EFFECT OF DIFFERENT SURFACE TREATMENTS ON THE BONDING STRENGTH OF ADDITIVELY MANUFACTURED INTERIM CROWNS ON TITANIUM BASE IMPLANT ABUTMENTS

A Thesis

by

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ABSTRACT

Statement of problem. Additively manufacturing (AM) is gaining popularity in dentistry, including the use for interim implant supported fixed dental prostheses. However, evidence regarding the bonding protocols of AM interim crowns on ti-base abutments is lacking.

Purpose. The purpose of the present in vitro study was to compare the effect of different surface treatments and bonding protocols on the retentive bonding strengths of implant-supported AM interim crowns on Ti-bases.

Material- and methods. A total of 50 AM fabricated interim crowns were cemented on Ti-bases. Five subgroups (n=10) were established testing different surface pre-treatments including Group C = no surface pre-treatment, Group AP = crown air-abraded with 50μm Al_2O_3, Group AMP = crown air-abraded with 50μm Al_2O_3 and silanized, Group MP = crown silanized, and Group CMP = crown air-abraded with 30μm silica-coated Al_2O_3 (CoJet) and silanized. The specimens were cemented using a resin cement and stored in distilled water for 24 hours. Following, the specimens underwent retention testing with a Universal Instron machine at 2mm/minute crosshead speed. Pull-out forces (N) and modes of failure were registered. Statistical analysis was performed using Mann-Whitney U tests with Bonferroni corrections for multiple tests (a=.05).

Results. The median retention force values were 233.27 ±79.28 N for Group Control, 398.59 ±68.59 N for Group MP, 303.21 ±116.80 N for Group AMP, 349.31 ±167.73 N for Group CMP, and 219.85 ± 55.88 N for Group AP. The pull-off forces were significantly greater for Group MP, while the differences between the remaining groups were not significant. (P<.05). Group AP showed the lowest retention values.

Conclusions. The surface pre-treatment of the intaglio AM crown significantly influenced the

bonding strength on Ti-bases. Pretreatment with an MDP-containing silane significantly improved the bonding strength, whereas pretreatment with 50μm Al2O3 air-abrasion alone is not recommended prior to cementation on a Ti-base abutment.

DEDICATION

To my parents, Gabriel and Kalliopi for their constant love and support.

To all my mentors who provide me with the motivation throughout the years of my education.

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CONTRIBUTORS AND FUNDING SOURCES

Contributors

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TABLE OF CONTENTS

Page

LIST OF FIGURES

Page

LIST OF TABLES

Page

1. INTRODUCTION AND LITERATURE REVIEW

Before any definitive prostheses, an interim phase with interim restorations is of utmost importance. Interim restorations serve as a tool to enhance soft tissue health, protect pulpal tissues, maintain space for the final restoration, and shape an optimal emergence profile. They provide esthetics, comfort, and function to the patient, serve as a prototype for the definitive restoration and a communication tool between dentists, patients, and laboratory technicians.[1,2]

Throughout the years conventional materials have been used for interim restorations, including bis-acryl composite resin and polymethyl methacrylate (PMMA) materials. The main disadvantages of these materials are dimensional change and porosity, as well as time-consuming and technique sensitive laboratory or chairside fabrication processes.[1]

Recent advancements in digital dental technology, however, have provided manufacturing alternatives that are able to compensate for some of these shortcomings. Nowadays, materials are available for CAD-CAM fabrication of interim restorations either by subtractive or additive manufacturing.[1]

Additive manufacturing technologies introduce a new manufacturing methodology to process polymer materials and present several advantages compared to conventional or subtractive manufacturing methods. These advantages include reduction in the material waste and manufacturing time, ability to incorporate fine details and complex morphology in a design, reduced cost, fabrication of larger objects and ability to print multiple objects at one time.[3,4] According to the ISO (ISO 17296-2:2015) there seven AM technologies: vat-polymerization, material jetting, material extrusion, binder jetting, powder bed fusion, sheet lamination and directed energy deposition.[5] The AM technologies that are most commonly selected to process polymers in dentistry are vat-polymerization and material jetting procedures.[6]

Vat-polymerization AM technologies for dental interim restorations use a liquid resin that is

polymerized upon exposure to a light source of specific wavelength. There are four main categories including stereolithography (SLA), direct light processing (DLP), liquid-crystal display (LCD) also called daylight polymer printing (DPP), and continuous liquid interface printing (CLIP),[7] with stereolithography being the most popular form of 3D printing in dentistry, incorporating a wide range of materials and applications.[3] In SLA printing a building platform moves up and down in the z-axis and is immerged into a liquid polymer resin which is polymerized using ultraviolet (UV) laser.[7] The laser is focused with a set of lenses and directed with two motorized scanning mirrors (galvanometer), to raster the surface of a vat of monomers, exposing voxels to create 3D polymer structures.[8,9] The UV laser draws a cross section of the object on the bottom of the resin tank to build up the first layer, and following, the building platform is lowered a few microns to build the next layer.[9] DLP technology is very similar to SLA, with the main difference being the light source. DLP AM uses light from a digital light projector navigated with a digital micromirror device (DMD). The DMD consists thousands of micromirrors the number of which determine the resolution of the projected image.[9] The projected mask of UV light displays the image of the 3D object onto the liquid photopolymer resin and polymerizes the whole layer of the 3D object at the same time,[7] thus making printing faster than SLA.[10] Following, the build platform moves up on the z-axis to allow polymerization of the next layer. DPP uses light from LCD screens to polymerize a photosensitive resin and CLIP uses a DMD to polymerize resin through an oxygen permeable window made of fluoropolymer.[7]

