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ELECTROLYTIC PLASMA OXIDATION FOR APPLICATION IN IMPLANTABLE DEVICES

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Abtract: Based on the constitutional principles of comprehensiveness and equity, it has become necessary to enable the incorporation of orthodontic procedures and dental implants for the population. Despite the implementation of a maximum of 6 implants per patient in dental specialty centers, the cost of surface treatment for implants is still high and has an impact on the final price for the industry. In order to solve this problem, the Plasma Electrolytic Oxidation (PEO) surface treatment method was used. This process is based on the generation of an ionized gas, through an electrochemical process, for the deposition of a ceramic layer on titanium. The SEM showed that the coatings were porous, with a well-adhered interface and no voids. The AFM images showed that with increasing treatment time there was greater roughness and increased homogeneity of the distribution of ceramic crystals on the surface. The wettability tests showed a smaller wetting angle for the PEO-treated samples. As a result of this technique, there was an improvement in the chemical and morphological quality of the surface, enhancing osseointegration and reducing the cost of treatment.It can be concluded that the Electrolytic Plasma Oxidation technique proved to be effective in depositing a ceramic coating, improving surface quality, reducing the cost of surface treatment and lowering the final value of the implant.
Keywords: Surface treatment, Dental implants, Titanium, PEO.

INTRODUCTION

Biomedical implants have emerged to solve a number of problems in the health sector, especially in the rehabilitation of patients through dental implants, improving their quality of life. In accordance with the constitutional principles of integrality and equity, it has become necessary to enable the incorporation of dental implant procedures by the

public health sector. According to Ministerial Order No. 718/SAS of the Ministry of Health, published by the Secretariat for Health Care, there has been a 35% reduction in the decay component. Despite the implementation of a maximum of 6 implants per patient in dental specialty centers, the cost of implant surface treatment is still high and has an impact on the final price for the industry.

The application of coatings on the surface of metals to improve properties such as appearance, resistance to wear and corrosion, adhesion, among others, is a viable and widely used solution (RAJ and MUBARAK, 2009). Dental implants have become the best tool for rehabilitating patients since the 70s. In dentistry, these implants were produced to follow a strict surgical protocol that allows the treatment of individuals with total or partial missing teeth, and they continue to be used to this day. Although the success rate of dental implants is high, there are still failures. Most of these occur after the implants have been inserted within the first year. Currently, the search is on for better and faster osseointegration, given that the implants are in direct contact with the biological environment (WISMEYER, WASS and VERMEEREN, 1995).

For better osseointegration, many technologies have been developed to enhance the chemical, mechanical and biological properties of these surfaces, introducing rapid, guided bone repair and enabling lasting function (PULEO and NANCI, 1999).

Among the biomaterials used, commercially pure titanium (cpTi), titanium alloys with aluminum and vanadium (Ti-Al -V_{64}) have become the most widely used metallic materials for making dental and orthopedic implants, due to the numerous properties these metals have. Chemical stability, biocompatibility, good biomechanical properties and bioinert surface oxide formation can be cited as characteristics for choosing an appropriate biomaterial (PIRES, BIERHALZ and MORAES, 2015). However, they do not form an extremely satisfactory chemical bond with bone tissue. To this end, studies have attempted to modify and even improve their surface properties and increase the degree of biocompatibility of the implant in relation to bone tissue, in order to promote better osseointegration (WISMEYER, WASS and VERMEEREN, 1995 and BECK, LANGE and NEUMANN, 2007).

Once implants come into contact with the biological environment, they are subjected to various dynamic changes in their bioliquid and surface properties, which trigger a sequence of reactions between the biological environment and the biomaterial. At this point, a "conditioning film" is formed which modulates the host's cellular responses. The first molecules to reach the titanium surface are water molecules. Many studies show that the osseointegration process takes place more quickly when the surface of the implant is textured, as explained by Kasemo in 2002, due to the increased wettability of the surface by the biological liquid and not just the roughness, as previously believed. Surface wettability influences proteins, molecules and cells that subsequently reach the water (KASEMO, 2002 and SILVA et al, 2013).

The development of the bone-implant interface is complex and involves numerous factors. Factors related to the implant as a material and factors related to the biological environment. These include the shape, topography and surface chemistry, but also the mechanical load, surgical technique, and patient variables such as the state of the recipient bed, bone quantity and quality (PULEO and NANCI, 1999 and BECKER et al, 2013).

