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ORIGINAL ARTICLE

Influence of photo-curing distance on bond strength and nanoleakage of self-etching adhesive bonds to enamel and dentin

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Abstract

Objectives. To assess the influence of light-curing unit tip distance on the microtensile bond strength (μ TBS) and nanoleakage of self-etching adhesives to enamel and dentin. **Materials and methods.** Flat buccal surfaces were prepared on 198 bovine incisors. The teeth were randomly assigned into nine groups for μ TBS ($n = 8$) and nanoleakage ($n = 3$) testing according to the adhesive system (Clearfil Protect Bond, Clearfil Tri-S Bond or One Up Bond F Plus) and distance from the light-curing tip (0, 3 or 6 mm). The bonded samples were tested in tension (0.5 mm/min) and nanoleakage was analyzed using SEM. **Results.** Clearfil Protect Bond exhibited the highest tensile strength on both enamel and dentin. Leakage was higher in samples exposed at a distance of 6 mm on enamel and 0 mm on dentin. One Up Bond F Plus experienced the greatest amount of nanoleakage on both substrates. **Conclusions.** Light-curing unit distance did not influence the μ TBS of the adhesives, but nanoleakage increased on enamel samples when photoactivation occurred at a distance of 6 mm.

Key Words: Microtensile bond strength, photoactivation, nanoleakage

Introduction

Self-etching adhesive systems were developed in an attempt to simplify the clinical procedure of dental adhesives application and to improve bond quality [1]. Self-etching adhesives are classified as two-step systems in which an acidic primer is used to etch the tooth surface for subsequent infiltration of a hydrophobic bonding resin [2] or one-step systems in which the acidic primer and bonding agent are applied simultaneously. One-step systems are packaged either in a single container or as two components [2,3], which are mixed immediately prior to application [4].

According to De Munck [5], adhesive systems are complex mixtures of hydrophobic and hydrophilic resin monomers dissolved in a mixture of solvents and water. One-step adhesives contain larger amounts of hydrophilic monomers [6]. Resins composed of

these monomers [3] behave as semi-permeable membranes [4] that permit water diffusion, which appears as nanoleakage indicated by the presence of water channels at the resin/dentin interface [7,8]. One-step self-etching adhesives are commonly associated with lower bond strengths because of their highly hydrophilic nature [3].

The clinical success of adhesive procedures depends not only on the bonding agent, but also on variables such as substrate condition and composition, clinical technique, restorative material, cavity shape and size and polymerization quality [9]. Regarding the polymerization quality, there are several clinical situations in which the curing procedure may be compromised, such as in Class II cavity preparations where the light cannot properly reach the bottom of proximal boxes. Although it is recommended that the light-guide be placed as near as possible to the bonded surface, this is

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Table I. Commercial names, manufacturers, composition and bonding procedures of the self-etching adhesives used in the present study.

Self-etching adhesives	Chemical composition	Bonding procedures
Clearfil Protect Bond - 2 steps, Kuraray, Okayama, Japan Batch Number: 61184	<i>Primer:</i> HEMA, 10MDP, hydrophilic DMA, 12-MDBP, water <i>Bond:</i> HEMA, 10MDP, BisGMA, N,N-diethanol-p-toluidine, hydrophilic DMA, Di-Camphorquinone, Silanated colloidal silica, surface treated sodium fluoride, bromide	(1) Primer application for 20 s (2) Mild air stream (3) Adhesive application (4) Light cure for 10 s
Clearfil Tri-S Bond 1 step, Kuraray, Okayama, Japan Batch Number: 61179	10MDP, BisGMA, HEMA, hydrophilic DMA, Di-Camphorquinone, Silanated colloidal silica, ethanol, water	(1) Adhesive application for 20 s (2) Dry with air stream (3) Light cure for 10 s
One Up Bond F Plus 1 step, Tokuyama Batch Number: UN36028	<i>Liquid A:</i> phosphoric acid monomer, MAC-10, bis-MPEPP, MMA <i>Liquid B:</i> HEMA, MMA, fluoroaluminosilicate glass, water, photoinitiator (aryl borate catalyst)	(1) Mix liquid A + liquid B (2) Application for 10 s (3) Light cure for 10 s

