Original paper

Characterization of Enamel and Dentin Surfaces after Removal of Temporary Cement — Effect of Temporary Cement on Tensile Bond Strength of Resin Luting Cement —

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This study was performed to investigate the effect of temporary sealing with cement on the tensile bond strength of resin luting cement to tooth substrate. Five temporary cements and five resin luting cements were used. Six hundred bovine incisor teeth were randomly divided between each group. Effect of temporary sealing on the tensile bond strength of resin luting cement varied in accordance with the temporary cement used. Temporary sealing with both the eugenol-containing and the eugenol-free temporary cements decreased the tensile bond strength of resin luting cement. The tensile bond strength with one of the resin luting cements was most stable on both enamel and dentin pretreated with all temporary cements tested.

Key words: Temporary cement, Resin luting cement, Tensile bond strength

INTRODUCTION

Progress in adhesive dentistry has been accompanied by the choice of resin luting cement for metal, ceramic, and composite resin restorations. As these procedures require multiple appointments, the use of temporary cement is indispensable for sealing the cavities or luting provisional restorations. Final restorations are then placed after removal of temporary cement.

Many temporary cements contain eugenol, which is a concern when the resin luting cement is applied, because eugenol has been shown to have harmful effects on the physical properties of resin. Eugenol decreases transverse bend strength\(^1\), surface hardness\(^2\), and shear bond strength of resin to resin\(^3\), and increases surface roughness\(^4\) and surface discoloration\(^5\). Eugenol also inhibits resin polymerization\(^4,6-9\). Thus, use of temporary cements containing eugenol are not recommended prior to resin luting cement because residual eugenol may inhibit the physical properties of the thin layer of the resin luting cement. Incomplete physical properitites of the resin would decrease the bond strength of resin luting cement to tooth substrate and increase microleakage between resin and substrate.

The purpose of this investigation was to determine the effect of temporary sealing with such cements on the tensile bond strength of resin luting cement to tooth substrate.
MATERIALS AND METHOD

Six hundred bovine mandibular incisor teeth were used within 12 hours of extraction. A flat area was prepared on the labial surface of the crown to expose an area of enamel or dentin surface. The surface was polished wet with 600 grit through 200 and 400 grit silicon carbide paper.

The temporary cements listed in Table 1 were mixed and applied to the flattened labial surface following each manufacturer's instruction. After the temporary cement was set, the specimens were stored in water at 37°C. After one week, the temporary cement was mechanically removed with a dental probe, and unaided visual inspection indicated that the surface was free of cement. Then, the specimens were washed with water and air-dried. Controls consisted of a flattened labial surface without temporary sealing.

Composite resin was placed in the stainless steel mold (diameter 4mm, height 3.5mm) as the composite inlay (Fig. 1). The top surface of composite resin was covered with a plastic matrix and a glass plate was gently pressed on top. Composite resin was first light-cured for 60 seconds with firm contact ensured between the matrix and the exit window of the visible light unit, and secondly polymerized in the visible light and heat unit for 10 minutes.

The resin luting cements listed in Table 2 were used. The manufacturers' recommendations for conditioning on the enamel or dentin surfaces were followed for each product. Composite inlay was cemented onto the enamel or dentin surfaces with each resin luting cement. The exit window of visible light unit was then positioned on the top surface of the composite inlay.

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**Table 1** Temporary cements

<table>
<thead>
<tr>
<th>Brand name</th>
<th>Code</th>
<th>Batch No.</th>
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<tbody>
<tr>
<td>Eugedain®️1</td>
<td>EUG</td>
<td>Powder</td>
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<td></td>
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<td>40250</td>
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<td>Liquid</td>
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<td>30237</td>
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<tr>
<td>Propac®️2</td>
<td>PRO</td>
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<td>220591</td>
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<td></td>
<td></td>
<td>Accelerator 220591</td>
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<td></td>
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<td>Accelerator 010587B</td>
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<td>Freegenol Temporary Cement®️4</td>
<td>FTC</td>
<td>Base</td>
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<tr>
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<td>061091</td>
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<tr>
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<td>Accelerator 061091</td>
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<td>HTC</td>
<td>Powder</td>
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<td>018843</td>
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<td>Liquid</td>
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®️1 Showa Yakuhinkakou Co., Tokyo, Japan.
®️2 GC Co., Tokyo, Japan.
®️3 Coe Laboratories Inc., Chicago, USA.
®️4 GC Co., Tokyo, Japan.
®️5 Shofu Inc., Kyoto, Japan.
EUG and PRO were eugenol-containing temporary cement.
NTC, FTC, and HTC were eugenol-free temporary cement.

