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The Journal of Adhesion

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713453635>

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Online publication date: 19 November 2009

To cite this Article Türkmen, Cafer , Durkan, Meral and Öksüz, Mustafa(2009) 'Shear Bond Strength of Indirect Composites Luted with Three New Self-Adhesive Resin Cements to Dentin', *The Journal of Adhesion*, 85: 12, 919 — 931

To link to this Article: DOI: 10.1080/00218460903307779

URL: <http://dx.doi.org/10.1080/00218460903307779>

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Shear Bond Strength of Indirect Composites Luted with Three New Self-Adhesive Resin Cements to Dentin

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The aim of this study was to evaluate the shear bond strengths of indirect composites (those cured outside the mouth) luted by three different, recently developed, self-adhesive resin cements to dentin. Seventy caries-free mandibular third molar teeth embedded in acrylic resin and with exposed dentin surfaces were used. Teeth were randomly divided into seven groups. The following application protocols were carried out: a) Group 1 (control group)—direct composite resin restoration (Alert) with total-etch adhesive system (Bond 1 primer/adhesive); b) Group 2—indirect composite restoration (Estenia) luted by a resin cement (Cement-It) combined with the same total-etch adhesive; c) Group 3—direct composite resin restoration with self-etch adhesive system (Nano-Bond); d) Group 4—indirect composite restoration luted by the resin cement combined with the same self-etch adhesive; e) Groups 5–7—indirect composite restoration luted with self-adhesive resin cements (RelyX Unicem[®], Maxcem[®], and Embrace WetBond[®], respectively) onto untreated dentin surfaces. Shear bond strengths of the groups were performed with a universal testing device. Results were statistically analysed by student-t and one way ANOVA tests. The fractured surfaces were also examined by SEM. The indirect composite restorations luted with the self-adhesive resin cements (Groups 5–7) showed successful results compared with the other groups ($p < 0.05$). Group 4 showed the weakest bond strength ($p > 0.05$). Open dentin tubules were observed on the total-etch adhesive applied surfaces whereas a smear rich layer was found by SEM on the self-etch adhesive applied surfaces. The new universal self-adhesive resins may be considered an alternative for luting the indirect composite restorations onto the untreated dentin surfaces.

Keywords: Bond strength; Dentin; Indirect composite; Resin cement; Self-adhesive resin

Received 5 March 2009; in final form 6 July 2009.

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INTRODUCTION

The physical properties of composite restorations are improved when the composite is free of voids, and the resin matrix is maximally polymerized. Generating dense, well-cured restorations is best accomplished in the dental laboratory using devices that polymerize the composite under pressure, vacuum, inert gas, intense light, heat, or a combination of these conditions [1,2].

Indirect composites are used in an attempt to overcome some shortcomings of direct composite resin restorations, such as polymerization shrinkage and degree of conversion. Material manipulation out of the mouth allows better proximal contacts, morphology, and adjustment of the occlusal surface. Clinical indications for indirect composite restorations are based on the evaluation of the remaining tooth structure, intraoral conditions, and cost [1–3].

Laboratory-processed composite (LPC) inlays/onlays are more resistant to occlusal wear than direct composites, particularly in occlusal contact areas. They are less wear-resistant than ceramic restorations, however, and might offer less resistance to debonding at interfaces than ceramic restorations. They offer easy adjustment, low wear of the opposing dentition, good esthetics, and potential for repair. Moreover, extra-oral polymerization allows a higher conversion rate, influencing the composite's mechanical properties. Processed composite restorations are indicated when (1) maximum wear resistance is desired from a composite restoration, (2) achievement of proper contours and contacts would be difficult otherwise, and (3) a ceramic restoration is not indicated because of concerns about wear of the opposing dentition. Regarding the last-mentioned, the indirect composite likely would cause less wear of the opposing dentition than a similar ceramic restoration [2–5]. The internal surface of indirect restorations can be treated with sandblasting, hydrofluoric acid, or silane coupling agents, and with the combination of these treatments. The air-abrasion technique produces a rough surface, while silane creates a chemical adhesion between the inorganic fillers and the organic matrix of the bonding agent. Hydrofluoric acid has been used to etch all-ceramic restorations; however, its effects on different filler particles of composite resins have not been effective in producing high bond strengths of resin cement bonded to indirect composite restorations [3].

