Efficacy of Dentin Bonding to Cervical Defects

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The bonding efficacy of sclerotic dentin was determined by measuring the polymerization contraction gap width of a commercial light-activated resin composite in a cervical defect and by measuring the micro-Vicker's hardness at the dentin adhesive surface; morphological characteristics were observed using a scanning electron microscope in extracted human incisors and premolars. Contraction gap formation was completely prevented when the cavity wall was primed with 35 vol% glyceryl mono-methacrylate solution after 0.5 mol/L EDTA conditioning. The contraction gap width was significantly decreased when the resin composite was filled into the sclerotic dentin cavity even when priming was omitted. These results suggest that the sclerotic dentin, which is frequently observed in cervical defects, should be preserved as a substrate because it exhibits an effect of dentin priming and is suitable for bonding.

Key words : Dentin bonding, Cervical defect, Sclerotic dentin

INTRODUCTION

The efficacy of dentin bonding systems have been evaluated using intact dentin as a substrate in many studies¹⁻⁸⁾. From a clinical point of view, however, the dentin adjacent to the caries, the dentin of a cervical defect or the exposed root surface requires resin composite restoration. It has been clarified that the physical and chemical characteristics of this type of dentin is most likely degenerated compared with those of the normal dentin. Fusayama *et al.*^{9,10)} reported that the caries dentin was classified into two layers, the inner and outer layer, which were either re-calcified or not re-calcified physiologically. They invented the caries detector, acid red diluted in propylene glycol, by which the two layers were identified. In addition, the dentin tubules in the inner layer of the caries dentin were filled with debris suggested to be the hardness of this sclerotic dentin. Yoshiyama *et al.*¹¹⁾ reported that the dentin tubules in the cervical defect indicate a good correlation with the clinical performance of the sensitivity of the tooth and closed dentin tubules did not cause hypersensitivity. These two findings supported the hydro-dynamic theory by Bränström¹²⁾ who suggested that dentin sensitivity was caused by fluid flow in the dentin tubules. The permeability of the dentin is limited in the sclerotic dentin because the dentin tubules are closed by the debris of the cubic crystals of calcium phosphate. Such a difference in the physical structure between the sclerotic and

normal dentin possibly affects the efficacy of the dentin bonding system. Ogata *et al.*¹³⁾ measured the micro tensile bond strength to the sclerotic dentin forming a cervical defect; the bond strength was higher in the occlusal dentin wall compared with the apical dentin although both dentin cavity walls exhibited lower bond strength than the normal dentin. Furthermore, Nakajima *et al.*¹⁴⁾ recommended the conditioning of the caries affected dentin using a strong acid such as 40% phosphoric acid for 15 sec to form a hybrid in the high acid resistance dentin. Many studies concerning dentin bonding materials to the sclerotic dentin, claim that the efficacy of the dentin adhesive deteriorates to the sclerotic dentin because the reduced permeability interrupts hybrid layer formation¹⁵⁾.

The primary requirement for the dentin adhesive, however, is to maintain attachment between the unpolymerized resin composite paste and the dentin cavity wall until polymerization of the resin composite is completed. We previously reported that the contraction gap width increased when the functional monomer was omitted from the dentin bonding agent or the dentin cavity wall was decalcified using a strong acid such as 40% phosphoric acid and the contraction gap was formed between the dentin cavity wall and the resin composite¹⁶⁾. Therefore, it is suggested that it is most important to prevent contraction gap formation to maintain a high concentration both of the adhesive monomer and the calcium in the substrate at the adhesive interface and to eliminate the inhibitors of monomer polymerization such as oxygen in the air and water from the dentin tubules¹⁷⁾. Thus, separation of the resin composite paste from the dentin cavity wall is prevented and the entire polymerization contraction stress is released from the free surface of the resin composite filling.

The purpose of the present study was to investigate the efficacy of dentin adhesive to sclerotic dentin in a cervical defect.

