Effect of Copper Salts Added to Primer on Bonding of MMA-TBB Resin to Dentin Treated with Phosphoric Acid

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The effects of addition of various copper salts to 2-hydroxyethyl methacrylate (HEMA) primer were studied with regard to tensile bond strength of methyl methacrylate-tributylborane (MMA-TBB) resin to dentin. Bovine dentin surfaces were treated with 10% phosphoric acid, then with a primer consisting of aqueous 35% HEMA and a copper salt, and finally bonded to acrylic rods with MMA-TBB resin. The types of copper salts and concentrations in the primers were varied. The optimal concentration of copper salts in the primers was 0.5-3 μmol/g. The primers containing copper salts of sulfate, methacrylate, methacryloyloxyethyl phthalate, or methacryloyloxyethyl succinate were especially effective among the eight copper salts studied in increasing bond strength, producing mean bond strengths of 14.2-16.1 MPa and mean minimum values of 9.9-11.7 MPa. These bond strengths were higher than those obtained with the commercial 4-META/MMA-TBB resin system. A model experimental study concerning the molecular weight of poly(MMA) formed in the presence of some copper salts suggested that the increase in bond strength was produced through an increase in molecular weight of MMA-TBB resin by the copper salts.

Key words: Dentin bonding, Primer, TBB resin

INTRODUCTION

The commercial 4-META/MMA-TBB resin (SB resin)* is one of the most effective bonding systems to dentin. Its high bond strength is believed to be due to 4-META (4-methacryloyloxyethoxycarbonylphthalic acid anhydride), which improves bond strength by infiltrating the dentin and forming a dentin-resin hybrid layer1), and thus 4-META has been proposed as the key component in this system. However, we demonstrated that a combination of TBB initiator and ferric ions supplied to dentin from a dentin treatment mixture of 10% citric acid and 3% ferric chloride plays the decisive role by an interfacial initiation mechanism2). The presence of Fe (III) or Cu (II) ions adsorbed onto dentin collagen is a prerequisite for obtaining high bond strength in bonding systems using TBB as a polymerization initiator.

In a previous study, we showed that a primer containing copper (II) salts that promoted the interfacial initiation of polymerization from the dentin interface was very effective in bonding dentin etched with 10% phosphoric acid with 4-META/MMA-TBB resin3). In this study, we examined the effects of copper salts added to the primer on bonding of MMA-TBB

* Super-Bond C&B (Sun Medical Co., Shiga, Japan) and Amalgambond (Parkell Co., Farmingdale, NY, USA)
To discuss the possible mechanism of the contribution of some selected copper salts to the improvement of bond strength in reference to a possible role of 4-META in the SB resin system.

**MATERIALS AND METHODS**

Materials and methods were practically the same as those described in a previous paper.8)

**Primers**

Eight primers containing copper (II) salts were prepared by adding 0.5 μmol/g of the salts in aqueous 35% HEMA, as shown in Table 1. CMP and CMS were prepared from copper (II) chloride and sodium salt of the corresponding carboxylic acids**, which were obtained commercially.

**Measurement of bond strength**

Bovine anterior teeth were ground on a model trimmer to expose adequate dentin surface for bonding. The exposed surfaces were then finished with 400- and 600-grit wet abrasive paper, treated with 10 wt% phosphoric acid (10 PA) or a mixture of 10 wt% citric acid and 3 wt% ferric chloride (10-3) for 30 s, rinsed with water for 30 s, and dried with compressed air for 30 s. The primer was applied to the surface with a small brush, left for 15 s, and then dried gently with compressed air. Adhesive masking tape with a 5-mm-diameter hole was attached to the surface to delineate the bonding area. The surface was bonded to an 8-mm-diameter acrylic rod using MMA-TBB resin, which consisted of a 1:1 mixture of PMMA powder and MMA containing 10 wt% TBB initiator. The bonded specimen was left for 30 min in air at room temperature, and immersed in distilled water at 37°C for 24 h. Tensile bond strength was measured on an Instron-type testing machine at a cross-head speed of 2 mm/min. Five specimens were tested in each group.