HEMA, 2-hydroxyethylmethacrylate; MDP, methacryloxydecyl di-hydrogen phosphate; DMA, dimethacrylate monomer; BisGMA, bisphenol A diglycidylmethacrylate; MAC-10, methacryloyloxy-1,1-undecandicarboxylic acid; MMA, methylmethacrylate; bis-MPEPP, 2,2'-bis[4(methacryloxypropyloxy) phenyl]propane.

often difficult to achieve in practice and it is not uncommon to have distances greater than 6 mm between the tip of the light curing unit and the gingival wall of the proximal box [10,11]. Extended distances between the curing unit tip and the bonded surface may reduce the intensity of the light reaching the resin, resulting in a lower degree of conversion [12] and incomplete polymerization, particularly near the gingival margins.

Adequate polymerization of bonding agents is important for ensuring the integrity of resin composite restorations and the distance of the light-curing unit from the bond may affect the performance of the bonded interface. Therefore, the objectives of the present study were to evaluate the bond strength of self-etching adhesives to enamel and dentin as a function of the distance of the light source and to determine whether the distance influenced the quality of hybridization in the restorations based on the amount of nanoleakage. The hypotheses tested were: (1) the distance of the light-guide from the resin does not influence the bond strength or quality of hybridization in self-etching adhesives applied to enamel and dentin and (2) there is no difference in bond strength or quality of hybridization among the adhesives tested.

Materials and methods

Specimen preparation

A total of 198 bovine incisors were selected and stored at 37°C in 0.1% thymol solution. The roots were separated from the crown at the cement–enamel junction using double-faced diamond disks (# 7020, KG Sorensen, Barueri, SP, Brazil) in a low-speed hand-piece. The lingual surface of each crown was removed and the buccal enamel and dentin were ground using

180, 320 and 600-grit silicon carbide abrasive papers (Carborundum, Saint-Gobain Abrasivos LTDA, Guarulhos, SP, Brazil) to create a flat surface. The thicknesses of the remaining enamel and dentin surfaces were measured using a digital caliper (Mitutoyo Sul Americana, São Paulo, SP, Brazil) to ensure similar substrate thickness in all specimens (2.5 mm enamel; 2.0 mm dentin). For teeth used in dentin bond strength testing, the enamel was entirely removed using silicon carbide abrasive paper. The teeth were randomly assigned to one of nine groups ($n = 8$) based on substrate (enamel or dentin), self-etching adhesive system (Clearfil Protect Bond, Kuraray, Okayama, Japan [CP], Clearfil Tri-S, Kuraray [CT] or One Up Bond F Plus, Tokuyama Dental Corporation, Tokyo, Japan [OU]) and curing-unit distance (0, 3 or 6 mm).

Immediately prior to adhesive application, the buccal enamel and dentin surfaces were ground using 600-grit silicon carbide abrasive paper under water cooling for 60 s to create a standardized smear layer. The adhesive systems were applied to the substrates according to the manufacturers' instructions (Table I).

A PTFE spacer 11 mm in diameter was placed between the light curing unit tip and the enamel/dentin surface to maintain curing distances of 3 or 6 mm. The specimens were light cured for 10 s. A silicone matrix of 4 mm in width and 6 mm in length was placed on the surfaces and two layers (2 mm thick) of resin composite (TPH3, A3, Dentsply Caulk, Milford, DE) were applied. Each layer was light cured for 40 s. The adhesives and composite resins were light cured using a halogen lamp (Optilux 501, Demetron LC, Sybron Kerr, Danbury, CT), with a constantly monitored irradiance of $\sim 650 \text{ mW/cm}^2$. Following restoration, the teeth were stored at relative humidity at 37°C for 24 h.

Table II. Means (MPa; standard deviations) of the microtensile bond strength test performed on enamel.

