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ORIGINAL ARTICLE

Effect of temporary cements on the microtensile bond strength of self-etching and self-adhesive resin cement

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Abstract

Objective. The aim of this study was to evaluate the microtensile bond strength (μTBS) of self-etching and self-adhesive resin cement systems to dentin affected by the presence of remnants of either eugenol-containing or eugenol-free temporary cements. ***Materials and methods.*** Thirty extracted teeth were obtained and a flat dentin surface was exposed on each tooth. Acrylic blocks were fabricated and cemented either with one of two temporary cements, one zinc oxide eugenol (ZOE) and one eugenol free (ZOE-free), or without cement (*control*). After cementation, specimens were stored in water at 37°C for 1 week. The restorations and remnants of temporary cements were removed and dentin surfaces were cleaned with pumice. Resin composite blocks were cemented to the bonded dentin surfaces with one of two resin cements, either self-etching (Panavia F 2.0) or self-adhesive (RelyX U-100). After 24 h, the specimens were sectioned to obtain beams for submission to μTBS. The fracture mode was evaluated under a stereoscopic loupe and a scanning electron microscope (SEM). Data from μTBS were submitted to two-way repeated-measure ANOVA and the Tukey test (alpha = 0.05). ***Results.*** The cross-product interaction was statistically significant ($p < 0.0003$). The presence of temporary cements reduced the bond strength to Panavia self-etching resin cements only ($p < 0.05$). Fracture occurred predominantly at the dentin–adhesive interface. ***Conclusions.*** The presence of eugenol-containing temporary cements did not interfere in the bond strength to dentin of self-adhesive resin cements.

Key Words: *self-adhesive luting cement, temporary cement, dentin, microtensile bond strength*

Introduction

The fabrication of dental prostheses requires laboratory time and any exposed dentin must be protected while the prostheses are being made. Maintaining pulp protection as well as aesthetics and functionality requires temporary restorations and cementation, for which materials containing eugenol are generally used [1].

Eugenol is a free radical that partially or completely inhibits the conversion of resin materials because it has an affinity for the free radicals that form during the polymerization process [2]; therefore, it can compromise the sealing and retention of adhesive restorations [2]. This can lead to leakage along the bond interface, which may cause pulp problems, hypersensitivity and secondary caries—the most common reason for restoration replacement [3,4].

Studies have yielded controversial results with regard to the bond strength to dentin of adhesive materials when zinc oxide eugenol (ZOE) is used as a temporary restoration material [4–7]. This is explained by the fact that eugenol molecules or eugenol-containing temporary restoration residues may be incorporated into hybridization materials, where they affect the bond strength to dentin [1,8,9] and especially by variation in the type of adhesive material used to evaluate the bond strength, i.e. etching-rise or self-etching.

Self-adhesive cement is a new concept in luting materials, recently developed, that does not require any pre-treatment of the tooth surface [10–12];

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according to Radovic et al. [13], once the cement is mixed, its application procedure is extremely simple. The goal behind the development of this material was to combine the favourable properties of conventional (zinc phosphate and/or glass ionomer) and resin luting agents [13]. According to information provided by its manufacturer, no post-operative sensitivity is expected with the use of self-adhesive resin cement as the smear layer is not removed.

When cement containing zinc oxide and eugenol is applied beforehand, however, molecules of eugenol or temporary restoration residues containing eugenol may still be present when self-adhesive cement is applied. This can reduce the free surface energy and compromise the wettability of self-adhesive resin cements on dentin [1]. Thus, the bonding effectiveness of self-adhesive resin cement systems may be seriously compromised by the presence of remnants.

In the same way, self-etching cements, which contain acidic monomers (10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate) and depend on Ca^{+2} to bond to the dental structure, may also be affected by temporary cement residues or the presence of eugenol. This may make it difficult for the acidic monomers to form a chemical bond to the Ca^{+2} in dentin.

