



RENATO ABDALLA PRATA

**The resin cement bond strength to lithium
disilicate ceramic can be affected by Try-
in paste removal method?**

Trabalho apresentado ao programa de Mestrado em Odontologia da Universidade de Uberaba - UNIUBE, para a obtenção do Título de Mestre em Odontologia - Área de concentração em Biomateriais.

Orientador: Prof. Dr. Fernando C. Hueb de Menezes

Co-orientador: Prof. Ms. Luciano de Souza Gonçalves

**UBERABA
2011**

AGRADECIMENTOS

Agradeço a DEUS pela oportunidade de realização e conclusão deste curso.

À minha querida e amada esposa e companheira Flávia, pelo apoio e compreensão nos momentos em que estive ausente.

Aos meus amados filhos Felipe e Rodrigo, por serem fontes constantes de energia e inspiração.

Aos meus queridos pais, Paulo e Maria Helena, que sempre me apoiaram em todas as buscas de novas realizações.

Ao meu “irmão” Danilo, parceiro e companheiro de todas as horas.

Ao estimado tio Salem, que tantos quilômetros percorreu por minha causa, para que eu pudesse alcançar este objetivo.

Aos meus padrinhos, Hermes e Maria Sara, à minha estimada tia Maria Marta e às minhas queridas primas Karen e Karine, por me acolherem tão bem como sempre fizeram.

Ao Prof.Dr. Amir Mattar pela presença incentivadora e carinhosa.

A todos os professores e funcionários do curso e, em especial, aos Profs. Drs. Fernando Carlos Hueb de Menezes e Luciano de Souza Gonçalves, pelos ensinamentos, apoio, paciência e expressiva orientação e dedicação, fundamentais para a conclusão deste curso e conseqüentemente na minha evolução profissional.

A todos que de alguma forma, contribuíram para que este sonho em ser MESTRE se realizasse.

SUMMARY

ABSTRACT	1
INTRODUCTION	2
MATERIALS AND METHODS	3
RESULTS	5
DISCUSSION	6
CONCLUSION	8
ACKNOWLEDGMENTS	9
CONFLICT OF INTEREST	10
REFERENCES	11

FIGURES AND TABLES LIST

Figure 1. A) SEM images of lithium disilicate crystals exposed after the hydrofluoric acid etching. (G1). B) Resin cement (RC) and Ceramic (Cm) interface. **13**

Figure 2. SEM images of G2 (A) show a surface topography with the presence of remains of Try-in adhered to lithium disilicate crystals. The bond interface between the resin cement (Rc) and ceramic (Cm) can be seen in image B. For G3, few crystals are observed in C. The SEM interface analysis of G3 shows the present of cracks (pointers) between the resin cement (Rc) and Ceramic (Cm) (D). **14**

Figure 3. Image A shows a large quantity of contamination demonstrated by the ellipses. In Image B small failures are pointed out in the resin cement and ceramic interface . For G5, a large quantity of contamination with a porous aspect was observed in image C. In image D can be seen the separation that occurred at the interface between the resin cement and ceramic. **15**

Figure 4. Distribution of failure mode **16**

Figure 5. In images A (dentin side) and A' (ceramic side) can be seen the adhesive failure between resin cement and dentin (ACD). In images B and B' is shown a failure mixed (FM) with portion of adhesive fracture between dentin and resin cement (Dn) and part of cohesive failure in resin cement (Rc). In the ceramic side (B') can be seen the opposite part of cohesive failure (Rc) and adhesive portion of failure(*). In the images C (dentin side) and C' (ceramic side) adhesive failure mode between ceramic (Cm) and resin cement (Rc) is shown, a small part of dentin can be seen (Dn). The details of this failure mode are observed at higher magnification in images D and E. **17**

Table 1: Groups and respective treatment **18**

Table 2: Push-out Bond strength Values (MPa). **19**

The resin cement bond strength to lithium disilicate ceramic can be affected by Try-in paste removal method?

Renato Abdalla Prata – DDS , Graduate Student of Biomaterials Division, University of Uberaba, Av. Nenê Sabino, 1801, Bairro Universitário, Cep 38.055-50 Uberaba – MG, Brazil

Vinicius Pires de Oliveira – Graduate Student of Dentistry, University of Uberaba, Av. Nenê Sabino, 1801, Bairro Universitário, Cep 38.055-50 Uberaba – MG, Brazil

Fernando Carlos Hueb de Menezes – PhD, Ms, DDS, Professor of Biomaterials Division, University of Uberaba, Av. Nenê Sabino, 1801, Bairro Universitário, Cep 38.055-50 Uberaba – MG, Brazil

Gilberto Antônio Borges - PhD, Ms, DDS, Professor of Biomaterials Division, University of Uberaba, Av. Nenê Sabino, 1801, Bairro Universitário, Cep 38.055-50 Uberaba – MG, Brazil

Oswaldo Scopin de Andrade - PhD, Ms, DDS, Professor of Restorative Dentistry, Senac – Tiradentes, Av. Tiradentes, 822, Bairro da Luz, Cep. 01102-000 São Paulo – SP, Brazil

Luciano de Souza Gonçalves – Ms, DDS, Professor of Biomaterials Division, University of Uberaba, Av. Nenê Sabino, 1801, Bairro Universitário, Cep 38.055-50 Uberaba – MG, Brazil

Correspondence: Prof. Dr. Fernando Carlos Hueb de Menezes, Departamento de Biomateriais, Faculdade de Odontologia da Universidade de Uberaba (UNIUBE), Av Nenê Sabino, 1801, 38055-500, Uberaba, MG, Brasil. Tel: +55-34-3319-8741 - e-mail: fernando.menezes@uniube.br

