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STUDIES ON NEWLY DESIGNED TOOTH SURFACE TREATMENTS FOR ADHESIVE COMPOSITE RESTORATION

BY

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ABSTRACT

Recently, problems have occurred frequently such as cervical and exposed dentin surface lesions or root caries and subsequent hypersensitivity because of further increase of the elderly population. This study was projected to devise a proper and simple treatment and materials for the adhesive composite restoration for such lesions. The 10–20Ca (10% citric acid containing 20% calcium chloride) solution was found to be a comparatively mild tooth surface conditioning agent and to be effective with a single application to both dentin and enamel. In addition, the use of the SA primer (3% N-methacryloyl 5-aminosalicylic acid in 80% ethanol) and the LVR (visible light-cured, 33% microfilled low viscous Bis-GMA resin) dramatically improved the adhesion and adaptability of the composite restoration in the saucer cavity at the cervical area.

Key words: Adhesive composite restoration, Tooth surface conditioning Primer, Adhesion promoter, Adhesion, Adaptation

INTRODUCTION

Phosphoric acid etching used along the bonding agent has provided excellent adhesion and adaptation of resinous materials to enamel, while it was not so effective for dentin (Hosoda et al. [1]; Fujitani et al. [2]). In addition, its decalcifying effect on dentin is so serious that the degeneration of the etched collagen fibers might cause some disadvantageous factors to the adhesion of composite restoration (Okamoto et al. [3]; Mizunuma [4]). Surface treatment with 10% citric acid + 3% ferric (III) chloride aqueous solution devised by Nakabayashi et al. [5] relatively suppressed the degeneration of the dentin collagen and improved the bond of the resin composites to dentin. However, it was not effective enough for enamel to obtain a good adhesion of the resins. Lately, the GLUMA system was developed by Munksgaard and Asmussen [6], which is considered to have the problem of pulpal irritation since it contains glutaraldehyde at a high concentration (Harnirattisai and Hosoda [7]). Furthermore, this system is quite complicated in the clinical manipulation as it requires another kind of conditioning agent for enamel, a phosphoric acid. Thus, a proper tooth surface treatment, which is comparatively mild especially for dentin and also is effective with a single application to both enamel and dentin, has not been devised yet.

Recently, problems have occurred frequently such as cervical and exposed root surface lesions or root caries and subse-

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quent hypersensitivity because of further increase of the elderly population. Most of those defects or cavities are composed of dentinal walls, to which the resinous materials cannot totally adhere, and thus their complete wall adaptation can be hardly obtained (Fujitani et al. [2]). There are also reports which showed that a subsequent application of an adhesive primer to the conditioned dentin surface enhanced the bond and improved the adaptation between the composite restorative materials and the cavity walls. The authors (Hosoda et al. [1]) have already reported that the adhesion promoting efficacy of N-methacryloyl 5-aminosalicylic acid, a monomer containing a salicylic acid derivative.

From these points of view, it is urgently needed to establish a simplified yet secure tooth surface treatment. This study was projected to develop a new tooth surface treatment. As conditioning agents, several low-concentrated citric acid aqueous solutions containing various metal salts, which have been considered to cause comparatively less damage to dentin (Mizunuma [4]), were tested. The effects of these conditioners with or without further application of the two kinds of adhesion promoters on the tensile bond strength to bovine dentin and also on the wall adaptation of the resinous materials were also examined, comparing with the phosphoric acid-etch technique.

Materials and Methods

I. Tensile bond test to flat dentin and enamel surfaces

For tooth surface conditioning agents, 10% citric acid aqueous solution (10citric) and 10% citric acid which was compounded with 3% ferric (III) chloride (10–3Fe), 10, 20, 30% calcium chloride (10–10Ca, 10–20Ca, 10–30Ca), 20% tannic acid (10–20Tan), 20% magnesium chloride (10–20Mg), 20% potassium chloride (10–20K), and 20% copper (II) chloride (10–20Cu) were prescribed. A phosphoric acid etchant (K-etchant, Kuraray Co.) and 0.5M EDTA (EDTA) were used as control (Table 1). Flat dentin and enamel surfaces were finished with #600 water-proof silicon carbide paper under running water, using freshly extracted bovine teeth. A black adhesive tape with a hole of ϕ5.5 mm

