Effect of sandblasting a titanium base abutment on the bonding stability and retention forces of hybrid abutment crowns after artificial aging

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RÉSUMÉ

Objectifs : évaluer l'influence des différents traitements de surface effectués sur les bases en titane (Ti-base) sur la stabilité de l'interface de collage et les forces de rétention dans le cadre de restaurations dentaires implanto-portées avec couronnes en disilicate de lithium après vieillissement thermomécanique.

Matériels et méthodes : 60 implants à connexion interne (Conelog, Camlog) ont été assemblés avec des couronnes en disilicate de lithium collées sur des Ti-bases (Conelog, Camlog). Les Ti-bases ont été divisées en 4 groupes (avec n = 15) en fonction du traitement de surface appliqué : [NoT] aucun traitement ; [30-SiO-AlO] sablage avec particules d'oxyde d'alumine 30 µm avec revêtement de silice (CoJet, 3M ESPE); [50-AlO] sablage avec particules d'oxyde d'alumine 50 µm (Cobra Aluoxyd, Renfert) et [110-SiO-AlO] sablage de particules d'oxyde d'alumine 110 µm avec revêtement de silice (Rocatec Plus, 3M ESPE).
Les surfaces des Ti-bases ont été sablées dans des conditions normalisées (10 secondes, 2,5 bars, 10 mm) en utilisant un appareil de sablage customisé. Toutes les couronnes ont été scellées avec un ciment de résine (Multilink Hybrid-Abutment, Ivoclar Vivadent AG). Après vieillissement (1'200'000 cycles, 49 N, 1,67 Hz, 5 ° C à 55 ° C, 120 s), tous les échantillons ont été observés et analysés au microscope optique (50x) afin de rechercher un échec au niveau de l'interface de collage. Les forces de rétention (N) ont été mesurées en pratiquant un test d'arrachement par traction verticale à 0,5 mm/min. Les modes de défaillance ont été classés selon 3 types (type 1, 2 ou 3). Une Ti-base supplémentaire de chaque groupe a été préparée pour mesurer les paramètres de rugosité de surface en µm (Ra, Rc, Rz) avec un profilomètre laser confocale sans contact. Des reconstructions 3D des balayages de surface et des images MEB représentatives ont été enregistrées (1000x). Des tests de chi-carré ont été réalisés pour analyser les échecs au niveau de

l’interface de collage et des modes de défaillance. Le test de Kruskal-Wallis a été utilisé pour évaluer les données de force de rétention. Enfin, un test ANOVA à un facteur a été utilisé pour analyser les valeurs moyennes des rugosités de surface ($\alpha = 0,05$).

Résultats : Les taux d'échec de liaison après vieillissement sont de 73,3% (NoT), 40% (30-SiO-AlO), 6,7% (50-AlO) et 40% (110-SiO-AlO). La stabilité de l'interface de collage est influencée par le traitement de surface appliqué ($p < 0,05$). Les valeurs moyennes de force de rétention varient de 206,3 ± 86,3 (NoT) à 420 ± 139,5 (50-AlO) et les différences entre ces deux groupes sont significatives ($p < 0,05$). Les modes de défaillance sont principalement de type 2 pour les groupes (30-SiO-AlO), (50-AlO), (110-SiO-AlO) et de type 3 pour (NoT). Les valeurs moyennes des rugosités de surface sont significativement différentes ($p > 0,001$) parmi les groupes pour tous les paramètres (Ra, Rc, Rz) ($\alpha = 0,05$).

Conclusion : Le traitement mécanique de la surface en titane a augmenté la stabilité de la liaison et les forces de rétention entre les Ti-bases et la couronne correspondante. L'utilisation d'une abrasion de particules en suspension dans l'air avec particules d’oxyde d’alumine 50 µm a fourni l'interface de liaison la plus stable parmi les différents traitements testés.

INTRODUCTION

Le choix de bases en titane (Ti-bases) ou d'inserts en titane en tant que pilier pour les reconstructions à base d'implants devient une option de plus en plus répandue dans le cadre des flux de travail numériques récemment introduits. Ces composants « préfabriqués » permettent un ajustement précis et une connexion implant-pilier mécaniquement stable de la même manière que les piliers conventionnels en titane (1-7).

En appliquant le concept de pilier hybride, consistant en un pilier intermédiaire en céramique ou en une couronne-pilier céramique (couronne hybride) collée à un Ti-base, il est possible de transformer un composant standardisé en une solution personnalisée (6, 8). Cette personnalisation fournie par la superstructure « tout céramique » permet un meilleur profil d'émergence et une meilleure adaptation des tissus mous. De plus, la partie métallique grisâtre de la reconstruction ne reste qu'en profondeur sur le niveau de l'implant. En effet, un meilleur résultat esthétique peut être obtenu par rapport aux piliers en titane personnalisés (9). En outre, un collage contrôlé est possible car le pilier intermédiaire ou la couronne-pilier est d'abord collé par voie extra-orale à la base en titane, puis seulement vissée comme un seul composant dans l'implant. De cette manière, le problème potentiel d'excès de ciment autour de la tête d'implant est évité, réduisant ainsi le risque de complications biologiques (10, 11).

Malgré tous les avantages du concept de pilier hybride, son succès dépend fortement de la stabilité de la liaison entre le Ti-base et les composants en céramique, couronne ou pilier. Une éventuelle instabilité de cette liaison pourra créer un micro-gap entre les respectives composants. Cependant, il n’est pas complètement prouvé que ce micro-gap prothétique proche dans la connexion implant-pilier peut entraîner une inflammation et, par conséquent, une perte osseuse autour de l'implant (12)

Afin d'obtenir une force de collage élevée et durable, plusieurs prétraitements de surface sont proposés. Malgré un protocole de liaison bien établi pour les vitrocéramiques (13), le meilleur traitement de surface mécanique à appliquer sur les bases en titane n'est pas encore clair (14). En outre, certains fabricants d'implant déconseillent explicitement le sablage des Ti-bases. Du sablage conventionnel avec des particules d'oxyde d'alumine aux systèmes utilisant des particules avec un revêtement de silice, de nombreux procédés ont été suggérés (15-20). Cependant, la plupart des études évaluant ces méthodes sont basées sur des tests de résistance au cisaillement ou à la traction en utilisant des échantillons à surface plane. Par la suite, la rétention donnée par les parois axiales du Ti-base, comme on le voit en milieu clinique, n'est pas prise en compte. Afin de prévoir le comportement des matériaux dans des conditions cliniques, des tests *in vitro* utilisant une simulation de mastication et des conceptions de prothèses cliniquement pertinentes sont nécessaires.

