



Retentive strength evaluation of resin cements in two-piece zirconia abutments

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ABSTRACT

OBJECTIVE: To evaluate, *in vitro*, the retentive strength between the two-piece zirconia abutments, comparing different types of resin cements.

METHODS: Thirty zirconia parts manufactured by CAD-CAM technology were divided into three groups, according to the resin cement used for their cementation on the metallic titanium bases: dual-curing self-adhesive, dual-curing conventional and chemically activated. The specimens were stored in distilled water (24 hours, 37°C), followed by thermocycling and submitted to 30 s in baths of 5°C and 55°C, 2 s displacement time, with 6,000 cycles. The mechanical cycling tests were carried out with specimens submerge in distilled water (37°C), with load of 100N, 2 Hz frequency and 200,000 cycles. After thermomechanical tests, the specimens were submitted to a tensile test using a universal testing device until the complete separation of, which were analyzed in stereomicroscope, to determine the cementation failure pattern.

RESULTS: The adhesive-type cementation failure occurred in all specimens. The retentive strength values did not differ statistically between the groups.

CONCLUSION: All three tested resin cements provided enough retentive to the two-piece zirconia abutments.

Keywords: ceramics; luting agents; dental implants; tensile strength.

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Avaliação da resistência de união de cimentos resinosos em pilares de zircônia de duas peças

RESUMO

OBJETIVO: Avaliar, *in vitro*, a força de retenção entre os constituintes dos pilares de zircônia de duas peças, comparando diferentes cimentos resinosos.

METODOLOGIA: Trinta peças de zircônia confeccionadas por meio de CAD-CAM foram divididas em três grupos de acordo com os cimentos resinosos a serem utilizados para cimentá-las sobre bases metálicas de titânio: auto adesivo de dupla ativação; dupla ativação convencional e quimicamente ativado. As amostras foram armazenadas em água destilada (24 horas, 37°C), seguidas de ciclos térmicos submetidos a 30 s em banhos de 5°C e 55°C, com tempo de deslocamento de 2 s, totalizando 6.000 ciclos. Os ensaios mecânicos foram realizados na amostra submersa em água destilada (37°C), com carga de 100 N, frequência de 2 Hz e 200.000 ciclos. Após testes termomecânicos, os espécimes foram submetidos a tração usando máquina de ensaio universal até a separação completa das superfícies cimentadas, que foram analisadas em estereomicroscópio, para determinar o padrão de falha de cimentação.

RESULTADOS: A falha de cimentação do tipo adesiva ocorreu em toda amostra. Os valores de resistência à tração não diferiram estatisticamente entre os grupos.

Conclusão: Os três cimentos resinosos testados proporcionaram retenção eficiente aos pilares de zircônia de duas peças.

Palavras-chave: cerâmica; cimentos dentários; implantes dentários; resistência à tração.

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INTRODUCTION

The demand for more naturalness in oral rehabilitation promotes the constant emerging of new techniques and of new dental materials, aiming the improvement of teeth esthetics and of gingival tissue. The search for more esthetic delivery culminated with the introduction of zirconia ceramic pillars in the implant-supported rehabilitation [1].

The interest in ceramic materials is on increase, since these materials have adequate physical and mechanical properties, excellent esthetic and biocompatibility that make them suitable for dental rehabilitation. However, the success of ceramic restorations depends largely on the reliable bonding between ceramic and luting materials [2].

The hybrid zirconia pillars gather both esthetic and compatibility-beneficial properties promoted by the zirconia and favorable mechanical properties of metals [3, 4]. These characteristics allow the fabrication of implant-supported two-piece abutments rehabilitation with natural aspects and, at the same time, resistant. In prosthesis over implants, regardless of connection prosthetic type, zirconia pillars associated with a titanium base promote better mechanical behavior than single-based zirconia pillars [4, 5, 6].

To promote the retentive of the zirconia ceramic part with the titanium metallic base, which remains in intimate contact with the implant surface and with the fixing screw, the adequate election of the cementation agent is of utmost importance for the long-term clinical success [7, 8].

The establishing of a strong and lasting retentive between the dental ceramics and the substrates to be cemented is an important challenge faced by the clinical longevity of indirect ceramic restorations [9, 10].