Adhesive system	Distance			
	0 mm	3 mm	6 mm	
Clearfil P	33.0 (11.4)	32.2 (7.8)	25.2 (6.9)	A
Clearfil T	26.7 (5.5)	26.9 (7.3)	23.0 (6.8)	B
One Up	25.2 (6.9)	21.9 (7.7)	21.7 (6.3)	B

Different letters indicate statistically significant differences (ANOVA two-way/Tukey ($\alpha = 0.05$) comparing adhesive systems ($p < 0.5$). There were no significant differences for the distance factor, as well as the interaction.

Microtensile bond strength test

The specimens were sectioned perpendicular to the adhesive interface in the incisio-cervical and mesio-distal directions using a low-speed diamond saw under water-cooling (Isomet 1000, Buehler, Lake Bluff, IL) to obtain 10 rectangular specimens from each tooth, each with an interfacial area of $\sim 0.8 \text{ mm}^2$. The specimens were stored in relative humidity for 24 h at 37°C .

The specimens were attached to the flat grips of a microtensile testing device using cyanoacrylate glue (Super Bond Gel, Locite Brasil Ltda) and were tested to failure in a Universal Testing Machine (Emic DL 500, São José de Pinhais, SC, Brazil) at a crosshead speed of 0.5 mm/min. The results were obtained in kgf and were converted to MPa by dividing the maximum load by the bonded surface area of each sample. The results were analyzed using two-way ANOVA (SAS Institute Inc., Cary, NC, v.9.1.3., 2002–2003) and Tukey's post-hoc test ($\alpha = 0.05$).

Nanoleakage

Leakage patterns were evaluated in three restored teeth from each experimental group. Samples obtained from each tooth were immersed in 50% ammoniacal silver nitrate (AgNO_3) [7] for 24 h at

Table III. Means (MPa; standard deviations) of the microtensile bond strength test performed on dentin.

Adhesive system	Distance			
	0 mm	3 mm	6 mm	
Clearfil P	23.5 (4.2)	23.1 (3.5)	23.1 (4.8)	A
Clearfil T	19.1 (4.6)	17.3 (8.4)	14.7 (4.4)	B
One Up	14.0 (5.6)	19.8 (7.7)	13.9 (3.5)	B

Different letters indicate statistically significant differences (ANOVA two-way/Tukey, $\alpha = 0.05$) comparing adhesive systems ($p < 0.5$). There were no significant differences for the distance factor, as well as the interaction.

Table IV. Means (%) of the nanoleakage analysis on enamel.

Adhesive system	Distance		
	0 mm	3 mm	6 mm
Clearfil P	10.6	13.1	14.8
Clearfil T	10.5	10.0	22.3
One Up	29.5	25.6	34.4

37°C in darkness. The specimens were then thoroughly rinsed in distilled water for 2 min and immersed in a photodeveloping solution (Kodak, Developer D-76, Kodak Brasileira, Ind. and Com. Ltda, São José dos Campos, SP, Brazil) for 8 h under fluorescent light, to reduce the silver ions to metallic silver grains within the voids along the bonded interface. The specimens were embedded in polystyrene resin (Erios Representações e Comércio Ltda, São Paulo, SP, Brazil) and wet ground using 600, 1200 and 2000-grit SiC papers and diamond paste (Arotec S.A. Indústria e Comércio, Cotia, SP, Brazil) compounds of $3.1 \mu\text{m}$ and $0.25 \mu\text{m}$ grain size. The specimens were cleaned in an ultrasonic bath (Ultrason Ultrason 1440 D - Odontobrás Ind. and Com. Med. Odont. Ltda, Rio Preto, SP, Brazil) for 10 min after each polishing procedure to remove debris, dried with absorbent paper, immersed in a solution of 50% phosphoric acid for 30 s, rinsed in distilled water and immersed in 10% sodium hypochlorite for 10 min. The specimens were then rinsed, dried at room temperature and immersed in increasing concentrations of ethanol (25%, 50%, 75%, 90% and 100%) for 10 min at each concentration.

The specimens were sputter-coated with carbon (Bal-Tec, SCD 050, Sputter Coater; MED 010, Balzers Union, Balzers, Liechtenstein) and examined in a field-emission scanning electron microscope (SEM) (JEOL JSM, 5600 LV, Tokyo, Japan), operating in \ high vacuum at 20 kV in backscattered electron mode. Images of specimens infiltrated with AgNO_3 were recorded and analyzed using the Image Tool 3.0 software (Periodontology Department, University of Texas, Health Science Center at San Antonio, TX), to measure the adhesive interface length and calculate the percentage of the area infiltrated by silver nitrate. The nanoleakage patterns were analyzed descriptively in terms of percentage of silver nitrate uptake.

