

EFFECT OF THERMOCYCLING ON THE BOND STRENGTH OF A GLASS-INFILTRATED CERAMIC AND A RESIN LUTING CEMENT

EFEITO DA CICLAGEM TÉRMICA SOBRE A RESISTÊNCIA DE UNIÃO ENTRE UMA CERÂMICA INFILTRADA COM VIDRO E UM CIMENTO RESINOSO

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The aim of the present study was to evaluate the effect of thermocycling on the bond strength between the surface of the glass-infiltrated alumina ceramic In-Ceram (VITA) and the Panavia F resin cement (Kuraray CO.). Four 5x6x6mm In-Ceram blocks were obtained. One of the 6x6mm faces of each block was conditioned with Cojet - System (tribochemical silica coating, ESPE-3M) and then luted under a constant 750g pressure with Panavia F cement to another identical face of a resin composit block (Clearfil AP-X, Kuraray) obtained by reproduction of the ceramic one from Express (3M) addition curing silicone impressions. The four sets so formed by ceramic, luting cement and resin have been each one serially sectioned in 20 sticks so that the adhesive surface in each presented 1mm² of area. The samples were divided in 2 groups (n=10): G1- stored for 7 days in deionized water at 36 ± 2°C; G2 – thermocycled 1500 times between 5 and 55°C dwell times. The microtensile tests were accomplished in an universal testing machine (EMIC) at a crosshead speed of 0,5 mm/min. The results showed that the mean tensile bond strength values (MPa) for the group G2: (22,815 ± 5,254) had not statistically differ of the values of group G1: (25,628 ± 3,353) (t = 1,427; gl = 18; p-value = 0,171), at the level of α = 5%. It can be concluded that the thermocycling technique used in the present experiment had not produced statistically significant differences between the bond strength results of the specimens obtained by the two used techniques.

UNITERMS: Dental ceramics; Resin cements; Air abrasion.

INTRODUCTION

In recent decades, patients have been pressing dental profession with a great demand for aesthetics and so, a large quantity of research are being developed for obtaining new materials with improved aesthetic properties, color stability, more resistance to fracture,

possibility of better marginal adaptation and good chemical compatibility with the new resin luting cements.

The ceramic system constituted of infiltrated by glass (15%) densely sintered alumina surface (85%) (In-Ceram, Vita, Bad Säckingen, Germany) is characterized by a framework that has flexural

resistance 75% larger than other dental ceramics and this kind of material was developed supposedly to substitute metallic frameworks of metal-ceramic crowns.

Although studies as one by McLaren¹⁹ indicate that the conventional cementation can be satisfactorily obtained with zinc phosphate and glass-ionomer cements in the In-Ceram system, Kern¹⁴ suggested that the cementation should be done with resin luting cements because they have qualities as resistance to the fracture which can improve the final result of restoration. So, a stable chemical bonding should be reached between resin luting cements and full crowns or ceramic restorations constituted of alumina as the In-Ceram system.

The bond strength between the conventional feldspathic ceramics and resin luting cements is obtained and increased by the etching of the ceramic surface with 2% fluoridric acid and treatment with a silane agent^{2,7,26}. However this method is not applied to the ceramic systems that have alumina infiltrated with glass, because it doesn't promote appropriate bond strength with resin luting cements^{16, 22}. This method is not indicated because it occurs the weakness of the bond union, due the elimination of the glass matrix of the ceramic surface after etching.

Searching a new method of superficial treatment of the glass-infiltrated alumina ceramic, Kern¹⁴ and Blixt³ verified the increase on bonding strength values when they used a BIS-GMA resin luting cement associated to the alumina surface sandblasting of the In-Ceram with a Tribochemical silica coating - Rocatec system (Espe-3M) (Rocatec-Pre: sandblasting with Al₂O₃ 110mm, Rocatec-Plus: silicoating with special powder of silica 30mm, Rocatec-Sil: silane coating).

Also Kraivixien-Vongphantuset¹⁶, Kern¹⁴, Marais¹⁸ and Kiyani¹⁵ demonstrated larger values of bond strength between resin luting cements and the glass-infiltrated alumina ceramic conditioned with tribochemical silica coating (Rocatec), even under thermic treatment⁹.

The aim of this study was to verify by using the microtensile test, the effect of the thermocycling on the bond strength between a resin luting cement containing phosphate monomer (Panavia F, Kuraray, Osaka, Japan) and a glass-infiltrated alumina ceramic In-Ceram, conditioned with the Cojet-System (tribochemical silica coating) similar to the Rocatec system^{4, 5, 6, 8, 10 13}.

