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Bond strength of two total-etching bonding systems on caries-affected and sound primary teeth dentin

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Aim. As bond strength of currently available adhesive systems in caries-affected teeth dentin on primary tooth dentin was not well known, the bond strength of two bonding systems (PQI and OptiBond Solo Plus) was evaluated on caries-affected and sound primary molar tooth dentin and observed the micromorphology of the adhesive–dentin interfaces.

Methods. By grinding both the sound (n = 30) and caries-affected (n = 30) approximal surfaces of teeth, flat dentin surfaces were obtained. The prepared surfaces were bonded with one of the each adhesive systems and a composite resin. After storing the bonded specimens in water at 37 °C for 24 h, the samples were sectioned and the bond strength of the adhesive systems was tested by the microshear test method. The data were statistically analysed.

Results. Microshear bond strengths of PQI group for caries-affected and sound primary tooth dentin were 9.43 ± 2.44 (MPa) and 9.32 ± 2.95 (MPa) ($P > 0.05$), respectively, and the bond strengths of OptiBond Solo Plus group for caries-affected and sound primary tooth dentin were 15.33 ± 3.59 (MPa) and 11.96 ± 2.30 (MPa) ($P < 0.05$), respectively. Micromorphological features between sound and caries-affected dentin were similar in both PQI and OptiBond Solo Plus groups.

Conclusion. Both the adhesives showed significantly different bond strengths in caries-affected dentin but showed similar bond strengths in sound dentin.

Introduction

Clinical restoration of primary teeth involves primarily dentin affected by caries. After removing outer carious and infected dentin, the remaining affected inner dentin is treated[1]. The efficacy of bonding of resin adhesives to caries-affected dentin of permanent teeth has been studied more than on resin bonding to caries-affected primary teeth dentin[1,2].

Caries-affected dentin is not normal dentin, because the tubules are occluded with mineral crystals, but it is bacteria free[3]. It is partially demineralized and caries intertubular dentin exhibits a higher degree of porosity than sound intertubular dentin, due to the loss of mineral. Therefore, resin infiltrated hybrid layers in caries-affected dentin are thicker than those in sound dentin, suggesting easier diffusion of acidic conditioners and adhesive monomers[4–6]. Conversely, resin infiltration into dentinal tubules was severely hampered by the presence of acid-resistant mineral casts within dentin tubules of both caries-affected and caries-infected dentin[7]. These differences may have important implications for bonding characteristic between caries-affected and sound dentin.

In addition, to date only the bonding performance of adhesive systems using occlusal dentin on the pulp chamber as a substrate has been studied[8–10]. But as approximal surfaces of Class II cavities have the disadvantage of limited visibility in contrast to occlusal cavities; the proper placement of resin materials is difficult for clinicians[11].

In primary dentition with space closure and formation of contact areas, the incidence of proximal caries greatly increases. Proximal caries also progresses more rapidly than occlusal caries causing a higher percentage of pulp exposure in primary teeth[12]. The thinness of

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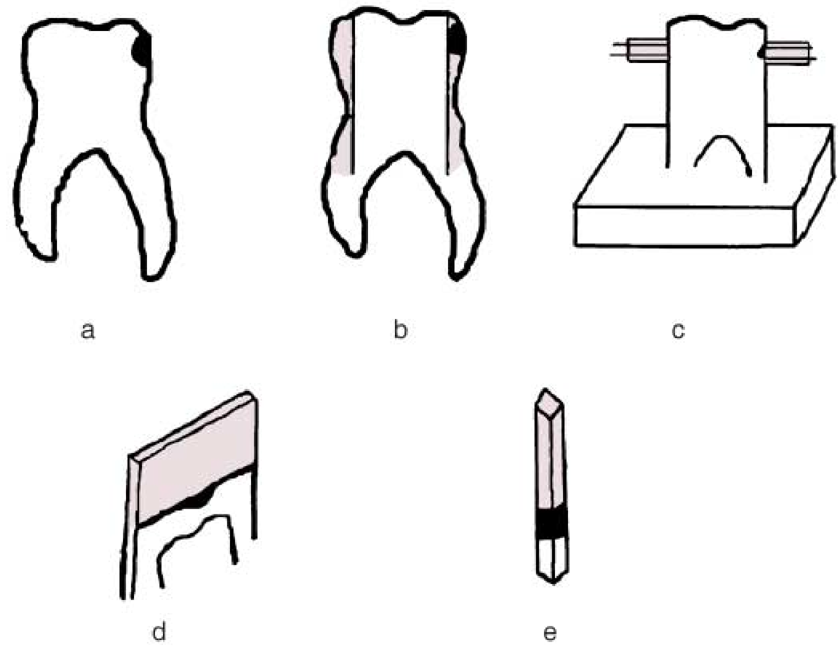

