

ISSN: 2178-7514

Vol. 16| Nº. 1| Ano 2024

ARTIGO ORIGINAL

EVALUATION OF BOND STRENGTH BETWEEN DIFFERENT SYSTEMS FOR ADHESIVE CEMENTATION TO FELDSPATHIC PORCELAIN AND ZIRCONIUM OXIDE

Avaliação da resistência de união entre diferentes sistemas para cimentação adesiva à porcelana feldspática e ao óxido de zircônio

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RESUMO

Introduction: New systems for adhesive cementation have been increasingly developed and used in daily clinical practice for the cementation of ceramic prostheses. Differences in the chemical composition of various system components can influence the bond strength between the resin cement and the ceramic. Objective: This study aims to evaluate the bond strength between two ceramic systems—feldspathic porcelain and yttria-stabilized zirconium oxide—and three different systems for adhesive cementation: Rely X ARC, Multilink System, and Rely X UCem. Methodology: Thirty discs of Noritake feldspathic porcelain and thirty discs of zirconium oxide ceramic were fabricated, each measuring 7.5 mm in diameter by 3.0 mm in thickness, according to the manufacturers' instructions. The discs were polished on a mechanical polisher with water sandpaper ranging from 320 to 1500 grit and embedded in ½ inch PVC tubes with self-curing acrylic resin. The feldspathic porcelain discs were conditioned with 10% hydrofluoric acid for 120 seconds, and the zirconium oxide discs were sandblasted with 40 psi alumina for 10 seconds at 10 mm. All discs were then washed and placed in an ultrasonic bath with distilled water for 10 minutes. The cementation systems were applied according to the manufacturers' instructions (n=10) on an adhesion area of 5.0 mm in diameter, delimited by a tripartite metal matrix. The samples underwent 1,000 thermal cycles ranging from 5°C to 55°C. Shear bond strength tests were performed on an EMIC DL2000 testing machine with a 20 kN load cell and a speed of 0.5 mm/ min. The mean values of the groups were as follows (in MPa): Feldspathic with Rely X ARC (G1) 6.21; Feldspathic with Multilink (G2) 8.98; Feldspathic with Rely X UCem (G3) 8.15; Zirconium Oxide with Rely X ARC (G4) 0.53; Zirconium Oxide with Multilink (G5) 9.25; Zirconium Oxide with Rely X UCem (G6) 4.37. Data were subjected to one-way ANOVA and Tukey tests. Results: There was no significant difference among the feldspathic porcelain groups. For zirconium oxide, Multilink showed higher bond strength values. Conclusion: The bond strength between the cements and feldspathic porcelain exceeded the intrinsic strength of this material, and Multilink showed the highest bond strength values regardless of the ceramic material.

Palavras-chave: Dental Porcelain; Resin Cements; Zirconium; Shear Strength.

ABSTRACT

Introdução: Novos sistemas para cimentação adesiva têm sido desenvolvidos e utilizados cada vez mais na clínica diária para cimentação de próteses cerâmicas. Diferenças na composição química de vários componentes do sistema podem influenciar na resistência de união entre o cimento resinoso e a cerâmica. Objetivo: O presente trabalho tem por objetivo avaliar a resistência de união entre dois sistemas cerâmicos, uma porcelana feldspática e uma cerâmica de óxido de zircônio estabilizada por ítrio e três diferentes sistemas para cimentação adesiva, o Rely X ARC, o Sistema Multilink e o Rely X UCem. Metodologia: Foram confeccionadas 30 pastilhas de porcelana feldspática Noritake e 30 de cerâmica de óxido de zircônio, medindo 7,5 mm de diâmetro x 3,0 mm de espessura, confeccionadas de acordo com as instruções dos fabricantes. As pastilhas foram polidas em Politriz mecânica com lixa d`água da granulação 320 até 1500 e incluídas em tubos de PVC de ½ polegada com resina acrílica autopolimerizável. As pastilhas de porcelana feldspática foram condicionadas com ácido fluorídrico 10% por 120 segundos, as pastilhas de óxido de zircônio foram jateadas com óxido de alumínio 40 psi por 10 segundos a 10 mm e então, todas foram lavadas e colocadas em cuba ultrasônica com água destilada por 10 minutos. Os sistemas de cimentação foram aplicados de acordo com instruções dos fabricantes (n=10), em área de adesão de 5,0 mm de diâmetro delimitada por matriz metálica tripartida. As amostras foram submetidas à 1.000 ciclos térmicos de 5°C a 55°C. Os ensaios de resistência ao cisalhamento foram realizados em máquina de ensaios EMIC DL2000, com célula de carga de 20 kN e velocidade de 0,5 mm/min. As médias dos grupos foram as seguintes (em MPa) Feldspática com Rely X ARC (G1) 6,21; Feldspática com Multilink
(G2) 8,98; Feldspática com Rely X UCem (G3) 8,15; Óxido de Zircônio com Rely X ARC (G4) 0,53; Óxido de
Zircônio ANOVA um critério e Tukey. Resultados: Não houve diferença significativa entre os grupos de porcelana feldspática. Para o óxido de zircônio, o multilink apresentou maiores valores de resistência de união. Conclusão: A resistência de união entre os cimentos e a porcelana feldspática superou a resistência intrínseca deste material e que o Multilink de união entre os cimentos e a porcelana feldspática superou a resistência intrínseca deste material e que o Multilink
apresentou os maiores valores de resistência de união, independentemente do material cerâmico.