Additively manufactured dental polymer materials for interim restorations are new to the dental market and their chemical composition remains unclear.[6,9] Methacrylates and phosphine oxides acting as UV photo initiators are often included in their composition but further details are proprietary.[2,9] In a study, Revilla-León et al[6] investigated the chemical composition of four AM polymer dental materials and found major differences in chemical composition between conventional

(PMMA and bis-acryl) and AM specimens. Different percentages of oxygen, silica, phosphorus, calcium, and titanium were found both between AM and conventional materials and between the groups of the AM materials.[6] Moreover, according to Lin et. al[11], a reasonable approach for the formulations of 3D printing resins used with DLP technology is to choose monomers commonly found in light-polymerized composite resins (Bis-GMA, UDMA, and TEGDMA).

Recent studies investigating hardness and flexural strength have shown similar or better performance of AM resins compared to bis-acryl resin.[2,8] However, flexural strength and fracture resistance of additively manufactured interim crowns has been found to be lower than subtractive manufactured or conventional PMMA interim crowns.[12,13] Moreover, in one study, the color stability of printed resins was found to be lower, an outcome possibly influenced by the reduced time of postprocessing that the authors followed.[2] Shade matching of AM manufactured interim materials using the commercially available shade guides, is another potential issue and further research is needed on the topic.[14]

Several parameters have been shown to influence the mechanical and physical properties of 3D-printed resins, such as printing parameters (build orientation, position in the building platform, layer thickness, supporting structures), post-polymerization protocols (removal, cleaning, post-polymerization and trimming of supporting structures),[8,15,16,7,17]type of resin material, and artificial aging.[15] Regarding the build orientation, the highest fracture load values have been found for specimens positioned in 90 degrees printing orientation. According to this finding, the load tolerance is higher when the layers of the printed restoration are orientated perpendicular to the direction of the load.[15,18] However, in terms of the dimensional accuracy, superior results have been found with 45 degrees[16] and 60 degrees printing orientation combined with a thin support type.[19] In addition, 45 and 60 degrees build orientation have been recommended for better results in terms of the marginal and internal gap of 3D printed resin restorations[20], while 45 degrees build orientation results in higher

surface roughness compared with 0 or 90 degrees.[21] The layer thickness is another important factor that determines the resolution of the object on the z-axis and is based on the morphologic features and the intended application of the object. For objects that require higher accuracy a reduced layer thickness is preferred.[19]

For AM implant-supported interim restorations, two types of stock abutments are widely used: either temporary cylinders or prefabricated titanium bases. In either case the interim crown needs to be bonded on the abutment. Titanium (Ti) bases are widely used to connect implants and ceramic screw-retained superstructures (abutments or crowns) ensuring the advantages of a titanium-titanium implant abutment connection.[22,23,24,25] The cementation protocol of different types of ceramic crowns, such as zirconia or lithium disilicate, on titanium bases has been widely investigated.[22,26,27,28,29,30,31] These cementation protocols recommend the use of adhesive agents such as silane for acid-etched ceramics and 10-MDP primer for zirconia and titanium surface pre-treatment.

Silane coupling agents are organosilane compounds that have at least two different reactive groups bonded to a silicon atom in a molecule. In dentistry, the commercially available silane coupling agents usually contain 3-methacryloxyproyltrimethoxysilane (MPS), which is a trialkoxysilane, and are effective in bonding silica-based restorative materials such as resin, composite luting cements and etchable ceramics.[32] However, for non- silica based materials such as zirconia or metal alloys (e.g. Co-Cr, Ni-Cr) a specific surface pre-treatment should be applied prior to use of silane for increased effectiveness.[33] An example of such a surface pre-treatment is tribochemical silica coating combined with silanization, where the surface is air-particle abraded with silica-coated alumina particles. The embedded particles increase its chemical reactivity, by resulting in chemical reaction between the silica layer and the silane and formation of silane molecules.[32] Moreover, the phosphate monomer 10-methacryloyloxydecyl dihydrogen (10-MDP)

was developed to achieve direct bifunctional adhesion with metal oxides (including zirconia and alumina) or calcium via a phosphate ester group and the resin bis-GMA matrix via a methacrylate group.[34] 10-MDP can bond to a variety of substrates such as tooth structure, ceramics, and metals including cast alloys.[35,36,37] Recently, universal adhesives containing a mixture of acidic ethanol solutions of silane with phosphate coupling agents have been marketed with the purpose to expand their applications in ceramic bonding and repair.[38] The effectiveness of those agents, however, remains controversial since combining hydrolyzed silanes with dental monomers possessing -OH groups results in condensation reactions and deactivation of the silanol (Si-OH) groups.[38]

Recently, a study also investigated the bonding protocol of milled PMMA interim crowns on titanium bases and suggested pretreatment of the intaglio surface of the interim crown with 30 μm silica-coated Al_2O_3 particles and an MMA-based liquid to improve retention.[1] However, to the author's knowledge there is no study investigating the bond strength and cementation protocols of AM interim crowns on titanium bases.