Table V. Means (%) of the nanoleakage analysis on dentin.

Adhesive system	Distance		
	0 mm	3 mm	6 mm
Clearfil P	34.9	27.8	26.4
Clearfil T	42.9	26.4	30.8
One Up	75.9	55.0	64.9

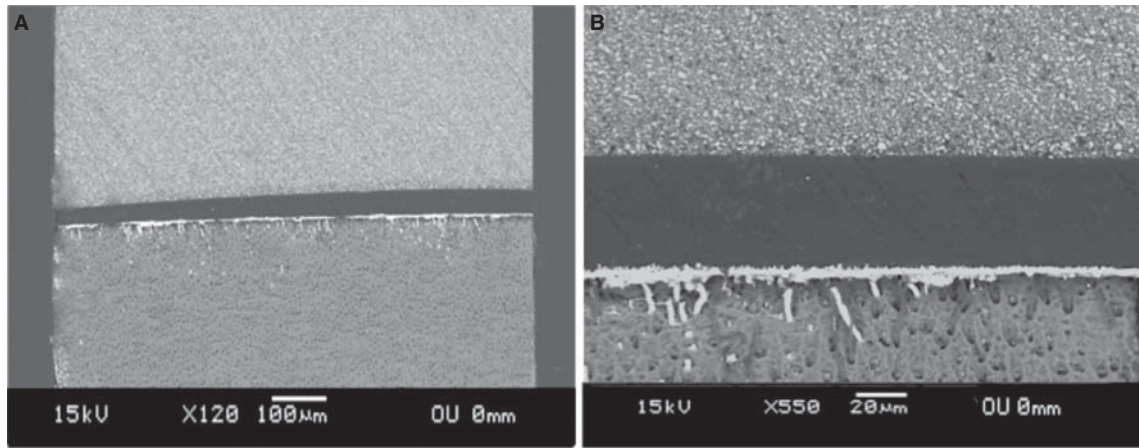


Figure 1. Interface of restoration using One Up Bond entirely infiltrated by the silver nitrate in dentin, presenting the worst sealing among the adhesive systems tested (A-X120) and (B-X 550).

Results

The results of the bond strength tests and nanoleakage evaluations are presented in Tables II–V. The bond strengths of the different adhesives to both substrates were similar. The adhesive system selection was statistically significant ($p = 0.0002$ for enamel and $p < 0.0001$ for dentin), while the irradiation distance ($p = 0.4994$ for enamel and $p = 0.2479$ for dentin) and the interaction of the factors ($p = 0.9169$ for enamel and $p = 0.1554$ for dentin) were not significant. The two-step self-etching adhesive exhibited greater bond strength than the two one-step adhesives, which were of similar strength when applied to either substrate.

All groups experienced leakage at the resin/dentin interfaces, with the greatest silver nitrate uptake occurring on dentin. The One Up Bond adhesive system was poorest in terms of leakage pattern (Figures 1 and 2), while the Clearfil Protect Bond and Clearfil Tri S adhesives exhibited similar leakage for all curing distances (Figures 3–6). In enamel samples, leakage was more frequently observed in samples cured at a distance of 6 mm

(Figures 2, 4 and 6), while in dentin samples greater silver nitrate uptake under the adhesive layer was observed in samples cured at a distance of 0 mm for all adhesives (Figures 1, 3 and 5).