Ainsi, l'objectif de la présente étude est d'évaluer l'influence, de différents traitements de surface appliqués sur les Ti-bases, sur les forces de stabilité et de rétention entre ces bases et les couronnes monolithiques tout céramique après un vieillissement artificiel. Les hypothèses nulles ont été définies comme suit :

1) Le traitement de surface n'influence pas la stabilité de l'interface de collage entre le Ti-base et couronnes tout céramique ;
2) Le traitement de surface n'influence pas les forces de rétention entre les Ti-bases et couronnes tout céramique.

RÉFÉRENCES

1. Sailer I, Asgeirsson AG, Thoma DS, Fehmer V, Aspelund T, Ozcan M, et al. Fracture strength of zirconia implant abutments on narrow diameter implants with internal and external implant abutment connections: A study on the titanium resin base concept. Clin Oral Implants Res 2018;29:411-423.

2. Stimmelmayr M, Heib PE, Schweiger J, Beuer F. Fracture resistance of different implant abutments supporting all-ceramic single crowns after aging. International Journal of Computerized Dentistry 2017;20:53-64.

3. Elsayed A, Wille S, Al-Akhali M, Kern M. Effect of fatigue loading on the fracture strength and failure mode of lithium disilicate and zirconia implant abutments. Clin Oral Implants Res 2018;29:20-27.

4. Kaweewongprasert P, Phasuk K, Levon JA, Eckert GJ, Feitosa S, Valandro LF, et al. Fatigue Failure Load of Lithium Disilicate Restorations Cemented on a Chairside Titanium-Base. J Prosthodont 2018. Epub ahead of print.

5. Pitta J, Hicklin SP, Fehmer V, Boldt J, Gierthmuehlen PC, Sailer I. Mechanical stability of zirconia meso-abutments bonded to titanium bases restored with different monolithic all-ceramic crowns. Int J Oral Maxillofac Implants 2019. Epub ahead of print.

6. Nouh I, Kern M, Sabet AE, Aboelfadl AK, Hamdy AM, Chaar MS. Mechanical behavior of posterior all-ceramic hybrid-abutment-crowns versus hybrid-abutments with separate crowns-A laboratory study. Clin Oral Implants Res 2019;30:90-98.

7. Roberts EE, Bailey CW, Ashcraft-Olmscheid DL, Vandewalle KS. Fracture Resistance of Titanium-Based Lithium Disilicate and Zirconia Implant Restorations. J Prosthodont 2018. Epub ahead of print.

8. Kurbad AK, S. CAD/CAM-bases implant abutments made of lithium disilicate. Int J Comp Dent 2013;16:125-141.

9. Linkevicius T, Vaitelis J. The effect of zirconia or titanium as abutment material on soft peri-implant tissues: a systematic review and meta-analysis. Clin Oral Implants Res 2015;26(suppl):s139-s147.

10. Wilson TG. The positive relationship between excess cement and peri-implant disease: a prospective clinical endoscopic study. J Periodontol 2009;80:1388-1392.

11. Korsch M, Obst U, Walther W. Cement-associated peri-implantitis: a retrospective clinical observational study of fixed implant-supported restorations using a methacrylate cement. Clin Oral Implants Res 2014;25:797-802.

12. Passos SP, Gressler May L, Faria R, Ozcan M, Bottino MA. Implant-abutment gap versus microbial colonization: Clinical significance based on a literature review. J Biomed Mater Res B Appl Biomater 2013;101:1321-1328.

13. Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. J Prosthet Dent 2003;89:268-274.

14. Bankoglu Güngör M, Karakoca Nemli S. The Effect of Resin Cement Type and Thermomechanical Aging on the Retentive Strength of Custom Zirconia Abutments Bonded to Titanium Inserts. Int J Oral Maxillofac Implants 2018;33:523-529.

15. Almeida Junior AA, Fonseca RG, Haneda IG, Abi-Rached FO, Adabo GL. Effect of Surface Treatments on the Bond Strength of a Resin Cement to Commercially Pure Titanium. Braz Dent J 2010;21:111-116.

16. Abi-Rached FO, Fonseca RG, Haneda IG, de Almeida-Júnior AA, Adabo GL. The effect of different surface treatments on the shear bond strength of luting cements to titanium. The Journal of Prosthetic Dentistry 2012;108:370-376.

17. Fonseca RG, Haneda IG, Almeida-Junior AA, de Oliveira Abi-Rached F, Adabo GL. Efficacy of air-abrasion technique and additional surface treatment at titanium/resin cement interface. J Adhes Dent 2012;14:453-459.

18. Elsaka SE, Swain MV. Effect of surface treatments on the adhesion of self-adhesive resin cements to titanium. J Adhes Dent 2013;15:65-71.

19. von Maltzahn NF, Holstermann J, Kohorst P. Retention Forces between Titanium and Zirconia Components of Two-Part Implant Abutments with Different Techniques of Surface Modification. Clin Implant Dent Relat Res 2016;18:735-744.

20. Ozcan M, Raadschelders J, Vallittu P, Lassilla L. Effect of particle deposition parameters on silica coating of zirconia using a chairside air-abrasion device. J Adhes Dent 2013;15:211-214.

(Article en anglais)

Effect of sandblasting a titanium base abutment on the bonding stability and retention forces of hybrid-abutment crowns after artificial aging

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ABSTRACT

Purpose: to evaluate the influence of different surface treatments of the Ti-base surfaces on the bonding stability and retention forces of adhesively cemented lithium-disilicate crowns after thermo-mechanical aging.

Materials and Methods: 60 internal connection implants (Conelog, Camlog) were restored with lithium-disilicate crowns bonded to Ti-bases (Conelog, Camlog). The Ti-bases were divided in 4 groups (n=15) according to the surface treatment: (NoT) no treatment; (30-SiO-AlO) tribochemical silica-coating with 30-µm silica-modified Al_2O_3 particles (CoJet, 3M ESPE); (50-AlO) airborne-particle abrasion 50-µm Al_2O_3 (Cobra Aluoxyd, Renfert); and (110-SiO-AlO) tribochemical silica-coating with 110-µm silica-modified Al_2O_3 particles (Rocatec Plus, 3M ESPE). Ti-bases were surface-treated (10 sec, 2.5 bar, 10-mm) under standardized conditions in a custom-made sandblaster device. All crowns were cemented using a resin-cement (Multilink Hybrid-Abutment, Ivoclar Vivadent AG). After aging (1'200'000 cycles, 49 N, 1.67 Hz, 5°C-55°C, 120s), all samples were assessed for bonding interface failure under optical microscope (50x). The retention forces (N) were tested using a pull-off test (0.5mm/min). Modes of failure were classified (type 1, 2 or 3). An extra Ti-base of each group was prepared for surface roughness (µm) calculation (Ra, Rc, Rz) with a non-contact laser profilometer. 3D reconstructions of the surface scans and representative SEM images were recorded (1000x). Chi-squared tests were performed to analyze the bonding interface failure and modes of failure, and Kruskal-Wallis test was selected to evaluate retention force data. 1-way ANOVA was used to analyze the mean roughness values (α=.05).

