CAN ULTRASOUND APPLICATION INFLUENCE THE BOND STRENGTH OF SELF-ADHESIVE RESIN CEMENTS TO DENTIN?

Diego F. F. da Silva[1], Maurem L. Marcondes[1], Niélli C. de Souza[1], Bruna Gomes Daudt[1], Luiz H. Burnett-Júnior[1], Ana M. Spohr[2]

[1] *Department of Restorative Dentistry, School of Dentistry, Pontifical Catholic University of Rio Grande do Sul, Brazil.*
[2] *Department of Dental Materials, School of Dentistry, Pontifical Catholic University of Rio Grande do Sul, Brazil.*

ABSTRACT
The aim of this study was to evaluate the influence of ultrasound application on the bond strength of self-adhesives resin cements to dentin. Twenty-four third molars were randomly divided into 4 groups (n=6/group): G1 - Rely X Unicem; G2 – Maxcem Elite; G3 – RelyX Unicem and ultrasound application; G4 – Maxcem Elite and ultrasound application. Composite resin blocks were luted to flat dentin with a load of 500 g for 2 min, followed by light polymerization in G1 and G2. In G3 and G4, the ultrasound device was applied for 20 s on the composite resin block, followed by 500 g load for 1 min and 40 s, and light polymerization. After storage in distilled water at 37ºC for 24 h, six tooth/resin sets were cut parallel to the long axis of the tooth, in the x and y directions, with a cross section area of ~0.80 mm². Twenty-four specimens were obtained for each group and submitted to microtensile bond strength (µTBS) testing in a universal testing machine at 0.5 mm/min crosshead speed.
According to two-way ANOVA, resin cement (p=0.000) and cementation method (p=0.002) were significant. Interaction was not significant (p=0.676). According to Student's-t test (α=0.05), the µTBS mean with ultrasound application (13.74 MPa) was statistically higher than without it (10.57 MPa). The µTBS mean of RelyX Unicem (13.95 MPa) was statistically higher than Maxcem Elite (10.36 MPa).
The ultrasound application increased the µTBS of the RelyX Unicem and Maxcem Elite to dentin.

Key words: *Dentin - bond strength - cements - ultrasound.*

A APLICAÇÃO DO ULTRASSOM PODE INFLUENCIAR A RESISTÊNCIA DE UNIÃO DE CIMENTOS RESINOSOS AUTOADESIVOS À DENTINA?

RESUMO
O objetivo desse estudo foi avaliar a influência da vibração ultrassônica na resistência de união de cimentos resinosos autoadesivos à dentina. Vinte e quatro dentes terceiros molares foram divididos aleatoriamente em quatro grupos (n=6/grupo): G1 – RelyX Unicem; G2 – Maxcem Elite; G3 – RelyX Unicem com vibração ultrassônica; G4 – Maxcem Elite com vibração ultrassônica. Blocos de resina composta foram cimentados sobre dentina plana com carga de 500 g por 2 min, seguido de fotoativação nos G1 e G2. Em G3 e G4, a vibração ultrassônica foi aplicada por 20 s sobre o bloco de resina composta, seguido de carga de 500 g por 1 min e 40 s e fotoativação. Após armazenagem em água destilada a 37º C por 24 h, seis conjuntos dente/resina foram cortados paralelamente ao longo eixo do dente, nos sentidos x e y, com secção de aproximadamente 0,8 mm². Foram obtidos 24 corpos de prova para cada grupo, sendo então submetidos ao teste de resistência à microtração (Rµt) em máquina de ensaio universal com velocidade de 0,5 mm/min.
De acordo com Análise de Variância Fatorial, as variáveis tipo de cimento resinoso (p=0,000) e tipo de cimentação (p=0,002) foram significativas. A interação das duas varáveis não foi significativa (p=0,676). De acordo com o teste t-student (α=0,05) a média de Rµt com uso da vibração ultrassônica (13,74 MPa) foi estatisticamente superior sem aplicação da vibração ultrassônica (10,57 MPa)). A média de Rµt do RelyX Unicem (13,95 MPa) foi estatisticamente superior ao Maxcem Elite (10,36 MPa).
A vibração ultrassônica aumentou a resistência de união dos cimentos resinosos autoadesivos RelyX Unicem e Maxcem Elite à dentina.

