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Influence of Surface Treatment and Resin Cements on the Bond Strength between the Y-TZP Zirconia and Composite Resin Interface

Influência do Tratamento de Superfície e dos Cimentos Resinosos na Resistência de União Entre a Interface Zircônia Y-TZP e Resina Composta

Lucas Campagnaro Maciel*[a]; Amanda Pádua Proeza[b]; Hélyda Coelho Guimarães Balbino[b]; Marcela Morão Corteletti[b]; Ricardo Huver de Jesus[c]; Laís Regiane da Silva Concílio[a]

[a]University of Taubaté, Stricto Sensu Graduate Program in Dentistry. SP, Brazil.
[b]Upper Education School São Francisco de Assis, Dentistry Course. ES, Brazil.
[c]Dentistry College São Leopoldo Mandic,Stricto Sensu Program in Clinical Dentistry. SP, Brazil.
*E-mail: lucascmaciel@hotmail.com
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Abstract

The evolution of dental materials and the improvement of ceramic systems stimulated the increased use of Y-TZP zirconia-based ceramics. Despite the excellent mechanical performance, this material has low adhesion potential. The objective of this work was to evaluate the surface treatments and resin cements influence on bond strength between Y-TZP zirconia and composite resin interface. A total of 60 blocks of Y-TZP zirconia (3x8x8mm) were prepared and divided into 3 groups according to the surface treatments: (C) control - extra fine diamond bur, (J) sandblasting with Al_2O_3 and (JP) sandblasting with Al_2O_3 + ceramic primer. Each group was subdivided into two groups according to type of resin cement used for cementing composite resin discs (2mm thick x 5mm diameter): self-adhesive and conventional (n=10). The samples were stored in distilled water for 24 hours at 37±1°C in a incubator and subsequently submitted to the shear bond test to determine the bond strength (RU). There was no significant difference in RU among the surface treatments when using conventional resin cement. For the self-adhesive resin cement, Al_2O_3 blasting and Al2O3 + primer blasting increased the RU but did not present significant differences between them ($p<0.05$). Comparing the cements, it was observed that regardless of the surface treatment, the highest values were for the self-adhesive resin cement ($p<0.05$). Application of the primer after blasting with Al_2O_3 did not increase RU.

Keywords: Dental Prosthesis. Ceramics. Dental cements. Shear Strength.

Resumo

A evolução dos materiais odontológicos e o aprimoramento dos sistemas cerâmicos impulsionaram o aumento da utilização da cerâmica a base de zircônia Y-TZP. Apesar do excelente desempenho mecânico, este material apresenta baixo potencial de adesão. O objetivo deste trabalho foi avaliar a influência dos tratamentos de superfícies e dos cimentos resinosos na resistência de união entre a interface zircônia Y-TZP e resina composta. Foram confeccionados 60 blocos de zircônia Y-TZP (3x8x8 mm) e divididos em 03 grupos de acordo com os tratamentos de superfícies que receberam: (C) controle - ponta diamantada extrafina, (J) jateamento com Al_2O_3 *e (JP) jateamento com* Al_2O_3 *+ primer cerâmico. Cada grupo foi subdividido em dois novos grupos de acordo com tipo de cimento resinoso utilizado para cimentação de discos de resina composta (2mm de espessura x 5mm de diâmetro): autoadesivo e resinoso convencional (n=10). As amostras foram armazenadas em* **água** *destilada por 24 horas a 37±1°C em estufa e posteriormente submetidas ao teste de cisalhamento para averiguar a resistência de união (RU). Não houve diferença significativa na RU entre os tratamentos de superfície quando utilizado o cimento resinoso convencional. Para o cimento resinoso autoadesivo o jateamento com* Al_2O_3 *e o jateamento de* Al_2O_3 *+ primer aumentaram a RU porém não apresentaram diferenças significativas entre si (p<0,05). Comparando os cimentos observou-se que, independente do tratamento de superfície, os maiores valores foram para o cimento resinoso autoadesivo (p<0,05). A aplicação do primer após o jateamento com* Al_2O_3 *não proporcionou aumento da RU.*

Palavras-chave: *Prótese Dentária. Cerâmica. Cimentos Dentários. Resistência ao Cisalhamento.*

1 Introduction

The increase in demand for aesthetic rehabilitation has boosted the development of restorative materials that are able to meet this need. In parallel, the dental ceramics have been the object of various studies due to the constant evolution of this material as well as the increase of its application[1]. Characteristics and limitations of the various ceramic systems available in the market predict the indications and limitations of its use dependent on the clinical need[2,3].

