Efficacy of dentin adhesives in primary and permanent teeth

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The purpose of the present study was to investigate the efficacy of dentin adhesives and to examine acid resistance in primary and permanent teeth. The efficacy of dentin adhesives was evaluated by SEM observation and by measuring the wall-to-wall polymerization contraction gap and dentin hardness before and after conditioning. The detailed mechanism of dentin bonding was the same in both primary and permanent teeth.

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INTRODUCTION

A resin composite restoration combined with a dentin bonding system has been frequently used in clinical practice in general for both permanent and primary teeth. Differences in the efficacy of dentin adhesives in primary and permanent teeth have been discussed.[1,2] In some papers, the bonding efficacy of the dentin adhesive was superior in the permanent teeth compared with the primary teeth, and it was claimed to be comparable both kind of teeth though primary teeth were scarcely reported to be more desirable for the dentin bonding than the permanent teeth.[3,4] This difference in the effect of the dentin adhesives on the two substrates is thought to be based on differences in the proportion of organic and inorganic components and acid resistance. However, there has been no consistent conclusion about the optimum technique for resin composite restoration of primary teeth.[5-7]

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In most papers about dentin adhesives, efficacy is based on the shear or the tensile bond strength measurement to the two-dimensional flat tooth substances.[8-10] However, Asmussen claimed that the efficacy of the dentin adhesives should be examined in a three-dimensional dentin cavity, and that it was essential to evaluate the interaction between the efficacy of the dentin adhesives and the polymerization contraction stress of the resin composite.[11] To simplify the steps of resin composite restoration, self-etching dentin primers, a total-etch wet bonding system, and single bottled dentin adhesives have been introduced.[12] However, little has been reported about the efficacy of these simplified systems in the dentin cavity in permanent and primary teeth. The purpose of the present study was to investigate the efficacy of dentin adhesives and to examine the characteristics of acid resistance in primary and permanent teeth.

MATERIALS AND METHODS

The dentin bonding systems and the resin composites that we tested are listed in Table 1. The efficacy of dentin adhesive was evaluated by measuring the wall-to-wall polymerization contraction gap width between the resin composite and the cylindrical dentin cavity margin. Seventy extracted human primary molars and also seventy permanent molars were used. The proximal enamel of each tested tooth was flatly eliminated with silicon carbide paper grit number 220 under running water. A cylindrical cavity approximately 2 mm in diameter and 1 mm in depth was prepared in the exposed dentin. The dentin cavity wall was treated with a commercial dentin bonding system according to the instructions of the manufacturer. The cavity was slightly over-filled with the resin composite and the composite surface was gently pressed on a glass plate mediated with a plastic matrix. The resin composite was then irradiated with a halogen lamp unit for 40 seconds. After storing the specimens in tap water at 24˚C ± 1˚C for

Table 1. Dentin bonding systems tested.

Bonding System	Conditioner	Primer	Bonding Agent	Resin Composite
Experimental 1	EDTA	GM	Cleafil. P. B	Silux Plus
Experimental 2	EDTA	GM	Cleafil. P. B	Cleafil. P. S. C
Experimental 3	H_3PO_4	GM	Cleafil. P. B	Cleafil. P. S. C
Single Bond	H_3PO_4	Adhesive		Cleafil. P. S. C
Single Bond	H_3PO_4	Adhesive		Z-250
Liner Bond II ∑	LB Primer A/B		∑ Bond A/B	Cleafil. P. S. C
One-up Bond F	Bonding agent A/B			Cleafil. P. S. C

10 minutes, the over-filled composite was eliminated with the wet carborundum paper and the resin composite surface, including the surrounding dentin surface, was polished on a linen cloth mediated with an alumina slurry grain size of 0.03mm.

The marginal adaptation of the resin composite was inspected under a light microscope, and the possible contraction gap width was measured with a screw micrometer mounted on the ocular lens of a microscope. The gap width measurement was performed at the eight points every 45 degrees along the cavity margin and the gap value was presented by the sum of the diametrically opposing gap width in per cent to the cavity diameter. The contraction gap value of the specimen was given by the maximum in four gap values.

For the control, the dentin cavity wall was conditioned with 0.5mol/L ethylene diamine tetra acetic acid (EDTA) neutralized to pH 7.4 or 40% phosphoric acid gel for 60 seconds. It was then rinsed and dried. The cavity was then treated with an experimental dentin primer composed of 35vol% of glyceryl monomethacrylate (GM) solution for 60 seconds followed by air blasting.