Abstract The aim of this study was to evaluate four methods of Try-in paste removal from the lithium disilicate ceramic surface before the cementation procedure, and assess their influence on the bond strength to resin luting cement. Slices of dentin 1,6 mm thick were sectioned from the crown of human third molars and truncated cone shape cavities were made with diamond burs. IPS e.max Press ceramic blocks were obtained for each cavity, etched with hydrofluoridric acid and fixed with temporary paste (Try-in, Ivoclar-Vivadent), except for Group G1 cemented according to manufacturer's instructions. Try-in paste was removed from the experimental groups using different techniques: G2, ultrasonic bath in distilled water (5 min.); G3, air/water spray (1min.); G4, etched with phosphoric acid (2 min) + air/water spray (30s) and G5, etched with phosphoric acid before and after hydrofluoridric acid application + air/water spray (30s). The ceramic restorations were cemented with the same procedures as in G1. Push-out bond strength test (MPa) was performed in a universal testing machine with a crosshead speed of 0.5mm/min. One way ANOVA and Tukey's test ($p < 0,05$) was applied: G4(19.21)a, G2(17.09)ab, G1(16.82)ab, G3(16.18)ab and G5(15.9)b. The results showed that all the experimental groups showed remaining particles of Try-in paste. The bond strength in G4 was statistically higher than in G5. In the other groups no significant difference was shown. In G5 there was predominance of adhesive failures between resin cements and ceramics. In conclusion, the techniques used were not totally effective at removing the paste Try-in, however the presence of remaining material that did not influence the bond strength of restorations. It was observed that the methods of the temporary paste removal may affect the bond strength of ceramic restorations.

Keywords Lithium disilicate ceramic · Cleaning · Contamination · Push out bond strength · SEM

Introduction

Dental offices have experienced a growth in the number of patients seeking enhanced esthetic appearance of the smile, which has encouraged the development and improvement of materials, allowing the professional to select the most suitable option for each case. In this respect, adhesive techniques have become necessary in practically all restorative procedures [1-3]. These techniques allied to the adhesive technique have made it possible to implement a conservative philosophy with regard to preparations, particularly in the anterior regions, where esthetics is of primordial importance. Therefore, the indication of indirect restorations that require planned and controlled wear may be much more effective in terms of esthetics and durability [4,5].

Dental ceramics have characteristics capable of mimicking the natural structures present in the tooth, and can be indicated in the majority of restorative treatments. In addition to being chemically stable, they present excellent optical properties when applied to dental structures, thus guaranteeing their outstanding place among the esthetic restorative materials [6,7]. Historically, the low strength and high friability of ceramics made them dependant on a metal reinforcement, and over the years, these have been replaced by metal-free reinforced ceramics. From this aspect, the indication of laminated ceramics reinforced with lithium disilicate may be pointed out as being outstanding among the esthetic restorative procedures [8-10].

The adhesive cementation systems have been considered the best option and the application of low viscosity resins on prepared teeth has demonstrated good biomechanical behavior, particularly in ceramic restorations [2,11-13]. Nevertheless, with the advent of more conservative preparations, the cement shade has a direct influence of the end result of the restoration. In many cases, the laminated ceramic is less than 1 mm thick and the end result depends on the shade of the substrate, shape and anatomy of the teeth, restoration thickness, and finally, on the cement [14-18].

In order to obtain a satisfactory esthetic result, the clinician may use the so-called Try-In pastes that simulate the final color of the restoration [19]. According to the manufacturer, these pastes contain water soluble glycerin, with a load of mineral elements and coloring agents, and when applied before definitive cementation, allow the professional to evaluate the resultant shade of the restoration, considering the color of the substrate, cement color, and the translucence of the ceramic, providing an adequate esthetic perspective of the work [18]. The use of Try-in pastes, accompanied by resin cements, allow both the dentist and patient to evaluate the shape and shadow of the restoration, in order to

guarantee the esthetic expectation will be achieved, making it an important artifact to estimate the end result of the restorative work [19]. However, the application of these agents may lead to a possible internal contamination of the ceramic, and interfere in the bond strength of the restoration. An effective bond largely depends on the condition of cleanliness of the surfaces [20]. Therefore, removal of these contaminants before ceramic cementation is a critical procedure for the long term clinical success of the restoration [21]. Various methods such as the use of an ultrasonic bath or acid agents have been used to obtain decontaminated surfaces, allowing a better bond quality [20,22], however, without there being any conclusive result about their efficiency.

Thus, the aim of this study was to evaluate the surface cleaning techniques after the use of a Try-in paste, and to verify their influence on bond strength to lithium disilicate-based ceramic restorations. The null hypotheses in the present study suggested that: i) there would be no difference among the techniques for removing the temporary Try-in cementation paste from the surface of the lithium disilicate-based ceramic surface. ii) The use of the temporary Try-in cementation paste would not interfere in the bond strength between the ceramic and dentin.

Materials and Methods

The study was approved by the Research Ethics Committee of the Dental School of the University of Uberaba, Brazil (Protocol **CAAE-0009.0.227.000-09**). Extracted sound human third molars were immersed in distilled water at 4°C and used within 4 months after extraction.

Dentin preparation

Slices of dentin were sectioned from crowns of human third molars with a water-cooled diamond saw (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA). The slices were embedded in epoxy resin (Buehler) and wet-ground with 600-grit SiC abrasive papers to obtain a flat surface only in dentin. The slices were standardized to 1.6 mm (± 0.1 mm) using a digital caliper (Mitutoyo Corporation, Kawasaki, Japan) with an accuracy of 0.01 mm. Cavities were prepared with #3131 diamond burs (KG Sorensen, Barueri, SP, Brazil) at high-speed, under air/water cooling using a custom-made preparation device allowed standardization of the cavity dimensions: widest diameter of 2.3 mm, smallest diameter 1.9 mm and height 1.6 mm.