Due to being able to reproduce the optical characteristics of the dental element and characteristics such as resistance to compression, thermal conductivity, chemical stability, radiopacity, marginal integrity and biocompatibility, ceramics became the main material in cosmetic restorative dentistry[2,4-8].

Due to the advancements in technology CAD/CAM (*Computer Assisted Design/Computer Assisted Machining*) and the improvement of ceramic materials as strong as capable of mimetizing dental characteristics, the prostheses based on polycrystalline ceramics, as for example the Y-TZP zirconia (tetragonal zirconia partly stabilized by yttrium), has become an excellent alternative to metallo-ceramics dentures[9].

Despite its mechanical performance (tenacity to fracture, resistance to flexion, behavior under mechanical fatigue) being superior to other ceramics, the absence or limited presence of glass in the Y-TZP zirconia makes it an acid-resistant ceramic to hydrofluoric acid and present as one of the main drawbacks, a low potential for adherence to the resin cements. Thus, the traditional treatment of its surface with hydrofluoric acid to 10% from 30 to 60 seconds followed by the application of silane becomes ineffective, preventing the accession process by means of adhesive cementation, when adopted such a strategy[10-12].

Even though the use of Y-TZP zirconia in dentistry is already consolidated , there is still no consensus on the ideal protocol for its cementation. Several have been the surface treatments described in the literature and among them are the mechanical treatment by means of sandblasting of the workpiece with aluminum oxide particles, promoting micro porosity responsible for micromechanical retentions, and the chemical treatment through the use of *specific primers* for zirconia responsible for the formation of chemical bonds among the cement, ceramics and the dental substrate[6,12-17].

In this context, it is important to emphasize that one of the clinical problems commonly associated with the use of the Y-TZP zirconia is related to loosening (adhesive) of the prosthetic piece[18-21]. And, due to the fact of dentistry experiencing a natural evolution at the beginning of this new century, offering a wide variety of techniques and materials, it becomes important the implementation of effective rehabilitation, with scientifically proven results, providing financial savings and optimization of time for both the professional and the patient.

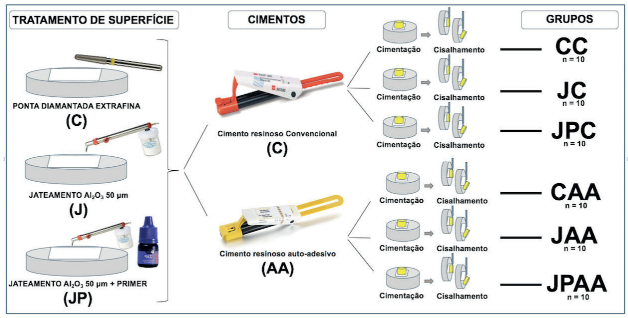
Thus, the objective of this work was to evaluate the surface treatments and resin cements influence on bond strength between Y-TZP zirconia and composite resin interface. The null tested hypotheses were the different treatments of surfaces will not influence the adhesive strength of Y-TZP zirconia interface/composite resin; and the conventional adhesive resin cements used in this study will provide the same adhesive resistance before different surface treatments of Y-TZP zirconia.