Fig. 1. Schematic illustrating the use of the microshear technique for measuring resin bond strength to normal and caries-affected dentin in the same tooth. The approximal surfaces (a) was ground flat after staining the carious lesion with caries-detector solution. After reaching caries-affected dentin (b), the entire surface was then bonded and a resin composite crown created that was sectioned to obtain slab (c, d) it was obtained stick which has approximately a cross-sectional area of 1 mm^2 (e).

enamel and its lower degree of mineralization may account for the more rapid progression of caries in primary teeth[13]. In addition, interproximal contact points are difficult to clean and as such, accumulate plaque, resulting in an increased risk for caries[14]. It can thus be speculated that caries-affected dentin could decrease in bond strength and the increase in microleakage of resin composite materials.

Conventional testing methods cannot be applied to clinical substrates such as caries-affected dentin because of the limited size and shape of these types of dentin. Recently, a microshear test was developed to measure bond strength of extremely small areas[15,16]. The purposes of this study were to examine the bond strength of two total-etching bonding systems on caries-affected and sound primary tooth dentin, and to observe the micromorphology of the adhesive–dentin interfaces.

Materials and methods

Specimen preparations

Thirty extracted human primary molars with approximal dentin caries stored in isotonic saline at 4 °C were used in this study. The teeth had only mesial or distal approximal caries. Both caries-affected and sound dentin surfaces were prepared on the axial wall of the same tooth.

The approximal surface with and without caries was prepared parallel to the long axis of the teeth to expose a flat surface by using a low-speed diamond saw under water-cooling (Isomet, Buehler Ltd, Lake Bluff, IL, USA) (Fig. 1a,b). To remove caries-infected dentin, grinding with 320-grit silicon carbide abrasive papers was used under running water using the combined criteria of visual examination, degree of hardness to a sharp excavator, and staining with a Caries-Detector dye (Kuraray Co., Ltd, Osaka, Japan) as previously described[6]. The surface was covered with caries detector solution and then rinsed with water. The surrounding yellow, hard dentin was classified as sound dentin while the discolored, harder dentin that stained pink as caries-affected dentin[8].

The flat dentin surfaces of teeth were then hand-polished with 600-grit silicon carbide abrasive paper under running water before bonding procedure. The teeth were randomly divided into two groups for two total-etching bonding systems (PQI, Ultradent, USA, and OptiBond Solo Plus, Kerr, USA). One approximal surface of each tooth was used for caries-affected dentin and the other proximal surface for sound dentin.

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Table 1. Manufacturers, components and application procedures of the dentin bonding systems used in the study.

Bonding systems	Components	Composite	Procedures
PQI Ultradent products, South Jordan, UT, USA, lot no. 84095	35% phosphoric acid, TEGDMA, Ethanol, HEMA, Florid	Amelogen Universal (Hybrid resin composite)	a (15 s) b (5 s), c e(15 s) f (20 s)
Optibond SoloPlus KERR Cor., Orange, CA, USA, lot no. 92867	37.5% phosphoric acid, Bis-GMA, HEMA, GPDM/fillers, Florid	Point 4 (Hybrid resin composite)	a (15 s) b d (3 s), e (15 s)d f (20 s)

Procedures: a, acid etching; b, air-water; c, remove surface water, do not desiccate; d, air-dry; e, apply adhesive; f, light-cure.
Abbreviations: TEG-DMA, triethyleneglycol dimethacylate; HEMA, 2-hydroxyethyl methacrylate; Bis-GMA, bisphenyl-glycidyl-methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; GDM, glycerol dimethacrylate; GPDM, glycerophosphoric acid dimethacylate.