Several other LPCs have been introduced in recent years. These are sometimes called *polymer glasses*, *filled polymers*, or *ceramic-optimized resins* (“ceromers”). Some manufacturers have recommended their use not only for inlays/onlays and some single-unit crowns, but also with fiber reinforcement, for splints, and short-span

fixed partial dentures. The long-term clinical performance of such applications is unknown [2].

LPC esthetic restorations constitute a substantial portion of contemporary esthetic restorative treatments. Tooth-colored inlays, onlays, veneers, and crowns are now routinely bonded to the tooth substrate *via* the use of adhesive resin cements. Adhesive resin cements have the ability to bond to both tooth structure and restoration. The integration produces reinforcement of both structures, and reduces microleakage at the restoration-tooth interface, postoperative sensitivity, marginal staining, and recurrent caries.

Adhesion of resin cement to processed composites has traditionally been difficult to achieve. Roughening the composite surface by bur or by sandblasting, followed by silanization has been recommended as a predictable means for enhancing retention between resin cements and the indirect composite restoration [5].

At the tooth surface, an adhesive system is used to bond the resin cement to the tooth substrate. Currently, all adhesives are categorized as either etch-and-rinse or self-etch adhesives. A multi-step application technique is time consuming and rather technique sensitive and, consequently, may compromise bonding effectiveness. And also, because of the complex nature of the anatomical structure of the dentin, its etch pattern may differ from enamel and may, as a result, affect the bonding of adhesive materials [5,6]. Recently, the self-adhesive universal resin cements (RelyX Unicem[®], Maxcem[®], Embrace WetBond[®]) without surface pre-treatment of dentin or LPC have been introduced. These self-adhesive resin cements (SRCs) are based on a new monomer, filler, and initiation technology. The manufacturers state that the organic matrix consists of newly developed multifunctional phosphoric acid methacrylates. The phosphate-based acidic methacrylates can react with the basic fillers in the luting cement and the hydroxyapatite of the hard tooth tissue [5,7].

Shear strength testing is perhaps more clinically applicable because resistance to shear stresses are thought to be important in retaining restorations that have been cemented to dentin surfaces [8–11]. Many *in-vitro* studies have reported the shear bond strength of different adhesive systems used in combination with dentin [4–7,11]. Little information, however, is available in the literature with regard to the shear bond strength of the indirect composite restoration cemented with self-adhesive luting cements and different luting composites categorized by their adhesive system.

The purpose of this study was to assess the shear bond strength of indirect composite restorations cemented with three new universal self-adhesive resin cements (SRCs), total-etch, and self-etch adhesive

systems to dentin compared with direct posterior composite restorations (control groups). In addition to the shear bond test, surfaces were observed for failure mode and the adhesive interface between the tooth and resin and the conditioned tooth surface without any bonding were evaluated morphologically under the scanning electron microscope (SEM).

METHOD AND MATERIALS

Specimen Selection and Involved Materials

Seventy intact, non-carious, unrestored human third molars, extracted for therapeutic reasons in patients (age range 20–40 years), were stored in an aqueous solution of 0.5% chloramine T at 4°C for up to 30 days. The teeth were embedded in chemically cured 2 × 2 × 2 cm acrylic resin blocks, approximately 2 mm from the cemento enamel junction. The un-mounted part of the teeth crowns were then cut by a diamond cylindrical bur parallel to the acrylic resin block surface. The specimens were randomly divided into seven experimental groups.