Keywords: Porcelana Dentária; Cimentos de Resina; Zircônio; Resistência ao Cisalhamento.

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DOI: 10.36692/V16N1-66

INTRODUCTION

The use of ceramics in dentistry dates back to the 18th century when Alexis Duchateau replaced animal teeth with ceramic teeth for prosthesis fabrication, seeking aesthetic improvements. In 1839, John Murphy achieved the first porcelain restoration by developing the platinum foil technique. At the beginning of the 20th century, with the advent of feldspathic or alumina porcelain, jacket crowns began to be used; however, issues such as low fracture resistance and considerable cervical misfit were observed. For these reasons, their application was limited to anterior teeth. In the 1960s, with the introduction of metal-ceramic systems, porcelains became classic in oral rehabilitations and remain the most widely used restorative modality today.

The bond between metal and ceramic provides great strength to restorations; however, aesthetic issues such as the inability to transmit light and darkening of the marginal gingiva have been factors to consider when choosing the material to be used, especially in the anterior region. Thus, the development of new materials aimed to eliminate the use of metal to improve aesthetic qualities. Currently, ceramic stands out as an alternative that meets the aesthetic, biological, mechanical, and functional requirements demanded of a restorative material and has its indication well established in dentistry 42.

However, when talking about ceramics, it does not necessarily refer to a single material, as there are several ceramic systems with different chemical compositions and processing methods for various indications, which often makes their classification difficult. The use of highdensity zirconia-based ceramics (ZrO2) has been proposed due to this material's excellent biocompatibility, high hardness, wear resistance, flexural strength, and high fracture toughness ¹⁸. Zirconia is used in dentistry as an opacifying agent, mechanical reinforcement in feldspathic porcelains, material for manufacturing dental implants and intra-radicular posts, substructures for implant-supported prostheses, orthodontic brackets, and as the main element in the fabrication of copings for prosthetic crowns ³⁶. Zirconia has several advantages over other ceramics, primarily due to its transformation toughening mechanism, which can confer very interesting mechanical properties to the parts, such as high mechanical strength and toughness 34,17.

Cementation is a vital process for the clinical success of all-ceramic restorations. According to recommendations from some manufacturers, these restorations can be cemented using zinc phosphate, glass ionomer, or resin-based cements. However, resin cementation is necessary for certain types of restorations 14,32. Currently, different types of cements are available on the market, including conventional resin cements and self-adhesive resin cements, with chemical, light, or dual activation. New cementation agents have been launched, and it is important to note that any change in their chemical composition can affect the bond strength with the different ceramic systems, which in turn also have diverse chemical compositions 21,27,29.

Therefore, considering the increasing use of zirconia ceramics in dentistry and the introduction of new cementation systems, it is necessary to develop a reliable technique for adhesive cementation for both high-strength ceramic systems like zirconia and conventional feldspathic porcelains, which are still widely used clinically.

PROPOSITION

"The objective of this study was to evaluate the bond strength between two ceramic systems, a feldspathic porcelain and a yttria-stabilized zirconia ceramic, and three different resin cements: Rely X ARC, Multilink System, and Rely X UCem".

 METHODOLOGY

In this study, the zirconia oxide ceramic systems ProtMat (ProtMat, Volta Redonda, Brazil) and feldspathic porcelain Noritake EX-3 (Noritake Kizai Co, Higashiyama, Japan) were used, along with the cementation systems Rely X ARC (3M-ESPE, Seefeld, Germany), Multilink (Ivoclar Vivadent, Schaan, Liechtenstein), and Rely X UCem (3M-ESPE, Seefeld, Germany). Tables 1 and 2 present the materials used in this study, including their commercial names, manufacturers, chemical compositions, and batch numbers.

Chart 1 - Ceramic materials used.

Source: own authourship.

