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SHORT COMMUNICATION

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EVALUATION OF MICROTENSILE BOND STRENGTH OF TWO LUTING CEMENTS BY TYPE AND REGIONAL VARIATIONS IN DENTAL SUBSTRATE

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ABSTRACT

To compare the microtensile bond strength of two adhesive luting cements depending on the type and regional variation in dental substrate. The 36 human molars used were divided into 6 groups: G1 occlusal dentin using Rely-X Unicem cement (3M ESPE), G2 occlusal dentin using Rely-X CRA (3M ESPE); G3 buccal dentin using Rely-X Unicem; G4 buccal dentin using Rely-X CRA; G5 enamel using Rely-X Unicem and G6 enamel using Rely-X CRA. Rectangular shaped cylinders measuring 4.0 x 4.0 x 6.0 mm, simulating indirect restorations that were cemented in all samples. After microtensile tests, the data were submitted to two-way analysis of variance α=5%. The mean microtensile bond strength values (MPa) were: G1: 21.3 ± 12.9; G2: 31.6 ± 9.9; G3: 31.1 ± 16.4; G 4: 40.5 ± 16.2; G5: 13.1 ± 9.0; G6: 39.3 ± 11.9. Rely-X CRA luting cement showed higher bond strength values than Rely-X Unicem to occlusal dentin and enamel substrates. When the two luting cements were compared, irrespective of the type of substrate, Rely-X CRA luting cement showed significantly higher bond strength than Rely-X Unicem. When comparing the substrate, buccal dentin showed significantly higher bond strength than the values obtained for occlusal dentin and enamel.

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INTRODUCTION

At present large esthetic restorations have been a great challenge in Restorative Dentistry. Extensive coronal destruction frequently requires the use of indirect restoration to improve restorative material retention, and better dissipation of masticatory forces (Yoshikawa *et al*., 1999). The main reason for failure of these restoration is damage to the bond interface (Van Dijken, 2003). Thus, clinical success of indirect esthetic restorations depends largely on the remaining dental structure and the properties of the luting cement used (Ozturk and Aykent, 2003). Luting cements such as zinc phosphate and glass ionomer have shown high failure rates, probably due their inability to bond to the restorative material (Van Dijken *et al*, 1998). When the adhesive properties of luting cement are combined, indirect restorations present high retention and good marginal adaptation (Blatz *et al*., 2003).

Several studies have demonstrated that indirect restorations fixed with adhesive luting cements presented greater bond strength when compared with those cemented with other types of luting cement (Burke and Watts, 1994; Sherrer *et al*., 1994). Thus, adhesive luting cements are most indicated and used for cementing esthetic indirect restorations and may be classified into two groups, according to treatment of the dental substrate (Radovic *et al*., 2008). The first group, denominated conventional, use acid etching and an adhesive system to prepare the enamel and dentin. The second refers to adhesive luting cement that have recently been introduced to the Dental market, and are denominated self-adhesive luting cements. These materials have been designed to overcome some of the deficiencies of conventional resin and glass ionomer cements, and aggregate the favorable characteristics of these different luting cements in a single product. Dual polymerization self-adhesive luting cements were introduced to the market with the intention of diminishing the operative steps. To achieve this, the dentin treatment stage was eliminated. The bond occurs by the demineralizing action of the acid monomer (ester of methacrylate phosphoric acid) on the dental substrate, which promotes infiltration and retention of the self-adhesive luting cement (Rosenstiel *et al*., 1998; Van Dijken *et al*., 1998). Whatever may be the adhesive luting cement used, the regional morphological variation of dentin may influence the quality of the bond to this substrate. Cavities with shallow depth present a greater concentration of mineralized (intertubular) dentin, which improves the quality of the hybrid layer and consequently the bond. Whereas deep cavities have a greater transdentinal flow, increase in the diameter of the hollow passages in tubules and reduction in the intertubular region. This is important, as the variation in bond strength depends not only on the materials themselves, but also on substrate characteristics such as dentinal depth (Benetti *et al*., 2011). Therefore, the aim of the present study was to evaluate the bond strength to different dentin substrates of two adhesive luting cements, a conventional and self-adhesive type to occlusal and vestibular enamel and dentin.