Therefore, the purpose of this study was to evaluate the effects of temporary cements (with eugenol and eugenol-free) on the immediate microtensile bond strength of two resin cements (self-etching and self-adhesive).

Materials and methods

Thirty extracted, caries-free human third molars were used. The teeth were collected after obtaining informed consent from the patients. The Local University Review Board approved and reviewed this study protocol (23115-006642/2011-88).

Two resin cements were used: Panavia F 2.0 (Kuraray Medical, Inc, Tokyo, Japan) as the self-etching type and RelyX U-100 (3M/ESPE, St. Paul, MN) as the self-adhesive type. The composition and application methods are described in Table I. The teeth were disinfected in 0.5% chloramine, stored in distilled water and used within 6 months after extraction.

Specimen preparation

Flat superficial dentin surfaces were created after removal of the occlusal enamel with a low-speed diamond saw (Isomet 1000, Buehler, Lake Bluff, IL) under water-cooling. The enamel-free exposed dentin surfaces were further polished on wet #600-grit SiC paper for 60 s to standardize the smear layer.

Thirty teeth were divided into six groups according to both of the main factors: Resin Cement (self-etching or self-adhesive) and Temporary Cement (ZOE, ZOE-free or no cement/control). Five teeth (n = 5) were used in each experimental group (Figure 1).

Resin block preparation

A 6 mm thick hand-layered build-up resin composite block (n = 30) was made on the flat dentine surface of each tooth, in the form of a block, by means of the application of 2 mm thick composite layers [8] (Herculite, shade A3.5, Kerr, Orange, CA). Each increment was light-activated with a halogen light appliance (Optilux 501, Kerr) for 40 s at an intensity of 600 mW/cm^2 (Radiometer, Kerr). These resin composite build-up blocks were detached from each tooth and they were stored in a dry environment until the time of its definitive cementation.

Immediately afterward, acrylic resin blocks 2 mm thick were fabricated for each tooth (JET Classic, Art. Odontológicos Ltda, Campo Limpo Paulista, SP, Brazil), those were used as temporary block and

Table I. Self-adhesive resin luting cements used in this study.

Material	Manufacturer	Composition		Application technique
Panavia F 2.0	Kuraray	ED Primer (Lot A: 00309A) (Lot B: 00183A)	Primer A: HEMA, 10-MDP, 5-NMSA, water, accelerator Primer B: 5-NMSA, accelerator, water, sodium benzene sulphinate	1. Mix (A+B) and apply for 30 s on dentin 2. Air-dry gently
		Paste A (Lot: 00254C) Paste B (Lot: 0031C)	Paste A: 10-MDP, silanated silica, hydrophobic aromatic and aliphatic dimethacrylate, hydrophilic dimethacrylate photoinitiator, dibenzoyl peroxide Paste B: silanated barium glass, sodium fluoride, sodium aromatic sulfinate, dimethacrylate monomer, BPO	1. Dispense equal amounts of pastes A and B and mix pastes for 20 s 2. Apply mixture on dentin 3. Light-activate (20 s) the margin on each side of the tooth
RelyX U-100	3M/ESPE	Base: glass fibre, methacrylate phosphoric acid esters, dimethacrylates, silanated silica, sodium persulphate (426343) Catalyst: glass fibre, dimethacrylates, silanated silica, p-toluene sodium sulphate, calcium hydroxide (426343)		1. Mix cement (20 s) 2. Apply mixture on dentin 3. Light activate (20 s) the margin on each side of the tooth

HEMA, 2-hydroxyethyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; 5-NMSA, N-methacryloyl 5-aminosalicylic acid; BPO, Benzoyl peroxide.