Chart 2 - Cementing systems used.

Source: own authourship.

Specimen preparation

Specimen preparation of Feldspathic Porcelain

The powder of Noritake EX-3 feldspathic porcelain (Noritake Kizai Co, Higashiyama, Japan), mixed with distilled water, was condensed into a metal mold with a perforation measuring 9.0 mm in diameter and 3.0 mm in thickness. For insertion, a 24 Duflex spatula (SSWhite, Rio de Janeiro, Brazil) and absorbent paper were used. Ten ceramic pellets were obtained and removed from the mold using a plastic plunger. After drying at a temperature of 600°C for 6 minutes, the ceramic pellets were placed in a Vulcano Platinum porcelain furnace (EDG, São Carlos, Brazil) with a heating rate of 55°C/min, under vacuum, until reaching 910°C, and then held at this temperature for an additional minute without vacuum. The ceramic was cooled to room temperature. This process was repeated three times to obtain 20 pellets of this material. After fabrication, the pellets were polished using a mechanical polisher with sequential Aquaflex water sandpapers (Norton, Guarulhos, Brazil) of grit sizes 320, 400, 600, 800, 1200, and 1500, each for about 30 seconds at a speed of 300 rpm.

Specimen preparation of Zirconia ProtMat

Prostheses made from yttria-stabilized zirconia oxide are milled from pre-sintered blocks using either pantographs or CAD/CAM systems. A pre-sintered yttria-stabilized zirconia block measuring 100.0 mm in diameter and 6.0 mm in thickness was acquired. Considering the shrinkage during sintering, which ranges from 20% to 25%, 15 square pellets measuring 9.0 mm by 9.0 mm with 6.0 mm thickness were obtained. These pellets were then halved to obtain 30 pellets measuring 9.0 mm by 9.0 mm by 3.0 mm thickness, with one surface remaining smooth for cementation purposes. The 30 cut pellets were sintered in a Zirkonzahn sintering furnace (Zirkonzahn, Gais, Italy) for 8 hours at a temperature of 1500°C and cooled to room temperature. These pellets did not require polishing since the surface of the block from which they were obtained was already smooth and standardized.

Inclusion

All ceramic pellets were embedded using clear autopolymerizing acrylic resin Jet (Artigos Odontológicos Clássico Ltda., São Paulo, Brazil). For this purpose, a glass plate was used with two markings: one for the diameter of the PVC tube and another centered with a overhead projector pen. The ceramic pellets and the 1/2-inch diameter and 15 mm tall PVC tubes were adhered to the glass plate surface at their respective markings using Pritt glue stick (Henkel Chile S.A., Santiago, Chile). The acrylic resin was manipulated according to the manufacturer's specifications and poured into the PVC tubes over the ceramic pellets until filled to the edge.

Surface treatment

Samples of each ceramic system received surface treatment according to previously conducted studies: feldspathic porcelain was conditioned with 10% hydrofluoric acid for 120 seconds, and yttria-stabilized zirconia was sandblasted with 50um aluminum oxide at 40psi pressure, from a distance of 10.0 mm for 10 seconds. All samples were rinsed with air/ water spray for 30 seconds and then placed in an ultrasonic cleaner (Thornton, Ipec Eletrônica Ltda., Vinhedo, Brazil) for a 10-minute bath with distilled water.

Cementation

After completing the surface treatments, the cementation procedure was carried out individually. Initially, a silane agent was applied to the treated ceramic body, varying according to the type of ceramic and the cementation system: for feldspathic porcelain, Ceramic Primer (3M/ ESPE, St Paul, Germany) was used with Rely X ARC and Rely X UCem cements, and Monobond

S (Ivoclar Vivadent, Schaan, Liechtenstein) was used with Multilink; for zirconia ceramic, Metal/ Zirconia Primer was used only with Multilink, while no primer was used with Rely X ARC and UCem cements. The silane was applied using a manual applicator (Applicator Tips, Dentsply, DeTrey), left to act on the surface for 60 seconds, and then dried with an air jet for 5 seconds. Next, a metal matrix with a circular perforation was fitted onto a PVC tube, into which another metal matrix was placed, now bipartite with a central perforation of 5.0 mm in diameter and 3.0 mm thick, to delimit the area for resin cement adhesion to the ceramic. With the matrices positioned, equal portions (0.0600 g each) of base and catalyst pastes of the resin cements were weighed on a precision balance (0.0001 g), model BL 210S (Sartorius, Gottingen, Germany), totaling 0.1200 g. The two portions of resin cement were manipulated with a plastic spatula (Clearfill, Kuraray, Tokyo, Japan) for 10 seconds and applied in excess with a composite resin spatula (American Eagle, Missoula, USA). A polyester strip and a glass slide were cut to fit the matrix surface to regularize the surface and prevent contact with oxygen, and left in place for 5 minutes to set before being light-cured for 40 seconds with an Elipar Free Light curing unit (3M-ESPE, Seefeld, Germany). The matrix was then removed, and the specimens were stored in a thermal cycling simulation machine.