MATERIALS AND METHODS

Contraction gap width measurement

Human incisors or pre-molars with cervical defects which were extracted for periodontal reasons were used. The surface of the defect was ground slightly using a round-shaped steel bur mounted on a low-speed cutting machine. The occlusal or incisal enamel cavity wall was etched with a commercial phosphoric acid gel (Etchant; 3M, MN, USA) (Table 1) for 30 sec and the cavity was rinsed and dried

Table 1 Experimental dentin bonding systems tested

Bonding System	Materials
Enamel etching	Etchant ¹
Dentin conditioner	neutralized 0.5 mol/L EDTA (pH 7.4)
Dentin primer	35 vol% of glyceryl mono-methacrylate (GM)
Dentin bonding agent	Clearfil Photo Bond ²

¹3M, St Paul, MN, USA, ²Kuraray, Osaka, Japan

completely. Then an experimental dentin conditioner, 0.5 mol/L EDTA neutralized to pH 7.4 was filled in the cavity for 60 sec followed by rinsing and drying. In half of the specimens, the cavity wall was primed with 35 vol% aqueous solution of glyceryl mono-methacrylate solution (GM) for 60 sec followed by air blasting to eliminate the primer completely, although in half of the specimens the cavity was not primed. A commercial dual-cured bonding agent (Clearfil Photo Bond; Kuraray, Tokyo, Japan) was applied in all the cavities and irradiated for 10 sec followed by a commercial light-activated resin composite (Silux Plus; 3M, MN, USA) filling and irradiated for 40 sec. After 10 min of storing the specimens in water at a room temperature of $24 \pm 1^\circ\text{C}$, a section through the center of the cavity of each tooth was prepared on wet carborundum paper. The cavity adaptation of the resin composite was inspected under a microscope and the maximum contraction gap width along the cavity margin was measured using a screw micrometer (Eyepiece Digital; Leitz, Wetzlar, Germany) mounted on the ocular lens of a light microscope (Orthoplan; Leitz, Wetzlar, Germany).

For the control group, V-shaped cavities with a cavo-surface angle of 135° were prepared using an experimental diamond point mounted on a high-speed cutting machine in carious-free extracted human incisors or pre-molars as shown in Fig. 1. The V-shaped cavity was restored with the dentin bonding system and resin composite using the same procedures as in the cervical defect. Gap width measurements on the section of the cavity were carried out using the same method as that in the cervical defect and V-shaped cavity, respectively; 40 in total were prepared. The results of wall-to-wall maximum contraction gap measurements were analyzed statistically with Fisher's PLSD test.

Vicker's Hardness measurement

Micro-Vicker's hardness both of the sclerotic and the normal dentin was determined by measurement at the hardness of the dentin adjacent to the resin cavity margin as presented in Fig. 2. The dentin hardness measurement was performed at five points

Fig. 1 Cervical defects of a tooth extracted for periodontal reasons, the V-shaped cavity (cavo-surface angle; 135°) and an experimental diamond point (Left to right).

Fig. 2 The micro-Vicker's hardness measurement. (A; Square mark with the micro-Vicker's hardness tester. MA : Measurement area was located less than $3\mu\text{m}$ from the cavity surface.)

for each specimen using the micro-hardness tester (MVK-E; Akashi, Tokyo, Japan), and the mean value of measurements was employed as the hardness of the specimen. The measurement of normal dentin and the dentin located in the cervical defect was analyzed statistically by Student's t-test.

Scanning Electron Microscopic observation

The microstructure of normal dentin and the cervical defects were observed with a scanning electron microscope (S-4700; Hitachi, Tokyo, Japan) after critical point drying and sputter coating the specimen with palladium and platinum. For the control group, the specimen in which dentin was etched with the 35% phosphoric acid gel for 60 sec was observed.

RESULTS

Maximum contraction gap measurement

When the dentin cavity wall was primed with GM, complete marginal adaptation was observed in all the specimens prepared regardless of the kind of dentin (Table 2). Without GM priming, cavity adaptation of the resin composite was not observed in all the specimens of the normal dentin whereas complete cavity adaptation was observed in seven specimens out of 10 with a cervical defect. The statistical analysis of difference was insignificant among the three tested groups (Fisher's PLSD test, $p < 0.05$).