The formation of bone tissue requires the recruitment and proliferation of osteoblast precursor cells, which will differentiate into osteoblasts producing the non-mineralized

extracellular matrix, which will subsequently be calcified. These events are greatly influenced by certain properties of the surface of titanium implants. These properties are: chemical composition, surface energy and surface texture, a combination of topography and roughness (SCHWARTZ, Z. & BOYAN, 1994).

One of the techniques used today to accelerate osseointegration is plasma technology (DEHNAVI et al, 2013). This is described as a gas containing neutral and electrically charged species such as electrons, positive ions, negative ions, atoms and molecules produced by applying a potential difference between two electrodes (ALVES JR et al, 2005 and DZHURINSKIY et al, 2015).

To obtain coatings that accelerate and improve adhesion of bone tissue to titanium, plasma electrolytic oxidation (PEO) has been used. The PEO process uses a liquid medium (electrolyte) and the composition of the coating can be controlled by adjusting the composition of the electrolyte (SRINIVASAN, BLAWERT and DIETZEL, 2009).

Today, surface treatments represent a high cost for the industry, which impacts on the final price of each implant manufactured. Reducing this cost will make it possible and easier for the disadvantaged population to access these implants, minimizing the national problem of the edentulous.

In order to solve this problem, the Plasma Electrolytic Oxidation (PEO) surface treatment method was used. This process is based on the generation of an ionized gas, through an electrochemical process, for the deposition of a ceramic layer on titanium. As a result of this technique, an improvement in the chemical and morphological quality of the surface is expected, enhancing osseointegration and reducing the cost of treatment

METHODOLOGY

This study used 18 cp grade II titanium cylinders, 3 mm in diameter and 25 mm long, purchased from Singular Implants, Parnamirim/RN. Until the surface treatment by Plasma Electrolytic Oxidation (PEO), the samples were subjected to various processes and subsequently characterized as shown in the Flowchart below (Figure 1), in 3 stages: the first, the preparation of the samples, the second, the PEO treatments and the third, the characterization of the samples.

SAMPLE CLEANING PROCESS

The titanium cylinders underwent a rigorous cleaning process to eliminate surface impurities that could interfere with the oxidation process. Using a diluted solution of hydrofluoric (HF) and nitric (HN) acids, 5 ml of HF in 100 ml of deionized water and 5 ml of HNO_3 in 100 ml of distilled water, with volume fractions of 10% and 40%, respectively, the samples were immersed for 30 seconds to remove the oxide layer and surface contaminants (WANG et al, 2014). After this stage, the samples were ultrasonically cleaned (Plana^{TC} - CBU 100/3L) with acetone and distilled water for 10 minutes, respectively. The samples were then dried using a commercial hot air dryer (Taiff Turbo 6000), ensuring the removal of impurities that could contaminate the electrolytic solution.

PREPARATION OF THE ELECTROLYTE SOLUTION

With the samples dried, 6 liters of electrolyte solution were prepared in the following proportions of reagents: 10 g/l of Tribasic Sodium Phosphate P.A. (TSOP, Na_3 PO4 - 12H2O), 2 g/l of Potassium Hydroxide (KOH) in 1 liter of distilled water. 3 g/l of Tris Hydroxymethyl Aminomethane (C H_{411} NO_3) was added to the base electrolyte as an additive, to enable an adherent coating favorable to osseintegration (HARIPRASAD

