Relationship Between the Surface Chemical Composition of Implants and Contact With the Substrate

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The purpose of the study was to use scanning electron microscopy and energy dispersive x-ray spectrometry to assess possible morphologic and chemical changes after performing double-insertion and pullout tests of implants of different shapes and surface treatments. Four different types of implants were used—cylindrical machined-surface implants, cylindrical double-surface–treated porous implants, cylindrical surface-treated porous implants, and tapered surface-treated porous implants—representing a total of 32 screws. The implants were inserted into synthetic bone femurs, totaling 8 samples, before performing each insertion with standardized torque. After each pullout the implants were analyzed by scanning electron microscopy and energy dispersive x-ray spectrometry using a universal testing machine and magnified 35 times. No structural changes were detected on morphological surface characterization, only substrate accumulation. As for composition, there were concentration differences in the titanium, oxygen, and carbon elements. Implants with surface acid treatment undergo greater superficial changes in chemical composition than machined implants, that is, the greater the contact area of the implant with the substrate, the greater the oxide layer change. In addition, prior manipulation can alter the chemical composition of implants, typically to a greater degree in surface-treated implants.

Key Words: dental implant, osseointegration, traction, scanning electron microscopy, topography

INTRODUCTION

During osseointegration, the implant surface reacts directly with body tissues; thus its characteristics significantly influence this process. 1–3 Currently, there are several types of implants, and they differ in shape, size, composition, and surface treatment. It is up to the dentist to choose the implant appropriate for each specific clinical case. 4–6 In addition, surface roughness, which is affected by different surface treatments, increases bone formation, osseointegration, and biomechanical fixation rates, 7–10 thereby promoting bone anchorage and positively influencing body tissue responses. 1,11,12

Mechanical testing of implants through insertion torque and pullout tests, 13,14 in addition to quantifying the anchorage of different implants tested, can also simulate surface and chemical changes. These changes can be evaluated through surface characterization by scanning electron microscopy (SEM) and by chemical analysis with energy-dispersive x-ray spectrometry (EDS) 15,16 assessments that are crucial to our understanding of tissue responses to osseointegration. 17

Implant surfaces can be machined with different types of treatments, roughnesses, and topographies, which can influence bone formation, thereby determining whether there is greater or lesser bone-implant contact and, consequently, whether the osseointegration rate is high or low. Thus, surface analysis can determine the best type of implant for each specific clinical case. 18

The surface characterization of titanium (Ti), besides being important for verification of possible morphologic and composition changes, is essential to assess the implant’s manufacturing process and prevent the introduction of residual particles that can cause deleterious effects on bone tissue formation due to the wide range of elements and chemicals that can appear during manufacture and storage 19—factors that can influence the implant’s primary stability. 20 Surface contamination by secondary hydrocarbon and exposure to air is not harmful to osseointegration. 21,22

To determine the actual influence of mechanical manipulation and its correlation with the shape and surface treatment of an implant for primary stability and osseointegration, we evaluated possible morphologic and chemical changes using SEM and EDS after performing double-insertion and traction tests on implants of different shapes and surface treatments.

MATERIALS AND METHODS

For the study, 32 implants and 8 artificial bone samples of polyurethane were used (Synbone, BaySystems, Malans, Swit-
The sample size was determined according to previously performed experiments. Polyurethane compounds have been suggested as bone substitutes after research comparing natural and synthetic bone by testing axial compression, bending, and twisting and by analyzing external geometry. Cristofolini et al. concluded that synthetic polyurethane bones flex and have geometry similar to that of cadaver bones; in addition, they allow for greater standardization of the study. Furthermore, polyurethane bones are used as a standard material for inserting implants in pullout testing according to the American Society for Testing and Materials (ASTM 1839-08).

Four different types of implants (Conexão, São Paulo, Brazil) were inserted into each sample: cylindrical machined-surface implants, 11.5 × 3.75 mm; cylindrical double-surface–treated porous implants (porous surface treatment consists of immersing the implant in etching solutions composed of nitric acid and sulfuric acid), 11.5 × 3.75 mm; cylindrical surface-treated porous implants, 11.5 × 3.75 mm; and tapered surface-treated porous implants, 11.5 × 3.5 mm.

Assays were performed by a single operator to standardize the study. The surgical insertions were performed with the sequence of bits specified by the manufacturer of an electric motor (MC 101, Omega; Dentscler, Ribeirão Preto, Brazil). Before each insertion, performed with standardized torque, the implants were subjected to physical (topography) and chemical (compositional) analysis by SEM and EDS, respectively. The SEM coupled with EDS (Carl Zeiss AG, EVO 50 Series, Cambridge, UK), operated in the high and low vacuum modes and magnified 35 times, detected differences between the implants.

The implants were removed from the samples with a pullout test performed in a universal testing machine (Emic, DL10000, São Paulo, Brazil) with 200-kg-F load and Tesc 3.13 software (Emic) to simulate the manipulation of the implants. For this test, the screw head was attached to the test machine by connectors that allowed for multidirectional movements and the application of axial load traction without applying torque. A preload of 5 N was applied for 10 seconds to accommodate the system, and then the axial load was applied at a constant pull of 0.2 mm/min until the implant was extracted. These variables were defined based on the need for the implant to adapt to the universal testing machine. After removal, each implant was again subjected to SEM and EDS.

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**FIGURES 1 AND 2.**

**FIGURE 1.** (a) Cylindrical machined-surface implants after the first extraction. (b) Cylindrical machined-surface implants after the second extraction.

