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Original Article

Retention of zirconia crowns to Ti-base abutments: effect of luting protocol, abutment treatment and autoclave sterilization

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Abstract

Purpose: To evaluate the influence of resin cement type, surface pretreatment and autoclave sterilization on the retention of Y-TZP crowns to Ti-base abutments.

Methods: Y-TZP crowns were designed and milled to fit Ti-base abutments. Crowns were cemented using either a conventional resin cement (conventional) with a universal adhesive or a self-adhesive resin cement (self-adhesive), both following no surface pretreatment (No) or Ti-base abutment sandblasting (SB) (n=20/group). Half of the cemented samples were subjected to in-office autoclave sterilization. Pullout testing was performed in a universal testing machine at a speed of 1 mm/min until crown displacement. Data were statistically evaluated through a linear mixed model following post hoc comparisons by LSD test.

Results: Pullout data as a function of cement type demonstrated higher retention for conventional relative to self-adhesive cement ($p<0.001$). Ti-base sandblasting (SB) favored crown retentiveness over No pretreatment ($p<0.001$). Sterilized crowns exhibited higher pullout values than non-sterile ($p=0.036$). All the two- and three-factor interaction analyses corroborated with the superior adhesive strength of conventional compared to self-adhesive cement (all, $p<0.011$), as well as, SB relative to No pretreatment (all, $p<0.024$). While autoclave sterilization maximized bond strength when self-adhesive cement (data collapsed over surface pretreatment, $p<0.050$) and No pretreatment were evaluated (data collapsed over surface pretreatment, $p<0.013$), no significant difference was observed for conventional resin cement ($p=0.280$) and SB ($p=0.878$) groups.

Conclusions: Conventional resin cement and/or Ti-base sandblasting increased Y-TZP crown retentiveness, with no significant influence of autoclave sterilization. Autoclaving increased retentiveness when self-adhesive cement and/or no Ti-base pretreatment were used.

Keywords: *Dental implants, Dental prosthesis, Dental prosthesis Retention, Resin cement, Sterilization*

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1. Introduction

Prefabricated or custom metallic abutments and metallic or yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) infrastructures along with porcelain veneering are the evidence-based standard reconstructive choices for single implant-supported rehabilitations [1-5]. Some recent findings support such a perspective through the presentation of high survival rates, more than 94%, for both metal-ceramic and Y-TZP single crowns after an observation period of up to 10 years [1,4]. Although results are encouraging, biological and technical complications have been frequently reported for implant-supported reconstructions and may influence clinical success, including retention loss (5%), abutment/screw loosening (3-8%), and veneer fracture (3-9%) [1-5]. Likewise, the incidence of complications has previously shown to be affected by the prosthesis retention mode, with screw-retained rehabilitations leading to higher levels of technical complications and cement-retained rehabilitations to more serious biological concerns [6,7]. Therefore, researches on prostheses components to provide retrievable systems with more favorable esthetic and biomechanical performance warrants further investigation in implant dentistry [8].

Technological advances have opened the opportunity to implement a unique digital workflow in the field of oral rehabilitation, where intraoral scanning and computer-aided designing/computer-aided manufacturing (CAD/CAM) allow reproducible and structured processes that may improve time-efficiency (approximately 50% reduction in the work time) and deliver a highly precise prosthetic reconstruction (marginal gaps below than 140 μm) [9-14]. Although favorable results in highly esthetic areas have been reported, Y-TZP

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abutments presented significantly higher fracture rates relative to metal abutments (1.8% and 0.08% 5-year facture rate, respectively), along with higher levels of implant connection wear [7,15]. Hence, CAD/CAM compatible titanium base abutments (Ti-base) have been used as a platform for subsequent milling of customized ceramic prostheses that are cemented chairside to the Ti-base and screwed to the implant [9-12]. This hybrid retention Ti-base concept advantages include esthetics, reproduction of a tailored emergence profile to reestablish soft tissue anatomic contour, retrievability, improved biomechanical performance, absence of ceramic material at the implant-abutment connection level, as well as, bonding procedure under controlled laboratory conditions [7,16-19].