Results: The percentages of bonding failure after aging were 73.3% (NoT), 40% (30-SiO-AlO), 6.7% (50-AlO), 40% (110-SiO-AlO). Bonding interface stability was influenced by the surface treatment applied ($p<.05$). Mean retention force values varied from 206.3±86.3 N (NoT) to

420±139.5 N (50-AlO), and the differences between these two groups were significant ($p<.05$). Modes of failure were predominantly type 2 (30-SiO-AlO; 50-AlO; 110-SiO-AlO) and type 3 (NoT). Surface roughness mean values were significantly different ($p>.001$) among the groups for all the parameters (Ra, Rc, Rz) ($\alpha=.05$).

Conclusion: Mechanical treatment of the titanium surface increased the bonding stability and retention forces between the Ti-base and the respective crown. The use of airborne-particle abrasion with 50-μm Al_2O_3 provided the most stable bonding interface among the different treatments.

INTRODUCTION

The use of titanium bases (Ti-bases) or titanium inserts as an abutment for implant-borne reconstructions is becoming an increasing popular option as part of the recently introduced digital workflows. These pre-fabricated components allow for a precise fit and mechanically stable implant-abutment connection similarly to the conventional titanium abutments (1-7).

Applying the hybrid abutment concept, that consists of an all-ceramic meso-abutment (hybrid abutment) or a full-contour all-ceramic crown (hybrid-abutment crown) bonded to a Ti-base, it is possible to transform a standardized component into a customized solution (6, 8). This customization provided by the all-ceramic superstructure allows for a better emergence profile and improved soft tissue adaptation. Moreover, the greyish metallic part of the reconstruction remains deeper than the soft tissue margin and close to the implant level. Indeed, a better esthetic outcome can be achieved compared to customized titanium abutments (9).

In addition, a controlled cementation is possible as the meso-abutment or the abutment-crown is first bonded extra-orally to the Ti-base, and only then, screw-retained in the implant as a complex. In this way, the potential problem of cement excess around the implant head is prevented, reducing the risk of biological complications (10, 11).

Notwithstanding all advantages of the hybrid abutment concept, its success is highly dependent on the bonding stability between the Ti-base and the ceramic components. In order to achieve a high and durable adhesive retention, several surface pre-treatments have been proposed. Despite the well-established bonding protocol to glass-ceramics (12), it is still not clear what is the best mechanical surface treatment to apply on the Ti- bases (13). Besides that, some implant manufacturers explicitly do not recommend the sandblasting of the Ti-bases. From the conventional airborne particle abrasion with aluminum oxide particles (Al_2O_3) to the tribochemical silica-coating systems, numerous sandblasting methods with different particles sizes have been suggested (14-19). However, most of the studies evaluating these methods are based in pure shear or tensile bond strength tests using flat surfaced samples. Subsequently the retention given by the Ti-base axial walls, as seen in a clinical setting, it is not taken into consideration. In order to predict the materials' behavior under clinical conditions, in vitro tests applying clinically relevant test settings, thermo-mechanical artificial aging and using clinically relevant prosthetic designs as test samples are needed.

The aim of the present study, therefore, was to evaluate the influence of different surface treatments of the Ti-bases surfaces on the stability and retention forces of adhesively cemented monolithic all-ceramic crowns after artificial aging. The null hypotheses were defined as follows: 1) the surface treatment does not influence the bonding interface stability between Ti-base abutments and the all-ceramic crowns; 2) the surface treatment does not influence the retention forces between Ti-base abutments and all-ceramic crowns.

MATERIALS AND METHODS

Sixty dental implants (Conelog, Camlog, Basel, Switzerland) with a diameter of 4.3 mm and 16 mm of length were used for this study. The implants were embedded into custom-made acrylic resin holders with a self-curing acrylic (Technovit 4071, Haraeus Kulzer, Hanau, Germany) following the ISO-Norm 14801 (20). A vertical distance of 3 mm was left from the implant shoulder to the top of the acrylic holder in order to simulate vertical bone loss (20).

To restore these implants, 60 commercially available Ti-bases (Conelog Titanium base CAD/CAM, Camlog) with 2.0 mm of gingival height were selected. The Ti-bases were composed of Ti-6Al-4V and their dimension are illustrated in Fig. 1. The Ti-bases were assigned to one of 4 groups (n=15), according to the mechanical surface treatment received (Table 1). The sample size was calculated based on a previous publication with similar methodology (21).

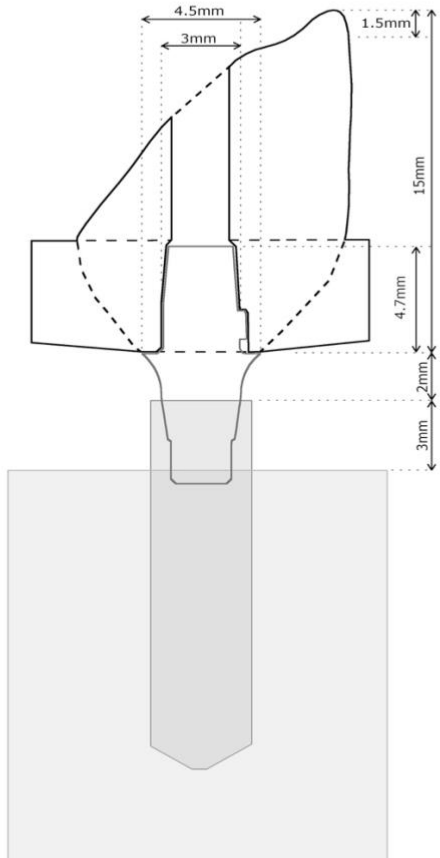

Figure 1. Illustration of the study sample with respective dimensions (mm)

Table 1. Group identification and respective surface treatment applied.

Group	Surface treatment	Commercial name
NoT	no surface treatment	---
30-SiO-AlO	tribochemical silica-coating with 30-µm SiO_2 - Al_2O_3 particles	CoJet powder, 3M ESPE
50-AlO	airborne-particle abrasion 50-µm Al_2O_3 particles	Cobra Aluoxyd, Renfert
110-SiO-AlO	tribochemical silica-coating with 110-µm SiO_2 - Al_2O_3 particles	Rocatec Plus powder, 3M ESPE

In order to achieve a uniform and replicable surface treatment of each Ti-base, a custom-made sandblasting-device was developed (Fig. 2). In one extremity, the sandblaster (Renfert, Hilzingen, Germany) was fixed allowing for only two positions in the horizontal plane (5 mm and 10 mm-distance), and in the opposite extremity the titanium base was fixed and rotated with the speed of 12 rpm. While the Ti-base was rotating, the sandblaster was turned on for 10 sec at each distance of the titanium base to apply the respective surface treatment with a pressure of 2.5 bar in a 45º angulation. Then, all Ti-bases were ultrasonically cleaned in an alcohol bath for 4 min (Micro 10+, Unident, Geneva, Switzerland), and then kept dry for cementation.

