A comparison of five adhesive systems to primary enamel

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Abstract

Purpose: *This study compared resin adhesion of five adhesive systems to primary enamel.*

Methods: *The labial surfaces of 115 bovine mandibular primary incisors and five different adhesive systems were used. Effects of tooth surface conditioners were observed using SEM. Shear bond strengths were tested, and the test surfaces of enamel and resin specimens were observed using SEM.*

Results: *All-Etch™, 10-3 solution, and K-etchant™ were effective tooth surface conditioners. In the nonthermocycled groups, the bond strengths of Superbond D Liner™, All Bond 2™, and Scotchbond Multi Purpose™ adhesive systems were significantly higher than those of K-etchant™/Clearfil Photo Bond™, and Clearfil Liner Bond™ adhesive systems (P< 0.01). However, in the thermocycled groups, no significant difference was obtained among the adhesive systems for bond strength. No correlation was found between the enamel–resin fracture mode and bond strength for any of the adhesive systems.*

Conclusions: *We disagree with the theory that the use of the latest developed adhesive resin systems significantly enhance bonding of resin to primary enamel. (Pediatr Dent 21:46–52, 1999)*

When an adhesive resin is placed on an etched enamel surface, it usually penetrates into the surface irregularities to produce retentive tags[1] to achieve clinical success. An important area of research focuses on new methods to produce a better bond between restorative materials and dentin.

A total-etch technique, in combination with improved formulations of hydrophilic primers and resins, has been advocated as a safe and effective method to achieve significant adhesion to dentin.[2, 3] Such a technique has recently been adopted as a standard protocol for the latest generation of dentin adhesive systems. The latest generation of resin materials shows sufficient bond strengths on both enamel and dentin. However, in a clinical study on primary anterior teeth under the observation period of 7 to 2,303 days by Hosoya et al.[4] some of the third or fourth generation resins showed significantly higher percent loss of resin restorations compared to the second generation resin. In the study,[4] the percent loss of resin restorations was 0% to 12.7% in the second generation groups, and 17.4% to 24.5% in the third or fourth generation groups.

Resin bond strength to dentin is lower than that to enamel,[4–9] therefore, resin adhesion to enamel significantly contributes to the clinical prognosis of composite resin restorations.

The purpose of this study was to compare the bond strength of composite resin to primary enamel using five adhesive systems, and to determine if new generation resin materials had a significant effect on the shear bond strength on primary enamel.

Methods

Labial surfaces of 115 extracted bovine mandibular primary incisors, which had dental pulp removed to avoid degeneration and frozen in physiologic saline were used. Five adhesive treatment groups were prepared; details of material compositions, manufacturers, application techniques and light curing times for each of the adhesive systems is given in Table 1.

Efficacy of tooth surface conditioners

Labial surfaces of 15 bovine primary incisors (three teeth for each of the enamel surfaces of the five adhesive systems) were used. To obtain a flat enamel surface, the labial surface was abraded with 400- and 600-grit wet silicon carbide papers. The enamel surfaces were treated with one of five tooth surface conditioners shown in Table 1 and then washed with an air–water spray for 10 sec. Observations of the

TABLE 1. ADHESIVE SYSTEMS TESTED

Group	*Tooth Surface Conditioner*	*Primer*	*Bonding Agent*	*Liner*	*Composite Resin*
1	K-etchant <40% phosphoric acid> [kuraray Co. Kurashiki, Japan] (40 sec.)		Clearfil Photo Bond <phosphoric ester monomer:MDP+HEMA> [Kuraray Co.] (wait 10 sec., air blow 1 sec., light irradiate 10 sec.)		Clearfil Anterior-A2 <hybrid, Bis-GMA, UTMA TEDGMA> [kuraray Co.] (light irradiate 40 sec.)
2	CA agent <10% citric acid 20% calcium chloride> [kuraray Co.] (40 sec.)	SA Primer <salicylic acid derivative monomer: 5-NMSA> [kuraray Co.] (wait 60 sec., airblow 1 sec.)	Clearfil Photo Bond		Clearfil Photo Anterior-A2
3	10-3 solution <3% ferric chloride into 10% citric acid> [Sunmedical Co. Moriyama, Japan] (40 sec.)			Superbond D Liner <MMA, 4-META polyfunctional monomer + TBB> {Sunmedical Co.} (air blow 1 sec., wait 120 sec.)	Clearfil Photo Anterior-A2
4	All-Etch <10% phosphoric acid> [Bisco Inc. Itasca, IL] (agitate 15 sec.)	Primer A and B Primer A <NTG-GMA> Primer B <BPDM> [Bisco Inc.] (apply 5 times, air blow 5 sec.)	Dentin/Enamel Bonding Resin <Bis-GMA, UDMA, Hema> [Bisco Inc.] (light irradiate 20 sec.)		Clearfil Photo Anterior-A2
5	Scotchbond Multi Purpose Etchant <10% maleic acid> [3M Co. St. Paul, MN] (15 sec.)	Scotchbond Multi Purpose Primer <HEMA> [3M Co.] (air blow 5 sec.)	Scotchbond Multi Purpose Adhesive <Bis-GMA, HEMA> [3M Co.] (wait 10 sec., air blow 1 sec., light irradiate 10 sec.)		Z100-A3.5 <microfilled, Bis-GMA, TEGDMA> [3M Co.] (light irradiate 40 sec.)