Mean as well as the minimum value data from five specimens in each group were compared. The mean values were analyzed by one-way ANOVA for each primer.

<table>
<thead>
<tr>
<th>Primer* code</th>
<th>Copper salt in primer</th>
</tr>
</thead>
<tbody>
<tr>
<td>35% HEMA</td>
<td>None</td>
</tr>
<tr>
<td>CC</td>
<td>Chloride</td>
</tr>
<tr>
<td>CN</td>
<td>Nitrate</td>
</tr>
<tr>
<td>CS</td>
<td>Sulfate</td>
</tr>
<tr>
<td>CA</td>
<td>Acetate</td>
</tr>
<tr>
<td>CAA</td>
<td>Acetylacetonate</td>
</tr>
<tr>
<td>CM</td>
<td>Methacrylate</td>
</tr>
<tr>
<td>CMP</td>
<td>Methacryloyloxyethyl phthalate</td>
</tr>
<tr>
<td>CMS</td>
<td>Methacryloyloxyethyl succinate</td>
</tr>
</tbody>
</table>

* Contained 0-5 μmol/g of copper (II) salt in 35% HEMA

** Kyoeisha Yushi Chemical Co., Osaka, Japan

# Autograph AGS 1000A, Shimadzu Co., Kyoto, Japan
Duncan's new multiple range test was used to compare mean values significantly different at $p<0.05$.

After the bond tests, the fracture surfaces were examined to identify the mode of bond failure. Failure at the dentin/resin interface, that occurring within the bonding resin and/or dentin, and failure partly at the interface and partly within the resin were termed adhesive (A), cohesive (C), and mixed failures (M), respectively.

**Preparation and determination of molecular weight of poly (methyl methacrylate)**

Six samples of poly (methyl methacrylate) (PMMA) were prepared by polymerization of a 1:1 (w/w) mixture of PMMA and MMA containing 10 wt% TBB, 0 or 2 $\mu$mol/g of copper salts (CAA, CMP, and CMS), and/or 4-methacryloyloxyethoxycarbonylphthalic acid anhydride (4-META) at 25°C for 24h or at 37°C for 1h.

Viscosity of the PMMA obtained was measured in chloroform at 25°C with an Ostwald type viscometer. Molecular weight of the PMMA was calculated from the intrinsic viscosity equation:

$$\text{Intrinsic viscosity} = 4.8 \times 10^{-5} \text{ (molecular weight)}^{0.80}$$

**RESULTS**

The mean, minimum, and maximum values of the tensile bond strengths with copper primers are plotted against the concentration of copper salts in 35% HEMA in Fig. 1. The data at higher concentrations of CM are lacking because preparation of the primer was difficult due to the limited solubility of CM in 35% HEMA. One-way analysis of variance for each copper primer showed that, for most salts, the mean bond strengths were not significantly affected by the concentrations over the considerably wide range studied ($p>0.05$). Thus, for the purposes of analysis, the results for the optimal concentrations showing no significant difference could be combined for each copper salt. Mean and minimum values of bond strength at the optimal concentration are compared with those in control groups including the SB resin system in Table 2, in which mean and range of minimum value are also shown.

The bond strengths with copper-containing primers were significantly higher than those obtained with 35% HEMA primer alone or without primer (10.9-16.1 MPa vs 5.4 and 4.2 MPa). The primers containing CS, CM, CMP, and CMS produced high mean bond strengths of 14.2-16.1 MPa and high mean minimum values of 9.9-11.7 MPa. These mean values were higher, although not significantly so, than the values obtained with the SB resin system (13.2 and 9.0 MPa, respectively; 2nd line in Table 2).

Test specimens producing bond strengths below 4 MPa, between 4 and 9 MPa, and over 9 MPa failed mostly in adhesive, mixed, and cohesive modes, respectively. All specimens with CS, CM, CMP, and CMS primers failed cohesively.