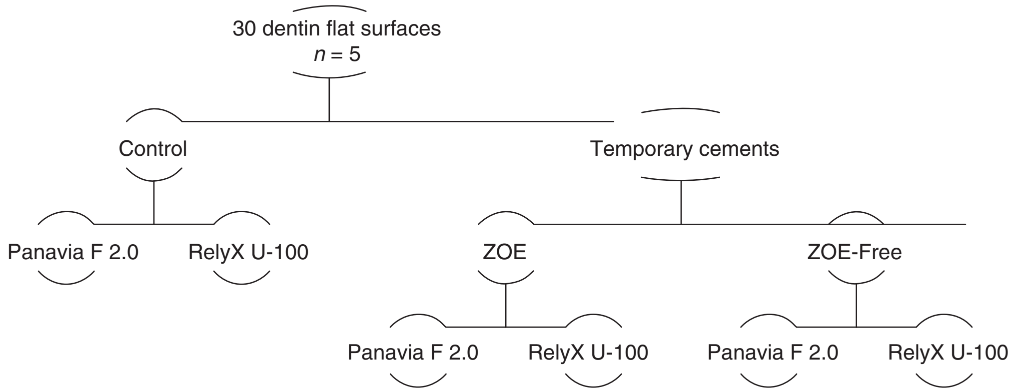

Figure 1. Diagram of experimental design.

cemented with a temporary cement (zinc oxide and eugenol-based temporary cement or eugenol-free zinc oxide cement).

Temporary cementation

Cementation was performed with either a zinc oxide and eugenol-based temporary cement (Provy, Dentsply, Petrópolis, RJ, Brazil) or an eugenol-free zinc oxide cement (RelyX Temp, 3M/ESPE). The cements were used in accordance with the manufacturers' recommendations. Temporary cements were applied to acrylic resin blocks and those were gently seated on the dentin surface using finger pressure (Figure 2A); excess cement was removed. The specimens were stored in distilled water at 37°C for 7 days.

After this period, the temporary blocks were mechanically removed (Figure 2B) and a stainless steel spatula was used to remove the temporary material (to avoid scaling of the dentin) until the dentin surface was visually (macroscopically) free of any temporary cement.

The teeth in the control groups received no temporary cementation. All teeth were cleaned with pumice-water (Pasom Materiais Odontológicos LTDA, SP, Brazil) slurry for 60 s using a slow-speed handpiece and rinsed with an air–water stream (20 s).

Final cementation

The internal surfaces of the resin composite blocks were treated with a bonding agent (Silano, Dentsply). Two different resin cements were used for the groups:

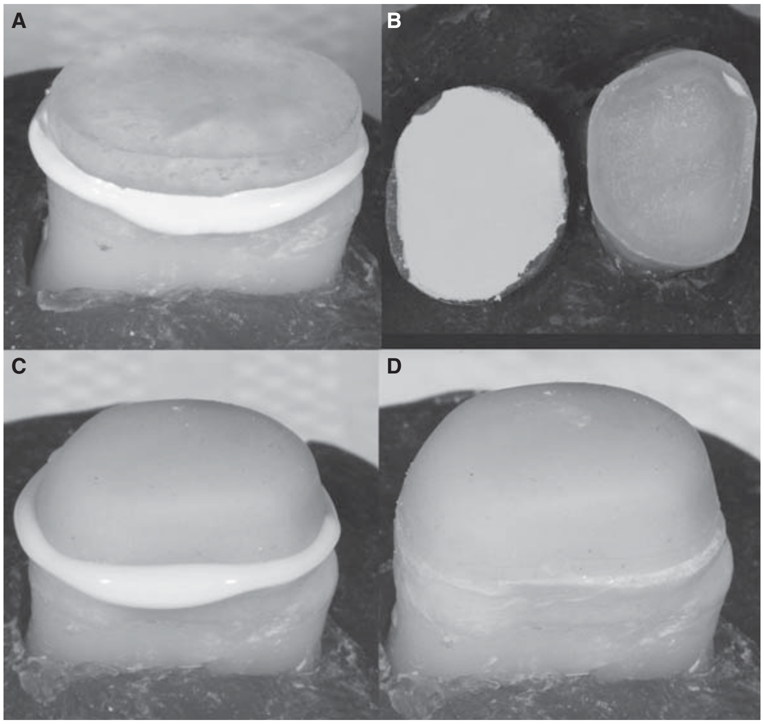

Figure 2. Cementation of temporary acrylic resin block and resin composite block: (A) Cementation of acrylic resin block. (B) Temporary block was removed mechanically. (C) Final Cementation: resin cement was applied onto the dentin and resin composite block. (D) Specimen final.

