Micro-shear bond strength of bioactive cement to translucent zirconia after thermocycling: a comparative in-vitro study

Resistência de união ao microcisalhamento do cimento bioativo à zircônia translúcida após termociclagem: estudo comparativo in vitro

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ABSTRACT

Objective: the purpose of the study was to evaluate the micro-shear bond strength of different cements to translucent zirconia before and after thermocycling aging. **Material and methods:** Twelve translucent zirconia ceramic discs were used in the study. Specimens were sandblasted using 50 μm aluminum oxide (Al_2O_3) particles. The specimens were divided into three groups (n = 4) according to the cement type: Panavia resin cement (control group), resin modified glass ionomer (RMGI), and Activa bioactive cement. Each group was further sub-divided into two equal subgroups (n = 2) according to whether the specimens were subjected to thermocycling or not. Thermocycling was performed in distilled water at 5000 cycles between 5 ºC - 55 ºC. The micro-shear bond strength test (μSBS) was measured using universal testing machine. Kruskal-Wallis test was used to compare between the three cements. Dunn's test was used for pair-wise comparisons when Kruskal-Wallis test is significant. Mann-Whitney U test was used to compare between micro-shear bond strength before and after thermocycling P $\leq$ 0.05. **Results:** In non-aged subgroups, there was no significant difference between Panavia and Activa; both showed significantly the highest mean μSBS values (22.9 MPa, 31.3 MPa respectively). While, RMGI showed the lowest μSBS values (4.7 MPa). In thermocycled subgroups, Panavia showed significantly the highest mean μSBS values (32.2 MPa). There was no significant difference between RMGI and Activa; both showed the lowest significant mean μSBS values (3.2 MPa and 8.7 MPa respectively). **Conclusions:** RMGI and Activa couldn't be considered long-term reliable materials for cementing zirconia. However, Panavia provided the most durable bond to zirconia.

KEYWORDS

Bioactive cement; Micro-shear bond strength; Resin cement; Translucent zirconia.

RESUMO

Objetivo: O objetivo do estudo foi avaliar a resistência de união ao microcisalhamento de diferentes cimentos à zircônia translúcida antes e após o envelhecimento da termociclagem. **Material e métodos:** Doze discos de zircônia translúcidos foram utilizados no estudo. As amostras foram jateadas com partículas de óxido de alumínio de 50 μm (Al_2O_3). Os espécimes foram divididos em três grupos (n = 4), de acordo com o tipo de cimento: cimento resinado Panavia (grupo controle), ionômero de vidro modificado por resina (RMGI) e cimento bioativo Activa. Cada grupo foi subdividido em dois subgrupos iguais (n = 2), dependendo se as amostras foram submetidas ou não a termociclagem. A termociclagem foi realizada em água destilada a 5000 ciclos entre 5°C - 55°C. O teste de resistência de união por microcisalhamento (μSBS) foi medido usando uma máquina de teste universal. O teste de Kruskal-Wallis foi utilizado para comparar os três cimentos. O teste de Dunn foi usado para comparações entre pares quando o teste de Kruskal-Wallis foi significativo. O teste U de Mann-Whitney foi utilizado para comparar a resistência de união ao microcisalhamento antes e após a termociclagem (P ≤ 0,05). **Resultados:** Nos subgrupos sem envelhecimento por termociclagem, não houve diferença significativa entre Panavia e Activa; ambos mostraram significativamente os maiores valores médios de μSBS (22,9 MPa, 31,3 MPa, respectivamente). Por sua vez, o RMGI apresentou os menores valores de μSBS (4,7 MPa). Nos subgrupos termociclados, Panavia mostrou significativamente os maiores valores médios de μSBS (32,2 MPa). Não houve diferença significativa entre RMGI e Activa; ambos apresentaram os menores valores médios significativos de μSBS (3,2 MPa e 8,7 MPa, respectivamente). **Conclusões:** RMGI e Activa não puderam ser considerados materiais confiáveis para cimentação à zircônia a longo prazo. No entanto, a Panavia apresentou a ligação mais durável à zircônia.

PALAVRAS-CHAVE

Cimento bioativo; Resistência a microcisalhamento; Resina de cimento; Zircônia translúcida.