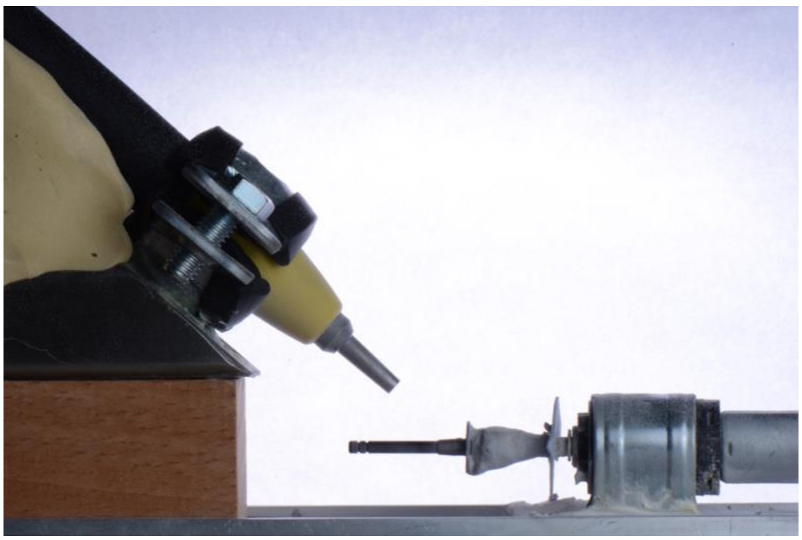

Figure 2. Custom-made sandblasting device.

From a master model used in a previous study (5), a maxillary central incisor implant abutment-crown was designed using a dedicated software (CEREC inLab 16.1, Dentsply Sirona, York, PA, USA). The design was adapted to allow for the pull-off set-up (Figure 1) by means of a circumferential thickness of material below the cingulum area. The abutment-crowns were milled (CEREC MC XL, Dentsply Sirona) out of lithium-disilicate ingots (IPS e.max CAD Cerec/inLab A16(S) – LT A2, Ivoclar Vivadent AG, Schaan, Liechtenstein) with an industrially prepared chimney to fit on the Ti-base. After surface polishing, the lithium-disilicate abutment-crowns were crystalized and finally glazed. Then, their internal surface was conditioned with hydrofluoric acid (HF 5%) for 20 sec (IPS Ceramic etching gel, Ivoclar Vivadent AG), ultrasonically cleaned in alcohol for 4 min, and conditioned with a universal primer agent for 60 sec (Monobond Plus, Ivoclar Vivadent AG). A resin-based cement (Multilink Hybrid Abutment HO-0, Ivoclar Vivadent AG) was used to adhesively bond the lithium-disilicate abutment-crowns to the Ti-bases following the manufacturer recommendations. This procedure was conducted always by the same operator and applying a constant pressure. While the cement was curing, a glycerin gel was applied on the margins for 7 min, and then rinsed. After cleaning all the cement excess, the one-piece abutment crowns were screw-retained onto the implant with the recommended torque (20Ncm) given by the manufacturer. The screw access holes were closed with a polytetrafluoroethylene (PTFE) tape (Teflon, Chemours, Wilmington, DE, USA) and a resin-based composite filling material (Tetric EvoCeram A3, Ivoclar Vivadent AG).

The samples were subjected to artificial aging by means of thermal cycling (5° to 55° C, dwelling time 120 s) and chewing simulation (1,200,000 cycles, 49 N, 1.67 Hz, CS-4.8 SD Mechatronik, Feldkirchen-Westerham, Germany). A steatite ball (diameter 6 mm), with similar hardness to enamel, was used as an antagonist indenter. The specimens were loaded 2 mm below the incisal edge at a 30° angle of the indenter to the palatal surface of the abutment-crowns. The vertical indenter movement for each chewing act was 2 mm. After aging, the samples were analyzed by 2 independent observers under 50x magnification (Olympus SZX9, Olympus, Tokyo, Japan) to asses bonding interface stability. Bonding failure was registered if a movement between the Ti-base and the crown was observed.

Retention forces (N) were measured using a pull-off test in a Universal Testing Machine (Shimadzu AGS-X series, Shimadzu, Kyoto, Japan) with a 10-kN calibrated load cell at a crosshead speed of 0.5mm/min. To ensure even force distribution between the crown and the holder, a 0.5mm thick tin foil (Dentaurum, Ispringen, Germany) was used. For each sample, the maximum retention force (N) was recorded using a dedicated software (TRAPEZIUM X, V.1.4.4, Shimadzu). After the pull-off test, the surfaces of the Ti-bases and the crowns intaglio were inspected under 20x magnification (Olympus SZX9) for remaining cement qualitative analysis and failure mode classification. Failure modes were classified in type 1 - cement remained predominantly on the abutment surface (> 90%), type 2 - cement remained on both abutment and crown surfaces (between 10-90%), type 3 - cement remained predominantly on the crown (> 90%) (22). Representative de-bonded specimens of each group were collected for surface observation using a scanning electron microscope (SEM) (Zeiss Sigma 300 VP, Carl Zeiss Microscopy GmbH, Jena, Germany).

An extra Ti-base was simultaneously prepared with the corresponding surface treatment of each group. The surface roughness (µm) was calculated using a high-resolution white light non-contact laser profilometer (CyberSCAN CT 100, Cyber technologies, Eching-Dietersheim, Germany) with a high-end confocal P-CHR-600 sensor achieving a z-resolution of 20 nm and a spot size of 4.0 µm. The treated surfaces were scanned for each sample with a controlled lateral displacement step of 1.0 µm. Depending on the sample size, areas ranging between [2800 - 3000 µm] x [900 - 1200 µm] were scanned to extract the average roughness (Ra), mean height of the profile (Rc) and maximum height of the profile (Rz). Gaussian profile filter with the cut-off wavelength (λc) was selected in concordance with the ISO-Norms 4288 and 11562 (23, 24). To calculate the surface roughness parameters, for each of the sample, 9 roughness profiles of 2000 µm length were extracted from the analyzed area. 3D reconstructions of the surface scans were registered (Surfer 12, Golden Software, Colorado, USA). The same samples were posteriorly prepared for SEM evaluation, and for each sample, representative images were recorded at 1000x magnification.