Palavras-chave: *Dentina - resistência de união - cimentos - ultrassom.*

INTRODUCTION

Resin cements are widely used because their mechanical properties, aesthetics and ability to bond to restorative materials when proper pretreatment is applied are better than those of conventional cements[1]. A new category of resin cements – self-adhesive resin cements – have gained popularity with clinicians because they are easy to use and the luting procedure takes less time than with resin cements that require the application of an adhesive system. Without the adhesive system, part of the sensitivity of the technique is eliminated[1,2]. Despite being easier to apply, it is important that these self-adhesive materials should be capable of bonding adequately to both the dental structures and the restorative material in order to strengthen the tooth. Self-adhesive resin cements interact superficially with tooth hard tissues[3,4]. They have lower bond strength with enamel than resin cements requiring an adhesive system do[2,5]. In relation to dentin, some studies have shown that self-adhesive resin cements perform comparably to multi-step systems on coronal dentin[2,3,5-7], although others have shown that they have significantly lower bond strengths to dentin[8-10]. To improve the bond, enamel etching with phosphoric acid has been suggested[7,11]; however, on dentin, this etching harms the effectiveness of the bond, which may be due to inadequate resin cement infiltration into the collagen fiber network[3]. With the aim of increasing the bond strength of self-adhesive resin cements to dentin, the application of weak acids, such as polyacrylic acid before the luting procedure has been tested[12,13]. Polyacrylic acid partially removes the smear layer[14], leaves the mineral phase of dentin and increases the chemical reaction between the material and substrate[15]. However, it would be interesting to test other techniques, such as ultrasound application, with the aim of improving the adhesion of self-adhesive resin cements to dental substrate.

Studies have shown that ultrasound application during cementation affects the thixotropic properties of luting agents, leading to a decrease in viscosity[16,17]. This may promote an adequate wetting and adaptation of the densely filled resin cements to the dental substrate[18]. Ultrasound vibration reduced porosities in glass ionomer cements[19], increased the temperature of the cement, shortened the setting reaction, and increased the bond strength to enamel[20].

The purpose of this study was to evaluate *in vitro* the effect of ultrasound application during the luting procedure on the bond strength of self-adhesive resin cements to dentin.

This study was conducted under the null hypothesis that ultrasound application does not influence the bond strength of self-adhesive resin cements to dentin.

MATERIALS AND METHODS

Twenty-four unerupted human third molars, extracted for therapeutic reasons, were cleaned of gross debris and stored in distilled water at 4ºC. The water was changed every week and the teeth were used within a period not exceeding 6 months. Roots were mounted in self-cured acrylic resin, and the occlusal enamel surface was removed with a low concentration diamond disc mounted on a low-speed laboratory cutting machine Labcut 1010 (Extec Corp., London, England), under cooling. The rest of enamel was removed with 400 grit silicon carbide abrasive paper in a polishing machine DPU-10 (Panambra, São Paulo, SP, Brazil) under water. The superficial dentin was exposed and finished with 600 grit silicon carbide abrasive paper in the polishing machine, and a flat dentin surface was obtained. After polishing, the teeth were randomly divided into four groups (n=6) according to the materials used (Table 1) and treatment applied.

Before the luting procedure, composite resin blocks of Z250 (3M, St. Paul, MN, USA) were obtained using a stainless steel mold with an inner diameter of 10 mm and a height of 5 mm. Three equal increments were inserted into the mold and each increment was light-cured for 40 seconds with a quartz-tungsten-halogen curing unit (XL 3000, 3M, St. Paul, MN, EUA). The surface of the resin composite block was treated with airborne particle abrasion with 50-µm aluminum oxide for 5 s at 4-bar pressure, and a layer of silane (Angelus, Londrina, PR, Brasil) and a layer of bond (Adhese, Ivoclar Vivadent, Liechtenstein) were applied, followed by light curing for 10 seconds. Group 1 - RelyX Unicem: equal quantities of base and catalyst pastes were mixed and applied on dentin

Table 1: Chemical composition and batch number of the resin cements.

