

ORIGINAL ARTICLE

Bonding of one-step and two-step self-etching primer adhesives to dentin with different tubule orientations

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Abstract

Objectives. To determine the microshear bond strengths of five ‘all-in-one’ adhesives and two 2-step self-etching primer adhesives to dentin with different tubule orientations and to compare bond strengths between the adhesives. **Material and methods.** Human adult molar teeth were sectioned to obtain dentin pieces with tubule orientations perpendicular or parallel/oblique to the bonded surface. Two 2-step self-etching primer adhesives (Clearfil SE Bond (CSE), Optibond Solo Plus SE Bond (Op. SE)) and five all-in-one adhesives (Clearfil S³ Bond (S³), Optibond All-in-One Bond (Op. AIO), G-Bond, Go!, and Xeno IV) were applied to dentin in accordance with the manufacturers’ instructions. Resin composite cylinders were bonded to superficial and deep dentin using microtubes (0.75 mm internal diameter and 1.5 mm high). Bonded specimens were stored in water at 37°C for 24 h, then stressed in shear at a cross-head speed of 1 mm/min until bond failure. Maximum load at failure (N) was recorded and converted to MPa. Statistical analyses were carried out using independent samples *t*-test and one-way ANOVA with Tukey’s HSD test. **Results.** Though bond strengths varied slightly, four all-in-one adhesives (Op. AIO, G-Bond, Go!, and Xeno IV) showed no significant differences in microshear bond strengths regardless of dentin depth (superficial or deep) or dentin tubule orientation (perpendicular or parallel/oblique). CSE, Op. SE, and S³ showed significantly lower bond strengths to deep dentin with a tubule orientation perpendicular to the surface. **Conclusion.** Most of the all-in-one adhesive systems used in this study appear to bond equally well to dentin independently of tubule orientation and depth. One two-step self-etching primer adhesive bonded significantly better than the all-in-one adhesives.

Key Words: *All-in-one adhesives, dentin depth, dentin tubules, dentin wetness, microshear bond strength*

Introduction

Bonding to dentin can be successfully achieved using resin-based adhesive systems that employ either the total-etch or self-etch approach. In the total-etch approach, dentin is etched with 30–40% phosphoric acid and washed prior to application of the resin adhesive. The smear layer is completely removed after etching; however, after washing there is the possibility of overdrying the etched dentin, which leads to collapse of the exposed collagen [1], resulting in incomplete resin infiltration into the demineralized areas and the formation of a porous zone beneath the hybrid layer [1]. Postoperative sensitivity has also been associated with the aggressive etching step of total-etch adhesives, which stimulated the development of self-etching primer adhesives. This approach involves dentin conditioning and priming by polymerizable carboxylic acid or

phosphate ester monomers [2,3], which simultaneously etch and infiltrate the dentin, theoretically ensuring a more complete resin infiltration [4]. Self-etching primers are more “user friendly” due to their fewer application steps, reduced technique sensitivity associated with the rinsing step of total-etch adhesives [5], and are generally associated with less clinical postoperative sensitivity [2,4]. Strong bonds to enamel and dentin have been reported [6–10]. For the older two-step self-etching adhesives, conditioning/priming and bonding are separate steps. In the newer “all-in-one” adhesive systems, the etchant, primer, and adhesive are combined in one application and therefore all three steps are effected simultaneously.

All-in-one adhesives are composed of hydrophilic and hydrophobic monomers, solubilized in water and/or organic solvents such as acetone or ethanol [2,3,11]. Such monomers can permeate the smear

layer and effect a mild demineralization of the underlying dentin [2,11]; they can also form covalent bonds with dentin collagen and ionic bonds with hydroxyapatite [2,3,11,12]. A high solvent content, in particular water, is present in all-in-one adhesives and this is necessary for adequate ionization of the acidic monomer [11,13]. Increasing the water concentration reduces monomer concentration and may reduce bond strengths [13], possibly by reducing the degree of monomer polymerization [14]. Several drawbacks have been associated with all-in-one adhesives, mainly due to their high water content and the hydrophilicity of their constituent monomers. These include concerns about their hydrolytic stability during storage, residual water after air drying [13], and phase separation [15], resulting in the formation of water droplets in the adhesive layer after curing [7,15,16]. A low degree of monomer polymerization on curing has been associated with self-etching primer adhesives [14,17,18], which may predispose to low bond strengths [13,17] and be responsible for initiation of bond degradation over the long term, thus contributing to the uncertainty surrounding their clinical efficacy.

Previous studies have reported bond strengths of several all-in-one adhesives comparable to those of 2-step self-etching primer adhesives to both enamel and dentin [6,7,19]. However, lower bond strengths for all-in-one adhesives have been reported [19].

Dentin variables such as tooth age, dentin region [9,20,21], dentin depth [22], dentin wetness, tubule density, and orientation [8,20,23] affect resin-dentin bonding. Owing to its varying structure, dentin bonding in the clinical situation will be to dentin with differing tubule orientations. Some studies have reported the microtensile and shear bond strengths of some 2-step self-etching primer adhesives to be unaffected by dentin tubule orientation [8,21,24]. Little is known about all-in-one adhesives in this regard. The bonding effectiveness of a restoration to dentin contributes significantly to its clinical success, and thus it may be important to determine the possible effect of tubule orientation and dentin depth on the bonding of recent all-in-one adhesive systems.