Cementation is a critical procedure for implant-supported reconstructions as titanium and Y-TZP are commonly used interfaces that challenge conventional adhesive bonding due to the high opacity and inadequate photopolymerization, as well as, the inefficiency of common surface pretreatments on polycrystalline surfaces involving silica coating (up to 5% retention loss after 5 years) [1-5,20-23]. The bond strength of implant-supported prostheses has previously shown to be influenced by abutment material, height and convergence angle, prostheses adaptation, surface pretreatment, cement type, and cement degradation rate [23-31]; however, there is limited scientific evidence concerning the bonding behavior of different luting systems to hybrid Ti-base reconstructions [16,17,31]. As previously evaluated, the retentive strength of resin cement resulted in up to threefold higher force to dislodgment relative to temporary and glass ionomer cement, irrespective of crown material and resin cement type [16,17]. Moreover, self-adhesive resin cements have emerged as a novel and simplified luting technology relative to conventional resin cement, with bonding performance yet to be elucidated when used to bond Y-TZP to Ti-base abutments [16,17,32,33]. Other possibilities to reasonably enhance adhesive bond strength of metal and Y-TZP consist of chemical and micromechanical surface pretreatments, including air particle abrasion and functional monomers coating (i.e. 10-methacryloyloxydecyl dihydrogen phosphate-MDP) [20-23,34].

After bonding procedures and to prevent tissue reaction to microbiological contaminants and pollutants, sterilization protocols have been indicated for some manufacturers prior to prostheses installation [35-38]. The effect of Ti-base non-thermal argon plasma sterilization on microbiologic parameters and soft and hard tissue response has previously presented promising results (reduced levels of pathogenic bacterial growth and bone loss) relative to non-sterile prosthetic components [35,36], however, concerns were subsequently raised since the method has not complied with approved disinfection norms for semi-critical medical devices, such as the in-office autoclave method (BS EN ISO 17664:2004) [39]. Nonetheless, it must be taken into account that the cleaning processes should be performed immediately before the abutment screwing since recontamination can promptly occur [38,39], whereas the effect of the heat treatment and pressure associated with autoclave sterilization protocol on crown retention still needs to be addressed [38].

Accordingly, this study sought to evaluate the effect of resin cement type, surface pretreatment, and in-office autoclave sterilization on the pullout retention of Y-TZP crowns to Ti-base abutments. The postulated null hypotheses were that: (i) cement type would not influence pullout retention values, (ii) surface pretreatment would not influence pullout retention values, and (iii) autoclave sterilization would not influence pullout retention values.

2. Materials and Methods

2.1. Sample preparation

A total of 80 Y-TZP crowns with identical external geometry were designed in a CAD software (Ceramill Mind, Amann Girrbach, Curitiba, PR, Brazil) to fit a 4mm-height Ti-base abutment (Ti-Base, Emfils, Itu, SP, Brazil). Projections beyond the occlusal surface were designed at the buccal and lingual cusps of the crowns presenting a round perforation of 2 mm to attach stainless-steel wires for pullout testing. Such projections were designed at the cusps to allow occlusal access for abutment screw torqueing [17]. In addition, an internal space of 50 μm for cementation was planned, starting 0.5 mm from the margin. The Ti-base abutments presented internal conical connection to fit their implant counterpart geometry (n=80, Novo Colosso, Emfils, Itu, SP, Brazil).

Ti-base abutments and their corresponding implants were embedded in acrylic resin (Jet, Clássico Artigos Odontológicos, São Paulo, SP, Brazil). For standardized long axis alignment, Ti-base abutments digitally torqued to the implants were fixed in a surveyor (Delineador B2 - Bio-Art, São Carlos, SP, Brazil) and acrylic resin poured in 25mm diameter PVC tubes with their long axis aligned to the tube long axis simulating the bone level of the implant for the tests. This procedure confirmed that the implant and Ti-base abutment were on the same axis for pullout testing. The acrylic base held the samples in the same position once fixed to a universal testing machine (Kratos KE, Kratos, Cotia, SP, Brazil).