MATERIAL AND METHODS

For the present study, four blocks of glass-infiltrated alumina ceramic (In-Ceram, Vita Zahnfabrik, Bad Säckingen, Germany) with dimensions of 5x6x6mm have been obtained according to the manufacturer's instructions (Figure 1).

For verifying the quality of these blocks they have been examined by x-ray and the samples with any air bubble have been eliminated. Exact reproductions of these ceramic blocks were then obtained in Kuraray Clearfil AP-X resin composit, by means of 3M Express addition curing silicone impressions.

One of the 6x6mm surfaces of each ceramic block, was planned by polishing with 300, 600, 800, 1000, 1200-grit sandpaper with the objective of creating a plane and coincident surface to the block made with composit resin. After this procedure, each ceramic surface was sandblasted with the Cojet-System (ESPE-3M), which consists of an initial sandblasting with aluminum oxide particles 110mm, at a standard distance of 10mm and perpendicular to the surface, for 20 seconds with pressure of 2,8bars, followed by other sandblasting with special particles of silica 30mm that promote the silicization (silica coating) and at last the application of a silane coat (ESPE-Sil).

The conditioned surfaces were cemented with Panavia F to the resin composit blocks. The cement was manipulated according to the manufacturer's recommendations and applied initially on the

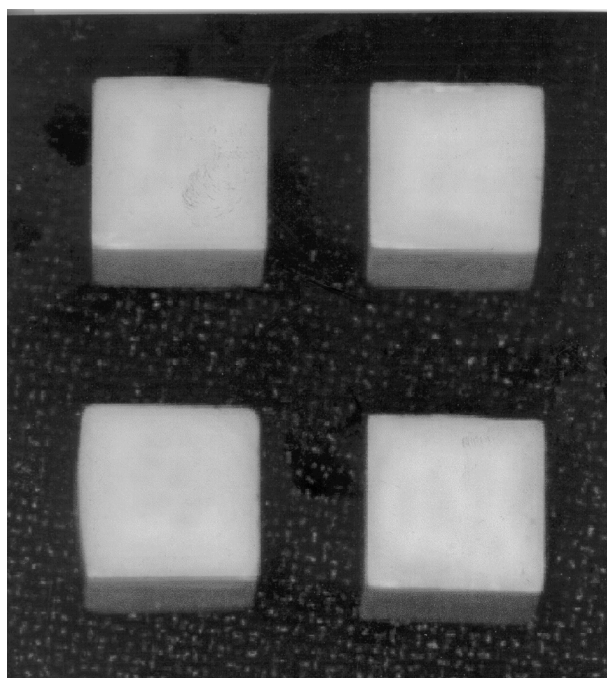


FIGURE 1- Glass-infiltrated alumina ceramic blocks (In-Ceram)

conditioned surface of the ceramic block, and after it was united to the resin composite block. It was then obtained a set constituted by ceramic, cement and resin that was positioned in a device to promote the cementation, under a constant load of 750g, for 10 minutes (Figure 2).

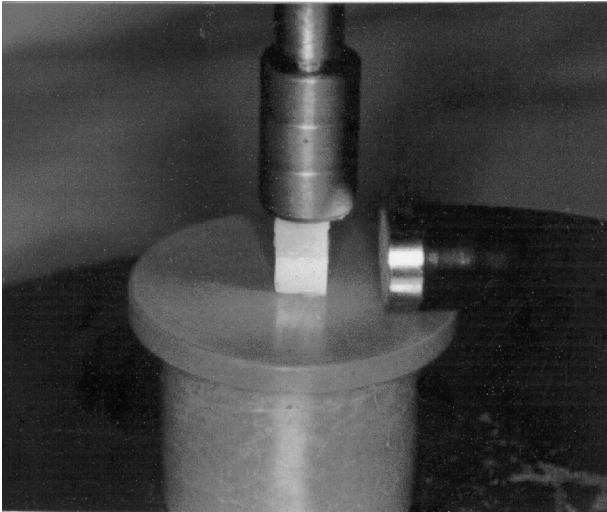


FIGURE 2- Blocks being luted on device for bonding

The excess of cement on the margins was removed and the light activation of the cement was reached applying the light for 40 seconds on each side of the sets. Cementation was concluded with the application of the oxygen-blocking gel (Oxyguard for the Panavia F). After the total curing of the cement, the sets were washed with air-water flush and stored in deionized water at $36 \pm 2^\circ\text{C}$ (Figure 3).

