Optimum Concentration and Application Time of 1, 6-hexanediol as a Dentin Primer

Mir Ayubur RAHMAN, Chihiro TANI, Kazuo ITOH, Sadao WAKUMOTO and Hisashi HISAMITSU
Department of Operative Dentistry Showa University School of Dentistry
2-1-1 Kitasenzoku, Ohta-ward Tokyo 145, Japan

Received August 3, 1995/Accepted October 27, 1995

The optimum concentration and application time for an experimental dentin primer composed of an aqueous solution of 1,6-hexanediol were determined by measuring the wall-to-wall polymerization contraction gap width of a commercial light-activated resin composite in a cylindrical dentin cavity prepared in an extracted human molar. Contraction gap formation was completely prevented only when the dentin cavity wall was primed with 45 wt% 1,6-hexanediol for 60 s. There were no significant differences in tensile bond strength among primers composed of 20.0-57.5 wt% 1, 6-hexanediol. These results suggest that the optimum concentration and application time are 45 wt% and 60 s, respectively.

Key words: 1, 6-hexanediol, Dentin primer, Contraction gap

INTRODUCTION

Priming of the dentin surface prior to application of a dentin bonding agent is believed to improve the efficacy of dentin bonding systems and to promote a complete seal between the resin composite restoration and the dentin cavity margin. This priming procedure was introduced by Munksgaard and Asmussen1), who developed a dentin primer composed of an aqueous mixture of hydroxyethyl methacrylate (HEMA) and glutaraldehyde. They speculated that the amino group in the dentin collagen was activated by the glutaraldehyde and made polymerizable with HEMA2). However, this bonding mechanism is unlikely, since we found that 35 vol% HEMA solution without aldehyde was equally effective as a dentin primer when priming was followed by a commercially available dentin bonding agent containing phosphoric acid ester; i.e., 10-methacryloxydecyl dihydrogen phosphate (MDP)3). Based on the speculation that an aqueous solution of esterified methacrylate with a polyvalent alcohol might be effective as a dentin primer, we developed three experimental dentin primers: glyceryl methacrylate (GM)4), erythritol methacrylate (EM)5) and xylitol methacrylate (XM)6). These were all definitely superior to the GLUMA and HEMA primers, since contraction gaps were formed by a commercially available light-activated resin composite in cylindrical dentin cavities in extracted human teeth in nearly half of the specimens prepared using HEMA or GLUMA priming. Methacrylic acid derivatives have been used not only in dentin primers but also in dentin bonding agents, because the hydrophilic and hydrophobic groups are believed to exhibit affinity for the dentin and the resin composite7), respectively. Furthermore, the double-bond between the carbon in the metha-
cryloyl group aids in polymerizing dentin adhesives after they are applied. It is recognized that some methacrylate monomers which have amino or hydroxyl groups cause a severe contact dermatitis, and the possibility of cross effects among the various methacrylate derivatives has not been overlooked. Therefore, it is advisable not only for the patient but also for the dentist to avoid direct contact with methacrylate monomers, or to simply not use methacrylate derivatives if possible. We recently found that contraction gap formed by a light-activated resin composite in a cylindrical dentin cavity can be completely prevented by using aqueous solutions of two diols: ethylene glycol and 1,6-hexanediol. This finding indicates that the dentin primer need not contain a polymerizable group. Furthermore, the use of methacrylate-free dentin primers may eliminate the possibility of contact dermatitis. The purpose of the present study was to examine the optimum concentration and application time of 1,6-hexanediol as a dentin primer.

MATERIALS AND METHODS

Experimental dentin primers were prepared by diluting 1,6-hexanediol in distilled water at concentrations ranging from 20 to 70 wt%. The efficacy of the primers was examined by measuring the contraction gap width of a commercial light-cured resin composite in a cylindrical dentin cavity and determining its tensile bond strength to a flat dentin surface.

