Original paper

Effect of Resin Composite Adhesion on Marginal Degradation

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Inappropriate bonding treatment tends to result in marginal fractures or microleakage caused by masticatory loading. The purpose of this study was to investigate the effect of adhesion of resin composites on marginal degradation. Five posterior resin composites were inserted into Class I preparations with and without etching or bonding treatment. These restored teeth were subjected to a three-body wear test for 400,000 cycles, and the exposed enamel height at the tooth/restoration interface was determined by profilometer and scanning electron micrography (SEM). It was found that use of appropriate etching and bonding agents reduced the exposed enamel height and maintained marginal integrity after repeated loading.

Key words: Marginal Integrity, Resin Composite, Adhesion

INTRODUCTION

Although posterior resin composite has recently come into use as a substitute for amalgam, there still exist a number of problems for clinical use. These include technique-sensitivity, poor wear resistance, postoperative sensitivity and marginal leakage. Marginal integrity also plays a crucial role in long-term clinical success; however, even when complete marginal integrity is obtained initially, marginal deterioration such as gap formation is accelerated as a function of time. Marginal integrity is usually obtained through good adhesion between the tooth structure and the restoration. An appropriate etching and bonding treatment is a prerequisite for good adhesion, and neglecting such treatment leads to tremendous marginal leakage at the tooth/restoration interface. Polymerization shrinkage of resin composites induces gap formation and marginal fractures, and occlusal loading yields additional marginal stress resulting in marginal deterioration. The object of this study was to investigate this concept and relate the effect of adhesion on marginal integrity. In this study using a three body wear testing device chewing force was loaded repeatedly for 400,000 cycles, whereupon marginal geography was recorded with a profilometer. In addition, marginal integrity was observed by SEM. As an index of marginal deterioration, the exposed marginal enamel height was measured and marginal integrity was evaluated.

MATERIALS AND METHODS

The materials used in the study are presented in Table 1. The posterior resin composites
Table 1  Resin composite systems used in this study

<table>
<thead>
<tr>
<th>Code</th>
<th>Resin Composite</th>
<th>Mean particle size (μm)</th>
<th>Batch No.</th>
<th>Bonding agent (Batch No.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HEL</td>
<td>Heliomolar RO¹</td>
<td>0.04-0.06</td>
<td>43290</td>
<td>Amalgambond⁶ (10342 + 104072)</td>
</tr>
<tr>
<td>APH</td>
<td>Prisma AP. H.²</td>
<td>0.5-1.0</td>
<td>0509912</td>
<td>Prisma Universal Bond 3² (042491)</td>
</tr>
<tr>
<td>HER</td>
<td>Herculite XR¹</td>
<td>0.5-1.0</td>
<td>20184</td>
<td>Amalgambond XR Bond⁵ (022065)</td>
</tr>
<tr>
<td>P30</td>
<td>P-30⁴</td>
<td>1-5</td>
<td>5E3P</td>
<td>Amalgambond Scotchbond²⁺ (8AB)</td>
</tr>
<tr>
<td>OCC</td>
<td>Occlusion²</td>
<td>1-5</td>
<td>SH17</td>
<td>Amalgambond Bonding agent⁶ (AN15)</td>
</tr>
</tbody>
</table>

¹ Vivadent, Schaan, Lichtenstein  
² L. D. Caulk, Milford, DE, USA  
³ Kerr, Romulus, MI, USA  
⁴ 3M Dental Products, St. Paul, MN, USA  
⁵ GC Dental Co., Tokyo, Japan  
⁶ Parkell, Farmingdale, NY, USA

included two proprietary composites in which the mean filler particle size was greater than one micron. The other three materials possessed a mean particle size of one micron or smaller¹⁵,¹⁶.

In vitro chewing test.

The in vitro chewing testing was carried out using a recently developed three-body wear testing device in accordance with the technique previously described in the literature¹²-¹⁴. Sound, non-carious extracted human molars were selected for mounting in a brass specimen holder. The occlusal surface was then ground flat using a series of metallographic papers. Care was taken so that the ground surface consisted entirely of enamel. A well-defined cylindrical cavity, 4.0 mm in diameter and 4.0 mm in depth, was created in the center of the tooth specimen. All the cavosurface margins were carefully finished with a sharp carbide bur.