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# Occlusin, GC Co., Tokyo, Japan
## Luxor, ICI Co., Cheshire, England
$ α-Light, J. Morita Co., Tokyo, Japan
Table 2  Resin luting cements

<table>
<thead>
<tr>
<th>Brand name</th>
<th>Code</th>
<th>Batch No.</th>
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<tr>
<td>Lite-Fil CR Inlay Cement</td>
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<td>029003</td>
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<tr>
<td>Palfique Inlay Cement</td>
<td>PI</td>
<td>80003M</td>
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<tr>
<td>Adhesive Cement</td>
<td>AD</td>
<td>036</td>
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<tr>
<td>Clearfil CR Inlay Cement</td>
<td>CF</td>
<td>11124</td>
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<tr>
<td>Super-Bond C&amp;B</td>
<td>SB</td>
<td>00603</td>
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</table>

@1 Shofu Inc., Kyoto, Japan.
@2 Tokuyama Soda Co., Yamaguchi, Japan.
@3 Kulzer GmbH, Wehrheim, Germany.
@4 Kuraray Co., Tokyo, Japan.
@5 Sun Medical Co., Kyoto, Japan.

The resin luting cement was light-cured through the composite inlay for 60 seconds. All specimens were left standing at room temperature for 5 minutes and stored in water at 37°C for 24 hours.

The tensile bond strength were measured with a universal testing machine at a cross-head speed of 0.5 mm/minute (Fig. 1). Ten specimens were measured for each testing group.

The data were statistically analyzed with a computer. All data from each group showed a normal distribution. These data did not include any abnormal measurements. After testing for homogeneity, all data were analyzed by t-test for significant differences between control and each temporary cement group (p<0.05).

Fig. 1  Scheme of tensile bond strength test.
RESULTS

Table 3 (on enamel) and 4 (on dentin) show the tensile bond strengths between composite resin inlay and enamel or dentin surfaces with resin luting cement.

On enamel, EUG did not decrease the tensile bond strengths of LF, PI, AD or CF, while it increased that of SB. PRO decreased the tensile bond strengths of LF, PI or CF. NTC decreased the tensile bond strength of AD, while it increased that of CF. FTC decreased the tensile bond strengths of LF, PI, AD or CF. HTC decreased the tensile bond strengths of LF, AD or CF.

LF, PI, AD or CF did not bond to dentin following pretreatment with any temporary cement. EUG, FTC or HTC decreased the tensile bond strength of SB on dentin.

DISCUSSION

It is well known that the length of storage time and the storage conditions of extracted teeth affect their characteristics9-12). To avoid these factors, we used freshly extracted teeth in this investigation.

The fact that eugenol inhibits resin polymerization is well documented4,6-8). This explains the results reported by Hansen and Asmussen13) who showed that the contraction gap was markedly increased in cavities previously filled with ZOE. However, Schwartz et al.14) reported that shear bond strength was unaffected by temporary cement pretreatment, provided that the enamel surface was cleaned with pumice and etched with 37% phosphoric acid.

Table 3  Tensile bond strength on enamel surfaces

<table>
<thead>
<tr>
<th></th>
<th>LF</th>
<th>PI</th>
<th>AD</th>
<th>CF</th>
<th>SB</th>
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<tr>
<td>Control</td>
<td>9.6±2.1</td>
<td>7.9±2.9</td>
<td>7.9±2.5</td>
<td>10.8±1.8</td>
<td>11.4±1.7</td>
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<tr>
<td>EUG</td>
<td>7.9±5.0*</td>
<td>7.6±5.4*</td>
<td>8.7±3.2*</td>
<td>13.5±4.3*</td>
<td>14.0±3.1</td>
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<tr>
<td>PRO</td>
<td>6.1±1.9</td>
<td>5.6±1.7</td>
<td>7.2±2.5*</td>
<td>7.3±2.3</td>
<td>11.3±1.7*</td>
</tr>
<tr>
<td>NTC</td>
<td>9.2±3.9*</td>
<td>8.0±2.9*</td>
<td>5.3±1.4</td>
<td>14.1±1.7</td>
<td>12.7±2.7*</td>
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<td>FTC</td>
<td>5.6±1.5</td>
<td>5.6±1.3</td>
<td>5.2±1.1</td>
<td>7.5±2.1</td>
<td>10.1±1.4*</td>
</tr>
<tr>
<td>HTC</td>
<td>6.1±1.8</td>
<td>5.7±1.8*</td>
<td>5.2±1.3</td>
<td>6.8±2.6</td>
<td>10.6±2.1*</td>
</tr>
</tbody>
</table>

