BONDING ABILITY OF ADHESIVE RESINS TO CARIES-AFFECTED AND CARIES-INFECTED DENTIN

RESUMO

As técnicas adesivas permitiram inovações nos tratamentos restauradores da atualidade. Este estudo investigou a qualidade de união de diferentes sistemas adesivos ao substrato dentinário afetado e infectado por cárie, através de testes de microtração e microscopia eletrônica de transmissão e varredura (MET). Os resultados sugerem que a resina adesiva pôde infiltrar na dentina infectada e envolver as bactérias na camada híbrida. Esse conceito de controle da cárie foi denominado de “Restauração-Selante Modificada” (RSM). Por outro lado, a empresa Kuraray Med. Inc. (Japão) desenvolveu um sistema adesivo com propriedades anti-bacterianas (ABF), o qual é comercializado nos EUA como Protect Bond. Para avaliar a efetividade do sistema ABF sobre cáries radiculares, empregou-se testes de resistência adesiva e análise por microscopia eletrônica. O sistema ABF foi capaz de formar uma estrutura interfacial semelhante à camada híbrida, através da infiltração na superfície da dentina cariada radicular e os valores de resistência de união foram em média de 23 MPa. Os achados sugerem que a combinação da técnica RSM com o sistema ABF parece ser uma alternativa para o emprego da terapia de mínima invasão em cáries radiculares.

Unitermos: Restauração selante modificada; Sistema adesivo anti-bacteriano; Dentina infectada por cárie; Resistência adesiva à microtração.

INTRODUCTION

Recently, new concepts of treatments for dentin caries by use of adhesive resins and glass-ionomer cements have been proposed. Dramatic changes of the roles of adhesive dentistry have occurred from the end of the 20th century. From the mainly reparative dentistry of the 20th century, contemporary dentistry shifts towards a minimal intervention (MI) approach, and contemporary operative treatment incorporates the MI philosophy in cavity design (Peters...
BONDING ABILITY OF ADHESIVE RESINS TO CARIES-AFFECTED AND CARIES-INFECTED DENTIN

Resin adhesion to caries-affected and caries-infected dentin

However, so as to establish the ultra-conservative therapy of caries-infected dentin, we should examine the resin adhesion to caries-affected and caries-infected dentin. Recently, a new bond strength testing procedure has been developed which permits the measurement of small (ca. 1mm²) cross-sectional bonded areas. It has been called the microtensile bond strength test (μTBS) (Sano, et al.14, 1994), and permits the testing of irregular surfaces such as sclerotic and carious dentin. By using μTBS, we were able to evaluate the adhesion of contemporary resin systems to cervical sclerotic dentin (Yoshiyama, et al.20, 1996; Tay, et al.24, 2000). We have also evaluated the interfacial morphology of two bonding system (Single Bond, 3M and Fluoro Bond, Shofu) to caries-infected dentin, coupled with the measurement of μTBS (Yoshiyama, et al.22, 2000), and reported that resin bonds made to caries-infected dentin were lower than to normal dentin using either self-etching primer or conventional adhesive systems. Nakajima, et al.23 (1999) could demonstrate that very high bond strengths could be obtained on caries-affected dentin if the dentin is etched with 35% phosphoric acid and the moist bonding technique is employed.

To establish a new dentin caries treatment involving embedding residual bacteria with adhesive resins, we need to evaluate the adhesive properties of bonding resins to caries-infected dentin. Therefore, we have clarified the adhesive property of a self-etching/self-priming system (ABF, Kuraray Medical Inc., Tokyo, Japan)(ABF) to normal, caries-affected and caries-infected dentin using μTBS and transmission electron microscopy (TEM)(Yoshiyama, et al.20, 2002). ABF contains 10-methacryloxyloxy methacrylate (MDP) as an adhesive monomer. The occlusal surfaces of extracted human third molars with coronal dentin caries were ground perpendicular to the long axis of the tooth to expose a flat surface where the carious lesion was surrounded by normal dentin (Figure 2).

The results of the μTBS to normal, caries-affected and caries-infected dentin are shown in Table 1. The μTBS of ABF to normal dentin was about 44.9 ± 14.6 MPa, while the bond strength of ABF to caries-affected dentin was significantly lower (25.5 ± 5.0 MPa) than to normal dentin (p<0.05). The μTBS of ABF to caries-infected dentin was only 15.2 ± 3.6 MPa which was significantly lower (p<0.05) than ABF bonds to caries-affected dentin.