The teeth were divided into three groups (I, II and III). In Group I, the cut enamel surfaces were acid etched with a 37% H₃PO₄ solution for 20s followed by washing and drying. At this point the bonding system recommended by the manufacturer for the individual resin composite was applied to the walls of the cavity preparation in Group Ib. For comparison, Amalgambond was used as a bonding agent in Group Ia. Care was taken to follow the exact recommendations of each manufacturer. In Group II, the cavity was etched but no bonding treatment was performed. As for Group III, no treatment preceded the insertion of composites. In all cases, an appropriate resin composite was inserted incrementally into the cavity. Each segment was light cured for 40s with a light curing unit*, followed by a final light cure of 1 min.

Using a custom made hand-held device, the surface of the restored tooth was finished with series of metallographic papers down to a 600-grit silicon carbide paper in the presence

* Optilux 400, Demtron, CT, USA
of water. This procedure was carried out to insure that the flat occlusal surface was optically parallel to a horizontal plane and hence parallel to the surface of the stylus of the testing device. The specimens were then stored in deionized water for 72h.

Using a specially designed aligning device\textsuperscript{11}, the mounted specimen was inserted into the wear testing apparatus. The mounted tooth was next surrounded by a tight fitting ring which was then filled with water. Next, a slurry of unplasticized poly methylmethacrylate (PMMA) beads averaging 44 µm was poured onto the surface of the restored teeth. The small acrylic resin beads served as the food bolus.

At this point, a flat planed stylus machined from polyacetal (DERLIN) was positioned over the restored area. The diameter of the optically flat stylus was approximately 6.0 mm; it was centered to completely cover the restoration. At a rate of 2.0 times per second the stylus was vertically loaded onto the restored surface under a load of 75.6 N. As soon as the stylus initiated contact with the spherical particles, it began to rotate 30 degrees. When maximum loading was achieved, the stylus counter rotated and moved vertically to its original position. The entire cycle was repeated 400,000 times, requiring approximately 56h of continuous operation.

In addition, silicon impressions were taken of the restored surface at baseline and at intervals of 50, 100, 200 and 400 thousand cycles. The surface of each restoration was replicated twice with a polyvinyl silicone impression material\textsuperscript{**}. These were then cast with an epoxy die. One replica was coated with a gold–platinum alloy in preparation for SEM\textsuperscript{#}, and the other was used for profilometric scanning of the surface. The restored surface and adjacent enamel were traced with a profilometer\textsuperscript{*} in four different planes at 45 degrees to one another. From the four profilometric tracings eight readings were generated for each specimen, and the distance from the original occlusal cavosurface margin to the resin composite was recorded as the exposed enamel height. The original cavosurface margins were obtained by drawing a line between two reference points outside the area contacted by the stylus. Mean values and standard deviations were then calculated.

\textit{Statistical analysis.}

The data on the average exposed enamel height of each resin composite were analyzed first by a one–way analysis of variance with a probability level set at 95 percent (p<0.05). Then a Fisher PLSD Test\textsuperscript{17} was used to identify the statistically significant differences (p<0.05) among the various adhesive treatments after 400,000 cycles.

\textbf{RESULTS}

The average exposed enamel heights of the five resin composites after 400,000 cycles is shown in Table 2. Analysis of the data shows that the exposed enamel height was least when a bonding agent was employed (Group I), next lowest when only an etching agent was

\begin{itemize}
  \item Reprosil, Caulk Co, DE, USA
  \item Epoxy–Die, Vivadent, Lichtenstein
  \item S–2100B, Hitachi Co, Tokyo, Japan
  \item Surfcorder4000, Federal, RI, USA
\end{itemize}
applied (Group II), and largest with neither etching nor bonding treatment (Group III). Statistical analysis indicated significant differences (p<0.05) between Groups Ib and III as well as between Groups II and III when P30 was excluded. Comparing Group Ia with Group Ib, the only significant differences (p<0.05) between the two proprietary bonding systems involved APH and HER. Of all the composites used in this experiment, HEL exhibited the smallest mean enamel exposure, followed by HER, OCC, P30 and APH, regardless of adhesive treatment.