INTRODUCTION

With the introduction of CAD/CAM technology, production of zirconia restorations has become a totally digitized process. Chemical and dimensional stability are among multiple factors that render zirconia as a good choice for prosthetic rehabilitation [1,2]. This group of materials has no glass matrix, which renders them to be more opaque and less translucent materials [1–3]. Due to the decreased translucency of the first generation of zirconia and compromised esthetics, this problem is solved by veneering the restoration framework with suitable glass-ceramics after being individually produced. However, some reports have documented fracture of ceramic veneers (chipping) [4,5] due to difference in the coefficients of thermal expansion of the framework material and veneering ceramic [4,6]. To overcome these problems of veneered zirconia, the full contoured monolithic zirconia was introduced as an alternative treatment option [5,6]. The fabrication of monolithic zirconia has many advantages as it reduces fracture possibilities and avoids chipping [6,7]. Besides, it is characterized by minimal occlusal adjustment, high strength, and accuracy in the marginal fit [7,8].

The clinical success of ceramic restorations depends on several factors among which is the cementation procedure [9]. Wide choices of materials for cementing metal-free restorations include: zinc phosphate, conventional and modified glass-ionomer cements, resin cements and self-adhesive cements [10]. However, bonding to zirconia is a challenge compared to glass ceramics because zirconia is silica free [11–13].Thus, several surface treatments have been introduced for bonding to zirconia including tribochemical silica coating, laser irradiation, and airborne particle abrasion [11–16]. Several studies concluded that 10-MDP containing resin cement in combination with airborne particle abrasion produced the highest shear bond strength [17–19]. On the other hand, RMGIC was suggested by some researchers as an alternative to resin bonding. Many advantages were pointed out such as its simplicity, being less technique sensitive and it requires fewer steps procedure with less probable postoperative sensitivity [19–22].

Nowadays, bioactive materials became an interesting topic in the field of restorative dentistry due to its biological compatibility. The concept of bioactivity refers to a specific property of a material that will induce a response from a living tissue or cell such as inducing the formation of hydroxyapatite. The direct function of the bioactive material is to induce growth factors and stimulate natural mineralization [23].

Activa bioactive cement was introduced in 2013 with claims to its high biological compatibility [24]. Activa contains three key components: bioactive ionic resin matrix, shock absorbing resin component, and reactive ionomer glass fillers [25]. The acquired bioactive properties of active cement is coupled with improved resiliency due to the resin matrix, which would contribute to enhanced wear resistance, fracture and marginal chipping [26].

Thermal cycling is a laboratory method used to simulate the clinical conditions with the accompanying deteriorating effects. Thermal stresses, water sorption, leakage and other destructive water and time related effects appear well after a period of use [27]. The majority of studies showed that thermocycling significantly reduces the bond strength [28,29].

Limited evidence is available regarding the bond strength of translucent zirconia to the bioactive cement. So, it would be worthy; however, to investigate the micro-shear bond strength of bioactive cement to translucent zirconia compared to those cemented with resin modified glass ionomer and resin cement after thermocycling. The first null hypothesis was that there is no differences in the micro-shear bond strength (μSBS) of translucent zirconia to different luting cements. The second null hypothesis was that thermocycling doesn't affect the bonding of different luting cements to translucent zirconia.

MATERIAL AND METHODS

Materials and experimental groups

Materials used in the study are described in table I. Twelve zirconia BruxZir anterior discs (Glidwell Dental Labs, Prismatik DentalCraft Inc. USA) were divided into three equal groups according to the cement used: Panavia SA (Kurary Noritake Dental Inc. Japan), RelyX Luting Plus (3M ESPE. USA), and Activa bioactive (PULPDENT Corporation USA). Each group was further sub-divided into two equal subgroups according to whether the specimens were subjected to thermocycling or not. For micro-shear bond strength test, each disc received 5 microtubules, giving a total of 60 microtubules (N = 60, n = 10) (Table II).

