

STUDY OF THERMOCYCLING EFFECT ON THE BOND STRENGTH BETWEEN AN ALUMINOUS CERAMIC AND A RESIN CEMENT

ESTUDO DO EFEITO DA CICLAGEM TÉRMICA NA RESISTÊNCIA DA UNIÃO ADESIVA ENTRE UMA CERÂMICA ALUMINIZADA E UM CIMENTO RESINOSO

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ABSTRACT

his study evaluated the effect of thermocycling on the bond strength between Procera AllCeram (Nobel-Biocare) and a resin cement (Panavia F, Kuraray CO). Nine ceramic blocks with dimensions of 5x6x6mm were conditioned at one face with Rocatec System (Espe). After, they were luted with Panavia F to composite resin blocks (Clearfil AP-X, Kuraray CO). The nine groups formed by ceramic, cement and composite resin were split up obtaining 75 samples with dimensions of 12x1x1mm and adhesive surface presenting $1mm^2\pm0.1mm^2$ of area. The samples were divided into 3 groups (n=25): G1 - 14 days in distilled water at 37° C; G2 - 6,000 cycles in water (5° C - 55° C - 30s); G3 - 12,000 cycles in water (5° C - 55° C - 30s). The samples were tested in a universal testing machine (EMIC) at a crosshead speed of 1mm/min. Data were analyzed by ANOVA and Tukey tests. The results indicated that mean values of rupture tension (MPa) of G1 (10.71 ± 3.54) did not differ statistically (p <5%) from G2 (9.01 ± 3.90), however there was statistical difference between G1 and G3 (7.28 ± 3.00). It was concluded that thermocycling significantly reduced the bond strength values when samples were submitted to 12,000 cycles. Uniterms: Ceramics; Resin cements; Tensile strength.

RESUMO

E ste trabalho avaliou o efeito da ciclagem térmica sobre a resistência adesiva entre a cerâmica aluminizada (Procera AllCeram, Nobel-Biocare) e um cimento resinoso (Panavia F, Kuraray CO). Nove blocos de cerâmica, com dimensões de 5x6x6mm, foram condicionados em uma de suas faces com o Sistema Rocatec (ESPE). A seguir foram cimentados a blocos de resina composta (Clearfil AP-X, Kuraray CO). Os conjuntos cerâmica-cimento-resina foram cortados em 75 corpos-de-prova com formato retangular com dimensões de 12x1x1mm e superfície adesiva apresentando 1mm² ± 0,1mm². Os corpos-de-prova foram divididos em três grupos (n=25): G1 - 14 dias em água destilada a 37°C, G2 - 6000 ciclos em água (5°C e 55°C) e G3 - 12000 ciclos em água (5°C e 55°C). Os corpos-de-prova foram ensaiados sob velocidade de 1mm/min em máquina de ensaio universal (EMIC). Os dados foram tratados estatisticamente com os testes de Anova e Tukey e indicaram que os valores médios de tensão de rupturas (MPa) de G1 (10,71 ± 3,54) não diferiram estatisticamente (p < 5%) de G2 (9,01 ± 3,90), porém houve diferença significativa entre G1 e G3 (7,28 ± 3,00). Concluiu-se que a ciclagem térmica diminuiu a resistência adesiva significativamente quando os corpos-de-prova foram submetidos a 12000 ciclos.

Unitermos: Cerâmica; Cimentos de resina; Resistência à tração.

INTRODUCTION

Metal-ceramic fixed partial dentures (FPD) have been for many years used with satisfactory clinical results. However, some factors can compromise and limit its esthetic indications, as the presence of metallic framework, which demands an opaque application under the porcelain and removes the vitality aspect of the prostheses. Besides, the metallic cervical margins can provide a grayish area near to periodontal adjacent tissues, compromising the esthetic result of the rehabilitated dental element.

Nowadays, one of the ceramic systems that allows the making of FPD without metallic base is Procera AllCeram (Nobel Biocare) that uses the CAD-CAM technology (Computer Assisted Design - Computer Assisted Machining) to produce a framework constituted by sintered aluminum oxide densely compacted with 99.5% of purity. With this system, the ceramic framework can present values of flexural resistance close to 687MPa, providing favorable mechanical conditions to make unitary and fixed partial dentures, besides highly esthetic results ^{11,29}.

Adhesive resistance between conventional feldspathic ceramic and luting agents, mainly resin cements, is increased when the internal surface of prosthetic pieces is previously conditioned by acid etching following by application of a silane agent. The acid etching of those surfaces promotes a surface with micro-retentions and exhibition of silica crystals that, together with silanization, increase the chemical union with resin cements, resulting in elevated and stable values of adhesive union ^{18, 27}.