Preparation of the Composite Blocks

Rectangular stainless steel metal 5 mm long, 4 mm wide, and 4 mm high were prepared and the inside of the molds were coated with Vaseline[®] before placing the composite material. The composite restorative materials were placed into this mold in two increments and each increment was light-cured for 180 s with a polymerization device (Estenia[®] CS-110 light and heat curing unit, Kuraray Dental, Osaka, Japan). After light polymerization, the composite blocks were removed and an air-barrier paste (Estenia[®], Kuraray Dental) applied to all of their surfaces. The blocks were then heat-polymerized at 160°C, for 15 min in the same device (Estenia CS-110). The surface of the blocks to be cemented or bonded to dentin was abraded with 50-µm aluminum oxide (Korox[®], Bego, Bremen, Germany) at 2 atm pressure in Topstar[®] Z3 device (Bego).

Bonding and Luting Procedures

One total-etch adhesive system (5th generation), Bond-1 Primer/Adhesive[®] (Jeneric Pentron, Wallingford, USA); one self-etch adhesive system (6th generation), Nano-Bond Self-etch Primer[®] and Adhesive (Jeneric Pentron); one resin-based, Cement-It[®] (Jeneric Pentron); and three SRCs, **a**) Rely X Unicem[®] (3M ESPE, Seefeld, Germany), **b**) Maxcem[®] (KerrHawe SA, Bogge, Switzerland), and

c) Embrace WetBond[®] (Puldent, Watertown, USA), were used for bonding and luting procedures. A posterior composite resin, Alert[®] (Jeneric Pentron), was also used as a direct restoration material (control group). Their application protocols are listed in Table 1.

For total-etch adhesive system groups (Groups 1 and 2), dentin surfaces were conditioned with 37% aqueous phosphoric acid etch for 15 s. Each surface was then rinsed thoroughly and the excess moisture was removed by using a light stream for 2 s, and the surface was left moist for the Bond 1 Primer/Adhesive (Group 1), while the surface was dried for 20 s for the Cement-It[®] (Group 2). The primer, adhesive, and resin cement applications to dentin were performed following manufacturers' directions. The adhesives were photo-activated for 20 s using a light unit (Bluephase[®] C5, Ivoclar Vivadent, Schaan, Liechtenstein). The intensity of the light (600 mW/cm²) was controlled by a radiometer (Caulk Dentsply, Milford, DE, USA).

The composite resin (Alert) was placed in two increments and each increment was light-cured (Bluephase C5) for 40 s on the pretreated dentin surface into the metal mold (5 × 4 × 4 mm) (Groups 1 and 3).

In Groups 2 and 4, the restorations (direct composite and indirect composite) were cemented with a resin cement (Cement-It) after adhesive application to the dentin surface.

In Groups 5–7, the following dual-cured self-adhesive cements (Rely X Unicem, Maxcem, Embrace Wet-Bond) were applied to the

TABLE 1 The Tested Materials and their Respective Application Procedures

Groups	Dentin pre-treatment	Bonding/resin cement	Restoration
1 (control gr.)	Etching (37% H ₂ PO ₄)	5th generation bonding (Bond-1 Primer/Adhesive [®])	Direct composite (Alert)
2	Etching (37% H ₂ PO ₄)	Resin cement (Cement-It [®])	Indirect composite (Estenia)
3	—	Self-etch adhesive system (Nano-Bond SEP and Adhesive)	Direct composite (Alert)
4	—	Self-etch adhesive system (Nano-Bond SEP and Adhesive) + Resin Cement (Cement-It [®])	Indirect composite (Estenia)
5	—	Self-adhesive resin cement (Rely X Unicem [®])	Indirect composite (Estenia)
6	—	Self-adhesive resin cement (Maxcem [®])	Indirect composite (Estenia)
7	—	Self-adhesive resin cement (Embrace Wet Bond [®])	Indirect composite (Estenia)

non-pre-treated dentin surfaces and then the composite blocks were pressed onto the adhesive under finger pressure for 30 s, after which excess cement was removed. Light-curing was performed on all four sides along the cement interface for 20 s. The specimens were stored in distilled water at 37°C for 24 h.