Table II. Mean and standard deviation (MPa) of microtensile bond strengths of tested groups as well as statistical analysis (*).

	Temporary cements		
Resin cements	Control	ZOE	ZOE-free
Panavia F 2.0	32.8 ± 4.4^A	10.8 ± 3.5^B	13.5 ± 4.0^B
RelyX U-100	14.9 ± 6.1^B	8.9 ± 3.0^B	12.2 ± 5.3^B

*Equal letters are not statistically significant (Tukey test, $p > 0.05$).

self-etching (Panavia F 2.0) and self-adhesive cement (RelyX U-100) were applied onto the dentin and resin composite blocks (Figure 2C) in accordance with the manufacturers' instructions (Table I).

After cementation, a pressure of 5 N was applied to the resin composite blocks with a weight for 5 min [8]. The vestibular, lingual and proximal surfaces were light-activated with a halogen light appliance (Opilux 501, SDS Kerr) for 20 s at an intensity of 600 mW/cm^2 (Radiometer, SDS Kerr). Next, the specimens were stored in distilled water at 37°C for 24 h (Figure 2D).

Microtensile bond test

Afterwards, teeth were serially sectioned into slabs that ran perpendicular to the adhesive tooth interface and the slabs were then sectioned into beams, each with a cross-sectional bonded area of ~0.8 mm^2, using a diamond saw (ISOMET 1000, Buehler). The cross-sectional area of each beam was measured to the nearest 0.01 mm using a digital caliper (Absolute Digimatic; Mitutoyo, Tokyo, Japan) and the measurements were recorded. The number of beams per tooth that prematurely debonded (D) during specimen preparation was recorded and those beams were not included in the statistical analysis.

In each storage time interval, individual bonded beams were glued to the Geraldelli device (Odeme, Joaçaba, SC, Brazil) with a cyanoacrylate adhesive (Super Bonder, Loctite, São Paulo, Brazil). Each stick was stressed to failure using a universal testing machine (Instron 3342, Canton, MA) at a crosshead speed of 1.0 mm/min. Maximum tensile load was divided by specimen cross-sectional area to express the results in units of stress (MPa).

Failure pattern analysis

The fractured surface of each test specimen was evaluated under a stereoscopic loupe (Kozo Optical and Electronical Instrumental, Nanjing, Jiangsu, China) at 40× magnification and classified as adhesive/mixed (A/M, failure at the resin–dentin interface that included cohesive failure of both neighbouring substrates), cohesive within dentin (CD, failure exclusively within the dentin) or cohesive within materials (CM, failure exclusively within the cement or resin material). The failure modes were expressed in percentages. Two specimens of each group were randomly selected for evaluation of the interface by scanning electron microscopy (Hitachi TM3000, Tokyo, Japan).

Statistical analysis

The data for each cement type were then subjected to two-way repeated-measure ANOVA and the Tukey test for pair-wise comparisons ($\alpha = 0.05$).

Results

The ANOVA revealed a significant effect for the interaction between the factors ($p = 0.0001$). Comparisons according to the Tukey test are shown in Table II. The highest bond strength values were found in control specimens (with no temporary cement) cemented with the self-etching resin cement system; the differences in bond strength values between this group and all others were statistically significant ($p < 0.05$). The two temporary cements evaluated led to a decrease in bond strength only when self-etching cement was used ($p < 0.05$). Neither temporary cement reduced the bond strength of self-adhesive cement ($p > 0.05$). In the absence of any temporary cement, self-adhesive cement exhibited a lower shear bond strength than self-etching cement ($p < 0.05$). In the presence of temporary cement, there was no statistical difference between the two resin cements ($p < 0.05$).