The purpose of the present in vitro study was to evaluate the effect of different surface treatments and bonding protocols on the retentive bonding strengths of implant-supported AM interim crowns on Ti-bases. The null hypothesis was that the surface treatment of the intaglio AM interim crown will not significantly affect the bonding strength on Ti-bases. Therefore, this in-vitro study aims to provide scientific guidelines regarding the preferred surface treatment of AM interim crowns prior to cementation on titanium base abutments.

2. MATERIALS AND METHODS

2.1 Crown fabrication

Fifty commercially available titanium (Ti) bases (Ti-Base- Engaging 4.3/5.0 [RP] Nobel Active compatible; Dess), were used. To design interim crowns, a Ti-base was screwed onto an implant analog (Implant Replica Conical Connection RP; Nobel Biocare) and scanned with a desktop scanner (D900 3D scanner; 3Shape) with 15 microns accuracy. The digital file of the Ti-base was saved as a Standard Tesselation Language (stl) file and subsequently imported into a CAD dental software program (DentalCAD, Galway; Exocad). A maxillary canine complete anatomic contoured crown was designed with a 300 μm cement space, to fit the Ti-base. The stl file of the crown was then imported to a non-dental CAD designing software program (Meshmixer; Autocad) and customized to allow for the pull-out testing by adding a rectangular shape beam with dimensions 10x10x30mm. Boolean difference was used to create the access hole (3mm in diameter) and the rectangular beam was combined with the crown and exported as an stl file. (Figure 1)

Fifty interim crowns (customized design) were additively manufactured with a DLP (digital light processing) printer with a XY resolution of 50μm (Pro55 S; SprintRay) and interim dental polymer material (Temporary CB resin A1; Dentca) (Figure 2). The crowns were printed with a 50 μm layer thickness and at a 40-degrees building orientation, per manufacturer's recommendation. The support structures were set at medium size and density. After printing, the specimens were detached from the building platform using a spatula, rinsed in a bath (Pro Wash/Dry; SprintRay) with a clean solution of isopropyl alcohol 99% for 15 minutes,[19] and air-dried. Support material was trimmed using a handpiece with a cutting disk and the crowns were post-polymerized for 60 minutes at 30°C using an UV-polymerization machine (Pro Cure 2; SprintRay), following manufacturer's instructions.

2.2 Cementation

The specimens were randomly assigned into five subgroups (n=10) testing different surface treatments of the intaglio interim crown and cementation protocols, namely Control (no surface treatment), AP group (surface treatment with 50μm Al_2O_3 air-abrasion), MP group (surface treatment with silane primer), AMP group (surface treatment with 50μm Al_2O_3 air-abrasion and silane primer), and CMP group (surface treatment with tribochemical silica coating and silane primer) (Table 1). The power analysis and sample size calculation was based on a previous study applying a similar methothology,[20] effect size, and α=0.05.

All Ti-bases were airborne particle-abraded with 50 mm Al_2O_3 particles for 20 seconds at a pressure of 2.5 bar and from a distance of 10 mm, cleaned in an ultrasonic alcohol bath for 3 minutes, and conditioned with Monobond Plus (Ivoclar Vivadent) for 60 seconds.[1,19] Subsequently, the surfaces were gently air dried, and the titanium bases were screwed into an implant analog (Implant Replica Conical Connection RP; Nobel Biocare) (Figure 3). The access hole of the abutment was filled with teflon tape and the interim crowns were cemented on their respective implant abutment, following one of the five different surface treatments and using the same composite resin cement (Rely X Unicem2; 3M ESPE).[1] To standardize the cementation procedure, all crowns were seated with a constant 50N force applied with a fixation device (Harvest Clip, Harvest dental laboratory products) and allowed a setting time of 5 minutes[9] (Figure 4,5).

Table 1. Summary of treatment groups (n=10 for each treatment group)

Groups	C	AP	MP	AMP	CMP
Surface treatment of the intaglio AM interim crowns	None	Air-abrasion with 50-µm Al_2O_3	Silane (Monobond Plus, Ivoclar)	Air-abrasion with 50-µm Al_2O_3 and silane (Monobond Plus, Ivoclar)	Air-abrasion with 30µm silica coated Al_2O_3 particles (CoJet) and silane (Monobond Plus, Ivoclar)
Cement	Self-curing composite resin cement (RelyX Unicem2, 3M)				

The five groups of different surface treatments were established as follows (**Table 1**):

Group C (control): No surface treatment was performed. The interim crowns were cemented with the selected self-curing resin cement over Ti -base abutments.

Group AP: The intaglio of the interim crowns was airborne particle-abraded with 50-µm Al_2O_3 particles for 15 sec at a pressure of 2.5 bar and from a distance of 10mm, and cleaned in an ultrasonic alcohol bath for 3 min. The crowns were then cemented with the selected self-curing resin cement over Ti -base abutments.

Group MP: The intaglio of the interim crowns was silanized (Monobond Plus; Ivoclar Vivadent) for 60 seconds and air-dried. The crowns were cemented with the selected self-curing resin cement over Ti -base abutments.

