Effect of Dentin Cleansers on the Bonding Efficacy of Dentin Adhesive

Mikio CHIBA, Kazuo ITOH and Sadao WAKUMOTO
Department of Operative Dentistry, Showa University, School of Dentistry, 2-1-1 Kitasenzoku, Ohta-ku, Tokyo 145, Japan

Accepted on April 1, 1989

The relation between the bonding efficacy of the dentin adhesive and the physical change of the dentin after treatment with various dentin cleansers was investigated.

The dentin surface was cleaned with one of the seven experimental dentin cleansers, 5, 10, 20, 30 and 40% of the phosphoric acid, neutralized 0.5 M EDTA solution (pH 7.4) and 9.1% pyruvic acid containing 9.1% glycin, prior to the combining application of 35% hydroxyethylmethacrylate solution (HEMA) and a marketed dentin bonding agent.

It was found that the bonding efficacy of the dentin bonding system employed in this study deteriorated with the degree of reduction of Vickers hardness on the dentin surface after the dentin cleaning.

Key words: Dentin cleanser, Dentin adhesive, Composite resin

INTRODUCTION

The treatment of the smear layer on the ground dentin surface, which disturbs the bonding between the resin and the cavity wall, is still being discussed although a number of researchers have reported various dentin cleansers1-7).

Most of these cleansers are composed of acid or mordants such as phosphoric acid, citric acid containing ferric chrolide, ethylenediamine tetraacetic acid (EDTA) or ferric oxalate.

The smear layer is easily dissolved by these cleansers and removed by water spray. In this procedure of dentin cleaning, however, it is considered that the dentin under the smear layer also becomes decalciﬁed, more or less, which might affect not only the pulp irritation but also the bonding efficacy of the dentin bonding system. The purpose of the present study was to examine the effect of the dentin cleanser on the physical properties of the dentin and on the bonding of the dentin adhesive.

MATERIALS AND METHODS

For the experimental dentin cleansers, phosphoric acid solution of 5, 10, 20, 30 and 40%, neutralized 0.5 M EDTA solution (pH 7.4) and 9.1% pyruvic acid containing 9.1% glycin (P-G solution) were prepared.

The flat dentin surface, prepared by grinding the occlusal enamel of the extracted human molars, was cleansed with one of the cleansers described above and sectioned along the tooth axis through the center of the treated dentin. They were then dehydrated by ethanol before the procedure of critical point dry and the morphological change of the dentin surface was
EFFECT OF DENTIN CLEANSERS ON DENTIN ADHESIVE

Fig. 1 Schema of specimen for measurement of Vickers hardness. Half of the surface was covered with an adhesive tape before dentin cleaning.

Fig. 2 The specimen for measurement of decalcified dentin layer. — The layer between arrows was measured as the decalcified dentin.

observed by the scanning electron microscope*

The effect of the dentin cleansers on the human dentin was determined by measuring the change in Vickers hardness (VHN) and the thickness of the decalcified dentin.

1. Measurement of micro VHN

The proximal surface of the extracted human molars was eliminated on a wet silicon carbide paper of #1,000 and the facial or lingual half of the flat dentin surface was covered by an adhesive tape. The exposed dentin was then treated with one of the experimental dentin cleansers by using a small sponge pellet for 60 s. The VHN of the treated and untreated dentin surface was measured by a micro-hardness tester** at ten points for each area as shown in Fig. 1.

The relative hardness of the treated dentin surface was shown in per cent against the

* S-700, Hitachi, Tokyo, Japan
** MVK-E, Akashi, Tokyo, Japan
hardness of the untreated dentin.

Five specimens were prepared for each cleanser.

2. Measurement of thickness of decalcified dentin

The flat dentin surface was prepared by grinding the occlusal enamel of the extracted human molars perpendicularly to the tooth axis on a wet silicon carbide paper and treated with experimental dentin cleansers.

The specimens were then dehydrated in ethanol, dried in the chamber of the critical point dry apparatus*** and finally embedded in an epoxy resin. The thickness of the decalcified dentin was measured by the screw micrometer**** mounted on the ocular lens of an optical microscope***** on the section surface of the treated dentin which was prepared by grinding the embedded specimens as shown in Fig. 2.

3. Measurement of the wall-to-wall polymerization contraction

The proximal surface of the extracted human molars was eliminated and the cylindrical cavity approximately 3 mm in diameter and 1.5 mm in depth was prepared in the exposed dentin.

The cavity wall was cleansed by one of the experimental dentin cleansers for 60 s, rinsed and dried by a blast of compressed air. Then the cavities were treated by an the aqueous solution of 35% hydroxyethylmethacrylate (HEMA)† for 60 s and dried completely.

After applying the dentin bonding agent‡ and removing the excess of the material by an air blast, the cavity was slightly overfilled with a light-curing composite§ and the resin surface was covered by a plastic matrix on a glass plate under gentle pressure.

The composite was then irradiated by a lamp unit¶ for 40 s, mediated by a plastic matrix, and the specimens were stored in water at a room temperature of 24±1°C.