Table 1. Materials Tested

		Code	Lot No.	Source
Conditioning agent	K-etchant	K-etch	ET-435	Kuraray
	0.5M EDTA	EDTA		Experimental
	10% citric acid	10citric		Experimental
	10% citric acid– 3% ferric (III) chloride	10–3Fe		Experimental
	10% citric acid–10% calcium chloride	10–10Ca		Experimental
	10% citric acid–20% calcium chloride	10–20Ca		Experimental
	10% citric acid–30% calcium chloride	10–30Ca		Experimental
	10% citric acid–20% tannic acid	10–20Tan		Experimental
	10% citric acid–20% magnesium chloride	10–20Mg		Experimental
	10% citric acid–20% potassium chloride	10–20K		Experimental
	10% citric acid–20% copper (II) chloride	10–20Cu		Experimental
Bonding agent	Clearfil Photo Bond	PB	Cat. 207 Uni. 107	Kuraray
Adhesive liner	SA Primer	SA	TSA-13	Kuraray
	Visible light-cured low viscous resin	LVR	RT-072	Kuraray
Resin composite	PhotoClearfil Bright	PhBr	HBX-7002	Kuraray

or ϕ4.0 mm was placed on the dentin or the enamel surface, respectively, in order to fix and limit the adhesion test area. The test surface was cleansed with 80% ethanol solution, conditioned for 40 seconds using various experimental conditioners and then well rinsed and dried. A dual-cured bonding agent, Clearfil Photo Bond (PB) (Kuraray Co.) was applied to the conditioned surface for 30 seconds and was irradiated by using Quick Light (Kuraray Co.) for 20 seconds. Then, a visible light-cured resin composite, PhotoClearfil Bright (PhBr) (Kuraray Co.) was placed with a thickness of 0.5 mm and polymerized for 60 seconds. The specimen was stored in distilled water at 37°C for 24 hours prior to the tensile bond test, which was performed at the crosshead speed of 2 mm/min at room temperature. Ten pieces of specimens for each conditioner were supplied for the test.

II. Tensile bond test to flat dentin surfaces using two kinds of adhesion promoters

The bond strength of the resin composites to the flat dentin surfaces under various tooth surface treatments were examined. Flat bovine dentin surfaces were prepared in the same way as in Experiment I. Ten teeth were supplied for each surface treatment. The test surface was primarily conditioned for 40 seconds with the conditioning agents which exhibited the highest bond among the conditioners examined in Experiment I. PhBr resin was placed with a thickness of 0.5 mm and polymerized for 60 seconds after the following procedure: 1) PB application and 20-second irradiation, 2) 30-second application of 80% ethanol solution, drying, PB application and 20-second irradiation, 3) SA (3% N-methacryloyl 5-aminosalicylic acid in 80% ethanol) priming for 30 seconds, drying, PB application and 20-second irradiation, 4) LVR (visible light-cured, 33% microfilled low viscous Bis-GMA resin) application and 20-second irradiation following the same surface treatment as in 1), 5) LVR application and 20-second irradiation following the same treatment as in 2), and 6) LVR application and 20-second irradiation following the same treatment as in 3). The thickness of LVR was regulated by placing a looped wire of 250 μm in diameter on to the adhesive tape. LVR was properly applied with a small brush and covered by a thin glass prior to photo-irradiation. As controls, the bond strengths were measured in the specimens where the dentin surfaces were not conditioned but treated in the same way as in 3) or 6). The specimens were stored in distilled water at 37°C for 24 hours. The tensile bond test was performed at the crosshead speed of 2 mm/min at room temperature.

III. Tensile bond test and leakage test in saucer type dentin cavities

The adhesion of the resin composites to the flat surfaces and saucer type cavity walls of bovine dentin under varying tooth surface treatments was studied by the method conventionally used in our laboratory (Okuya [8]). Flat surfaces and standardized saucer cavities were prepared in the freshly extracted bovine dentin (Fig. 1) and were treated for 40 seconds with the conditioner which exhibited the highest bond in Experiment I. The PhBr resin was placed with a thickness of 1 mm onto the flat surface or in the cavity after the following procedure; 1) PB application and 20-second irradiation, 2) SA priming for 30 seconds, drying, PB application and 20-second irradiation, and 3) LVR application (approximately 200–250 μm thick) and 20-second irradiation following the same surface treatment as in 2). The specimens were then thermally stressed

