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ORIGINAL ARTICLE

Bond strength of self-adhesive resin cements to different treated indirect composites

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Abstract
Objectives The objective of this study was to determine microtensile bond strength (μTBS) to dentin of three self-adhesive and a total-etch resin cements used for luting different treated indirect composites.
Materials and methods Composite overlays (Filtek Z250) were prepared. Their intaglio surfaces were ground with 600-grit SiC papers and randomly assigned to three different surface treatments: no treatment, silane application (RelyX Ceramic Primer), and silane agent followed by a bonding agent (Adper Scotchbond 1 XT). The composite overlays were luted to flat dentin surfaces of extracted human third molars using the following self-adhesive resin cements: RelyX Unicem, Maxcem Elite and G-Cem, and a total-etch resin cement, RelyX ARC. The bonded assemblies were stored in water (24 h, 37 °C) and subsequently prepared for μTBS testing. Beams of approximately 1 mm^2 were tested in tension at 1 mm/min in a universal tester (Instron 3345). Data were analyzed by two-way ANOVA and Student–Newman–Keuls tests (α=0.05).
Results A significant influence of the resin cement used was detected. Composite surface treatment and the interaction between the resin cement applied and surface treatment did not affect μTBS.
Conclusions Surface treatment of indirect resin composite did not improve the μTBS results of dentin/composite overlay complex. Self-adhesive resin cements tested obtained lower μTBS than the total-etch resin cement RelyX ARC. Specimens luted with Maxcem Elite exhibited the highest percentage of pretesting failures.
Clinical relevance Surface treatment of indirect resin composite with silane or silane followed by a bonding agent did not affect bond strength to dentin.

Keywords Bond strength · Self-adhesive resin cement · Luting · Dentin · Surface treatment · Indirect composite

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Introduction

The bond between indirect composite restorations and tooth structure is challenging, as two different interfaces need to be considered: the one established between dentin/enamel and the resin cement, and the one between resin cement and the precured resin composite. The bond strength of these interfaces should be optimized because the weakest one will determine the final bond strength of the cemented restoration [1].

Indirect composite restorations are subjected to an additional postcure of light and/or heat to increase resin conversion that enhances their wear resistance but lessens the potential for chemical bonding as the quantity of residual free carbon double bonds decreases [2, 3]. Bonding relies upon mechanical retention and residual free carbon bonds for chemical adhesion [4]. Several surface treatments of indirect composite restorations have been proposed in order to improve the bond between indirect composite restoration and resin cement [3, 5]. Increasing roughness of indirect composites by several techniques provides better mechanical interlocking and increases the probability of finding residual free carbons through the larger surface area [8]. Some of these techniques include airborne particle abrasion [3], hydrofluoric acid [5], roughening with diamond points, or carbide burs [6].

Other specific treatments aim to improve chemical bonding between resin cement and indirect composite restorations. The most common method used in everyday clinical practice is silanization of indirect composite surface [4, 7]. Silane agents are bifunctional molecules used to create a chemical bond between the inorganic fillers of the indirect composite and the methacrylate monomers of the resin cement matrix. The silanol group of a silane molecule reacts with silica on the glass filler surface, and the methacrylate group in silane molecule forms a covalent bond with the resin matrix [5]. Moreover, silane agents increase the wettability of the composite by making the surface hydrophobic [3, 4, 7]. The following application of an unfilled resin agent would improve the wettability of the indirect composite and allow the composite to flow into the irregularities of the precured composite [8, 9]. Nevertheless, the effect of treating the indirect composite surface with a silane agent, alone or followed by a bonding agent, on the adhesive performance of dentin/indirect composites is not entirely clear [8].

