Resistance of dentin coating materials against abrasion by toothbrush

Iori GANDO¹, Meu ARIYOSHI¹, Masaomi IKEDA², Alireza SADR³, Toru NIKAI¹ and Junji TAGAMI^{1,3}

¹ Cariology and Operative Dentistry, Oral Restitution Department, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan

² Clinical Oral Science, Department of Oral Health Care Sciences, Faculty of Dentistry, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan

³ Global Center of Excellence (GCOE) Program; International Research Center for Molecular Science in Tooth and Bone Diseases, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan

Corresponding author, Toru NIKAI; E-mail: nikai@tmd.ac.jp

Thin-film coating of root dentin surface by all-in-one adhesives has been shown to be an effective option to prevent root surface caries. The purpose of this study was to investigate the wear resistance against toothbrush abrasion of two all-in-one coating materials; Shield Force (SF) and Hybrid Coat (HC). Bovine dentin surfaces were covered with one of the coating materials; SF or HC. After storage in water for 24 h, the testing surface was subjected to the toothbrush abrasion test up to 50,000 cycles either in water or toothpaste slurry. The remaining thickness of the coating material was measured using SEM. Toothpaste slurry significantly increased rate of tooth brush abrasion of the coating materials. While SF and HC wore at a similar pace under toothbrush abrasion, SF had a thicker coat and could protect dentin longer, up to 50,000 cycles.

Keywords: All-in-one adhesive, Coating material, Wear resistance, Toothbrush, Surface roughness

INTRODUCTION

The growing geriatric population in many developing or developed countries is expected to retain their teeth into an old age. However, soft tissue recession due to aging, traumatic toothbrush habits, periodontal disease or surgical periodontal treatment will unavoidably result in exposure of susceptible root surface¹ and higher risk of root caries and dentin hypersensitivity. Prevention of primary root caries is attempted mainly by the daily use of fluoride-containing mouth rinse^{2,3} or toothpaste^{2,4}. While this treatment is cost-effective and less stressful to the patient, success depends upon the compliance of patients. In this regard, simple single-visit methods to protect the exposed root surfaces from caries attack in the long-term are advantageous⁵.

Thin-film coating materials have been developed through the technology of all-in-one adhesive systems; the coatings were used to seal the exposed dentin after cavity and crown preparation⁶. It was also reported that the thin-film coating with an all-in-one adhesive system could prevent marginal leakage beneath full cast crown⁷ and improve the bond strength of resin cement to dentin⁸⁻¹⁰. While the use of various resin-based restorative materials⁵ have been suggested as protective layers for exposed dentin surfaces, a durable thin-film coating should bear advantages over conventional restoration procedures in terms of minimal invasiveness, ease of application and reduced complications associated with restoration margins¹¹. In addition, application of the all-in-one adhesives to dentin creates the acid-base resistance zone beneath the hybrid layer¹², which is a reinforced dentin, so called “Super Dentin” to resist against caries attack¹³. Thin-film coating of root dentin

surface by using an all-in-one adhesive reportedly prevented demineralization by acid attack to the root dentin surface and reduced biofilm attachment formation on to surface^{14,15}. Covering the exposed dentin surfaces using the coating materials and sealing tubules can also potentially reduce dentin hypersensitivity¹⁶. The coating should be the most beneficial option for the patients, as they can be performed as a single-visit treatment, provided that their effects last for a long-time.

On the other hand, toothbrushing may be responsible especially for wear of cervical restorations¹⁷. The wear resistance of the coating materials on root surface is of great clinical significance; the success of this simple treatment literally depends on the durability of the coating materials on the dentin surface. Although a number of studies have described wear resistance of restorative materials to toothbrush abrasion¹⁸⁻²⁰, a limited number of studies have been conducted on the wear of the coating materials. Therefore, the purpose of this study was to investigate the wear resistance of two all-in-one coating materials against toothbrush abrasion. In addition, the hardness and surface roughness of the coating materials were evaluated. The null hypothesis of this study was that toothbrush wear of the coating materials were not affected by materials, toothbrush abrasion cycles and abrasion media.