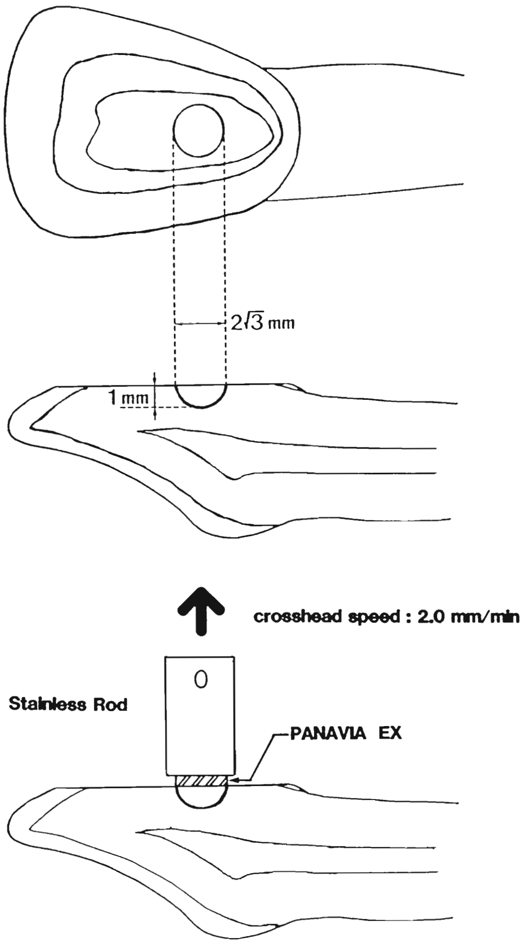

Fig. 1. Standardized Saucer Type Cavity Prepared in Bovine Dentin

for 100 cycles between the water baths of 4°C and 60°C, being kept in each bath for 30 seconds. The tensile bond strengths were measured after 24 hours, followed by the microleakage examination using the dye penetration method (Fujitani et al. [2]). Furthermore, correlations between them were evaluated. Ten teeth were used for each surface treatment.

IV. Marginal leakage and adaptation of composite restoration in saucer cavities at the cervical area

The marginal integrity and the wall

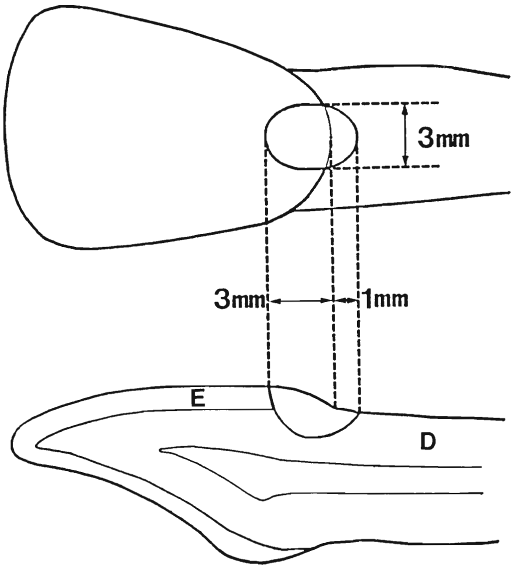

Fig. 2. Saucer Type Cavity Prepared at Cervical Area of Bovine Tooth
E: Enamel, D: Dentin.

adaptation at the cervical area of composite restoration inserted in the saucer type cavities were evaluated using the dye penetration method. Saucer cavities with cervical margins finished approximately 1 mm below the CEJ were prepared in freshly extracted bovine teeth (Fig. 2). The cavities were restored with PhBr after the same cavity surface treatments as mentioned in Experiment II. The resin restorations were finished and then thermally stressed for 100 cycles. They were stored in distilled water at 37°C for 24 hours until the time of the test. A dye penetration test was performed on the longitudinal section surface of each restored tooth by applying 15% acid fuchsin aqueous solution for 60 seconds. Ten teeth were carefully examined for each surface treatment under the light microscope and scanning electron microscope (SEM). Following the test, the boundaries between the restorations and the cavity walls were observed in some of the specimens under the SEM, using a replication technique (Endo et al. [9]).

V. pH measurement of various tooth surface conditioning agents

The pH values of the tooth surface conditioning agents tested in Experiment I were measured using the pH meter (pHI 12, Beckman Instruments, Inc.) at room temperature. The measurement was made five times per conditioner.

VI. Observation of the conditioned enamel and dentin surfaces

Three human incisal teeth freshly extracted due to the periodontal disease were used. Flat enamel and dentin surfaces were finished with #600 water-proof silicon carbide paper under running water. The surfaces were cleansed with 80% ethanol solution, conditioned for 40 seconds with K-etch or 10–20Ca and then well rinsed and dried. The morphology of the conditioned enamel and dentin surfaces was observed under the SEM.