Resin cements are the materials of choice to lute indirect resin composite restorations [10]. Currently, these cements can be classified as total-etch, self-etch, and self-adhesive resin cements, according to dental tissues treatment or adhesion strategy [10–12]. total-etch, or etch-and-rinse resin cements, require the use of phosphoric acid followed by a bonding agent before the application of the resin cement [11]. Self-etch resin cements use an acidic primer, which is not rinsed away, to modify the dental tissue surfaces before bonding [10, 11]. Self-adhesive resin cements have been recently developed to simplify clinical procedures and overcome the technique sensitivity of multistep systems. These resin cements do not require any pretreatment of the tooth surface, and their application is accomplished through a single clinical step, similar to the more conventional zinc phosphate and polycarboxylate cements [13, 14]. RelyX Unicem (3M ESPE, St. Paul, MN, USA) was the first self-adhesive cement to be introduced in the market. Since then, the number of new materials is increasing [11, 15]. Nonetheless, RelyX Unicem continues to be the most tested material both in vitro [10, 12–20] and clinically [21–24].

The scientific evidence on the adhesive properties of many of the newly released self-adhesive resin cements is scarce [11, 18, 25], and the influence of composite surface treatment on bond strength of the tooth indirect composite complex is still largely unknown. Therefore, the purpose of this in vitro study was to determine microtensile bond strength to dentin of three self-adhesives and a total-etch resin cement after different indirect composite surface treatments. The null hypothesis was that the bond strength to dentin of indirect composites is not influenced by chemical treatment of the indirect composite surface, and that self-adhesive resin cements and a total-etch resin cement provide similar bond strengths to dentin.

Material and methods

Tooth preparation

Intact caries-free third human molars were selected for this study. All teeth were stored in a thymol solution at 4 °C until their use in the experiment. Flat coronal dentin surfaces were exposed by removing occlusal enamel and superficial dentin with a slow-speed, water-cooled diamond saw (Accutom 50; Struers GmbH, Copenhagen, Denmark). The exposed dentin surfaces were abraded with wet 600-grit SiC papers to create standardized smear layers. Prior to the luting procedure, the dentin surfaces were rinsed copiously with water and blot-dried with a cotton gauze for 5 s.

Composite overlays preparation

Composite cylinders were prepared by layering 2-mm-thick increments of a microhybrid light-cured resin composite (Filtek Z250, A3 shade; 3M ESPE) into a silicone mold (8 mm in diameter and 4 mm high). Each increment was light cured for 40 s with a LED unit (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) applying the high-intensity program (1,200 mW/cm^2). Specimens were removed from the mold, and the remaining five surfaces not previously light exposed were additionally light cured for 40 s each. The resin disk surface to be bonded was wet abraded on a polishing machine (EXAKT-Apparatebau D-2000 Nerderstedt, Germany) using 600-grit SiC papers in order to simulate the clinical condition of sandblasting [14]. The composite blocks were then ultrasonically cleaned for 5 min in distilled water and air-dried.

Before bonding the composite overlays, the intaglio surface of each overlay was cleaned with 35 % phosphoric acid gel (Scotchbond Etchant, 3M ESPE) for 15 s. The use of phosphoric acid etching on the ground surfaces removes smear debris caused by grinding, exposing the underlying surface and fillers, and according to Fawzy et al. [26] does not change the morphological pattern of the composite surface.

After water rinsing and air drying, the composite overlays randomly received one of the following treatments:

Group 1 (NOT): No additional chemical surface treatment.
Group 2 (S): A silane solution (RelyX Ceramic Primer, 3M ESPE) was applied and left undisturbed

on the surface of the indirect composite for 60 s, after which the surface was air-dried.

Group 3 (SA): The silane solution (RelyX Ceramic Primer) was applied as described above followed by a bonding agent. A thin layer of Adper Scotchbond 1 XT (3M ESPE) adhesive was applied and light cured for 20 s with a LED Bluephase unit.

Indirect composite overlays bonding

Teeth were randomly distributed into 12 experimental groups according to the previous surface treatments applied on the composite overlays and to the resin cement used. Three self-adhesive resin cements were investigated: RelyX Unicem (3M ESPE), Maxcem Elite (Kerr, Orange, CA, USA) and G-Cem (GC Corp., Tokyo, Japan), and a total-etch resin cement, RelyX ARC (3M ESPE).

The resin cements were mixed and applied according to the manufacturers' instructions listed in Table 1. Each composite cylinder was luted on the dentin substrate maintaining a constant pressure of 1 kg during the first 5 min [14], leaving the material to set in the self-curing modality. Finally, curing was completed by light irradiation from the top of the 4-mm-thick composite cylinder for 40 s with a LED curing unit (Bluephase, output 1,200 mW/cm^2). The bonded specimens were stored in a laboratory oven for 24 h at 37 °C and 100 % relative humidity until the microtensile bond strength test was performed.