The aims of this study were to measure and compare the microshear bond strengths of two 2-step self-etching primer adhesives and five all-in-one adhesive systems to dentin with different tubule orientations and depths. The null hypothesis was that the 2-step self-etching primer and the all-in-one systems bond equally well to dentin with different tubule orientations and depths.

Material and methods

Forty-two non-carious adult molar teeth collected from the Royal Dental Hospital, Melbourne were used in the study after appropriate ethics approval for teeth collection had been obtained from the

University of Melbourne Human Research Ethics Committee. The teeth were stored in 1% chloramine T solution (pH = 9.1) for 2 weeks, and transferred into phosphate buffered saline solution (sodium hydrogen orthophosphate anhydrate (Na_2HPO_4), 5.92 g; potassium dihydrogen orthophosphate (KH_2HPO_4), 1.72 g; sodium chloride (NaCl), 28.8 g; dissolved in 4 liters of distilled water, (pH 7.4)) at 4°C. The teeth were used within 3 months of extraction.

The teeth were randomly divided into seven groups of six teeth. Each group was to be bonded with one of seven dentin adhesive systems: two 2-step self-etching primer adhesives and five all-in-one adhesives (Table I). In each group, the teeth were further divided into two subgroups of three teeth and each subgroup prepared for bonding in one of two ways: in the first subgroup, the teeth were sectioned approximately 1.5 mm from and parallel to the occlusal surface of the tooth using a water-cooled diamond saw (Struers, 230-CA, USA). The exposed occlusal surface was ground on 240-grit silicon carbide (SiC) paper under copious running water on a polishing machine (Tegrapol-25, Struers, Ballerup, Denmark) until all traces of the occlusal enamel and fissures were removed and only superficial dentin was present. A second cut on the crown was made approximately 2 mm parallel to the first and a third cut made at the cemento-enamel junction. Two dentin disks per tooth, the occlusal surfaces of which corresponded to superficial and deep dentin, were therefore obtained. In the second subgroup, the crowns of the teeth were first sectioned at the cemento-enamel junction; then each crown was sectioned vertically and approximately 2 mm from the most convex aspect of the buccal surface in a mesiodistal orientation. A second cut was placed 1.5 mm parallel to the first and a third cut approximately 1.0 mm parallel to the second. In this way, four enamel-dentin slabs (one buccal, one lingual, and two central) were obtained per tooth. Figure 1 shows the details of the specimen preparations. The second central slab obtained (1 mm thick) was discarded, thus three slabs per tooth were used. The buccal and lingual enamel surfaces of the buccal and lingual slabs were ground flat (approx. 200–400 μm tissue being removed). The dentin-enamel surfaces of the dentin disks and slabs were ground with 600-grit SiC paper in order to obtain uniform smear layers on the dentin, and the specimens mounted in dental stone in plastics molds. The embedded dentin specimens were placed in a tray containing water in an incubator at 37°C and covered using wet paper towels to maintain hydrated dentin until just before bonding. Bonding was carried out immediately after specimen preparation and in accordance with the manufacturers' instructions. The dentin was bonded in a moist state for all specimens.

The methods of application of the dentin adhesives are explained in Table II. After application of

Table I. Materials.

Material	Code	Type	Contents	pH	Manufacturer	Batch no.
Clearfil SE Bond	CSE	2-SEA	Primer: 10-MDP, HEMA, hydrophilic dimethacrylate, di-camphoroquinone, N,N-diethanol-p-toluidine, water.	2	Kuraray Medical, Okayama, Japan	00629A
			Bond: 10-MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, di-camphoroquinone, N,N-diethanol-p-toluidine, silanated colloidal silica			00891A
Clearfil S ³ Bond	S ³	AIO	10-MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, di-camphoroquinone, ethyl alcohol, water, silanated colloidal silica	2.7	Kuraray Medical, Okayama, Japan	00043A
Optibond Solo Plus SE Bond	Op. SE	2-SEA	Self-etch Primer: Hexafluoroglutaric anhydride-glycerodimethacrylate adduct, GPDM, ethyl alcohol, water	1.5	Kerr Dental, Orange, CA, USA	444794
			Optibond Solo Plus: Bis-GMA, GDMA, GPDM, HEMA, ethyl alcohol, fillers	2.2		449067
Optibond All-in-One Bond	Op. AIO	AIO	GPDM, mono and di-functional methacrylate monomers, nanofillers including sodium hexafluorosilicate, water, acetone and ethyl alcohol, photoinitiators	2.5	Kerr Dental, Orange, CA, USA	450585
G-Bond	–	AIO	4-Methacryloxyethyltrimellitate anhydride, UDMA, dimethacrylate component, phosphoric ester monomer, water, photoinitiators	2	GC Corp, Tokyo, Japan	0511081
Go!	–	AIO	Phosphoric acid ester monomer, dimethacrylate monomer, monomethacrylate monomer, silicon dioxide filler, water, acetone, sodium fluoride	2	SDI Ltd, Bayswater, Victoria, Australia	070682
Xeno IV	–	AIO	Mono-, di- and trimethacrylate resins, dipentaerythritol penta acrylate monophosphate, cetylamine hydrofluoride, acetone, water	2.1	Dentsply-Caulk, Milford, DE, USA	070323
Palfique Estelite	–	–	Methacrylates, silica-zirconia fillers		Tokuyama Dental, Tokyo, Japan	EW809394