MATERIAL AND METHODS

To conduct this study, 20 whole human third molars were used, obtained from patients in the age-range of 19 to 22 years. The teeth were stored in physiological solution at 4ºC., for a period not exceeding 3 months (Reis *et al*., 2003). The teeth were randomly divided into 6 groups according to the dental substrate region and luting cement used (Table 1).

Preparation of Teeth: The teeth were fixed in chemically activated acrylic resin (JET Clássico®) inside PVC cylinders (Tigre Industrial) 20 mm in diameter by 30 mm high. Each tooth was sectioned with a double-faced diamond disc (NTI – Kahla GmbH - Germany). For preparation of teeth with cuts in the occlusal region, 1/3 of the coronal height was removed and a flat dentin surface was exposed. The enamel at the periphery of the occlusal region was removed with a cylindrical, flat diamond tip ≠3100 (KG Sorensen). The dentinal surfaces were examined under a stereoscopic microscope (Baush & Lomb) at 25X magnification to ensure absent of enamel. To create a standard smear layer, Hidrolix silicone carbide abrasive papers grains 220, 320, 400 and 600 (GK Abrasivos, Indústrias Brasileiras), were used manually and under cooling, for 10 second each, with grain 600 being used for 60 seconds. Initially, the clinical crowns (space comprised between the amelo-cement limit and vertex of the cuspid) of the teeth forming the group in occlusal dentin were measured with a digital caliper (Absolute Digimatic, Mitutoyo, Tokyo, TYO, JPN), and this value was divided by 3 to delimit the area corresponding to the occlusal 1/3 of the clinical crown of each tooth. After this, the teeth were fixed by their roots in chemically activated acrylic resin (JET Clássico®), with the amelo-cemental junction above the resin, taking care to ensure that the amelo-cemental junction remained above the embedding resin. After acrylic resin polymerization following the occlusal 1/3 line of demarcation, the occlusal ½ of the crown was cut to expose a flat dentin surface, with complete removal of all the occlusal enamel. The peripheral enamel was removed with cylindrical, flat diamond tip ≠ 3100 (KG Sorensen) with a high speed pen under cooling so that the supposed bond to this enamel remnant would not interfere in the results. In the groups corresponding to the vestibular face in dentin, the crowns were sectioned in the mesio-distal direction, parallel to its long axis, to remove 1/3 of the vestibular thickness of the crown, and obtain a flat surface in dentin. After this, the crowns were cut at the level of the amelo-cemental junction, discarding the roots and area relative to the vestibular 1/3 of the crown initially sectioned. The coronal remainder was embedded so that the exposed vestibular dentin was above the cylinder margin at a height of approximately 2 to 3 mm. The teeth with the vestibular face in enamel were sectioned at the height of the amelo-cemental junction, separating the crown from the root, which was discarded. After embedment in the acrylic base, the vestibular enamel that remained exposed was used for evaluating the luting cements.

Table 1. Distribution of Experimental Groups

Group	Dental substrate region and luting cement used
G1	Occlusal Dentin (OD) and Rely-X Unicem (3M ESPE)
G2	OD and Rely-X CRA (3M ESPE)
G3	Vestibular Dentin (OD) and Rely-X Unicem (3M ESPE)
G4	VD and Rely-X CRA (3M ESPE)
G5	Enamel and Rely-X CRA (3M ESPE)
G6	Enamel and Rely-X CRA (3M ESPE)