Microtensile bond strength evaluation

After a 24-h storage period, the bonded assemblies were sectioned with a water-cooled diamond saw (Accutom 50; Struers, Copenhagen, Denmark) in both x and y directions, perpendicular to the adhesive interface to obtain sticks with a cross-sectional area of approximately 1 mm^2. The exact dimensions of sticks were measured using a digital caliper (Mitutoyo Corp., Kanogawa, Japan). Specimens were attached to the fixtures of a universal testing machine (Instron 3345; Instron Co., Canton, MA) with a cyanocrylate adhesive (Loctite Super Glue-3 gel; Henkel, Düsseldorf, Germany) and subjected to a tensile load at 1 mm/min until failure. The bond strength values were calculated in megapascals.

Failure modes were evaluated by a single operator under a stereomicroscope (Olympus SZX7; Olympus Co., Tokyo, Japan) at ×40 magnification and classified as cohesive (in cement, in dentin, or in composite), adhesive (between cement/dentin, AD, or composite/cement, AC, or both cement/dentin and composite/cement fractures, AD+AC), or mixed (adhesive and cohesive fractures occurred simultaneously). Each type of failure mode was expressed as a percentage of the total number of specimens in that group.

Statistical analysis

Pretesting failures were not included in the statistical analysis. A two-way ANOVA was applied to analyze the effect of the composite surface treatment and resin cement used on microtensile bond strength. Posterior comparisons were performed by Student–Newman–Keuls test. All statistical testing was performed at a pre-set alpha of 0.05 by means of SPSS 16.0 for Windows software (SPSS Inc., Chicago, IL, USA).

Results

The means and standard deviations of the microtensile bond strength of each experimental group are summarized in Table 2. All specimens luted with Maxcem Elite when indirect composite was treated with silane agent and bonding agent suffered pretesting failures. Thus, they were excluded from statistical analysis. Two-way ANOVA revealed that bond strength values were significantly influenced by resin cement used (F=18.060, p<0.001). However, composite surface treatment (F=0.891, p=0.413) and the interaction between the resin cement applied and surface treatment (F=1.164, p=0.330) did not significantly affect dentin bond strength. Post hoc comparisons among the cements revealed that the total-etch resin cement RelyX ARC displayed the highest microtensile bond strength mean values. The bond strengths of all self-adhesive resin cements tested, G-Cem, RelyX Unicem, and Maxcem Elite, were comparable and significantly lower than that of RelyX ARC.

The distribution of failure modes among resin cements is shown in Table 3. For the self-adhesive resin cements, the predominant failure mode was adhesive between the resin cement and dentin. For RelyX ARC, the distribution of the fracture modes was heterogeneous with no preference for one fracture type. More adhesive failures were observed for resin cements that yielded lower bond strength values, as Maxcem Elite.

Discussion

This study examined the effect of two chemical composite surface treatments on the adhesive properties of dentin-indirect composite using three self-adhesive resin cements and one total-etch resin cement. The first null hypothesis was accepted as the composite surface treatment did not affect the bond strengths.