Contraction gap measurement

A cylindrical cavity approximately 3 mm in diameter and 1.5 mm deep was prepared in the proximal dentin of an extracted human molar after eliminating the enamel with wet carborundum paper (grit number 1000). The cavity wall was cleaned with neutralized 0.5 mol/l EDTA (pH 7.4) for 60 s, then rinsed and dried, since it was found in our previous study that contraction gap width significantly increased with the degree of softening of dentin by the cleanser, and that EDTA exhibited complete efficacy as a dentin cleanser. An aqueous solution of 1,6-hexanediol was applied for 60 s and the cavity was dried completely. The cavity was then filled with a light-activated resin composite after applying a commercially available dual-cured dentin bonding agent and irradiating for 10 s. The cavity was slightly overfilled and the composite surface was gently pressed on a glass plate mediated with a plastic matrix before irradiation for 40 s, care being taken to ensure a tight fit between the light source and the plastic matrix.

The overfilled resin composite was removed with wet carborundum paper and the exposed margin was polished on a linen cloth mediated with an alumina slurry after the specimens had been stored in water at 24±1°C for 10 min. The marginal adaptation of the resin composite was inspected under a light microscope and the width of any contraction gap was measured at eight points, every 45 degrees, along the cavity margin. The contraction gap was calculated as the sum of each pair of diametrically opposed gap widths, and

* Wako Pure Chemicals Industrial, Osaka, Japan
** Silux Plus, 3M, St. Paul, MN, USA
# Clearfil Photo Bond, Kuraray Osaka, Japan
## Metaloplan, Leitz, Darmstadt, Germany
expressed as a percentage of the cavity diameter; the maximum of these four values was considered the maximum contraction gap. Ten specimens were prepared for each concentration. For the group with the optimum concentration of 1, 6-hexanediol, the priming time was then reduced successively from 60 s to momentary.

**Tensile bond strength measurement**
The dentin surface of an extracted human tooth embedded in epoxy resin was flattened with wet carborundum paper (grit number 1000). The dentin surface was cleaned with neutralized 0.5 mol/L EDTA for 60 s, then rinsed and dried. A split Teflon mold (inner diameter 3.6 mm, outer diameter 20 mm, height 5 mm) was then clamped onto the substrate. The experimental dentin primer was applied to the dentin through the top of the mold and the dentin surface was dried thoroughly. The lower part of the mold was filled with the light-cured resin composite to a thickness of less than 2 mm after application of the dual-cured dentin bonding agent and irradiation for 10 s. The light-cured resin composite was irradiated for 40 s and the upper part of the mold was filled with a chemically activated resin composite. A round bur was inserted into the unpolymerized resin composite paste perpendicular to the substrate to provide a grip for the measurement device. The Teflon mold was removed from the specimen and the tensile bond strength was measured after storing the specimen in water at 24±1°C for 24 h. The tensile bond strength of the specimens was measured using a universal testing machine with a cross-head speed of 5 mm/min. Ten specimens were prepared for each concentration of 1, 6-hexanediol (50 total).

**RESULTS**
The maximum contraction gaps measured are listed in Table 1. When the dentin priming

<table>
<thead>
<tr>
<th>Concentration (wt%)</th>
<th>Contraction gap (%)</th>
<th>Gap-free</th>
<th>Tensile bond strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.145±0.058</td>
<td>0</td>
<td>3.7±1.4*</td>
</tr>
<tr>
<td>20.0</td>
<td>0.025±0.031</td>
<td>5</td>
<td>15.6±4.0</td>
</tr>
<tr>
<td>32.5</td>
<td>0.074±0.061</td>
<td>3</td>
<td>17.6±6.8</td>
</tr>
<tr>
<td>45.0</td>
<td>0</td>
<td>10</td>
<td>16.2±5.2</td>
</tr>
<tr>
<td>57.5</td>
<td>0.096±0.055</td>
<td>2</td>
<td>18.1±7.7</td>
</tr>
<tr>
<td>70.0</td>
<td>0.055±0.080</td>
<td>5</td>
<td>8.6±5.4</td>
</tr>
</tbody>
</table>

N = 10
The dentin cavity wall or the flat dentin surface was cleaned with neutralized 0.5 mol/L EDTA (pH 7.4) for 60 s and the resin composite was applied, mediated with a commercial dentin bonding agent.