Material	Batch number	Composition	Manufacturer
RelyX Unicem	440148	Powder: glass powder, silica, calcium hydroxide, pigment, substituted pyrimidine, peroxy compound, initiator Liquid: methacrylated phosphoric ester, dimethacrylate, acetate, stabilizer, initiator	3M/ESPE, St. Paul, MN, USA
Maxcem Elite	3668021	Glycerol phosphate dimethacrylate (GPDM), barium, silica, fluor-aluminium-silicate	Kerr, Orange, CA, USA

at approximately 1 mm thickness, and the composite resin block was luted to the tooth under a 500-g load by means of a metallic tool for 2 min. The excess resin cement was removed, followed by light curing for 40 s on each side (mesial, distal, buccal, lingual and occlusal) with the curing unit XL 3000. The light intensity was controlled by a radiometer (model 100, Demetron/Kerr, Danbury, CT, USA) in the interval between 450 and 500 mW/cm^2. The specimens were stored for 24 h at 37°C in distilled water.

Group 2 - Maxcem Elite: the material was applied on dentin at approximately 1 mm thickness using the syringe supplied by the manufacturer, followed by the composite resin block as described for group 1.

Group 3 - RelyX Unicem with ultrasound application: the cement manipulation was the same as described for group 1. The composite resin block was luted under ultrasound vibration. An ultrasound tip provided with a rubber cap (C20 tip applicator, Gnatus, Ribeirão Preto, SP, Brazil) was mounted on an ultrasound handpiece (Jet Sonic Four Plus, Gnatus, Ribeirão Preto, SP, Brail), set at 30% of the power according to the manufacturer's recommendation. The operator held the handpiece and the tip was oriented perpendicular to the surface of the composite resin block. The vibration was applied for 20 seconds, in an intermittent mode, under a load of approximately 100g. This load was previously calibrated by the operator using a digital weighing-machine. After the ultrasound application, the 500-g load was applied for 1 min and 40 seconds. The excess resin cement was removed, followed by light-curing as described for group 1.

Group 4 - Maxcem Elite with ultrasound application: the cement manipulation was the same as described for group 2. The luting procedure was the same as described for group 3.

Specimens were stored for 24 h at 37^{o}C in distilled water, and then sectioned perpendicular to the bonding surface using a Labcut 1010 laboratory cutting machine at a speed of 400 rpm with a diamond disk under water cooling. The specimens had a cross-section of approximately 0.90 × 0.90 mm, measured with a digital caliper (Mitutoyo Sul Americana Ltda., Suzano, SP, Brazil). Six sticks from the central region of each tooth were used, which were examined with a stereomicroscope (Olympus Corp., Tokyo, Japan) at 25× magnification to analyze the adhesive area. The specimens with defects such as bubbles, lack of material or irregular areas were discarded. Twenty-four specimens were selected for each group.

The specimens were then fitted to the microtensile testing device, which has two stainless steel grips with an area of 8 × 10 mm and sliding shafts that prevent torsion movements during the tests. These shafts have a fixing screw that prevents the specimen from moving during bonding. The specimens were fixed with cyanoacrylate glue (Loctite, São Paulo, SP, Brazil), associated with the Zip Kicker accelerator (Pacer Technology, Rancho Cucamonga, CA, USA), and stressed at a crosshead speed of 0.5 mm/min until failure in a universal testing machine (EMIC DL-2000, São José dos Pinhais, PR, Brazil) using a cell load of 50 N. The μTBS was expressed in MPa and derived by dividing the force (N) at the time of fracture by the bond area (mm^2). The fractured surfaces of all specimens were observed by scanning electron microscopy (SEM) (Philips XL 30, Philips Electronic Instruments Inc., Mahwah, NJ, USA). The failures were classified as adhesive (failure between dentin and resin cement), cohesive in dentin (dental substrate failure), cohesive in resin cement (failure inside the resin cement) or mixed (two or more types of failure). The specimens were further analyzed regarding the percentage of remaining resin cement on the dentin surface. Images of the fractured areas from SEM at X200 magnification were viewed on a 15-inch computer screen. A grid divided into 16 squares was placed over the specimen image (Fig. 1). The criterion used to classify a "square with cement" was the presence of remaining cement in at least half of the square. The remaining cement area on the fractured surface of each

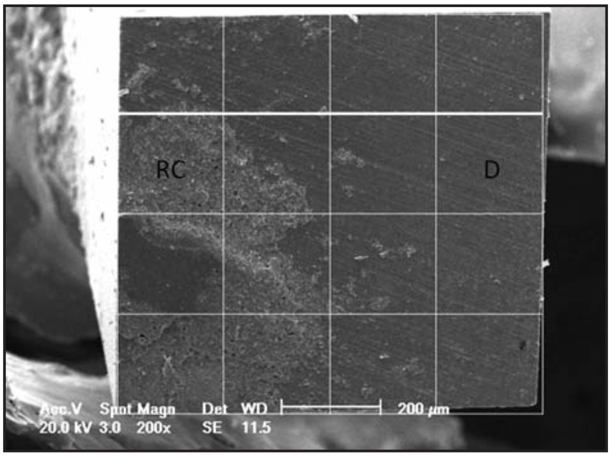

Fig. 1: Dentin μTBS fracture surfaces of a representative sample treated with RelyX Unicem showing mixed failure. RC: resin cement; D: dentin.

specimen was calculated as percentage of the total area. Data on the percentage of remaining cement for each specimen were recorded, and the mean values for each surface treatment were calculated.