Table I - list of brands name, materials description, manufacturers and lot numbers used in this study

Brand name	Material description	Manufacturer	Lot number
BruxZir anterior	Zirconia milling blank Anterior shaded 150 A1, B1	Glidwell Dental Las, Prismatik Dentalcraft, Inc CA	Z0853787
Panavia SA	Self adhesive resin cement	Kuraray Noritake dental Inc. Japan	8NO111
RelyX luting plus	Resin modified glass ionomer cement	3M ESPE, St. Paul, MN, USA	N903754
Activa	Bioactive cement translucent	Pulpdent, watertown, MA,USA	161027
Clearfil ceramic primer	Dental universal prosthetic primer	Kuraray Noritake Dental Inc, Japan	CE0031
Sandblasting abrasive powder	50 µm aluminum oxide	Renfert GmbH, Hilzin-gen, Germany	A722A30

Table II - Samples grouping

Type of luting cement	Panavia SA (control)		RelyX luting plus		Activa	
Number of discs	4		4		4	
Subject to thermo-cycling	Yes	No	Yes	No	Yes	No
	2	2	2	2	2	2
Micro-shear bond strength test (5 microtubules/disc)	10	10	10	10	10	10
Total number	60					

Sample preparation

Two disc-shaped objects were designed in Meshmixer CAD software (Autodesk Meshmixer, version 1.0.544) with 3 mm thickness and 12 mm diameter. Twelve samples were soft dry-milled from BruxZir anterior blocks from the 3 mm thickness design, using a Roland DWX50 5-axis dental milling machine (Roland DWX 50, Roland DGA Corp, California, USA). All zirconia discs (n = 12) were sintered following the manufacturer's recommendations, using Sirona inFire HTC (Dentsply Sirona, Salzburg, Ausrtis). For ease of handling, each specimen was embedded in a 3D printed mold using Meshmixer CAD software. The STL design file was transferred to the slicing software of DLP 3D printer (Wanhao duplicator 7, China). The cylindrical mold was printed using photo-curable resin (FTD resin, ALKmaar,Netherland) which was filled with cold cure acrylic resin.

All zirconia disc specimens of each group were subjected to sandblasting. The surfaces of the ceramic disc specimens were air abraded with 50-μm white aluminum oxide ($Al2O3$) particles in a dental sandblasting unit (Renfret, Germany) under two bar pressure (30 psi) at a distance of 15 mm between the nozzle of the sandblaster and the ceramic surface for 20 seconds each. All ceramic disc specimens were ultrasonically cleaned with distilled water for three minutes then air-dried gently.

Clearfil Ceramic Primer Plus (Kuraray Noritake Dental Inc) was applied in one coat to all disc specimens with a brush and left to dry for one minute following the manufacturer's recommendations.

For micro-shear bond strength test (μSBST), five transparent polyethylene microtubules were cut from a 6 FG nelaton catheter, of internal diameter of 0.9mm and 1 mm height were placed on each ceramic surface (Figure 1).

For each luting cement, a clear automix tip with a bendable 20-gauge metal cannula was attached to the cartilage of the luting cement to fit easily in each microtubule. After filling

the luting cement in the microtubules, all the cement filled-microtubules were light cured (3M ESPE, Elipar light cure) on each disc specimen, following the manufacturer's recommendation. After curing, the microtubules were cut using a surgical blade size 15 by making a vertical cut along the microtubule wall and each was carefully removed leaving the cement micro-cylinders properly bonded on the ceramic surface disc.

Thermocycling

Six zirconia disc specimens (2 discs/group) were subjected to thermocycling in distilled water in a thermocycling unit (Julabo FT200, Julabo, Seelback, Geramany) for 5000 cycles at changing temperature between -5 °C and 55 °C with a dwell time of 30 seconds [30].

Microshear bond strength testing

Micro-shear bond strength test (μSBST) was performed out using a universal testing machine (Instron 3345, Instron Corporation, MA, USA). An orthodontic wire (0.2 mm diameter) was used to loop around the base of each cement micro-rod. Micro-shear force was applied on each micro-rod at a crosshead speed of 0.5 mm / minute until fracture occurred. Micro-shear bond strength was calculated by dividing the maximum load at failure (N) over the bonded surface area (mm2) and recorded in megapascals (MPa).