A commercial dual-cured dentin bonding agent (Clearfil Photo Bond, Kuraray, Okayama, Japan) was applied to the cavity and irradiated for 10 seconds after the excess material was removed with a gentle stream of air. The resin composite filling and gap width measurements were done with the same method previously described. Ten specimens were prepared for each group.

After gap width measurement, the specimens were dehydrated in gradual alcohol solutions, critically point dried, and coated with palladium and platinum ions prior to SEM observation.

The effect of conditioning on dentin hardness was examined by measuring dentin hardness before and after conditioning. The proximal enamel of an extracted human primary or permanent molar was flatly eliminated with wet carborundum paper grit number 220. After measuring the hardness of the dentin adjacent to the enamel-dentin junction, the dentin surface was conditioned with one of three commercial dentin conditioners or self-etching dentin primers according to the instructions of the manufacturer. After rinsing and drying, the hardness of the conditioned dentin was measured. For the control, the dentin surface was conditioned with EDTA or 40% phosphoric acid gel for 60 seconds. The Vickers hardness test was conducted on both the conditioned and non-conditioned dentin surfaces with a micro hardness tester with an indention load of 50g for 20 seconds. The hardness of the specimen was determined to be the mean value of five measurements, and five specimens were prepared for each conditioning.

The results of the wall-to-wall polymerization contraction gap width measurement and the Vickers hardness test were analyzed statistically with the Kiuskal-Wallis one-way analysis of variance by ranks and the Student's t-test at a 5% level of significance.

RESULTS

The results of the wall-to-wall polymerization contraction gap width measurement are shown in Table 2. Complete marginal integrity was obtained in the experimental dentin bonding system with EDTA conditioning regardless of whether the teeth were primary or permanent. In the commercial dentin bonding systems, gap formation was prevented completely in the group that tested a single bottled dentin bonding system on primary teeth.

In the statistical analysis by Student's t-test by ranks, there was a statistically insignificant difference between permanent and primary teeth in all tested groups without phosphoric acid treatment of a experimental 3 system. On the other hand, in a statistical analysis by Kiuskal-Wallis test of ANOVA by ranks, the difference between all tested groups of permanent or primary teeth were divided to each two groups.

With the SEM study, marginal integrity was observed with EDTA conditioning, GM priming, Clearfil Photo Bond application, and Silux Plus filling in one of the experimental groups of permanent and primary teeth and the Liner Bond II S / Clearfil Photo SC in primary teeth (Figures la, b, 2). In addition, there

Table 2. Comparison of contraction gap width between permanent or primary teeth and each bonding systems.

Bonding System	Permanent Teeth		Primary Teeth
Experimental 1 + Silux Plus	0 (10)	A	0 (10)
Experimental 2 + Cleafil. P. S. C	0.01 ± 0.04 (9)		0.05 ± 0.08 (5)
Experimental 3 + Cleafil. P. S. C	0.08 ± 0.13 (6)	B	0.12 ± 0.14 (5)
Single Bond + Cleafil. P. S. C	0.10 ± 0.09 (2)		0.12 ± 0.13 (3)
Single Bond + Z-250	0.15 ± 0.12 (3)		0.15 ± 0.15 (4)
Liner Bond II ∑ + Cleafil. P. S. C	0.06 ± 0.66 (4)	A	0.07 ± 0.07 (5)
One-up Bond F+Cleafil. P. S. C	0.02 ± 0.04 (7)		0 (10)

%, N=10

mean±SD of the gap values and the number of gap-free specimens are in ( ).
Groups joined by the same line were insignificantly different (ANOVA)

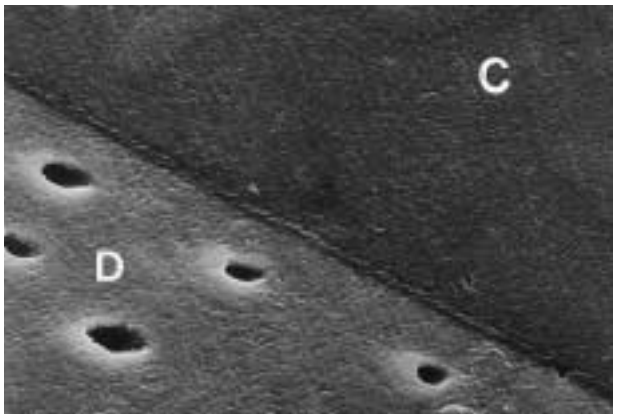

Figure Ia. SEM photograph of complete marginal adaptation between Silux Plus and permanent dentin cavity margin treated with EDTA conditioning, GM priming and Clearfil Photo Bond application (X2000). The bar represents 10mm.

