Evaluation of Enamel Acid Resistance Acquired under a Temporary Esthetic Coating Material

Izumi IWAYA, Yoshiharu MUKAI, Hiromi FUKUKAWA and Toshio TERANAKA

Abstract: In this study, we assessed the acid resistance of bovine enamel coated with a temporary fluoride-releasing flowable composite resin (BeautiCoat: BC) following vital bleaching with the HiLite (Hi) system. Each tooth received nine Hi applications before BC was applied. The teeth were stored in remineralization solution for 10 days. BC was then removed and the specimens underwent acid resistance testing for 10 days. The mineral profile and integrated mineral loss (IML) were obtained using transversal microradiography. Subsurface lesions were present in all specimens; however, the mean IML of specimens in the HiBC (no-primer) group was significantly lower than that in the Hi-alone group. On the other hand, the HiBC with primer group showed a similar IML to the Hi group. This suggests that the remineralization solution penetrates into the micro-spaces between the enamel and BC when the primer is not used. In addition, fluoride released from the surface pre-reacted glass-ionomer filler contained in BC may disperse in the micro-spaces and adhere to the enamel surface, thus inhibiting the progression of lesions.

Key words: Acid resistance, Whitening, Coating material, Fluoride

Introduction

Recently, the demand for tooth whitening has rapidly increased; consequently, materials for esthetic dental treatment are being diversified. Demand for office bleaching of vital teeth is rising because this method does not require the removal of healthy tooth structures and the whitening effect can be achieved in a short period of time. On the other hand, researchers have reported some disadvantages: postoperative hypersensitivity\(^1,2\), the dissolution of mineral elements\(^3,4\), and breakdown of organic elements\(^5\), caused by an increased frequency of bleaching, in addition to the demineralizing effect of acid\(^7\).

Recently, BeautiCoat (BC; Shofu, Kyoto, Japan) (Table 1) was commercially offered as a white fluoride-releasing flowable composite resin that is used as a temporary esthetic material to coat tooth surfaces. By applying it to the entire tooth surface, it works not only as an esthetic coating material for whitening, but also as a temporary protective material until the tooth surface is stabilized after bleaching with HiLite (Hi; Shofu, Kyoto, Japan) or other bleaching agent. In addition, it is expected that the enamel will acquire acid resistance due to fluoride release that takes place while this material is applied temporarily. In this study, we evaluated the acid resistance of enamel surfaces coated with BC using a demineralizing system in vitro.

Materials and Methods

1. Preparation of specimens

Thirty-five extracted bovine incisors were used. Enamel blocks (6 × 10 × 3 mm) were cut using a diamond disk. The surfaces of the blocks were sequentially smoothed using 1,500 and 2,000 grit waterproof abrasive paper. A rectangular window (2 × 3 mm) was outlined on the enamel surface of each specimen, and the remainder of the tooth surface was painted with acid-resistant var-

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nish. The thirty-five specimens were randomly divided into five groups.

2. Treatment protocol
The treatment procedure for each group (n = 7) was as follows (Fig. 1).
1. Control group: Each specimen was stored for 10 days at 37°C in 20 mL of remineralization solution (1.5 mM CaCl₂, 0.9 mM KH₂PO₄, 130 mM KCl, 20 mM Hepes, pH 7.0) that simulates human saliva.
2. Hi group: Hi application was performed following the manufacturer’s instructions. In brief, one scoop of powder and three drops of liquid were mixed, and the resulting paste (pH 4.1) was applied onto the surface of each specimen to a thickness of 1–2 mm using a special brush. Five minutes after the start of mixing, Hi was irradiated using a visible-light curing unit (JET LITE 3000, J. Morita, CA, USA) for 30 seconds. After two minutes, the Hi was removed with a wet cotton swab. Each treatment consisted of three applications of Hi. Finally, specimens were rinsed and stored in deionized water (DW) at 37°C. Each specimen received one treatment per week for a total of three treatments. The specimens were then stored in remineralization solution for 10 days at 37°C.
3. BC (+ pr) group: Primers A and B provided with the BC kit (Table 1) were mixed (pH 1.7), applied to the surfaces of specimens, and air-blown for three seconds. BC was then applied and photo-polymerized for 30 seconds. The un polymerized surface layer was removed with gauze and specimens were stored in remineralization solution for 10 days at 37°C.
4. Hi/BC (+ pr) group: This group underwent Hi application in the same manner as the Hi group (one treat-