The retention of Y-TZP crowns to Ti-base abutments was evaluated using two different resin cement types and surface pretreatments, as detailed for the following four groups : (i) self-adhesive resin cement (self-adhesive) (RelyX U200, 3M Oral Care, St. Paul, MN, USA) without Ti-base sandblasting (SB), (ii) conventional resin cement (conventional) (RelyX Ultimate, 3M) with universal adhesive containing MDP monomers for chemical coating of titanium and Y-TZP substrates (Scotchbond Universal, 3M) without Ti-base SB, (iii) self-adhesive resin cement and Ti-base SB with alumina particles of 45 μm, at 2 bars, for 15 seconds, at a 5 mm step-over distance, and (iv) conventional resin cement and Ti-base SB (n=20/group). To standardize cementation, after cement mixing and crown insertion on the abutment, a weight of 5kg was used for 10 minutes until cement polymerization. Excess cement was removed from the abutment margin and LED light was initially applied on each surface for 20 seconds (Valo Cordless, Ultradent, South Jordan, UT, USA). Half of the samples of each group underwent autoclave sterilization at 134°C, at 2 bars for 10 minutes before testing. Thereafter, Ti-base abutments were digitally torqued as per manufacturer recommendations (32 N.cm, Tohnichi America Corporation, Buffalo Grove, IL, USA). The tested groups description is illustrated in the figure 1.

2.2. Pull-out retention testing

A stainless-steel wire of approximately 1.8 mm was inserted through both occlusal projections of each crown and fixed in a device positioned directly below the load-cell. Pull-out testing was performed in a universal testing machine (Kratos KE, Kratos) at a cross-head speed of 1 mm/min until crown displacement and load drop. The force (N) was recorded for statistical analysis.

2.3. Statistical analysis

Preliminary data analysis has shown indistinguishable variances in the study of the dependent variable (Levene test, all $p>0.25$). Pullout retention data were statistically evaluated through linear mixed model with fixed factors of resin cement type (self-adhesive and conventional), surface pretreatment (No and SB), and Ti-base sterilization (sterile and non-sterile). Post hoc comparisons were determined by Fisher's least square difference (LSD) test. Data are presented as a function of mean values with the corresponding 95% confidence interval (CI). All analyses were accomplished using SPSS (IBM SPSS 23, IBM Corp., Armonk, NY).

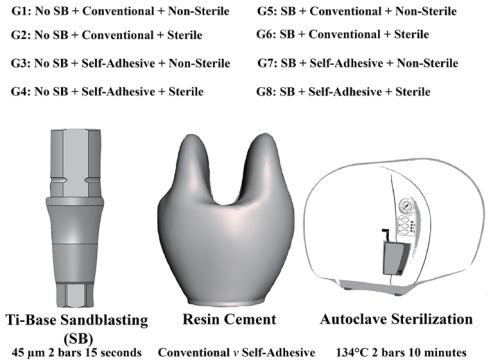


Fig. 1. Schematic of tested groups.

3. Results

Pullout data as a function of cement type demonstrated higher retention values for conventional resin cement associated with universal adhesive (244 ±18 N) relative to self-adhesive resin cement (181±18 N) ($p<0.001$). Analysis of surface pretreatment showed higher retentiveness when Ti-base abutments were sandblasted (SB: 250 ±16 N and No: 175 ±21 N) ($p<0.001$) (data collapsed over cement type and sterilization). Autoclave sterilization increased the retention of adhesively cemented Ti-base abutments (sterile: 226 ±18 N and non-sterile: 198 ± 18 N) ($p=0.036$) (data collapsed over cement type and surface pretreatment).

The interaction between cement type and surface pretreatment resulted in higher crown pull-out retention of conventional relative to self-adhesive resin cement, irrespective of surface pretreatment ($p<0.001$). Likewise, higher pullout retention was observed when the Ti-base surface was sandblasted relative to no surface pretreatment, regardless of resin cement type ($p<0.005$) (Table 1).

Pullout data as a function of cement type and sterilization showed higher values for conventional compared to self-adhesive resin cement for sterile and non-sterile Ti-base abutments ($p<0.004$). While similar force to dislodgement between sterile and non-sterile assemblies that followed a conventional adhesive luting procedure ($p=0.280$), sterile Ti-base exhibited significantly higher pullout retention relative to non-sterile for self-adhesive cement ($p<0.05$) (Table 2).

Evaluating the influence of the surface pretreatment following autoclave sterilization confirmed the higher retentiveness of sandblasted Ti-base abutments relative to no surface pretreatment ($p<0.008$). While a similar force to dislodgement between sterile and non-sterile sandblasted Ti-base abutments ($p=0.878$), sterile assemblies without Ti-base surface pretreatment exhibited statistically significant higher pullout retention in comparison to non-sterile ($p=0.013$) (Table 3).

