silica suspension solution. After completion, the specimens were divided into 2 groups (n=6) according to the tests performed (Table 4).

Table 4. Group Division According to the Tests Performed on the Samples.

Group	Tests	Sequence of Tests
G1	C	OCP + PA (at -250 mV) + EIS (at -250 mV)
G2	E.M. + C	Mechanical brushing (14,600 cycles) + C1

Legend: OCP. Open Circuit Potential. PA. Anodic Potential. EIS. Electrochemical Impedance Spectroscopy.

2.2 Testing Stages

2.2.1 Mechanical Brushing Test

For simulated brushing, a Pepsodent-type brushing machine (MAVTEC - Com. Peças, Acess. e Serv. Ltda. ME, Ribeirão Preto, SP, Brazil) was used. For each test, a soft brush (Tek, Johnson & Johnson Ind. Com. Ltda., São José dos Campos, SP, Brazil) was used per specimen. The handles of the brushes were removed with the aid of a low-speed motor, straight handpiece, and maxicut bur (Edenta AG, Au, Switzerland) to fit the brush heads into the machine's holders and secure them with screws on the sides and top of the holders. The weight exerted by the machine on the specimen, with the brush attached, is 200 grams. The brush stroke length corresponds to 3.8 centimeters.

To ensure the perfect adaptation of the sample and maintain the same position during brushing, the specimens were fixed in plexiglass plates (Acrilpress Artefatos de Acrílico Ltda, Brazil) designed with a circle in the center matching the dimensions of the specimen and fixed in the machine's tubs. Six samples were brushed simultaneously at a speed of 356 rotations per minute. A volume of 20g of toothpaste was suspended in 20ml of distilled water (1:1 ratio) and mixed in a vacuum mixer A 300 (Polidental Ltda., Cotia, SP, Brazil) and then poured with a plastic syringe (10 ml of solution) equally into each tub over the specimens.

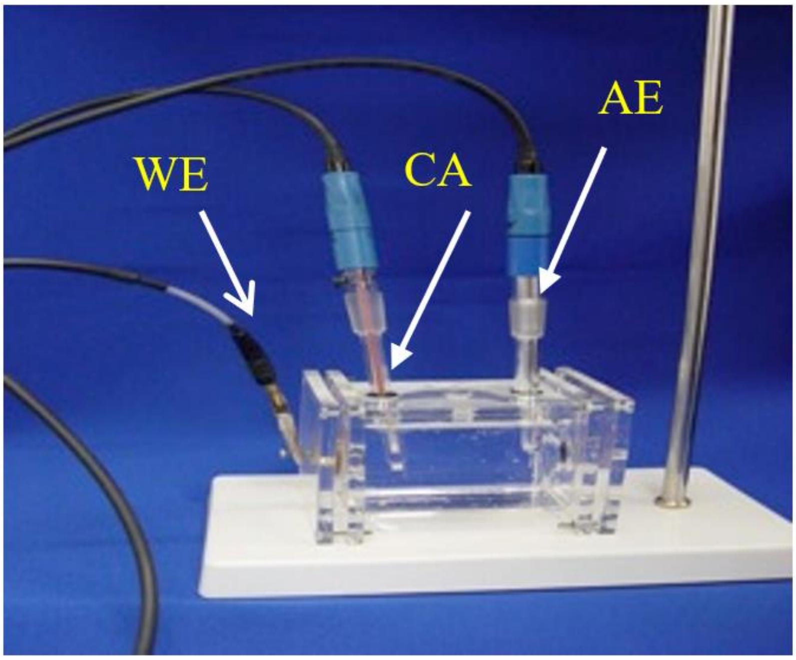
The machine was run until its display reached 14,600 brushing cycles, representing one year of brushing by a healthy individual [5], which corresponds to 41 minutes of machine operation. After this period, the machine was turned off, the specimens removed, washed in running water, and dried. For the samples in Group 2, the corrosion test was performed after brushing. Group 1 was not subjected to mechanical brushing.

2.2.2 Electrochemical Corrosion Test

To determine the corrosive behavior of the alloy, samples from all groups were placed in an ultrasonic cleaner (Ultrasonic 1440 D Comércio de Equipamentos Médico - Odontológicos Ltda, Brazil) for cleaning and removal of any grease residue from the surface of the specimens with isopropyl alcohol for 15 minutes. Then, the surface to be analyzed was again placed in the ultrasonic cleaner in distilled water for 10 minutes to remove the alcohol, which could otherwise alter the analysis. After the ultrasonic cleaning, the surface was dried with hot air.