* No significant differences between control and temporary cement pretreated groups. (MPa, Mean ± S. D., n=10).

Table 4  Tensile bond strength on dentin surfaces

<table>
<thead>
<tr>
<th></th>
<th>LF</th>
<th>PI</th>
<th>AD</th>
<th>CF</th>
<th>SB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>2.7±2.0</td>
<td>3.5±2.2</td>
<td>2.4±1.3</td>
<td>3.7±1.7</td>
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<tr>
<td>EUG</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>9.3±2.7</td>
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<tr>
<td>PRO</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>10.1±2.1*</td>
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<td>—</td>
<td>—</td>
<td>—</td>
<td>12.0±2.2*</td>
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<tr>
<td>FTC</td>
<td>—</td>
<td>—</td>
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<td>—</td>
<td>9.3±1.7</td>
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<tr>
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<td>—</td>
<td>—</td>
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<td>9.7±2.2</td>
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</tbody>
</table>

* No significant differences between control and temporary cement pretreated groups. (MPa, Mean ± S. D., n=10).

— Did not bond to tooth substrate.
acid. Lacy et al.\textsuperscript{15} also reported that there were no significant differences in shear bond strength between untreated dentin and that pretreated with either eugenol-containing or eugenol-free temporary cements. The results of present investigation indicated that temporary sealing with cement, regardless of whether it contains eugenol or not, decreased the tensile bond strength of resin luting cement on both enamel and dentin surfaces. The reason for this is not fully understood at present. Not only EUG and PRO which contain eugenol but also NTC, FTC and HTC which do not contain eugenol decreased the tensile bond strength of resin luting cement, despite the purported adverse effects of eugenol on resin. These findings imply that factors other than the presence of eugenol were responsible for the reduction in tensile bond strength of resin luting cement. These results are in agreement with those reported by Woody and Davis\textsuperscript{16} who demonstrated that there was no significant differences in leakage between groups treated with eugenol-containing and the eugenol-free temporary cements. Therefore, the bonding of final restorations to teeth with resin luting cement could be adversely affected by temporary sealing with any cement.

There were some interesting exceptions; on enamel, EUG increased the tensile bond strength of SB, and NTC had a similar effect on that of CF. However, the cause of these exceptions is not completely understood.

LF, PI, AD or CF did not bond to dentin pretreated with any of the temporary cements used. In controls, the tensile bond strengths of these resin luting cements to dentin were markedly less than those to enamel. The findings of this and of our earlier investigation in which SEM examination showed a gap visible at the interface between composite and dentin which had been a previously treated with temporary cement suggested that the effect of temporary sealing with the cement on dentin was greater than that on enamel\textsuperscript{17}. Therefore, the effect of temporary sealing with the cement may inhibit the adhesion of resin luting cements with low tensile bond strengths to dentin. Although the tensile bond strength of SB to dentin pretreated with EUG, FTC or HTC was reduced relative to that to untreated dentin, it did form stable bonds to both enamel and dentin.

A previous investigation\textsuperscript{18} indicated that mechanical removal with a dental probe did not completely remove all of the temporary cement from both enamel and dentin surfaces, and moreover that etching with 37\% phosphoric acid effectively removed the temporary cement that remained on enamel surfaces after mechanical removed but that the etchant was not effective on dentin surfaces. The resin luting cements used here required various etching treatments before cementing; LF and PI required etching with 37\% phosphoric acid only for enamel, AD and CF required etching with 37\% phosphoric acid for both enamel and dentin, and SB required etching for both enamel and dentin with 10–3 solutions (an aqueous mixture of 10\% citric acid and 3\% ferric chloride which has a weaker effect on tooth structure than 37\% phosphoric acid). Although 37\% phosphoric acid was used as an etchant for LF, PI, AD and CF on enamel surfaces, the tensile bond strength of some groups were decreased by temporary sealing with the cement. The temporary cement was usually used provisionally for one week. However, even after temporary cement removal with a dental probe, some traces remained on tooth surfaces\textsuperscript{16–18}, and some of the ingredients probably penetrated the tooth surface\textsuperscript{19–21}. This residual cement and penetration may have changed the characteristics of tooth structure, for example, contact angle\textsuperscript{18} and dentin permeability\textsuperscript{22} and this
possibility requires further investigation.