Indirect Restorations: Indirect resin composite VMLC (Veneering Material Light Cure – Vita- Germany) was selected for fabricating the indirect restorations. To do this, standardized rectangular-shaped resin blocks measuring 4.0 x 4.0 x 6.0 mm were fabricated. Before cementation, the indirect resin composite block surface was submitted to airborne particle abrasion with 110 – 250 μm aluminum particles at a pressure of 2.5 – 3.5 bar and the application of Rely-X Ceramic Primer (3M ESPE), and drying for 5 seconds. Cementation was performed with Rely-X ARC (3M ESPE) and Rely-X Unicem (3M ESPE) luting cements in accordance with the manufacturer's instructions (Table 2). After placing the indirect resin composite block in position, uniform pressure of 1 Kg was applied, with the help of a metal device especially fabricated for doing the work. After this, excess cement was removed and light polymerization performed for 20 seconds on each of the faces, using a Curing Light XL 3000 (3M) appliance, with power density of 550 mw/cm², gauged by a radiometer, before cementation. The teeth were stored in an oven in distilled water at 37o C, for 24 hours up until the time the sticks for the microtensile test were fabricated.

Microtensile Test: Each tooth was sectioned with a low speed diamond disc coupled to a cutting machine (ISOMET 1000, Buheler) under constant cooling, to obtain stick-shaped test specimens, with a cross-sectional area of approximately 0.5 mm2, measured with a digital caliper with a precision of 0.01mm, resulting in between eight and twelve sticks per tooth. The test specimens were placed in receptacles filled with distilled water, duly identified, and stored again until the time they were submitted to the microtensile test. After one week, the microtensile test was performed. Each test specimen was individually fixed to the device developed for the microtensile tests, using a cyanoacrylate-based adhesive, and submitted to the test in a Universal test machine EMIC DL500 - 500N – at a speed of 0.5 mm/min (CDC-Bio, School of Dentistry, Federal University of Pelotas, RS, Brazil). The data were submitted to the Kolmogorov-Smirnov test of normality and test of equality of variances, performed with the Sigmastat 3.1 program (Systat Inc, San Jose, CA USA). After attaining the requisites of normal distribution and equality of variances the parametric analysis was selected. Statistical analysis was performed with two-way Analysis of Variance (Two-Way ANOVA), considering the factors substrate, material and interaction between the two, and the complementary Tukey test at a level of significance of α=5%.

RESULTS

The Table 3 presents the two-way Analysis of Variance, revealing significant difference in bond strength for the variables substrate (P<0.009), and luting cement (P<0.001). There was significant interaction between the substrate and luting cement (P<0.025).

In the table 3, table 4 and figure 1 it can be verified that when the luting cement were compared between them, within the factor substrate, luting cement Rely-X CRA presented statistically higher bond strength values than luting cement Rely-X Unicem for the substrate occlusal dentin and enamel (p<0.05). No statistical difference was observed for the factor vestibular dentin. For the variable substrate, vestibular dentin presented statistically higher bond strength values than enamel (p<0.05). No statistical difference was observed between vestibular and occlusal dentin. Whereas, for luting cement

Table 2. Description of Materials Used

Rely-X ARC		Rely-X	
Technique for Use	**Composition**	**Technique for Use**	**Composition**
Substrate Preparation: • Prophylaxis with pumice stone and water • Etching with 37% phosphoric acid for 15 seconds. • Washing for 10 seconds. • Remove excess water leaving the surface humid. Adhesive System • Apply two consecutive coats of 3M ™ Single Bond • Dry for 5 seconds. Avoid excess adhesive on all prepared surfaces. Light Activation: • Light activation of the adhesive system for 20 seconds. Cementation: • Distribute luting cement on a glass plate and mix for 10 seconds. • Apply a thin coat of cement on the resin composite block surface. • Place the resin block into position and remove excesses.	**Paste A:** is composed of approximately 68% by weight of zirconium/silica particles. Pigments, tertiary amine and photoinitiator. **Paste B:** composed of approximately 67% by weight of zirconium / silica particles. Benzyol Peroxide.	Substrate Preparation: Prophylaxis with pumice stone and water. Cementation: • Distribute luting cement on a glass plate and mix for 10 seconds. • Apply a thin coat of cement on the resin composite block surface. • Place the resin block into position and remove excesses.	**Powder:** Glass powder, initiator, silica, substituted pyrimidines, Calcium hydroxide, peroxide composite, pigments. **Liquid:** Methacrylate ester of phosphoric acid, dimethacrylate, acetate, stabilizer, initiators.