Due to their crystalline content and lack of vitreous phase, the zirconia-based ceramics have a high superficial smooth surface, are acid-resistant, do not present mechanical micro-retentions on their surface after fluoridric acid application, and are not subject to surface chemical treatment with silane [7].

The interaction improvement between the resin cement and the zirconia is possible with the use of cementing agents that contain phosphate monomer molecules, which may be associated to previous chemical treatment with retentive agents containing this type of monomer [7].

The phosphate monomers are bi functional molecules with the ability to chemically bond to metallic oxides present in both zirconia bodies (zirconium oxide) and titanium bases (titanium oxides). Therefore, these monomers act on the surface of both two-piece abutments constituents and reinforce the retentive of these components with the resin cements [8, 11].

In a laboratorial study, the fracture resistance of three different types of zirconia pillars was tested and it was shown that the failure type was different for each evaluated group. In two of these groups, hybrid zirconia pillars were used, and the authors found that failure occurred on the cementation between the zirconia body and the titanium base [6].

With this stated, the present study aims to carry out an *in vitro* comparative analysis of the retentive strength promoted by different resin cements (dual-curing self-adhesive, conventional dual curing and chemically-activated) in two-piece zirconia abutments, with cone-morse type prosthetic connections, submitted to thermocycled aging.

METHODS

Completion of the zirconia body

For this study, 30 specimens were manufactured, each of them composed of a zirconia body cemented on a titanium metallic base (SMARTDENT, São Carlos, São Paulo, Brazil) and lastly, screwed on an indexed cone morse implant analog (CONEXÃO, Arujá, São Paulo, Brazil). For the fabrication of the zirconia bodies, the CAD/CAM technology was used, aiming to obtain standardized samples with adaptation precision with the metallic bases. With the assistance of the Zirkonzahn Modellier software (ZIRKONZAHN, Gais, Bousano, Italy), the mandibular molar anatomic-shaped zirconia pillar was projected (element 46) and the established cement thickness was of 30 μm . Then, with the Zirkonzahn M1Met milling machine ZIRKONZAHN, Gais, Bousano, Italy), the Zirkon Translucent 95H22 zirconia block milling (ZIRKONZAHN, Gais, Bousano, Italy) was used for the manufacturing of 30 zirconia bodies, followed by their sintering.

Completion of the metallic base

The titanium bases had vertical length of 4 mm (surface in contact with the zirconia) and semi-circular section, one of the faces was flattened, providing an anti-rotational characteristic to the ceramic body. Besides this, the surface external metallic bases were sandblasted with aluminum-oxide particles and presented horizontal elevations along their surface. The cone-morse implant analogs were included in PVC tubes (polyvinyl chloride) with the aid of a prosthetic parallelometer (PRO-DEL, São Paulo, São Paulo, Brazil), in a perpendicular and centralized fashion with the tube base and its interior. The PVC tube received sufficient chemically activated, colorless acrylic resin (JET, Campo Limpo, São Paulo, Brazil) to cover its inner volume, with the purpose to keep the analog in position after resin polymerization. The mobile parallelometer handle was lowered until the analog reached the acrylic resin level. The handle was then locked and kept in position until final material polymerization.

The thirty specimens were randomly distributed into three groups (n=10) according to the cement to be used: dual-curing/self-adhesive: RelyX U200 (3M ESPE, Seefeld, Bayern, Germany); conventional dual-curing: Panavia F 2.0 (KURAKAY, Okoyama, Okoyama, Japan); chemically activated: Multilink (IVOCLEAR-VIVADENT, Schaan, Liechtenstein). Before cementation, the internal and external surfaces of the 30 zirconia specimens were cleaned with cotton impregnated with alcohol at 70%, followed by ultrasonic tank agitation during 10 min. Afterwards, they were sprayed with distilled water and dried with oil-free/

moist-free air jets. Once the cleaning was concluded, the metallic interface was positioned and screwed (torque of 10N/mm) over an analog which was previously isolated with solid Vaseline with the purpose to facilitate the overflow cementation agent removal and to avoid cementation of the hybrid pillar to the analog. The screw access was protected with utility wax (TECHNEW, Rio de Janeiro, Rio de Janeiro, Brazil) so that overflowing of cement to the inner metallic interface and to the retention screw head did not occur.