Table 1 Chemical composition and application technique of the tested materials

Resin cement	Delivery system (cement)	Composition	Application technique
RelyX Unicem Aplicap 3M ESPE, St. Paul, MN, USA Shade: A3 opaque Batch:326313	Capsules, mechanically mixed 10 s	Powder: glass fillers, silica, calcium hydroxide, pigments, light cure initiators; liquid: methacrylated phosphoric ester, dimethacrylates, acetate, stabilizers, self-cure initiators, light-cure initiators 72 wt%	Activate the capsule for 2 s and mix it for 10 s with Rotomix (3M ESPE)[a]
Maxcem Elite Kerr Corp., Orange, CA, USA Shade: yellow Batch:2986507	Paste/paste dual syringe, direct dispensing through mixing tip	Methacrylated ester monomers, inert mineral fillers, ytterbium fluoride, activators and stabilizers 69 wt% filler weight	Automix cement[a]
G-Cem GC Corporation, Itabashi-Ku, Tokyo, Japan Shade: AO3 Batch:0707041	Capsules, mechanically mixed 10 s	Powder: fluoroaluminosilicate glass, initiator, pigment Liquid: 4-META, phosphoric acid ester monomer, water, UDMA, dimethacrylate, silica powder, initiator, stabilizer 65–70 wt%	Activate the capsule and mix it for 10 s with Rotomix[a]
RelyX ARC 3M ESPE, St. Paul, MN, USA Shade: Universal, A3 Batch:FAGH	Automatic dispenser, 2 pastes, hand mixed for 10 s	Etchant: 35 % H_3PO_4 Adper Scotchbond 1 XT: bis-GMA, HEMA, UDMA, dimethacrylates methacrylate functional copolymer (polyacrilic and polyitaconic acids), ethanol, water, silica nanofillers (5 nm; 10 wt%) Cement: bis-GMA, TEGDMA polymer, zirconia/silica filler	Etch dentin surfaces with Scotchbond etchant (15 s), rinse, blot excess water using a cotton pellet (keep dentin slightly moist). Apply 2–3 consecutive coats of adhesive for 15 s with gentle agitation; gently air thin (5 s); light cure for 10 s. Mix cement[a]
RelyX Ceramic Primer 3M ESPE, St. Paul, MN, USA Batch:6XM		Ethyl alcohol (70–80 wt%), water (20–30 wt%), methacryloxypropyl trimethoxysilane (<2 %)	Apply for 60 s and dry
Filtek Z250 3M ESPE, St. Paul, MN, USA Shade: A3		Organic matrix bis-GMA, UDMA, bis-EMA, TEGDMA. Filler: 60 % in volume (range of 0.19–3.3 μm)—zirconia and silica	

HEMA 2-hydroxyethyl methacrylate, *MEHQ* 4-methoxyphenol, *CHPO* cumene hydroperoxide, *TiO_2* 4-methacryloxyethyltrimellitate anhydride titanium dioxide, *4-META* 4-methacryloxyethyl trimellitate anhydride, *UDMA* urethane dimethacrylate, *bis-GMA* bisphenol glycidyl methacrylate, *TEGDMA* triethyleneglycol dimethacrylate

[a]Apply mixture onto the substrate. Let the cement autocure for 5 min under pressure; light cure through the composite overlay for 40 s

In contrast to our findings, a positive effect of silane application has been reported on bond strength when resin cements were luted to composite inlays [4, 5, 7, 27]. The silane agent used in our study, RelyX Ceramic Primer, is a single-phase prehydrolyzed solution containing 3-methacryloxypropyltrimethoxysilane diluted in an ethanol–water solution. According to manufacturer's instructions, RelyX Ceramic Primer must be used with a bonding agent. The subsequent application of a bonding agent as intermediate agent would ease the penetration of resin monomers and the resin cement into surface irregularities allowing a micromechanical interlocking [3]. Therefore, the application of a bonding agent has been advised to enhance bond strength [28, 29]. However, previous studies have also reported a failure of silane agents to predictably increase the bond of composite to composite, either in repair or luting procedures [3, 30–33].

The surface treatments based on mechanically increasing the surface roughness of resin composites seem to produce a more relevant effect on bond strength [34]. According to these authors, treatments based in sandblasting, aluminum oxide or silica coating, produced higher mean bond strength values of repaired and aged Filtek Z250, independently of the application of a silane, a bonding agent or the association of both. It should be highlighted that although silica

Table 2 Means microtensile bond strength in megapascals (standard deviation) and pretesting failures (PTF, in percent) of resin cements to different treated indirect composites (*n* = number of microbars tested)

Surface treatment/cement	NOT			S			SA			Total	
	mTBS	*n*	PTF	mTBS	*n*	PTF	mTBS	*N*	PTF	mTBS	*n*
RelyX Unicem	15.3 (6.2)	11	0	14.7 (7.2)	12	22.2	13.4 (8.7)	15	21.1	14.4 (7.41) b	38
Maxcem Elite	13.5 (8.6)	16	60.8	11.2 (8.6)	13	74.1	–[a]	0	100	12.5 (8.53) b	29
G-Cem	11.7 (4.6)	10	23.5	15.9 (5.4)	10	0	17.4 (5.6)	12	6.7	15.3 (5.8) b	32
RelyX ARC	24.8 (7.2)	14	0	21.7 (6.3)	12	20	27.1 (9.6)	15	0	24.7 (8.04) a	41

Different lowercase letters indicate statistically different microtensile bond strength values ($p<0.05$)

[a] No microbars were available due to premature debonding prior microtensile bond strength test

coating enables the chemical interaction with silane, no significant increase in mean bond strength values was detected in comparison with aluminum oxide sandblast [34].