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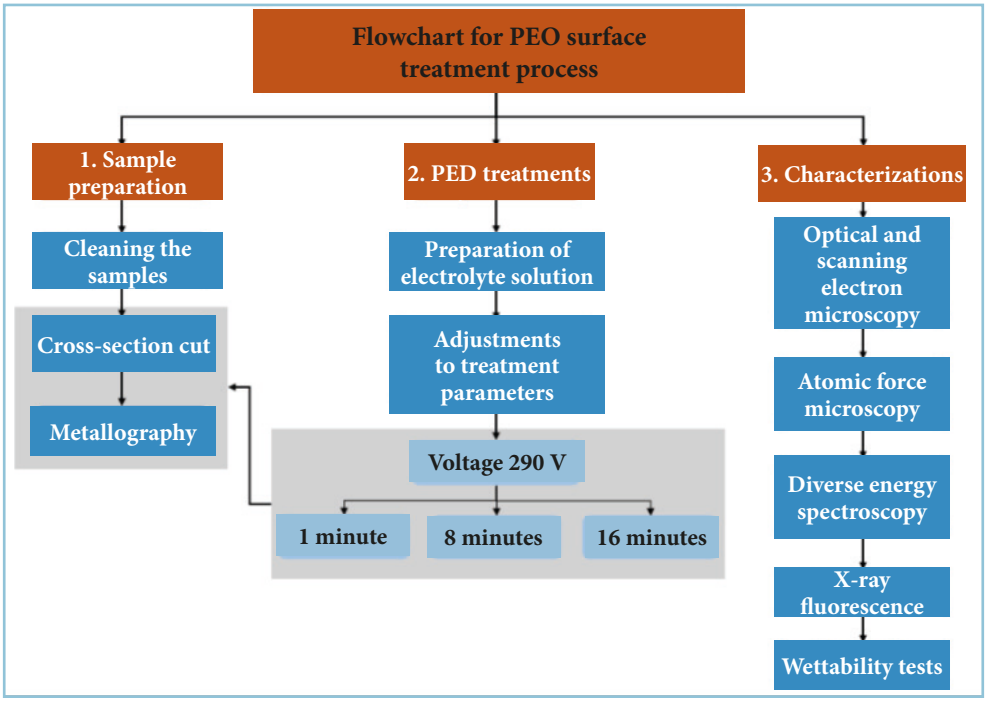

Figure 1 - Flowchart with the process for surface treatment by Electrolytic Plasma Oxidation. Author's own work (2018)

Source: Prepared by the author (2018).

et al, 2016). The aforementioned substances were weighed on an analytical balance (Quimis^{R} Q-500L210C) and then added to a 600 ml beaker and dissolved in 400 ml of distilled water. The solution was then poured into a 1 liter volumetric flask, topped up with distilled water and mixed for 1 minute. 600 ml of solution was used for each treated sample and, to ensure that equal treatment conditions were maintained, the electrolyte solution was changed at each experiment.

ELECTROLYTIC PLASMA OXIDATION

Figure 2(a) shows the experimental apparatus used to treat the titanium samples. The equipment has three PEO coating reactors, a magnetic stirrer, an electrolyte recirculation system, a flow control valve and a Tic 17RGTI digital thermocouple (-50 to 105° C), as shown in Figure 2(b). The titanium samples and the stainless steel tube were used as the anode and cathode, respectively.

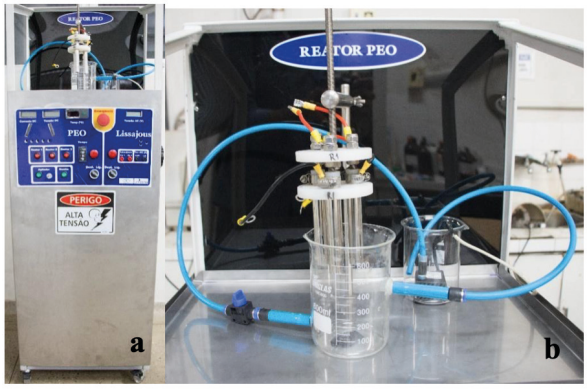

Figure 2 - (a) Electrolytic Plasma Oxidation (PEO) treatment equipment and (b) reactors, flow control system and thermocouple. Author's own work (2017).

Source: Prepared by the author (2018).

The cleaned samples were placed in the reactors of the PEO equipment and immersed in a 600 ml beaker with 400 ml of electrolytic solution. The treatments were carried out at times of 1, 8 and 16 min, subjected to a voltage of 290 V in direct current (DC), chosen because it was the best condition found for

this process. For each time adopted in this work, three placements were carried out (P1, P2 and P3), thus using 6 samples. Therefore, 18 samples were used for the three times. The electrical voltage, current and temperature of the solution were monitored and recorded every minute.

METALLOGRAPHIC PREPARATION

For optical microscopy and scanning electron microscopy analysis, the post-treated samples were selected for cross-section cutting, 4 mm from the end. After this stage, they were hot-embedded in bakelite, sanded with silicon carbide sandpaper with grain sizes of 120, 220, 360, 600, 1000 and 1200 mesh, and finally polished with colloidal silica composed of 60% hydrogen peroxide (H O_{22}) and 40% colloidal silica 0.06 μm. After this stage, the surfaces were cleaned with water and acetone and dried using a commercial hot air dryer.