2 Material and Methods

2.1 Design and experimental groups

After fabricated and included in acrylic resin, 60 blocks of Y-TZP zirconia (Prettau Zirkon , Zirkonzahn GmbH, Bruneck, Italy) were randomly divided into three groups (20 blocks each) in accordance with the surface treatment that they received: (C - control) - extra fine diamond tip of 30 µm (Diamond tip, 4138 FF, KG Sorensen, Barueri, SP, Brazil), (J) Sandblasting with Al_2O_3 50 µm (Bio-Art Dental Equipment Ltda, São Carlos, SP, Brazil) and (JP) sandblasting with Al_2O_3 50 µm followed by the application of *ceramic primer* (*Primer* Yzap, Yller, Pelotas, Rio Grande do Sul, Brazil). Then, each group was again divided in accordance with the cement used (n=10): conventional resin (Rely X ARC, 3M ESPE, St Paul, MN, Unites Stated of America) and self-adhesive (Rely X U200, 3M ESPE, St Paul, MN, United State of America). Composite resin discs (Filtek Z350 XT, 3M ESPE, St Paul, MN, United States of America) were cemented on the treated ceramic surface and then, samples were subjected to the shear. The research flowchart and the materials used in this study are described, respectively, in Figure 1 and Table 1.

Figure 1 - Flowchart of the study



Source: Research data.

Table 1 - Materials used in the study

Materials	Composition	Trademark
Ceramics	Zirconia Y-TZP based ceramic: Zirconium Oxide 97% Yttrium oxide 3%	Zirkon Prettau, Zirkonzahn GmbH, Bruneck, Italy
Conventional resin cement	Paste A: 68% of particles of load (zirconia/silica), pigments, amine and photo initiating system. Paste B: 67% of particles of load, benzoyl peroxide.	Rely X ARC, 3M ESPE, St. Paul, MN, United States of America
Self-adhesive resin cement	Base Folder: Phosphatic methacrylate monomers, methacrylate monomers, marked loads, initiating components, stabilizers, reactive additives Catalyzing paste: Methacrylate monomers, fillers, alkaline (basic), marked, Initiating Components, stabilizers, Additives reactive pigments	Rely X U200, 3M ESPE, St. Paul, MN, United States of America
composite Resins.	Bis-GMA, UDMA, TEGDMA, PEGDMA, Bis-EMA Particles: silica/ zirconia, photo-initiator	Filtek Z350XT, 3M ESPE, St Paul, United States
Extra fine diamond tip	Diamond tip coated with granules of diamond of 30 µm	KG Sorensen, São Paulo, SP, Brazil
Aluminum oxide Sandblasting	Particles of aluminum oxide (Al_2O_3) of 50 µm sandblasted through a micro-jet	Bio-Art Equip Odon-tol Ltda, São Carlos, São Paulo, Brazil
Silane	Pre-hydrolyzed silane	*Primer* Yzap, Yller, Pelotas, Rio Grande do Sul, Brazil

Source: Research data.

2.2 Fabrication and inclusion of ceramic blocks

Sixty blocks that are pre-synthetized of Y-TZP zirconia (Zircon Prettau , Zirkonzahn GmbH, Bruneck, Italy) of 3x8x8 mm (thickness, length and width) were prepared using a diamond double face disc (KG Sorensen, Barueri, São Paulo, Brazil), mounted on a straight part associated with a micro-motor (Kavo, Joinville, Santa Catarina, Brazil). Before being sintered, adhesive surfaces of these blocks were settled with sandpaper of fine-grained silicon carbide (#600 - Norton, Guarulhos, São Paulo, Brazil). Then the blocks were sintered in a furnace-specific (Oven Zirkonofen 600, Zirkonzahn, Bruneck, Italy) according to the manufacturer's instructions.

For inclusion, the ceramic blocks were positioned on a blade of utility wax (Lysanda Dental Products, São Paulo, SP, Brazil), and then a cylindrical array of PVC (Tigre tubos e conexões, Rio Claro, São Paulo, Brazil) of 20mm in diameter and 15 mm height was adapted around each ceramic block. An acrylic self-polymerizable resin (Jet – Clássico Artigos Odontológicos, São Paulo, SP, Brazil) was proportionated and handled in accordance with the manufacturer's instructions, and, in its plastic phase, poured into inside of the matrix of PVC.