Table III. Number of specimens in each fracture pattern mode (percentage) and premature failure under different experimental conditions.

	Experimental Conditions											
	Control				ZOE				ZOE-free			
Resin cements	A/M	CM	CD	PF	A/M	CM	CD	PF	A/M	CM	CD	PF
Panavia F 2.0	47 (81.0)	3 (5.2)	8 (13.8)	3	21 (95.5)	0 (0.0)	1 (4.5)	23	35 (100)	0 (0.0)	0 (0.0)	18
RelyX U-100	31 (81.6)	4 (10.5)	3 (7.9)	16	34 (82.9)	7 (17.1)	0 (0.0)	11	36 (85.7)	6 (14.3)	0 (0.0)	18

A/M, predominant failure at the resin–dentin interface; CM, predominantly cohesive failure in resin or cement; CD, cohesive failure in dentin; PF, premature failures.

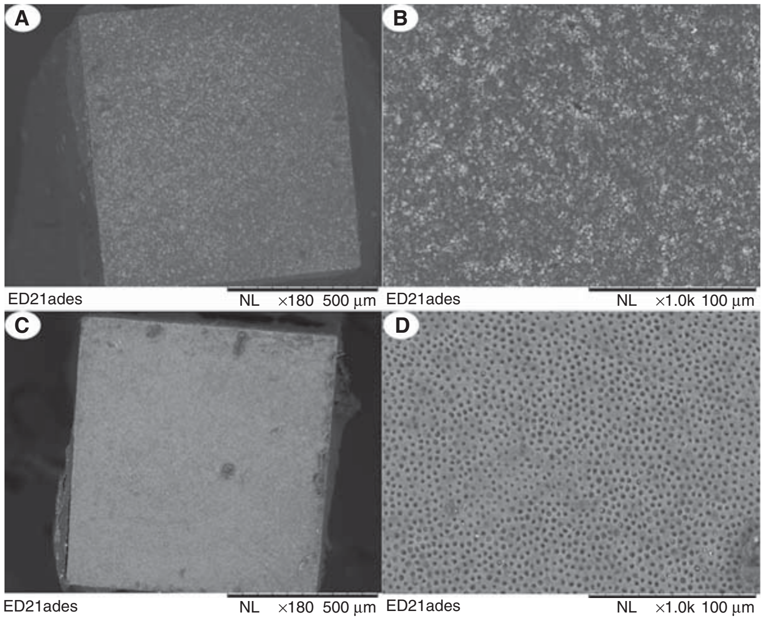

Figure 3. Representative SEM images of an adhesive failure of the self-etching resin cement Panavia F 2.0 (Control Group). (A) Material side of fractured specimen. (B) Higher magnification of the material side. (C) Dentin side of fractured specimen. (D) Higher magnification of the resin side of the same specimen shows adequate demineralization in the dentin, with visualization of some open dentinal tubules.

The failure mode distributions for all conditions tested are shown in Table III. The predominant failure mode of the cements was adhesive/mixed, occurring in both the resin cement and the adjoining dentin (Figures 3 and 4).

Discussion

The present study found that the bond strength of self-adhesive cement was not influenced by temporary restorations performed with eugenol-containing or eugenol-free materials. The bond strength of