The second null hypotheses that several self-adhesive and a total-etch resin cement provide similar bond strength of dentin composite overlay complex was rejected. According to our results, selection of the resin cement seems to be a more relevant factor when bonding indirect composite to dentin than its surface treatment.

In the present study, the total-etch resin cement RelyX ARC displayed the highest mean bond strength values. These results are in accordance with previous studies that reported a higher bonding capacity to dentin for RelyX ARC in comparison with RelyX Unicem [35–38], G-Cem, and Maxcem (the previous version of Maxcem Elite) [37]. In contrast, Piwowarczyk et al. [39] showed comparable bond strength values between RelyX Unicem and RelyX ARC. The higher mean bond strength values attained by RelyX ARC are in agreement with the observation of a higher prevalence of cohesive and mixed failures (Table 3), this trend being previously observed [12, 39]. Regarding self-adhesive resin cements tested, they all exhibited statistically similar mean bond strength values. Previous studies have also reported a similar performance for RelyX Unicem and G-Cem [12, 14, 25, 37].

It should be borne that in the present study, premature failures were not included in the statistical analysis. The presence of many pretesting failures has been associated to low bond strengths [12]; therefore, there may be an overestimation of the bonding potential [12, 37]. This is evident for Maxcem Elite resin cement that exhibited a percentage of pretesting failures ranging between 60 and 100 %, depending on the surface treatment of the indirect composite. Moreover, all the posttesting failures observed were adhesive between the dentin and the resin cement. A relatively poor bonding ability had been reported for Maxcem, [12, 40], as well as the occurrence of many pretesting failures [12, 37, 40]. However, no information is available about the bonding effectiveness to indirect composite of the self-adhesive resin cement Maxcem Elite. A recent study revealed high variation in micromechanical properties within self-adhesive resin cements [41], showing RelyX Unicem and G-Cem higher modulus of elasticity and microhardness than other self-adhesive resin cements tested, included Maxcem Elite. The authors suggested a correlation between modulus of elasticity obtained in their study with microtensile bond results of previous reports [41]. When other critical properties for clinical success have been assessed, such as marginal adaptation in dentin with self-adhesive resin cements, RelyX Unicem has shown comparable results to conventional resin cements tested [17, 42, 43] and better than those exhibited by Maxcem [17, 43].

Table 3 Failure mode distribution (in percent)

Resin cement	Failure mode				
	C	AD	AC	AD+AC	M
RelyX Unicem					
NOT	0	82	9	0	9
S	0	100	0	0	0
SA	0	66	0	27	7
Maxcem Elite					
NOT	0	100	0	0	0
S	0	100	0	0	0
SA	0	100	0	0	0
G-Cem					
NOT	0	80	0	0	20
S	0	50	0	20	30
SA	8	26	0	33	33
RelyX ARC					
NOT	21	14	22	0	43
S	33	8	8	18	33
SA	20	7	7	20	46

C cohesive within the cement, dentin, or composite; *AD* adhesive between the cement and dentin; *AC* adhesive between the composite and the cement; *AD+AC* adhesive at the dentin/cement level and composite/cement simultaneously; *M* mixed

Self-adhesive cements are structurally similar to conventional (total-etch or self-etch) resin cements but differ in monomer chemistry and setting reactions [44]. They contain multifunctional monomers with phosphoric acid groups that react simultaneously with tooth substrate and alkaline fillers to rapidly increase the initial low pH of the system. Water is generated as by-product and reused within the setting reactions to form a hydrophobic polymer chain [11]. However, despite low initial pH of this newly resin cements, limited decalcification/infiltration into the underlying dentin has been reported for the self-adhesive cements tested in the present study [13, 35–37, 40, 45, 46]. The insufficient demineralization effect of these cements has been attributed to the relatively high viscosity of the densely filled material [13, 37], resulting in insufficient adaptation of the cement even upon the application of seating pressure [45, 47]. With the aim to improve thixotropic properties and decrease the viscosity of this kind of resin cements, the influence of ultrasound application on inlays luting has been tested for RelyX Unicem and G-Cem, being more effective for the former [48].