The statistical description of pullout retention data as a function of all factors, resin cement type, surface pretreatment, and sterilization, highlighted the significantly higher retentiveness presented when using conventional relative to self-adhesive cement for all pairwise comparisons (all, $p<0.011$), except non-sterile with no surface pretreatment ($p=0.321$). Sandblasting the Ti-base abutment demonstrated significantly increased force to dislodgement in comparison to its counterpart with no surface pretreatment, for all studied combinations ($p<0.024$). There was a trend of the sterilization procedure in increasing the pullout retention values when using self-adhesive cement, however, no significant difference has been observed ($p>0.164$). For conventional adhesive cement protocol with no surface pretreatment, sterile assemblies presented significantly higher retention than non-sterile ($p<0.031$). In contrast, no significant difference between non- and sterile groups was observed when using conventional adhesive cementation protocol and Ti-base sandblasting ($p=0.259$) (Table 4).

Table 1. Pullout retention values (N) as a function of cement type and surface pretreatment (data collapsed over sterilization).

	No Sandblasting	Sandblasting
Self-Adhesive	154 (±29) bA	208 (±22) aA
Conventional	195 (±29) bB	293 (±21) aB

Different lowercase letters represent significant difference within rows. Different uppercase letters represent significant difference within columns.

Table 2. Pullout retention values (N) as a function of cement type and sterilization (data collapsed over surface pretreatment).

	Non-Sterile	Sterile
Self-Adhesive	163 (±26) bA	199 (±26) aA
Conventional	234 (±26) aB	254 (±26) aB

Different lowercase letters represent significant difference within rows. Different uppercase letters represent significant difference within columns.

Table 3. Pullout retention values (N) as a function of surface pretreatment and sterilization (data collapsed over cement type).

	Non-Sterile	Sterile
No Sandblasting	148 (±29) bA	201 (±29) aA
Sandblasting	249 (±22) aB	251 (±22) bB

Different lowercase letters represent significant difference within rows. Different uppercase letters represent significant difference within columns.

Table 4. Pullout retention values (N) with all factors depicted, cement type, surface pretreatment and sterilization.

	No Sandblasting		Sandblasting	
	Non-Sterile	Sterile	Non-Sterile	Sterile
Self-Adhesive	133 (±42) cA	175 (±42) bcA	193 (±31) abA	223 (±31) aA
Conventional	163 (±42) cA	228 (±42) bB	305 (±31) aB	280 (±30) aB

Different lowercase letters represent significant difference within rows. Different uppercase letters represent significant difference within columns.

4. Discussion

The current data have shown higher crown-to-abutment retention for conventional resin cement and/or Ti-base surface sandblasted with alumina particles, with a synergistic effect when combined. While autoclave sterilization had no significant influence on Y-TZP crown retentiveness for conventional resin cement and/or Ti-base sandblasting autoclaving enhanced retention for self-adhesive resin cement and/or no pretreatment condition. Therefore, the three null hypotheses postulated that (i) cement type, (ii) surface pretreatment, and (iii) autoclave sterilization would not influence pullout retention values were rejected.

The comparison of different resin cement types in the current study demonstrated higher retention for conventional resin cement associated with universal adhesive relative to self-adhesive resin cement for all factorial analyses. Previous studies have also demonstrated more favorable adhesive bond strength to Y-TZP for conventional relative to self-adhesive resin cement types, especially after artificial aging [17,21,40]. The rationale behind this result may lie on the presence of functional acidic monomers, 10-methacryloyloxydecyl dihydrogen phosphate - MDP in the self-adhesive cement composition instead of in the primer/adhesive itself (components of conventional resin cement systems), which may impact on the polymerization reaction, especially by inhibiting the action of the amine initiator required for the camphor quinone-amine photo initiator system [32]. A decreased degree of conversion (DC) has already been demonstrated for self-adhesive resin

cement (DC Self-adhesive: 26 - 68%/ DC Conventional: 61 - 84%), which may be critical for mechanical performance [41,42].