Group AMP: The intaglio of the interim crowns was airborne particle-abraded with 50-μm Al_2O_3 particles for 15 sec at a pressure of 2.5 bar and from a distance of 10mm and cleaned in an ultrasonic alcohol bath for 3 minutes. Following, the crowns were silanized (Monobond Plus; Ivoclar Vivadent) for 60 seconds, air-dried and cemented with the selected self-curing resin cement over Ti -base abutments.

Group CMP: The intaglio of the interim crowns was airborne particle-abraded with 30μm silica coated Al_2O_3 particles (CoJet), using an intraoral sandblaster (MicroEtcher IIA sandblaster; Zest Dental Solutions), for 15 seconds and from a distance of 10mm and cleaned in an ultrasonic alcohol bath for 3 minutes. Subsequently, the crowns were silanized (Monobond Plus; Ivoclar Vivadent) for 60 seconds, air-dried and cemented with the selected self-curing resin cement over Ti-base abutments.

The chemical composition provided by the manufacturers of the materials used in the study are shown in Table 2. After the cementation procedures, the excess of cement was cleaned, and the specimens were kept in a lightproof box. Prior to testing, the specimens were stored in distilled water at 37°C for 24h. Following storage, the interim crowns were screwed into an implant analog (Implant Replica Conical Connection RP; Nobel Biocare) at a 20N torque value. The analog was embedded in a vertical position in a custom-made acrylic resin holder with an autopolymerizing acrylic resin (Jet tooth shade powder and Jet liquid; Lang Dental), following ISO 14801.[19,26]

2.3 Retention testing

Retention testing was performed with a universal testing machine (Instron; Instron Corp.) at a crosshead speed of 2 mm/minute (Figure 6,7). After the tension tests, the fractured interfaces were examined by using an optical microscope (SZX7; Olympus) under x20 magnification for

remaining cement and failure mode classification. An adhesive remnant index (ARI) system was used to determine the amount of adhesive resin cement that remained on the crown surface after debonding. Failure modes were classified as: ARI 0 = all of the adhesive remaining on the crown, ARI 1 = >50% of the adhesive remaining on the crown, ARI 2 = <50% of the adhesive remaining on the crown, ARI 3 = no adhesive remaining on the crown.[39, 40]

2.4 Calculations and Statistical Analysis

Axial pull-out forces were measured in kilograms force (kgf) and converted to Newtons (N). The peak force measured prior to bonding failure was recorded for each specimen. All statistics and analyses were computed using SPSS software program (SPSS Statistics, version 27; IBM). Each group was tested for normal distribution with a Kolmogorov-Smirnov test and for homogeneity of variance using Levene's tests. Due to non-normal distributions, non-parametric Mann-Whitney U tests with Bonferroni corrections for multiple tests were performed for between and within groups comparisons with an α=.05 for all tests.

Figure 1: Customized design for pull-out apparatus

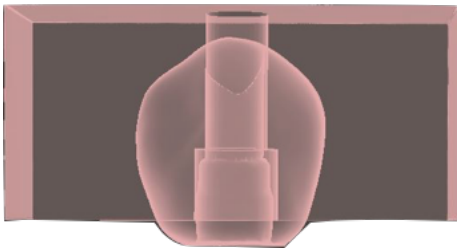

Figure 2: AM crowns

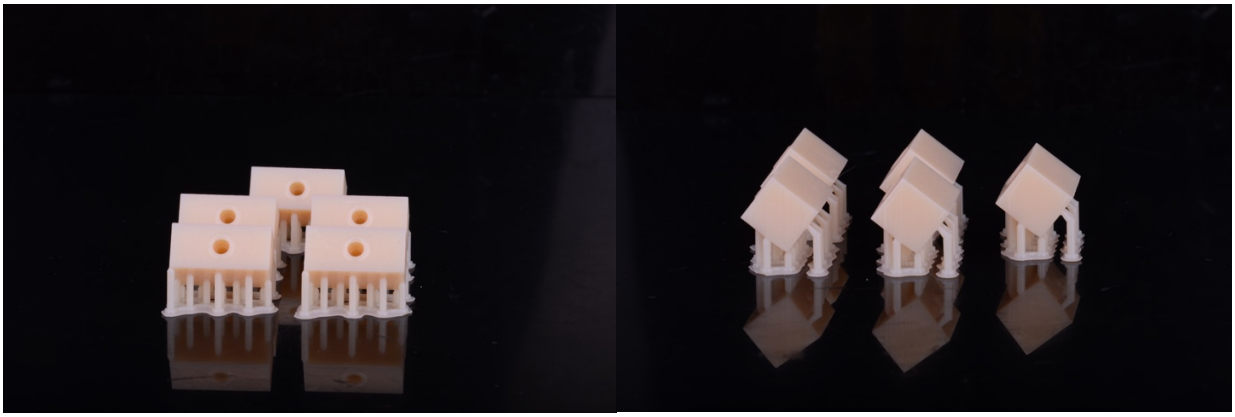

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Figure 3: Titanium base screwed into implant analog