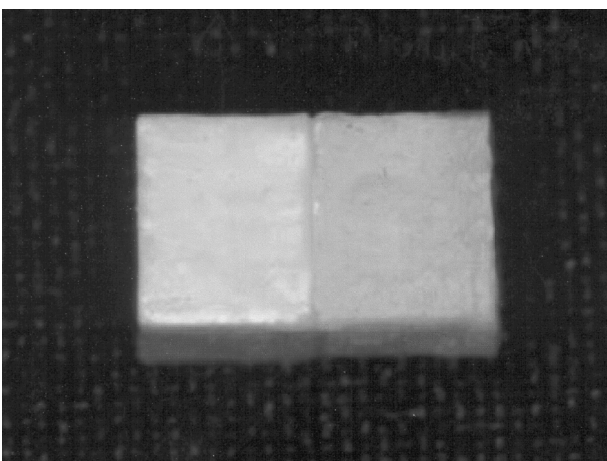


FIGURE 3- Sample constituted by ceramic and resin block after cementation

It was obtained four sets of ceramic blocks cemented to resin blocks, that were fixed in a lathe

adapted by Andreatta¹ to accomplish precision cuts with diamond disks of 0,15mm of thickness and 22mm of diameter (Figure 4).

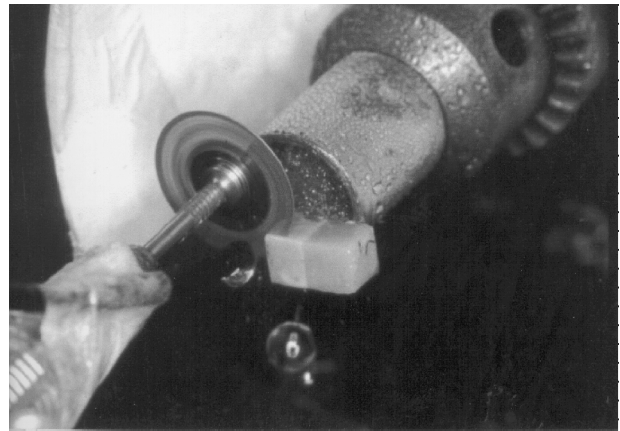


FIGURE 4- Initial cut to eliminate excess of cement on the lateral surface

The 1mm external faces of the sets were splited up in order to avoid that excess of cement on the lateral walls could influence the final results. After this, the blocks were cut in slices of 10x6x1mm. At last, this slices were splited up longitudinally for obtaining 20 samples with dimensions of 10x1x1mm \pm 0,1mm (Figure 5).

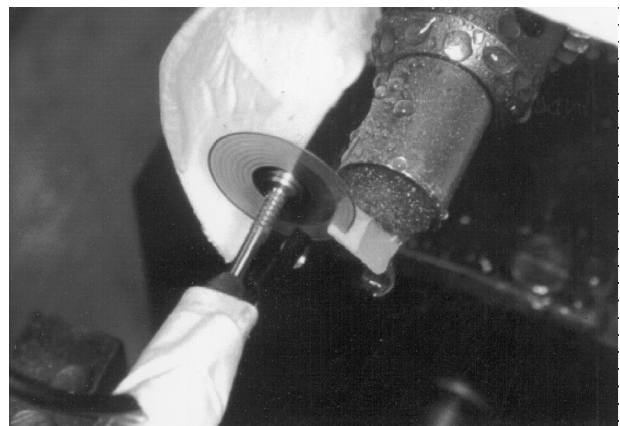


FIGURE 5- Cut for obtaining the samples

Two groups were established with ten samples (n=10) each one, varying the type of thermal treatment. In group I (no thermocycled) the samples were stored for 7 days in deionized water at $36 \pm 2^\circ\text{C}$ and in group II (thermocycled) the samples were submitted to 1500 thermal cycles in baths at 5°C and 55°C , with 30 seconds dwell times. After the thermal treatments, each sample was glued with cyanoacrylate adhesive to an adapted calliper for accomplishment of the

microtensile test. In this equipment the applied load is perpendicular to the long axis of the sample for avoiding the occurrence of sprain forces and shear on the bond area. Each sample glued to the calliper was adapted to the universal testing machine (DL-1000 - EMIC - Equipamentos e Sistemas de Ensaio Ltda - São José dos Pinhais - PR) and loaded to failure under tension at a crosshead speed of 0,5 mm/minute (Figure 6).