Results

I. Bond strengths of various tooth surface conditioners

Figs. 3 and 4 present the bond strengths of PhBr to the flat dentin and enamel surfaces treated with various conditioning agents. In the dentin surfaces, 10–20Ca showed the highest bond of 79.5±12.4 kg/cm^2 (mean±S.D.), and no significant differences (U-test, p>0.01) could be found among the five conditioners (10–20Ca, EDTA, 10–20Cu, K-etch, 10–20K) which indicated relatively higher bond strengths. In the enamel surfaces, K-etch exhibited the highest bond of 184.4±29.9 kg/cm^2 and 10–20Ca was 179.4±20.4 kg/cm^2. Generally, however, the bond strenghs to enamel showed no significant differences (U-test, p>0.01) among the five condition-

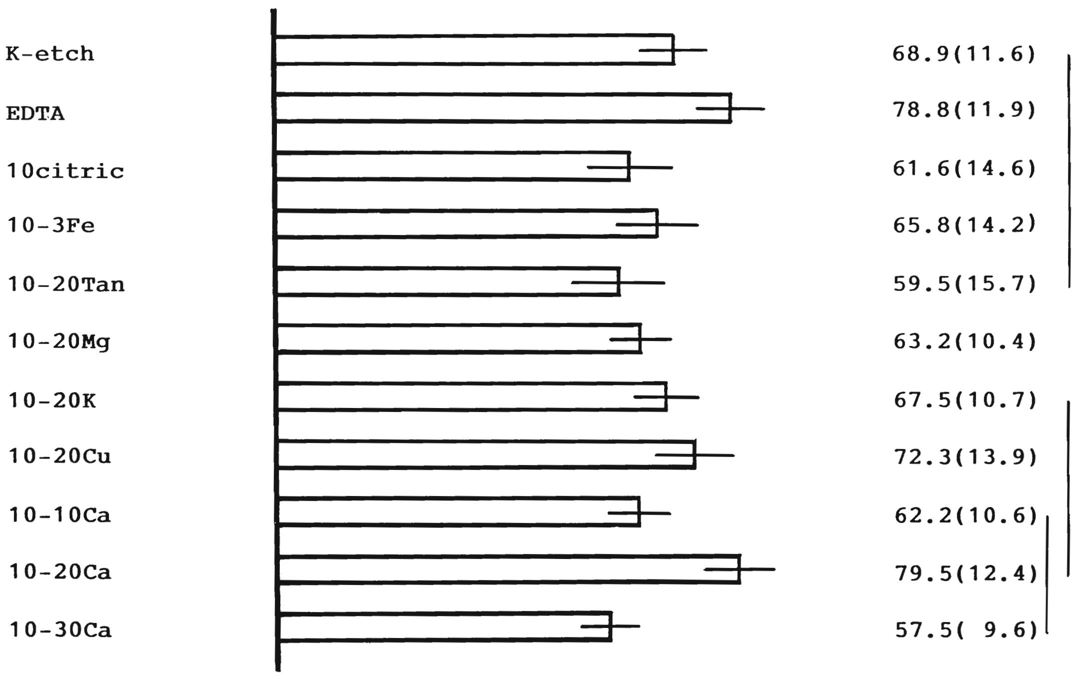

Fig. 3. Tensile Bond Strengths of PhotoClearfil Bright to Flat Bovine Dentin Surfaces Treated With Various Conditioning Agents [Mean(S.D.), kg/cm^2]
The vertical lines indicate no significant difference between the groups by the U-test at the 99% confidence interval.