10-MDP = 10-methacryloyloxydecyl dihydrogen phosphate, HEMA = 2-hydroxyethyl methacrylate, Bis-GMA = Bisphenol A diglycidylmethacrylate, GPDM = Glycerol phosphate dimethacrylate, UDMA = Urethane dimethacrylate, Glycerol dimethacrylate. 2-SEA = 2-step self-etching and primer adhesive. AIO = All-in-one adhesive. The pH values stated were obtained from MSDS for Clearfil SE Bond, Clearfil S³ Bond, private correspondence with company representatives for Go! Optibond All-in-One Bond and for the remaining adhesives, from the paper by Van Landuyt et al. [3].

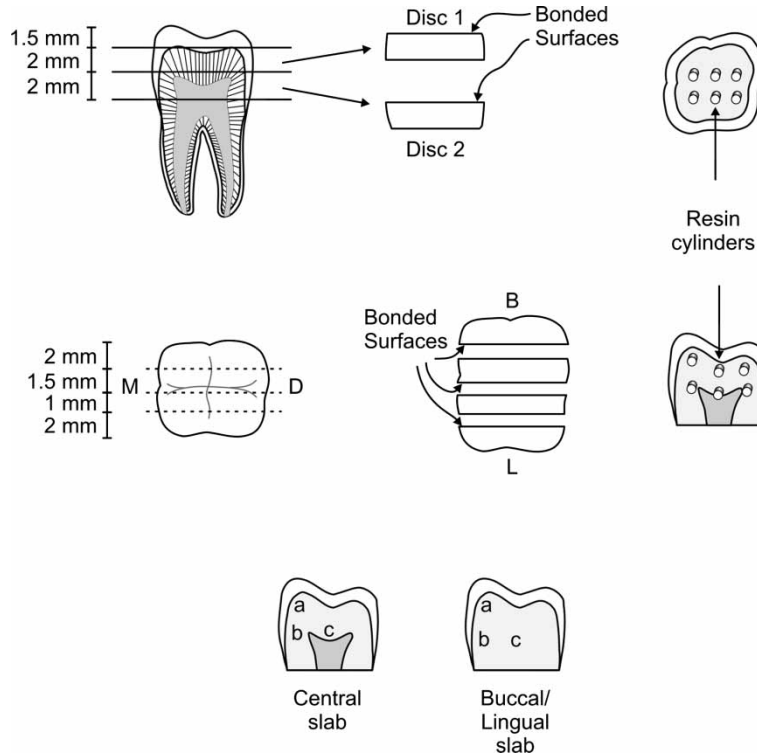


Figure 1. Schematic diagram of specimen preparation. Region “a” indicates superficial dentin; regions “b” and “c” indicate deep dentin, tubules parallel/oblique to the surface; region “c” on the buccal/lingual slabs exhibited tubules perpendicular to the surface on FE-SEM.

each adhesive, before light-curing was carried out, translucent polyvinylchloride microtubes (0.75 mm internal diameter and 1.5 mm high) were placed on the adhesive-covered dentin surface and the adhesive light-cured using a light-emitting diode light unit with a 800 mW/cm² intensity (BluePhase C8; Ivoclar Vivadent AG., Schaan, Liechtenstein). The intensity of the emitted light was previously checked using the appliance’s in-built light meter. A hybrid resin composite (Palfique Estelite) (Table I) was used to fill the tubes and light-cured for 30 s. Six microtubes (three to superficial and three to deep dentin) were

bonded to each dentin slab and six to eight microtubes to each dentin disk (Figure 1).

After bonding, the specimens were placed in distilled water in an incubator at 37°C. Twenty-four hours after bonding, the bonds were checked under a light microscope at ×10 magnification and defective bonds were excluded from the study; then microshear bond strength testing was carried out on a universal testing machine (Imperial 1000; Mecmesin, West Sussex, UK) using the corresponding computer software (Emperor v. 01). Additional specimens were prepared to make up the group

Table II. Methods of dentin adhesive application.