Table 3 shows the results of molecular weight determination of various types of PMMA prepared under different conditions. Addition of copper salts and/or 4-META resulted in an increase in the molecular weight from $23 \times 10^4$ to $36-38 \times 10^4$ or $41 \times 10^4$ at 25°C and from $40 \times 10^4$ to $41-46 \times 10^4$ at 37°C, respectively. The presence of the copper salt CAA with 4-META increased the molecular weight further by $5 \times 10^4$. PMMA obtained in the presence of 4-META at 37°C were cross-linked slightly and insoluble in chloroform.
Fig. 1 Effects of copper salt concentration in HEMA primer on mean, standard deviation, minimum, and maximum values of tensile bond strength of MMA-TBB resin to dentin treated with 10% phosphoric acid.

- mean; ◇, minimum; ○, maximum.
Table 2  Effects of primers containing copper (II) salts on mean and minimum values of tensile bond strength in bonding of MMA-TBB resin to dentin

<table>
<thead>
<tr>
<th>Dentin treatment</th>
<th>primer</th>
<th>Optimal concentration of Cu (II) in primer (μmol/g)</th>
<th>Bond strength (MPa)</th>
<th>Minimum value</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Mean SD n</td>
<td>Mean SD n</td>
<td>Range</td>
</tr>
<tr>
<td>10-3</td>
<td>None</td>
<td>9.3 ± 4.0</td>
<td>1.8 1</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>None</td>
<td>13.2 ± 3.5</td>
<td>6.3 1</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>35%HMA</td>
<td>12.8 ± 6.1</td>
<td>1.6 1</td>
<td>1</td>
</tr>
<tr>
<td>10PA</td>
<td>None</td>
<td>4.2 ± 2.4</td>
<td>2.0 1</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>35%HMA</td>
<td>5.4 ± 2.0</td>
<td>2.0 1</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>CAA</td>
<td>0.5-5.0 ± 10.9 ± 3.8 ± 25</td>
<td>7.1 ± 0.5 5</td>
<td>6.3-7.5</td>
</tr>
<tr>
<td></td>
<td>CC</td>
<td>0.5-5.0 ± 12.5 ± 3.7 ± 30</td>
<td>8.1 ± 3.1 6</td>
<td>6.4-9.3</td>
</tr>
<tr>
<td></td>
<td>CN</td>
<td>0.5-5.0 ± 12.7 ± 4.0 ± 30</td>
<td>8.1 ± 3.1 6</td>
<td>5.5-11.8</td>
</tr>
<tr>
<td></td>
<td>CA</td>
<td>0.5-2.0 ± 12.8 ± 4.7 ± 25</td>
<td>7.7 ± 3.1 6</td>
<td>5.0-9.9</td>
</tr>
<tr>
<td></td>
<td>CMS</td>
<td>0.5-3.0 ± 15.0 ± 4.7 ± 25</td>
<td>11.0 ± 3.1 4</td>
<td>8.3-13.7</td>
</tr>
<tr>
<td></td>
<td>CMP</td>
<td>0.5-5.0 ± 15.3 ± 4.3 ± 30</td>
<td>10.2 ± 3.1 4</td>
<td>10.4-14.9</td>
</tr>
<tr>
<td></td>
<td>CS</td>
<td>0.5-3.0 ± 16.1 ± 4.8 ± 20</td>
<td>10.3 ± 3.1 4</td>
<td>7.5-14.6</td>
</tr>
</tbody>
</table>

* Treated with a mixture of 10% citric acid and 3% ferric chloride (10-3) or 10% phosphoric acid (10PA)
** MMA containing 5% 4-META was used
# Means with the same letters were not significantly different at the level of 0.05

Table 3  Intrinsic viscosity and molecular weight of PMMA obtained in the presence of copper salts and/or 4-META at 25 or 37°C

<table>
<thead>
<tr>
<th>Additive*</th>
<th>25°C</th>
<th>37°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Intrinsic viscosity</td>
<td>Molecular weight×10⁻⁴</td>
</tr>
<tr>
<td>None</td>
<td>0.94</td>
<td>23</td>
</tr>
<tr>
<td>CMP</td>
<td>1.35</td>
<td>36</td>
</tr>
<tr>
<td>CMS</td>
<td>1.39</td>
<td>38</td>
</tr>
<tr>
<td>CAA</td>
<td>1.37</td>
<td>37</td>
</tr>
<tr>
<td>CAA/4-META</td>
<td>1.63</td>
<td>46</td>
</tr>
<tr>
<td>4-META</td>
<td>1.48</td>
<td>41</td>
</tr>
</tbody>
</table>