Bonding procedure

The line of the caries-affected dentin area was marked by waterproof pen before application adhesive system. Following application of the adhesives to the prepared surfaces according to the manufacturers' instructions (Table 1), the respective composite of bonding agents were built up incrementally in three to four layers to a height of 4–5 mm (Fig. 1c). Bonding agents and composite resins were cured with a curing light (Hilux 250, Benlioðlu, Ankara, Turkey) according to the stipulated curing time, where light intensity was at least 400 mW/cm^2. Specimens were, then, stored in water at 37 °C for 24 h.

Microshear bond strength testing

The specimens were vertically sectioned both mesial-distally and buccal-lingually along their long axis into approximately 1 × 1-mm wide sections using a low-speed diamond saw. 'I' shaped longitudinal cuts, the top half consisting of composite and the bottom half consisting of dentin from each tooth were obtained (Fig. 1d–e). Each stick was carefully examined in a dissecting microscope (×20) to ensure that the test site was homogeneous with regard to caries-affected dentin (Olympus SZ 4045 TRPR, Tokyo, Japan). The cross-sectional area and remaining dentin thickness were measured using digital caliper (Mitutoyo, Tokyo, Japan).

For microshear bond testing, the sticks were mechanically fixed to the microshear bond testing apparatus and a test machine (Harvard Apparatus Co. Inc., Dover, MA, USA) (Fig. 2). Shear forces were applied to the resin–dentin interface at a crosshead speed of 1 mm/min until failure occurred. To determine the shear bond strengths, the maximum load (N) was recorded and converted to megapascals.

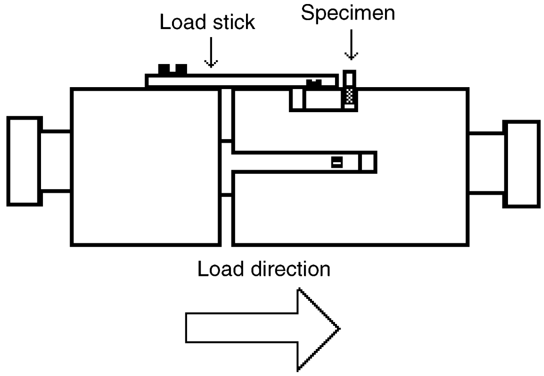

Fig. 2. Microshear test apparatus.

Fracture analysis

The fractured surfaces were examined using a stereomicroscope at ×20 magnification. The failure modes were categorized into one of three types:

1 adhesive failure between the composite resin and the bonded dentin surface;

2 cohesive failure in either dentin or composite; and

3 mix failure is which a combination of adhesive and cohesive failure[17].

Scanning electron microscopy evaluation

The second aim of this study was to observe the micromorphology of the interface between the adhesives and sound and caries-affected dentin. Six additional primary molar teeth with approximal dentin caries were used to

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evaluate the morphology of the interface by scanning electron microscopy (SEM). The preparation of the teeth was as the same as previously mentioned methods. The resin composites were bonded to the prepared dentin surfaces using bonding agents according to the manufacturers' instructions. The bonded specimens were longitudinally sectioned perpendicular to the bonded interface. The cut surfaces were fixed in 10% formaldehyde solution for 24 h, ground with 600 grit silicon carbide abrasive paper and highly polished with a diamond paste 6-3-1-1/4 μm (Struers, Copenhagen, Denmark). Then, the specimens were immersed in 10% phosphoric acid solution for 3–5 s, rinsed with distilled water, and treated for 5 min with 5% sodium hypochlorite solution and again rinsed thoroughly with distilled water. After drying at room temperature (27 °C), the specimens were coated with Polaron Sc500 Sputter Coater (VG Microtech Inc., Tokyo, Japan) and examined under SEM (JSM-5600, JEOL, Tokyo, Japan) at an accelerating voltage of 20 kV.