Ceramic block production and cementation process

After the cavity preparations, the impression was taken with a polyvinyl siloxane material (Virtual - Ivoclar-Vivadent, Liechtenstein) and reproduced in dental stone type V Exadur (Polidental, Cotia, SP, Brasil) to obtain the wax pattern. The e.max Press ceramic blocks (Ivoclar-Vivadent, Schaan, Liechtenstein) were fabricated by the heat-pressed ceramic procedure following the manufacture's instructions.

Ceramic restorations were etched with hydrofluoridric acid (IPS Ceramic Etching Gel - Ivoclar-Vivadent, Liechtenstein) for 20 s, rinsed with distilled water in an ultrasonic bath for 5 min and air-dried. The specimens were divided into five groups (n=10) and treated as shown in Table 1. After the Try-in removal procedures, the silane agent was applied for 15 s and air-dry for cementation. In the all groups the dentin was etched with 37% phosphoric acid (Total Etch - Ivoclar Vivadent) for 60 s, rinsed for 15 s and dried using a cotton pellet. A one-bottle adhesive system (Excite – Ivoclar Vivadent) was applied according the manufacturer's instructions and photoactivated for 10 s and 1200 mW/cm² with a LED light source Radium-cal (SDI Ltd. - Bayswater, Victoria, Australia). Dual curing resin cement Variolink II (Ivoclar Vivadent) was mixed and inserted in the cavity and the ceramic blocks were placed under static load (0.454kgF for 1 min.). The excess cement was removed with a disposable microbrush. A LED Radium-cal source (SDI Ltd.), 1200 mW/cm², was used to expose the specimen surfaces to two 40s photoactivations, one from top and other from bottom surface. The specimens were stored in 100% relative humidity at 37°C, for 24 h.

Push out bond strength test and Scanning Electron Microscope (SEM) Analysis

The push out test was performed in a universal testing machine (EMIC DL 3000, Emic, São José dos Pinhais, PR, Brazil). A metal cylinder with a central orifice adapted to the machine was used as the base for the specimens. Each specimen was placed on the cylinder with the larger base of its cavity against the orifice on the metal cylinder surface. The small base was then loaded with a 1,9 mm diameter cylindrical plunger (touching only the ceramic base) at a cross-head speed of 0.5mm/min until failure of the bond on the lateral walls between the ceramic and cavity. The load was recorded by the testing machine and used to calculate the push-out force (PF) in N as follows: $PF=(Lg/A1)$, where L is the fracture load (kgf), g the acceleration of gravity (9.8 m/s²) and A1 the lateral area of the ceramic cone (mm²). The push-out bond strength data were submitted to one-way ANOVA and Tukey's test ($p>0.05$).

After testing, the fractured specimens were coated with gold and examined by SEM (JSM 5600LV, Jeol Inc., Peabody, MA, USA) in order to classify their failure modes. For the interface analyses the SEM examinations were performed as described before the bond strength tests. After storage the specimens were longitudinally sectioned through the middle portion of the ceramic cone base and the specimens were embedded cross-sectionally in epoxy resin (Buheler). After 24 hours (resin polymerization) , the specimens were wet-polished with 600-, 1200- and 2000-grit SiC paper, and then polished with 3 μm , 1 μm and 0.5 μm Al_2O_3 solutions. The cross-sectional profiles were examined by SEM, focusing on integrity, homogeneity and continuity along the bond interface.

In order to observe the topography of the surfaces and presence of Try-in paste contamination, etched and non-cemented specimens (n=3) for each cleaning procedure and control were coated with gold and examined by SEM.

Results

SEM images of topography of surfaces and ceramic/cement bond interface are shown in Figures 1, 2 and 3. For the G1, the dissolution of the vitreous phase was observed, with complete exposure of the lithium disilicate crystals (Figure 1A) providing an adequate substrate to cement adhesion resulting in high quality cement interface (Figure 1B). In the other groups (G2 to G5), the presence of contamination and the cross-sections of the ceramic-cement interfaces can be seen in the Figures 2 and 3.

The mean Push-out bond strength values are shown in Table 2. The G4 means were statistically higher than G5. The other groups did not show statistically difference. The failure mode distributions obtained for the groups are shown in Figure 4. The adhesive failures between cement and dentin (ACD) had predominance in G1 and G2. Mixes failures between cement and dentin (MIX) were predominant in G3 and G4. In G5 was the only one group that shown adhesive failure between ceramic and resin cement (ACC). Detail on the failure modes are shown in the Figure 5. Adhesive failure can be seen in the Figures 5A (dentin side) and 5A' (ceramic side) respectively. The Figure 5B shows the mixed failure. ACC is shown in the Figure 5C (dentin side) and Figure 5C'(ceramic side). Details of this failure are shown in the Figures 5 D and 5E.

Discussion

Bonding ceramic to dental structures by means of the application of resin adhesive systems, increases the fracture strength of the tooth and restoration, in addition to minimizing minicrack formation, a determinant factor in treatment success or failure [23]. However, contaminants on the internal surface of the material may diminish the bond strength and must be removed from the surface before definitive cementation [24]. In the present study, all the methods for removing the temporary cementation agent Try-in were not shown to be efficient, as the presence of residues of the material were left behind. Nevertheless, the presence of remnants of the material did not interfere in the bond strength results of all the groups, as Group G4 results were statistically superior to those of G5. Therefore, the null hypotheses of the study were partially rejected.