CHARACTERIZATIONS

The samples were subjected to Optical Microscopy (OM) characterization of reflected light to analyze the thickness of the coatings using the Image Pro Plus surface morphology software with 5 layer thickness measurements for each sample (WHEELER et al, 2010). An Olympus BX 60M - Japan optical microscope was used, coupled with Image-Pro Plus software version 4.5.1.22 for Windows (serial number 41N41000-29998) Copyright 1993- 2002 Media Cybernetics, Inc. Scanning Electron Microscopy (SEM), using Shimadzu secondary electron equipment, was used for more precise thickness analysis, with 5 measurements for each sample. The chemical composition of the films was analyzed using X-ray and Energy Dispersive Spectroscopy (EDS).

WETTABILITY TESTS

The wettability measurements of the coatings after treatment were carried out by a goniometer using the pinacle software from the Plasma Materials Processing Laboratory (LabPlasma) at UFRN. The cylindrical samples were fixed horizontally. A fixed-volume micropipette was used, positioned perpendicular to the horizontal plane of the samples, depositing 5 μl of distilled water on the surface under study (ALVES, JR et al, 2005). The wettability values correspond to the arithmetic mean of 3 measurements taken after 5 seconds for each drop deposited on the surface.

RESULTS AND DISCUSSIONS

After the Electrolytic Plasma Oxidation treatments, it was observed that the reactor coated titanium rods with good performance and did not leak current in undue places. The equipment proved effective in depositing a ceramic layer. It was possible to control all the parameters in an ergonomic, practical and safe way and to monitor the variations in the oxidation process. The results of the voltage, current and temperature of the electrolytic solution were obtained at intervals of time for each sample, shown in Tables 1, 2 and 3, for 1, 8 and 16 minutes respectively. For 1 minute of treatment, the analysis intervals were every 0.5 min; for 8 minutes they were at an interval of 1 min; and for 16 minutes of treatment, the analysis interval was 2 min.

Sample with 1 minute of treatment	Average time (min)	Temperature (° C)	Voltage (V)	Current (A)
	0	19	240	0,60
	0,5	25	240	0,50
	1	26	280	0,40

Table 1 - Temperature of the electrolytic solution, Voltage and Current varying every 30 seconds for a 1-minute treatment. Author's own work (2017).

Source: Prepared by the author (2018).

	Average time (min)	Temperature (° C)	Voltage (V)	Current (A)
Sample after 8 minutes of treatment	1	31	282	0,40
	2	34	285	0,27
	3	35	287	0,25
	4	36	287	0,20
	5	37	288	0,19
	6	37	288	0,15
	7	39	288	0,16
	8	39	288	0,17

Table 2 - Temperature of the electrolytic solution, Voltage and Current varying every 1 minute for an 8-minute treatment. Author's own work (2017).

Source: Prepared by the author (2018).

	Average time (min)	Temperature (° C)	Voltage (V)	Current (A)
Sample with 16 minutes of treatment	0	25	284	0,47
	2	30	287	0,30
	4	31	286	0,16
	6	32	286	0,14
	8	32	287	0,11
	10	32	287	0,09
	12	32	288	0,07
	14	32	288	0,09
	16	33	288	0,09

Table 3 - Temperature of the electrolytic solution, Voltage and Current varying every 2 minutes for a 16-minute treatment. Author's own work (2017).

Source: Prepared by the author (2018).

It can be seen that the results are in line with the theoretical review, where the current decreases with increasing deposition time, since the ceramic layer formed increases the dielectric strength, decreasing conduction, promoting glow discharge and plasma formation, as shown by Parfenov et al, 2015.

Ceramic coating occurred on the titanium surface at all treatment times. During the general anodizing stage, a porous oxide film forms on the surface of the titanium alloy as described by Gowthan et al, 2016. The samples showed homogeneous and similar coatings, as seen in the example of a treated sample in Figure 3 (a) with a white color and matte appearance due to the deposition of the oxide. The untreated sample, Figure 3 (b), has the normal smooth visual appearance of titanium.