Statistical analysis was performed using SPSS statistical software released version 25.0 (IBM Corporation, Armonk, NY, USA) with a significance value set at α=.05. Pearson's chi-squared tests were performed to analyze the bonding failure types among the groups after artificial aging. The data on the retention forces were tested using a non-parametric test Kruskal-Wallis followed by the Dunn-Bonferroni adjusted post-hoc test as normality was not confirmed according to a Shapiro-Wilk test. Mean roughness values (Ra, Rc, Rz) were normally distributed and submitted to a 1-way ANOVA, followed by Tukey HSD post-hoc tests. Failure modes after pull-off testing were analyzed descriptively.

RESULTS

The percentages of bonding failure after aging for each group were 73.3% (NoT), 40% (30-SiO-AlO), 6.7% (50-AlO), 40% (110-SiO-AlO) (table 2). Bonding failure was influenced by the surface treatment applied (x^2 =13.889, df=3, $p<.05$). The means and standard deviations of the retention force values (N) were calculated for each group (table 3). The application of a surface treatment had a significant effect on retention force values (N) ($p<.05$) (table 3).

Table 2. Bonding failures detected under optical microscopy (x 50) after chewing simulation. (x^2 =13.889, df=3, $p< .05$).

Group	No detectable debonding (n)	Debonding (n)	debonding (%)
NoT	4	11	73.3 %
30-SiO-AlO	9	6	40 %
50-AlO	14	1	6.7 %
110-SiO-AlO	9	6	40 %

Table 3. Means and standard deviations of the retention force values (N). Different letters represent a statistical difference between groups ($p < .05$).

Group	Retention forces (N) mean ± SD
NoT	206.3 ± 86.3 (b)
30-SiO-AlO	346.9 ± 273.0 (a, b)
50-AlO	420.0 ± 139.5 (a)
110-SiO-AlO	376.1 ± 295.1 (a, b)

Failure mode was type 1 for all samples in group NoT, and predominantly type 2 for the remaining groups (30-SiO-AlO, 50-AlO, 110-SiO-AlO). These differences were significant different ($p<.001$) (table 4). Fig. 3 shows the SEM images of the Ti-bases showing representative failures for each group.

Table 4. Failure modes after pull-off testing. (x^2 =55.227, df=3, $p< .001$).

Group	Type 1	Type 2	Type 3
NoT	0	0	15
30-SiO-AlO	0	15	0
50-AlO	0	14	1
110-SiO-AlO	0	15	0

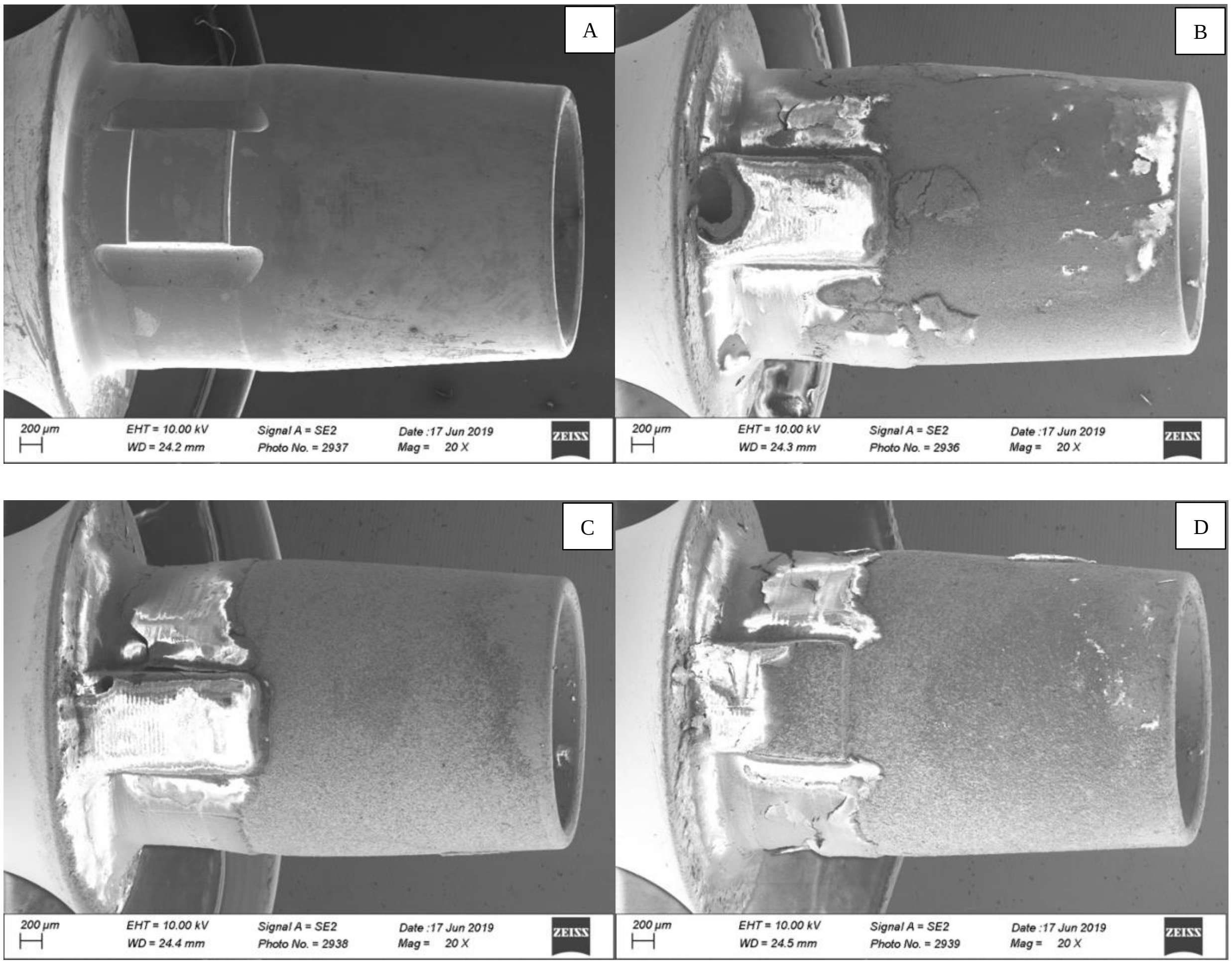

Fig 3. SEM photographs of representative Ti-base after the pull-off test showing the remaining cement on the surface (20x) (representative samples). A, NoT. B, 30-SiO-AlO. C, 50-AlO. D, 110-SiO-AlO.

The roughness values are described in table 5. The surface treatments had a significant effect on all the roughness parameters (Ra, Rc, Rz) ($p<.001$). Differences in roughness parameters were significant among all groups ($p< .001$). Fig. 4 shows 3D reconstructions images of the profilometer surface scans. SEM images of the respective Ti-base for each surface treatment are illustrated in Fig. 5.