After preparing the surface, the specimens were mounted, one at a time, in an appropriate electrochemical cell for corrosion tests. For corrosion tests, a potentiostat/galvanostat (PGP201 model, Radiometer Copenhagen, Denmark) was used, assisted by the Voltamaster 1 software provided by the same company. The electrochemical cell consisted of a 500 ml container and an acrylic lid with holes. Through these holes, the following electrodes were introduced and fixed: (WE) - working electrode, made of the material to be tested; the saturated calomel reference electrode (SCE); and the platinum auxiliary electrode with a 1 cm^2 area (AE). Figure 4 shows the assembled electrochemical cell.

Figure 4. Electrochemical cell assembled for the start of the study.



The electrolyte used was artificial saliva at a pH of 5.5, based on the Fusayama formula modified by Meyer, at a temperature of 37°C, controlled by an incubator made in the precision workshop of the Department of Dental Materials and Prosthesis at the School of Dentistry of Ribeirão Preto – USP, to simulate the temperature of the human body (Figure 5).

For the Open Circuit Potential (OCP) analysis, the measurement period was 1 hour, with the sample potential recorded every 0.6 seconds. The corrosion potential is the OCP value after stabilization. In Group G1, OCP analysis was performed, followed by the application of an Anodic Potential at -250 mV for 1 hour, and then Electrochemical Impedance Spectroscopy (EIS) at -250 mV. Group G2 was first subjected to the mechanical brushing test, with the machine running until it reached 14,600 cycles, following the methodology proposed by Wiegand et al. [5], representing 1 year of brushing by a healthy individual, which corresponds to 41 minutes of machine operation. Subsequently, the same corrosion test sequence as Group G1 was performed, namely: OCP + Application of Anodic Potential at -250 mV + EIS at -250 mV.

Figure 5. Incubator made at FORP/USP.

2.2.3 Surface Characterization

2.2.3.1 3D Digital Interferometer

After the mechanical brushing and electrochemical corrosion tests, the specimens were subjected to surface analysis using a 3D Digital Interferometer (Zygo New View 6300, Zygo Corporation, Middlefield, CT, USA) at Rush University Medical Center – Chicago, Illinois, to check for possible changes in surface roughness after the abrasion and corrosion processes.

2.2.3.2 Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) was performed as an additional method for surface characterization of the specimens after the proposed tests for each group.

2.2.3.3 Statistical Analysis

The values obtained after the electrochemical tests and roughness measurements were subjected to one-way ANOVA statistical analysis and Duncan's statistical analysis. The ANOVA test was used to compare the means between the different studied groups, aiming to find statistically significant differences between the samples, without indicating which means differ from each other. To detect exactly where the differences between the means lie, Duncan's Test was used, complementing the variance analysis (ANOVA).

3. Results

3.1 Open Circuit Potential (OCP)

The mean values from the OCP test were grouped in Table 5 and Figure 6.

Table 5. Mean Open Circuit Potential Values.

Group	Mean OCP (V)
Group 1 (C)	-0.212 ± 0.041a
Group 3 (E.M.+C)	-0.210 ± 0.027a

Figure 6. Mean Open Circuit Potential (OCP) values for Groups 1, 2, 3, and 4.