Two-way analysis of variance (ANOVA) was used to test the effect of the resin cements and the cementation method on the µTBS. Furthermore, Student *t*-test was used to determine differences in µTBS between the resin cements, and with and without ultrasound application. $P<0.05$ was considered significant. The software used was SPSS 10.0 (SPSS Inc., Chicago, IL, USA).

RESULTS

Two-way ANOVA analysis showed that the resin cement ($p=0.000$) and the cementation method ($p=0.002$) had a significant effect on the µTBS, while the interaction effect was not significant ($p=0.676$) (Table 2).

According to Student-*t* test, the µTBS mean with ultrasound application (13.74 MPa) was statistically higher than without ultrasound application (10.57 MPa) ($p=0.003$). The µTBS mean for RelyX Unicem (13.95 MPa) was statistically higher than Maxcem Elite (10.36 MPa) ($p=0.001$) (Table 3).

All specimens showed mixed failure (adhesive + cohesive in resin cement). The percentage values of remaining resin cement on the dentin surface was 16.8% for group 1, 25% for group 2, 15.3 % for group 3 and 19.2% for group 4.

DISCUSSION

The null hypothesis was rejected, because ultrasound application increased the µTBS of RelyX Unicem and Maxcem Elite self-adhesive resin cements to dentin.

In this study, ultrasound vibration was applied for 20 s during the cementation procedure of the composite resin block, allowing the resin cement to flow and the resin block to become seated on the dentin. After this, a static load of 500g was applied. Although 20 s correspond to a rapid procedure, they were sufficient to influence the µTBS value.

Whenever two materials are mixed, whether they are powder and liquid, or a paste – paste system, air bubbles are incorporated, making the material more porous. This porosity reduces the intrinsic strength of the material[21], and may reduce its bond capacity to some substrate. Cantoro *et al.*[22] used SEM to verify that ultrasound vibration reduced the porosity of the self-adhesive resin cements RelyX Unicem and G-Cem, making them more homogeneous. The same effect was observed on glass ionomer cements[19]. The reduction in resin cement porosity might have allowed these materials to wet the dentinal substrate better, promoting a higher mean µTBS when cementation was associated with ultrasound application.

It has been reported that ultrasound vibration increases the mechanical properties of glass ionomer cements, especially in the first 24 h[23], as well as the bond strength to dentin[24]. Algera *et al.*[20]

Table 2: Results for the two-way ANOVA.

Sources of Variation	Sum of Squares	DF	Mean Square	f	p
Resin cement	310.61	1	310.61	13.20	0.000**
Cementation method (with or without ultrasound)	241.36	1	241.36	10.25	0.002**
Resin cement x cementation method	4.14	1	4.14	0.18	0.676 ns
Error	2165.53	92	23.54		
Total	16902.16	96			

** significant $p \leq 0.05$; ns = not significant

Table 3: µTBS for the experimental groups.

Variable	Comparison	n	Mean (MPa)	Standard deviation (MPa)	Coeficient of variation (%)	P
Cementation method	With ultrasound	48	13.74	5.45	39	0.003
	Without ultrasound	48	10.57	4.80	45	
Resin cement	RelyX Unicem	48	13.95	5.49	39	0.001
	Maxcem Elite	48	10.36	4.60	44	

studied resin-modified glass ionomer cements, reporting that the local heat produced by ultrasound vibration may catalyze free radicals during polymerization, favoring improvement in the mechanical properties and a better bond to the substrate. As regards self-adhesive resin cements, Cantoro *et al.*[22] reported that ultrasound vibration could also favor a greater initial reaction between the calcium hydroxide and acid resin monomers of RelyX Unicem, a reaction in which the water required for ionization of the functional monomers is generated. In addition, the acid-base reaction between the acid monomers and basic inorganic particles of the material must also be increased[22]. It is therefore suggested that ultrasound application favored a greater setting reaction in both the self-adhesive resin cements studied, providing an increase in the mechanical properties in the first 24 h, and thus, greater bond strength.