Adhesive	Priming	Bonding
Clearfil SE Bond	Clearfil SE Primer applied, left in place for 20 s, dried with a mild air blast for 5 s.	Clearfil SE Bond applied, air-thinned gently for 3 s, light-cured for 10 s.
Clearfil S ³ Bond	Clearfil S ³ Bond applied, left in place for 20 s, dried using a strong air blast for 5 s while spreading the adhesive, light-cured for 10 s.	
Optibond Solo Plus SE Bond	Optibond Self Etch Primer applied with brushing motion for 15 s, air-thinned for 3 s.	After thoroughly shaking the bottle, Optibond Solo Plus applied with a brushing motion for 15 s, air-thinned for 3 s, reapplied for 15 s, air-thinned for 3 s, light-cured for 20 s.
Optibond All-in-One Bond	The bottle was shaken well, Optibond All-in-One Bond applied using an applicator brush with a brushing motion for 20 s and repeated. Adhesive air-thinned for 5 s, light-cured for 10 s.	
G-Bond	The bottle was shaken well. G-Bond applied using the micro-tip applicator, left in place for 10 s, dried for 5 s with a strong air blast, light-cured for 10 s.	
Go!	Go! was applied and left in place for 20 s, dried with a strong air blast for 5 s, light-cured for 10 s.	
Xeno IV	Xeno IV applied with a scrubbing action for 15 s, reapplied in the same manner for 15 s, gently air-thinned for 5 s, light-cured for 10 s.	

sizes. The embedded specimens were mounted in a jig with the dentin surface flush with the external surface of the jig and a wire loop (0.35 mm diameter) wound round the cylinder at the composite-dentin interface at one end and the other end to a load cell connected to the computer. The bonds were stressed in shear at a cross-head speed of 1 mm/min until failure occurred, the maximum load at failure recorded, converted to MPa stress by dividing the failure load by the surface area of the bonded surface. Bond failure modes were also assessed using a light microscope at $\times 100$ magnification and classified as: A = adhesive bond failure – involving more than 50% of the bonded surface; C = cohesive failure in resin composite – involving more than 50% of the bonded surface; M = mixed failure involving up to 50% each of adhesive and cohesive resin composite failures.

Bond failure modes observed under the light microscope were confirmed by field emission scanning electron microscopy (FE-SEM). Representative specimens for the bond failure modes recorded were selected, removed from the plastics molds, and ultrasonicated for 30 s to remove debris. Six teeth were also prepared, three for each of the sectioning methods used in the study, treated with 35% phosphoric acid solution for 15 s to remove the smear layer and to expose underlying dentin for confirmation of dentin tubule orientation. The prepared specimens were mounted on aluminum stubs and left to dry at room temperature for 5 days, gold-coated, and examined under the FE-SEM at 2.0 kV and 140, 500, and 1000 magnifications.

The independence of values within dentin disks and slabs at different dentin depths and regions (buccal, lingual, and central) was checked by calculating the intraclass correlations. These were found to be negligible. The variations of MPa values within slabs/disks from different teeth were large when compared to the variations in the mean MPa values for the slabs/disks for the same tooth; therefore independent samples *t*-tests were used to compare results obtained for each adhesive at the dentinal tubule orientations and dentin depths at an alpha level of 0.01 (incorporating Bonferroni's correction as 4 *t*-tests were carried out per adhesive). One-way ANOVA and post hoc Tukey's HSD test were used to compare microshear bond strengths between adhesives for the same dentin tubule orientations and dentin depths after normality of the data distribution and equality of the group variances were checked and confirmed. Statistical analyses were carried out using the SPSS 15 software for Windows (SPSS Inc., Chicago, Ill., USA) at a significance level preset at 5%.

Results

The microshear bond strength values recorded for the adhesives and results of statistical analyses are given in Table III. Independent samples *t*-test revealed that, though bond strengths varied slightly, four all-in-one adhesive systems (Op. AIO, G-Bond, Go!, and Xeno IV) showed no significant differences in microshear bond strengths regardless of dentin depth (superficial or deep) or dentin tubule orientation (perpendicular or parallel/oblique). Of the all-in-one adhesives, only S³ showed significant variations in bond strengths.

For S³ and the 2-step self-etching primer adhesives, CSE and Op. SE, microshear bond strength was statistically lower in deep dentin compared with superficial dentin when tubule orientation was perpendicular to the surface ($p < 0.01$). When tubule orientation was parallel to the surface, dentin depth did not significantly affect bond strengths of S³, CSE, and Op. SE. While comparisons of bond strengths to superficial dentin revealed no significant differences, there were significant differences in bond strengths to deep dentin ($p < 0.01$) between the two dentin tubule orientations for S³, CSE, and Op. SE.

One-way ANOVA revealed that one of the 2-step self-etching primer adhesives used, CSE, exhibited significantly higher bond strengths compared to the five all-in-one adhesives, as well as to the other 2-step adhesive used (Op. SE), except in deep dentin when tubule orientation was perpendicular to the surface ($p < 0.05$). The all-in-one adhesives exhibited comparable bond strengths to Op. SE.

Bond failure mode results revealed over 70% of mixed failures followed by adhesive failures (Table IV). There were few cohesive failures recorded. A greater number of adhesive failures in deep dentin with perpendicular tubule orientation was recorded for CSE and S³. A higher incidence of adhesive failures was also recorded for Go! in superficial and deep dentin with tubule orientation parallel to the surface.