Discussion

We evaluated the influence of curing distance and adhesive system on bond strength and leakage for both enamel and dentin substrates. The curing distance did not affect the bond strength to either enamel or dentin and the hypothesis that the distance from the light-guide does not compromise bond strength was accepted. Power density measurements of the light guide were performed using a radiometer. The power density was 650 mW/cm^2 at 0 mm, 400 mW/cm^2 at 3 mm and 350 mW/cm^2 at 6 mm. Although operation at a distance of 6 mm reduced the power density by ~ 53%, even 350 mW/cm^2 is greater than the minimum recommended power density for light-curing units employing a quartz-tungsten-halogen bulb [13]. Several researchers have reported that the degree of conversion of adhesives is dependent

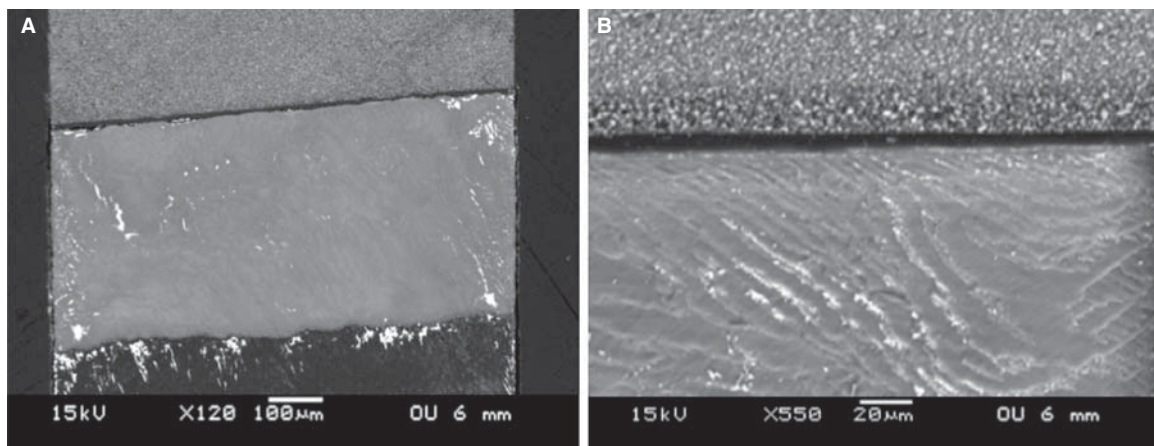


Figure 2. Interface of restoration using One Up Bond in enamel, presenting the lower sealing among the adhesive systems tested (A-X120) and (B-X 550).

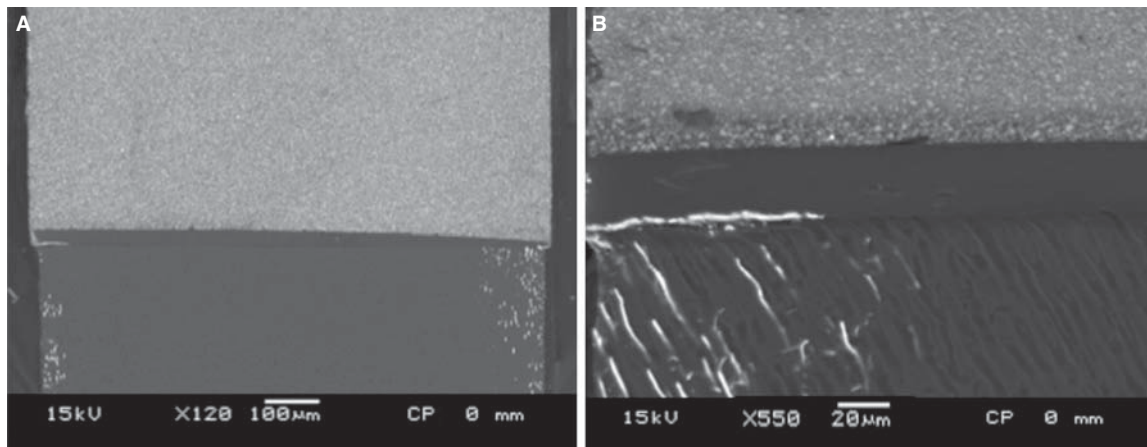


Figure 3. SEM figures of the silver nitrate uptake in dentin of interface of the restoration performed with Clearfil Protect Bond showed few points of silver nitrate infiltration (A-X120) and (B-X 550).