After ten minutes, the cavity margin was exposed on the silicon carbide paper and polished with linen and alumina slurry, and the width of the contraction gap was measured by the screw micrometer mounted on the ocular lens of an optical microscope. Ten specimens were prepared for each cleanser and the maximum contraction was shown in per cent of the cavity diameter.

4. Measurement of tensile bond strength

The proximal surface of the extracted human teeth embedded in the epoxy resin was ground on the wet silicon carbide paper of #1,000 and the flat dentin surface was prepared.

The surface was cleansed with experimental dentin cleansers and treated with a dentin primer of aqueous solution of 35% HEMA for 60 s. Then the split teflon mold, inner diameter of 3.6 mm, outer diameter of 20 mm and height of 5 mm, was clamped on to the dentin and the dentin bonding agent was applied from the top window of the mold. The lower half of the mold was filled with a light-curing composite and irradiated for 40 s, and then the upper

---

*** HCP-2, Hitachikouki, Tokyo, Japan

**** Model RZDO-DO, Leitz, Wetzlar, W, Germany

***** Orthoplane, Leitz, Wetzlar, W, Germany

† Merck, Darmstadt, W, Germany

‡ Clearfil New Bond, Kuraray, Okayama, Japan

§ Silux, 3M, St. Paul, MV, USA

¶ Focus activater Light, Teledyne Getz, Chicago, USA
half was filled with the chemical curing composite†. A round bar of #6 was inserted into the unpolymerized composite to make a grip for the tensile bond strength measurement.

The teflon mold was removed and the specimens were stored in water at a room temperature of 24±1°C for 24 hours and the bond strength was measured by the use of a universal testing machine‡, at a crosshead speed of 5 mm/min.

Ten specimens were prepared for each cleanser.

RESULTS

The smear layer on the ground dentin surface was completely removed by the dentin cleansers employed in this study, whereas the dentinal plug remained in the EDTA and P-G solution. In the phosphoric acid group, a fibrous structure without the fine crystal-like substance was observed in the superficial dentin. And, in the specimens of the 30% phosphoric acid group, all of the fine structures were destroyed as shown in Fig. 3a-3h.

In the measurement of both the decalcified dentin thickness and relative hardness, a high correlation was observed with the increase in the concentration of the phosphoric acid as shown in Figs. 4-5.

In the EDTA and P-G solution, 85% of the relative hardness were maintained after cleansing and the thickness of the decalcified dentin by EDTA was too thin to measure under the optical microscope as shown in Fig. 6.

---

‡ P-10, 3M, St. Paul, MN, USA

†† TCM-20, Minebea, Tokyo, Japan
Fig. 3c  Sectioned dentin surface treated with 9.1% pyruvic acid containing 9.1% glycine.

Fig. 3d  Sectioned dentin surface treated with 5% H$_2$PO$_4$.

Fig. 3e  Sectioned dentin surface treated with 10% H$_2$PO$_4$.

Fig. 3f  Sectioned dentin surface treated with 20% H$_2$PO$_4$.

In the measurement of the contraction gap, the most desirable cleansing effect was obtained with the EDTA group in which seven out of ten specimens showed a complete seal.
EFFECT OF DENTIN CLEANSERS ON DENTIN ADHESIVE

Fig. 3g Sectioned dentin surface treated with 30% H₃PO₄.

Fig. 3h Sectioned dentin surface treated with 40% H₃PO₄.

Fig. 4 Correlation between relative hardness and concentration of H₃PO₄ solution.

between the resin and the dentin cavity margin.

And, in the phosphoric acid group, the width of the contraction gap increased, although a considerably high tensile bond strength was measured.
Fig. 5 Correlation between the decalcified dentin thickness and concentration of H$_3$PO$_4$ solution.

Fig. 6 Decalcified dentin layer treated with EDTA, which was too thin to be measured.

DISCUSSION

In this study, a high correlation was found between relative hardness after dentin cleaning and the wall-to-wall polymerization contraction gap as shown in Fig. 7. The most excellent bonding was obtained by EDTA cleansing, showing that the mean value of contraction gap was as small as 0.036% and seven out of ten were gap free. There is no significant
EFFECT OF DENTIN CLEANSERS ON DENTIN ADHESIVE

Fig. 7 Correlation between the relative hardness and maximum contraction of light-curing composite in the cylindrical dentin cavity mediated by HEMA and a marketed dentin bonding agent.

Fig. 8 Correlation between relative hardness and tensile bond strength of combining application HEMA and a marketed dentin bonding agent to human dentin.
correlation between relative hardness after dentin cleaning and tensile bond strength as shown in Fig. 8. These findings indicate that the primary requirement for a dentin cleanser is not to decalcify the sound dentin under the smear layer, and it was impossible to obtain a tight adaptation between the resin and the softened dentin.

As observed by the scanning electron microscopic study, the phosphoric acid removed the fine crystal-like substance, which might be a hydroxyapatite, at the concentration of only 5% and the collagen fibers were exposed in the superficial layer to the depth of 5 μm.