Cementation of the zirconia body

The zirconia pillars were cemented on the ceramic bases by a single operator, according to manufacturer's instructions for each cement, following the chemical surface treatment protocol indicated for zirconia/metallic parts cementation.

For dual-curing, self-adhesive specimens cementation, the RelyX U200 resin cement, associated with the Singlebond Universal retentive agent (3M ESPE, Seefeld, Bayern, Germany) was used. It was applied to the Single Bond Universal for 20 s on each piece, in order to act as a primer for metal and zirconia and performing a chemical treatment of surfaces. For dual-curing conventional specimens' cementation, Panavia F 2.0 resin cement, associated with the Alloy Primer chemical agent (KURAKAY, Okoyama, Okoyama Japan) was used. It was applied to the metal Primer for five seconds in each piece, in order to carry out a chemical treatment and achieve a greater retention of the treated surface would Panavia F 2.0 cement. Lastly, chemically activated specimens were cemented with Multilink resin cement after chemical surface treatment with Metal/Zirconia Primer (IVOCLAR_VIVADENT, Schaan, Liechtenstein). Was applied to the Metal/Zirconia Primer for 180 s in each piece, in order to carry out a chemical treatment and achieve a greater retention of the treated surface would chemically with Multilink cement. After cementing agent's application, the two-piece abutments were submitted to a constant load of 5 kg, applied to the long axis of specimens during 10 min.

During this period, the specimens from dual-curing self-adhesive and dual curing conventional had their vestibular, lingual, mesial, distal, and occlusal faces polymerized during 40s, each face, with Optilight LD Max photopolimerizer (Gnatus, Ribeirão Preto, São Paulo, Brazil), with intensity of 400 mW/cm², previously measured with the assistance of a radiometer (RD 7, ECEL, Ribeirão Preto, São Paulo, Brazil).

Thermocycling test

After the cementation process, the specimens were stored in a sealed container with distilled water at 37°C for 24 hours. Then, they were submitted to thermocycling with proper equipment (ETHIK 521-6D, ETHIK TECHNOLOGY, Vargem Grande Paulista, São Paulo, Brazil) and submitted to 30 s baths at 5°C and 55°C, 2 s transitional time, with 6,000 cycles.

After thermocycling, the specimens were adapted and screwed on the analogs previously included in the PVC tubes. For this process, the PVC cylinders were stabilized

in a workbench vise and the screw torque was performed with a manual torquimeter (CONEXÃO, Arujá, São Paulo, Brazil) with torque of 20 N/mm for each specimen.

Mechanical cycling test

The mechanical cycling tests were carried out in a ER-11000 mechanical fatigue simulator (ERIOS, São Paulo, SP, Brazil) with specimens submerge in distilled water at 37°C, with load of 100 N, 2 Hz frequency and 200,000 cycles. For the mechanical assay, the studied cements – dual-curing self-adhesive, dual-curing conventional and chemically activated – were subdivided into three mixed subgroups. They were created with the purpose of promoting more homogeneity between the samples during mechanical cycling. Hence, factors such as cementation time and alterations in the assay device would not affect a group in isolation.

Check torque

After removing the specimens from the mechanical assay device, they were adapted in a workbench vise and the screws torque was verified with a manual torquimeter with the application of 20 N/mm.

Tensile strength

The tensile mechanical assays were performed with the universal testing machine EMIC DL-2000 (EMIC Equipamentos e Sistemas de Ensaio Ltda, São José dos Pinhais, Paraná, Brazil), and was set to print at the speed of 0.5 mm/min, with a load cell of 500 kgf. The two-piece abutments were submitted to an axial tensile strength, perpendicular to their long axis, until retentive of their constituents was broken. The bond strength values were provided by the EMIC's computer and were reported in Newton (N), for each specimen. During the execution of tensile tests, none of the specimens presented catastrophic failure and all of them presented cementation failure in this study's area of interest.

Failure analysis

After complete separation of the two parts of the two-piece abutments, all specimens were analyzed with the stereomicroscope (Discovery V20, Carl-Zeiss, Oberkochen, Göttingen, Germany) for zirconia/resin cement/titanium interface failure pattern determination.