Figure 2A–F and 3A–C show representative FE-SEM photographs taken revealing that dentin tubule orientation in the buccal and lingual slabs was mainly parallel (Figure 2A), but sometimes oblique (Figure 2B,C), in the superficial dentin and mesial/distal deep dentin regions (marked a and b in Figure 1) for the dentin slabs. However, dentin tubule orientation appeared to be almost perpendicular (Figure 2D) in the deep-central regions for the buccal and lingual slabs (marked c in Figure 1). In the central slab, the dentin tubule orientation was parallel and/or oblique (Figure 2A–C) in both superficial and deep dentin. For the superficial and deep dentin disks, open dentin tubules perpendicular to the surface (Figure 2E,F) were observed with greater tubular density and less intertubular dentin in deep

Table III. Microshear bond strength results.

	CSE (n = 19)	S ³ (n = 18)	Op.SE (n = 18)	Op.AIO (n = 19)	G-Bond (n = 18)	Go! (n = 19)	Xeno IV (n = 19)
Mean (SD), MPa							
SPP	22.5 (4.04) ^a	16.4 (3.75) ^{b, c}	18.8 (4.04) ^b	17.2 (2.97) ^{b, c}	14.1 (2.82) ^c	14.5 (2.63) ^{c, d}	17.9 (4.60) ^{b, d}
DPP	15.2 (6.35) ^a	10.2 (2.82) ^b	14.2 (5.34) ^{a, b}	16.1 (3.66) ^a	12.9 (2.81) ^{a, b}	12.8 (3.11) ^{a, b}	16.7 (5.92) ^a
SP	23.3 (5.96) ^a	14.4 (3.61) ^{b, c}	15.6 (3.91) ^{b, c}	16.7 (4.67) ^{b, c}	12.7 (4.25) ^b	14.0 (4.25) ^b	18.6 (4.82) ^c
DP	20.4 (4.60) ^a	14.8 (2.51) ^{b, d}	19.1 (4.60) ^c	18.1 (4.46) ^{b, c}	11.2 (2.92) ^d	13.8 (2.15) ^d	18.0 (5.85) ^{b, c}
P values recorded (2-tailed sig.)							
SPP × SP	0.619	0.116	0.021	0.734	0.248	0.666	0.648
DPP × DP	0.006	<0.001	0.006	0.135	0.085	0.286	0.503
SPP × DPP	<0.001	<0.001	0.006	0.326	0.222	0.092	0.486
SP × DP	0.104	0.717	0.020	0.358	0.243	0.872	0.723

a) SPP = Superficial Dentin-Perpendicular tubule orientation; DPP = Deep Dentin-Perpendicular tubule orientation; SP = Superficial Dentin-Parallel tubule orientation; DP = Deep Dentin-Parallel tubule orientation. b) CSE = Clearfil SE Bond; S³ = Clearfil S³ Bond; Op.SE = Optibond Solo Plus SE Bond; Op.AIO = Optibond All in One Bond. c) Within the same columns, statistical analysis was carried out using independent samples t-test at $\alpha = 0.01$ (incorporating Bonferroni's correction). P-values of the results are as listed. Within the same rows, adhesives with the same lower case letters are not significantly different and those with different upper case letters are significantly different (one-way ANOVA and Tukey's HSD test at $\alpha = 0.05$).

dentin (Figure 2F). The increase in tubular density was estimated to have been up to five times in deep compared to the superficial dentin disks. FE-SEM also revealed wider diameter obliquely cut and more open tubules (Figure 2C) in deep dentin close to the pulp (area marked c in Figure 1) in the central dentin slabs than in the buccal and lingual slabs. For the buccal and lingual slabs, the variations in tubular density were not quite distinct and some tubules were not open to the surface. Figures 3A–C are shows scanning electron micrographs of representative bond failure modes recorded in the study.

Discussion

Previous studies [6,10,25,26] have reported that 2-step self-etching primer adhesives exhibit comparable bond strengths to enamel and dentin as some total-etch adhesives. Some all-in-one adhesives have been reported [6,7,19] to bond as well as their 2-step counterparts to enamel and dentin; however, lower bond strengths have also been reported [19].

The higher bond strength of CSE may be related to the ability of the functional monomer 10-MDP (Table I) to bond to residual hydroxyapatite [12] in addition to the micromechanical interlocking. The relative concentration of this monomer in the adhesive, pH, solvent type, and content of the adhesive, however, will determine contributions of the monomer to the ultimate adhesive bond strength and may explain the relatively lower bond strengths of S³. Bonding of resins to dentin is affected by dentin wetness [1] in addition to extent of resin polymerization [17]. The polymerization and resultant mechanical strength of a dentin adhesive resin are affected by the degree of ionization of its functional monomers [13], the degree of polymerization [14], and cross-linking density of its functional and cross-linking monomers [15]. The extent of polymerization for simplified systems has been reported to be low

[14,17,18] due to their lower monomer content. Incomplete adhesive solvent removal following air-drying for all-in-one adhesives [7,16] may impair polymerization [17] and result in reduced bond strength [27]. These factors could explain the lower bond strengths recorded for the all-in-one adhesives used in the study, compared with the 2-step self-etching primer adhesives. The significantly higher bond strengths exhibited by CSE to the dentin compared to the other adhesives may be indicative of the structural strength of the cross-linked monomer as well as its ability to bond to hydroxyapatite.