Shear Bond Strength Testing and SEM Examination

Shear bond strength tests were performed on a Zwick testing device (Z010 model, Zwick GmbH, Ulm, Germany) using a metal profile (100 × 10 × 4 mm) loading head (50 kg). Tests were performed at a crosshead rate of 0.5 mm/min until the composite and composite rectangles were dislodged from the dentin surfaces. Bond strength was calculated in megapascals (MPa). The obtained shear bond strength data were analyzed with a two-factor analysis of variance (ANOVA) and student-t test with level of significance of $p < 0.05$. Statistical software was used for statistical data analysis (Version 11.0; SPSS Inc., Chicago, IL, USA).

Following shearing, two failed specimens of each group (total 14 specimens) were randomly selected for scanning electron microscopy (SEM). The surfaces were gold sputter coated (BioRad-SC502, Fison, U.K.) and observed under SEM (JEOL JSM-5200, Tokyo, Japan) at 20 kV. Dentin surfaces and some mode of fracture surfaces were viewed and photographed usually at magnification × 1000.

RESULTS

Shear Bond Test

The mean shear bond strength values and standard deviations in MPa for cementing/luting the direct composite resin and indirect composite blocks to dentin are shown in Table 2.

The indirect composite restorations luted with Embrace Wet Bond (Group 7) exhibited the highest shear bond strength (10.45 MPa) while the composite blocks luted with self-etch adhesive + resin cement (Group 4) exhibited the lowest strength (6.42 MPa).

When comparing the groups with each other,

- a. The direct composite and indirect composite restorations bonded with total-etch adhesive systems (Groups 1 and 2) have almost equal bond strength values (8.19 and 8.26 MPa, respectively).
- b. The direct composite restoration bonded with the total-etch adhesive system (Group 1) showed a higher bond strength than that

TABLE 2 Shear Bond Strength Means (MPa) of Direct Composite and Indirect Composite Restorations Bonded/Luted to Dentin

Groups	Bond strength mean \pm SD	n
1 (total-etch adhesive \rightarrow composite)	8.19 \pm 0.87	10
2 (total-etch adhesive + resin cement \rightarrow ind. composite)	8.26 \pm 1.68	10
3 (self-etch adhesive \rightarrow composite)	7.41 \pm 2.80	10
4 (self-etch adhesive + resin cement \rightarrow ind. composite)	6.42 \pm 1.91	10
5 [self-adhesive resin cement (Rely X Unicem [®]) \rightarrow ind. composite]	8.51 \pm 3.03	10
6 [self-adhesive resin cement (Maxcem [®]) \rightarrow ind. composite]	7.49 \pm 2.01	10
7 [self-adhesive resin cement (Embrace Wet Bond [®]) \rightarrow ind. composite]	10.45 \pm 2.19	10

bonded with the self-etch adhesive system (Group 3) (8.19 and 7.41 MPa, respectively).

- c. The indirect composite restorations bonded/luted with total-etch adhesive system + resin cement (Group 2) showed a higher bond strength than those bonded/luted with the self-etch adhesive system + resin cement (Group 4) (8.26 and 6.42 MPa, respectively).
- d. The direct composite restorations bonded with the self-etch adhesive system (Group 3) showed a higher bond strength than indirect composite restorations bonded/cemented with self-etch adhesive + resin cement (Group 4) (7.41 and 6.42 MPa, respectively).

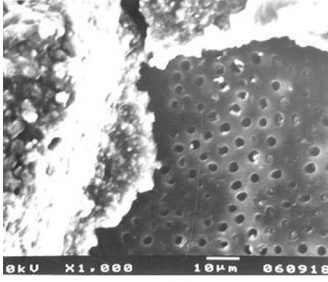
But no statistically significant differences were found between the above groups ($p > 0.05$).

Of the indirect composite restorations cemented with SRCs, the highest bond strength was recorded for Embrace Wet Bond (Group 7) (10.45 MPa), and Maxcem (Group 6) showed the lowest bond strength (7.49 MPa). This was statistically significant ($p < 0.05$).