The bonding efficacy of self-adhesive resin cements can also be attributed, in part or primarily, to their ability to chemically interact with dentin hydroxyapatite [37]. Due to insufficient demineralization effect of RelyX Unicem, Hiraishi et al. [35] reported that chemical bonding may have occurred between RelyX Unicem and the smear layer-covered dentin, leaving behind the interface between the unbound smear layer and the underlying intact mineralized dentin as the weakest link [35]. G-Cem also contains the functional monomer 4-MET which seems to have a low affinity of chemical bonding to hydroxyapatite [49].

The lower bonding efficacy to dentin detected for self-adhesive resin cements in comparison with the total-etch resin cement is in accordance with the high prevalence of adhesive failures detected when fracture surfaces were analyzed. Traditionally, it was considered that the bond between processed composite inlays and the resin cement was the weakest link in indirect composite restorations [50]. However, when using self-adhesive resin cements, adhesion to dentin is the weakest link in the indirect restoration/tooth complex, especially for cements that exhibit many pretesting failures [12]. Thus, efforts should be made in order to improve the bond capacity to dentin, either through adequate pretreatment of dentin or by modifying the chemistry of these new self-adhesive resin cements.

Besides a lesser bonding capacity, a deficient polymerization has been reported for several self-adhesive resin cements. In a recent study, Giraldez et al. [51] determined the early microhardness, as a measure of the degree of conversion, for several resin cements. Following the same procedure as in the present study, all resin cements evaluated were tested in dual curing mode and light irradiation was applied from the top of 4-mm-thick resin composite cylinder for 40 s, in order to simulate the clinical situation in which an indirect resin composite is luted in a proximal box. In that study, light irradiation was reduced by the presence of composite cylinder, but it was not completely blocked. Maxcem Elite exhibited an inadequate efficiency for not only the chemical-curing component but also the light-curing component [51]. The lower degree of cure in dual cure mode of the previous version of Maxcem Elite has also been reported by Vrochari et al. [15]. Interestingly, these findings are consistent with the lower microtensile bond strength values in the present study. G-Cem and RelyX Unicem exhibited intermediate values in both studies.

Acidic monomers, present in self-adhesive resin cements, have been shown to negatively affect the degree of cure of dual-cured materials, since they seem to interact chemically with the amine initiator that dual-cured resins contain [15]. The pH profile during the setting of self-adhesive cements differs from total-etch or self-etch resin cements as they present a high initial acidity [13, 52] and gradual pH rise [52]. Such low pH values allow the etching of enamel and dentin [53]. While RelyX Unicem displays a unique rapid rise in pH (neutrality was achieved only 15 min after dual curing and exhibited the final pH values within 1 h) [52], Maxcem and G-Cem still have pH values of less than 4 at 48 h after mixing [53]. There is no information about the setting pH profile of Maxcem Elite, and it is possible that only RelyX Unicem reveals a unique chemistry that allows a rapid rise despite the mode of cure, as it has been reported by Saskalauskaite et al. [52]. It has been speculated that if a low pH condition is maintained for too long, it might adversely influence the adhesion of mixed cement to the tooth structure [53].

Conclusions

According to the results of the present study, self-adhesive resin cements tested do not require application of intermediary agent (silane alone o silane plus bonding agent) to microretentive Filtek Z250 overlays to improve the bonding capacity of dentin-indirect composite complex. Lower bonding efficacy to dentin and a high prevalence of adhesive failures between the resin cement and dentin were detected for self-adhesive resin cements tested in comparison with the total-etch resin cement. Although the bond strength values were comparable for all self-adhesive resin cements tested, Maxcem Elite exhibited many pretesting failures.

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

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