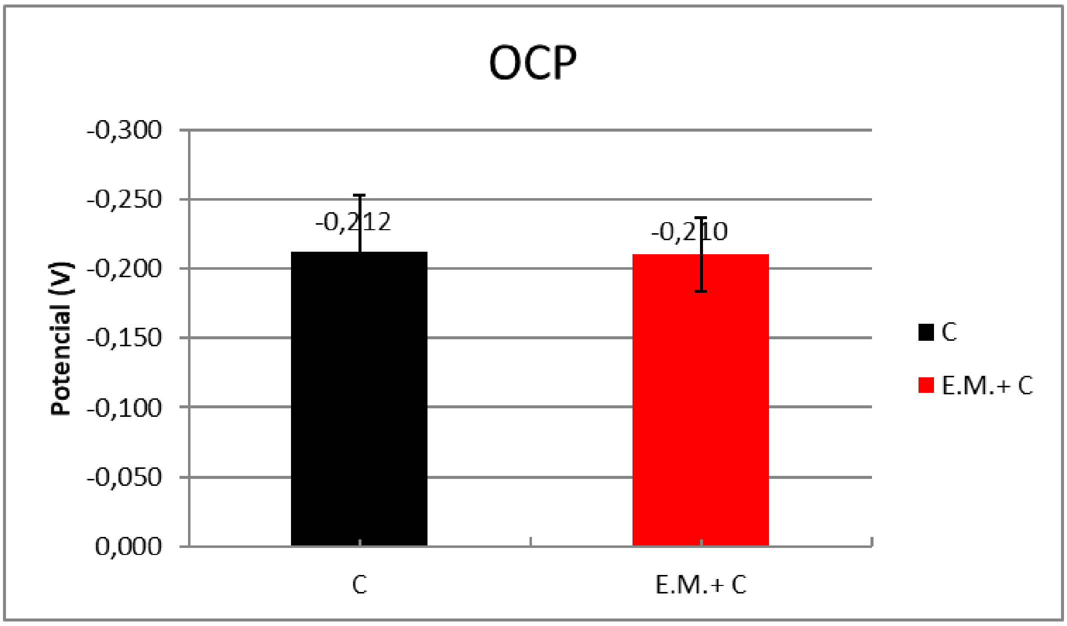
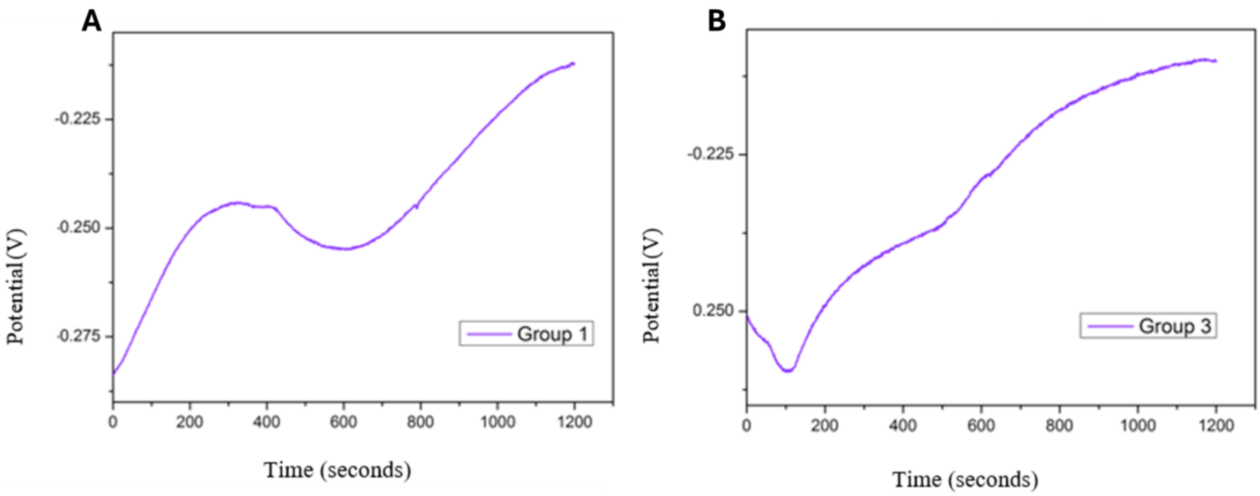


Figure 7 presents the OCP evolution curves obtained from the average values recorded for each specimen in each group during the 20-minute test. Observations of Figures 7A and 7B show that during the electrochemical test, there was an increase in potential for all groups with different curve patterns, indicating that all samples experienced similar corrosion.

The initial average potential of the groups was different, with Group 1 showing the lowest value (-0.285 V) compared to Group 2 (-0.250 V). The average potential values recorded for the Group 1 specimens show a gradual increase in corrosion during the first 5 minutes, followed by a slight decrease from the fifth to the tenth minute, and a new increase in the last 10 minutes, reaching an average value of -0.212 V at the end of the 20-minute test. For Group 2, the OCP averages showed a decrease in the first few minutes followed by a gradual increase, reaching an average value of -0.210 V at the end of the electrochemical test. However, even with the variations between the groups, statistical analysis (one-way ANOVA) showed no significant difference between the tested groups ($p > 0.05$), indicating that mechanical brushing prior to OCP tests had no significant statistical impact.

Figure 7. A and B. Evolution of open circuit corrosion potential for groups 1, 2, 3, and 4.



3.2 Electrochemical Impedance Spectroscopy (EIS)

The mean values from the Electrochemical Impedance Spectroscopy (EIS) test were grouped in Table 6 and Figure 8. They represent the average polarization resistance of the specimens in each group after the electrochemical and mechanical tests. These results were analyzed using the Z View 2 Software. The Simple Circuit (Figure 9) was found to be more compatible with the obtained data and was used as a standard during the analysis of the results.

Table 6. Roxide Values (MΩ/cm²) Obtained from EIS.