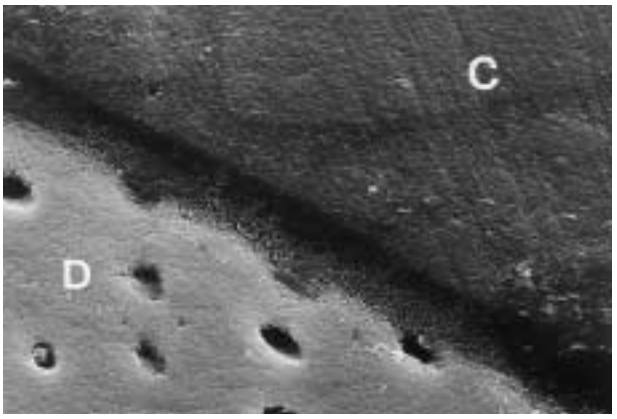

Figure 1b. SEM photograph of complete marginal adaptation between Silux Plus and primary dentin cavity margin treated with EDTA conditioning, GM priming and Clearfil Photo Bond application (x 2000). The bar represents 10mm.

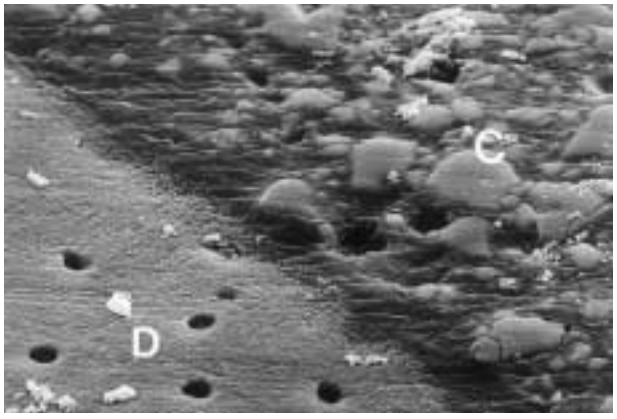

Figure 2. SEM photograph of complete marginal adaptation between Clearfil Photo SC and primary dentin cavity margin treated with Liner Bond II S (x 2000). The bar represents 10mm.

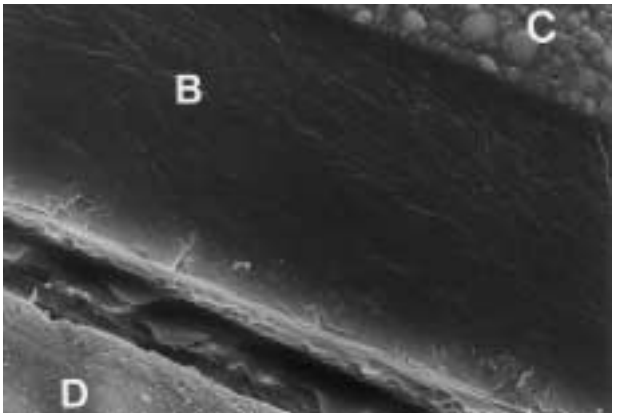

Figure 3. Marginal discrepancy between Z-250 and permanent dentin cavity margin treated with Single Bond (x 2000). The bar represents 10mm.

was no distinct hybrid layer formation when the dentin cavity wall was conditioned with EDTA solution. In the specimens of Single Bond/Z-250 with permanent teeth and phosphoric acid/GM/ Photo SC with primary teeth, a contraction gap formed between the thick bonding agent layer and the dentin (Figures 3, 4).

The results of the Vickers hardness measurement are shown in Tables 3 and 4. In the statistical analysis by

Table 3. Comparison of contraction gap width between permanent and primary teeth.