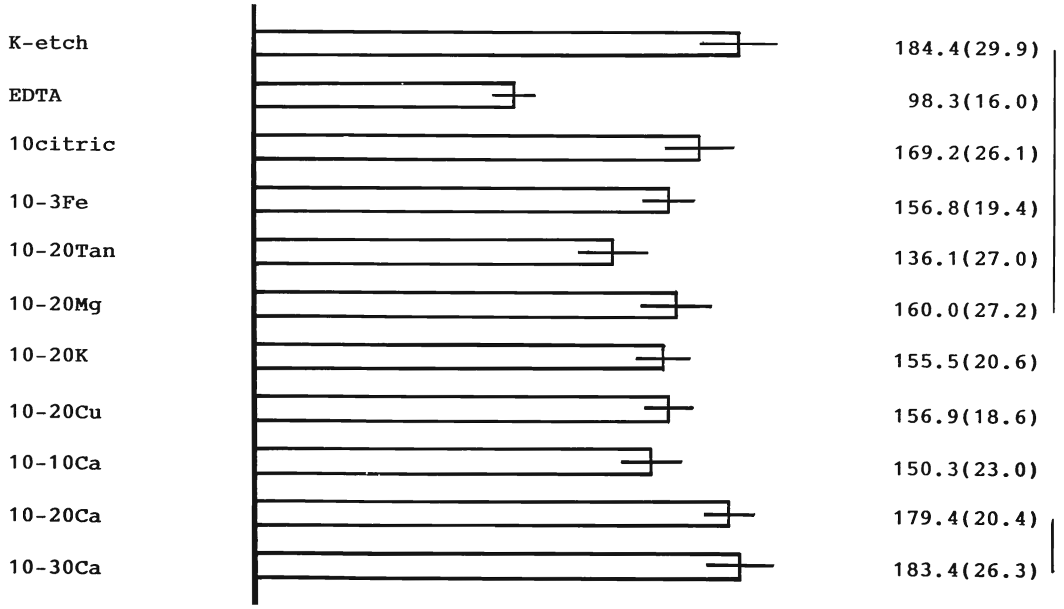

Fig. 4. Tensile Bond Strengths of PhotoClearfil Bright to Flat Bovine Enamel Surfaces Treated With Various Conditioning Agents [Mean(S.D.), kg/cm^2]
The vertical lines indicate no significant difference between the groups by the U-test at the 99% confidence interval.

Table 2. Tensile Bond Strengths of PhotoClearfil Bright to Flat Bovine Dentin Surfaces Under Varying Tooth Surface Treatments [Mean(S.D.), kg/cm^2]

Surface treatment	Bond strength
K-etch + PB	68.9(11.6)
10–20Ca + PB	79.5(12.4)
10–20Ca + 80%EtOH + PB	76.1(18.8)
No conditioning + SA + PB	43.5(14.2)
10–20Ca + SA + PB	109.7(15.8)
10–20Ca + PB + LVR	87.8(19.7)
10–20Ca + 80%EtOH + PB + LVR	90.4(16.7)
No conditioning + SA + PB + LVR	56.7(19.1)
10–20Ca + SA +PB + LVR	123.5(19.1)

The vertical lines indicate the nonsignificant differences between the groups by the U-test at the 99% confidence interval.

ers which were K-etch, 10–30Ca, 10–20Ca, 10citric and 10–20Mg. From these findings, the conditioning agents which demonstrated significantly high bond strengths through both dentin and enamel were 10–20Ca.

II. Bond strengths of various tooth surface treatments

Table 2 showed the tensile bond strengths of PhBr to the flat dentin surfaces with various tooth surface treatments. When the dentin surfaces were not conditioned at all, significantly lower bond

Table 3. Tensile Bond Strengths of PhotoClearfil Bright to Flat Surfaces and Saucer Type Cavity Walls of Bovine Dentin [Mean(S.D.), kg/cm^2]

Surface treatment	Flat surface	Saucer cavity
K-etch + PB	73.1(13.9)	55.4(11.4)
10–20Ca + PB	76.8(12.8)	63.6(15.4)
10–20Ca + SA + PB	106.2(14.3)	110.8(17.9)
10–20Ca + SA + PB + LVR	126.7(14.4)	115.6(23.7)

The vertical lines indicate no significance between the groups (U-test, $p>0.01$).

strengths were demonstrated as compared with the 10–20Ca-conditioned specimens (U-test, $p<0.01$). Greater adhesion could not be obtained when 80% ethanol solution, which is the solvent of the SA primer, was used instead of SA. When the SA primer or both SA and LVR were applied following the dentin conditioning with 10–20Ca, the bond strengths considerably improved to 109.7±15.8 kg/cm^2 or 123.5±19.1 kg/cm^2, respectively (U-test, $p<0.01$).

III. Bond strengths of resin composites to saucer type dentin cavities and their marginal leakage

The tensile bond strengths of PhBr to the flat surfaces and saucer type cavity walls of bovine dentin are tabulated in Table 3. The bond strengths to the flat surface considerably improved when the SA primer or both SA and LVR were applied following the surface treatment with the 10–20Ca solution (U-test, $p<0.01$). The adhesion to the dentin cavity greatly improved when SA was used (U-test, $p<0.01$), however, subsequent application of LVR did not have remarkably the adhesion promoting-effect (U-test, $p>0.01$). In both the flat surface and the cavity, the highest bond was obtained when both SA and LVR were used, 126.7±14.4 kg/cm^2 for the flat surface and 115.6±23.7 kg/cm^2 for the cavity. These data demonstrated the efficacy of these materials as adhesion-promoting agents. The number of the specimens which showed marginal leakage around the cavity walls was two for K-etch and one for the rest of each surface pretreatment. Correlation between the bond strength and marginal adaptation was hardly detected in this experiment.