flat enamel surfaces were conducted using a scanning electron microscope (SEM). According to previous studies,[11, 12] if the clear prism structure was observed on the etched enamel surface, it was judged that the efficacy of the tooth surface conditioner was high. If no prism structure could be observed on the etched enamel surface, it was judged that the efficacy of the tooth surface conditioner was low. The etching patterns were judged in the same way. The SEM views were studied under 25x to 20,000x magnifications. All data were analyzed using the chi-square test.

Bond strength

Labial surfaces of 100 bovine primary incisors (10 teeth for the nonthermocycled groups and the thermocycled groups for each of five adhesive systems)

TABLE 2. EFFICACY OF TOOTH SURFACE CONDITIONERS

Tooth Surface Conditioners	Prism Structure #	Prism Structure +	Prism Structure —	Etching Patterns [11,12] Prism Peripheries	Prism Cores	Peripheries & Cores	Poorly Structured	Number of Cases (%)
K-etchant	3 (100)	0	0	3 (100)	0	1 (33.3)	0	3
CA agent	1 (33.3)	2 (66.7)	0	3 (100)	1 (33.3)	2 (66.7)	1 (33.3)	3
10-3 solution	3 (100)	0	0	3 (100)	0	0	0	3
All-Etch	3 (100)	0	0	3 (100)	1 (33.3)	3 (100)	1 (33.3)	3
SMP Etchant	0	3 (100)	0	3 (100)	0	1 (33.3)	1 (33.3)	3

were used. Flat enamel surfaces were obtained in the same manner as for tooth surface conditioners. These surfaces were treated with tooth surface conditioners (Table 1). All of the specimens were washed with an air–water spray for 10 sec and then dried. Self-adhesive tape was used on the enamel to mask off a circular area 3 mm in diameter. A primer, bonding agent, and liner were applied to the exposed surfaces according to the application techniques shown in Table 1. A brass ring with an inside diameter of 4 mm and a height of 2 mm was placed on the test surfaces. The ring was filled with the composite resin and irradiated with a visible light activation unit VCL 300 (Demetron Co, Danbury, CT) for 40 sec. The specimens were left in air for 30 min then immersed in water and stored at 37°C for 24 h (non-thermocycled groups). In the thermocycled groups, specimens were then cycled 10,000 times between water baths maintained at 4°C and 60°C. The dwell time in each bath was 1 min. All specimens were embedded in a large metal ring with a self-curing resin Plastik kit™(Buehler Co., Lake Bluff, IL).

TABLE 3. SHEAR BOND STRENGTHS ON THE PRIMARY ENAMEL (UNIT:MPA)

Group	Thermocycling Times	Mean ± SD	Max.	Min.	Number of Cases
	0	20.33±5.20 •	27.46	12.62	10
1	10000	32.10±10.68	47.84	13.17	10
	0	16.14±7.15 •	27.73	4.71	10
2	10000	27.83±9.08	41.05	11.23	10
	0	36.47±10.80 †	62.12	22.46	10
3	10000	26.62±9.98	45.48	9.57	10
	0	29.91±7.25	40.08	20.66	10
4	10000	31.31±20.21	59.90	10.26	10
	0	35.78±5.70	42.29	25.52	10
5	10000	29.88±11.19	45.76	13.31	10

• $P < 0.01$. † $P < 0.05$.