2.3 Preparation of composite resin discs

Through a pre-fabricated array, 60 composite resin discs (Filtek Z350 XT - 3M ESPE, St. Paul, Minessota,United States of America) of 5 mm in diameter by 2 mm thickness were manufactured. The matrix was placed on a tape of polyester (Maquira Dental Products, Maringá, Paraná State, Brazil) and then with the help of a spatula of insertion no. 1 (Golgran, São Caetano do Sul, São Paulo, Brazil) a single increment of resin was inserted in its interior. In order to standardize the compaction of the increment, elimination of excesses and smoothness of the disc surface, a second strip of polyester was interposed on the resin and on it, a glass plate was placed and maintained under pressure of 1kg for a minute. The photo-polymerizer (Bluephase N, Ivoclar Vivadent, Schaan, Liechtenstein, Austria) was positioned perpendicular to the increment of the composite resin for activation by twenty seconds. After finishing this step, the disks were removed from the array cleaned in ultrasonic cleaner for 2 minutes (Cristófoli Equipamentos de Biosseguraça Ltda, Campo Mourão, Paraná, Brasil) and stored in plastic containers.

2.4 Surface treatments of y-tzp zirconia

The Y-TZP zirconia based ceramic blocks received three different surface treatments before being cemented and the composite resin discs: (C) extra fine diamond tip, (J) blasting with aluminum oxide particles and (JP) blasting with aluminum oxide particles followed by the application of the *primer* ceramic.

2.5 Extra fine diamond tip

Through extra fine diamond tips of 30 μm of granulation (diamond tip 4138, FF, KG Sorensen, Barueri, SP, Brazil) coupled in high speed motor (Kavo, Joinville, Santa Catarina, Brazil), the ceramic blocks of these groups had their surfaces adjusted. Manual multi-directional repetitive movements were executed for 10 seconds on the surface for adhesive interface at an approximate speed of 300,000 rpm. Every 5 Samples treated, a new diamond tip was used. The blocks were cleaned in ultrasonic cleaner for 2 minutes (Cristófoli Equipamentos de Biosseguraça Ltda, Campo Mourão, Paraná State, Brazil) and were ready to receive the cements.

2.6 Blasting with aluminum oxide

Aluminum oxide particles with a diameter of 50μm (Bio-Art Equip Odontol Ltda, São Carlos, São Paulo, Brazil) were sandblasted perpendicular to the adhesive surface of the ceramic blocks with a pressure of 70 psi at a distance of 10 mm, for 10 seconds. The blocks were also cleaned in ultrasonic cleaner for 2 minutes.

2.7 Blasting with aluminum oxide followed by the application of ceramic primer

After sandblasting with aluminum oxide particles with a diameter of 50μm, as already described, the *primer* was applied (single bottle) to zirconia (*Primer* YZap, Yller, Pelotas, Rio Grande do Sul, Brazil) in the adhesive surface of the ceramic block. Through a disposable applicator (Microbrush, KG Sorensen, Cotia, São Paulo, Brazil), the *primer* was deposited on the surface previously sandblasted and cleaned and left to act for 3 minutes followed by air blasts for 10 s, according to the manufacturer's instructions.

2.8 Cementation of composite resin discs

Before being cemented, the disks of the composite resin received the following sequence of treatment of their adhesive surface: prophylaxis with brush Robinson and pumice stone-based paste (Polidental Ind. Com. Ltda., Cotia, São Paulo, Brazil) for 15 seconds, then washed with tap water for 15 seconds and drying with air blasts.

For the cementing process, the conventional resin cement RelyX ARC (3M ESPE) and the self-adhesive resin cement RelyX U200 (3M ESPE) were provided and handled according to the manufacturer's instructions and, with the aid of a spatula number 1 (Golgran, São Caetano do Sul, São Paulo, Brazil), deposited on the surface of the ceramic blocks previously treated. Then, the disk of the composite resin was positioned and digitally pressed on the ceramic surface filled resin cement. The set was taken to a delineator (Bio-Art Equipamentos Odontológicos, São Carlos, São Paulo, Brasil) in which a device was adapted with weight corresponding to 3 Kg of pressure. The excesses of cement were removed with the aid of a paintbrush micro brush (KG Sorensen, Cotia, São Paulo, Brazil) and the cement agent was cured (Bluephase

N, Ivoclar Vivadent, Schaan, Liechtenstein, Austria) in four different regions for 20 seconds each.

2.9 Storage of samples

After cementing the sets of ceramic blocks/composite resin discs were packaged in a plastic container immersed in distilled water and stored in an oven ((Quimis Científica Equipamentos Ltda, Diadema, São Paulo, Brazil) for 24 hours with controlled temperature of 37±1 ^oC.