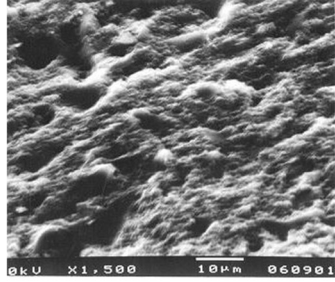
SEM Evaluation

The SEM photographs of the fractured surfaces of all groups are shown in Figure 1a–g. Some adhesive and cohesive failure modes were observed on the surfaces of all the specimens:

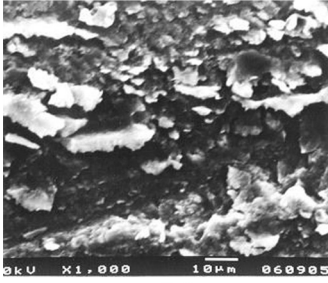
- a. In Group 1 (total-etch adhesive syst. + direct composite rest.)—adhesive failure between the dentin-bonding substrate interface, cohesive failure within the composite resin, and opened dentin tubules (Fig. 1a).



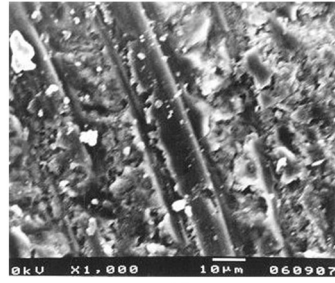
(a)



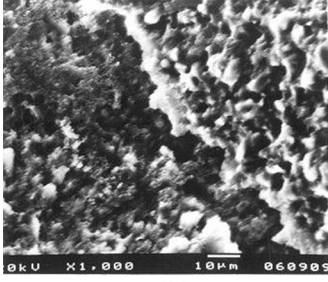
(b)



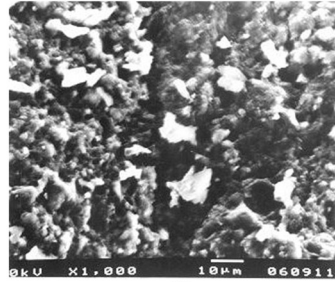
(c)



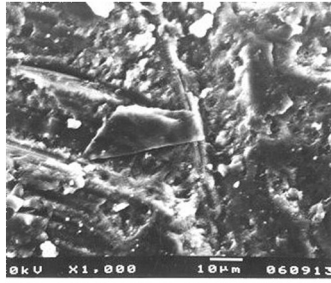
(d)



(e)



(f)



(g)

- b. In Group 2 (total-etch adhesive syst. + resin cement + composite block)—adhesive failure between resin cement-bonding and dentin-bonding interface, and also smear layer on the dentin surface (Fig. 1b).
- c. In Group 3 (self-etch adhesive syst. + direct composite rest.)—adhesive failure between the dentin-bonding substrate, cohesive failure within the composite resin, and smear layer on the dentin surface (Fig. 1c).
- d. In Group 4 (self-etch adhesive syst. + resin cement + composite block)—adhesive failure between composite block-resin cement substrate and resin cement-bonding interfaces, cohesive failure within the resin cement (Fig. 1d).
- e. In Groups 5, 6, and 7 (SRCs + composite blocks)—adhesive failures between the composite block-SRC, cohesive failures within the SRCs, only cement remnants were observed on the surface of the dentin (Fig. 1e–g).

DISCUSSION

Dentin-adhesive resins were originally formulated with separate etchants, primers, and adhesives, but they have evolved such that in some products the adhesive and primer are combined (a), in others the etchant and primer are combined (b), whereas in some, all three are combined. The (b) and (c) categories are considered “self-etching,” but products in the (c) category have been termed “self-etching adhesives.” Whether self-etching products are equivalent to earlier systems with separate etchants, also termed “total-etch” or “etch-and-rinse” systems, has not been established [12]. The self-etching product, Nano-Bond, evaluated in the present study has etchant, primer, and adhesive combined into a single component, allowing the resin