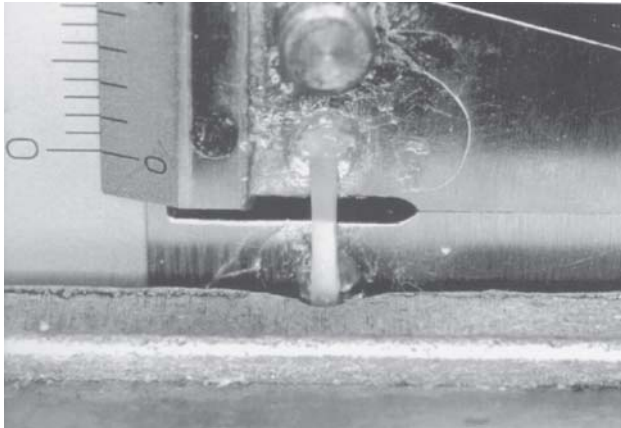


FIGURE 6- Sample fixed to the calliper mounted in testing machine

Results have been compared by the Student's test with 5% of significance.

RESULTS

The data of the microtensile test when submitted to the normality and homogeneity tests of variance were considered a normal population (p -value $> 0,05$) and of same variance (p -value $> 0,05$). The Student's test made for comparison of the thermocycling effects didn't indicate statistical difference (Table 1).

The variability on the data is represented in Figure 7. The very close values of variation coefficient ($VC = 5,254 / 22,815 = 23\%$ for the situation with thermocycling, and, of $VC = 3,353 / 25,628 = 13\%$ for the situation without thermocycling), denoted that the values are homogeneous and that the mean can be considered a measure of central tendency, representative, of the same ones.

TABLE 1- Number of samples per group (n), mean tensile strength (Mpa), standard deviation (SD) and variation coefficient values for the microtensile bond strength test of In-Ceram and Panavia F

Group	n	(MPa)	(SD)	(VC)
Group I (no thermocycled)	10	25,628	± 3,353	13%
Group II (thermocycled)	10	22,815	± 5,254	23%

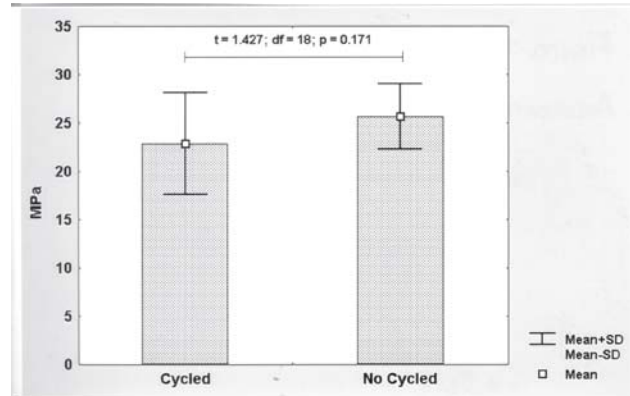


FIGURE 7- Graphic showing results of tensile strength of bonding to In-Ceram ceramic after different storage technics

DISCUSSION

Laboratory thermocycling is the simulation of the physical process in the oral environment that more frequently influences the integrity of the bonding union between restorative materials and luting agents. Chang⁵ verified that several bonding systems are influenced by cyclical variations of temperature. Due to this fact, the present work evaluated the effect of the thermocycling on the bond strength between a glass-infiltrated alumina ceramic surface conditioned with a sandblasting with silica powder (silicoating) and a resin luting cement containing in its composition phosphate monomer. However, other methods of superficial etching, have not been compared in the present study because it was based on studies that report more efficient results of the surface treatment by silicatization (tribochemical silica coating - Rocatec) of the glass-infiltrated alumina ceramic^{3, 12, 14, 15, 20, 21}.

Pape²² and Kraivixien-Vongphantues¹⁶ reported that bond strength of resin luting cements to In-Ceram surface is not appropriate when procedures are used to condition feldspathic ceramics, such as, acid etching adding silane coating, because in the In-Ceram system acids do not promote similar micromechanical retentive surface like on the conventional ceramics²⁵. Some authors^{11, 12} suggested a surface treatment with sandblasting of aluminum oxide 110mm (Al₂O₃) to

create micromechanical retention in the surface of the aluminous ceramics, however only sandblasting doesn't promote effective chemical bond with the silane agents, because of the low amount of silica present in the glass matrix of the In-Ceram ceramic system^{11, 25}.

In spite of these surface treatment methods presenting a high value of bond strength immediately after the cementation procedures, when submitted to treatments of thermocycling even under storage in larger periods than 30 days in distilled water at 37°C, the values of bond strength decreased to very low levels and do not promote a stable chemical union between ceramic and luting cement¹⁴.