The 2-step self-etching primer adhesives CSE and Op. SE and one all-in-one adhesive S³ exhibited significantly lower bond strengths to deep dentin with the tubule orientation perpendicular to the surface. Dentin wetness increases towards the pulp because of increasing tubular density and luminal diameter [1,28]. Therefore, the hydrolytic solubility of the functional and cross-linking adhesive monomers may become more important in dentin bonding at this depth due to the greater water content. Following on from this, bonding in deep dentin may be affected by the reduced fibril network area and hence contribute to the reduced bond strengths in deep dentin with the tubule orientation perpendicular to the surface.

The characteristics of the monomer in the adhesives could also have influenced resin bonding. 10-MDP, the functional monomer in CSE and S³, is to some extent hydrophobic [3]. This may explain the lower bond strengths of CSE and S³ in deep dentin with tubule orientation perpendicular to the surface than to dentin with tubule orientation parallel/oblique to the surface due to increased dentin wetness. A recent study [29] reported significantly lower microtensile bond strengths of S³ and G-Bond under simulated pulpal pressure. The authors reported a 70% and 32% reduction in bond strengths for the two adhesives when bonded under pulpal

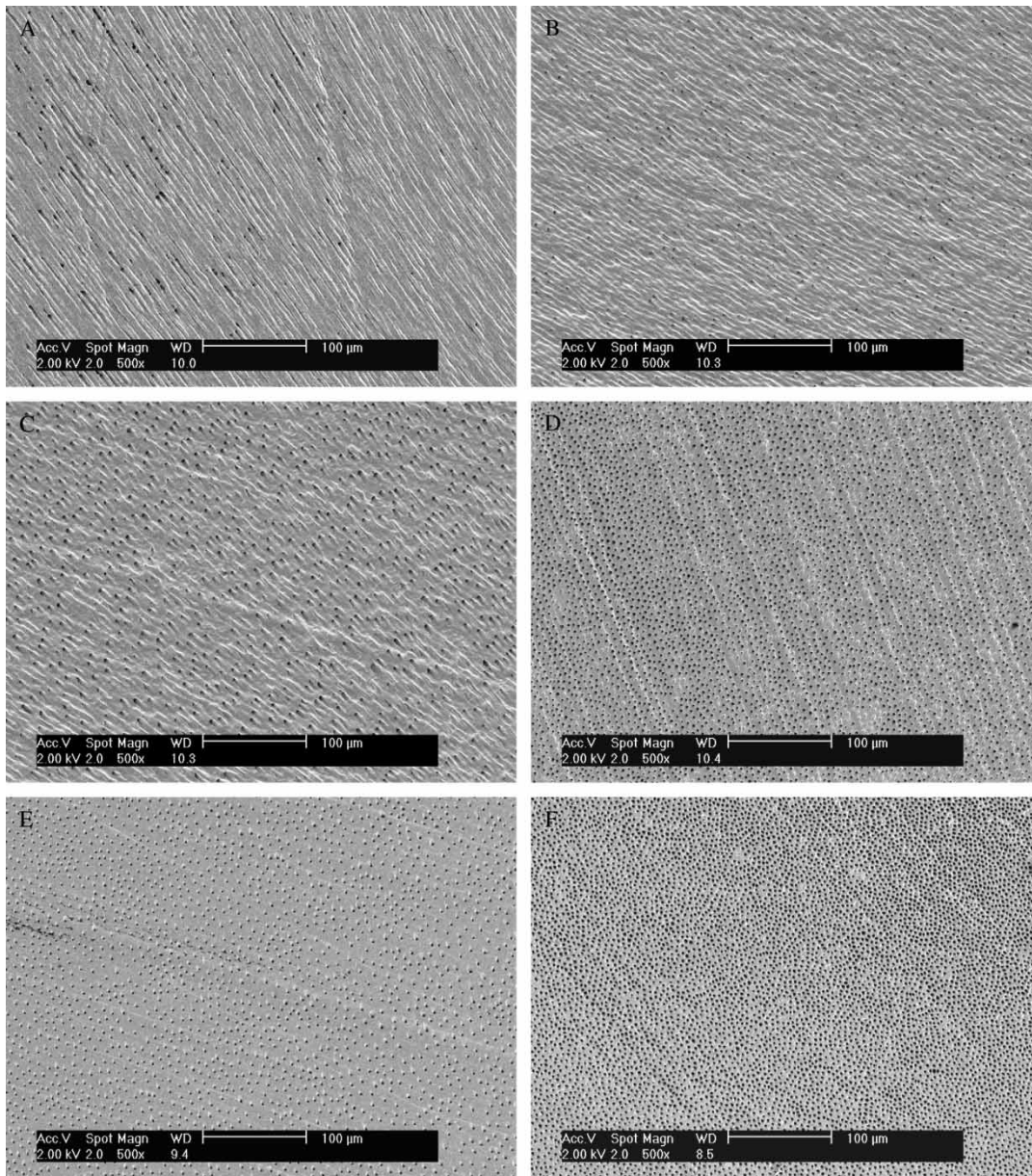


Figure 2. A. FE-SEM showing tubule orientation parallel to the surface observed in superficial coronal dentin and in mesial and distal aspects of deep dentin. This tubule orientation was more commonly observed in the buccal and lingual slabs. B. FE-SEM showing tubule orientation oblique to the surface, frequently observed in superficial coronal dentin and in the mesial and distal aspects of deep dentin. C. FE-SEM showing tubule orientation oblique to the surface in superficial mid-coronal dentin and in the mesial and distal aspects of deep dentin. This tubule orientation was more frequently observed in the central dentin slab than