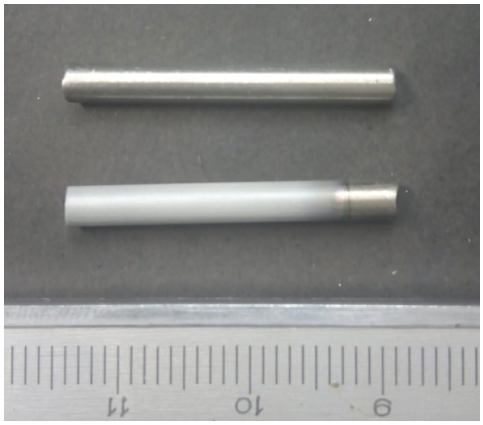

Figure 3 - Titanium rods with PEO surface treatment (a) and untreated (b). Author's own work (2017).

Source: Prepared by the author (2018).

The PEO process causes temperature peaks that melt the materials present in the medium and when they are rapidly cooled by the electrolyte, the molten oxide solidifies on the surface of the substrate. Due to the repeated melting and solidification process induced by the discharges, the temperature allowed the crystallization and transformation of the titanium oxide phase (TiO_2) from anatase to rutile, as described by Yeung et al, 2013. Also identified in this work through chemical analysis by X-ray fluorescence, TiO_2 in addition to the elements of the compounds that make up the electrolyte solution (TSOP, Na_3 PO_4 - $12H_2$ O), (KOH), ($(HOCH_2)$ $3CNH_2$). As a complementary analysis, Energy Dispersive Spectroscopy showed the elements present in the solution and in the deposited layer.

The micrographs analyzed by optical microscope and scanning electron microscope (Figures 4 and 5) show the formation of the ceramic layer deposited with the electrolyte solution. Deposition took place for all the samples between 1 and 8 minutes with an approximate thickness of 11 μm, with an

approximate thickness of 21 μm over 8 minutes. There was no significant increase in layer thickness when the treatment time exceeded 8 minutes, due to the high electrical resistance.

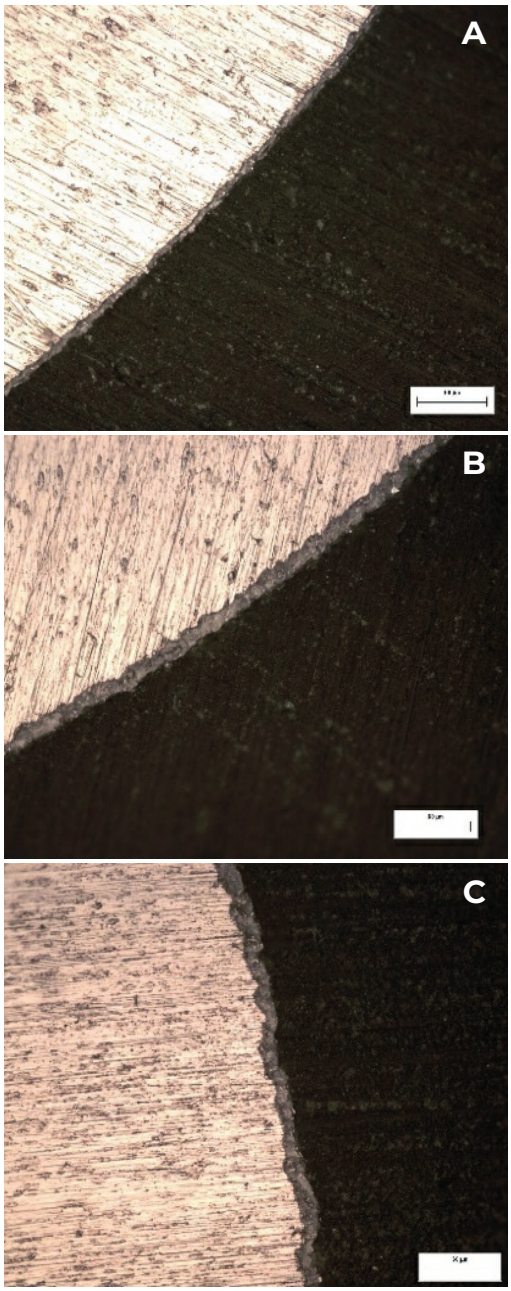

Figure 4 - Optical microscope micrographs at 500x magnification for: a - 1 minute of treatment; b - 8 minutes of treatment; c - 16 minutes of treatment.