←
FIGURE 1 SEM photograph of the fractured surfaces. (a) Group 1—adhesive failure at the dentin/bonding interface, cohesive failure within the composite resin, and hybrid layer with some partially open dentin tubules; (b) Group 2—adhesive failures at the resin cement/bonding and dentin/bonding interfaces, and smear layer on the dentin; (c) Group 3—adhesive failure at the dentin/bonding interface, cohesive failure within the composite resin, and smear layer on the dentin; (d) Group 4—adhesive failure at the dentin/bonding interface, cohesive failure within the composite resin, and also separation through lengths of dentinal tubules with smear layer as a result of shear artifact; (e), (f), and (g) Groups 5–7, respectively—adhesive failures at the indirect composite/self-adhesive resin cements (SRCs), cohesive failures within the SRCs, and remnants of the SRCs remaining on the dentin surface.

restorative material to be placed more quickly than with other types of bonding agents. All self-etching products have the advantage of producing little discomfort when applied to unanesthetized but sensitive dentin, because rinsing and air drying are unnecessary. Limitations of the self-etching adhesive tested include the following: no capability for dual polymerization, which can be desirable for indirect adhesive restorations, and difficulty in preventing the acid resin from splashing onto adjacent teeth or soft tissue during air-drying.

Dual-polymerizing resin cement, Cement-It, can be polymerized by light or by chemical polymerization. These two polymerizing mechanisms form the basis for the wide spread use of these materials for definitive cementation of all-ceramic as well as composite and metal-based indirect restorations. Furthermore, dual polymerizing resin cements are characterized by high mechanical strength and excellent esthetic properties. Their chemical composition allows adherence to many dental substrates. However, resin cements require skillful handling, especially during the time-consuming bonding procedure, and when removing excess cement. The use of resin cements in clinical practice is complicated and technique-sensitive [13].

Some self-adhesive, dual-polymerizing universal resin cements (Rely X Unicem, Maxcem, and Embrace Wet Bond) have been recently introduced. The objective in developing these cements was to combine ease of handling (no pretreatment steps required) offered by glass ionomer cements with favorable mechanical properties, attractive esthetics, and good tooth adhesion. According to the manufacturer, bonding to the tooth structure can be achieved without any pretreatment steps, for example, without etching and priming. These self-adhesive universal resin cements are based on a new monomer, filler, and initiator technology. The manufacturer states that the organic matrix consists of newly developed multifunctional phosphoric-acid methacrylates. The phosphoric-acid groups of these molecules condition the tooth surface and contribute to adhesion [13]. The present study results showed the highest shear bond strength values in the restoration groups luted with SRCs.

In the present study, we also wanted to demonstrate pronounced differences among the adhesives in their bonding performance on dentine with the general trend that conventional systems with separate primers and bonding agents perform better than simplified systems that combine the functions of priming and bonding, irrespective of the etch-and-rinse or the self-etch approach [6,11,14]. But our results were different and less than the data from manufacturer's catalog and the literature [4,9,15]. This is related to the push-off piece (upper jaw) of the shear bond testing device. This rectangular piece (4 mm in

thickness) was used rather than a conventional cylinder because it impacted the specimen widely and so the load was applied to the flat surface (90°) of the restoration. The load in shear bond testing is a static load. Therefore, there is no difference between a cylinder and the rectangular piece that was used because the range of the restoration surfaces tested is the same.

The stresses at the interface of restorations are complex, but can be identified as mainly a tensile or shear type of stress, created either by forces working perpendicular to or parallel to the tooth surface [15]. Since the restorative materials evaluated were usually used in luting/cementing the inlay-onlay/crowns, the forces of displacement tend to be closer to that of the shear test. Therefore, in this study, a conventional shear bond test was used. Loads were applied by a metal profile on the rectangular restoration. As a consequence, different shear bond strengths may be reported from different laboratories directly related to the method of shear bond testing. The knife edge exerts the load on a more concentrated area of the bonded sample. Because the initial contact of the knife was at one point on the edge of the bonded sample, the stress was concentrated in a smaller area, resulting in premature failure of the dentin-adhesive or -luting cement bond [9].