These results showed clearly that temporary cement, whether it contains eugenol or not, should not be used on surfaces to later be cemented with resin luting cement and bonding systems. If temporary sealing with cement is necessary, SB which was most stable on both enamel and dentin should be used as the resin luting cement.

CONCLUSIONS

This study showed the temporary sealing decreased the tensile bond strength of resin luting cement. The effects of temporary sealing on the tensile bond strength of resin luting cement varied in accordance with the temporary cement used. Temporary sealing with both eugenol-containing and the eugenol-free temporary cements decreased the tensile bond strength of resin luting cements. The tensile bond strength with SB was most stable on both enamel and dentin pretreated with all temporary cements tested.

ACKNOWLEDGMENT

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REFERENCES

仮封材除去後のエナメル質および象牙質の特性
—仮封材がレジンセメントの歯質接着性に与える影響—
寺田林太郎，中島薫，小原雅彦，久保田稔
岩手医科大学歯学部歯科保存学第一講座

この研究は，仮封処置がレジンセメントの歯質接着性に与える影響を検討したものである。5種の仮封材と5種のレジンセメントを実験に用いた。600本の乳歯を被検歯として使用した。仮封処置がレジンセメントの歯質接着性に与える影響は，仮封材により異なっていた。ユージノール系，非ユージノール系のいずれの仮封材も，レジンセメントの歯質接着性を減少させていた。いずれの仮封材を使用しても，エナメル質および象牙質において4-META系レジンセメントが安定した歯質接着性を示していた。

MMA-TBB系レジンによるエナメル質の接着における酸塩含有プライマーの効果
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長崎大学歯学部歯科補綴学第一講座
東京医科歯科大学医器研究所生体機能材料部門

TBB系レジンセメントの接着耐久性改善の試みとして，エナメル質とステンレス鋼の接着におよぼす酸塩含有プライマーの効果を検討した。乳歯エナメル質を10％リン酸でエッチング後，鋼塗を含むアセトンプライマーで処理し，TBB系レジンで接着した。水中での熱サイクル試験を行った後，引張り接着強さを測定した。市販のコンポジット型レジンセメントと比較した。鋼塗の塗布はMMA-TBBレジンによる接着強さの耐久性を全般に向上させたが，2メタクリロイルオキシエチルカハ酸塩を含むプライマーを用いて，4-META/MMA-TBBレジンで接着した場合に最もよい耐久性が得られ，市販品よりもすぐれていた。熱サイクル2,000回後でも接着強さの平均値21MPa，最低値12MPaが維持されていた。

陶材中元素の酸化チタンへの拡散
塚隆夫，今政幸，大川昭治，浅岡憲三
徳島大学歯学部歯科理工学講座
北海道大学歯学部歯科理工学講座

3種類の市販チタン用焼結陶材中の元素が加熱中に酸化チタンへどのように拡散するかを調べた。円板状に形成した陶材上にチタンを3種類の厚さに真空蒸着し，大気中で加熱した。蒸着厚は，表面アラサ計およびエリプソメーターによって測定した。加熱によってチタンは酸化し，薄い酸化チタンが陶材を覆った。この表面およびチタンを蒸着していない陶材の表面をX線光電子分光で解析した。その結果，チタン用焼結陶材はホウ酸の量が従来型よりも多かった。これは，チタンに合わせて熱膨張係数を小さくするためと考えられる。加熱後の酸化チタン中のチタンは3種と4種の間の価数であり，ナトリウム，カリウム，バリウムのみが加熱中に酸化チタンに拡散した。これらの元素は酸化チタン中に混合しているのではなく，チタンと複合酸化物を形成していた。これらの元素の拡散は陶材とチタンとの結合に係していると考えられる。