Bonding System	Permanent Teeth	Primary Teeth
Experimental 1 + Silux Plus	0 (10)	0 (10)
Experimental 2 + Cleafil. P. S. C	0.01 ± 0.04 (9)	0.05 ± 0.08 (5)
Experimental 3 + Cleafil. P. S. C	0.08 ± 0.13 (6)*	0.12 ± 0.14 (5)*
Single Bond + Cleafil. P. S. C	0.10 ± 0.09 (2)	0.12 ± 0.13 (3)
Single Bond + Z-250	0.15 ± 0.12 (3)	0.15 ± 0.15 (4)
Liner Bond II ∑ + Cleafil. P. S. C	0.06 ± 0.66 (4)	0.07 ± 0.07 (5)
One-up Bond F+Cleafil. P. S. C	0.02 ± 0.04 (7)	0 (10)

%, N=10

mean±SD of the gap values and the number of gap-free specimens are in ( ).
* were insignificantly different (Student's t-test.

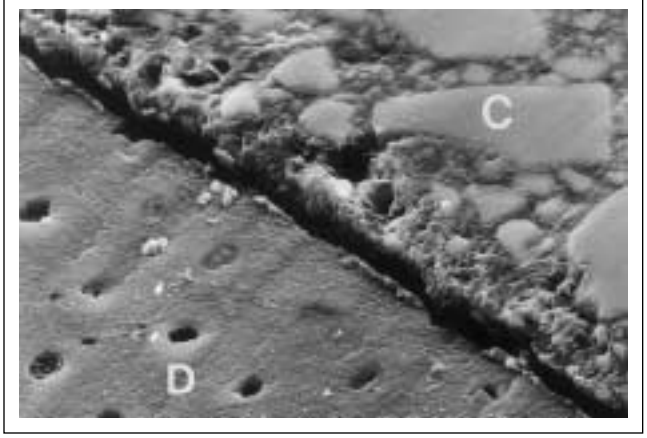

Figure 4. Marginal discrepancy between Clearfil Photo SC and primary dentin cavity margin treated with phosphoric acid etching, GM priming and Single Bond Clearfil Photo Bond application (x 2500). The bar represents 10mm.

Student's t-test by ranks, there was a statistically significant ($p<0.05$) difference between the conditioned dentin surface and the non-conditioned surface in all tested groups except the EDTA group. Furthermore, in the statistical analysis by ANOVA, a difference between non-conditioned permanent and non-conditioned primary teeth was found in all tested groups, but it was not statistically significant. And the statistically insignificant difference between permanent and primary teeth after conditioning was found phosphoric acid conditioning group and Liner Bond II S of permanent teeth.

DISCUSSION

In 1982, Nakabayashi proposed that the detailed mechanism of dentin adhesives is explained by the hybrid layer formation in the superficial dentin layer which is decalcified by the dentin conditioner.[13] In 1984, Munksgaard and Asmussen reported on the pretreatment of the dentin surface with an aqueous mixture of glutaraldehyde and 2 hydroxyethyl methacrylate (2-LIEMA) after which the efficacy of the dentin adhesive was greatly improved.[14] This pretreatment was introduced as the GLUMA dentin primer. They speculated that the amino group in the dentin collagen was chemically activated and might be polymerizable with 2-EDEMA by glutaraldehyde. In 1991, Sugizaki *et al.* reported that the collapsed dentin collagen, which had been exposed by the acidic dentin conditioner was expanded by the dentin primer.[15] Thus, the dentin bonding agent penetrated and polymerized in the enlarged microspace in the collagen network resulting in improved efficacy of dentin adhesives.

In 1991, Gwinnett and Kanca reported that maintaining the acid-etched dentin collagen prevented shrinkage of the collagen network, which was helpful when the dentin adhesive was immersed in the superficial dentin.[16] The expanding effect of the dentin collagen on the dentin primer was based on the observation of the resin-dentin adhesive interface, which was prepared by sandwiching the dentin adhesives between two dentin discs, as proposed by Inokoshi, or coating the dentin rod with a dentin adhesive.[17] In these papers, the dentin bonding agent was found to bond to organic components in the dentin, although most of the adhesive monomers had a chemical structure that bonded to inorganic components. It is widely known that the resin materials exhibited a greater bonding efficacy with enamel than with dentin. Thus, the above mentioned inorganic bonding mechanism conflicted with the high bonding efficacy of the dentin adhesives to the inorganic-rich enamel.

In addition, the efficacy of the dentin adhesives has been widely evaluated by measuring the bond strength to the flat tooth substances. During bond strength measurement, the specimens were frequently fractured inside the resin composite cylinder or inside the substrate dentin, although such fractures were never experienced in clinical practice in general. Furthermore, the bond strength was influenced by the mechanical properties of the resin composite, and higher bond strength was obtained when the resin composite contained a higher amount of inorganic filler.[18] It exhibited more improved cavity adaptation sometime but not all the time. No consistent conclusion has been reported about

Table 4. Comparison of contraction gap width between permanent and primary teeth.