Group	Roxide (MΩ/cm²)
Group 1 (C)	0.62 ± 0.12a
Group 2 (E.M.+C)	2.10 ± 0.68b

Figure 8. Roxide Values (MΩ/cm²) Obtained from EIS.

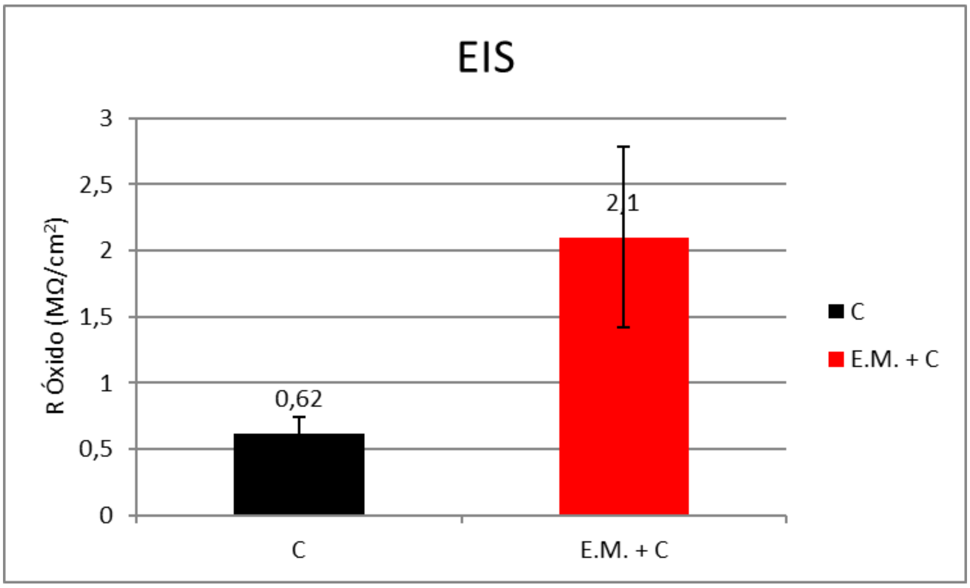
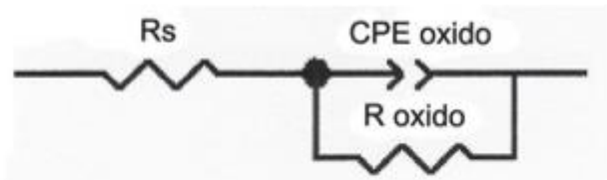


Figure 9. Simple Circuit.



The oxide resistance (Roxide) observed in the tested groups shows statistically different values ($p \leq 0.05$), with Group 1, subjected only to the electrochemical test, demonstrating lower oxide resistance, which means increased corrosion susceptibility of the tested surface, compared to Group 2 that was mechanically brushed before the electrochemical test. This brushing technique demonstrated greater effectiveness in protecting the specimen surfaces against corrosion, thereby increasing the oxide resistance.

3.3 Surface Characterization

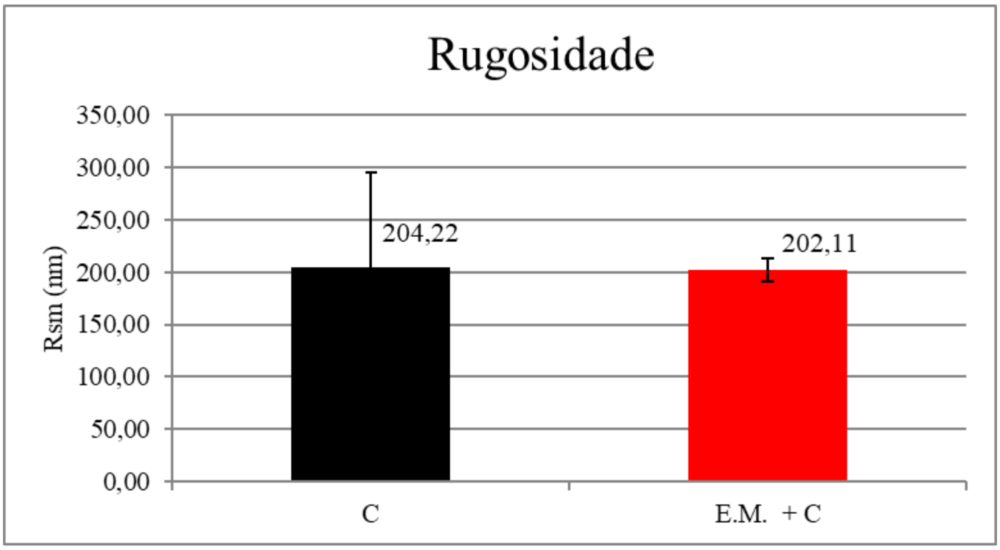
3.3.1 3D Digital Interferometer

Surface analysis using the 3D Digital Interferometer (Zygo - Rush Medical Center, Chicago, Illinois) was conducted to characterize the surfaces of the specimens and measure the roughness obtained after the mechanical and electrochemical tests. The roughness values (Rms and Ra) obtained for each group, using MetroPro Software, were compiled in Table 7 and Figures 10 and 11.