* Two μmol/g of copper salt (CMP, CMS, and CAA) and/or 5 wt% of 4-META were added to MMA
** PMMA was insoluble in chloroform (cross-linked)

DISCUSSION

The use of primers containing copper salts significantly improved bond strength. Optimal copper salt concentrations producing high bond strengths were in the range between 0.5 μmol/g and 3 μmol/g, mostly 1-2 μmol/g. Copper salts of methacrylate (CM), methacryloyloxyethyl phthalate (CMP), methacryloyloxyethyl succinate (CMS), and sulfate (CS) were especially effective in improving mean and minimum bond strengths. The increase in
minimum bond strength is especially important from a clinical viewpoint to increase reliability of the bonding. In this paper, we report and discuss the minimum values obtained in each group, based on the suggestion that the minimum value is critical for clinical success in practice and to be included in all data reported\textsuperscript{5-7}). Minimum values should also be compared in evaluating bond strength, when we consider the safety factor to be taken into account in a clinical situation and can guarantee that even the minimum bond strengths are sufficiently strong to prevent debonding.

For improved bond strength, we postulated that CM, CMP, and CMS would have the potential to function as cross-linking agents and thus result in an increase in molecular weight and bond strength. We then performed a model experiment concerning the effects of the addition of copper salts on the molecular weight of the PMMA formed. The molecular weight of the PMMA increased with addition of some copper salts and/or 4-META (Table 3).

Almost all the failures above 9 MPa occurred cohesively within the resin. In these cases, the tensile bond strengths were indicative of the cohesive strength of the resin. The actual bond strength of the system to dentin was greater than those values. An increase in molecular weight of a polymer generally increases mechanical strength. Therefore, it was reasonable to assume that dimethacrylic copper salts such as CM, CMP, and CMS and/or 4-META functioned as cross-linking agents to increase the molecular weight of PMMA resulting in an improvement of bond strength.

This mechanism of the contribution of 4-META to improvement in the bond strength is completely different from that proposed by Nakabayashi who postulated that 4-META improved the bond strength by infiltrating the dentin and forming a dentin-resin hybrid layer\textsuperscript{1}). With regard to the role of 4-META in the SB resin system, we have clearly demonstrated previously that an additional role of 4-META in bonding is not comparable with a decisive role of ferric ions which are contained in the dentin treating solution\textsuperscript{2,3). This will again be clear from Table 2 when we compare the bond strengths from the 1st line through the 5th. Moreover, the mechanism of an additional effect of 4-META itself has already been postulated; (1) 4-META may have the capacity to act as a cross-linking agent to increase the molecular weight of the resin and consequently the bond strength, and (2) ferric ions may enhance the cross-linking effect of 4-META\textsuperscript{4}). In this study, we confirm the postulates in a model experiment by determining the molecular weight of PMMA. The molecular weight of PMMA increased with addition of 4-META, and the presence of copper salts with 4-META resulted in a further increase in the molecular weight.

A possible role of cross-linking in improving bond strength has been suggested in a bonding system using propanal by Asmussen and Munksgaard\textsuperscript{5}). Although the authors have not referred to it, they, in a recent separate paper, reported significant improvement in mechanical properties of composite resins by the addition of propanal, suggesting a potential role of this compound as a cross-linking agent\textsuperscript{6}). Therefore, it appears natural to correlate the improvements both in bond strength and mechanical properties with cross-linking by propanal. While they have postulated condensation reaction mechanisms for the cross-linking, we would rather suggest a chain transfer mechanism involving free radicals as we proposed previously in a bonding system using glutaraldehyde\textsuperscript{7}).
The primary roles of copper salts were as interfacial polymerization promoters. Additionally, some copper salts acted as cross-linkers increasing the molecular weight of PMMA resin. Copper salts such as CM, CMP, and CMS with polymerizable double bonds have an advantage over other common copper salts such as CC, CN, CA, and CAA in increasing the cohesive resin bond strength not only as polymerization promoters, but also as cross-linking agents.