![Table 1 Composition of BeautiCoat](image)

<table>
<thead>
<tr>
<th>Primer A</th>
<th>Water, Acetone</th>
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<tbody>
<tr>
<td>Primer B</td>
<td>6-MHPA, Ethanol</td>
</tr>
<tr>
<td>Paste</td>
<td>Bis-GMA, TEGDMA, Photo Initiator, S-PRG filler</td>
</tr>
</tbody>
</table>

1) 6-MHPA: 6-methacryloxyhexylphosphonic acid acetate
2) Bis-GMA: 2,2-bis [4-(2-hydroxy-3-methacryloxy-propoxy) phenyl] propane
3) TEGDMA: triethylene glycol dimethacrylate
4) S-PRG filler: surface reaction-type pre-reacted glass-ionomer filler

![Fig. 1 Experimental procedure](image)

* : A single Hi treatment consists of three repetitions of this procedure. Each specimen received one treatment per week for a total of three treatments. Specimens were stored in deionized water after each treatment.
ment per week for a total of three weeks). The specimens were then rinsed with DW for one minute, and primer and BC were applied. The specimens were stored in remineralization solution for 10 days at 37°C.

5. Hi/BC group: The specimens were treated in the same manner as the Hi/BC (+pr) group, except that no primer was applied.

After the 10-day remineralization period, all of the BC coating was removed from the total specimens with an explorer.

3. Acid resistance test

All specimens were tested for acid resistance following the method of Ingram et al. Each sample was fixed to the bottom of a plastic container and covered with 20 ml of 8% methyl cellulose gel (Methocel MC gel, Fluka, Buchs, Switzerland), to which 20 ml of 0.1 M lactic-acid solution (pH 4.6) was added. Specimens were demineralized for 10 days at 37°C, followed by sequential dehydration with ethanol and propylene oxide.

4. Transversal microradiography

Each specimen was embedded with Spurr’s embedding resin (Taab, Berkshire, UK), and three sections (150 ± 10 μm) perpendicular to the testing surface were cut in the direction of the tooth axis using a diamond-coated wire-sectioning machine (Well 3242, Walter Ebner, Mannheim, Germany).

Each section was placed on a perspex holder and covered with a thin polyester sheet. We then radiographed each section on a high-resolution glass film plate (Konica Minolta, Tokyo, Japan) with a nickel-filtered Cu-Kα source operating at 25 kV and 15 mA for 20 minutes (PW 3830, Spectris, Surrey, UK) with a 13-step aluminum wedge ranging from a 0 to 300-μm thickness. Radiographic images of the sections and the aluminum step wedge were analyzed using a microscope/video camera/microcomputer setup and TMR2000 software (Inspektor, Amsterdam, The Netherlands). Data obtained were the mineral content profiles of the lesions and integrated mineral loss (IML, vol% × μm).

5. Statistical analysis

We statistically analyzed the experimental results with one-way ANOVA and Duncan’s multiple range test (SPSS, Ver. 10.1) (p < 0.05).

Results

Fig. 2 shows representative microradiographs of each group. Thicker surface layers (arrowhead) were observed in the three groups in which BC was applied than in the control and Hi groups. The Hi/BC group showed a thicker surface layer with marked radiopacity.
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The group showed higher mineral concentrations than the Hi/BC (+ pr.) group, and deminer-arlzation was effectively inhibited.

Fig. 3 Average mineral profiles of each group.
The BC-coated groups [BC (+ pr.), Hi/BC (+ pr.), Hi/BC] displayed a greater inhibition of demineralization in the body of the lesion than control and Hi groups.
The Hi/BC group retained higher mineral concentrations than the Hi/BC (+ pr.) group, and deminer-alization was effectively inhibited.