TEM observation of the ultrathin sections of the interface from resin-bonded normal dentin showed a thin hybrid layer (less than 1.0 µm) that was formed by ABF in the dentin (Figure 3). Higher magnification TEM revealed that smear

![Figure 1](image)

**FIGURE 1**- The concept of Modified Sealed Restoration (MSR). Carious bacteria may be embedded with hybrid layers.

<table>
<thead>
<tr>
<th>TABLE 1</th>
<th>Tensile bond strength of ABF (Protect Bond) in normal, caries-affected and caries-infected dentin</th>
</tr>
</thead>
<tbody>
<tr>
<td>Normal dentin</td>
<td>44.9 ± 14.6*</td>
</tr>
<tr>
<td>Caries-affected dentin</td>
<td>25.5 ± 5.0*</td>
</tr>
<tr>
<td>Caries-infected dentin</td>
<td>15.2 ± 3.6*</td>
</tr>
</tbody>
</table>

Values are χ ± S.D. in MPa N=7 in each group. Different superscript lowers ease letters identify statistically significant differences (p<0.05) using Student-Keuls test.
layer was completely dissolved. TEM observation of the interfaces of bonded caries-affected dentin showed a much thicker hybrid layer (6-8 µm) than was seen in normal dentin. A gradient of resin could also be identified from the surface of the hybrid layer downward, with the base of the hybrid layer poorly identified (Figure 4). Bacteria were rarely observed within the dentinal tubules or on dentin surfaces of the bonded caries-affected dentin. TEM observation of the resin-bonded interfaces of deep caries-infected dentin revealed much substrate variation. In the simplest form, exceptionally thick hybrid layers (ca. 25-30 µm) could be identified, along with the entrapment of bacteria within some dentinal tubules (Figure 5). Similar to the caries-affected dentin, a gradient of resin could be observed in these thick hybrid layers. Moreover, parts of the dentin surface were not completely wetted by the filled, bonding resin component of the adhesive systems. Other sections revealed thick, erratic hybrid layers that consisted of a superficial layer of completely disorganized and denatured intertubular and peritubular dentin, and an underlying layer of intact hybridized dentin (Figure 6). Bacteria were also trapped within disfigured dentinal tubules.

**Bonding of antibacterial fluoride-releasing adhesive system (ABF) to root carious dentin**

Root caries is an important dental disease, especially because the number of elderly dentate people is increasing (Simons, et al., 1999). Root surfaces become exposed to oral environment due to gum recession. This gum recession increases with age and the risk of root caries is high because of the change in saliva flow rate, the change in cleaning effectiveness and the change in type of bacteria in older patients. Thus, diagnose and rapid treatment of root caries is required for these patients.

Treatment strategy for root caries include the repair of the decay occurred by the carious progression and the use of antibacterial agents or fluorides for inhibition of the
root caries (Doi, et al.1, 2004). To expose the normal coronal and root dentin, but also caries-affected and caries-infected root dentin in human carious teeth so as to establish remineralization of the dentin around the restoration (Itota, et al.7, 2002). In addition, it has been reported that MDPB on the surface of a resin-based material showed a bacteriostatic effect and anti-adhesion property against oral streptococci. Thus, the use of this adhesive system for the root carious treatment may inhibit secondary caries occurrence by invasion of bacteria into the resin-dentin interface and adhesion of bacteria on the restorative surface. Fluoride ions released from the adhesive system may also inhibit secondary caries by the restorative surface. Fluoride-releasing adhesive system (ABF) was developed by Kuraray Medical Inc. (Tokyo, Japan). The self-etching primer in this system contains the antibacterial monomer, methacryloylpyridinium bromide (MDPB) synthesized by combining an antibacterial agent and methacryloyl group, and the adhesive contains sodium fluoride. Imazato, et al.4,5 (1994, 2001) have reported that MDPB on the surface of a resin-based material showed a bacteriostatic effect and anti-adhesion property against oral streptococci. Thus, the use of this adhesive system for the root carious treatment may inhibit secondary caries occurrence by invasion of bacteria into the root caries treatment. Moreover, even in normal dentin, the bond strength to normal dentin differs in crown and root sites (Yoshiyama, et al.17, 1996; Yoshiyama, et al.18, 1998).

Recently, an antibacterial primer in this system contains the antibacterial monomer, methacryloylpyridinium bromide (MDPB) synthesized by combining an antibacterial agent and methacryloyl group, and the adhesive contains sodium fluoride. Imazato, et al.4,5 (1994, 2001) have reported that MDPB on the surface of a resin-based material showed a bacteriostatic effect and anti-adhesion property against oral streptococci. Thus, the use of this adhesive system for the root carious treatment may inhibit secondary caries occurrence by invasion of bacteria into the resin-dentin interface and adhesion of bacteria on the restorative surface. Fluoride ions released from the adhesive system may also inhibit secondary caries by the remineralization of the dentin around the restoration (Itota, et al.6, 2001; Itota, et al.7, 2002). In addition, it has been reported that this adhesive system had a good adhesion to normal dentin (Imazato, et al.3, 1997).

However, the adhesion of this system to dentin in carious teeth is unknown regardless of the use of this adhesive for the carious treatment. Moreover, even in normal dentin, the bond strength to normal dentin differs in crown and root sites (Yoshiyama, et al.17, 1996; Yoshiyama, et al.18, 1998).