IV. Cavity-wall adaptation of composite restoration in saucer cavities at the cervical area

The resinous fillings exhibited complete adaptation to the enamel walls of the cervical cavities regardless of the surface treatment methods. Thus, the degree of the marginal seal and adaptation of PhBr to the dentinal walls was evaluated. When replacing the conditioning agents from K-etch to 10–20Ca, the adaptation of PhBr was somewhat improved. Unlike the tensile bond test, subsequent application of SA only did not appear to significantly improve the adaptability. However, greater improvement in the marginal seal and adaptation was demonstrated when both SA and LVR were used. Some specimens showed no penetration of the dye and no gap formation on the SEM image especially when both SA and LVR were applied (Figs. 5 and 6).

V. pH values of tooth surface condition-

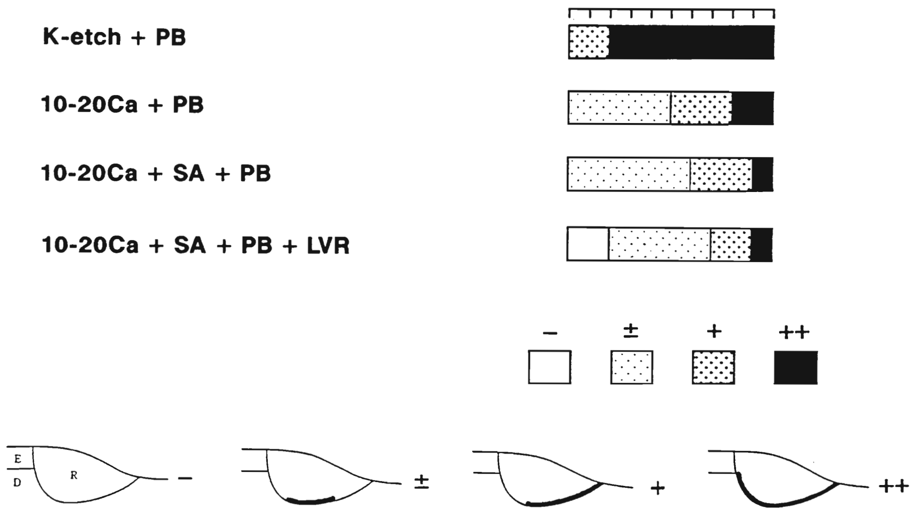

Fig. 5. Degree of Dye Penetration Into Boundaries Between Cervical Cavity Walls and Inserted Resins (Longitudinal Section)
It was evaluated and graded as either none(−) for excellent adaptation, slight (±) for gap formation along the cavity wall but no marginal leakage, moderate(+) for gap formation with marginal leakage, or severe(++) for gap formation all along the cavity wall with leakage.