Vicker's Hardness measurement

Dentin hardness of the dentin located in the cervical defect was significantly decreased compared with that of the dentin located in the artificially prepared V-

Table 2 Mean and standard deviations of the contraction gap width and Vicker's hardness

Bonding Substrates	Contraction gap (μm)		Vicker's Hardness
	GM Priming	Without Priming	
Normal Dentin	0 (10) *	2.34 ± 0.09 (0) **	59.53 ± 2.66 #
Cervical Defect	0 (10)	0.59 ± 1.18 (7) *	49.74 ± 1.08 ##

N=10

Mean $\pm$ SD of the maximum contraction gap width and the number of gap-free cases out of 10 are ().

*, The values of the contraction gap measurement joined by the line were not significantly different by Fisher's PLSD ($p < 0.05$).

#, The values of the Vicker's hardness measurement joined by the line were significantly different between two groups by Student's ttest ($p < 0.05$).

Fig.3 Scanning electron microscopic (SEM) observation of the normal dentin which was conditioned with 0.5 mol/L EDTA for 60 sec ($\times 5,000$). The bar represents $5\mu\text{m}$.

Fig.4 SEM observation of the sclerotic dentin which was conditioned with 0.5 mol/L EDTA for 60 sec ($\times 5,000$).

shaped cavity (Student's t-test, $p < 0.05$).

Scanning Electron Microscopic observation

In the case of the normal dentin conditioned by EDTA, the smear layer and debris on the dentin surface was completely removed although a dentinal plug and opening of the dentin tubules were observed (Fig.3). In the case of the sclerotic dentin condi-

Fig. 5 SEM observation of normal dentin tubules ($\times 500$). The bar represents $20\mu\text{m}$.

Fig. 6 High magnification of Figure 5 ($\times 5,000$). The bar represents $5\mu\text{m}$.

Fig. 7 SEM observation of sclerotic dentin tubules ($\times 500$). The bar represents $20\mu\text{m}$.

Fig. 8 High magnification of Figure 7 ($\times 5,000$). The bar represents $5\mu\text{m}$.

Fig.9 SEM observation at the adhesive interface of the sclerotic dentin which was cut through in a longitudinal section ($\times 5000$).

Fig.10 High magnification of Figure 8 ($\times 30,000$). The bar represents $1\mu\text{m}$.

tioned with EDTA, the debris was removed from the dentin surface and opening of the dentin tubules was observed (Fig.4). When the normal dentin was etched with the phosphoric acid, the dentin tubules were opened widely compared with the EDTA conditioning. The intertubular dentin was not decalcified compared with that of the normal dentin. The dentin tubules with cervical defects were closed by crystal-like substances (Figs.5-10).

DISCUSSION

The findings in this study were; 1) Complete marginal adaptation was observed in the sclerotic dentin of the cervical defects and intact dentin of the artificially prepared V-shaped cavity when the resin composite was filled into the cavity followed by the experimental dentin bonding system application, which was composed of EDTA conditioning, GM priming, Clearfil Photo Bond application. 2) In seven specimens with cervical defects, the gap formation was prevented even when the GM priming was omitted and cavity adaptation of the resin composite was clearly superior in the cervical defect compared with that in the artificially prepared V-shaped cavity. 3) In the SEM observation, the dentin tubules of a cervical defect were closed by debris with various crystal-like substances. 4) The sclerotic dentin showed an acid-resistance compared with the intact dentin, even though the Vicker's hardness of the dentin of the cervical defect was slightly reduced.

In the contraction gap measurements, such a discrepancy between the sclerotic and the intact dentin in the un-primed dentin cavity can be explained by the mechanism of GM priming.

As reported by Chigira *et al.*¹⁸⁾, monomer infiltration through the dentin is considered to be limited by the GM solution. Thus, the concentration of the functional monomer in both the hydrophilic and hydrophobic groups remained high at the adhesive interface. Furthermore, the fluid flow through the dentin tubules was also

interfered by GM priming; consequently, polymerization of the adhesive monomer was ensured. Such a high monomer concentration at the adhesive interface was detected as a high destiny zone by TEM observations.

As described above, in the contraction gap measurements, such a discrepancy between the sclerotic and the intact dentin in the un-primed dentin cavity can be explained by the mechanism of GM priming.