Table 5. Roughness values. (all the values revealed significance differences ($p < .05$))

Groups and Roughness amplitudes parameters		Average ± SD (µm)
NoT	Ra	0.17 ± 0.01
	Rc	0.76 ± 0.05
	Rz	1.29 ± 0.11
30-SiO-AlO	Ra	0.45 ± 0.02
	Rc	1.90 ± 0.09
	Rz	3.42 ± 0.22
50-AlO	Ra	1.02 ± 0.06
	Rc	4.12 ± 0.30
	Rz	7.14 ± 0.42
110-SiO-AlO	Ra	0.79 ± 0.04
	Rc	3.36 ± 0.22
	Rz	6.21 ± 0.51

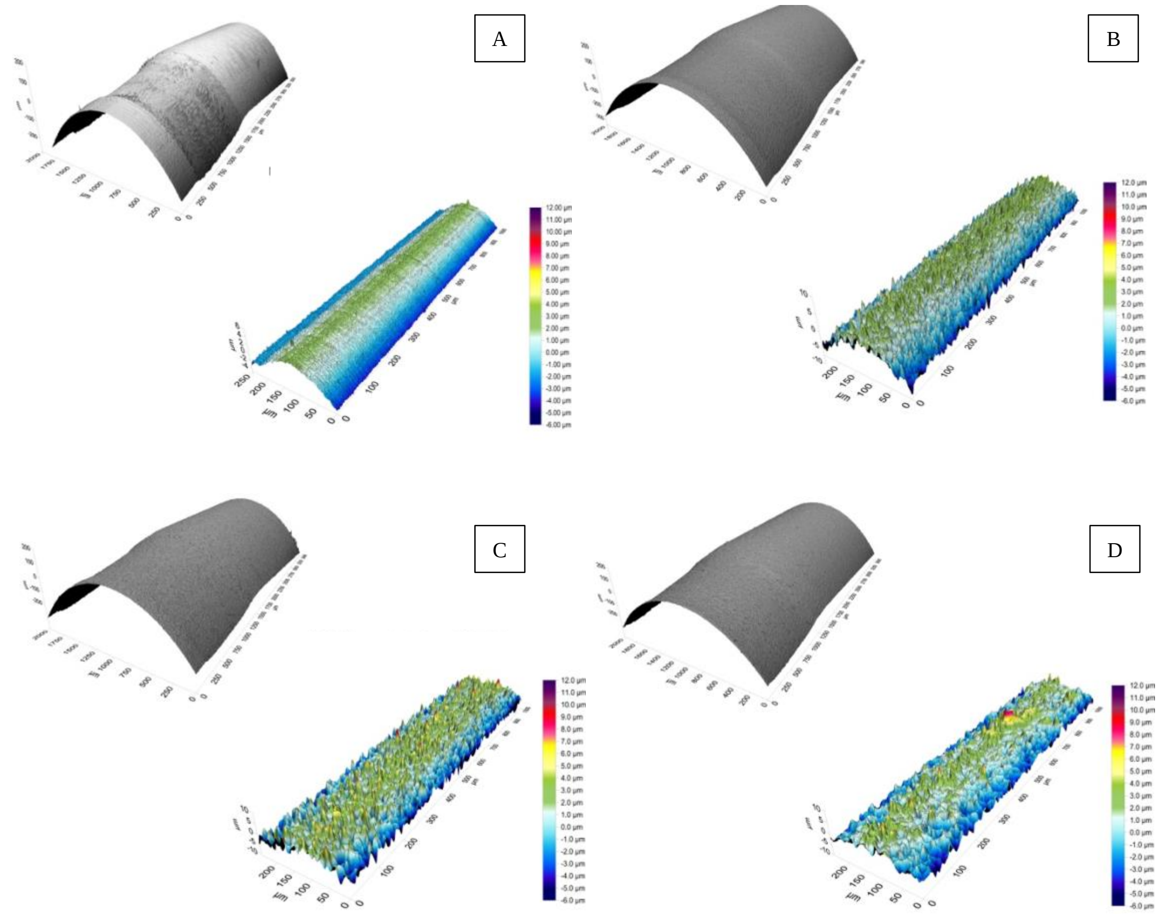

Fig 4. 3D reconstructions images of the profilometer surface scans. A, NoT. B, 30-SiO-AlO. C, 50-AlO. D, 110-SiO-AlO.