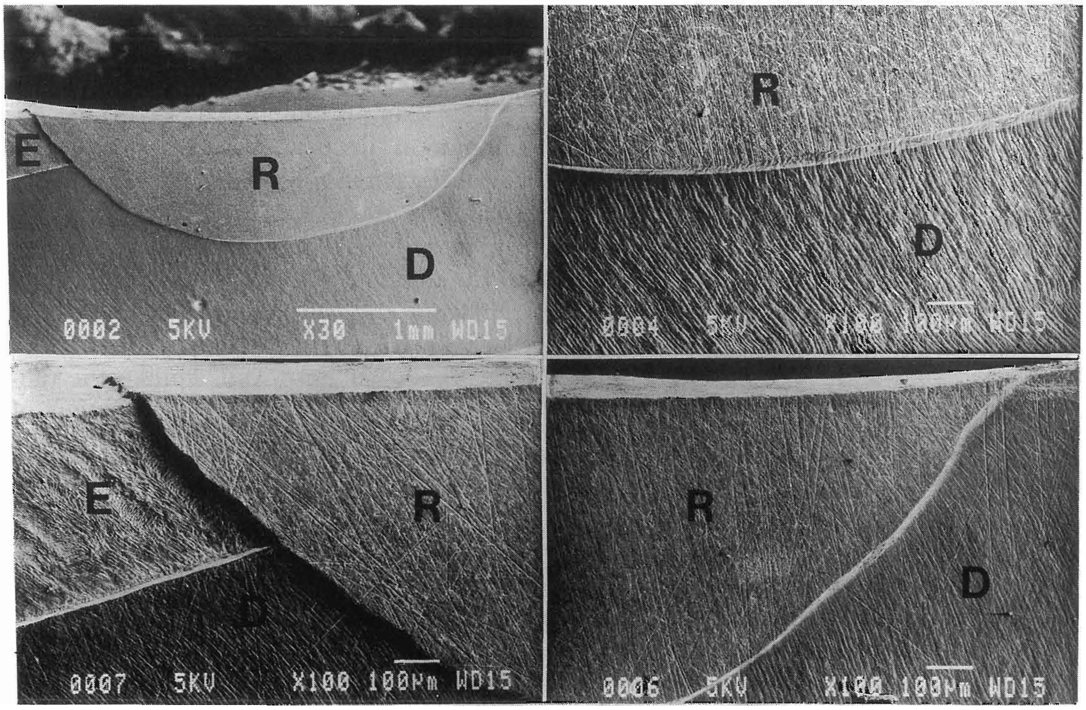

Fig. 6. SEM Images of Specimen Showing Excellent Adaptation of Composite Restoration to Cervical Cavity Wall (10–20Ca+SA+PB+LVR+PhBr)
The sectioned surface was slightly etched with 6N HCl for 20 seconds. E: Enamel, D: Dentin, R: Resin composite.

Table 4. pH Values of Tooth Surface Conditioning Agents

Conditioning agent	pH
K-etch	0.04
EDTA	7.40
10citric	1.48
10–3Fe	0.91
10–20Tan	1.21
10–20Mg	0.88
10–20K	1.08
10–20Cu	0.15
10–10Ca	0.74
10–20Ca	0.44
10–30Ca	0.25

ing agents

Table 4 presents the pH values of various tooth surface conditioners examined in Experiment I. K-etch or 10 citric showed the lowest or the highest pH, respectively, among the acids.

VI. Morphology of conditioned enamel and dentin

Different conditioning patterns of the enamel and dentin surfaces were observed (Fig. 7). When the tooth surface was etched with K-etch, clear honeycomb patterns were revealed on the enamel surface, and also most of the orifices of the dentinal tubules were opened. When 10–20Ca was used, microcrystalline structures were exhibited without the honeycomb patterns on the enamel surface, and the smear layer in some areas of the cut dentin surface was removed and the dentinal tubule apertures were exposed but not so opened. The conditioning effect of 10–20Ca was comparatively mild on the enamel rods, the intertubular dentin and the orifices of the tubules.

Discussion

For adhesive composite restoration, the

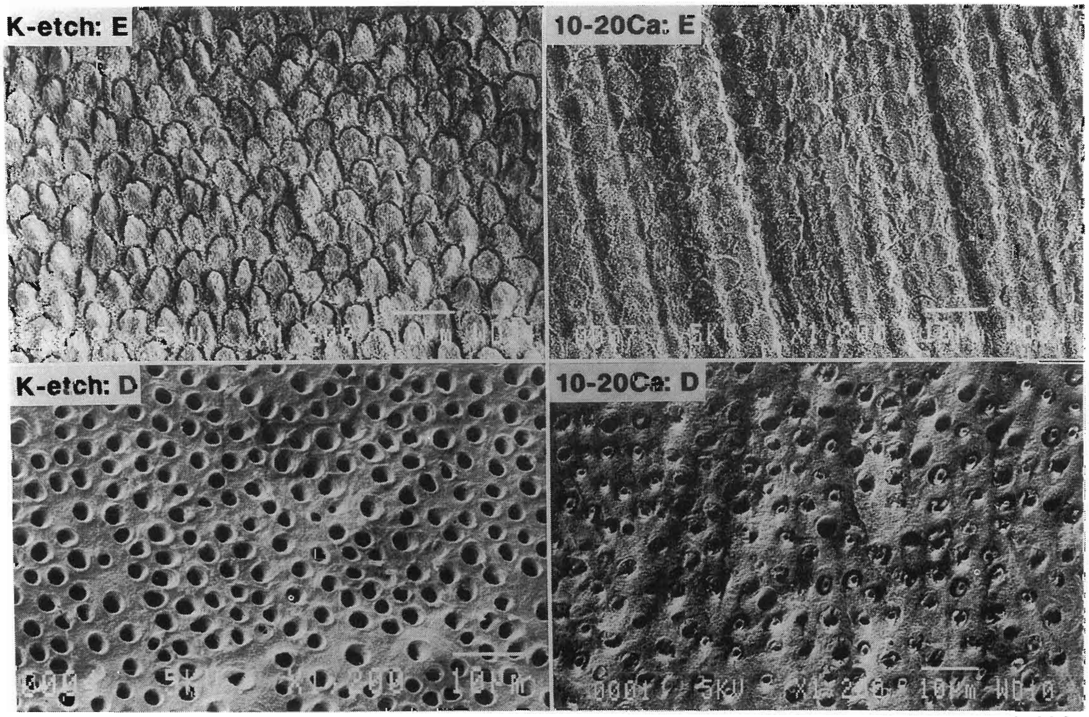

Fig. 7. SEM Images of Human Enamel(E) and Dentin(D) Surfaces Conditioned With K-etchant or 10–20Ca Solution