The longitudinal changes of the average exposed enamel depth of the five resin composites are presented in Figs. 1 to 5. These results reveal that the amount of enamel exposure decreased as a function of time: all the resin composites showed the greatest increase of

<table>
<thead>
<tr>
<th>Treatment</th>
<th>HEL</th>
<th>APH</th>
<th>HER</th>
<th>P30</th>
<th>OCC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group Ia</td>
<td>11.8 (8.5)</td>
<td>57.1 (11.6)</td>
<td>21.1 (5.7)</td>
<td>44.3 (14.8)</td>
<td>28.1 (9.5)</td>
</tr>
<tr>
<td>Ib</td>
<td>13.5 (5.2)</td>
<td>42.5 (8.3)</td>
<td>11.3 (3.7)</td>
<td>42.9 (7.9)</td>
<td>31.3 (5.2)</td>
</tr>
<tr>
<td>Group II</td>
<td>20.9 (4.5)</td>
<td>61.4 (15.7)</td>
<td>23.9 (5.5)</td>
<td>46.8 (24.5)</td>
<td>36.0 (5.0)</td>
</tr>
<tr>
<td>Group III</td>
<td>32.3 (2.9)</td>
<td>78.5 (19.9)</td>
<td>39.5 (7.9)</td>
<td>49.1 (9.5)</td>
<td>43.9 (3.0)</td>
</tr>
</tbody>
</table>

*: Mean (SD) Unit: μm
1: Amalgambond was used as the bonding agent
2: Attached bonding agent was used as the bonding agent
3: Etching agent (37% H₃PO₄) only was used
4: Neither etching nor bonding agent were used

Values connected by vertical lines were not significantly different (p<0.05).

Fig. 1 Longitudinal change of the average exposed enamel height of HEL.
exposed enamel height during the initial 50,000 cycles.

SEM pictures of the marginal interface of the one resin composite after 400,000 cycles

Fig. 2 Longitudinal change of the average exposed enamel height of APH.

Fig. 3 Longitudinal change of the average exposed enamel height of HER.
are shown in Fig. 6, illustrating various bonding statuses. From optical surveys of the SEM pictures, gaps of 5 to 50 μm were observed all around the margins of cavities when neither

Fig. 4  Longitudinal change of the average exposed enamel height of P30.

Fig. 5  Longitudinal change of the average exposed enamel height of OCC.
etching nor bonding agent was employed (Group III). However, when appropriate etching and bonding agents were used (Group I), no marginal gaps could be detected in most cases. Those detected were in the range of 1 to 3 μm.

DISCUSSION

It is generally accepted that appropriate etching and bonding treatments lead to good marginal adaptation and excellent marginal integrity9-11). Furthermore, Retief and Denys18) reported that when enamel was properly etched, microleakage did not exist at enamel cavosurface margins. The results of the present study showed broad gap formation occurred when a proper adhesive treatment was not given. However, SEM observation revealed that marginal gaps formed around restorations even when the manufacturers' recommended adhesive treatments were used. Even when no gap is formed initially, severe conditions such as occlusal and thermal stress are thought to make a number of negligible microgaps visible and problematic in the long run. Regarding Group I, gap–free percentages differed according to the bonding agents used. These differences are thought to depend on the compatibility between the resin composite and the bonding agent.

In this experiment the exposed marginal enamel height was measured from profilometric tracings. Because the exposed enamel height indirectly shows the amount of contact free area (CFA) wear, the "Leinfelder", "Moffa–Lugassy (M–L)" and "Vivadent" optical standard systems have been developed for measuring CFA wear in clinics. However, when the resin composites are filled without appropriate adhesive systems, these measuring systems would not indicate the true wear values even if the three–body wear testing machine was used. The significantly larger values in Group III compared to those of Group I suggest that the polymerized resin composite would be packed and transformed by the repeated load. If the composites adhered sufficiently to enamel or the dentin cavity wall treated by appropriate bonding systems, the loading force could be absorbed and supported by adhesion between the cavity and composites. This is the reason why the values of exposed enamel height differed significantly among the respective treatments, particularly at the early stage before 50,000 cycles. In most cases, after 50,000 cycles there were no significant differences in exposed enamel height among Groups I, II and III. These increments probably indicate the actual amount of wear of the respective resin composites due to repeated loading, but they are generally too small to detect. Although the actual effect of adhesion on wear of the restoration has not been clarified by this study, there is a possibility that good adhesion might prevent marginal microfracture formation followed by wear loss. The occlusal load is supposed to induce repeated movement or vibration of the unbonded marginal ends. Since the ends are always hitting each other during load–cycling, marginal fractures will tend to occur from this weakened location. Kubo19) has reported that under load testing, marginal fractures occurred sooner among specimens receiving neither etching nor bonding treatment than in those with both treatments. Although the substantial loss brought by these fractures might be regarded as CFA wear, further study is needed concerning the distinction between fracture and wear loss.