TABLE 4. FRACTURE MODES BETWEEN PRIMARY ENAMEL AND RESIN (%)

Group	Thermocycling Times	Enamel Fracture	Adhesive Fracture	Cohesive Resin Fracture	Mixed Fracture	Number of Cases
	0	0	8 (80)	0	2 (20)	10
1	10000	0	10 (100)	0	0	10
	0	0	4 (40)	0	6 (60)	10
2	10000	0	7(70)	0	3 (30)	10
	0	0	5 (50)	0	5 (50)	10
3	10000	0	6 (60)	0	4 (40)	10
	0	0	3 (30)	1 (10)	6 (60)	10
4	10000	0	4 (40)	0	6 (60)	10
	0	0	5 (50)	0	5 (50)	10
5	10000	0	6 (60)	0	4 (40)	10

The shear bond strength was tested with an autograph DCS-500™ (Shimazu Pro-duct Inc, Kyoto, Japan) at a cross-head speed of 0.5 mm/min in the same manner of the previous study.[13] Shear bond strengths were analyzed using the Student *t*-test.

Fracture mode

After the shear bond strength test, the test surfaces of the enamel and the resin were observed using SEM. The SEM views were studied under 25x to 20,000x magnifications. The fracture modes of each specimen were determined by 30x magnification. The modes of fracture were designated as enamel fracture if the bonded enamel surface was fractured (regardless of the range of the fracture); adhesive fracture if 100% of the bonded interface failed between the enamel and the bonding resin; cohesive resin fracture if 100% of the failure was in the resin composite; or mixed fracture if the failures were partially adhesive and partially cohesive resin fracture and/or enamel fracture.

The relationships between the fracture modes and the shear bond strengths were observed according to the previously reported standard[14–16] and the data were analyzed using the chi-square test.

Results

Efficacy of tooth surface conditioners

K-etchant, 10-3 solution, and All-Etch showed clear prism structures (higher efficacies) than did the CA agent and SMP Etchant™. The efficacy of SMP Etchant was the lowest. The peripheral etching patterns [11, 12] (Type II by Silverstone et al.[17]) were the most commonly seen pattern for all the treated enamel surfaces (Table 2).

However, tooth-to-tooth differences influenced tooth surface conditioning efficacy. The differences were noted not only on various teeth within the same groups but also on the particular portions within an individual tooth.

Bond strength

Table 3 shows the shear bond strengths on the primary enamel. In the comparison of the bond strengths of the nonthermocycled group and the thermocycled group, in group 1 and group 2 the bond strengths of the thermocycled groups were significantly higher than those of the nonthermocycled groups ($P<0.01$). In group 3, the bond strength of the nonthermocycled group was significantly higher than that of the thermocycled group ($P<0.05$). In the comparison of the bond strengths among the adhesive systems, in the nonthermocycled groups the bond strengths of groups 3, 4, and 5 were significantly higher than those of groups 1 and 2 ($P<0.01$). However, in the thermocycled groups, no significant difference was obtained among the adhesive systems.

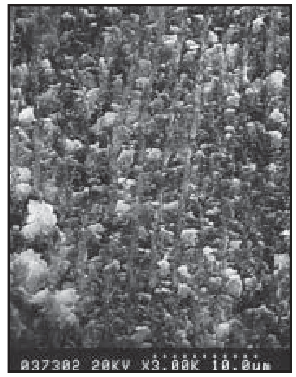

Fig 1. Enamel specimen of Group 1 thermocycled group. The bond strength was 47.84MPa MPa.

Fig 2. Resin specimen of the Fig 1 case.

Fig 3. Enamel specimen of group 3 nonthermocycled group. The bond strength was 62.12 MPa.

Fig 4. Resin specimen of the Fig 3 case.

Fracture mode

The enamel–resin adhesion pattern differed among the adhesive systems.

Table 4 shows the fracture modes between primary enamel and resin. Enamel fracture was not observed on any enamel specimen. Cohesive resin fracture was observed in only one case in group 4 nonthermocycled group. In both the non-thermocycled and the thermocycled groups of all adhesive systems, the difference in numbers of cases that showed adhesive fracture and mixed fracture were not significant among the five adhesive systems.

Figs 1–4 show the scanning electron microscopy views of the enamel specimens and the resin specimens after shear bond strength tests. In both the non-thermocycled and the thermocycled groups of all adhesive systems, in groups 3 and 5, the number of the cases that showed numerous clear resin tags on the resin surface (Figs 2 and 4) was significantly higher than those in groups 1 and 2 ($P<0.01$). There was no correlation between the enamel–resin fracture mode and the bond strength for any of the adhesive systems.