Figure 4: Fixation device

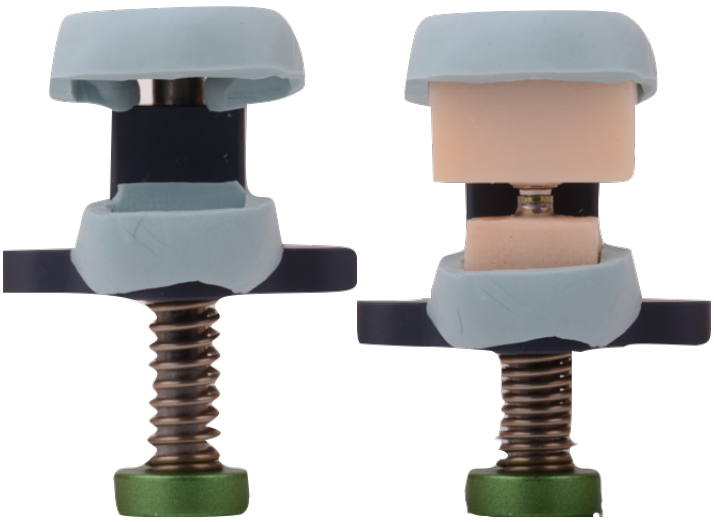


Figure 5: Cemented crown

Figure 6: Pull-out testing apparatus

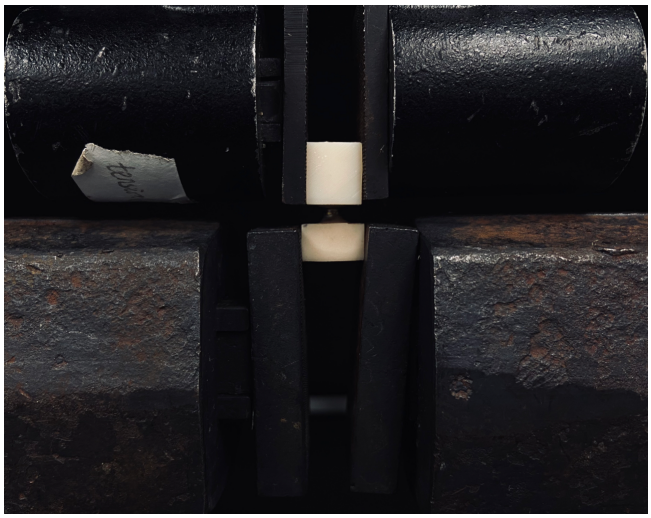
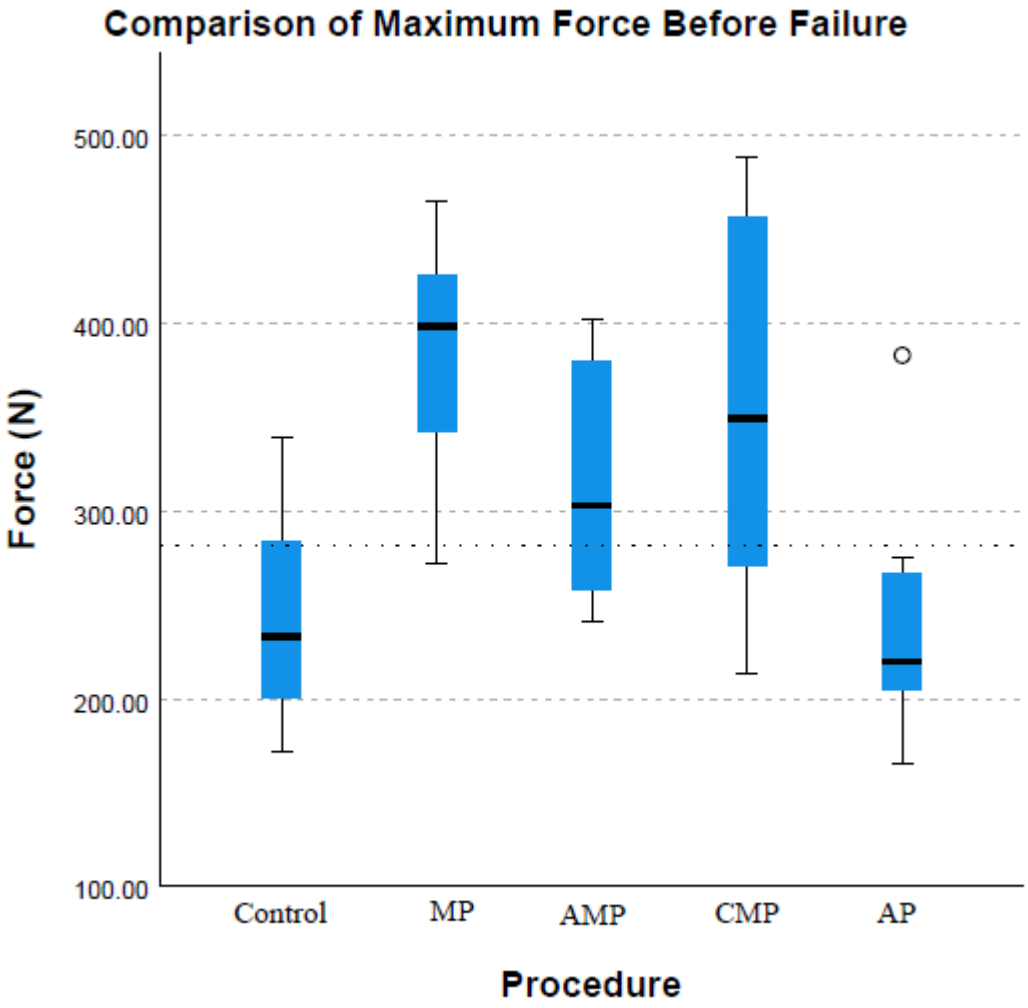