Although the results of this experiment showed that mechanical loading had a significant
effect on the marginal integrity, there have also been reports showing no statistically
significant change in the marginal integrity of restorations after load-cycling\textsuperscript{20,21}. Munks-
gard and Irie\textsuperscript{21} found that no gaps formed after load-cycling when an adequate adhesive was
used. On the other hand, Jørgensen \textit{et al.}\textsuperscript{22} showed that occlusal loading increased the
amount of deformation at the margins of composites. In addition, Mandras \textit{et al.}\textsuperscript{23} demonstrated that occlusal stress significantly increased microleakage at the tooth/restoration
interface. Since repeated loading enlarges horizontal gaps as well as vertical steps, it must
again be emphasized that adequate adhesive treatments, following manufacturers’ recom-
mendations, should be employed for clinical success.

\textbf{CONCLUSION}

Although good adhesion was obtained with appropriate etching and bonding treatments,
marginal seals certain of long-term clinical success could not be achieved under repeated
masticatory load. However, the present study demonstrates that good adhesion decreases
the marginal degradation which is exhibited as the exposed enamel height at the tooth/
restoration interface after load-cycling.

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より導入されたアミノ基及びグルタルアルデヒドにより
表面に固定され、被膜表面の密度は約1 μg・cm⁻²であることが明らかとなった。また、pFNを表面に修飾した
合金上では、研磨状態の試料と比較して、ヒト歯周由来
の線維芽細胞はより扁平に伸展しており、細胞の伸展が
促進されていることが確認された。以上の結果より、機
能性タンパク質を金属表面に修飾し、金属-細胞間の相
互作用を制御することにより、バイオアクティブな金属
インプラントの開発が可能であることが示唆された。

機能性タンパク質薄膜によるインプラント用金属表面の化学修飾
（第2報）化学修飾したNiTi合金の耐食性
遠藤一彦
北海道医療大学歯学部歯科理工学講座
機能性タンパク質を表面に化学修飾したインプラント
用NiTi合金の耐食性を生理食塩水中ならびに細胞培養
液で評価した。アミノ酸コンとグルタルアルデヒドを
架橋剤として用い、ヒト血漿フィブロネクチン(pFN)を
表面に固定化した試料のアノード分極挙動と腐食速度を
電気化学的手法を用いて調べた。また、NiTi合金上に生
成した酸化物被膜の組成、厚さ、ならびに表面に吸着し
たタンパク質をX線光電子分光装置を用いて調べた。不
働態保持電流密度は、血清タンパク質の存在により増大
した。これからの種々の方法で得られる試料は、酸化物
の耐食性が大きく影響されるため、本表面処理法は、金
属表面に生体活性を付与すると同時に耐食性を向上させるため、バイオアク
ティブな金属インプラントの開発に有用と考えられる。

コンポジットレジンの歯質接着性が辺線の劣化に及ぼす影響
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†Department of Biomaterials, School of Dentistry, University of Alabama at Birmingham

コンポジットレジンの歯質接着性を変えることによ
り、繰り返し負荷後、窩洞辺線部の劣化度にどのような
影響がみられるのかを検討した。接着条件として、エッ
チングおよびポーディング処理の有無や種類を変えて1
級窩洞にコンポジット充填を行い、40万回の繰り返し荷
重負荷後、辺線部の露出エナメル質の幅を劣化度として
測定した。その結果、エッティングおよびポーディング処
理を併用した場合は、そのどちらかまたは両者を省略し
た場合と比べ、1種のコンポジットレジンを除いて露出
エナメル質の幅は有為に小さかった。辺縁でのSEM観察
でもほぼ同様の所見が得られた。いずれの接着条件にお
いても、劣化度は最初の5万回の間は最も大きかったが、
繰り返し回数とともに減少した。