Bond quality, however, should not be assessed on strength data alone, because the mode of failure is also important; this information may yield predictions of clinical performance. This is because, in the clinical situation, there are many fracture patterns [13,15]. Failure analysis revealed adhesive modes at the dentin-bonding, resin cement-bonding, composite block-resin cement, and composite block-SRC substrate interfaces and cohesive modes within the resin cement, SRCs, and composite resin. Cohesive failure was not observed within the composite blocks.

Thermocycling was not done in this study, because the thermocycling regimens used in reported studies differ with respect to the number of cycles, temperature, and dwell time (immersion of specimens in hot and cold fluids). The number of cycles ranges from 100 up to 50,000 cycles. The number of cycles is usually arbitrarily set, which makes it difficult to compare published results. It is estimated that approximately 10,000 thermal cycles correspond to 1 year of clinical function. This estimate is based on the hypothesis that such cycles might occur 20 to 50 times a day, which makes the 500-cycle regimen proposed by the ISO standard (ISO TR 11450) insufficient to simulate the long-term challenging of bond durability. Many reports that used the ISO protocol concluded that thermocycling did not affect the bond strength and microleakage of adhesive systems [16]. Thermocycling

seems to be a valid *in vitro* method to accelerate the aging of restorative materials. However, reasoning for the choice of temperature and timing conditions is rarely given. The varied number of cycles, temperatures, dwell time, and intervals between baths hinder comparison of the study results. Consequently, results obtained from thermocycling are contradictory. Abo-Hamar *et al.* [4] also concluded that thermocycling did not significantly affect the bond strength of the tested luting systems (Rely X Uncem, Syntac/Variolink II[®], Panavia F2.0[®], Dyract Cem Plus[®], Ketac Cem[®]) to dentin, whereas it significantly affected their bond strengths to enamel.

Thermocycling has been the most used method to stress the adhesive interface, while water storage has been shown to reduce bond strength, even after short period of storage, indicating that bonds degrade over time [16–18]. Biodegradation of restorative materials has been associated with undesirable effects on the surface and sub-surface, including the resin matrix, the filler content, and the matrix-filler interface. Morphological changes in the bond structure of the tooth-restoration interface aged in the oral environment for long periods have been reported [17,18]. The very hydrophilic nature of the self-etching resin blends will increase water sorption and permeability of resin-dentin interfaces, contributing to their hydrolytic instability after water aging [18]. Although a single-step adhesive is desirable clinically, the combination of acidic, hydrophilic, and hydrophobic monomers into a single solution may compromise the function of each one of these components. These simplified adhesives are more sensitive to water degradation. Fluid transudation from the underlying dentin was augmented, resulting in dilution and phase separation of the adhesive solution, as there is a reduced tendency of the hydrophilic and hydrophobic monomers to polymerize as copolymers. Water entrapment within the adhesive-rich layer may also occur, adversely affecting adhesive polymerization and bond longevity [16–18]. Therefore, in the present study, the specimens were not aged in the water.

This *in vitro* comparative study allowed an immediate assessment of the bond created between the SRC and the indirect composite restorative material. *In vitro* tests cannot adequately simulate clinical conditions in every detail. Additional clinical factors, such as the retentive and resistance form of the preparation, were not considered. The results of *in vitro* tests should be applied to the clinical situation with caution. It is admissible, however, to compare the measured *in vitro* results obtained under identical conditions. *In vitro* studies have limitations and long-term clinical studies would provide a better evaluation of material performance. A long-term study pertaining to Estenia would be of great benefit to the dental readership.

CONCLUSIONS

Based on the limitations of this *in vitro* study, the following conclusions may be drawn:

1. The self-adhesive universal resin cement, Embrace WetBond, produced the highest shear-bond strength to composite blocks, while the self-etch adhesive, NanoBond[®] + resin cement, and Cement-It[®] together produced the least bond strength.
2. There was no statistically significant difference between the total-etch and self-etch adhesive systems in shear bond strength.
3. Some adhesive and cohesive failures were observed in all groups.
4. The piece design of the shear bond testing device has a very important effect on the results of the bond strength values.

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