Table 2 Mean mineral loss values

<table>
<thead>
<tr>
<th>Group</th>
<th>IML, vol% × μm</th>
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</thead>
<tbody>
<tr>
<td>Control</td>
<td>3,320.3 (424.3) a</td>
</tr>
<tr>
<td>Hi</td>
<td>3,366.8 (302.1) a</td>
</tr>
<tr>
<td>BC (+ pr.)</td>
<td>2,366.3 (634.9) b</td>
</tr>
<tr>
<td>Hi/BC (+ pr.)</td>
<td>3,033.5 (691.3) a, b</td>
</tr>
<tr>
<td>Hi/BC</td>
<td>2,490.6 (757.2) b</td>
</tr>
</tbody>
</table>

Mean (± SD), n = 7
Values with the same letters are not significantly different (p > 0.05).
BC (+ pr.) and Hi/BC groups demonstrated more marked acid resistance than the control and Hi groups.

The Hi/BC (+ pr.) group displayed a wider lesion (asterisk) with lower radiopacity on the surface layer than the Hi/BC group.

Fig. 3 shows the average mineral profile of each group. The BC (+ pr.), Hi/BC, and Hi/BC (+ pr.) groups showed higher mineral contents in the body of the lesions than the Hi group. The Hi/BC group exhibited a higher level of mineral vol% on both the surface layer and in the body of the lesion than the Hi/BC (+ pr.) group.

Table 2 shows the average mineral loss for each group. There was no significant difference between the control and Hi groups. However, the inhibition of demineralization was significantly higher in the BC (+ pr.) and Hi/BC groups than in the control and Hi groups (p < 0.05).

Discussion

The demand for esthetic dentistry procedures has greatly increased in recent years. The Hi system consists of a mixture of catalyst powder and a 35% hydrogen peroxide solution, which is 10 times more concentrated than solutions used in home-bleaching products. Because of the low pH of the mixture (pH 4.1), some adverse effects have been reported, including dissolution of the inorganic contents and the reduction of tooth-surface hardness. Coating the treated surface after bleaching may provide beneficial effects of stabilizing the enamel surface and providing acid resistance. BC is a composite resin containing surface pre-reacted glass- ionomer (S-PRG) filler. The filler’s particles are produced using pre-reacted glass-ionomer (PRG) technology. With this technology, a glass-ionomer phase is formed on glass particles through the reaction of fluoroalumi-
osilicate glass and a polycarboxylic acid in the presence of water. It was reported that a composite resin containing S-PRG filler released fluoride. On the first day, the rate of fluoride release was 9.32 µg/cm²/day, and 2.98 µg/cm²/day after 60 days\(^{13}\). Kamijo et al.\(^{14}\) reported that the amount of fluoride released from four experimental denture base resins containing 5, 10, 20, and 30 wt% S-PRG filler were evaluated. In the results, specimens with 5, 10, 20, and 30 wt% of S-PRG filler released 0.70, 1.00, 1.88, and 2.91 µg/cm²/day of fluoride on one day, respectively. Further, S-PRG filler was also found to release inorganic elements such as Al, Si, and Sr\(^{15}\).

In this study, Hi/BC and BC (+ pr.) groups exhibited higher levels of acid resistance than the control and Hi groups. We speculate that when specimens coated with BC and stored in remineralization solution, which simulates human saliva, fluoride from the S-PRG filler is released into the micro-spaces between the enamel and BC. We analyzed the fluoride intensity on the enamel surface of the Hi/BC group using an electron probe microanalyzer (EPMA 8705, Shimadzu, Kyoto, Japan) before the acid resistance test. The fluoride intensity of the Hi/BC-treated surface was almost twice that of the control surface (data not shown). Although enamel was dissolved during the subsequent storage in demineralization solution, it is possible that fluoride deposited on the surface was dissolved and released again and contributed to the improvement in acid resistance. On the other hand, there was no difference between the control and Hi groups in their acid resistance. The following reasons for this result were considered: 1) although the surface area of the Hi group specimens might be partially demineralized by the total of nine Hi applications, the degree of demineralization would be comparatively mild; 2) storing the Hi specimens in the remineralization solution for 10 days contributed to the recovery of the tooth surface, so no effect on acid resistance was observed.