Termocycling

For the thermal cycling, a thermal cycling simulation machine (MSCT-3, São Carlos, Brazil) was used. The specimens were placed inside a mesh bag and tied to prevent any loss during the thermal cycling process. Initially, the samples were stored in distilled water at 37°C for 24 hours.

Subsequently, 1,000 cycles were performed, with each cycle ranging from 5°C to 55°C, each bath lasting 30 seconds and with a 5-second transition time between baths. After completing the cycles, the specimens were again stored for 24 hours in distilled water at 37°C.

Shear bond strength

Mechanical Test

The shear bond strength mechanical test was performed using an EMIC DL2000 testing machine (EMIC, São Paulo, Brazil) equipped with a 20 kN load cell and an actuator speed of 0.5 mm per minute. Data will be collected and printed by the machine.

For the test, a cylindrical rod with a wedgeshaped tip was used, which tangentially contacted the flat surface of the specimen, applying vertical compressive force at the ceramic/resin cement adhesive interface.

Shear bond strength values were recorded in MPa. The data were tabulated and then subjected to D'Agostino normality test, followed by one-way ANOVA and Tukey's post hoc test using BioEstat version 3.0 software.

Verification of failure modes

For such verification, a stereoscopic microscope (Carl Zeiss, Jena, Germany) with 30 times magnification was used, and images were captured using the Leika software (CK Comércio Ltda., São Paulo, Brazil) in TIFF format. Black and white images of each fractured specimen were obtained. The images could be classified as follows: (a) adhesive failure, (b) cohesive failure of the cement, (c) cohesive failure of the ceramic, (d) mixed – adhesive and cohesive failure of the cement, (e) mixed – adhesive and cohesive failure of the ceramic, and (f) mixed – cohesive failure of the cement and cohesive failure of the ceramic.

RESULTS

Bond Strengh

The measured values for the variable shear bond strength of ceramic/resin cement union, expressed in MPa, are found in Table 1.

Source: own authourship.

The normality of the distribution of this variable was tested using the D'Agostino method, resulting in p-values > 0.05, indicating that the distribution is 'NORMAL'. The normality test is presented in Table 2.

Source: own authourship.

A parametric test, one-way analysis of variance (ANOVA), was selected, which showed (Table 3).

significant differences between the averages

Table 3 - One-way Analysis of Variance.

Source: own authourship.

"The p-value less than 0.05 indicates significant differences between the studied groups. Tukey's test was used for pairwise mean Table 4 - Algorithm for calculating the modulus of differences between averages.

comparisons with a significance level of ≤ 0.05 . Table 4 shows the algorithm for calculating the magnitude of differences between the averages.

* Significant value

Based on the data from Table 4, the observations are as follows:

a) There were no significant differences within the groups that used feldspathic porcelain as the substrate, meaning all cements yielded the same result for cementation on feldspathic porcelain.

b) For zirconia substrate, significant differences were observed among the three cements. Multilink cement achieved the highest bond strength values, while Rely X ARC obtained the lowest values, with UCem showing intermediate values.

c) The resin cements Rely X ARC and UCem showed better bond strength results when the cementation substrate was feldspathic porcelain.

d) Multilink cement showed consistent effectiveness for cementation on both feldspathic porcelain and zirconia substrates.

The means of the groups are also depicted in Graph 1.

Graph 1 - Average bond strength values of the studied groups (in MPa).

Regarding the analysis of the types of

failures observed, you can refer to Table 5, which details these occurrences in the studied groups.

Table 5 - Types of failures resulting from the mechanical test, juxtaposed with their respective averages (in MPa) and their occurrences.

- A. Cohesive failure of the ceramic
- B. Adhesive failure
- C. Cohesive failure of the cement
- D. Mixed failure A + B

E. Mixed failure A + C

F. Mixed failure B + C

Source: own authourship.

Feldspathic Porcelain

A failures (cohesive ceramic failures) regardless of the cement used.

There were approximately 80% of Type

Figure 1 - Cohesive ceramic failure in a sample from the feldspathic porcelain group.

Source: own authourship.

Zirconium Oxide Regardless of the treatment performed, there were 100% of failures classified as Type B (adhesive).

Figure 2 - Adhesive failure in a sample from the zirconia oxide group.