Statistical analysis

Numerical data were explored for normality by checking the distribution of data and using tests of normality (Kolmogorov-Smirnov and Shapiro-Wilk tests). Micro-shear bond strength data showed non-normal (non-parametric) distribution. Data were presented as mean, standard deviation (SD), median and range values. Kruskal-Wallis test was used to compare between the three cements. Dunn's test was used for pair-wise comparisons when Kruskal-Wallis test is significant. Mann-Whitney U test was used to compare between micro-shear bond strength before and after thermocycling. The significance level was set at $P \leq 0.05$.

Statistical analysis was performed with IBM® SPSS1® statistics version 20 for windows.

Scanning electron microscope observations

To identify the effect of surface treatment protocol (sandblasting), representative disc specimens (2 discs) were subjected to scanning electron microscope (SEM) analysis (Quanta 250 FEG, ThermoFisher Scientific, MA, USA) before and after surface treatment at magnification 5000X.

To identify the failure mode after micro-shear bond strength test, disc specimens of each group were subjected to scanning electron microscope (SEM) at a magnification of 200X. The mode of failure was classified as follows:

a. Adhesive failure in the cement/ceramic interface;

b. Cohesive failure within the cement or the ceramic surface;

c. Mixed failure combining both parts of cement and ceramic interface (both cohesive and adhesive).

RESULTS

Micro-shear bond testing

The means and standard deviations of micro-shear bond strength values in the non-aged groups showed no significant difference between Panavia and Activa cements; both showed the highest significant mean micro-shear bond strength values. The lowest bond strength was observed in the samples luted with RMGI cement ($P < 0.001$) (table III). In thermocycled groups, Panavia cement showed the highest significant mean bond strength values. No significant difference was found between samples luted with RMGI and Activa cements; both showed the lowest bond strength values ($P < 0.001$) (table III).

Table III - Means and standard deviations of micro-shear bond strength values of the tested group

Thermocycling	Panavia		RMGI		Activa		P-value
	Mean	SD	Mean	SD	Mean	SD	
Non-aged	22.9 A	6.4	4.7 B	2	31.3 A	3.8	<0.001*
Thermocycled	23.2 A	5.6	3.2 B	1.8	8.7 B	3.9	<0.001*

*: Significant at P ≤ 0.05, Different superscripts in the same row are statistically significantly different

Effect of thermocycling

The results of Mann-Whitney U test showed no significant difference in the bond strength values in non-aged and thermocycled groups luted either with Panavia or RMGI cements (p = 0.821, p = 0.295 respectively), while significant decrease in the bond strength values were observed on thermocycled group luted with activa cement compared to non-aged group (P < 0.05) (Table IV).

Table IV - Means and standard deviations in comparison between micro-shear bond strength of specimens subjected to thermocycling or not

| Cement	Non-aged		Thermocycled		P-value
	Mean	SD	Mean	SD	
Panavia	22.9	6.4	23.2	5.6	0.821
RMGI	4.7	2	3.2	1.8	0.295
Activa	31.3	3.8	8.7	3.9	<0.001*

*: Significant at P ≤ 0.05

Scanning electron microscope observations

Scanning electron microscope (SEM) images of Monolithic zirconia surface before sandblasting revealed totally uniform crystalline mostly prismatic and plate shaped grains. After sandblasting, SEM observations clearly showed wavy surface to produce rough surface ready for the cementation (Figure 2).