We evaluated the effect of using primers before BC application in specimens treated with Hi. The no-primer group exhibited a somewhat greater inhibition of demineralization than the primer-using group. The pH of the primer is 1.7, and it seems to have worked as an etching material on the enamel surface. The etched enamel surface would mean that the demineralization solution could penetrate to a deeper depth. Furthermore, it is widely known that adhesion to enamel is achieved by the application of phosphoric acid or acidic primer\(^{16,17}\). In other words, this may be because the absence of an acidic primer may have allowed the remineralization solution to more easily penetrate between the BC and enamel, thus enhancing the uptake of fluoride released from the S-PRG filler.

**Conclusion**

Our study confirmed that BC coating facilitates the effective acid resistance of underlying Hi-bleached enamel *in vitro*.

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審美性歯面コート材下エナメル質に獲得される耐酸性

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概要：ホワイトニング薬の歯面は脱灰を受けやすいという報告がある。BeautiCoat（以下 BC と示す）は、S-PRGフィラー配合の白色系フッ素含有性フロアブルレジンであり、歯面全体を薄くカバーすることによりホワイトニングとしての役割をもった暫定的な審美回復材料としての特徴をもつ。また、同時に HiLite（以下 Hi と示す）等でホワイトニングを行った歯面が安定化するまでに使用する臨時的なホワイトニング回復材としての性格も兼ね備えている。この研究では、BC で被覆した Hi 处理エナメル質の耐酸性を測定した。35本のウシ下顎中切歯歴面より切り出したエナメル質片を 2×3 mm の試験面を残してパッケージした後、以下の 8 群、すなわち、Control 群：非処理の試験片を人工唾液（15 mM CaCl₂、0.9 mM KH₂PO₄、130 mM KCl、20 mM Hepes、pH 7.0）に浸漬した群、Hi 群：試験面に Hi を 3 回適用後、人工唾液に浸渍した群、BC (+pr) 群：処理の前にプライマーを塗布後、BC 处理し人工唾液に浸漬した群、Hi/BC (+pr) 群：Hi を 3 回適用した試験片にプライマーを塗布後、BC 处理し人工唾液に浸漬した群、Hi/BC 群：Hi を 3 回適用した試験片に BC 处理し人工唾液に浸漬した群に分類した。人工唾液に 10 日間浸漬後、BC を一塊として除去し、脱灰液（0.1 M乳酸 2 塩化物 pH 4.6）に 37℃、10 日間浸漬した。これらをレジン包埋後、150 μm の薄切片を作製し、ミエラルプロファイルと病巣のミエラル喪失量（IML）を求めるため、transversal microradiography を用い、ミエラルプロファイルを作成した。耐酸性能の比較は、ミエラル喪失量（IML）を測定することにより検討した。ミエラルプロファイルでは、Control 群、Hi 群に比較し、他の BC 处理群は、病巣体部での脱灰抑制傾向が認められた。Hi/BC 群は Hi/BC (+pr) 群に比較し、ミエラル密度が高く維持され、脱灰がさらに抑制される傾向を示した。IML の比較では、BC (+pr) 群と Hi/BC 群は Control 群、Hi 群に比較し、脱灰がより抑制された（p<0.05）。エナメル質が BC で覆われた場合、特にプライマー非使用下では唾液をシミュレートした人工唾液への浸漬によってエナメル質と BC 間の微小空間領域で S-PRG フィラーから徐放されたフッ化物の一部がエナメル質表面に沈着すると考えられる。このフッ化物が病巣進行の抑制に寄与し、その後脱灰液への浸漬によってエナメル質は溶解し、表面に沈着したフッ化物から遊離したフッ化物イオンがエナメル質から遊離したカルシウムおよびリン酸イオンと相まって再石灰化が生じ、結果として脱灰が抑制されたものと考えられる。以上の結果から、Hi 群で被覆されたエナメル質を BC で一定期間被覆することにより、適用下エナメル質に耐酸性を付与できることが確認された。

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