Source: Prepared by the author (2017).

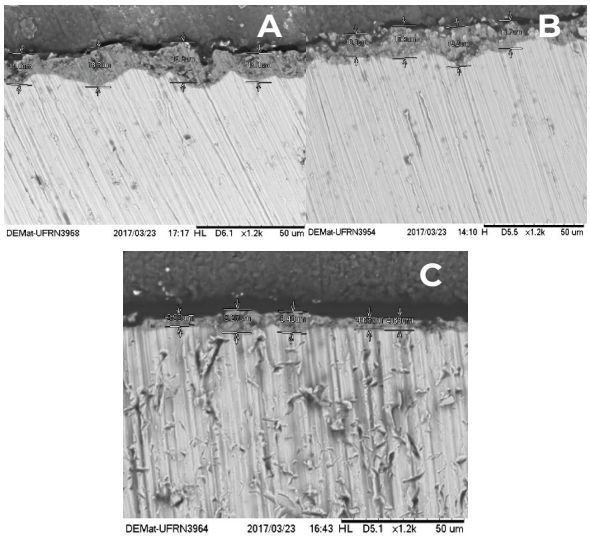

Figure 5 - Scanning Electron Microscope micrographs at 1200 x magnification for: a - 1 minute of treatment; b - 8 minutes of treatment; c - 16 minutes of treatment.

Source: Prepared by the author (2017).

All the coatings exhibit a common characteristic of a PEO process, presenting a porous structure in the outer layer, with a well-adhered titanium and coating layer interface and no voids, which provides greater resistance to wear of the layer, as in the study carried out by Hariprasad et al, 2016. With the addition of Tris Hydroxymethyl Aminomethane ($C\ H_{411}\ NO_3$), it was possible to increase the conductivity of the electrolyte, thereby reducing the dielectric strength between the poles and consequently increasing the density of discharges for the same value of voltage supplied, which in turn favors discharges resulting in greater porosity (BAYATI, MOSHFEGH and GOLESTANI-FARD, 2010).

The Atomic Force Microscopy images (Figure 6) show a variation in roughness and texture due to the ceramic deposition, which enables the surface to be wettable. It can be seen that with increasing treatment time there was greater roughness and increased homogeneity of the distribution of ceramic crystals on the surface.

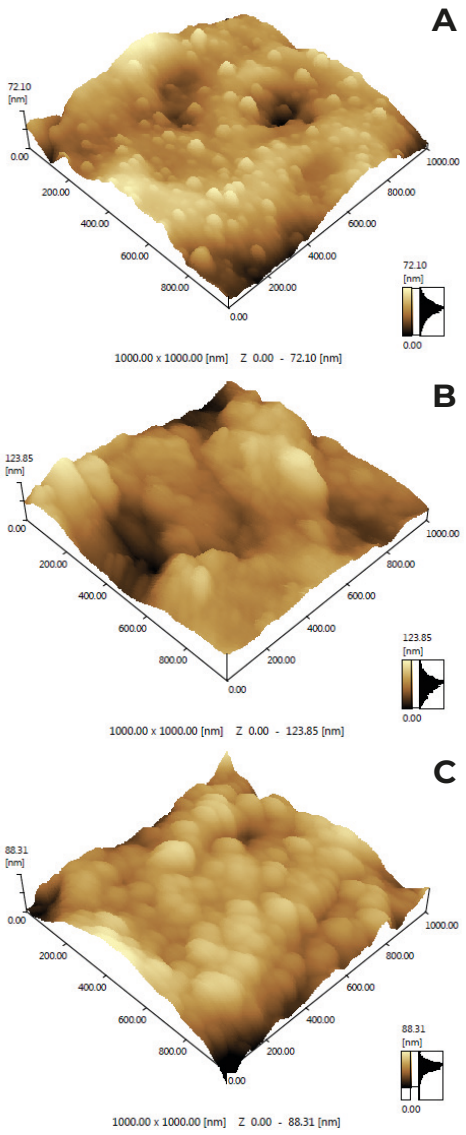

Figure 6 - Atomic Force Microscopy micrographs for: a - 1 minute of treatment; b - 8 minutes of treatment; c - 16 minutes of treatment. Author's own work (2017).