In Figure 1, note the ideal condition for the bond performed in G1 as recommended by the restorative material manufacturer, and without the application of the temporary cementing agent Try-in. The application of hydrofluoric acid for 20 s removed the vitreous matrix exposing the lithium disilicate crystals, leaving the surface free of impurities, which favored the adhesive procedure, as has been demonstrated in previous studies [12,25,26]. The time of 20 s for etching ceramics reinforced with lithium disilicate is recommended by the manufacturer, although removal of the vitreous matrix did not affect the integrity of the crystal reinforcement, as seen in Figure 1A. This condition allowed the formation of an interface without interrupting the continuity between the resin cement and ceramic, thus contributing to no gap formation between the materials (Figure 1B).

Ultrasonic cleaning bath for 5 min. (G2) (Figure 2A) partially removed the temporary cementation paste, nevertheless, residues of the material remained adhered to the lithium disilicate crystals. The vibration promoted by the ultrasonic bath appears to have fragmented the Try-in residues, particularly when compared with the other experimental groups, which presented plates of the material. The bath time of five minutes was based on cleaning ceramic test specimens in a previous study [27], however, this time seems too short for the complete removal of Try-in although this was composed of water soluble glycerin, as informed by the manufacturer. However, the presence of spaces could be perceived between the crystals, thus allowing the interaction between the resin cement and ceramic, and not affecting the bond strength. This resulted in a bond interface similar to that observed in Group G1, continuous and without the presence of gaps. The cement ceramic bond interface of G2 can be seen in Figure 2B.

The samples submitted to cleaning with a jet of air/water (G3) presented a larger amount of contamination, resulting in the formation of plates with a porous aspect, with the exposure of few lithium disilicate crystals (Figure 2C). This shows that the mechanical action of the jet of air/water was less efficient than the ultrasonic bath for removing Try-in. Although the difference in surface topography was perceptible, this difference was not reflected in the bond strength values, although the analysis of the interface of this group demonstrated the presence of gaps between the cementing agent and ceramic (Figure 2D). This leads one to believe that even with less interaction between the cement and ceramic, the bond between dentin and resin cement was still superior, which was confirmed by the high rate of adhesive fractures between the resin cement and dentin found in this group.

Although the manufacturer recommends these methods for removing the temporary cementation paste, no bath time or any form of application of water or any other solvent was informed in their recommendations, in spite of the manufacturer itself emphasizing that the presence of residues could affect the bond of the material.

Some studies have shown that the use of phosphoric acid could be efficient for cleaning and decontaminating ceramic surfaces before cementation [20,24]. In the present study, the use of phosphoric acid provided distinct results both as regards the surface topography and the bond strength. Figure 3A shows a large quantity of contamination demonstrated by the ellipses. The partially exposed crystals present small porosities (white arrows). The porosities may have resulted from the exposure to acid, since in G1, G2 and G3 this was not observed. This corroborates a previous study that showed irregular etching patterns when a leucite-based ceramic was submitted to different time intervals of hydrofluoridric acid application [12]. This may also explain the small failures pointed out in Figure 3B. The bond strength in this group did not differ statistically from G1, G2 and G3, however, it was statistically higher than that of G5, which also did not differ from the others. The number of mixed failures in G4 was similar to that of G3, which also presented failures at the interface, however the bond strength to dentin still appears to be the weakest link as regards the bond to ceramic, equaling the bond strength values.

For G5, a large quantity of contamination with a porous aspect was observed in Figure 3C. The crystals pointed out by the white arrows present a large number of porosities, which possibly weakened the lithium disilicate crystals. Another aspect observed only in Figure 3C was the presence of empty spaces. In a previous study, Naves et al. [12] also related the appearance of these spaces in vitreous ceramics reinforced with leucite, when submitted to prolonged etching times or successive applications of

acid, as performed for cleaning in this group. In this previous study [12], a reduction in bond strength values and an increase in cohesive fractures in ceramic and mixed fractures were also observed. Figure 3D shows the separation that occurred at the interface between the resin cement and ceramic, probably caused by the action of successive applications of acids on the crystals and vitreous matrix, causing it to weaken. This is reflected in the failure pattern found in the bond strength samples of G5 (Figure 5 C and C'), in which adhesive failure between the ceramic and resin cement predominated. The details of this failure mode are observed at higher magnification in Figures 5 D and E.

Another hypothesis that could explain the adhesive behavior of the restorations in G5 would be the repeated application of Hydrofluoric acid, before and after the use of the tested paste. This procedure increased the etching time recommended by the manufacturer and could have caused the accumulation of crystalline residues deposited on the ceramic surfaces and greater depth of dissolution promoted by the increase in etching time [11]. The Hydrofluoric Acid in contact with silica-based ceramics produces insoluble silicon fluoride salts, which may remain on the surface as a byproduct [11,20,26,28].

The first hypothesis was partially rejected, since the Try-in removal methods showed different surface conditions resulting in the presence of Try-in paste contamination (G2 and G4) or unconventional vitreous matrix dissolution (G3, G4 and G5) when compared with Group G1. The second hypothesis was partially rejected, since the bond strength for G4 was higher than for G5, and there was no significant difference for the other groups.

The low influence of the treatments on the bond strength may have occurred as a result of the test having been performed 24 hours after cementation. It is necessary to conduct a study that takes into consideration the degradation of the interface by aging to provide more precise data about the longevity of indirect restorations cemented after use of the temporary cementation paste Try-in.

Conclusion

The techniques used were not totally effective at removing the paste Try-in, however the presence of remaining material did not influence the bond strength of restorations. It was observed that the methods of the temporary paste removal may affect the bond strength of ceramic restorations.

Acknowledgments This study was supported in part by PAPE – Programa de Apoio à Pesquisa – University of Uberaba. We thank Ivoclar-Vivadent, Liechtenstein, for the financial support for this study. We also thank Marcos Massao Shimano for help us push-out test.

Conflict of interest The authors declare that they have no conflict of interest.