The Rocatec system introduced by Guggenberger⁹ to condition metallic surfaces, was used initially by Neikes²⁰, Kraivixien-Vongphantuset¹⁶ and then by Kern^{12, 14} to condition the glass-infiltrated alumina ceramic surface (In-Ceram), promoting the increase of silica content to 19,7% in weight. This treatment method by sandblasting (silicoating), according to these authors' results, presented values of bond larger than other adhesive methods of superficial treatment mentioned before and higher bond strength between ceramic and BIS-GMA resin luting cements.

In the works above mentioned, the maintenance of the integrity of the bond strength between In-Ceram ceramic and resin luting cements was submitted to thermo cycles in water that varied from 200 to 37500 cycles between 5°C and 55°C with 30 seconds of immersion in each bath. It was however verified in these studies that even in thermal tests with great amounts of cycles the bond strength between aluminous ceramic and BIS-GMA resin luting cements was not significantly altered.

The conditioning of In-Ceram ceramic surface by Cojet-System, presented as results, lower values of bond strength when compared to previous studies. However, in the present study the results demonstrated that the values between the group I and group II were not altered in a significant way by the effect of the thermocycling. There was a stability of the adhesive unions, corroborating with the results found by Kern¹⁴. A fact to be considered in the author's studies^{14, 16, 21} mentioned above is that they did not link comparative values between numbers of thermal cycles and time in oral environment so, our work was based on Leibrock¹⁷ that establishes an approximate relationship of these values. Based on this fact, the use of 1500 thermal cycles would be similar in physiologic normal conditions during the period of 1 and a half of year. So the numbers of cycles used in the methodology was considered reasonable.

Regarding the values of bond strength obtained with the conditioning of the In-Ceram surface with the Cojet-System had been lower than the presented in other works¹⁴ that evaluated the conditioning with the Rocatec system, we believed that such fact could be explained because Panavia-F cement's composition differs by it containing phosphate-monomer and not BIS-GMA as in the resin luting cements used in other works. The fact that the mean values obtained for groups I and II have not been statistically different, indicates that thermocycling did not compromise the stability of the bond strength between ceramic and resin cement. Other fact to consider is the method used for measuring bond strength because in our study we verified adhesive failures only, while in other methods it was observed cohesive failures of the materials, so the results could not supply a real evaluation of the bonding strength between restorative materials and luting cements^{23, 24}. On the limitations of our study, that just evaluated one method of conditioning the glass-infiltrated alumina ceramic, we suggest that other works must be developed using the microtensile methodology to verify the values of bond strength created by other surface treatments as the Rocatec system and resin luting cements containing in its composition BIS-GMA. Such tensile method is suggested because we think it can be obtained trustful results of adhesive failures.

CONCLUSION

The results obtained demonstrated that the thermocycling technique here used had not produced statistically significant differences between the bond strength results of the specimens obtained by the two techniques employed in the present experiment.

RESUMO

O objetivo deste estudo foi avaliar o efeito da ciclagem térmica sobre a resistência de adesão entre a superfície da cerâmica In-Ceram Alumina (VITA) e o cimento resinoso Panavia F (Kuraray). Foram confeccionados quatro blocos de cerâmica In-Ceram com dimensões de 5x6x6mm. Uma das faces com 6x6mm de cada bloco cerâmico, após condicionamento com o sistema Cojet (ESPE-3M) (jateamento com óxido de alumínio/jateamento com óxido de sílica/ silanização) foi cimentada com Panavia F, sob peso constante de 750g, a outro bloco idêntico de resina composta Clearfil AP-X (Kuraray).

Os blocos de resina foram obtidos por meio de duplicação daqueles de cerâmica a partir de moldes com silicão de adição Express (3M). Os quatro conjuntos formados por cerâmica, cimento e resina foram seccionados em 20 corpos-de-prova com forma de palitos, de modo que a região adesiva apresentasse 1mm² de área. Dois grupos (n=10) foram constituídos: G1- estocagem por 7 dias em água deionizada à 36 ± 2°C; G2- 1500 ciclos entre 5°C e 55°C com intervalos de 30 segundos. A seguir, foi realizado o teste de microtração em máquina de ensaio universal (EMIC) com velocidade de 0,5 mm/min. Os resultados mostraram que os valores médios de tensão de ruptura (MPa) para o grupo G2: (22,815 ± 5,254) não tiveram diferenças estatisticamente significantes daqueles do grupo G1: (25,628 ± 3,353) (t= 1,427; gl = 18; p-valor = 0,171), ao nível de significância de 5%. A partir destes resultados, entendemos lícito concluir que o efeito da ciclagem térmica não produziu alterações estatisticamente significantes nos valores da resistência adesiva.

UNITERMOS: Cerâmica dental; Cimentos resinosos; Abrasão dental por ar.

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