Failure Mode Analysis

SEM images showed cohesive failure in the resin interface which was predominant in all Panavia cement groups in non-aged and thermocycled groups (figure 3), while for RelyX Luting Plus it was adhesive failure at the cement/zirconia interface in non-aged and thermocycled groups (figure 4). Moreover, Activa bioactive cement showed mainly cohesive failure within the cement in non-aged and adhesive failure at the cement/zirconia interface in thermocycled groups (figure 5).
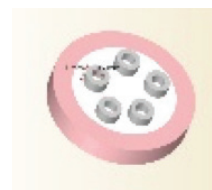
Figure 1 - Schematic diagram showing disc specimen with five microcylinders with height of 1 mm and internal diameter of 0.9 mm.
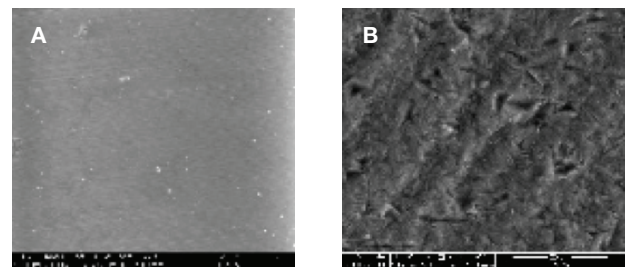
Figure 2 - SEM photographs of Monolithic zirconia surface: A: before sandblasting showing totally uniform crystalline mostly prismatic and plate shaped grains. B: after sandblasting showing wavy surface to produce rough surface (at 5000X magnification).
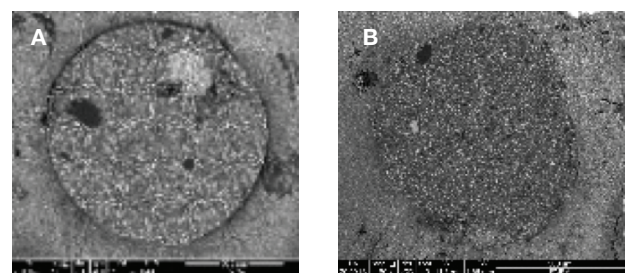
Figure 3 - SEM images of Monolithic zirconia cemented with Panavia resin cement showing cohesive mode of failure in the resin interface. A: Non-aged. B: thermocycled (At 200X Magnification).
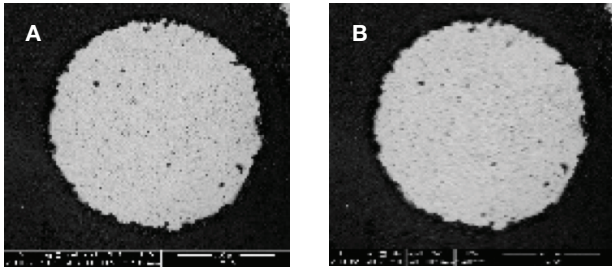
Figure 4 - SEM images of Monolithic zirconia cemented with RelyX luting plus cement showing adhesive mode of failure at the cement /zirconia interface. A: Non-aged. B: thermocycled (At 200X Magnification).
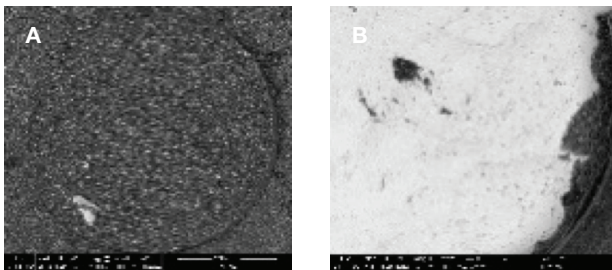

Figure 5 - SEM images of Monolithic zirconia cemented with Activa cement showing A: cohesive mode of failure within the cement for non-aged group. B: Adhesive mode of failure at the cement/zirconia interface for thermocycled group (At 200X Magnification)

DISCUSSION

In this study, the results showed that the micro-shear bond strength to zirconia was affected by the type of the luting agent. Thermocycling played a role in the degradation at the cement/zirconia interface and decreased the bond strength of Activa cement to zirconia. So, the proposed first and second null hypotheses were partially rejected.

Several studies [11,31–34] have investigated the bond strength and durability of various bonding methods to zirconia. It seems that the best results are obtained with air-particle abrasion which was selected as a surface treatment protocol in the current study. As this protocol is considered to be the gold standard one for treating zirconia as it increases the surface roughness and the surface area for bonding with zirconia through micromechanical bond mechanism [18,35,36]. SEM analysis of air abraded surface in the present study revealed wavy surface to produce rough surface ready for bonding (figure 2).

In the current study, the high micro-shear bond strength of the non-aged specimens luted with Panavia SA, could be attributed to the presence of 10-MDP monomer group within Panavia. These findings are consistent with those of previous studies [18,37–39] who reported that Panavia luting cement contains 10-MDP- monomer have been identified as a key factor in achieving durable bond with zirconia based ceramics as they have chemical bond to zirconium oxide, and create water proof bond with zirconia.