Dentin Treatment	Before	After	Before	After
EDTA	55.32 ± 2.20 ^{IB}	52.52 ± 3.14 ^{IB}	53.92 ± 2.46 ^{IA}	50.49 ± 2.44 ^{IA}
H_3PO_4	51.88 ± 1.85	36.60 ± 0.60	52.00 ± 2.53	36.52 ± 1.38
Primer (Liquid A +B)*1	51.36 ± 1.21	42.48 ± 2.55	54.75 ± 3.08	50.57 ± 2.21
Bonding Agent (A + B)*2	59.60 ± 3.48	50.60 ± 2.91	54.12 ± 1.20	48.22 ± 1.98

N=5

*1; Liner Bond II Σ System
*2; One-up Bond F System
Mean±SD of the Micro Vicker's hardness measurements.
The groups joined by the line were insignificantly different by Student's t-test.

what degree of bond strength is required to obtain adequate clinical performance of dentin adhesives.

In 1975, Asmussen reported on the consistent method to estimate the efficacy of a dentin adhesive in the cylindrical dentin cavity.[11] The primary requirement for dentin adhesives used for resin composite restoration is to prevent marginal gap formation caused by the polymerization contraction stress of the resin composite. As suggested by Asmussen, the interaction between the efficacy of the dentin adhesive and polymerization contraction stress should be assessed soon after completion of the polymerization of the composite under a light microscope. When the efficacy of the dentin adhesive is perfect, the unpolymerized resin composite paste is bonded to the dentin cavity wall during irradiation and the flow of the composite from the free surface into the cavity during polymerization should compensate for the polymerization contraction.

We reported many factors that influenced contraction gap formation. Chiba *et al*. reported that gap width was increased in conjunction with the degree of reduction of dentin hardness by dentin conditioning[19]. In addition, Manabe *et al.* reported that contraction gap formation was promoted when an adhesive monomer, such as 4-methacryloxyethyl trimellitate anhydride (4-META) or the 10-metharyloxydecyl dihydrogen phosphate (10-MDP), was omitted from the dentin bonding agent, whereas tensile bond strength was not affected by the presence or absence of the adhesive monomer in the dentin bonding agent.[20] Thus, it was possible to speculate that contraction gap formation was prevented by the chemical interaction between the calcium in the dentin and the functional monomer in the dentin bonding agent. Chigira *et al*. suggested that the mechanism of GM priming prevented both adhesive monomer infiltration into the dentin and the liquid flow through the dentin tubules.[21] Thus, GM priming prevented the reduction in the adhesive monomer concentration at the adhesive interface and the polymerization was not inhibited by the water coming up through the dentin tubules.

As demonstrated in this study, the marginal adaptation of the resin composite deteriorated after acid etching of the dentin cavity wall in both primary and permanent teeth. The calcium loss in the substrate dentin indicated less bonding to the target in the cavity wall resulting in the separation of the resin composite paste from the decalcified dentin cavity wall. Incomplete marginal adaptation of the commercial total-etch wet bonding system was also caused by a reduction in calcium in the dentin cavity wall. However, dentine hardness was the same in both primary and permanent teeth. Therefore, in order to discuss bonding efficacy, it was necessary to measure and compare calcium concentrations on the superficial dentin surfaces of permanent and primary teeth. In the specimens of the single bond bottled system tested in this study, a significantly thick bonding layer was formed between the resin composite and the dentin cavity wall despite its perfect marginal adaptation in primary teeth.

The bonding agent of this system was composed of an adhesive monomer of 10-methacryloxydecyl propane dioic acid diluted in the monomer as Bis-GMA and TEGDMA. The high viscosity of this material caused a thick bonding layer. It was thought to be important to minimize the intermediate layer between the resin composite and the cavity wall in order to reduce the possibility of marginal discoloration, and the low mechanical resistance of the unfilled bonding layer though the longevity of this system was unknown.

CONCLUSION

Therefore, there was no difference in the detailed mechanism of dentin bonding between primary and permanent teeth. Dentin adhesives were thought to consistently bond to the inorganic component in the tooth regardless of whether it was primary or permanent, and it was essential to avoid decalcification of the dentin cavity wall and to employ the optimum combination of the dentin conditioner, primer, bonding agent, and resin composite.

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