The depth of the decalcified dentin increased with the concentration of phosphoric acid. Such decalcification deteriorated the physical property of the dentin although the dentin primer and the dentin bonding agent might be able to penetrate into the more profound part of the dentin.

It was previously suggested that the decalcification by 40% phosphoric acid gel was effective both in removing the smear layer and in making it easy for the dentin bonding agent to penetrate into the deep layer of the dentin to produce a resin tag in the dentin tubules. It was, however, claimed that this dentin cleansing technique should be prohibited in practice in the clinic because it decreases the ability of the dentin adhesive and increases the chance of pulp irritation.

The findings obtained in this study, did not support the pretreatment by 40% phosphoric acid of the dentin surface.

It is possible to explain this discrepancy by the difference of the effect of contraction stress of comsite resin between tensile bond strength and wall-to-wall contraction gap measurement, which was extremely increased in the latter measurement, as claimed by Davidson et al.

We also recognized that the primary requirement for the dentin bonding system, was to completely prevent the contraction gap and secondly, to keep the bond strength for long periods under stress in the mouth. Therefore, it was not necessary to measure the tensile bond strength if the contraction gap formation couldn’t be interrupted just after polymerization.

Furthermore, the dentin primer employed in this study was previously reported to improve the bonding efficacy of the dentin bonding agent, although this solution was not effective in combination with the dentin cleanser of P–G solution and phosphoric acid.

Bowen et al. reported that the P–G solution was effective as a cleanser for both the dentin and enamel. However, we found that the superficial part of the dentin of a thickness of 3 μm deteriorated, although relative hardness was comparable with that of the EDTA. The difference between the adaptation to the dentin cavity wall cleansed with EDTA and P–G solution may be explained by the thickness of the deteriorated dentin.

Further effort is required to produce a dentin cleanser which does not affect the sound dentin under the smear layer.

**CONCLUSION**

The effect of the physical change of the dentin caused by various dentin cleansers on the efficacy of the dentin adhesive was investigated.
A high correlation was observed between the concentration of the phosphoric acid solution as a dentin cleanser and relative hardness on the dentin surface after cleansing.

The bonding between the composite and the dentin cavity wall deteriorated with the reduction of the dentin hardness.

Finally, the most desirable cleansing effect was obtained with the neutralized 0.5 M EDTA which softened the dentin at the degree of only 15%.

REFERENCES

象牙質強度はレジンの接着性に与える影響について
千葉幹男，伊藤和雄，和久本貞夫
昭和大学歯学部第二歯科保存学教室

Dentin cleanser によって象牙質の受ける変化が接着性に与える影響を及ぼすかについて、リン酸、グリシン含有ピルビン酸エタノールに水溶液を行なった後の象牙質の物理的特性を計測し、これらのレジンとの接着性の相関を検討した。その結果、レジンと象牙質との接着性は、cleanser によって象牙質に与える変化のうち Micro Vickers Hardness の低下と最も高い相関を示した。すなわち、cleanser によって残存象牙質硬度が 85% 以上に保たれる EDTA-2Na による処理が最も良好な接着性を示した。このことから、良好な接着性を得るためには smear layer のみを除去し、象牙質に全く影響を及ぼさない cleanser が理想的な材料であると結論付けられた。

Self Etching Dentin Primer の効果
千木良尚志*，小池斗誌江*，長谷川篤司*，伊藤和雄*，和久本貞夫*，早川徹**
*昭和大学歯学部第二歯科保存学教室
**日本大学松戸歯学部歯科理工学教室

35%HMEA 水溶液に希釈されたジカルボン酸または、そのエステル構造物を含む市販及び試作の Self Etching Dentin Primer の効果を象牙質円柱窩洞での可視光線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の測定と象牙質平面に対する引張り接着強さによって評価した。

試作した primer はすべて市販のリン酸エステル系ボンディング材と、また市販のセラフェッティングタイプの primer は付属のレジンモノマーを併用し、全ての窩洞および被着面には、市販の光重合型コンポジットレジンを塗塗または接着せたものとする。さらに、HMA にメタクリル酸エチルコハク酸または、メタクリル酸エチルフタル酸を溶解した 2 種類の試作 primer が、今回用いられたボンディング材の象牙質に対する接着性を著明に向上させた。また、マレイン酸を含む市販の primer は、24 時間後の引張り接着強さが 11.9±5.7 MPa と高い値を示したが、コンポジットレジンと象牙質窩洞との適合性を改善することはできなかった。

水中浸漬下におけるコンポジットレジンの圧縮クリープ
平野 進，平沢 忠
鶴見大学歯学部歯科理工学教室

コンポジットレジンの圧縮クリープに 4 種の異なる応力（圧縮応力 0～3.5 kg/mm²）で水中浸漬下で 500 時間により行った。得られた 4 種のクリープ曲線から、一定時間経過したときの各クリープひずみと圧縮応力と