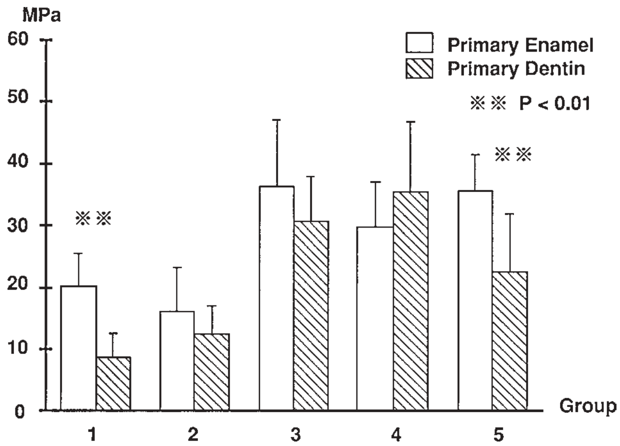

Fig 5. Comparison of the shear bond strengths on primary enamel and primary dentin in the nonthermocycled groups.

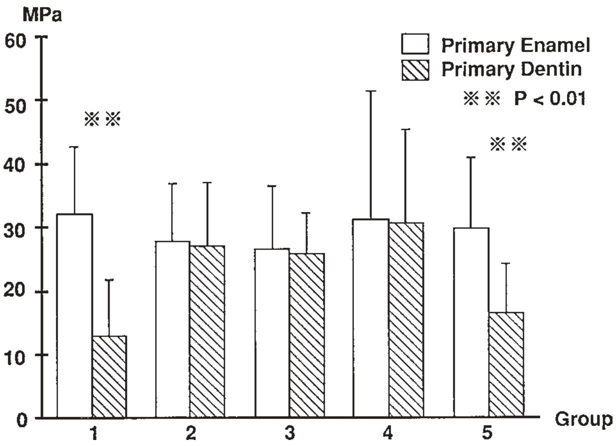

Fig 6. Comparison of the shear bond strengths on primary enamel and primary dentin in the thermocycled groups.

Discussion

Optimal tooth surface conditioning times to obtain sufficient bond strength of resin to enamel and to dentin are different. If the highest bond strengths of resin to enamel and to dentin can be obtained by the same tooth surface conditioning time, it would be beneficial for clinical use. We have previously reported the bond strength and bonding mechanism of resin to primary dentin using six adhesive systems, including all adhesive systems used in this study.[15, 16] In this study and our previous studies,[15, 16] the application times of tooth surface conditioners for enamel and dentin were the same, except for the 10-3 solution. The 10-3 solution was applied for 40 sec on enamel, but applied for 10 sec on dentin[15, 16] based on data reported by Nakamura et al.[18]

The etching patterns of primary ground enamel produced by 40% phosphoric acid have been reported previously[11] and are regarded as necessary to mechanically bond adequately.[13] It is generally understood that smear layer removal is required to obtain high bond strength with adhesive resinous materials. Recently, a total-etch technique has been adopted as a standard protocol for the latest generation of dentin adhesive systems. Pashley et al.[19] have shown that removal of the smear layer barrier will allow bacterial invasion of dentinal tubules and pulp if microleakage should occur after placement of the composite resin restoration. To allay this concern, dental material manufacturers have either replaced the tooth surface conditioner component with an acid that has a higher pH, or reduced the phosphoric acid concentration.

In this study and in our previous studies,[15, 16] All-Etch (10% phosphoric acid) and 10-3 solution (3% ferric chloride into 10% citric acid) were effective tooth surface conditioners on both enamel and dentin, and caused clear prism structures on enamel (Table 2) and complete removal of the smear layer on dentin. K-etchant (40% phosphoric acid) showed a clear etching pattern on enamel, although the efficacy on dentin[15, 16] was lower than All-Etch, SMP Etchant, and 10-3 solution. SMP Etchant (10% maleic acid) was efficacious on dentin,[15, 16] although the efficacy on enamel was low. CA agent (10% citric acid, 20% calcium chloride) showed the lowest tooth surface conditioning efficacy on both enamel and dentin.[15, 16] On the dentin treated with K-etchant or CA agent, small particles were observed. The particle is considered to be the silica included in the tooth surface conditioner.

In this study and our previous ones,[15, 16, 20] enamel or dentin fracture was not observed on the primary enamel or the primary dentin specimens in any of the adhesive treatment groups, and there was no correlation between the enamel–resin fracture mode and the bond strength, or between the dentin–resin fracture mode and the bond strength in any of the adhesive systems.