Table 7. Roughness Values (Rms and Ra) after Proposed Electrochemical and Mechanical Tests for Each Group.

Group	Rms (nm)	Ra (nm)
Group 1 (C)	204.22 ± 90a	162.62 ± 75a
Group 3 (E.M.+C)	202.11 ± 11a	164.06 ± 9a

Figure 10. Roughness Values (Rms) for Groups 1, 2, 3, and 4.



The final condition of the specimen surfaces after the proposed tests for each group is detailed in different presentation formats. The surface can be qualitatively analyzed from a flat, golden-colored image that portrays the nanometric characteristics of the surface. Quantitative analyses can be performed based on the images in Figures 12 and 13, which quantify the surface irregularities in nanometers, with the data presented in bi- and three-dimensional formats, facilitating a better understanding of the obtained data.

After statistical analysis (One-way ANOVA), it was found that there was no significant difference in the roughness values obtained between Groups 1 and 2, regardless of whether the surfaces were subjected to mechanical brushing. This indicates that repeated brushing cycles do not disadvantage or harm the metal.

Figure 11. Roughness Values (Ra) for Groups 1, 2, 3, and 4.

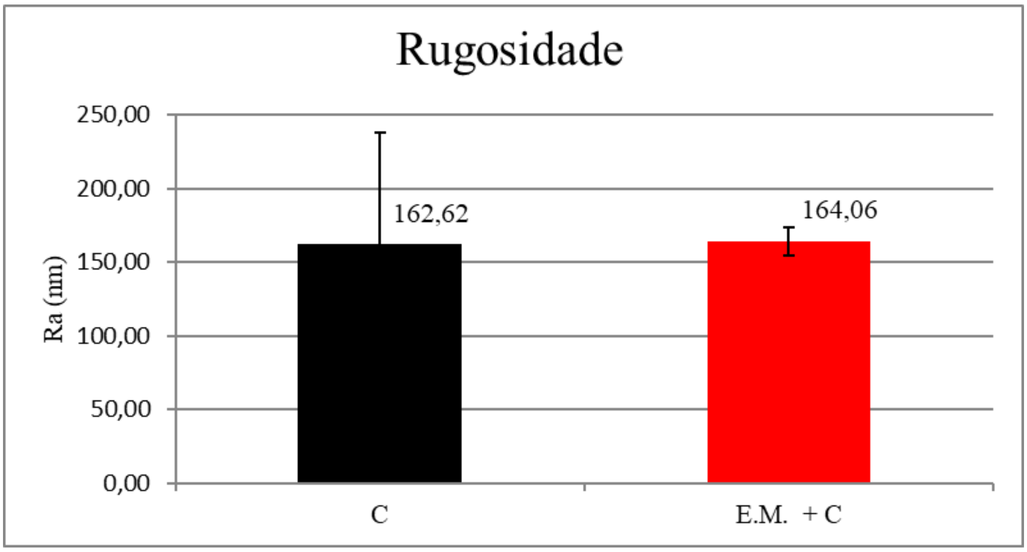


Figure 12. Quantitative analysis in nanometers of the surface irregularity of the metals analyzed in Group 1.