acid-etch technique has been accepted in the routine clinic. The application of an acid etchant and subsequently a bonding agent can obtain a good prognosticated resin restoration. However, the approximately 40% phosphoric acid solution is considered to cause certain damage to dentin, especially to the collagen fibers (Okamoto et al. [3]; Mizunuma [4]). It is also reported to affect the pulp, though temporarily, through the opened dentinal tubules (Fujitani [10]). Intensive researches have been carried out to develop a new tooth surface conditioning agent to substitute for a phosphoric acid etchant. Consequently, the 10–20Ca solution (10% citric acid containing 20% calcium chloride aqueous solution) has been devised. It increased the adhesion to dentin better than the other tooth surface conditioners examined and had the same bond strength as the phosphoric acid etchant (Figs. 3 and 4). These results showed that it is feasible to finish the surface treatment of both enamel and dentin by a single application of 10–20Ca. In addition, it offers less damage to the tooth substance in spite of its comparatively low pH (Table 4 and Fig. 7).

As for the effect of acidity of 10–10Ca, 10–20Ca or 10–30Ca, 10–20Ca and 10–30Ca were effective on the adhesion to enamel, which is considered to be due to their lower pH values than that of 10–10Ca. However, 10–30Ca was not so efficacious on the dentin bonding. It appears to offer more damage to dentin than 10–20Ca. On the other hand, as for the addition of calcium chloride, it is believed that calcium phosphate from the decalcified dentin might be facilitated to deposit on the exposed collagen and the conditioned surfaces since a small amount of the conditioners applied was ready to increase the pH value. The existence of calcium phosphate may also reinforce the decalcified dentin collagen and further promote the adhesion of the bonding resin to dentin. In the 10–10Ca solution, calcium phosphate would be less produced due to a weaker decalcification than that of 10–20Ca, while 10–30Ca possibly produces a more decalcified dentin layer but makes it weaker as compared with 10–20Ca. Thus, 10–20Ca seems to have an optimum concentration for exhibiting such a reaction. It is found in the similar way, for example, that the Fe ion in the 10–3Fe solution not only suppresses the degeneration of the dentin collagen but also reinforces the weak portion of both the dentin and resin (Kadoma et al. [11]). Therefore, the interaction between the effect of the acidity of the conditioner and the concentration of $CaCl_2$ may have an effect on the adhesion of the bonding resin.

The adhesive composite restorative technique, using 10–20Ca as the tooth surface conditioning agent, and SA and LVR as adhesion-promoting agents were employed in a standardized saucer type dentin cavities, where the behavior of polymerization shrinkage of the inserted restorative resin could be examined under a condition similar to the actual clinical application. Furthermore, this system was applied in the saucer cavities having enamel and dentin margins at the cervical area, where it is reported that the composite restoration can hardly obtain a good adaptation (Fujitani et al. [2]). While SA and LVR proved to be efficacious adhesion promoters, their effect seemed to vary slightly with the configuration of the adherend surface. In the tensile bond test, LVR was more effective at the flat surface than at the saucer cavity (Table 3) and both were efficient for the adaptation of the restoration at the cervical cavity (Fig. 5). Therefore, the adhesion and adaptability of the resin composites should not be evaluated through the results of the bond test using flat adherend surfaces alone.

Since the SA primer has a desensitizing effect on the hypersensitive dentin as well as on the adhesion-promoting one (Tagami et al. [12]), it is considered to be indispensable for the adhesive composite restoration of the cervical lesions with hypersensitivity. The mechanism of the adhesion-promoting effect of SA is not clarified, but it is believed that it impregnates into the decalcified layer of the conditioned dentin and probably rearranges the affected collagen fibers in the nearly original position. These effects may allow for a better penetration of the bonding resin and may accelerate the formation of a firm complex of the penetrated resin and dentin, resulting in a strong bond of the resin composites (Sugizaki [13]).

LVR also demonstrated the adhesion-promoting effect. Its mechanism is considered to be the following: LVR can sufficiently wet the adjoining surfaces between the bonding resin and the inserted restorative resin composite, so that the possible strains caused by the polymerization shrinkage of the composite may be relieved, leading to an improved adaptation.

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