On the other hand, the increase of alumina concentration in aluminous ceramic composition significantly reduces the silica content, making the conditioning procedures with acid substances contraindicated, because such procedure does not create micro-retentions as occurs with the feldsphatic porcelains^{3, 16, 20}.

In this way, some researches tried to develop alternative methods to condition the surfaces of alumina ceramics in order to promote larger and stable values of adhesive union. One of those systems is Rocatec (ESPE), which promotes silica coating on the ceramic surface, allowing its adhesion to silane agents and resin cements.

In 1995, Kern and Thompson¹⁵ used the Rocatec system for the first time as an option to etch ceramic surfaces with high alumina concentrations, as In-Ceram system, creating a silica layer that presents larger affinity with the ceramic surface, silane agent and BIS-GMA resin cements. In the literature, there are few defined methods to treat the ceramic surface of Procera AllCeram system that provides clinically acceptable values of bond strength.

This study aimed at evaluating, using microtensile methodology, the thermal cycling effects on the bond strength between a resin luting cement containing phosphate monomer (Panavia F, Kuraray CO. - Japan) and the surface of an alumina ceramic conditioned with silica coating system (Rocatec).

MATERIAL AND METHODS

Three blocks of alumina ceramic were used (Procera AllCeram) with dimensions of 15x6x6mm, obtained by CAD-CAM technology (Computer Assisted Design - Computer Assisted Machining) that makes frameworks constituted of sintered aluminum oxide. The production of the ceramic material is made by a scanner coupled to a computer that reads the surface of a matrix and develops a three-dimensional draw. The images are sent by modem for Procera Sandvik AB, in Stockholm, Sweden, where the material is made. An acrylic block with dimensions of 15mm of length, 6mm of height and 6mm of thickness was used as matrix to obtain the scanned images for Procera Scanner (Nobel Biocare).

As quality control, the ceramic blocks were radiogaphed to verify if there were bubbles inside them. The presence of bubbles led to disposal of the block.

Each ceramic block was split up, forming three small blocks with dimensions of 5x6x6mm each.

One of the faces of each ceramic block, with dimensions of 5x6mm, was planed out with water sandpapers with granulation of 300, 600, 800, 1000, 1200 to create a plane and uniform surface. Afterwards, each ceramic block was molded with addition silicone (Express - 3M Dental Products, St. Paul, MN - USA), in order to obtain an impression. This impression was used to make the blocks of resin composite (Clearfil AP-XTM, Kuraray CO. - Japan) that were used to allow construction of the specimen. The blocks of composite resin presented one of the faces with similar characteristics to the planed faces of the ceramic blocks, providing a good contact between them. After impression, the planed faces of the ceramic blocks were conditioned with Rocatec system (ESPE, Seefeld - Germany), that comprises initial sandblasting with 110µm aluminum oxide particles (Rocatec-Pre), for 20 seconds with pressure of 2.8 bars, at a 10mm standard distance perpendicular to the surface, followed by further sandblasting with 30µm silica particles (Rocatec-Plus), which promotes formation of the silica layer, and at last, application of the silane coating (Rocatec-Sil).

The ceramic blocks were cemented to the resin blocks with resin cement (Panavia F, Kuraray CO. - Japan), manipulated according to the manufacturer's recommendations and applied on the surface of the two blocks. The set was positioned in a device to promote cementation under a constant 750g load for 10 minutes.

The excess cement was removed with an appropriate brush, followed by light curing (XL 3000 - 3M Dental Products, St. Paul, MN - USA), with a light intensity of 450mW/cm². The light was applied for 40 seconds at each side of the set, followed by application of an oxygen inhibitor agent (Oxyguard, kuraray CO. - Japan), for 5 minutes on all faces. After curing of resin cement, the sets were washed with an air-water spray and stored in deionized water at 37°C.

Nine sets of ceramic blocks cemented to resin blocks were obtained, which were fixed with cyanoacrylate in a lathe adapted by Andreatta et al 1 to accomplish precision

cuts with diamond disks measuring 0.15mm in thickness and 22mm in diameter.

The sets had 0.5mm of their external aspects sectioned to eliminate cement excesses that could change the final bond strength values. Afterwards, the sets were cut in slices of 12x6x1mm of area.

At last, each slice was fixed in the lathe for accomplishment of new cuts to obtain the samples.

From the nine sets of cemented blocks, 75 samples were randomly obtained with parallelogram form, with 12mm of length and square transverse section with 1mm \pm 0.01mm² of adhesive area. Three groups were established with 25 samples (n=25) each , varying the type of thermal treatment. Group 1 was not thermocycled and the samples were stored for 14 days in deionized water at 37°C. In Group 2, the samples were submitted to 6,000 thermal cycles in baths at 5°C and 55°C, with 30-second dwell times. In Group 3, the samples were thermocycled 12,000 times in baths at 5°C and 55°C, with 30-second dwell times (Table 1).