in the buccal and lingual slabs and shows wider diameter tubular openings than those observed in A and B. D. FE-SEM showing tubule orientation in deep central coronal dentin just above the cemento-enamel junction. The tubule orifices are densely distributed and almost perpendicular to the surface, with varying diameters. E. FE-SEM showing dentin tubule orientation observed in superficial dentin with narrow openings perpendicular to the surface and prominent intertubular dentin. F. FE-SEM showing deep dentin with densely distributed tubules perpendicular to the surface.

pressure compared with no pressure. The 2-step adhesive used in the study, Clearfil Protect Bond, exhibited a 54% lower microtensile bond strength. The study reiterates the effect of dentin wetness on resin bonding using these adhesives.

Bis-GMA, a cross-linking monomer used in Op. SE, is reported to have a high viscosity and low water solubility. Glycerol dimethacrylate (GDMA) resin, also in this adhesive, has a low viscosity and improved water solubility. The concentrations of individual monomers in the adhesive and their interactions will

determine the extent of infiltration, ionization, and cross-linking obtained on polymerization and subsequent mechanical properties of the adhesive. The scrubbing action recommended by the manufacturer probably assists adhesive infiltration into the dentin but may also increase technique sensitivity.

Interestingly, the HEMA-containing CSE, S³, and Op. SE were the only adhesives to exhibit differences in bond strengths with respect to varying dentin tubule orientations. HEMA serves as a wetting agent and transitional solvent (carrier) in dentin adhesives.

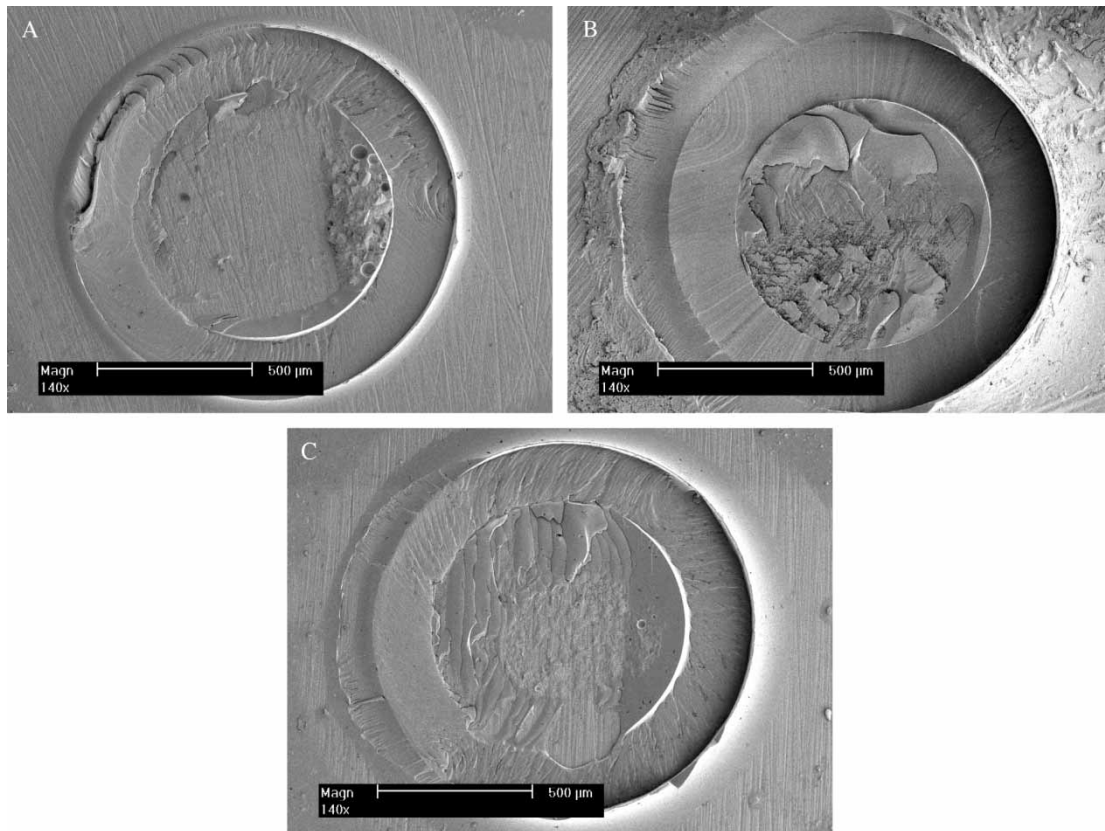


Figure 3. A. FE-SEM of adhesive failure, i.e. involving greater than 50% of the bonded surface. B. FE-SEM of cohesive failure in resin composite, i.e. involving greater than 50% of the bonded surface. C. FE-SEM of mixed failure, i.e. involving up to 50% each of adhesive and cohesive resin composite failures.