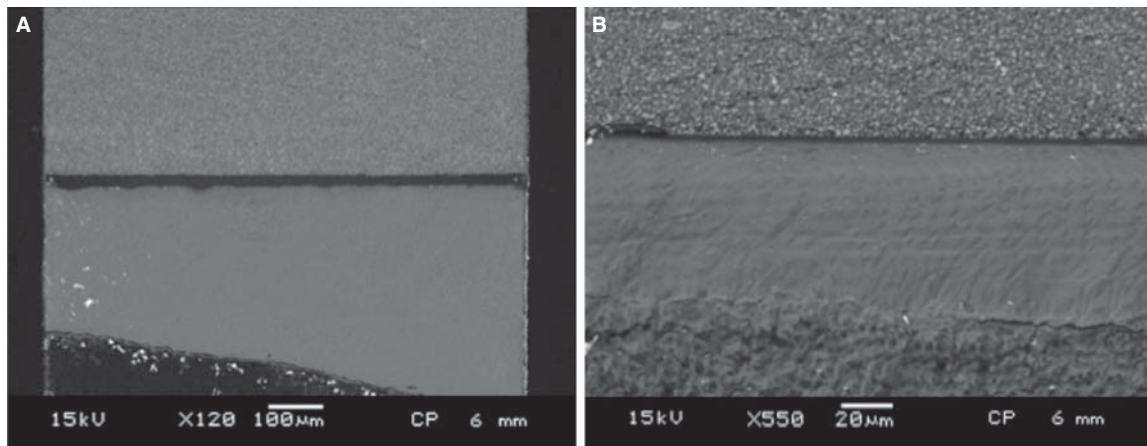


Figure 4. SEM figures of the silver nitrate uptake in enamel of interface of the restoration performed with Clearfil Protect Bond showing no points of silver nitrate infiltration (A-X120) and (B-X 550).

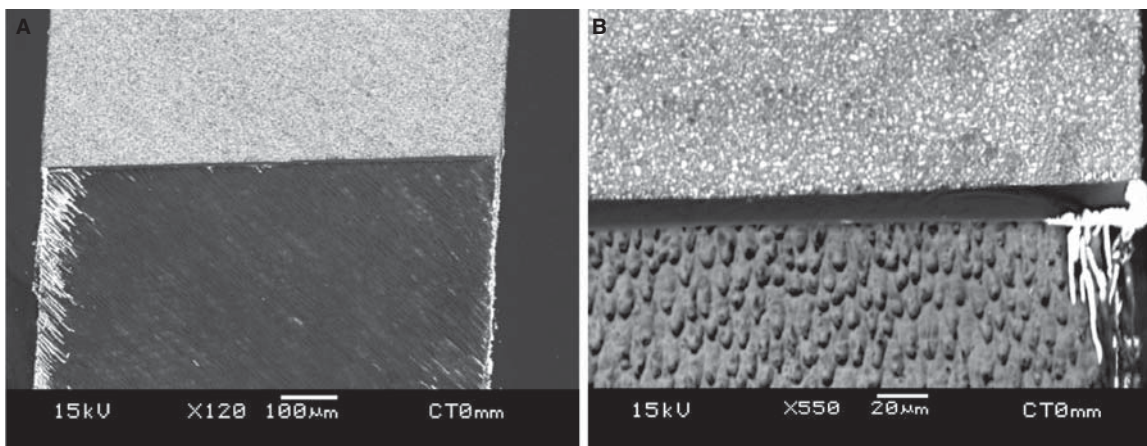


Figure 5. SEM photomicrography of the restoration using the Clearfil Tri S, demonstrating satisfactory sealing with some areas of infiltration in dentin similar to the Clearfil Protect Bond (A-X120) and (B-X 550).