**FIGURE 2.** (a) Cylindrical double-surface–treated porous implant after the first extraction. (b) Cylindrical double-surface–treated porous implant after the second extraction.
In the second step, the screws were reinserted, reextracted, and then analyzed again by SEM and EDS, so that we could verify the amount of organic matter retained in the screw threads and the influence of the amount of mechanical manipulation on the possible topographic and compositional changes of the implants tested.

**RESULTS**

**Surface characterization of implants**

Visual inspection of the implant images obtained by SEM analysis showed no surface changes in structure. In contrast, there was accumulated organic matter between the spirals after the first and second insertions, and this accumulation was quantitatively different for each type of implant.

When the machined implants (Figures 1a and b) were compared with those with double-surface treatment, there was greater accumulation in the latter (Figures 2a and b), especially after the second insertion.

**Chemical characterization of the implants**

An implant remains pure only under vacuum and conditions equivalent to those of a Ti surface, because when the implant comes into contact with air, it is instantly coated with an oxide layer, which may consist of a combination of TiO, TiO₂, and TiO₃, a stable coating that prevents direct contact between bone and metal but does not interfere with the osseointegration process.²⁵

After using EDS to evaluate the composition of the implants, we observed a concentration reduction in the Ti element in the first analysis (for the first mechanical test) and a concentration increase in the carbon (C) element. When comparing the implants with each other, we observed that the machined implants maintained the highest Ti concentration and the lowest C concentrations (Figures 3a and b), whereas implants with the double surface treatment showed the greatest reduction in Ti concentration and the highest C concentrations (Figures 4a and b) compared with the concentrations in the first test. Among the surface-treated implants, the double-treated implant had a lower quantity of Ti and a higher quantity of C, whereas the tapered implant retained more Ti and less C (Table).

**DISCUSSION**

Both Ti and Ti alloys are widely used in rehabilitation to replace body parts; they exhibit such favorable characteristics as fatigue strength, corrosion resistance, and biocompatibility.²⁶ To expand the indications and osseointegration performance of Ti, changes to its surface are made through chemical treatments to improve its quality for body tissue responses.²⁷

To evaluate the implants for surface stability after surface manipulation, we subjected them to insertion torque and pullout tests, simulating mechanical manipulation, to analyze the effect of this manipulation on possible chemical and topographic changes. The pullout test determines the force required to break the bone-metal link and the implant’s ability to withstand masticatory forces.²⁹ Studies have indicated that pullout strength is correlated with bone quality and the surface and diameter of the implant.³⁰ In this study, the density of the substrate was homogeneous, hence allowing for possible changes in the structural and morphologic differences of the implants.
The different surface treatments influence roughness, increasing the extent of the bone-implant interface.\textsuperscript{31,32} and the increase in bone volume in rough-surfaced Ti implants is due to the lower bone remodeling rate. Roughness is a factor that helps determine the balance between bone formation and resorption at the bone-implant interface.\textsuperscript{33}

According to SEM analysis, the different types of implants used in the study demonstrated surface changes due to the accumulation of organic material particles, but the spirals did not undergo any deformation after mechanical manipulation. The different implants retained a greater or lesser amount of organic matter, and the machined-surface implants had the lowest accumulation of particles and therefore a lower concentration of C, to the detriment of implants with that surface treatment, among which the double–surface-treated implants showed a higher accumulation of organic matter and higher concentrations of C.

The different surface treatments did not have a great influence on the implants’ chemical composition and native oxide surface layer, essentially showing little influence on surface roughness.\textsuperscript{33} However, in this study, changes in the chemical composition were observed, probably because of the mechanical manipulation.

The implants’ concentration showed a reduction in the Ti element from the first extraction; this was reduced even more after the second test, which suggests the need for greater care in the handling of implants to protect the oxide layer of the Ti surface, as Ti in high concentrations favors osseointegration.\textsuperscript{34} In contrast, the C element, which had previously appeared in small or nonexistent concentrations, greatly increased in concentration after the tests, probably because of the accumulation of organic material between the screw threads. However, according to the literature, surface contamination by secondary hydrocarbons due to air exposure, manufacturing, and storage is not detrimental to osseointegration.\textsuperscript{21}

The cylindrical machined-surface porous implant kept the highest percentage of Ti and the lowest percentage of C after mechanical manipulation. Among the surface-treated porous implants, the cylindrical one with double-surface treatment showed the greatest reduction of Ti and the highest concentrations of C. The tapered surface-treated porous implant had the lowest reduction of Ti and the lowest concentration of C. Several authors have reported that surface treatment does not change the chemical composition of the implant;\textsuperscript{32,33} however, it was shown that the surface can be changed with respect to Ti and C levels due to previous manipulation. Thus, although implants with surface treatment are confirmed to be greater inducers of osseointegration because of the presence of roughness, manipulation should be performed with care, as it can change the oxide layer formed by the surface treatment.

There is also the possibility of associating the design of the screw with the surface treatment. Within the group of surface-treated implants, the tapered implant, which has a slightly smaller diameter than the other implants, had a smaller reduction of Ti and a lower accumulation of C, indicating that the greater the contact area, the greater the change in the oxide layer.

### Conclusions

Implants with surface acid treatment undergo more superficial changes in chemical composition than do machined implants. The greater the contact area of the implant with the substrate, the greater the change in the oxide layer. Prior manipulation can also change the chemical composition of the implants, and to a greater degree in surface-treated implants.

### Abbreviations

- C: carbon
- EDS: energy dispersive x-ray spectrometry
- SEM: scanning electron microscopy
- Ti: titanium

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