Table 3. Results of the two-way Analysis of Variance for microtensile bond strength

Factors	DF	SS	MS	F	P
Substrate	2	1647.453	823.727	4.948	*0.009*
Luting Cement	1	4881.622	4881.622	29.320	*<0.001*
Substrate x Cem. Ag.	2	1284.573	642.286	3.858	*0.025*
Residual	78	12986.414	166.492		+
Total	83	21100.848	254.227		+

Note: DF – Degree of Freedom, SS -Sum of squares, MS - Mean of squares, F – Value F, P – level of significance.

Table 4. Mean microtensile bond strength values (MPa) considering the luting cement and dental substrates

Luting Cement	Substrate		
	Occlusal Dentin	Vestibular Dentin	Enamel
RelyX Unicem	21.3(±12.9) ab, B	31.1(±16.4) a, A	13.1 (±9.0) b, B
RelyX CRA	31.6 (± 9.9) a, A	40.5 (± 16.2) a, A	39.3 (±11.9) a, A

Note: Means followed by different lower case letters on the line and capital letters in the column differ statistically among them, at a level of 5% by the Tukey test. ( ) Standard Deviation

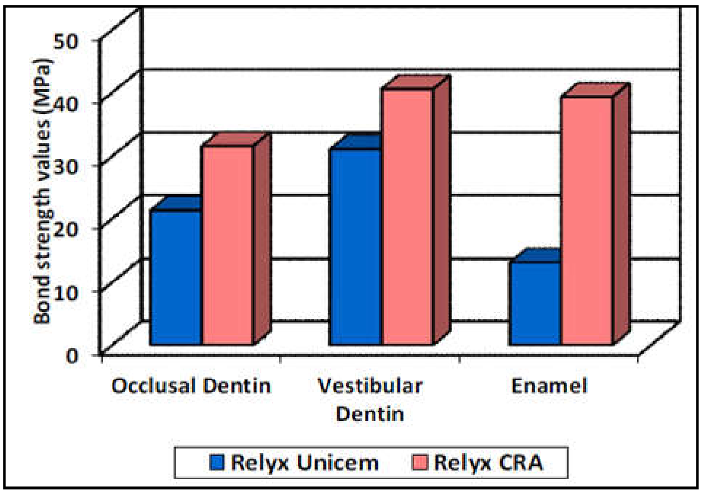

Figure 1. Mean microtensile bond strength values (MPa) considering the luting cement and dental substrates

Rely-X CRA no statistical difference was observed among the three substrates.

DISCUSSION

The bonding capacity of adhesive materials to dental structures has been extensively studied over the last few years. When used on dentin the variation in bond strength depends not only on the materials themselves, but also on substrate characteristics such as: dentinal depth, calcium concentration, patient's age, surface humidity, relative humidity, caries-affected dentin, sclerotic cervical erosion, or polymerization shrinkage, related to cavity configuration (Sano *et al*., 1999). Nevertheless, few studies have reported the influence of dentinal tubule direction on bond strength to dentin (De Munck *et al*., 2004). For the luting cement Rely-X ARC the highest bond strength values, to a significant extent, were found for occlusal dentin and enamel, in comparison with the luting cement RelyX Unicem. The most accepted hypothesis for these findings in enamel is that the pre-treatment with phosphoric acid increases the interprismatic spaces in enamel, where the resin monomers of the adhesive system are able to penetrate and create a high degree of micro-mechanical retention. The results of this study are in agreement with those of other researches (De Munck *et al*., 2004; Swift Jr. *et al*., 1995; Benetti *et al*., 2011), who demonstrated that the tooth surface treated with acid etching and adhesive promotes greater bond strength for conventional luting cements when compared with self-adhesive luting cement.