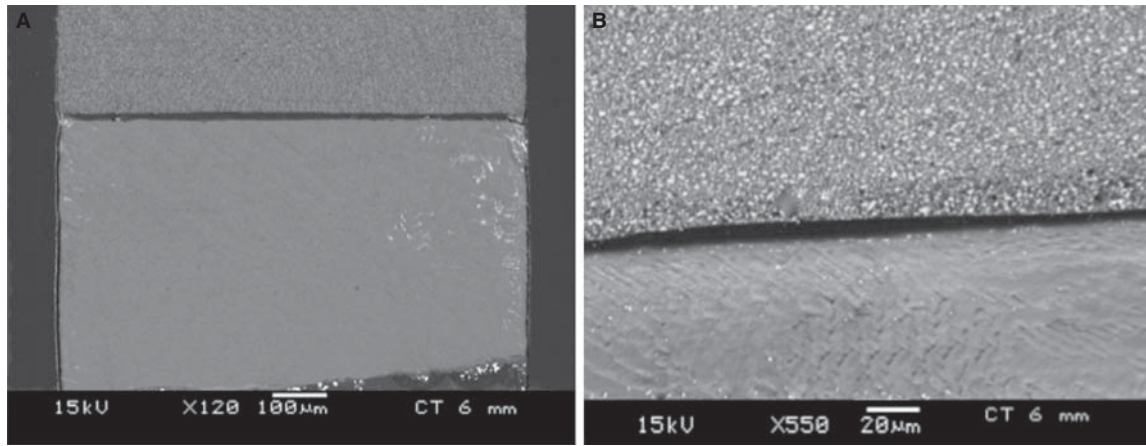


Figure 6. SEM photomicrography of bond interface of Clearfil Tri S, demonstrating satisfactory sealing in enamel, similar to the Clearfil Protect Bond (A-X120) and (B-X 550).

on the curing protocol and is affected by variables such as the light source (light emitting diode-LED or quartz tungsten halogen-QTH), emission spectrum and curing time [14–16]. However, most of these studies measured the degree of conversion in bulk specimens prepared from the adhesives. In clinical situations such as those simulated in the present study, the adhesive layer is only a few micrometers in thickness. A thin layer of adhesive would require less energy for complete curing and this could explain the lack of influence of curing distance on the bond strength of the adhesives.

Clearfil Protect Bond produced stronger bonds than Clearfil Tri-S Bond or One Up Bond F Plus, both of which were similar in strength. These results led to the rejection of the second hypothesis. Following primer application in two-step self-etching adhesive systems, both the enamel and dentin substrates are covered with a layer of low-permeability hydrophobic resin, resulting in more reliable bonding between the composite and dental substrate [17–19].

The hypothesis that the curing unit distance would not influence the quality of hybridization of self-etching adhesives was partially accepted, since the distance did not affect the leakage pattern on dentin substrates. However, on enamel substrates the quality of hybridization was better when the tip of the light source was nearer (3 mm) the substrate and a separation of 6 mm caused an increase in leakage. The negative influence of increased distance on hybridization was perceptible only on enamel substrates. Adhesion to enamel relies on monomer infiltration into the gaps formed during etching. The difficulty of achieving an adequate bond to enamel when using self-etching adhesives has been described in several studies [20,21] and is due to the poor etching performance of the acidic monomers. This effect, combined with inadequate polymerization of the adhesive system due to low light intensity, results in a situation in which the potential for leakage is at its greatest.

In contrast, self-etching adhesives are capable of achieving reliable bonding to dentin [22–24] and this characteristic was apparently sufficient to suppress the deleterious effect of extended curing distance on the quality of hybridization.

Samples prepared using the one-step Clearfil Tri-S Bond exhibited leakage patterns similar to the two-step Clearfil Protect Bond. These results may be related to the adhesive formula, which contains the hydrophobic resin bis-GMA. This monomer produces a unique polymer network with improved properties and interfacial integrity [25], resulting in better sealing.

General practitioners have adopted self-etching adhesives for routine restorative purposes due to their ease of use [26]. Unfortunately, the bond strength and leakage susceptibility of these adhesives is dependent on the adhesive composition and the intensity of irradiation during curing. Although the distances used in this study did not strongly influence the results, the probability of failure should be considered when light curing units of low irradiance are employed.

The results of this study clearly indicate that the use of two-step self-etching adhesives should be encouraged compared to the one-step self-etching adhesives, due to their better bond strength and hybridization to both enamel and dentin substrates. There was no significant dependence of bond strength on curing distance. In order to avoid a reduction in sealing effectiveness when using one-step self-etching adhesives to bond to enamel, an increase in polymerization time should be considered [27].

Declaration of interest: The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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