Commercial HEMA usually contains a small amount of methacrylic acid as an impurity. Therefore, even in bonding with HEMA primers containing inorganic copper salts, there will be the possibility of formation of copper methacrylate (CM) between the copper salts and methacrylic acid, resulting in an increase in the cohesive strength of the resin at the dentin interface. Whether this interpretation could also be applied to the improved bond strength using the CS primer remains to be investigated further.

This study demonstrated again that the presence of an adequate amount of metal ions such as ferric or copper ions at the dentin interface is essential for strong bond formation using TBB resin. Metal ions adsorbed on the dentin surface can promote polymerization of MMA in combination with TBB, and therefore can cause interfacial initiation of polymerization\(^{12}\). Once interfacial initiation occurs, polymerization of resin proceeds from the interface outward. Therefore, the interfacially-initiated polymerization can minimize the undesirable effect of contraction force generated by shrinkage of polymerizing resin on the interface. Thus, interfacial polymerization can contribute to increasing the bond strength and marginal adaptation or decreasing contraction gap and microleakage. We have discussed the advantages of interfacial polymerization previously\(^2\), and also, the effects of polymerization contraction stress and initial polymerization site on tooth-resin bonding have been well reviewed by Van Meerbeek et al.\(^{13}\).

The statement that “Amalgambond catalyst (TBB) actually requires water and oxygen to initiate polymerization.”\(^{14}\), Amalgambond and Super-Bond, a similar 4-META product, \(^{14}\) appears to represent a typical understanding of SB resin. As we have shown in detail previously\(^3\), there is no significant specific effect, except in the role as a cross-linking agent as observed in this study, of 4-META in increasing the bond strength to dentin. The MMA-TBB bonding system does not work without ferric or copper (II) ions. Therefore, the copper-containing primers could work effectively in bonding to dentin treated with 10% phosphoric acid without 4-META or the so-called 10-3 solution treatment. In this respect, the statement cited above could better be replaced with “Amalgambond catalyst (TBB) actually requires ferric or copper ions to initiate polymerization.”\(^{14}\), Amalgambond and Super-Bond, a product based on a similar TBB/ferric ion system.\(^{14}\). The reaction product at the dentin interface between 4-META and ferric ions, which are supplied from 10-3 solution to dentin, would have a structure analogous to CMP and CMS, ferric polymethacrylates, and could also be similarly effective in bonding.

The advantage of the use of a copper primer in the TBB resin system is that it enables dentin and enamel to be treated simultaneously with 10% phosphoric acid, thus eliminating the troublesome two-step treatment (with phosphoric acid for enamel and 10-3 solution for dentin) recommended for the SB resin system.
CONCLUSION

Primers composed of aqueous 35% HEMA containing 0.5-3.0 μmol/g of copper (II) salts were very effective in improving bond strength of MMA-TBB resin to dentin treated with 10% phosphoric acid. Primers containing copper salts of sulfate, methacrylate, methacryloyloxyethyl phthalate, or methacryloyloxyethyl succinate were especially effective among the eight copper salts studied. These salts appeared to function as cross-linking agents in addition to their roles as polymerization promoters at the dentin interface.

REFERENCES

Megafillerを充填したコンポジットレジンの耐摩耗性

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臼歯咬合面に修復されたコンポジットレジンの臨床予後を決定づける重要な物性は耐摩耗性である。筆者らはさきに、Bowenによって提案されたβ-Quartz Glass-Ceramic (Megafiller) の粗粒をコンポジットレジン中に混入する新しい臨床技法が、修復物の重合収縮率を効果的に減少させることを報告した。今回、6種の光重合型コンポジットレジンを用いて、Megafiller 塩入の有無が耐摩耗性に与える効果を検討した。摩耗試験は、筆者らが考案したグラスビーズ摩耗試験および通常のハブラシ摩耗試験の2種類の方法で評価した。摩耗深さは、精度μmの万能投影機を用いて摩耗試験前後の厚さを計測して求めた。実験の結果、Megafillerの塩入によって、概ね数値的には摩耗深さは小さくなる傾向が認められたが、1,2の材料を除いて統計的有意差はみられなかった。また、コンポジットレジンのMegafillerへの"ぬれ性"はコンポジットレジンの種類によって僅かに差異が認められた。