Owing to its high affinity for water, it imbibes and may retain the water within the cured adhesive layer, and can result in mechanical weakening of the adhesive layer and hence to lower bond strengths.

In contrast to the results obtained in this study, one study [24] found no difference in shear bond strength of CSE in deep dentin with tubule orientation perpendicular to the surface, while another [22] reported higher shear bond strengths in deep dentin for CSE. Toba et al. [9] reported a lower microshear bond strength of CSE to the floor of the pulp chamber than to mid-coronal dentin. Phrukkanon et al. [21] reported no difference in microtensile bond strength of an experimental 2-step self-etching primer adhesive to perpendicular and parallel dentin tubule orientations. Pereira et al. [20] also reported no regional differences in microtensile bond strength of a 2-step self-etching adhesive system; Clearfil Liner Bond II. The differences in the results may have been due to other factors that affect bonding, such as tooth age, regional variations in dentin, duration and solution of tooth storage, and test method [30].

FE-SEM findings revealed mainly parallel/oblique tubule orientations in the buccal, lingual, and central dentin slabs. Bonding in this plane is more to solid dentin structures of intertubular and exposed peritubular dentin and to a lesser extent via dentinal tubule lumens. The role of dentin wetness in

bonding in this plane may therefore have been minimized. This could explain the similar bond strength results observed in superficial and deep dentin in this plane for all adhesives tested. The FE-SEM finding on dentin tubule orientation in buccal/lingual deep central coronal dentin exhibiting perpendicular tubule orientation has been reported previously [1,31].

The similar bond strength results recorded for Xeno IV, Op. AIO, G-Bond, and Go! in this study may be related to the relatively high water content of all-in-one adhesives which facilitates dentin permeation. Increasing dentin wetness may be less likely to impair resin infiltration of the adhesives. In addition, these adhesives contain structurally relatively less hydrophobic monomers. The acidic (functional) monomers and cross-linking monomers are in one solution for all-in-one adhesives and may theoretically allow monomer infiltration to the same depth of demineralization, whatever the substrate depth may be. These factors may explain why bond strengths to dentin were similar to dentin with a tubule orientation perpendicular and parallel to the surface for the four all-in-one adhesives.

The higher occurrence of adhesive bond failures for CSE and S³ in deep compared with superficial dentin with tubule orientation perpendicular to the surface is consistent with bond strength results and

Table IV. Bond failure modes.

Tubule orientation	Perpendicular		Parallel/Oblique	
	Superficial A/C/M	Deep A/C/M	Superficial A/C/M	Deep A/C/M
Dentin depth				
CSE	4/1/14	12/0/7	3/0/16	4/1/14
S ³	3/1/14	10/0/8	7/1/10	5/2/11
Op. SE	2/1/15	5/0/13	1/0/17	1/0/17
Op. AIO	1/2/16	4/1/14	4/0/15	1/0/18
G-Bond	1/1/16	5/0/13	2/0/16	4/0/14
Go!	5/0/14	7/0/12	12/0/7	8/2/9
Xeno IV	5/1/13	4/0/15	2/1/16	7/0/12

CSE = Clearfil SE Bond; S³ = Clearfil S³ Bond; Op. SE = Optibond Solo Plus SE Bond; Op. AIO = Optibond All in One Bond. Bond failure mode: A = Adhesive bond failure-involving more than 50% of the bonded surface; C = Cohesive failure in resin composite-involving more than 50% of the bonded surface; M = Mixed failure involving up to 50% each of adhesive and cohesive resin composite failures (Figure 3a, b and c).

may be due to reduced resin infiltration, particularly for the more hydrophobic adhesive constituents. The higher occurrence of adhesive failures recorded for Go! may also be related to the extent of demineralization and micromechanical interlocking it effects to the dentin in this plane.

The results presented in this laboratory study should be interpreted with caution as clinical conditions of pulpal pressure, the presence of odontoblastic processes, and mineral deposits in dentinal tubules may limit dentin tubule permeability, reduce the effect of the penetration of acids and resins, and thus affect bonding effectiveness [1].

As the microshear bond strengths of the 2-step self-etching primer adhesives and one all-in-one adhesive varied with dentin tubule orientation perpendicular to the surface; and the bond strength of one 2-step self-etching primer adhesive was significantly higher than those of the all-in-one adhesives at the different dentin tubule orientations and superficial dentin, the null hypothesis is rejected. Under the limitations of this study, it can be concluded that the all-in-one adhesive systems appear to bond equally well to dentin with tubule orientation perpendicular or parallel/oblique to the surface. This may ensure a more uniform stress distribution on restorations placed in deep cavities using these adhesives. One of the 2-step self-etching primer adhesives bonded significantly better than the all-in-one adhesives.

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