2.10 Bond strength test

The bond strength test (RU) was carried out in a universal testing machine (Shimadzu AG-X, Shimadzu Corporation, Kyoto, Japan) with a load cell corresponding to 5KN. The samples were positioned and fastened in a metallic device located in the lower part of the machine, so that the line of cementation remained in a vertical position for application of the load (shear). The force was applied by the metal tip attached to the upper part of the machine (chisel) with a constant speed of 0.5 mm/minute until the collapse of the bond. The values needed to resistance (bond strength) were recorded in Megapascal (MPa) by software (Trapezium-X Software, Shimadzu Corporation, Kyoto, Japan) dedicated to the assay machine. All samples were prepared and tested by the same researcher, previously calibrated.

After tabulated, the results were statistically analyzed with the aid of the software Bioestat 5.0 (Bioestat, Maringá, Paraná, Brazil). The komolgorov-Smirnov tests were performed for normality analysis and then applied the ANOVA 1 Factor and t test for comparative analysis of the effect of different surface treatments and the student t test for comparison of two different cements when subjected to the same surface treatments, adopted for the comparisons ($p<0.05$.

3 Results and Discussion

Table 2 shows the values of bond strength (RU) to zirconia provided by resin cements in association with different surface treatments. There was no significant difference in RU among the surface treatments when using conventional resin cement. However, for the self-adhesive resin cement it was possible to observed that the blasting with Al_2O_3 (JAA) and the blasting of Al_2O_3 followed by the application of the primer (JPAA) significantly increased the RU ($p<0.05$).

Table 2 - Mean and standard deviation of bond strength (MPa).

Surface Treatment	Resin Cement	Group	Bond strength
Diamond tip (control)		CC	4.73 (2.35)^A
Blasting Al_2O_3	Conventional	JC	6.82 (1.68)^A
Blasting Al_2O_3 + Primer		JPC	6.06 (1.86)^A
Diamond tip (control)		CAA	8.88 (3.61)^A
Blasting Al_2O_3	Self-adhesive	JAA	13.66 (3.01)^B
Blasting Al_2O_3 + Primer		JPAA	13.77 (3.71)^B

ANOVA1 Factor and t test ($p<0.05$).

Source: Research data.

Comparing the cement agents, it was observed that regardless of the surface treatment, the highest values were found when the for the self-adhesive resin cement ($p<0.05$) was used.

Table 3 - Comparison of bond strength between the zirconia and the different resin cements, subjected to the same surface treatments.

Group	Bond strength (MPa)
CC	4.73 (2.35)^A
CAA	8.88 (3.61)^B
JC	6.82 (1.68)^A
JAA	13.66 (3.01)^B
JPC	6.06 (1.86)^A
JPAA	13.77 (3.71)^B

Student t test ($p<0.05$.)

Source: Research data.

The zirconias Y-TZP are known as polycrystalline ceramics of high mechanical resistance, presenting little or no glassy phase. Thus, much has been discussed regarding the surface treatments, its reliability and stability of accession, since the lack of vitreous content in the composition of zirconia has as a disadvantage the low potential for adherence to the resin cements[22].

Within this context, the present study evaluated the influence of different surface treatments associated with the two resin cements. According to the results, it was possible to observe that both null hypotheses tested in this research were rejected. Despite not having been observed statistically significant difference among the treatments of surface when used conventional resin cement ($p<0.05$), both the blasting with Al_2O_3(13.66 +/- 3.01) as well as the combination of sandblasting with Al_2O_3 followed by the application of *ceramic primer* (13.77 +/- 3.71) showed higher bond strength resistance values (RU) than the extra fine diamond tip (8.88 +/- 3.61) when associated with the adhesive resin cement ($p<0.05$). In addition, when compared with the performance of the different cements, regardless of the surface treatment used, the highest values of RU were observed for the self-adhesive resin cement.