References

1. Conrad HJ, Seong WJ, Pesun IJ (2007) Current ceramic materials and systems with clinical recommendations: a systematic review. *J Prosthet Dent* 98 (5):389-404.
2. Garcia RN, Reis AF, Giannini M (2007) Effect of activation mode of dual-cured resin cements and low-viscosity composite liners on bond strength to dentin. *J Dent* 35 (7):564-569.
3. Luhrs AK, Guhr S, Gunay H, Geurtsen W (2010) Shear bond strength of self-adhesive resins compared to resin cements with etch and rinse adhesives to enamel and dentin in vitro. *Clin Oral Investig* 14 (2):193-199.
4. Belser UC, Magne P, Magne M (1997) Ceramic laminate veneers: continuous evolution of indications. *J Esthet Dent* 9 (4):197-207.
5. Derand T, Molin M, Kleven E, Haag P, Karlsson S (2008) Bond strength of luting materials to ceramic crowns after different surface treatments. *Eur J Prosthodont Restor Dent* 16 (1):35-38.
6. Ccahuana VZ, Ozcan M, Mesquita AM, Nishioka RS, Kimpara ET, Bottino MA (2010) Surface degradation of glass ceramics after exposure to acidulated phosphate fluoride. *J Appl Oral Sci* 18 (2):155-165.
7. Rahme HY, Tehini GE, Adib SM, Ardo AS, Rifai KT (2008) In vitro evaluation of the "replica technique" in the measurement of the fit of Procera crowns. *J Contemp Dent Pract* 9 (2):25-32.
8. Guazzato M, Albakry M, Ringer SP, Swain MV (2004) Strength, fracture toughness and microstructure of a selection of all-ceramic materials. Part I. Pressable and alumina glass-infiltrated ceramics. *Dent Mater* 20 (5):441-448.
9. McLean JW (2001) Evolution of dental ceramics in the twentieth century. *J Prosthet Dent* 85 (1):61-66.
10. Zortuk M, Kilic K, Gurbulak AG, Kesim B, Uctasli S (2010) Tensile bond strength of a lithium-disilicate pressed glass ceramic to dentin of different surface treatments. *Dent Mater J* 29 (4):418-424.
11. Fabianelli A, Pollington S, Papacchini F, Goracci C, Cantoro A, Ferrari M, van Noort R (2010) The effect of different surface treatments on bond strength between leucite reinforced feldspathic ceramic and composite resin. *J Dent* 38 (1):39-43.
12. Naves LZ, Soares CJ, Moraes RR, Goncalves LS, Sinhoreti MA, Correr-Sobrinho L (2010) Surface/interface morphology and bond strength to glass ceramic etched for different periods. *Oper Dent* 35 (4):420-427.
13. Abo-Hamar SE, Hiller KA, Jung H, Federlin M, Friedl KH, Schmalz G (2005) Bond strength of a new universal self-adhesive resin luting cement to dentin and enamel. *Clin Oral Investig* 9 (3):161-167.
14. de Andrade OS, de Goes MF, Montes MA (2007) Marginal adaptation and microtensile bond strength of composite indirect restorations bonded to dentin treated with adhesive and low-viscosity composite. *Dent Mater* 23 (3):279-287.
15. De Goes MF, Giannini M, Di Hipolito V, Carrilho MR, Daronch M, Rueggeberg FA (2008) Microtensile bond strength of adhesive systems to dentin with or without application of an intermediate flowable resin layer. *Braz Dent J* 19 (1):51-56.
16. Kaneshiro AV, Imazato S, Ebisu S, Tanaka S, Tanaka Y, Sano H (2008) Effects of a self-etching resin coating system to prevent demineralization of root surfaces. *Dent Mater* 24 (10):1420-1427.

17. Nikaido T, Kitasako Y, Burrow MF, Umino A, Maruoka R, Ikeda M, Tagami J (2008) Effect of resin coating on dentin bond durability of a resin cement over 1 year. *Am J Dent* 21 (1):64-68.
18. Xing W, Jiang T, Ma X, Liang S, Wang Z, Sa Y, Wang Y (2010) Evaluation of the esthetic effect of resin cements and try-in pastes on ceromer veneers. *J Dent* 38 Suppl 2:e87-94.
19. Good ML, Mitchell CA, Pintado MR, Douglas WH (2009) Quantification of all-ceramic crown margin surface profile from try-in to 1-week post-cementation. *J Dent* 37 (1):65-75.
20. Phark JH, Duarte S, Jr., Kahn H, Blatz MB, Sadan A (2009) Influence of contamination and cleaning on bond strength to modified zirconia. *Dent Mater* 25 (12):1541-1550.
21. Marshall SJ, Bayne SC, Baier R, Tomsia AP, Marshall GW (2010) A review of adhesion science. *Dent Mater* 26 (2):e11-16.
22. Ozden AN, Akaltan F, Can G (1994) Effect of surface treatments of porcelain on the shear bond strength of applied dual-cured cement. *J Prosthet Dent* 72 (1):85-88.
23. Cekic I, Ergun G, Lassila LV, Vallittu PK (2007) Ceramic-dentin bonding: effect of adhesive systems and light-curing units. *J Adhes Dent* 9 (1):17-23.
24. Quaas AC, Yang B, Kern M (2007) Panavia F 2.0 bonding to contaminated zirconia ceramic after different cleaning procedures. *Dent Mater* 23 (4):506-512.
25. Borges GA, Sophr AM, de Goes MF, Sobrinho LC, Chan DC (2003) Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. *J Prosthet Dent* 89 (5):479-488.
26. Ozcan M, Vallittu PK (2003) Effect of surface conditioning methods on the bond strength of luting cement to ceramics. *Dent Mater* 19 (8):725-731.
27. Brackett MG, Lockwood PE, Messer RL, Lewis JB, Bouillaguet S, Wataha JC (2008) In vitro cytotoxic response to lithium disilicate dental ceramics. *Dent Mater* 24 (4):450-456.
28. Kiyani VH, Saraceni CH, da Silveira BL, Aranha AC, Eduardo Cda P (2007) The influence of internal surface treatments on tensile bond strength for two ceramic systems. *Oper Dent* 32 (5):457-465.