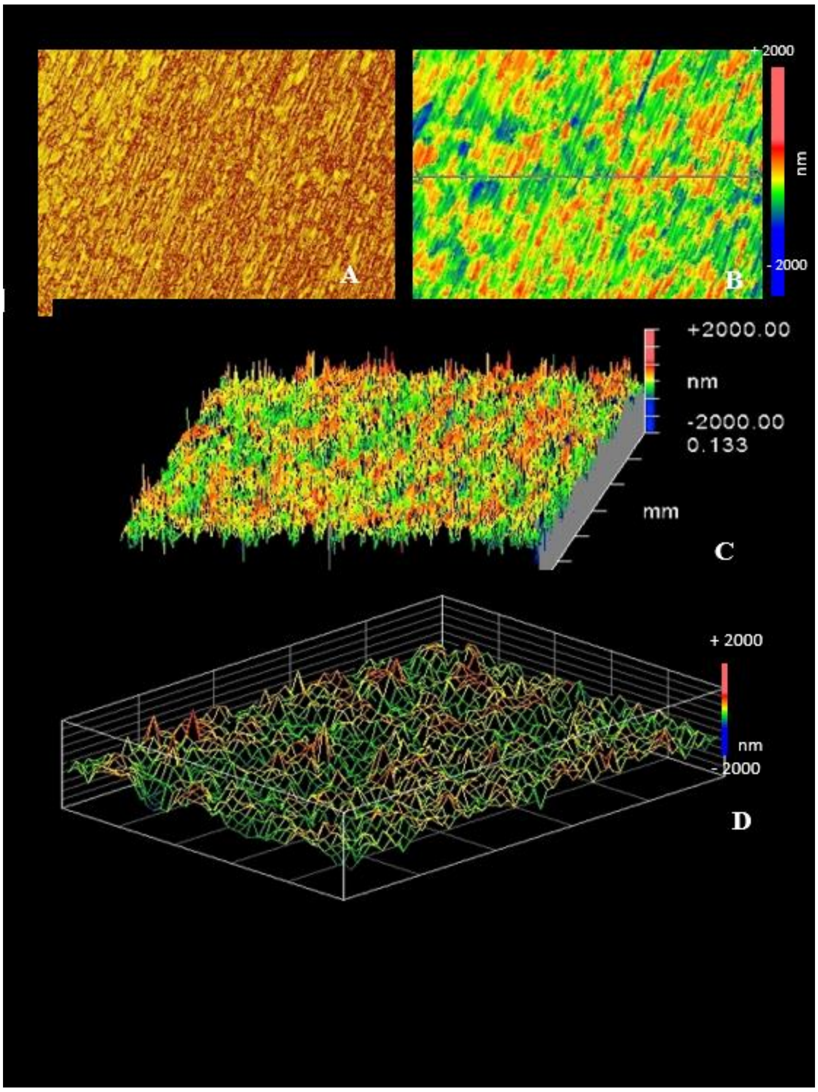
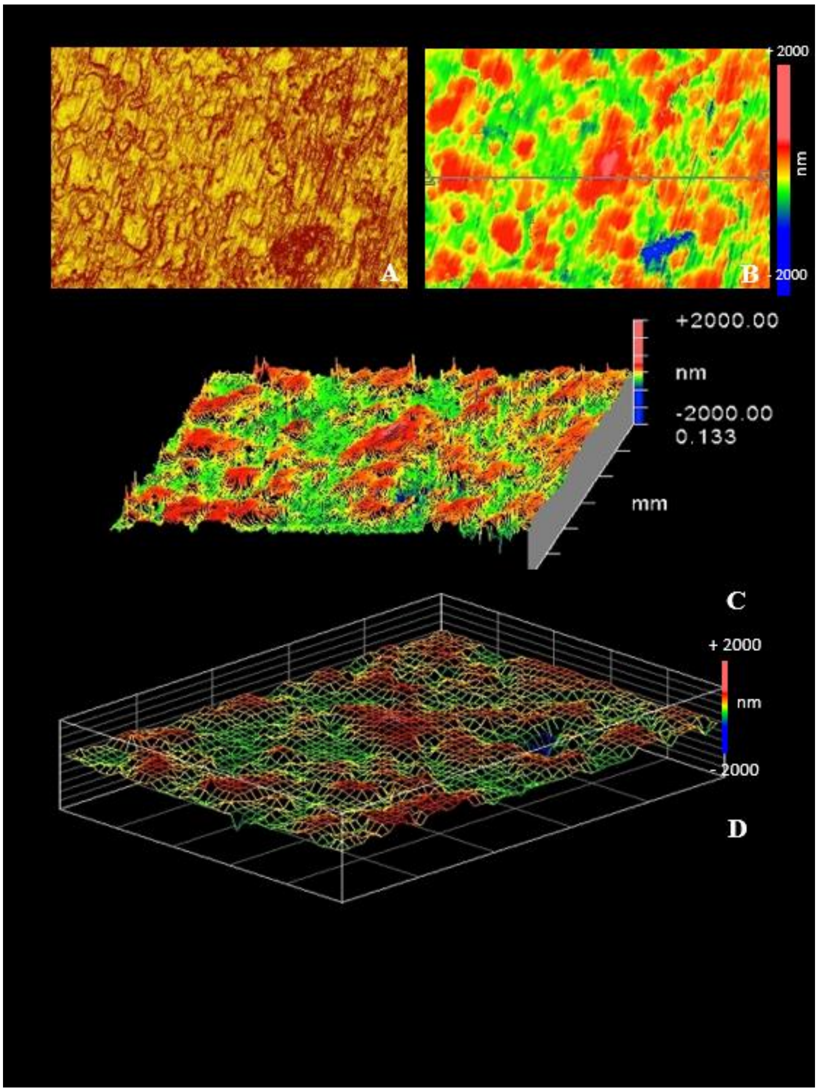


Figure 13. Quantitative analysis in nanometers of the surface irregularity of the metals analyzed in Group 2, where a flatter surface with fewer irregularities can be observed compared to Group 1.