Various types of surface treatments are used with the aim of increasing considerably the adhesive capacity between the zirconia Y-TZP based ceramic and the restorative substrates. Mechanical treatments stand out by offering increase of the area of inner surface of the restorations, establishing a porous /erratic surface and free from impurities favoring the mechanic imbrication of cement on the piece. Whereas the chemical bonds allow among the structures, by means of the bifunctional molecules in which one of its parties join the metallic oxides of zirconium and the other to the resin matrix of the cements[6.23]. In this study, the efficacy of blasting with aluminum oxide particles (mechanical treatment) and a *ceramic primer* (chemical treatment) were investigated.

Sun et al.[24] describe that the mechanical treatment of zirconia surface through the blasting with aluminum oxide particles provides higher values of bond strength when compared to non-treated surface. However, in the present study, despite this fact has also been observed for samples cemented with the cement adhesive the blasting with Al_2O_3 did not provide higher strength bond values when associated with conventional resin cement, this fact is also observed in the work of Re et al.[25]. The discrepancy among the results may be related to different compositions of ceramic and cement agent systems used in studies. As described by Blatz et al.[26] it is worth emphasizing that it is important to consider that the adhesive performance is related not only to the treatments of surfaces of Y-TZP zirconia-based ceramics but also the proper selection of the resin cement.

Dentistry has experienced a constant evolution of materials and in this context arise the *ceramic primers* as a promising alternative in terms of improving the adhesion between the resin cements and polycrystalline ceramics such as the zirconias Y-TZP.

Yun et al.[27], Amaral et al.[28] and Lee et al.[29] describe that the combination of the application of *ceramic primers* after the blasting of the surface of the zirconia with particles of Al_2O_3 offer the best bond strength values when compared to the blasting with particles of Al_2O_3 alone. They suggest that this strategy is as an excellent option to increase the bond strength of Y-TZP zirconia interface/resin cement, but they consider the importance of conducting more research to better grounding. Different from the results mentioned, in this study, the application of *ceramic primer* after the particle blasting with Al_2O did not prove be more efficient than when applied to only the blasting as surface treatment (Table 2).

Although the cementation of Y-TZP zirconia based prosthetic parts through conventional cements (glass ionomer cement and zinc phosphate) ensures good mechanical retention, the risk of failure must be considered, due to the fact that such cements have relatively high solubility in oral environment. Therefore, due to providing better sealing/ adaptation marginal and consequently a lower risk of micro infiltration, greater retention and resistance to fracture of the prosthesis and the tooth, it is preferable the use of cements resin through the adhesive cementation technique[30].

In order to simplify the process of accession to the structures, minimize the occurrence of failures and optimize the clinical time, the self-adhesive resin cements emerged [31]. However, the evolution of resin cements may also be observed in terms of their composition, where the addition of phosphate monomers (10-MDP - metacriloxidecil 10-di-hydrogen phosphate) to its array, enable the chemical bond with metallic oxides, such as zirconium oxide, enabling its use for cementation of Y-TZP zirconia[32] based ceramic restorations .

In the present study, regardless of surface treatment proposed, the largest bond strength values were observed for the cemented samples with the self-adhesive cement(Rely X U200) (Table 3). Bearing in mind that this cement contains in its composition the monomer 10-MDP, and, despite De Souza et al.[33] describe that the Mdp based cements do not provide a significant increase in bond strength to the zirconia, the results show consistent convergence to several studies that also demonstrate the effectiveness of this monomer when it comes to accession to the TZP[26,29,34]zirconia based ceramics

However, due to the limitations of this research, it is important to conduct more laboratory studies and clinical trials that could clarify the best combination of pre-cementation strategy for zirconia Y-TZP based ceramics enabling comparison to existing studies as well as the consolidation of concepts currently stated. It is worth noting also that this research is an independent production and that there is no conflict of public or private interests.

4 Conclusion

Through the proposed methodology and based on the results it was concluded that when used the self-adhesive resin cement that contains 10 monomer-MDP in its composition, the proposed surface treatments influenced the bond strength of the interface Y-TZP zirconia/composite resin. The strategy of the application of *primer* after the ceramic aluminum oxide blasting did not provide increased bond strength. Regardless the type of surface treatment proposed in this study, the bond strength was always higher when using the adhesive resin cement that contains 10 monomer-MDP.

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