It was recognized that permeability through the dentin was significantly limited in the sclerotic dentin. Such a reduction of infiltration into the dentin was still limited by GM priming; consequently, the monomer concentration at the adhesive interface was kept sufficiently high to prevent contraction gap formation. For the normal dentin, monomer diffusion into the dentin was not prevented without GM priming and the monomer concentration was too reduced to prevent gap formation. As discussed above, the sclerotic dentin decreased monomer diffusion into the dentin and exhibited an effect such as a dentin priming.

In many studies, however, the mechanism of dentin adhesives is based on hybrid layer formation in the superficial substrate dentin as demonstrated by Nakabayashi^{19,20}. Therefore, acid etching the dentin with a dentin conditioner and monomer infiltration into the collagen-rich dentin layer has been considered essential to obtain bonding between the resin materials and the dentin. Furthermore, the effect of the 2-HEMA based dentin primer has been explained by promoting monomer diffusion into the etched dentin²¹. We previously reported that the priming effect of the 2-HEMA primer was clearly inferior to that of the GM primer probably because the 2-HEMA primer increased permeability of the monomer into the dentin and decreased the adhesive monomer concentration at the adhesive interface¹⁸.

In 1991, the mechanism of the dentin primer was explained by expanding the microspace among the dentin collagen network which was collapsed by dentin conditioning²². Subsequently, the adhesive monomer easily penetrated into the expanded microspace and formed the hybrid layer. In these studies, however, the efficacy of the dentin bonding system was estimated by measuring the tensile bond strength to the flat dentin surface. In addition, the fracture inside the resin composite or the dentin substrate was frequently observed even when the specimen was dumb-bell shaped to fracture at the adhesive interface. It is noteworthy that the clinical contraction gap was formed between the dentin cavity wall and the resin composite paste and never inside the resin composite or inside the dentin along the cavity margin. This clinical phenomenon suggested the conflict of estimating the efficacy of the dentin adhesive by the bond strength measurement. Hasegawa *et al.*²³ claimed that the mechanical properties of the resin composite significantly affected the bond strength of the dentin bonding systems because the higher tensile bond strength was measured by the resin composite that exhibited the higher tensile strength. It was consistent that the resin composite with higher mechanical properties did not always exhibit improved adaptation in the dentin cavity. It is noteworthy that the efficacy of the dentin bonding system did not deteriorate in the cervical defect even though the hardness of the dentin of the cervical defect was slightly reduced. We previously

reported that decalcification or the reduction of hardness on the dentin cavity wall increased the contraction gap width²⁴⁾. The results in the present study suggested that the closed dentin tubules or the limited permeability in the sclerotic dentin played a more important role for the establishment of marginal adaptation of the resin composite than the slight reduction in dentin hardness. We previously demonstrated that the dentin hardness was decreased by only 15% by the EDTA conditioning²⁴⁾. It is easily suggested that conditioning with a strong acid such as 40% phosphoric acid possibly deteriorated the marginal integrity of the resin composite in the sclerotic dentin cavity.

As discussed above, the dentin tubules of a cervical defect were closed by debris with various crystal-like substances. Such substances in the dentin tubules of the sclerotic dentin not only improved the resistance to acid, but the fluid flow through the dentin tubules from deep inside was also affected, and decreased the monomer diffusion into the dentin. Consequently, the monomer concentration at the adhesive interface was kept sufficiently high to prevent contraction gap formation. These effects occurred after the monomer had infiltrated through the dentin and are considered to be limited by the GM priming.

The efficacy of the experimental dentin bonding system to the sclerotic dentin was superior to that to the normal dentin especially when the dentin cavity was not primed with a GM solution. It was suggested that such improved bonding efficacy was based on the limited permeability of the sclerotic dentin which causes a high adhesive monomer concentration on the adhesive interface. Clinically, it is recommended that the sclerotic dentin in a cervical defect be conditioned with a mild conditioner such as EDTA and conservatively restored with a resin composite.

CONCLUSIONS

The sclerotic dentin in a cervical defect and root dentin is more suitable as a substrate for a dentin adhesive than the intact dentin. The sclerotic dentin shows effects such as a priming, and cavity adaptation of the resin composite does not deteriorate even when the cavity is not primed with a GM solution.

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