* The tensile bond strength of the no priming group is from our previous report, which employed in the same method.

* P-10, 3M. St. Paul., MN. USA
Table 2  Efficacy of an experimental dentin primer composed of 45 wt% of 1, 6-hexanediol solution

<table>
<thead>
<tr>
<th>Priming time (s)</th>
<th>Contraction gap (%)</th>
<th>Gap-free</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.145±0.058</td>
<td>0</td>
</tr>
<tr>
<td>momentary</td>
<td>0.051±0.073</td>
<td>6</td>
</tr>
<tr>
<td>15</td>
<td>0.134±0.092</td>
<td>2</td>
</tr>
<tr>
<td>30</td>
<td>0.092±0.0101</td>
<td>5</td>
</tr>
<tr>
<td>45</td>
<td>0.077±0.074</td>
<td>4</td>
</tr>
<tr>
<td>60</td>
<td>0</td>
<td>10</td>
</tr>
</tbody>
</table>

N=10
The dentin cavity wall was cleaned with neutralized 0.5 mol/L EDTA (pH 7.4) for 60 s and the resin composite was applied, mediated with a commercial dentin bonding agent.

Dentin priming is believed necessary to obtain marginal integrity in resin composite restoration of a dentin cavity. Most commercially available dentin primers contain a HEMA solution because they were developed by modifying the components of GLUMA primer, which is an aqueous solution of HEMA and glutaraldehyde. We demonstrated that HEMA solutions are not particularly desirable for practical use as a dentin primers because contraction gap formed by a light-cured resin composite can not be completely prevented by HEMA priming\(^\text{11}\). In addition, HEMA also causes contact dermatitis on the skin of the operator\(^\text{12}\). The possibility of a delayed allergic skin reaction can not be eliminated by the use of GM, EM, or XM, due to the possibility of cross-reaction between the methacrylate derivatives, although contraction gap formation can be prevented by aqueous solutions of these three methacrylate derivatives. We recently determined that the dentin primer need not contain a polymerizable group, since two diols, ethylene glycol\(^\text{13}\) and 1, 6-hexanediol\(^\text{14}\), were completely effective as dentin primers. This finding may help us avoid the side effects of methacrylate derivatives.
The significance of the water content of dentin primers has not yet been explained in detail, although most dentin primers contain significantly large amounts of water. However, we can speculate that dentin priming affects the physical surface characteristics and that the diol might play a role as a hydrophilic surface activator. The priming time of 1, 6-hexanediol could not be reduced below 60 s without formation of a contraction gap. Nakamura et al.\textsuperscript{15} and Shono et al.\textsuperscript{16} reported that priming for 30 s with a self-etching dentin primer composed of an aqueous solution of 20 wt% methacryloxyethyl hydrogen phenyl phosphate (phenyl-P) and 45 wt% 1, 6-hexanediol or 62.5 wt% ethylene glycol was effective in that it completely prevented gap formation. However, this experimental self-etching dentin primer is associated with the possibility of side effects from the methacrylate derivative phenyl-P. In the present study, the dentin cavity wall was cleaned and primed separately, and the dentin surface was conditioned for more than 90 s before application of the dentin bonding agent, which contrasts markedly from the self-etching dentin primer procedure. Ohhashi et al. reported that GM solution, as a primer, needed to be applied only momentarily if the dentin cavity wall was first cleaned with EDTA for 60 s\textsuperscript{17}. The results of this study indicate that momentary priming with 1, 6-hexanediol solution not can be recommended for clinical applications. In addition, the dentin cavity wall should be cleaned with EDTA for 60 s and primed with 1, 6-hexanediol solution for 60 s, even though this may complicate and prolong the restoration procedure. It is difficult to explain why priming with 1, 6-hexanediol for 60 s yielded the best results. This diol has no chemical groups which can be polymerized or chemically bond to the dentin structure. Further studies are required to determine the significance of primer water content and the effect of diol solution on the dentin surface.