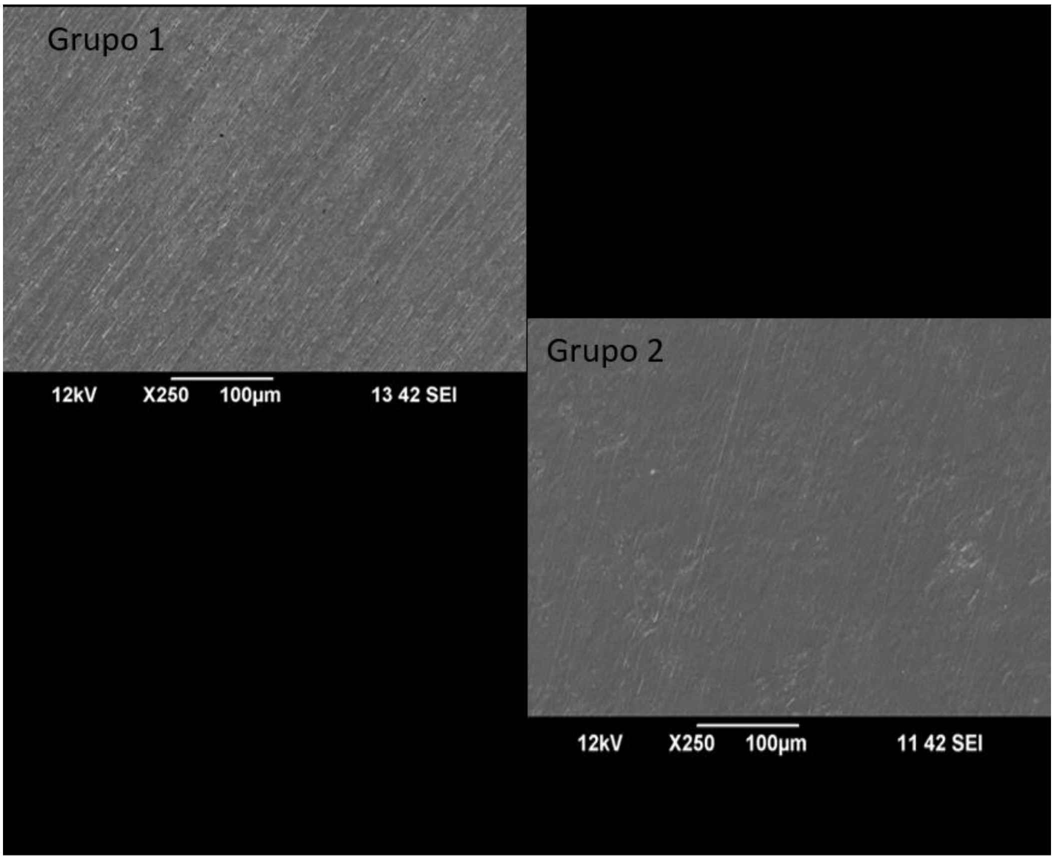
3.3.2 Scanning Electron Microscopy (SEM)

Similar to the analysis performed with the 3D Digital Interferometer, SEM (Figure 14) was conducted to provide qualitative analysis of the specimen surfaces after the proposed mechanical and electrochemical tests. The images show that the surface of the specimens in Group 1 is more irregular compared to the surfaces of the specimens in Group 2, which were brushed and subsequently subjected to the same corrosion tests. These findings are consistent with the results from the 3D Digital Interferometer analysis, demonstrating that specimens subjected to mechanical brushing followed by corrosion or oxidation tests exhibited less surface irregularity or corrosion, thus providing better protection to the metal.

4. Discussion

The corrosion resistance of dental alloys has been studied and tested for years to prevent failures in dental treatments due to corrosion-related material failures. Over the years, a significant amount of metal alloys has been intensively studied, ranging from dental amalgam, still widely used for direct restorations, to gold alloys, including silver, copper, cobalt-based, nickel-based, and titanium-based alloys. The latter is almost always associated with procedures involving oral rehabilitation through implant placement [6-9]. With the increasing use of titanium-based alloys due to the growing popularity of implantology techniques, numerous tests and studies have been proposed and conducted to assess the biocompatibility and performance of this material in the oral environment. This study aimed to evaluate the degradation or oxidation of titanium surfaces after brushing cycles and electrochemical surface interference, demonstrating that brushing was beneficial in reducing these parameters.