Statistical analysis

The obtained tensile strength values were compared between studied cements: dual-curing self-adhesive, dual-curing conventional and chemically activated with a variance analysis (ANOVA), to the level of 5% significance. Before this analysis, data were verified from a normality and homoscedasticity perspective, with the application of Shapiro-Wilk and Bartlett tests, respectively. It was verified that the data presented normal distribution (FAILURE CEMENTATION=0.90, p=0.22) and homogeneous variance (Bartlett's K-squared=1.26, GL=2, p=0.53). All analyses were performed using free software (R, R Core Team, 2013).

RESULTS

The tensile strength values registered for cements dual-curing self-adhesive, dual-curing conventional and chemically activated were not statistically different ($F=0.29$, $p=0.745$), and the mean values for dual-curing self-adhesive were 354.019 N; for dual-curing conventional, 382.335 N; and chemically activated, 372.048 N (**Figure 1**). The adhesive-type pattern of cementation failure between the resin cement and the zirconia surface occurred in all analyzed specimens (**Figure 2**).

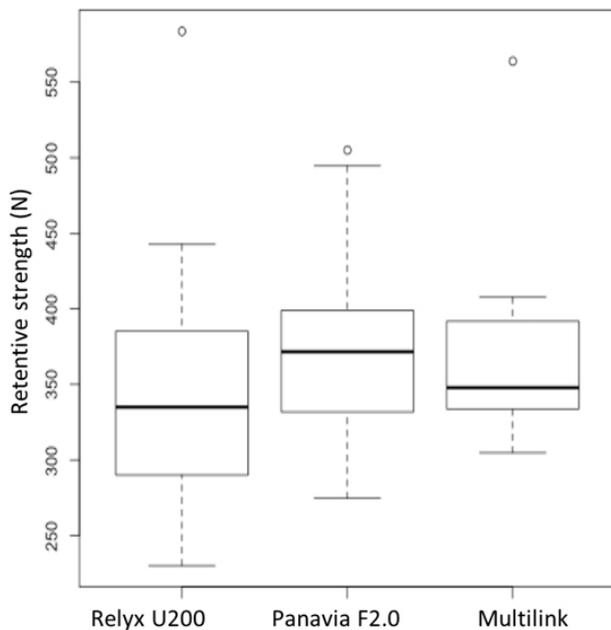


Figure 1. Retentive strength promoted by resin cements Relyx U200, Panavia F 2.0 e Multilink.

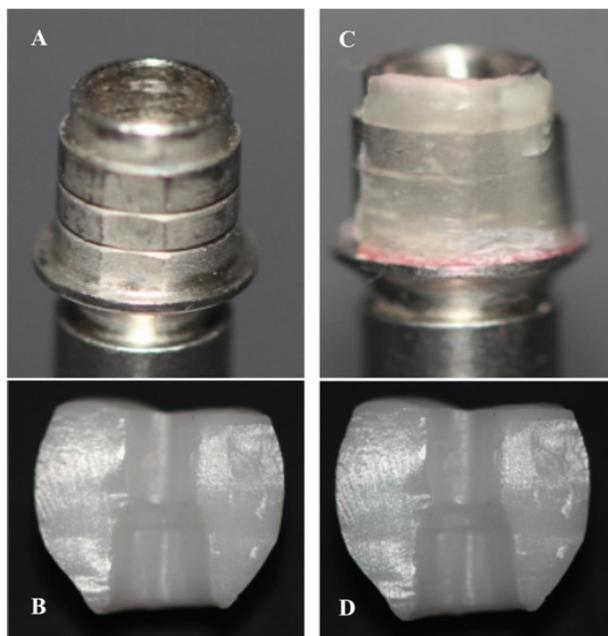


Figure 2. Surfaces before cementation (A-B) and after traction test (C-D).

DISCUSSION

The present study was developed based in the existence of a few scientific studies that relate resin cements and the retentive strength of hybrid zirconia pillars. This study demonstrated that there are no statistically significant differences between the retentive strength promoted by the three resin cement types used in the cementation of the hybrid zirconia pillars. This study is similar to another in which the retentive strength promoted by resin cements in CAD-CAM-made zirconia two-piece abutments [8]. Although the self-adhesive resin cements presented higher water absorption and high solubility when compared to conventional resin cements, so self-adhesive resin cements are more likely to suffer hydrolytic degradation [12, 13], union values were similar. It should be noted that, despite statistically similar values between the different groups tested, dual-cure cements them, had higher high standard deviation (A-Mean \pm standard deviation; B-Mean \pm standard deviation) compared to chemical polymerization cement (C-Mean \pm standard deviation). This may be related to the conversion of monomers, which may have been influenced by virtue of the thickness of the ceramic piece. In the present study, it was decided to use the chemical surface treatment in the inner part of the zirconia specimens, as this method is conservative and has proved efficiency, which means it does not promote structural damage to the zirconia specimens.