The high bond strength of Activa cement compared to the RMGIC may be due to their structural differences and better mechanical and physical properties [24,40]. It is the first bioactive dental material with reactive ionomer glass fillers and a shock-absorbing resin component which improves the resilience and the physical properties that may provide improved clinical performance and durability [24]. These coincides with those findings reported by Pameijer et al. [40] who found that the flexural strength and the flexural fatigue of the Activa bioactive restorative material was significantly greater than the other commercial glass ionomer cements.

Moreover, the lowest bond strength of RMGIC may be attributed to the disability of the cement to produce chemical bond with zirconia, and that the micromechanical interlocking was weak to produce durable bond [21]. These are in agreement with Zhang et al 2010 [41] who found that the bond durability of RelyX Luting Plus Cement was not enough to obtain good bond service for zirconia ceramic in comparison to resin cement. This might indicate that no chemical reaction occurred between zirconia and RMGI cement [18].

On the contrary, these findings disagreed with those reported by Alnassar et al [42] who found a relatively high shear bond strength with RMGIC. They claimed that

RelyX Luting Plus cement which contains MDP has the ability to bond to zirconia and also has crosslinking branches (polymerization groups), which react with the resin matrix (Bis-GMA or HEMA) and create strong bonds when the resin polymerizes. The variations in the results were probably due to different research methodology as they tested the bond strength by shear test while in the present study the bond strength was tested by micro-shear bond strength test. The micro-shear bond strength test has more advantages than other testing methodologies for bond strength such as shear and micro-tensile bond strength tests. Micro-shear bond strength test is relatively simple compared to micro-tensile test as it does not require careful handling of fragile disc specimens [43]. In micro-shear bond strength test, a precise mapping of different regions of tested disc surfaces can be done. Stress distribution is uniform because an ultra-small area of bonding interface is tested [44]. Also, a wire loop rather than knife-edge chisel was used for testing the bond strength. This reduces the magnitude of stress concentration adjacent to the interface [45,46].

After thermocycling, the highest micro-shear bond strength was achieved with specimens luted with Panavia luting agent. Aside from the high mechanical properties and sealing ability of Panavia luting agent, the functional monomer in Panavia has been rated as a relatively hydrolysis stable due to the presence of a long carbonyl chain [41]. Fujishima et al [47] revealed that the bond strength of 15 to 21 MPa is necessary for clinical use. In the present study, the mean micro-shear bond strength values of the non-aged Panavia specimens was 22.9 MPa while it was 23.2 MPa for the thermocycled specimens, with no significant difference in between. These results are in agreement with those of other studies [37,41] who concluded that MDP-based cements provided more favorable adhesion to zirconia with non-significant decrease in the bond strength even after thermocycling.

The lower bond strength of RMGIC and Activa cements could be due to their low mechanical properties and the higher solubility of their glass ionomer content [48,49]. Besides, researches [50,51] claimed that water thermocycling is responsible for bond deterioration. It causes repeated thermal expansion and shrinkage of the materials used, which causes fatigue in the interphase and, therefore, reduction in the bond strength. These results are in accordance with those of Zhang et al. [41] who found that resin modified glass ionomer cement (RelyX Luting Plus) showed the lowest shear bond strength compared to Panavia and Fuji Plus. They claimed that thermocycling showed detrimental effects of hydrolytic degradation at RMGI/zirconia interface and decreased the bond strength.

The failure mode analysis after micro-shear bond strength test found that specimens luted with Panavia SA luting agent had predominately cohesive failure in the resin whether in the non-aged or thermocycled specimens, indicating durable bond to zirconia (Figure 3). This supports previous reports on this topic [18,52]. The RMGIC demonstrated predominately adhesive failure in the non-aged and thermocycled specimens, indicating a weak unstable bond to zirconia (Figure 4). A previous SEM study confirmed this fracture pattern [18]. Activa cement demonstrated cohesive failure in the non-aged specimens (Figure 5). This may be attributed to the efficient strong micromechanical interlocking which induced initial high adhesive bond strength. However, after thermocycling this bond could not be maintained and significantly decreased, with consequent adhesive failure (Figure 5).

CONCLUSIONS

Within the parameters used and the limitations of this study, it can be concluded that neither resin modified glass ionomer cement nor Activa bioactive cements could be considered long-term reliable materials for cementing

zirconia. However, Panavia resin cement provided the most durable bond to zirconia.

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