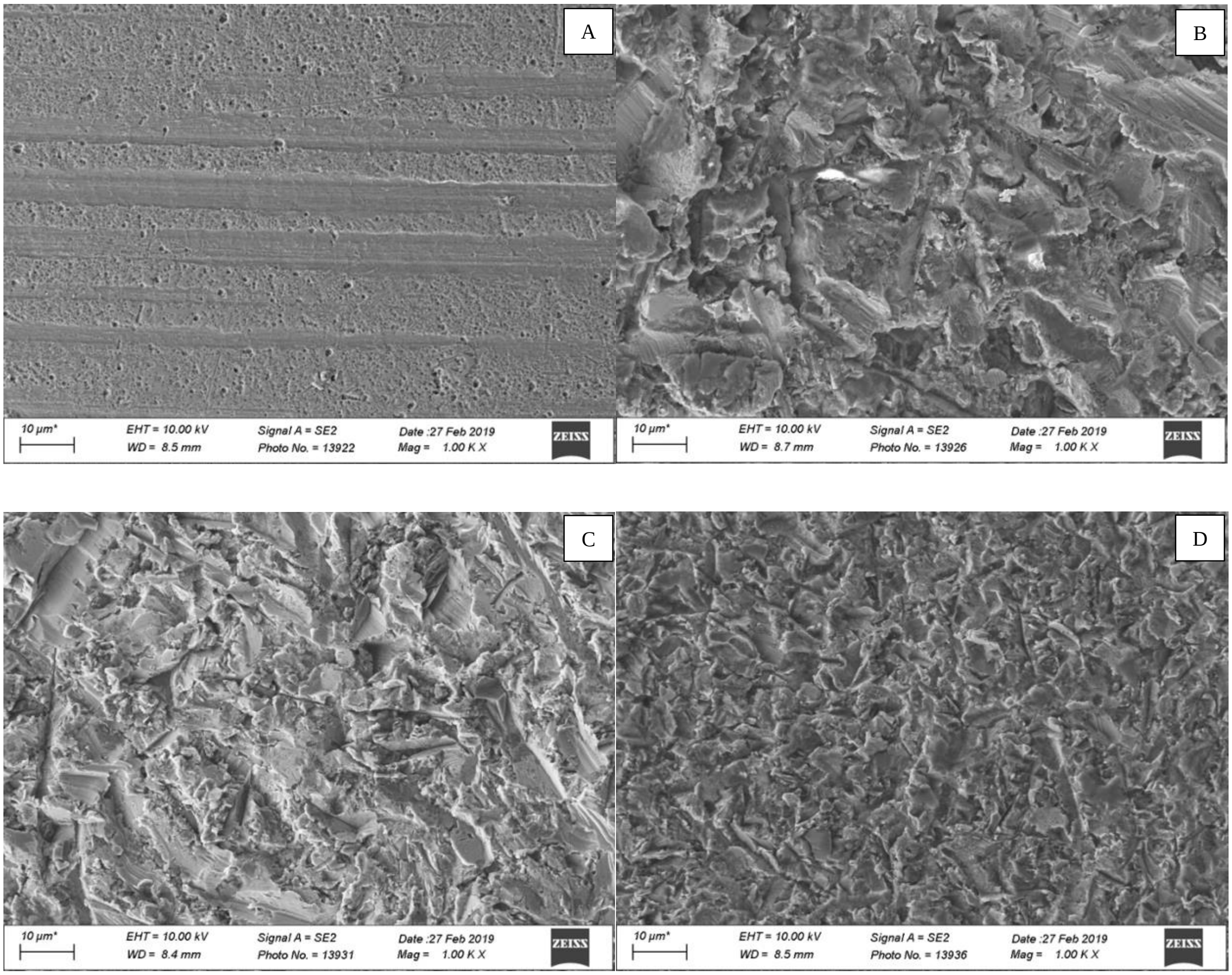

Fig 5. SEM photographs of the extra Ti-bases surface after each surface treatment (1000x). A, NoT. B, 30-SiO-AlO. C, 50-AlO. D, 110-SiO-AlO.

DISCUSSION

The application of a surface treatment on the Ti-bases influenced the bonding interface stability and retention forces between Ti-base abutments and all-ceramic crowns. Therefore, the two null hypotheses were rejected. More specifically, sandblasting with 50-µm Al_2O_3 particles resulted in higher bonding interface stability when compared to other surface treatments. This surface treatment also improved the retention values when compared to the use of no treatment.

In this study, each of the three sandblasting systems applied to the Ti-bases demonstrated to have a positive effect on the bonding interface stability of the hybrid abutment-crowns. Regardless the application of a universal primer agent and a resin-based cement, the samples that were not mechanically surface-treated revealed a 73.3 % of bonding failure after aging. In contrast, only 6.7 % failed when airborne-particle abrasion of 50-µm Al_2O_3 was applied. The latter treatment has been constantly used by multiple authors (3-7, 13) for treating titanium base surfaces in order to increase the bonding potential. However, only two studies applied this surface treatment on Ti-bases supporting lithium-disilicate abutment-crowns and submitted to chewing simulation (3, 6). While one did not report any de-bonding event (3), the other revealed de-bondings in 12.5% of the hybrid abutment-crowns after the aging process (6). Surprisingly, no other study with similar testing set-up including chewing simulation reported debonding events. One possible explanation could be that most of the studies tested Ti-bases supporting ceramic meso-abutments (hybrid abutment) with cemented crowns on top. It could be hypothesized that the presence of a second bonding interface might allow for a better force distribution, and consequently, less stress to the relatively small interface Ti-base / abutment-crown. Another possible reason might be related to limited reporting on the evaluation of microscopic bonding gap after chewing simulation. In the present study, a meticulous analysis of interface was performed allowing the detection of micromovements between the crowns and the Ti-base which might not be visible by the naked eye.

Although the crowns still remained attached to the Ti-bases due to a given physical retention and friction, these micromovements represent the initial failure of the bonding interface. Whether or not this should be considered a debonding, the consequences of the presence of a micro-movement for the peri-implant soft-tissues remain uncertain (25).

The retention force results of this investigation indicated the positive effect of the application of a mechanical surface treatment to the surfaces of Ti-bases, namely 50-µm Al_2O_3 particles. These findings are in agreement with other studies, that demonstrated a positive effect of sandblasting before application of a silane or adhesive primer on the bond strength to titanium using shear or tensile testing protocols (14-17, 26). Albeit, two recent studies revealed a negative effect of sandblasting micro-grooved Ti-bases on the retention forces of zirconia crowns (21, 27). One justification suggested by the respective authors was the elimination of the original micro-retentive grooves as an effect of sandblasting (21, 27). In the present study, the untreated Ti-bases presented originally a smooth surface with only one micro-retentive area designed as an anti-rotational feature. This smooth surface topography explains the benefit of sandblasting, by increasing the surface roughness and consequently the contact area, which contributes to improved retention values (14-16).

The use of silica-coated particles has also been demonstrated to ameliorate the bonding potential, due to a chemical reaction between the silica layer and the silane (15, 28). Nonetheless, in the present study, no significant effect of the type of particles on the retention forces could be detected. It can be hypothesized that the retentive form conceded by the geometry of a Ti-base abutment have a more relevant effect than the chemistry of the particles applied (silica-coated or not).

Other possible reason might be linked to the aging protocol applied. Under non-aged or uniquely thermo-aged conditions the tribochemical silica coating have demonstrated improvements on the bond strength comparing to just alumina-particle abrasion (14, 16, 29). However, in the present study, the application of a thermo-mechanical stress may have negatively affected the chemical liaison provided by the silica-silane bonding. It may be assumed that in the tested conditions, the roughness of the titanium surface was the most crucial factor for bonding stability and improved retention forces. This assumption was supported by the roughness measurements under a non-contact laser profilometer, that shown a higher surface roughness on the group using 50-µm Al_2O_3 airborne-particle abrasion. Previous authors have also detected, under SEM analysis, rougher morphology for 50-µm Al_2O_3 particles than for 30-µm or 110-µm SiO_2 - Al_2O_3 particles (29). These findings explain why 50-µm Al_2O_3 particles can promote higher surface roughness than bigger silica-coated particles, when applied with the same pressure.

Although no statistical differences were found between the retention forces of tribochemical silica coating with silica-modified Al_2O_3 particles (30-µm or 110-µm) and no surface treatment, both mechanical treatments still demonstrate a trend to improved retention values. The lack of significance might be related to the considerable amount of bonding failures in the silica-modified Al_2O_3 particles groups (30-SiO-AlO and 110-SiO-AlO), that resulted in very low retention values for some samples and contributed for a high standard deviation of these values.

Failure modes after the pull-off test revealed predominantly a mixed distribution of the cement between abutment and crown surfaces (type 2) for the air-abraded samples. In contrast, in the non-treated titanium bases (NoT), a predominantly adhesive pattern of failure between the titanium surface and the cement was identified, with most of the cement remaining on the intaglio of the crowns (type 3). These observations clearly support the benefit of applying a surface treatment in order to improve the adhesion to titanium surfaces.

One limitation of the current investigation is that the sandblasting was applied in a 45º angulation in order to allow standardization. Although it has been shown that an airborne-particle abrasion with silica-coated particles at 45º angulation results in a more effective silica deposition (19), the manufactures still recommend an application perpendicular to the surface. Future research should focus on clarifying the effect of sandblasting angulation and also to assess the influence of using titanium bases with different geometries.