Some studies have demonstrated that the mechanical surface treatment with the sandblasting of aluminum-oxide particles, associated to the use of a retentive agent containing phosphate monomer molecules are efficient methods for the promotion of a strong and lasting bond between resin cements and zirconia [11]. However, other studies have also demonstrated that the mechanical surface treatment with the sandblasting of aluminum-oxide particles may cause micro cracklings and induce the zirconia ceramic phase transformation, causing a considerable decrease in the mechanical resistance of this material [14, 15].

The resin cements and/or retentive agents utilized in this study contained phosphate monomer molecules in their chemical composition. They have the ability to make stable chemical connections with metallic oxides, present in both the zirconia surface and the titanium metallic base. The effectiveness of this type of monomer in the resistance increase and retentive durability between resin cements and zirconia was documented in literature [7, 16]. However, there is some disagreement as of its long-term effectiveness. Some authors describe a lasting bond, whereas others state an early degradation of the retentive interface [7].

The specimens aging in laboratorial tests through the simulation of buccal cavity conditions is of utmost importance for the result of a research project [11].

When the artificial aging of specimens is not conducted in laboratorial assays, the clinical predictability becomes limited. The aging performed with water storage, thermocycling, or a combination of both, are the most common method to simulate the buccal cavity conditions. The

specimens' temperature variation during the thermocycling produces a thermal stress onto the cementing agent and cemented substrates due to expansion and contraction, causing retentive failure [17]. Despite thermocycling seems to be a trustworthy aging method, contradicting results have been reported. Although some studies have demonstrated a decrease in the retentive strength after thermocycling [18], other studies showed no effect or an increase in retentive strength [19]. Besides this, a recent study reported that a standard thermocycling protocol determination that compares laboratorial research is flawed [20].

The strength generated during mastication may be simulated by mechanical cycling, which tends to be closer to the physiological conditions of a buccal cavity. Some studies reported that 1,200,000 cycles, with load of 50N simulate five years of clinical material use [21,22]. However, other studies affirm that there is no exact correlation between the number of cycles in laboratorial research and the clinical conditions [23,24].

The retentive strength evaluation between zirconia and resin cement using tensile resistance tests may be verified in literature [11,24]. For the tensile test to present trustworthy results, it is important that the long axis of specimens are aligned and coincident with the central line of force application by the assay machine, according to the uniaxial force application concept. Based in this principle, in this study, a prosthetic parallelometer was used to position the specimens, perpendicular to the support base, enabling the long axis of specimens to be aligned to the load application axis by the tensile assay device. In this study, it was verified that all stereomicroscope-analyzed specimens presented an adhesive-type failure pattern between the zirconia surface and the resin cements after chemical surface treatment by thermomechanical cycling. The same type of failure was found in another study, which evaluated the retentive of zirconia two-piece abutments constituents with resin cements after mechanical surface treatment with sandblasting of aluminum-oxide particles and thermocycling aging [8]. Despite the different surface treatment types, results demonstrated that the retentive failure type on both studies were of adhesive type, between the researched zirconia and the resin cements. The limitations of this *in vitro* study shall be overcome with well-defined *in vivo* studies and with long term follow-up. Laboratorial studies results may not be directly extrapolated to the clinic, as they may be clinically different. The retentive strength between resin cements and the zirconia two-piece abutments shall be clinically followed-up on a long-term basis with the objective of verifying the clinical performance of cementation agents evaluated in this study.

CONCLUSION

As a result of the applied methodology and obtained data, it can be concluded that all three tested resin cements resisted to the proposed aging tests provided enough retentive to the two-piece zirconia abutments, regardless of activation

type and adhesive characteristics of the evaluated cements. Failure mode of all tested specimens were completely adhesive, leaving the detached zirconia coping and titanium insert undamaged.

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