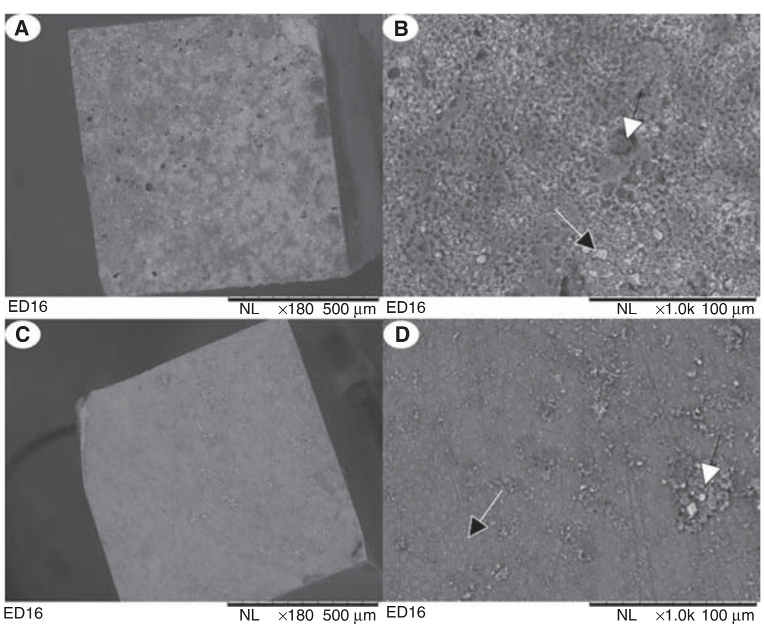

Figure 4. Representative SEMs of an adhesive failure of the self-adhesive resin cement Relyx U-100 (ZOE Group). (A) Material side of fractured specimen. (B) Higher magnification of the material side shows porosities (white arrow) and the presence of granular particles on a dentin surface that had been in contact with temporary cements, even after cleaning with pumice (black arrow). (C) Dentin side of fractured specimen. (D) Higher magnification of the dentin side of the same specimen shows poor acid conditioning on the dentin and inadequate visualization of dentinal tubules (black arrow) and particles of the temporary cements (white arrow).

self-etching cement, on the other hand, was reduced by temporary restorations, irrespective of the type of temporary cement (ZOE or ZOE-free).

Usually, the use of a temporary eugenol-free cement reduces the bond strength of the subsequent permanent cement to the dentin [9]. This negative effect may not be caused by eugenol but rather by residual particles of temporary cement [10]. Frankenberger et al. [14] showed that contamination of dentin with temporary cement is detrimental to the strength of the subsequent bond of permanent cement to the dentin and the issue of contamination with temporary cement is crucial.

As regards the chemical influence of any remaining temporary materials on material bonding, cement residues may act as a physical obstacle, preventing proper contact between resin cements and dentin. Finally, the accumulation of large amounts of salts around the remains of temporary cements may prevent the reaction of acidic monomers with dentin [15].

It has been reported that mechanical removal of temporary cements is not fully effective, as cement remnants can be observed by means of SEM [1,9] or atomic force microscopy [8] on dentin surfaces which had appeared to be macroscopically clean. Figures 4B and D clearly show the presence of temporary cement residues on such a surface.

When self-etching cement systems are used, residual particles of temporary cements that are not dissolved by the acidic primer may impair the permanent cement's penetration into the smear layer and decrease the bond strength. It is reported that the resin cement Panavia F 2.0 contains the acidic monomer 10-methacryloyloydecyl dihydrogen phosphate (MDP), which forms chemical bonds with ions calcium (Ca^{+2}) around the collagen fibrils of the hybrid layer. A stable MDP-Ca^{+2} bond enhances the strength of the adhesive layer [16].

Therefore, it appears that the low bond strength values found for Panavia F 2.0 cement in the groups with temporary cementation may be explained by the inability of the monomer 10-MDP to react with tissues rich in Ca^{+2} due to the barrier created by the presence of temporary cement particles on the dentin surface. This sensitivity of Panavia cement may be deduced from the larger number of beams lost in the groups with temporary cementation in comparison with the control group (Table III).