Source: own authourship.

DISCUSSION

The use of indirect restorations cemented onto prepared teeth is a common practice in daily dental clinical practice. Dental porcelains, due to their aesthetic characteristics resembling natural teeth and highly satisfactory mechanical and biological properties, have been one of the most demanded materials in oral rehabilitation for quite some time 19. This use was mostly associated with porcelain and a metallic reinforcement structure, due to their high friability and low resistance ⁸. However, the development of new ceramic systems that do not require metal has provided a highly satisfactory treatment option in terms of aesthetics, allowing both patients and professionals to achieve high levels of satisfaction ²⁶.

As a result of this, it is important to note that there was also a need for the development of suitable cementing agents for this new type of prosthetic parts. Many authors and manufacturers report the possibility of cementing all-ceramic crowns or fixed partial dentures using a nonadhesive cement such as zinc phosphate or glass ionomer, due to the high strength of these prostheses, which is provided by the ceramic infrastructure 32,35 . However, adhesive cementation is a necessary condition for the fixation of veneers, inlays, onlays, and adhesive fixed prostheses. Therefore, it is crucial to have sufficient bond strength between the resin cement and the ceramic. This is why several authors seek to observe the adhesive strength behavior between ceramic systems and resin cements of various compositions and commercial brands 1,2,4 ,5,6,7,9,10,11,12,13,15,20,22,25,38.

Currently, one of the most promising ceramic systems for use in dentistry is the one that utilizes yttria-stabilized tetragonal zirconia polycrystals (Y-TZP), due to its high mechanical strength, excellent biocompatibility with the oral environment, and suitable optical properties when compared to metals ^{16,24,34,41}. However, some authors also report poor bonding of this material with existing cementing agents in the market, potentially limiting its use, especially regarding the effectiveness of cementing components made with this ceramic system 6,7,21,27,29,32,35,36,43,44,45.

One of the most commonly used mechanical tests to evaluate the bond strength between ceramic and resin cements is the mechanical shear bond strength test 3,23,31,37,40. Therefore, this test was chosen for conducting this study.

In this study, we used a feldspathic porcelain for coverage, manufactured by sintering (Noritake Ex-3, Noritake), and Prot Mat zirconia.

These two ceramic materials received the same surface treatment recommended by the manufacturers of the cements used in the study (Multilink, Ivoclar/Vivadent; Rely X ARC, 3M/ ESPE; and Rely X UCem, 3M/ESPE).

Given the methodology employed and the results obtained in this study, it is observed that there was no significant difference within the groups using feldspathic porcelain as the substrate. In other words, all cements yielded the same result for cementation on feldspathic porcelain. However, for zirconia substrate, significant differences were found among the three cements used in the study, with Multilink cement showing the highest adhesive strength values for this substrate. Furthermore, Multilink cement demonstrated similar adhesive strength results for both feldspathic porcelain and zirconia. Therefore, the distinct composition of these cements may lead to more efficient interaction with the zirconia substrate, resulting in better performance of one cementing agent over another in terms of adhesive strength with this substrate. The same reasoning applies when observing that Rely X ARC and UCem cements showed higher adhesive strength results with feldspathic porcelain in this study compared to when the substrate was zirconia.

All these results can be further interpreted through a subjective analysis using microscopy to examine the type of fracture at the interface of the test specimens used for the assays. In this study, such analysis revealed that in all samples

where zirconia was used, adhesive failures occurred. In contrast, in samples with feldspathic porcelain, failures were predominantly cohesive within the ceramic or mixed adhesive and cohesive failures within the ceramic. This finding is particularly significant because despite similar bond strength values found for Multilink cement on both types of substrates, the observation that cohesive ceramic failures were predominant in feldspathic porcelain, whereas zirconia exhibited predominantly adhesive failures, leads us to believe that the adhesive strength of this cement is higher with feldspathic porcelain. This is inferred because the adhesive interface for this substrate did not undergo rupture."It can be said that, based on the results obtained from this study, ceramic systems that use zirconia as a reinforcement structure constitute a valid treatment modality for the fabrication of prosthetic parts that will be cemented with resin cements. However, it should be emphasized that more laboratory and clinical studies are necessary to fully elucidate the complete range of possibilities for using this system."

CONCLUSION

The bond strength between the studied resin cements and the feldspathic porcelain surpassed the intrinsic strength of this material, meaning the bond strength of the cement/ ceramic interface is greater than the strength of the feldspathic porcelain.

The Multilink cementation system exhibited the highest bond strength values, regardless of the ceramic material.

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Observação: os/(as) autores/(as) declaram não existir conflitos de interesses de qualquer natureza.