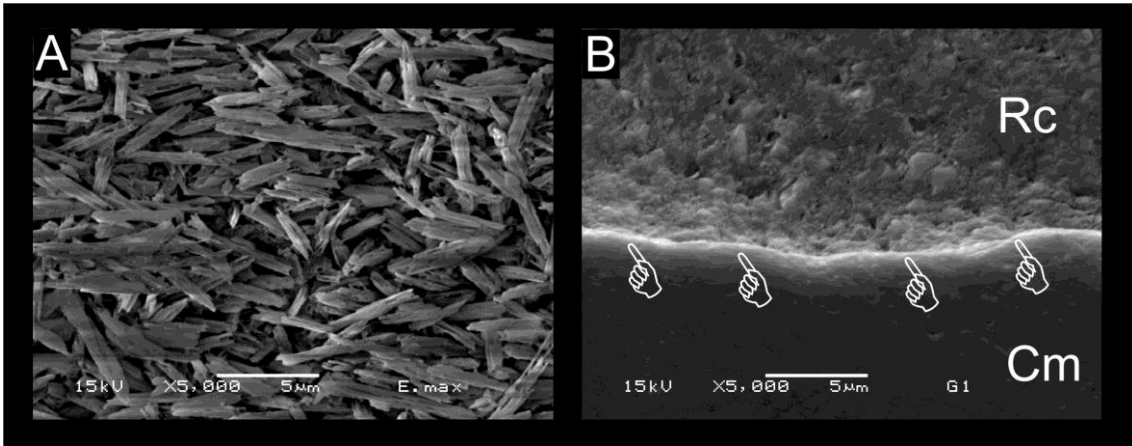


Figure 1. A) SEM images of lithium disilicate crystals exposed after the hydrofluoric acid etching. (G1). B) Resin cement (RC) and Ceramic (Cm) interface. The pointers show a continuous interface without gaps or unfilled voids.

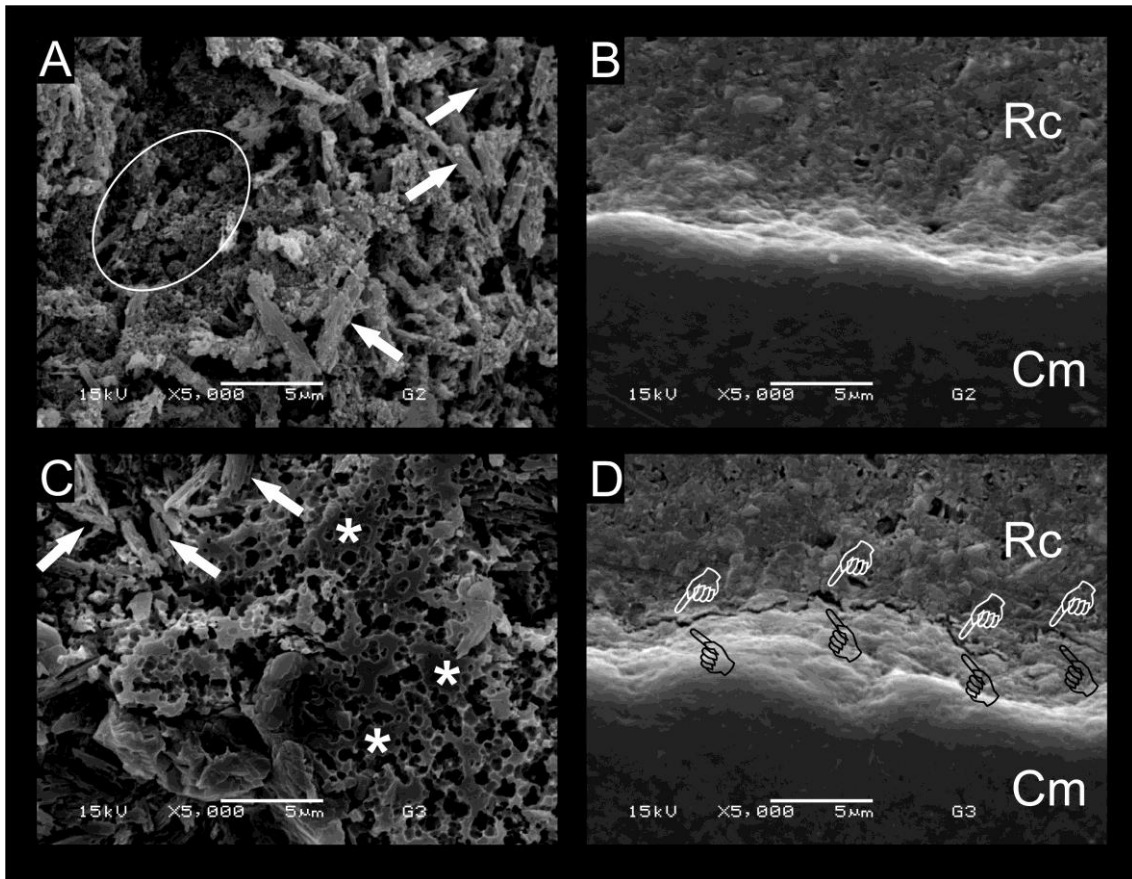


Figure 2. SEM images of G2 (A) show a surface topography with the presence of remains of Try-in adhered to lithium disilicate crystals (white arrows); the ellipse shows an area where the debris prevent the crystals observation. The bond interface between the resin cement (Rc) and ceramic (Cm) can be seen in image B, no gaps or unfilled voids were observed in this group. For G3, few crystals are observed in C (white arrows), as the largest amount of contamination with porous aspect is identified for (*). The SEM interface analysis of G3 shows the present of cracks (pointers) between the resin cement (Rc) and Ceramic (Cm) (D).