The thickness of the hybrid layer was interpreted as the mean distance between the top of the layer and the height of the scalloped convexities at the base of the hybrid layer between adjacent resin tags as comparing by bar (=10 μm).

Statistical analyses

The bond strength and remaining dentin thickness were statistically analysed using the two-way analysis of variance (ANOVA) and post-hoc Tukey HSD tests. The Pearson chi-squared test was used to compare the failure modes. A confidence level of 95% was set for all statistical evaluations.

Results

Bond strength to primary teeth is shown in Table 2. In respect of bond strength to sound dentin, the difference between the two bonding agents was not statistically significant ($P > 0.05$). The bond strength of OptiBond Solo Plus to caries-affected dentin was higher than PQI ($P < 0.05$).

The bond strength of OptiBond Solo Plus to caries-affected dentin was significantly higher than that to sound dentin ($P < 0.05$). However, PQI showed no significant difference between sound and caries-affected dentin ($P > 0.05$).

The remaining dentin thickness (RDT) in all groups was not significantly different ($P > 0.05$) (Table 2).

The modes of failure in the various groups were shown in Table 3. There were significant differences between the failure modes of all groups ($P < 0.05$).

SEM observation

Scanning electron micrographs of the resin–dentin interface showed that the hybrid layer thickness of the OptiBond Solo Plus in sound

Table 2. The mean microshear bond strength (MSBS) (MPa) and remaining dentin thickness (RDT) (mm) values (mean ± SD) to sound and caries-affected primary dentin teeth (*n* = 15).

Dentin type	Bonding systems	MSBS*	RDT†
Sound	PQI	9.43 ± 2.44[a]	1.37 ± 0.28
	OptiBond Solo Plus	11.96 ± 2.30[a]	1.18 ± 0.32
Caries-affected	PQI	9.32 ± 2.95[a]	1.27 ± 0.28
	OptiBond Solo Plus	15.33 ± 3.59[b]	1.20 ± 0.30

*There was no statistically difference between groups having the same letters ($P > 0.05$). †There was no statistically difference according to the two-way ANOVA ($P > 0.05$).

Table 3. Distribution and percentage of failure modes of groups.

Dentin type	Bonding systems	Adhesive (%)	Cohesive (%)	Mix (%)
Sound	PQI	9 (60)	6 (40)	0
	OptiBond Solo Plus	15 (100)	0	0
Caries-affected	PQI	13 (86.7)	1 (6.7)	1 (6.7)
	OptiBond Solo Plus	9 (60)	3 (20)	3 (20)

Pearson $\chi^2 = 16.748$, $P = 0.010$.

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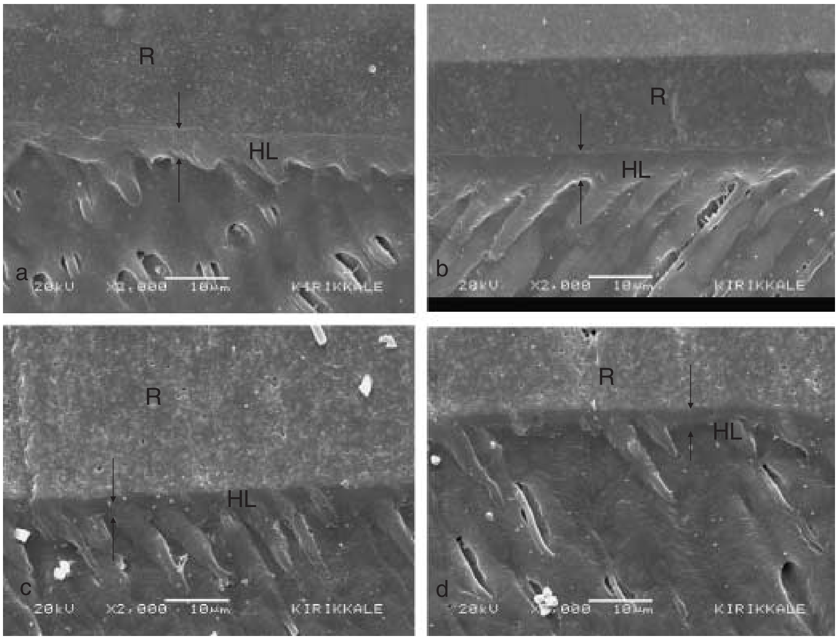