Bayati, Moshfegh and Golestani-Fard (2010) describe that as the electrical voltage increases in the treatment, a film is formed over the entire surface and simultaneously new layers develop in parallel, occupying more of the surface as the treatment time increases. Ceramic deposition (formation of the TiO_2 layer) and organic compounds occurred gradually with increasing time, but the discharges decreased after 8 minutes, due to the increase in the ceramic layer, which in turn is more insulating than the titanium rod. The AFM images show surfaces with rounded grains for this type of deposition, with a morphology conducive to the formation of pores.

The wettability tests showed a significant decrease in the wetting angle for the PEO-treated samples, a change that was more evident at the 8 and 16 minute treatment times (Table 4 and Figure 7).

Nº	Sample	Wetting angle (°)
1	No treatment	42
2	With 1 min of treatment	30
3	With 8 min of treatment	12
4	With 16 min of treatment	9

Table 4 - Wettability test for untreated sample and for 1, 8 and 16 minutes of PEO treatment. Author's own work (2017).

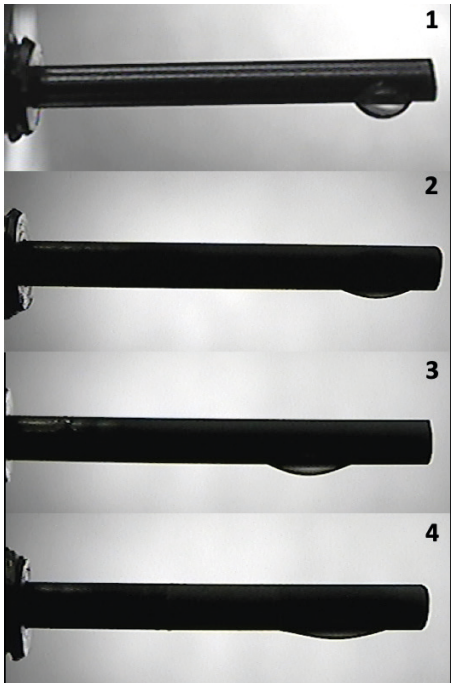

Figure 7 - Drop wettability analysis: (1) - Untreated; (2) - Treated for 1 min; (3) - Treated for 8 min; (4) - Treated for 16 min. Own authorship (2017).

According to researchers Gowtham, Arunnellaiappan and RameshbabuRameshbabu (2016) a hydrophilic surface is a necessary factor to show favorable bioactivity. The wettability tests showed very promising results in

terms of good wettability for the deposited ceramic surface. Compared to the literature, the closer the wetting angle is to 180 degrees, the more wettable the surface is, a necessary factor to show favorable bioactivity, and favoring the surface for osseointegration. It was observed that as the treatment time increased above 8 degrees, the greater the wetting angle, which can be explained by the large presence of porosity and roughness on the surface resulting from the PEO coating.

Wheeler et al, (2010) revealed that the electrolyte coating containing phosphate has a greater degree of porosity on its surface. In this way, all the conditions performed obtained results of contact angles greater than the reference sample (untreated). These results indicate that PEO coatings produce hydrophilic surfaces. This can be explained by the increase in porosity with increasing treatment time.

FINAL CONSIDERATIONS

Based on the above, it is fair to say that the Electrolytic Plasma Oxidation technique proved to be effective in depositing a ceramic layer on the surface of the titanium alloy. It was possible to control all the parameters in an ergonomic, practical and safe way and to monitor the variations in the oxidation process. Chemical analysis by X-ray fluorescence identified the presence of TiO_2 on the surface of the sample. As a complementary analysis, Energy Dispersive X-ray Spectroscopy showed the elements present in the solution and in the deposited layer.

Analysis by optical microscope and scanning electron microscope showed that all the samples were deposited at 1 minute with a thickness of approximately 11 μm. And for the times of 8 and 16 minutes, a thickness of approximately 21 μm was observed. The SEM images of the coatings show an interface suggestive of good adhesion with no voids. The Atomic Force Microscopy images showed that as the treatment time increased from 1 to 8 minutes, there was greater roughness and increased homogeneity in the distribution of ceramic crystals on the surface. The wettability tests showed a smaller wetting angle for the PEO-treated samples at 8 and 16 minutes.

We can conclude that the Electrolytic Plasma Oxidation technique proved to be effective in depositing a ceramic coating, improving the quality of the surface, reducing the cost of surface treatment and lowering the final value of the implant for implementation in the SUS.

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