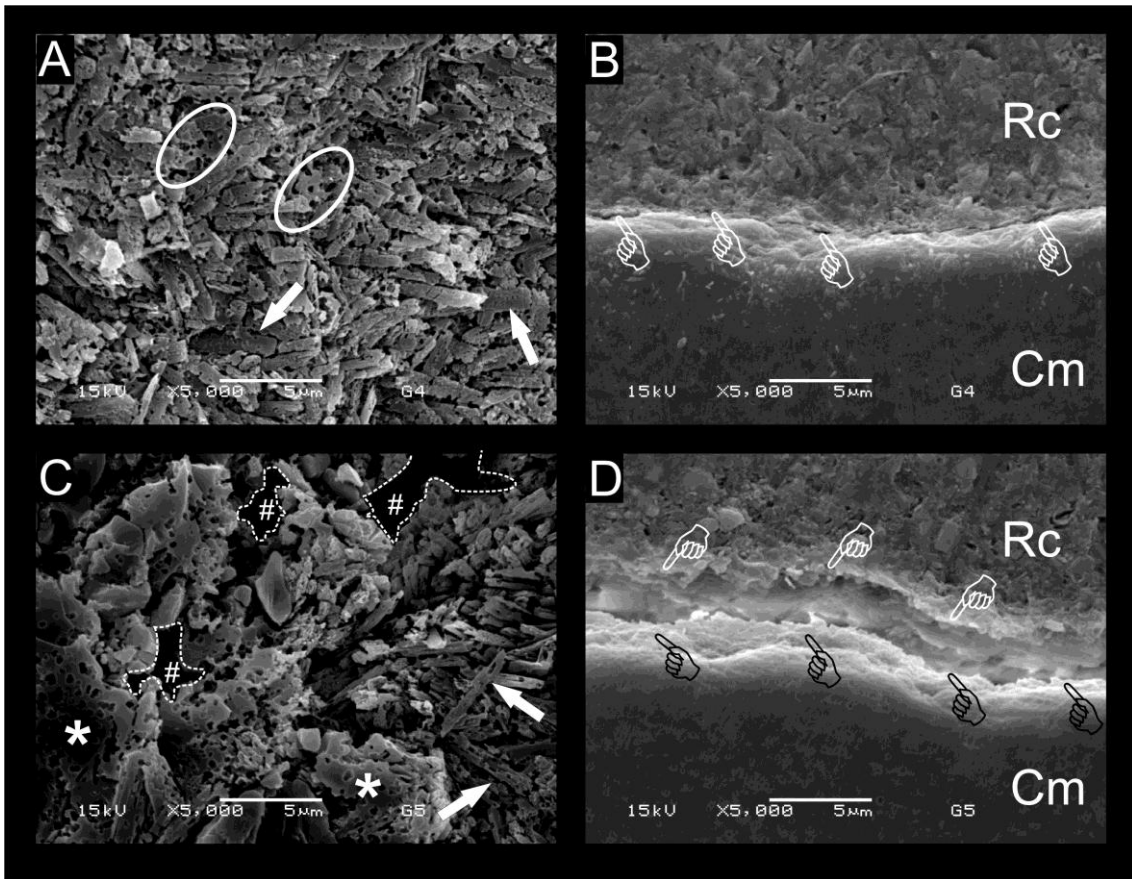


Figure 3. Image A shows a large quantity of contamination demonstrated by the ellipses. The partially exposed crystals present small porosities (white arrows). In Image B small failures are pointed out in the resin cement and ceramic interface . For G5, a large quantity of contamination with a porous aspect was observed in image C (*). The crystals pointed out (white arrows) present a large number of porosities. Another aspect observed only in image C was the presence of empty spaces (marked by # and delimited by dotted line). In image D can be seen the separation that occurred at the interface between the resin cement and ceramic.

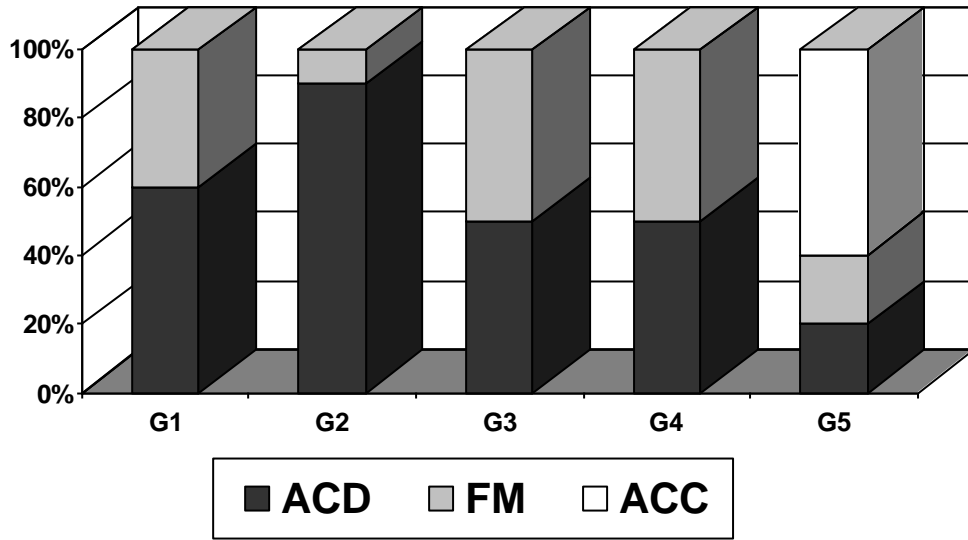


Figure 4. Distribution of failure mode.