Bond strength is suggested to be improved with the use of chemical and/or micromechanical surface pretreatments, involving grit-blasting the titanium surface with alumina particles [20]. The current study data exhibited an increased force to dislodgement when Ti-base abutment was sandblasted in comparison to its counterpart with no surface pretreatment, for all studied combinations. Sandblasting the titanium surface has been associated with an increased surface roughness by loss of surface material, which supports an increased micromechanical interlocking through the higher surface area available for bonding, and with an enhanced wettability of the luting system by surface physicochemical modifications [20,21,33,34]. It is noteworthy that the combination of Ti-base sandblasting and conventional adhesive luting provided approximately 30% higher retention values relative to any other group. Also, the combination of SB and self-adhesive cement increased the pullout retention of Y-TZP crowns to the same level of conventional adhesive cement, without surface pretreatment. Therefore, grit-blasting the Ti-base surface has effectively maximized the retention of the resin cement, mainly for conventional resin cement, and the combination of protocols may be clinically important to provide durable adhesive bonds, and, eventually improve implant-supported success rates through fewer reports of technical complications, such as retention loss [20-22,34].

Despite the favorable laboratory-controlled cementation protocol of Ti-base reconstructions, which allows removal of cement residues and final surface polishing [18,19], the presence of contaminants and debris after abutment customization has also been suggested to trigger a tissue-damaging inflammatory process [35,36]. Therefore, autoclave sterilization has been proposed prior to prostheses installation. The present data analysis indicated an increased force to dislodgement of adhesively cemented Y-TZP crowns to Ti-base abutments after autoclaving, mainly for crowns cemented with self-adhesive resin cement and in the absence of Ti-base pretreatment. A previous study has indicated that dual cure resin cement polymerized at increased temperature (～60℃) tended to show higher DC (～8-10% higher) and improved mechanical properties than resin cement polymerized at room temperature, mainly for clinical scenarios of impaired light transmission and predominantly chemical cure of the resin cement (DC: 45-56% at room temperature, DC: 60-67%, at 60℃) [43]. Hence, it is reasonable to speculate that a resin cement with a delayed polymerization (24 hours - 7 days) [32], such as self-adhesive resin cement, as well as, circumstances with no micromechanical and/or chemical pretreatment, may have presented a higher DC after autoclaving due to increased radical mobility and collision frequency of the unreacted active groups [32,43]. In fact, a parabolic relationship has been reported between DC and mechanical properties and once a threshold conversion value has been reached, as in favorable scenarios with conventional resin cement and Ti-base SB, the retention of Y-TZP crowns showed to not markedly increase with additional heat curing from autoclave [32,43,44].

The use resin cements has raised some concerns about their hydrothermal aging resistance since a detrimental effect of bond strength has been previously reported [21]. Nonetheless, a recent systematic review and meta-analysis on resin cement bond strength has shown hardly any effect of artificial aging when the bond surface was pretreated, such as sandblasted and/or functional acidic monomers coated [20]. Therefore, more studies are warranted to evaluate the kinetics of DC of different resin cements and their degradation rate after autoclaving.

The in vitro pullout test conducted in the current study has been widely used to evaluate the retentive strength of tooth- and implant-supported restorations [23-31]. This methodology allows the evaluation of individual variables, such as geometry, surface pretreatments, cement types, and luting protocols on the retentiveness of a restorative system [25]. Nonetheless, the pullout test does not accurately reproduce all oral conditions that can cause crown dislodgement since multidirectional forces are applied on the prostheses [27,31]. Hence, the extrapolation of this in vitro pullout results to in vivo situations must be done with caution and the data should be considered as an initial bonding data ranking the highest immediate bonding scores for different resin cement types and surface pretreatment subjected or not to in-office autoclave sterilization. The findings should guide subsequent studies where not only long-term artificial aging and cyclic fatigue are performed to simulate the oral environment, but also clinical studies are conducted to report patient-centered outcomes, survival, and complications rates (biological, technical, and esthetic) of hybrid retention implant-supported prostheses.

5. Conclusion

Based on the present findings, the following conclusions were obtained:
1) Conventional resin cement associated with self-etch adhesive exhibited higher pullout retention of Y-TZP crowns cemented to Ti-base abutments than self-adhesive cement;
2) Ti-base surface sandblasting increased the retention of Y-TZP crowns relative to No pretreatment;
3) Autoclave sterilization maximized the bond strength of Y-TZP crowns cemented with self-adhesive resin cement, as well as, following no Ti-base surface pretreatment. No significant influence of autoclave was observed when used a conventional adhesive cement or Ti-base surface sandblasting.

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Conflict of Interest Statement

Authors declare no conflict of interest.

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