The results of this study are in agreement with those found by Ribeiro et al. [8], namely that self-etching adhesive exhibited reduced bond strength values in the groups in which eugenol-containing temporary cements were used. In the groups in which an eugenol-free temporary cement was used, on the other hand, the results were statistically similar to those of the control group. Ribeiro et al. [8] used an aggressive self-etching adhesive (Adper Prompt, 3M/ESPE) [17], which may have contributed to the dissolution/removal of the temporary cement particles when no eugenol was present. In the present study, a primer with moderate acidity was used (pH 2.4), but it appears to be incapable of removing all of the contaminated tissue.

The bond strength values in the control group for self-etching cement (Panavia F 2.0) were close to those found in the literature [18,19]. In addition to the formation of chemical bonds, the high bond strength values of self-etching resin cement used in this study are due to this product's capacity to remove the smear layer and expose the dentinal tubules, guaranteeing a micromechanical bond (Figure 3D).

The self-adhesive provisional cements groups exhibited no reduction in bond strength values compared with the control group. The self-adhesive cement interacted only superficially with the underlying dentin; no hybrid layer or resin tags were observed when this material was applied to dentin [20]. Therefore, its bond to dentin consists solely of macromechanical bonding through the irregularity of the interface. De Munck et al. [20] observed an irregular interaction zone where this occurs, ranging from almost 0 to 2 mm in thickness. The low bond strength values of the self-adhesive resin cement may be caused by two factors: (1) the cement's high initial pH was shown to be incapable of removing the smear layer and infiltrating into the dentin layer; and (2) its high viscosity makes penetration into small sites, which would enable micromechanical bonding to occur, more difficult. An initially low pH is important for enamel and dentin etching, but high pH (5.0) values were found for another self-adhesive cement with the same composition as the one used in the present study [21].

This superficial interaction of the self-adhesive cement with dentin may be observed in Figure 4D, in which it is possible to note an almost intact smear layer, with no resin tags and no exposure of dentinal tubules. According to Monticelli et al. [22], self-adhesive cements were unable to demineralize/dissolve the smear layer completely and the absence of micromechanical retention with dentin may be responsible for the previously reported bond strength values, which have consistently been weaker than those of conventional resin-based cements.

All of these factors causing very superficial interaction led to the bond strength being very low in the control group of self-adhesive resin cement compared to that in the control group of self-etching resin cement. Nevertheless, self-adhesive resin cement still offers an advantage in that it does not appear to be very sensitive, given that a reduction in bond strength occurred, although it was not statistically significant.

With regard to the presence of eugenol, it is believed that the presence of residues rather than the presence or absence of eugenol is the main factor that may compromise the quality of a permanent bond to dental substrate. Takimoto et al. [1] demonstrated that the application of either eugenol-containing or eugenol-free temporary cement reduced the free surface energy value and changed the wettability of dentin.

One of the limitations of the present study is that, usually when the microtensile bond strength test is employed, the bonding performance is tested in a flat dentin surface. Results from flat dentin surfaces do not mimic what occurs in most of the clinical situations. Under more realistic conditions, lower bond strength values with a high variability of the data is obtained as there are many other factors challenging the bonding to the dental substrates.

For instance, Silva et al. [23] evaluated the bond strength values when tensile forces were applied in adhesive junctions prepared at 10, 20 and 30° to the usual perpendicular interfaces, trying to simulate a dentin surface, that occur clinically. Reduced bond strength results were obtained for the higher angle (30°). The higher the angulation, the higher was the cross-sectional areas. Higher cross-sectional areas contain more flaws (bubbles, etc.), which lower the bond strength values. Besides that, the higher angulation produced a cross-sectional area with different adhesive thickness, which was also reported to be an important factor on bond strength values [23–25]. Additionally, as the samples had different angles onto the joint interface, the off-axis load applied produced a bending moment, consequently reducing the bond strength values. Future studies need to be done for testing this hypothesis.

Within the limitations of the present study, the previous use of eugenol-containing or eugenol-free temporary restorations did not interfere with the microtensile bond strength of self-adhesive resin cements.

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