Figure 7: Pull-out test

Table 2. Chemical composition of materials used (according to manufacturer)

Material; Manufacturer	Composition
MicroBlaster, Comco Inc	50 µm aluminum oxide particles
CoJet (Tribochemical silica coating sands)	30 µm alumina particles modified by silica
Monobond Plus; Ivoclar Vivadent	Ethanol, 3-trimethoxysilylpropyl methacrylate, MDP and disulfide acrylate
Rely X Unicem; 3M ESPM	Methacrylate monomers, methacrylated phosphoric acid esters, dimethacrylate, silanated fillers, sodium persulfate, substituted pyramidine, calcium hydroxide, initiator components, stabilizers
Crown and Bridge A1; Dentca	Urethane Dimethacrylate (UDMA), methacrylates

Figure 8: Comparison of Maximum Force before failure between groups



3. RESULTS

The descriptive statistics of the maximum force before failure (N) are presented in Table 3. The average mean retention force values for all the specimens were 307.04N with a standard deviation of 88.97N and a median of 281.29N. The median retention force values were 233.27 ±79.28 N for Group Control, 398.59 ±68.59 N for Group MP, 303.21 ±116.80 N for Group AMP, 349.31 ±167.73 N for Group CMP, and 219.85 ± 55.88 N for Group AP. Mann-Whitney U Tests for differences between sample groups (Table 4) revealed that the forces were significantly greater for Group MP compared to Group Control (P<.05) and Group AP (P<.05). There was no statistically significant difference between Group AMP and the rest of the Groups (P<.05). Also. There was no statistically significant difference between Group CMP and the rest of the Groups (P<.05), although the difference between Group CMP and Group AP was approaching significance. Results for Group CMP showed the greatest variations in force values. The median maximum force was the lowest for Group AP and lower compared with group Control, although there was no statistically significant difference (Figure 8).

The failure modes were predominantly adhesive and substrate failure of the AM crown. 66% of the specimens presented a combination of debonding and fracture of the AM crown after pull-out testing. The remaining 26% presented predominantly debonding, whereas 4% did not present loss of retention between the crown and the abutment after the pull-out test. For these specimens the failure corresponded to the fracture of the printed material. The ARI scores are shown in Table 5. The majority of the samples had <50% cement remaining on the crown surface.

Table 3. Descriptive Statistics for Maximum Force (N)

Group	Mean	Standard Deviation	Median	Interquartile Range
Control	243.18	55.45	233.27	79.28
MP	385.48	61.29	398.59	68.59
AMP	314.97	63.09	303.21	116.80
CMP	352.28	99.26	349.31	167.73
AP	239.49	60.46	219.85	55.88

Table 4. Mann-Whitney U Tests for differences between sample groups.

Comparison	Test Statistic	Adjusted Significance*
Control – AP	0	1.000
AP – AMP	3.20	0.736
AP – CMP	7.20	0.073
AP – MP	12.80	0.003
Control – AMP	.80	1.000
Control – CMP	.80	1.000
Control – MP	12.80	0.003
AMP – CMP	.80	1.000
AMP – MP	3.20	0.736
CMP – MP	3.20	0.736