カルシウムイオンがアパタイト生成に及ぼす影響

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カルシウムイオン（Ca²⁺）がアパタイト（HAP）生成に及ぼす影響を検討した。Ca²⁺が存在しない条件を含む種々のCa²⁺濃度（0〜200mM）において無水リン酸水素カルシウム（DCPA）の加水分解をpH スクラッチを用いて行った。加水分解速度は滴定速度から、また得られたHAP結晶の結晶性を粉末X線回折法により測定した。Ca²⁺が存在する場合、非存在下と比較して、加水分解速度が非常に促進され、また HAPの収率は2倍になった。一方、加水分解速度、HAPの収率はCa²⁺濃度（25〜200mM）には影響を受けなかった。溶液化学平衡計算によればCa²⁺が存在する場合、その非存在下に比較して非常に少量のDCPAが溶解すれば溶液が HAPと平衡になることが示された。従って我々はCa²⁺がDCPAのような酸性リン酸カルシウムの加水分解に重要な役割を果たすと結論した。

リン酸で処理した象牙質へのMMA-TBBレジンの接着におけるプライマーへの銅塩の添加効果

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メチルメタクリレート（MMA）－トリプチルボラン（TBB）系レジンを用いた象牙質への接着において、
HEMA プライマーへの各種銅塩の添加効果を検討した。ウシ象牙質表面を 10 %リン酸水溶液で処理した銅塩を含む 35 % HEMA 水溶液プライマーを塗布し、MMA-TBB レジンでアクリル樹脂を接着した。銅塩の種類・濃度を変えて接着したところ、プライマー中の銅塩の最適濃度は 0.5-3.0μmol/g であった。検討した 8 種類の銅塩の中で、硫酸、メタクリル酸、メタクリロイルオキシエチルフルアルデヒド、メタクリロイルオキシエチルホルム酸の銅塩が特に有効であり、14.2-16.1MPa の平均接着強さおよび最低の接着強さの平均値として 9.9-11.7MPa が得られた。これらの接着強さは、市販の 4-META/ MMA-TBB レジン接着システムで得られる値よりも大きかった。ある種の銅塩存在下で重合したポリ MMA の分子量が増加したことから、いくつかの銅塩では MMA- TBB レジンに対するその分子量増加効果によって接着強さが向上したことが示唆された。

光重合コンポジットレジンの重合性に及ぼす光強度の影響

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照射距離を変化させて得られた種々の光強度で光重合コンポジットレジンを重合し、硬化面を初期硬化体内部の反応率、重合率およびベンダクト二重結合の深さ方向への分布を測定し、これら重合特性に及ぼす光強度の影響を調べた。照射する照射量（光強度と照射時間の積）を一定にすると光強度、照射時間に関わらず硬化深さはほぼ一定の値を示し、硬化体内部の反応率、重合率およびベンダクト二重結合の各分布もよく一致した。硬化深さは照射量の対数で表され、この直線回帰式から使用した光照射器に対する材料の吸光係数および材料が硬化するのに必要な照射量（透界照射量）を求めることができた。

チタン鍛造におけるガスバージの効果

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溶解雰囲気と鍛型内のガス雰囲気を目的としたダブルガスバージを特徴として開発されたチタン専用鍛造機を用い、ダブルバージの効果について検討することを目的に、溶解雰囲気中ならびに純チタン鍛造体中の酸素濃度分析、鍛造体の硬さ試験および耐食性試験を行った。ダブルバージを採用することで溶解雰囲気の極低酸素化が可能となり、純チタン鍛造体中の酸素量は他機種に比べて著しく低くなった。さらにチタン鍛造体表面の硬さもバージを採用することで減少した。しかし、鍛造性に対しては影響はみられなかった。以上の結果から、ダブルバージを採用して開発されたチタン鍛造機により酸素の混入を抑えたチタン鍛造体を作製できることが示された。