Another important effect of ultrasound vibration is on the thixotropic properties of cementing agents. It was demonstrated that oscillatory load on the cement increased its fluidity, leading to a reduction in viscosity[17]. De Munck *et al.*[3] observed high viscosity and presence of spaces in the resin cement layer for RelyX Unicem, resulting in insufficient adaptation to dental substrate. It is therefore suggested that when the ultrasound vibration reached the resin cements RelyX Unicem and Maxcem Elite, it caused an intrinsic vibration of the molecules and particles of the cements precisely at the moment at which the shear forces were being placed on the material by the cementation load. This favored a reduction in viscosity of the resin cement, greater wetting capacity and adaptation to the dentin, as well as a higher bond strength value, since the chemical and physical interactions with dentin are favored by the greater adaptation between the cement and substrate.

Regardless of the resin cement and whether or not ultrasound was applied, all specimens showed mixed failures, characterized by rupture at the level of the dentin – resin cement interface (adhesive), with the presence of cohesive failure in the resin cement itself, resulting in a certain quantity of resin cement remaining adhered to the dentin after the microtensile bond strength test. This type of failure demonstrates that the bond interface is the most fragile site, and is susceptible to rupture. However, a certain bond exists between self-adhesive resin cements and dentin, because part of the resin cement remained bond to the substrate. It was also observed that the presence of remaining cement was similar between the experimental groups.

RelyX Unicem had higher μTBS (13.95 MPa) than Maxcem Elite (10.36 MPa), in agreement with other studies[5,9]. In general, self-adhesive resin cements have a limited capacity to demineralize tooth hard tissues[3-5]. The following hypotheses might explain these findings: (1) the pH of these cements, which is approximately 2.1[4], is not low enough; (2) the high viscosity of the cement[3], and (3) neutralization may occur during mixture due to the chemical reaction that releases water or alkaline particles, which may increase the pH[4]. Studies evaluating the bond interface of self-adhesive resin cement using SEM [3,4,6] and TEM [3,25] showed no formation of a hybrid layer or resin tags.

The bonding mechanism of RelyX Unicem to dentin appears to be chemical rather than micromechanical in nature[26]. This bond is established by the specific multifunctional phosphoric-acid methacrylates, which are ionized at the time of mixing and which react with the hydroxyapatite of the mineral tissues of the tooth[26]. According to information from the manufacturer, Maxcem Elite also contains an acid monomer, glycerol dimethacrylate dihydrogen phosphate (GPDM), which is partly responsible for the effect of etching and adhesion to the dental structure (Technical Bulletin, 2007).

Various factors may influence the bonding capacity and adhesion of resin cements to dentin, including chemical composition, viscosity and pH. Maxcem Elite tends to maintain its low pH (2.2), while the pH of RelyX Unicem increases after 48 h (from 2.8 to 7.0). Although low pH is necessary for adequate dentin etching, it has been speculated that if the pH is maintained for a long time, as in the case of Maxcem Elite, there could be an adverse effect on the bond between this cement and the dental structure[27]. Therefore, this characteristic of Maxcem Elite pH could be one of the factors that led to this material having lower bond strength than RelyX Unicem.

The mean difference in μTBS with and without the ultrasonic application was 3 MPa. Although this difference is statistically significant, it is doubtful whether it would be of any clinical significance. To date, the literature has not established the minimum bond strength required between the material and the substrate to guarantee the success and longevity of the cementation procedure. Nevertheless, this study demonstrated that ultrasound may be applied without producing a negative effect on the process of bonding to dentinal substrate, but rather, a positive effect. Moreover, cementation with ultrasound application results in the restoration adapting to the preparation more rapidly, requiring a lower cementation load to seat the restoration in comparison with a static load, and a thinner cementation line[18].

According to the methodology used and within the limitations of this study, the results suggest that the ultrasound application increased the µTBS of the self-adhesive resin cements RelyX Unicem and Maxcem Elite to dentin.

CORRESPONDENCE
Dr. Ana Maria Spohr
PUCRS - School of Dentistry - block 6
Avenida Ipiranga, 6681 - CEP: 90619-900
Porto Alegre, RS, Brazil
e-mail: ana.spohr@pucrs.br

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