Figure 14. SEM images of the sample surfaces from Groups 1 and 2. Greater irregularity is observed on the surface of Group 1.



In 1979, Duc and Tissot [10] developed a closed cell for corrosion testing and a polarization program to evaluate metal surfaces, being pioneers in this type of testing. Following the studies initiated by these authors, different methodologies and techniques have been developed to characterize the metallic surfaces used in dentistry [11-13]. Currently, similar techniques are still employed in electrochemical tests of dental alloys. Mathew et al. [14] investigated the combined effect of chemical corrosion and wear (tribocorrosion) on the degradation of dental implant materials (CP Ti) under various pH conditions in the oral environment. Although experiments demonstrated that CP Ti is electrochemically stable up to pH 2.0, these authors found peaks of metal surface degradation at different pH levels, including near-neutral values. This demonstrates the importance of saliva and mechanical cleaning of the metal surface to reduce its degradation.

To simulate an in vitro environment as close as possible to the oral cavity and the durability of metal alloys in oral rehabilitations, various studies and methodologies are tested in different media, such as metals exposed to artificial saliva [8, 9, 11], different fluorides [13, 15], peroxide [16], and biofilm [2, 6, 7, 9]. The results vary and are presented through different methodologies but converge in emphasizing the importance of mechanical cleaning of the metal to reduce its corrosion and degradation over time. Studies on the interaction of corrosion with abrasion (tribocorrosion) have shown that this association plays an important role in the longevity of dental alloys [7,14], findings that can be considered similar to those demonstrated in our study. Without a doubt, the mechanical brushing process tends to promote greater durability of titanium alloys, as clearly observed by the reduced surface wear of our samples in the images presented by the 3D Digital Interferometer and SEM.

The oxide resistance (Roxide) results observed in the tested groups show statistically different values ($p \leq 0.05$), with Group 1, subjected only to the electrochemical test, demonstrating lower resistance compared to Group 2. These data suggest that brushing before the electrochemical test contributed to increasing the resistance of the oxide layer formed on the specimen surfaces. Similar results related to the corrosion resistance of metal alloys subjected to daily abrasion were reported by Benatti and colleagues in 2000 [17]. The authors conducted in vitro and in vivo studies to determine the corrosion resistance of different alloys immersed in artificial saliva, 0.9% sodium chloride, and 1.0% sodium sulfide. For the in vivo test, the samples were included in full dentures, with one surface exposed to the oral cavity of patients at different sites. They observed that alloys maintained in the self-cleaning regions of the oral cavity did not show substantial corrosion, demonstrating the importance of cleaning or brushing the metal surface for greater longevity and reduced wear. In the present study, we used commercially pure titanium alloy to complement previous studies using this alloy in mechanical tests [7-9, 13, 14].

When exposed to the environment, titanium promotes the formation of a dense and stable titanium oxide layer on its surface, playing an important role in the material's corrosion resistance. When this same metal is exposed to an acidic environment, fluorides, and saliva, the protective layer formed on the surface tends to be removed, and the corrosion process begins. The corrosion process is also influenced by the metal surface's contact with other metals, food, and brushing trauma [11, 13, 15, 17, 18]. However, few studies have been conducted on the corrosion or oxidation behavior of titanium surfaces after repeated brushing cycles simulating daily use. In this study, we observed that, contrary to expectations, mechanical brushing before electrochemical tests revealed better results regarding oxidation and surface characterization of the analyzed metals. These results encourage further research to find more concrete explanations for the observed behavior.

4. Conclusions

It is concluded that brushing positively interferes with the corrosion resistance of CP Ti, making the surface oxide layer more regular and resistant to corrosion, indicating that brushing can be beneficial in cases where the metal is exposed in the oral cavity after rehabilitation with dental implants. With these initial results, we can affirm that the removal of biofilm or plaque through manual brushing reduces the oxidation and wear of the titanium alloy, promoting greater longevity of oral rehabilitations. We believe that further studies using similar methodology are necessary to confirm our results. One suggestion is to use this same methodology to test titanium that has undergone different surface treatments to accelerate the osseointegration process.

Funding: None.

Research Ethics Committee Approval: None.

Acknowledgments: None.

Conflicts of Interest: The authors declare no conflict of interest.

Supplementary Materials: None.

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