Fig. 3. Scanning electron micrographs of the resin/dentin interface of the polished cross-sectional dentin bonded with Optibond Solo Plus (top) and PQI (bottom). (a, c) caries-affected dentin; (b, d) sound dentin. R, resin; HL, hybrid layer.

and caries-affected dentin was approximately 4–5 μm (Fig. 3a,b). In the PQI group, the thickness hybrid layer was approximately 2–3 μm in both sound and caries-affected dentin (Fig. 3c,d). The resin tags were seen in the two bonding groups. No gaps were seen between composite resin and dentin surfaces for all test groups.

Discussion

It was found that the microshear bond strength of OptiBond Solo Plus to caries-affected dentin was significantly higher than that to sound dentin, however PQI showed no significant difference between sound and caries-affected dentin.

The recently developed microtensile test was used in many studies to measure bond strength[6,18–20]. Although an effective method in terms of test small areas, the microtensile test is difficult to conduct and time consuming for specimen preparation, especially primary tooth[21]. In this method, each stick is glued to the flat stainless steel ‘grip’ with a cyanoacrylate. In actual testing, the cyanoacrylate covers the entire surface of both ends of stick to increase the surface area of cyanoacrylate-stainless steel bond. When the sticks are short, it is very difficult to glue them to the stainless steel grip by cyanoacrylate. In this study, microshear bond tests was performed to measure the bond strength between adhesive material and primary tooth dentin as remaining dentin thickness of primary tooth especially in caries-affected primary tooth dentin was too thin to glue to the microtensile test apparatus. In addition, there is no necessity for preparations of the bonding surface of the specimens in the microshear test method which will alter its bonding surface[21,22]. Hosoya *et al.*[23] suggested that because of the lower physical properties of primary tooth dentin, especially for inner dentin, preparation might damage the bonded interface and this could change bond strength. Therefore, the advantage of the microshear test method is that it is not necessary to alter the bonding surface[21,22].

Microshear method uses smaller surface areas such as microtensile method, which shows generally adhesive failure at bonded interface[15]. In the present study, most of the specimens showed also adhesive failure. However, 20% cohesive and mix failure was observed in the OptiBond Solo Plus group. Caries-affected dentin is softer and more porous than normal dentin as it is partially demineralized. The weaker and softer caries-affected dentin may explain higher rate of cohesive and mixed failures in the OptiBond Solo Plus group.

For PQ1, fracture mode significantly changed to cohesive in resin from adhesive failure

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although there was no difference in bond strength between sound and caries-affected dentin. The reasons are not clear, but some reports suggest that there was no correlation between cohesive resin failure and bond strength[24–27]. Almuammar *et al.*[28] suggested that no direct relationship between the mode of fracture and bond strength values, which means that high bond strength values were not necessarily correlated with a cohesive type of fracture. It was suggested that fracture mode was affected the material properties of all components of the bonded joint, i.e. dentin, adhesive system and composite resin restorative material, as well as the mechanics of the test assembly[23,29]. It was observed similar results in our study as mentioned above.

The mean RDT values of specimens in the four groups were similar, indicating that all samples were bonded at approximately the same dentin level.

Although bonding performance of adhesive system using occlusal caries-affected dentin were evaluated in most studies[8,9], proximal caries-affected primary teeth dentin were used in this study. Because in primary dentition with space closure and formation of contact areas, the incidence of proximal caries greatly increases. Proximal caries also progresses more rapidly than occlusal caries causing a higher percentage of pulp exposure in primary teeth[12]. Dentin has a highly orientated microstructure with tubules arranged in a radial form running from the pulp chamber outward to the dentin–enamel junction in the crown[30–32]. In a proximal primary tooth cavity preparation with the absence of occlusal extension, one important factor on adhesion to dentin to be considered is the orientation of dentin tubules[32,33].