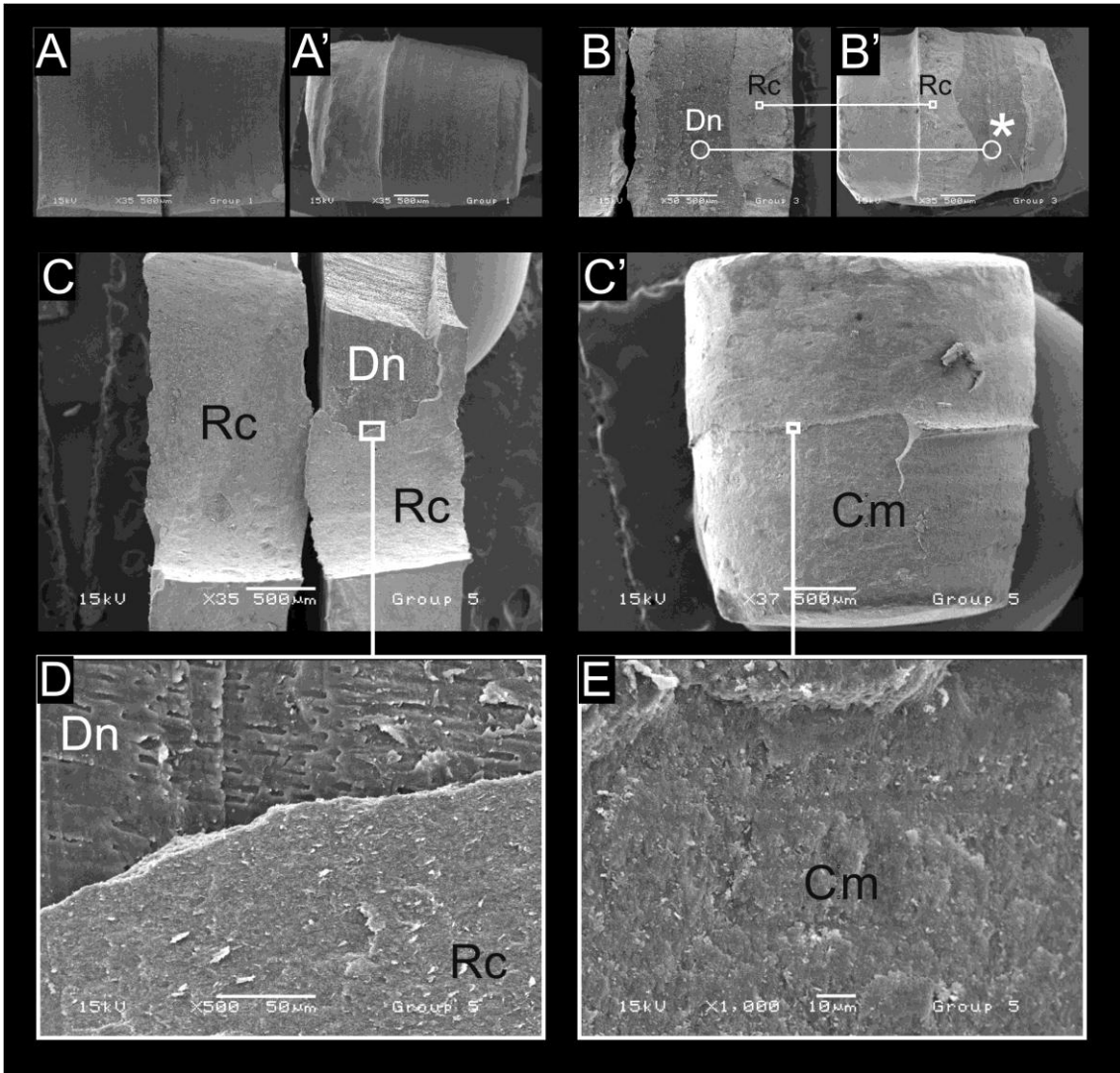


Figure 5. In images A (dentin side) and A' (ceramic side) can be seen the adhesive failure between resin cement and dentin (ACD). In images B and B' is shown a failure mixed (FM) with portion of adhesive fracture between dentin and resin cement (Dn) and part of cohesive failure in resin cement (Rc). In the ceramic side (B') can be seen the opposite part of cohesive failure (Rc) and adhesive portion of failure(*). In the images C (dentin side) and C' (ceramic side) adhesive failure mode between ceramic (Cm) and resin cement (Rc) is shown, a small part of dentin can be seen (Dn). The details of this failure mode are observed at higher magnification in images D and E.

Table 1: Groups and respective treatment

Groups	Try-in	Try-in removal procedure
G1	No	No.
G2	Yes	5 min. ultrasonic bath.
G3	Yes	Air/water spray for 1 min.
G4	Yes	Application of phosphoric acid 37% for 2min + Air/water spray for 1 min.
G5	Yes	Phosphoric acid 37% for 2min + Air/water spray for 1 min + Application of hydrofluoridric acid 5% for 2min + Application of phosphoric acid 37% for 2min + Air/water spray for 1min.

Table 2: Push-out Bond strength Values (MPa)

Groups	Bond Stregth Values (SD)
G1	16.82 (2.98)ab
G2	17.09 (2.27) ab
G3	16.18 (2.32)ab
G4	19.21 (2.20)a
G5	15.90 (2.92)b

Different letters in column indicate statistics difference.