In groups 1 and 2, the bond strengths on the enamel of the nonthermocycled groups were significantly lower than those of the thermocycled groups (Table 3). In groups 1 and 2, the degree of polymerization of diffused resin monomer might be low, and lower bond strengths were obtained in the nonthermocycled groups. Polymerization of the residual monomer was accelerated with time and heat, which led to improved resin physical properties and increased bond strengths in the thermocycled groups. In the nonthermocycled groups, the bond strengths on the enamel of groups 1 and 2 were significantly lower than those of groups 3, 4, and 5. For the above reasons, the adhesive systems used in groups 1 and 2 were not suitable for primary enamel.

In group 3, on the other hand, the bond strength on enamel of the thermocycled group was significantly lower than that of the nonthermocycled group (Table 3). In group 3, reduction of the bond strength may be caused by deterioration of adherence between the enamel and resin due to the physical properties of the resinous material as affected by the long-term thermocycling test. However, in the thermocycled groups, a significant difference was not obtained among the adhesive systems used on the enamel.

Figs 5 and 6 show the bond strengths to the primary enamel and the primary dentin.[15, 16] Both in the nonthermocycled groups and in the thermocycled groups, in groups 1 and 5, the bond strengths to enamel were significantly higher than that to dentin. In group 1, 40% phosphoric acid was used as a tooth surface conditioner, but a primer and a liner were not applied on dentin. The bond strengths to dentin, both in the nonthermocycled group and in the thermocycled group of group 1, were significantly lower than those of groups 3, 4, and 5.[15, 16] The bond strength to dentin in the thermocycled group of group 5 was significantly lower than those of groups 2, 3, and 4.[15, 16]

Significantly lower bond strengths to dentin in group 1 (K-etchant/Clearfil Photo Bond) and group 5 (SMP system)[15, 16] may suggest that these adhesive systems are not as effective on primary dentin compared with the other adhesive systems. Bordin-Aykroid et al.[21] and Elkins et al.[22] have studied the shear bond strengths to human primary dentin. They reported that the bond strengths using SMP system were each of 12.3 MN/cm[21] and 6.99 MPa,[22] the bond strength using All-Bond with Valux was 13.01 MPa,[22] and the bond strength using Amalgambond (Superbond D Liner) with Valux was 13.03 MPa.[22] Hallett et al.[23] reported that the shear bond strengths to human primary enamel using SMP system with Z-100 was 11.18 MPa. Compared with these bond strengths, the bond strengths to primary enamel and primary dentin obtained in this study and in our previous studies[15, 16] showed significantly higher values. On the other hand, Triolo et al.[24] studied the shear bond strengths to permanent dentin. They reported that the bond strengths using SMP system and All Bond 2 with Bis-Fil composite were 23.1 MPa and 21.4 MPa respectively. Bond strength to enamel and to dentin varies according to several factors. The type of bond strength test and the resinous materials[16] are direct factors. Recently, Sano et al.[25] reported that the tensile bond strength was inversely related to the bonded surface area. It is difficult to compare the bond strengths obtained with the different methods.

In group 3 (Superbond D Liner system) and in group 4 (All Bond 2 system), high bond strengths were obtained to both enamel and dentin (Figs 5 and 6). However, all of the bond strengths to enamel and dentin obtained in this study and in our previous studies[15, 16] were significantly lower than the bond strengths on the bovine primary enamel previously reported by Hosoya et al.[13] In that study,[13] each of the highest bond strengths in the nonthermocycled group and in the thermocycled group to primary enamel using K-etchant/Clearfil Photo Bond with Photo Clearfil A were 80.1 MPa and 61.6 MPa and 37% phosphoric acid/Scotch Bond with Silux were 76.8 MPa and 63.6 MPa respectively. The method for the shear bond strength test used in the previous study[13] also was used in this study.

The results of the clinical study by Hosoya et al.,[4] and the results of this study disagree with the theory that the use of the latest developed adhesive resin systems may significantly enhance bonding of resin to primary enamel.

Conclusions

1. All-Etch, 10-3 solution, and K-etchant were effective tooth surface conditioners on the primary enamel.
2. In the nonthermocycled groups, the bond strengths of Superbond D Liner, All Bond 2, and Scotchbond Multi Purpose adhesive systems were significantly higher than those of K-etchant/Clearfil Photo Bond and Clearfil Liner Bond adhesive systems.
3. In the thermocycled groups, no significant difference of bond strengths among the adhesive systems was observed.

The authors gratefully acknowledge the assistance by Dr. Kyoko Kakazu and Dr. Yoko Kashiwabara, Department of Pediatric Dentistry, Nagasaki University School of Dentistry.

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