CONCLUSION

Mechanical treatment of the titanium surface increased the bonding stability and retention forces between the titanium bases abutments and the respective crown. The use of airborne-particle abrasion with 50-µm Al_2O_3 provided the most stable bonding interface among the different tested treatments.

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REFERENCES

1. Sailer I, Asgeirsson AG, Thoma DS, Fehmer V, Aspelund T, Ozcan M, et al. Fracture strength of zirconia implant abutments on narrow diameter implants with internal and external implant abutment connections: A study on the titanium resin base concept. Clin Oral Implants Res 2018;29:411-423.

2. Stimmelmayr M, Heib PE, Schweiger J, Beuer F. Fracture resistance of different implant abutments supporting all-ceramic single crowns after aging. International Journal of Computerized Dentistry 2017;20:53-64.

3. Elsayed A, Wille S, Al-Akhali M, Kern M. Effect of fatigue loading on the fracture strength and failure mode of lithium disilicate and zirconia implant abutments. Clin Oral Implants Res 2018;29:20-27.

4. Kaweewongprasert P, Phasuk K, Levon JA, Eckert GJ, Feitosa S, Valandro LF, et al. Fatigue Failure Load of Lithium Disilicate Restorations Cemented on a Chairside Titanium-Base. J Prosthodont 2018. Epub ahead of print.

5. Pitta J, Hicklin SP, Fehmer V, Boldt J, Gierthmuehlen PC, Sailer I. Mechanical stability of zirconia meso-abutments bonded to titanium bases restored with different monolithic all-ceramic crowns. Int J Oral Maxillofac Implants 2019. Epub ahead of print.

6. Nouh I, Kern M, Sabet AE, Aboelfadl AK, Hamdy AM, Chaar MS. Mechanical behavior of posterior all-ceramic hybrid-abutment-crowns versus hybrid-abutments with separate crowns-A laboratory study. Clin Oral Implants Res 2019;30:90-98.

7. Roberts EE, Bailey CW, Ashcraft-Olmscheid DL, Vandewalle KS. Fracture Resistance of Titanium-Based Lithium Disilicate and Zirconia Implant Restorations. J Prosthodont 2018. Epub ahead of print.

8. Kurbad AK, S. CAD/CAM-bases implant abutments made of lithium disilicate. Int J Comp Dent 2013;16:125-141.

9. Linkevicius T, Vaitelis J. The effect of zirconia or titanium as abutment material on soft peri-implant tissues: a systematic review and meta-analysis. Clin Oral Implants Res 2015;26(suppl):s139-s147.

10. Wilson TG. The positive relationship between excess cement and peri-implant disease: a prospective clinical endoscopic study. J Periodontol 2009;80:1388-1392.

11. Korsch M, Obst U, Walther W. Cement-associated peri-implantitis: a retrospective clinical observational study of fixed implant-supported restorations using a methacrylate cement. Clin Oral Implants Res 2014;25:797-802.

12. Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. J Prosthet Dent 2003;89:268-274.

13. Bankoglu Güngör M, Karakoca Nemli S. The Effect of Resin Cement Type and Thermomechanical Aging on the Retentive Strength of Custom Zirconia Abutments Bonded to Titanium Inserts. Int J Oral Maxillofac Implants 2018;33:523-529.

14. Almeida Junior AA, Fonseca RG, Haneda IG, Abi-Rached FO, Adabo GL. Effect of Surface Treatments on the Bond Strength of a Resin Cement to Commercially Pure Titanium. Braz Dent J 2010;21:111-116.

15. Abi-Rached FO, Fonseca RG, Haneda IG, de Almeida-Júnior AA, Adabo GL. The effect of different surface treatments on the shear bond strength of luting cements to titanium. The Journal of Prosthetic Dentistry 2012;108:370-376.

16. Fonseca RG, Haneda IG, Almeida-Junior AA, de Oliveira Abi-Rached F, Adabo GL. Efficacy of air-abrasion technique and additional surface treatment at titanium/resin cement interface. J Adhes Dent 2012;14:453-459.

17. Elsaka SE, Swain MV. Effect of surface treatments on the adhesion of self-adhesive resin cements to titanium. J Adhes Dent 2013;15:65-71.

18. von Maltzahn NF, Holstermann J, Kohorst P. Retention Forces between Titanium and Zirconia Components of Two-Part Implant Abutments with Different Techniques of Surface Modification. Clin Implant Dent Relat Res 2016;18:735-744.

19. Ozcan M, Raadschelders J, Vallittu P, Lassilla L. Effect of particle deposition parameters on silica coating of zirconia using a chairside air-abrasion device. J Adhes Dent 2013;15:211-214.

20. Internation Organization for Standardization. ISO 14801. Dynamic loading test for endosseous dental implants. Geneva, Switzerland: ISO; 2016.

21. Arce C, Lawson NC, Liu PR, Lin CP, Givan DA. Retentive Force of Zirconia Implant Crowns on Titanium Bases Following Different Surface Treatments. Int J Oral Maxillofac Implants 2018;33:530-535.

22. Palacios RP, Johnson GH, Phillips KM, Raigrodski AJ. Retention of zirconium oxide ceramic crowns with three types of cement. J Prosthet Dent 2006;96:104-114.

23. Internation Organization for Standardization. ISO 4288. Geometrical product specifications (gPS) – Surface texture: Profile method – Rules and procedures for the assessment of surface texture. Geneva, Switzerland: ISO; 1998.

24. Internation Organization for Standardization. ISO 11562. Geometrical Product Specifications (GPS) -- Surface texture: Profile method -- Metrological characteristics of phase correct filters. Geneva, Switzerland: ISO; 1996.

25. Passos SP, Gressler May L, Faria R, Ozcan M, Bottino MA.. J Biomed Mater Res B Appl Biomater 2013;101:1321-1328.

26. Kern M, Thompson VP. Durability of resin bonds to pure titanium. J Prosthodont 1995;4:16-22.

27. Linkevicius T, Caplikas A, Dumbryte I, Linkeviciene L, Svediene O. Retention of zirconia copings over smooth and airborne-particle-abraded titanium bases with different resin cements. J Prosthet Dent 2019;121:949-954.

28. Matinlinna JP, Lassila LV, Vallittu PK. Evaluation of five dental silanes on bonding a luting cement onto silica-coated titanium. J Dent 2006;34:721-726.

29. Ozcan M, Pekkan G, Khan A. Does rinsing following particle deposition methods have a negative effect on adhesion to titanium? J Adhes Dent 2013;15:307-310.

n methods have a negative effect on adhesion to titanium? J Adhes Dent. 2013;15(4):307-10.

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