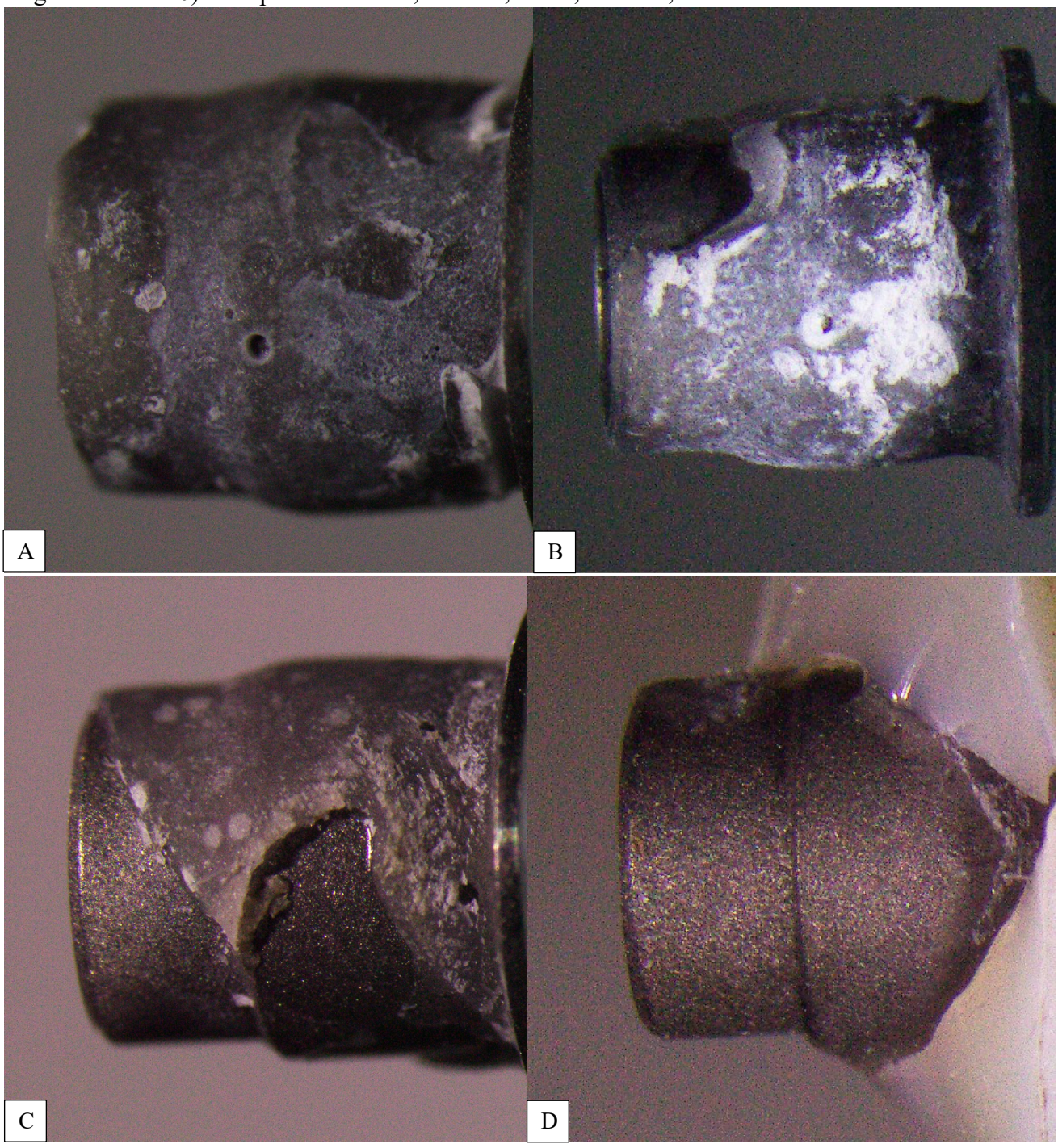
*Bonferroni correction for multiple tests

Table 5 Adhesive remnant index (ARI) scores of interfaces of all test groups

Group	**Surface**	**ARI 0**	**ARI 1**	**ARI 2**	**ARI 3**	**Mode of failure**
Control	Titanium	0	0	9 (90%)	1 (10%)	Adhesive/ substrate
MP	Titanium	2(33.3%	4(66.7%)	0	0	Adhesive/ substrate
AMP	Titanium	0	2 (22.2%)	5 (55.6%)	2 (22.2%)	Adhesive/ substrate
CMP	Titanium	0	0	7 (77.8%)	2 (22.2%)	Adhesive/ substrate
AP	Titanium	1 (10%)	1 (10%)	7 (70%)	1 (10%)	Adhesive/ substrate
Total	Titanium	3	6	28	6	Adhesive/ substrate

ARI 0, all of the adhesive remaining on the crown; ARI 1, > 50% of the adhesive remaining on the crown; ARI 2, <50% of the adhesive remaining on the crown; ARI 3, no adhesive remaining on the crown

Figure 9: Representative photographs of remaining cement on titanium base surfaces (original magnification x20) after pull-off test. A, CMP B, AP C, AMP D, MP



4. DISCUSSION

The present study evaluated the bonding strength of AM interim crowns cemented on Ti-bases with resin cement, following different surface pre-treatments of the intaglio AM crown. The surface treatment of AM crowns influenced the retention forces to Ti-bases. Therefore, the null hypothesis was rejected. Pre-treatment of the intaglio AM crown with an MDP-containing silane (Monobond Plus; Ivoclar Vivodent) improved bonding strength when compared to no pre-treatment or pre-treatment with 50μm alumina air-abrasion.

Silane coupling agents establish a covalent bond to substrates having silica particles by forming siloxane bonds and to the resin matrix monomers by carbon double bond polymerization. In dentistry, silanes have been used to achieve bonding between silica-base ceramics and resin, adhesion between the polymeric matrix and the fillers of resin composites and for resin composite repairs.[36,41,37] These agents are often marketed in combination with organophospate monomers such as MDP, which contain a phosphate ester group that reacts with metal oxides and a terminal double bond group to copolymerize with resin.[37] Several studies have shown enhanced bonding to zirconia, silica based ceramics and resin with the use of MDP-containing silanes.[30] However, it has also been demonstrated that combining hydrolyzed silanes with phosphate monomers -OH like 10-MDP deactivates the silanol (Si-OH) groups and may diminish the bonding potential with the substrate.[38] In agreement with these studies, the present study showed highest bonding strengths of AM crowns after pre-treatment with an MDP-containing silane (Monobond Plus; Ivoclar, Vivodent) which could result from the chemical reaction of the silane with the AM resin. However, to further understand the mechanism of reaction, more information should be revealed regarding the composition of commercially available AM resin materials.