CONCLUSIONS

The optimum concentration and application time of the 1, 6-hexane diol solution as a dentin primer was determined to be 45 wt% and 60 s, respectively, since contraction gap formation of a commercial light-cured resin composite in a cylindrical dentin cavity was prevented completely only when this diol solution was applied in the manner described. Dentin priming using this diol solution is beneficial not only for the patient but also for the dentist in avoiding the side effects on skin tissue caused by methacrylate derivatives.

REFERENCES


パルビツル酸/塩化第二銅を開始剤とするレジンによる象牙質の接着

鈴木明子，今井庸二

東京医科歯科大学医用器材研究所生体機能材料部門

パルビツル酸/塩化第二銅を重合開始剤とするレジンの象牙質への接着において，パルビツル酸の構造ならびに前処理剤の効果を検討した。4種類のパルビツル酸/塩化第二銅を開始剤とするMMA-PMMAレジンを用いて，硬化時間とウシ象牙質への接着強さを測定した。象牙質面を3%の塩化第二銅または塩化第二鉄を添加あるいは添加していない10%クエン酸またはリン酸水溶液6種類で前処理した。4種類のパルビツル酸で硬化時間は有意差が認められたが，3%塩化第二銅を含む10%クエン酸水溶液処理した象牙質への接着強さには有意差はなかった。前処理剤は接着強さに有意に影響し，リン酸系処理剤の方がクエン酸系処理剤よりも有意に接着強さが大きかった。パルビツル酸/塩化第二銅系レジンを用いて最適条件下で接着すると，MMA-TBBレジンに匹敵する大きな接着強さが得られた。

試作デンティンプライマー1,6-ヘキサジオール水溶液の
至適濃度および至適作用時間の検討

Mir Ayubur Rahman，谷 千尋，伊藤和雄
和久本貞雄，久光 久
昭和大学歯学部歯科保存学第二講座

1,6-hexanediol水溶液からなる試作デンティンプライマーの至適濃度および，至適作用時間を決定する目的で，ヒト抜去大臼歯に形成された円柱窩洞にEDTAによるクリーニング，試作プライマーによる前処理，および市販デンティンボンディング材を併用して塗装した市販可視光重合型コンポジットレジンのコントラクションギャップを計測した。その結果，コントラクションギャップの形成は45 wt%の1,6-hexanediol水溶液を60秒間作用させた場合のみ完全に抑制され，この条件が最適と評価された。また引張り接着性試験においては20.0 wt%から57.5 wt%濃度の1,6-hexanediol水溶液を用いた場合で統計学的に有意差は認められなかった。

コンピュータによる陶材の自動築盛の試み

菊地聖史，高久田和夫¹，宮入裕夫¹，奥野 攻

東北大学歯学部歯科理工学講座

¹東京医科歯科大学医用器材研究所精密機械部門

従来の陶材技工システムを発展させた新しい補織物の自動製作システムの開発を目的とし，コンピュータを用いた陶材の自動築盛に関する基礎的・研究を行うとともに築盛作業を自動化する上での問題点について検討した。実験は陶材吐出装置，蒸留水噴射装置，XY軸ステージ，制御用コンピュータから構成される装置を試作し，陶材を平板状に自動築盛することを試みた。水量，面積，厚みの各築盛条件を変えて自動築盛を行った結果，試作装置のように水と陶材粉末を築盛対象の局所に交互に供給し，泥化させて盛り上げていく方法では水量が築盛体の