For primary teeth, a few studies have compared the bond strength between caries-affected dentin and sound primary teeth. Way *et al.*[34] compared the bond strength of resin modified glass ionomer cement of non-carious and carious primary dentin. No difference was found between the bond strength to the carious and non-carious primary teeth dentin[34]. The bond strength of total-etching system on the caries-affected primary teeth dentin was found significantly higher than that on sound primary teeth dentin[1].

Similar results were obtained by Sengun *et al.*[35], who found that bond strength of total-etching systems, One Coat Bond and Prime Bond 2.1, to caries-affected dentin were significantly higher than to sound permanent teeth dentin. However, there was difference on data on permanent teeth, in which the bond strength was significantly lower for caries-affected dentin than for sound dentin when a different total-etching adhesive system was used[36]. We found that the bond strength of OptiBond Solo Plus to caries-affected dentin was significantly higher than that of sound dentin, but PQI was showed no significant difference between sound and caries-affected dentin. Our data confirm those of Sengun *et al.*[35] suggesting that the strength of adhesion to dentin depends on both the adhesion system used and the type of dentin.

Caries-affected dentin contains crystals of calcium phosphate which is less soluble in acidic conditions than is sound apatite. Thus, stronger acids may be required to solubilize the mineral phase of caries-affected dentin to obtain sufficient resin infiltration for high bond strengths[3]. Both of the bonding materials used in this study have strong and similar concentration of phosphoric acid conditioner. However, the bond strength of OptiBond Solo Plus to caries-affected dentin was higher than the bond strength of PQI. This difference may be result of bonding technique used. In this study, the materials were used according to the manufacturers' instructions. The manufacturer of PQI recommended using the moist bonding technique, while that of OptiBond Solo Plus recommended 'air-dry' after etching and rinsing. In normal demineralized dentin, the spaces between the collagen fibrils are maintained by water during the moist bonding technique. If demineralized dentin matrix is air-dried, collagen fibrils are brought closer together resulting in a demineralized zone with reduced permeability to resin monomers[37–39]. However, it was suggested that whether this same sequence of events occurs in acid-etched, caries-affected dentin is unclear[39].

The SEM examination of the resin–dentin interface in our study showed no morphological difference in the thickness of the hybrid layer between sound and caries-affected

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dentin in both adhesive groups. Nakornchai *et al.*[1] have suggested that owing to the shorter period of demineralization and remineralization of dentin in primary teeth, the calcium crystal deposits in dentinal tubules of carious dentin in primary teeth may have been dissolved by phosphoric acid. Therefore, the resin tags found in caries-affected dentin were not different from those found in sound dentin.

In PQI adhesive system, there were no differences between the caries-affected dentin and sound dentin in SEM examinations. There were also no differences in bond strength of caries-affected dentin in the PQI group. No differences were seen in the SEM examination of resin–dentin interface between sound and caries-affected dentin in the Optibond Solo Plus group. Nevertheless, the bond strength to caries-affected dentin was higher than that to sound dentin. There was no correlation between hybrid layer thickness and bond strength[38–40] and further research is needed to improve resin bonding to primary teeth dentin.

Conclusion

Our study confirms that differences in bond strength of adhesive systems depends on both the adhesive system used and the type of dentin.

Since specimen size, especially in caries-affected primary teeth dentin, is limited by dentin thickness in primary teeth, the micro-shear test method where these can be used for adhesion to primary teeth dentin.

What this paper adds
- The chemical composition of bonding system effects on bond strength to caries-affected primary tooth dentin.

Why this paper is important to paediatric dentists
- It is difficult to evaluate bond strength of caries-affected primary tooth dentin because of limited dentin thickness.
- The microshear bond strength test can be used in vitro studies for primary teeth.

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