Air-particle abrasion is a technique used to roughen the surface of a material before bonding, aiming to increase the bonding area and enhance the micromechanical interlocking of the cement.[36] The combination of 50 μm alumina particles air particle abrasion with an MDP primer has been proven to effectively improve bonding to zirconia.[26,27] Moreover, air-particle abrasion with silica-coated alumina particles (tribochemical silica coating) in combination with silanization has been found to improve bonding of resin cements and composites to metal alloys and ceramic restorations, and increase the bond strength of repaired resin composite restorations.[33] In a recent study, the combination of 30 μm silica-coated Al_2O_3 air-particle abrasion with an MMA-based liquid was recommended to improve retention of CAD-CAM PMMA interim crowns on Ti-bases.[1] Contrary to those results, in the current study, surface pre-treatment of the AM interim crown with air-particle abrasion in combination with an MDP-containing silane did not statistically improve the bond strength compared to no surface pre-treatment. Moreover, whenever 50 μm air-abrasion alone was used before cementation, the results showed reduced bonding strength, although the difference was not statistically significant. These results could be explained by the stepwise connection between different layers of the AM interim material and the increased surface roughness[21,42](4), thus enabling adequate surface area for bonding and micromechanical interlocking without the use of air-abrasion. However, more studies need to be conducted in order to evaluate the effect of surface roughness of 3D-printed materials on the bond strength to ti-bases.

In the present study pre-treatment with 50μm Alumina air-abrasion resulted in the lowest bonding strengths. This finding may be attributed to an alteration of the surface geometry of the AM material potentially by smoothening the step edges between the layers and resulting in a less micro-retentive surface and an increased cement gap. A similar finding was observed in a study by Arce et al[22] measuring the retention between titanium base abutments with microgrooves and zirconia crowns. The study found that pre-treatment of the abutments with Alumina air-abrasion resulted in

lower retention values caused by an increased cement gap and a change in the surface topography of the microgrooves.

Tribochemical silica coating followed by silanization has been used especially with non-silica based materials to improve bonding because of a chemical reaction between the silica layer and the silane[29] and an increase in the hydrophobicity which favors energetically bonding to resins.[33] The silane can be either applied as a different step or be encapsulated in the silica microspheres of the coating, thus resulting in simultaneous roughening, silica implantation and silanization of the substrate.[33,38] In the present study the use of tribochemical silica coating as an additional step prior to application of an MDP-containing silane resulted in increased retention values. However, this pre-treatment did not significantly improve the retention, showed high variability of results and reduced strength compared to the application of an MDP-containing silane alone, thus leading to the conclusion that tribochemical silica coating was not beneficial prior to silanization. This might be explained from the composition of the AM printed material and the chemical reaction with the applied primer.

Failure modes after the pull-out testing were predominantly adhesive and substrate failure of the printed material. The majority of the samples exhibited a combination of debonding and fracture of the crown which is suggestive of increased retention forces compared with the tensile strength of the AM material. In most of the samples exhibiting adhesive failure mode for Groups Control, AM, AMP and CMP, the cement remained predominantly on the abutment surface (AR2) which shows a stronger bonding between the abutment and the cement compared to the crown and the cement. In contrast the cement remained predominantly on the crown (AR1) for group MP, supporting the increased retention values that were found in that group. Moreover, Group MP had the highest

number of crown fractures without debonding (30%). This observation is also suggestive of increased retention values.

Dess Ti bases compatible with Nobel Replace CC implants were used which have 4.2mm height and a patented laser surface treatment which ensures greater adhesion of the structure with cement. Previous studies have shown increased bonding strengths of Ti bases following Alumina particle air-abrasion[29] and application phosphate based primers.[30] This protocol was followed in the present study for the pre-treatment of Ti-base abutments prior to cementation.

In general, all the groups showed good retention values comparable to CAD-CAM fabricated PMMA crowns cemented on Ti-bases.[1] However, the present study had several limitations including the in-vitro design, which may not represent clinical conditions and the use of only one 3D printing technology and AM interim material with specific printing parameters and post-processing procedures. This constitutes the generalization of the results difficult since there is a great variability in the chemical composition and reactivity of 3D printing materials, as well as adhesive materials. Moreover, the specimens did not undergo thermocycling prior to pull-out testing which could influence the mechanical properties of the AM material and decrease the bonding strength values.[1,44] Other limitations include the non-anatomical shape of the crown and the increased cement space used to compensate the material shrinkage. Finally, mounting of the specimens on the Instron machine might have potentially introduced off-axis forces during retention testing.

5. CONCLUSIONS

Within the limitations of this in vitro study the following conclusions can be drawn:

1. Surface pre-treatment of the intaglio AM crown with a 10-MDP containing silane statistically increased the retention forces between the AM crown and the Ti-base abutment.
2. The use of tribochemical silica coating or 50-μm Al2O3 particle air-abrasion prior to silanization did not prove beneficial for the retention forces between the AM crown and the ti-base abutment.
3. Surface pre-treatment of the intaglio AM crown with 50-μm Al2O3 particle air-abrasion is not recommended prior to cementation on a Ti-base abutment.
4. Extrapolation of the results should be based on the specific AM technology and materials used and generalizations should be done carefully.

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