After thermal treatments in a thermal cycling machine (Model 521-4D - Nova Ética Ind., Com. and Serv Ltda, São Paulo Vargem Grande - SP, Brazil), each sample was fixed with cyanoacrylate sticker (Super Bonder) to the modified caliper for accomplishment of microtensile tests. In this method, the applied force is perpendicular to the long axis of the specimen, avoiding strain forces and shear of the adhesive area. Each sample fixed to the caliper was adapted to the universal testing machine (DL-1000, EMIC - Equipments and Systems Ltda, São José dos Pinhais - PR, Brazil) and loaded to failure under tension at a crosshead speed of 1.0mm/minute. After testing, the fracture modes of each sample were determined by examination in a stereoscopic microscope at 18x magnification (Tecnival Carl Zeiss - JENA).

TABLE 1- Groups and thermocycling treatments

Group	Number of samples	Thermocycling	
G1	25	Stored for 14 days in deionized water at 37°C	
G2	25	6,000 cycles/5-55 °C - 30s	
G3	25	12,000 cycles/5-55 °C - 30s	

Statistical analysis

The data (MPa) were statistically tested to verify the differences between groups. Anova and Tukey tests at 0.05 of significance were used.

RESULTS

The means and standard deviations of bond strength data (MPa) obtained in the microtensile test are presented in Table 2.

It was verified that standard deviation values were close and did not statistically differ (Bartlet test: $X^2 = 1.61$; df = 2; p = 0.446>0.05).

The data were analyzed by one-way ANOVA, at α = 0.05. This parametric test indicated that the bond strength values had significant difference between experimental groups (ANOVA, $F_{df(2:72)}$ = 5.99; p = 0.004<0.05).

Tukey test ($\alpha = 0.05$) showed that the bond strength mean of group 1 (10.71 ± 3.54) was statistically higher (p<0.05) than group 3 (7.28 ± 3.00). Bond strength mean of Group 2 (9.01 ± 3.90) did not differ from group 1 and group 3.

Regarding the fracture modes, all samples showed adhesive failures when observed at 18x magnification in stereoscopic microscope.

DISCUSSION

Nearly one decade after its development, the microtensile methodology is still researched with the purpose of determining a pattern of specimen dimensions. In that way, based on studies of Sano, et al.²² (1994); Shono, et al.²³ (1997); Bianchi ⁴ (1999); Pashley, et al.²¹ (1999); Shono, et al. ²⁴ (1999), Della Bona, et al.⁸ (2000), microtensile methodology was used to make specimens with transverse section areas of approximately 1mm², fixed in the test device (caliper) with cyanoacrylate adhesive.

In the present study, methods of superficial conditioning were not compared, because the study was based on studies that reported more efficient results for conditioning of the alumina surface with sandblasting, mainly with aluminum oxides and silica, because those promoted larger values of adhesive resistance than the acid conditioning used in conventional feldspathic ceramics.

Kraivixien-Vongphantuest, et al. 16 (1992) and Kern and

TABLE 2- Descriptive statistics (mean, standard deviation and coefficient of variation) for data (MPa) obtained in the microtensile test

Statistics	Groups		
	No thermocycling	6,000 cycles	12,000 cycles
Mean (MPa)	10.71	9.01	7.28
SD	3.54	3.90	3.00
CV (%)	33.05	43.33	41.29

Thompson ¹³⁻⁴ (1994) reported that the adhesive union between resin cements and the In-Ceram surface was not appropriate when acid etching and silanization were performed, because the acids do not provide formation of micro retentions similar to those that occur in conventional feldspathic ceramics ^{7,27}.

Some authors $^{15,\,20}$ suggested conditioning with $110\mu m$ aluminum oxide sandblasting (Al $_2$ O $_3$) to create micro retentions in the surface of the alumina ceramic; however, it was verified that only with that conditioning there is no chemical union with the silanization agents, due to the low amount of silica present in the glass of In-Ceram ceramic matrix $^{13\text{-}4}$.

Even though those conditioning methods presented a high value of adhesive resistance immediately after cementation procedures when submitted to thermal cycling or even under storage in larger periods than thirty days in distilled water at 37°C, the values of adhesive resistance decreased at very low levels, not promoting a stable union between ceramic and cement.

The Rocatec-ESPE system, used to condition metallic surfaces with good results of adhesive union¹², was initially used by Kraivixien-Vongphantuset, et al.¹⁶ (1992) and Neikes, et al.¹⁹ (1992) and later by Kern and Thompson¹³⁻⁴ (1994, 1995) to condition the surfaces of alumina ceramic (In-Ceram), promoting the increase of silica content to 19.7% in weight by the tribochemical covering of the ceramic surface. That conditioning method, according to those authors' results, presented bonding values larger than other adhesive methods of superficial conditioning, besides a stable adhesive union between ceramic and BIS-GMA resin cements.