Recently, self-adhesive luting cements that do not require pretreatment of the dental substrate were introduced to the Dental market. These materials are composed of organic matrix, load particles and initiation technology. The organic matrix consists of the development of methacrylate groups with phosphoric acid. These acid monomers are able to react with the particles of cement and hydroxyapatite of dental tissue (Hikita *et al*., 2007). The self-adhesive luting cement RelyX Unicem does not require dental treatment and achieved the lowest bond strength values, to a significant extent, for enamel and occlusal dentin, in comparison with the conventional luting cement, which is in agreement with the findings of Benetti *et al*. (2011) and Viotti *et al*. (2009). The bond quality between the cement-tooth surface is intimately related to the extension of monomer infiltration. In spite of its low pH value (approximately 2), the acid monomers present in the composition of self-adhesive cements promote less interprismatic hybridization in enamel, and consequently lower bond strength when compared with the values achieved by the conventional technique (De Munck *et al*, 2004; Hikita *et al*., 2007). Another factor that could justify the low bond strength values of self-adhesive cements to enamel is their high viscosity, which makes it difficult for the acid monomers to penetrate and generate insufficient contact between the cement and dental substrate before light polymerization (Benetti *et al*., 2011).

The values found for occlusal dentin differ in recent studies (Hikita *et al*., 2007) in which conventional luting cement presented similar bond strength values for the two types of cement. Various studies have shown that self-adhesive cement can be used for indirect esthetic restorations, due to its capacity to provide high bond strength values to dentin and efficient marginal sealing (Sano *et al*., 1999; Takahashi *et al*., 2002). In addition, the multi-step application technique (conventional) is slow, very sensitive and may compromise bond effectiveness (Mak *et al*., 2002). Nevertheless, various studies have demonstrated that simplification may facilitate manipulation for the general clinician, but is incapable of improving the bond quality. (Frankenberger *et al*., 1999; 2000; 2005). Bond strength to the vestibular face presented statistically similar bond strength values for the two luting cement investigated, which is in agreement with the study of Ogata *et al*. (2001). The cut to obtain vestibular dentin exposes the dentinal tubules in an oblique manner in relation to their orientation. The bond of resin materials is more stable in this region due to the diminished density and diameter of the dentinal tubules (Adebayo *et al*., 2008). When the bond to this surface occurs, acid etching removes the peritubular dentin along the cut tubules (20-30 μm), thus enabling greater radial diffusion around the intertubular dentin. Thus, the percentage of area occupied by the hybrid layer is wider, due to the absence of resin tags. The bond interface in dentin with obliquely sectioned tubules represents a unique condition in which the entire bond area is composed of a hybrid layer free of resin tags (Ogata *et al*., 2001). Whereas the cut to obtain occlusal dentin exposes the openings of dentinal tubules in a manner perpendicular to their orientation. This promotes greater surface wetting due to the larger tubule diameter and density. (Marshall *et al*., 1997; Tagami *et al*,1990). When this surface is etched, the peritubular dentin is removed, and there is formation of funnel-shaped resin tags. The resin monomers diffuse into the etched intertubular dentin, both in a direct manner on the surface, and indirectly by radial diffusion into the hollow center of the tubule, however, the depth of peritubular dentin removal limits the depth of the hybrid layer in a distance of 4-5 □m and contributes to the reduced bond strength of self-adhesive cements (Adebayo *et al*., 2008). The bond strength of conventional luting cements to the different regions of dental substrate is known. However, self-adhesive cements present low bond strength to occlusal dentin and enamel without pretreatment. Thus, based on the results found, the need for further studies is perceived, in order to evaluate the interactions of self-adhesive cements with enamel and dentin, including variations to potentiate the bond capacity of dental substrate to self-adhesive luting cement.

Conclusion

According to the results obtained, and considering the materials tested and methodology used in this study, it could be concluded that:

- With regard to the type of substrate, the conventional luting cement *Rely-X* CRA presented significantly higher bond strength values than the self-adhesive luting cement *Rely-X Unicem* when tested in enamel and occlusal dentin. There was no statistical difference in the values obtained in vestibular dentin.
- In the comparison between the two cements evaluated, irrespective of the type of substrate, the conventional luting cement *Rely-X* CRA presented significantly higher bond strength values than the self-adhesive luting cement *Rely-X Unicem*.

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