We have evaluated the μTBS of ABF to not only normal coronal and root dentin, but also caries-affected and caries-infected root dentin in human carious teeth so as to establish the modified sealed restoration (MSR) combined with ABF to root caries (Doi, et al.1, 2004). To expose the normal coronal, normal root, caries-affected root and caries-infected root dentin, the surfaces of human premolars with root carious lesions were roughly polished using diamond saw (Morita Corp., Tokyo, Japan) with a coolant and then polished with a #600-grit SiC paper under wet condition as shown in Figure 7. The exposed surfaces were distinguished into normal coronal dentin area without dye, normal root dentin area without dye, caries-affected root dentin area with the slightly weak dye or caries-infected root dentin area with the strong dye by caries detector (Kuraray Medical Inc., Tokyo, Japan). The surfaces were then treated with ABF according to manufacturer’s instruction, and then covered with resin composite (Clearfil AP-X, Kuraray Medical Inc., Tokyo, Japan) to provide sufficient bulk for μTBS test. After immersion in 37°C water for 24h, specimens were serially sectioned into multiple slabs about 0.8 mm thick. Each slab was distinguished into normal coronal, normal root, caries-affected root or caries-infected root dentin specimens and then trimmed for μTBS testing and SEM observation.

Table 2 shows the results of the μTBS of ABF to the various types of dentin. In normal coronal dentin, ABF could produce quite high bond strength (43.2 MPa), and in normal root dentin, the bond strength of ABF was 33.7 MPa. However, in caries-affected and caries-infected root dentin, the bond strength of ABF were significantly lower than those to normal coronal and root dentin. SEM micrographs revealed that there was the hybrid or hybrid-like layer at the resin-dentin interfaces in all specimens as shown in Figure 8. However, the hybrid-like layers formed in caries-affected root and caries-infected root dentin showed porous.

![FIGURE 4-TEM of the interface of ABF bonded to caries-affected dentin. Although the hybrid layer (H) was about 3 μm thick, the underlying undemineralized dentin (U) was highly porous (arrow head). The dentinal tubule was covered with a smear plug (SP), and was partially filled with large caries crystals (arrows).](image1)

![FIGURE 5-Bonding of the ABF system to caries-infected dentin showing the pathological bonding substrate. Stained, undemineralized section. In its simplest form, the hybrid layer (between arrows) consisted of a thin layer of carious-infected dentin (CI) that contained colonized bacteria (arrowhead) that was connected with a layer or caries-affected dentin (U) that was highly porous. The dentinal tubules were completely obliterated with minerals that accounted for the reduction in the permeability in carious dentin.](image2)

![FIGURE 6-Stained, undemineralized section. A thick layer of caries-infected dentin was present, containing loose dentin fragments (L) and bacteria (B). The extent of the hybrid layer could not be clearly discerned. It is likely that the self-etching primer did not reach and infiltrate beyond the layer of caries-infected dentin and did not form a hybrid layer into the underlying caries-affected dentin (CA). A gap (asterisk) was present between the unfilled primer caries-infected dentin. This gap was subsequently filled up with the more electron-lucent epoxy resin that was used for laboratory specimen preparation.](image3)
structures compared to normal coronal and root dentin.

The results of our latest study indicated that the µTBS of ABF to caries-affected root and caries-infected root dentin were significantly lower than those to normal coronal and root dentin. These findings agree with the previous reports that the bond strengths of the current commercial self-etching adhesive systems to caries-affected or caries-infected dentin were significantly lower than that to normal dentin (Yoshiyama, et al.22, 2000; Kimochi, et al.9, 1999). However, the bond strength of ABF to caries-affected dentin was higher than that of other self-etching adhesive systems reported by several authors (Yoshiyama, et al.22, 2000; Nakajima, et al.12, 1995). This result suggests that the application of ABF to root caries-affected dentin can contribute to the effectiveness of the restoration. Moreover, ABF adhesive system has an antibacterial effect and fluoride-releasing property. Imazato, et al.5 (2001) have reported that the incorporation of MDPB into dentin primer could be beneficial for eliminating the residual bacteria in cavities. This report suggests that the use of ABF can eliminate the residual bacteria in caries-affected dentin and caries-infected dentin.

CONCLUSION

Recent studies have indicated that the bond strengths to caries-affected and caries-infected dentin were lower than that to normal coronal and root dentin even when ABF was used for root carious treatment. However, the antibacterial and fluoride-releasing properties of ABF can be expected to inhibit secondary caries by elimination of the residual bacteria and the invasion of bacteria, and by remineralization of the dentin around the restoration. Further investigations will be necessary for the establishment of the root carious treatment strategy by MSR combined with ABF.

TABLE 2- Microtensile bond strength of the ABF to dentin in human root carious teeth

<table>
<thead>
<tr>
<th>Dentin</th>
<th>Means ± s.d.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coronal normal dentin</td>
<td>43.2 ± 8.1a</td>
</tr>
<tr>
<td>Root normal dentin</td>
<td>33.7 ± 6.7b</td>
</tr>
<tr>
<td>Caries-affected root dentin</td>
<td>23.0 ± 15.3c</td>
</tr>
<tr>
<td>Caries-infected root dentin</td>
<td>14.5 ± 5.5d</td>
</tr>
</tbody>
</table>

Distinct superscript letter indicate significantly different (ANOVA and Fisher’s PLSD, p<0.05)

REFERENCES