The adhesive union between resin cement and phosphate monomer (Panavia F) and the surface of the In-Ceram ceramic conditioned with the Rocatec system was evaluated by Soares, et al. ²⁵ (2002). This study showed values significantly greater than the association of that cement with surfaces sandblasted only with 110µm aluminum oxide; however, the effect of thermal cycling was not evaluated. Andreatta, et al. ² (2002), Galhano, et al. ¹⁰ (2002), evaluating the effect of 1,500 thermal cycles on the adhesive union between surfaces of In-Ceram conditioned with silica coatings (Cojet and Rocatec systems) and Panavia F resin cement, obtained similar results with stable adhesive union, even after thermal cycling.

Although for the In-Ceram alumina ceramic the combination of superficial treatment with silica covering (Rocatec) and resin cement (BIS-GMA) has promoted a satisfactory and durable adhesive union, Friederich and Kern⁹ (2002) reported that, under the thermal cycling effect, the adhesive union between Procera AllCeram's surface conditioned with Rocatec system and a resin cement (BIS-GMA) was not stable. In that same study, higher values of adhesive resistance were obtained for the group in which the resin cement containing phosphate monomer (Panavia 21 EX) was used with Procera AllCeram's surface conditioned with aluminum oxide sandblasting (110µm). The authors attributed the higher values of that group to the differences in the processes to make the ceramic, because for the Procera AllCeram system the glass infiltration is not performed as for the In-Ceram system, and the glass infiltration could be necessary for formation of the silica layer by sandblasting with Rocatec system.

Blixt, et al.⁵ (2000) obtained favorable results of adhesive union associating conditioning of Procera AllCeram's surface with the Rocatec system and resin cement (BIS-GMA); however, the storage and thermal cycling effect was not verified.

Regarding the conditioning of Procera AllCeram surface with acids, Awliya, et al.³ (1998) verified that this method, in agreement with studies of Madani, et al.¹⁸ (2000), did not increase the values of adhesive union. With scanning electron microscope, the authors verified that Procera AllCeram's surface after conditioning with hydrofluoric acid (9.6%) maintained the same morphological structure.

However, studies were not found in the literature that evaluated the thermal cycling effect on the adhesive union between resin cement containing phosphate monomer (Panavia F) and the surface of Procera AllCeram ceramic conditioned with the Rocatec system.

Based on Leibrock et al.¹⁷ (1999), 6,000 and 12,000 thermal cycles were used between 5 and 55°C, that is similar, in physiologic normal conditions, approximately to the period of five and ten years, respectively.

The results of the present study demonstrated that thermal cycling negatively influenced the quality of adhesive resistance, promoting the decrease in union values when the number of thermal cycles was increased.

The negative effect of thermal cycling on the adhesive union can be explained by the fact that materials with different lineal thermal expansion coefficients (LTEC) also presented different degrees of shrinkage and expansion. That process promotes the fatigue phenomenon of the materials, leading to rupture of the union between them⁶. The LTEC of conventional resins ranges from 25 to $35 \times 10^{-6} \text{K}^{-1}$, and for alumina ceramics this value is approximately $6.6 \times 10^{-6} \text{K}^{-1}$. Therefore, the effects of temperature variations when those two materials are associated can lead to fatigue of the adhesive union.

According to Söderholm and Roberts²⁶ (1990), Wegner et al.²⁸ (2002), the storage in water and thermal cycling can alter the properties of resin materials, because they contain components (matrix and inorganic particles) with different LTEC and can suffer fatigue under thermal variations that accelerate their structural weakness, promoting union flaws.

Another factor that might have contributed to the decrease in the adhesive resistance values was the sample dimension that had a small area, receiving larger influence of thermal cycling effects on its surface. Shono, et al.²⁴ (1999) verified that adhesive unions in samples with different dimensions presented reductions of bond strength in samples with small areas, after similar periods of storage and thermal cycling.

It should be considered that the methodology and the variables in the present study did not simulate all clinical conditions happening in masticating physiologic processes. It is believed that the use of traditional adhesive resistance data should be restricted to comparison of effects from properties and microstructure of materials and from treatment conditions that could improve the fracture resistance, and

not to accomplish categorical clinical interpretations about resistance union.

CONCLUSION

With the results of this study, it can be concluded that thermal cycling with 6,000 cycles did not influence the resistance values of the adhesive union between aluminous ceramic (Procera AllCeram) conditioned with Rocatec System and resin cement (Panavia-F). However, 12,000 thermal cycles significantly decrease the adhesive resistance in relation to the group that has not been submitted to thermal cycling.

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