MATERIALS AND METHODS

Materials used in this study

Two coating materials; Shield Force (SF, Tokuyama Dental, Tokyo, Japan) and Hybrid Coat (HC, Sun Medical, Moriyama, Japan) were used in this study. The compositions of these coating materials are shown in

Table 1. SF is a single-bottle all-in-one adhesive system, which contains three dimensional self-reinforcing (3D-SR) monomer and glass fillers. The several phosphate groups of the 3D-SR monomer are capable to interact with calcium at multiple sites and form ionic bonds. SF has been developed as a coating material for reduction in dentin hypersensitivity. HC is also an all-in-one adhesive system, which is composed of an adhesive and a sponge. The adhesive contains 4-methacryloxyethyl trimellitate anhydride (4-META) as a functional monomer. The sponge supplied by the manufacturer contains co-activators, such as p-toluene sulfinate salt and amine, which promote interfacial polymerization. HC was developed as a resin coating material to protect dentin prepared for indirect restorations⁸⁾.

Specimen preparation

The specimen preparation procedure is schematically illustrated in Fig. 1. One-hundred twenty eight freshly extracted bovine incisors, stored frozen were used as the test substrates. A flat superficial dentin surface was prepared by trimming labial enamel using a model trimmer (Y-230, Yoshida, Tokyo, Japan) under copious water lavage. The roots were then removed and each tooth specimen was trimmed to obtain a dentin block 8×10×3 mm in size. The blocks were embedded in a methylmethacrylate resin (Unifast III, GC, Tokyo, Japan). Afterwards, the specimen surface was wet-polished with #4000-grit silicon carbide paper followed by rinsing in deionized water for 1 min to obtain a smooth dentin surface as a base for measuring thickness of the coating material. Following this, the specimens were randomly divided into two groups. The dentin surface in each group was covered with a single coat of one of the coating materials (SF or HC) using the sponge supplied in the package, according to the manufacturers' instructions. Table 1 summarizes the compositions and application instructions for each material. A halogen

light curing unit at an output intensity of 500 mw/cm² (Optilux 501, Demetron-Kerr, Danbury, CT, USA) was used to polymerize the coating material. After storage in distilled water at 37°C for 24 h, the surfaces of the specimens were covered by a masking tape (Plastic Tape, Asahipen, Osaka, Japan) with the exception of the central 2×10 mm window. The masking tape protected the original flattened surface of the coating material from abrasion for thickness measurements after abrasion.

Toothbrush abrasion test

Testing surface of each specimen was subjected to the toothbrush abrasion test. The test was performed in an automatic brushing machine (K236, Tokyo Giken, Tokyo, Japan). The coated specimens were divided into two subgroups; the specimens were abraded either in toothpaste slurry prepared by mixing 100 g of a regular toothpaste (White and White, Lion, Tokyo, Japan) in 700 mL of tap water (S), or in 700 mL of tap water (NS). Prospec-Plus (GC, Tokyo, Japan) toothbrushes (23 mm in length, 8 mm in width, medium hardness, 9.5 mm in filament length) were used. The toothbrushes were fixed at the holder of the brushing machine, allowing movement of the brush head parallel to the coated surface of the specimens at a rate of 100 cycles per min. Each cycle consisted of two straight 10 mm strokes along the longer side of the exposed window under a load of 300 g placed on the center of the holder. Eight specimens were prepared respectively for each period; 1,000, 10,000, 20,000 or 50,000 cycles. The toothpaste slurry was renewed after every 10,000 cycles. After each period, the specimen was removed from the storage container, and thickness of the coating material was confirmed as described below.

Thickness measurement

After the toothbrush abrasion test, the masking tape on the specimens was peeled off and the coated surface

Table 1 Coating materials used in this study

Code	Brand Name	Manufacturer	Batch No.	Composition	Application instructions
SF	Shield Force	Tokuyama Dental, Tokyo, Japan	051060	3D-SR monomer, HEMA, TEGDMA, Bis-GMA, Glass filler, Isopropyl alcohol, Photoinitiator, Water	Apply the coating material for 20 s Mild air-blow for 5 s and strong air-blow for 5 s Light-cure for 10 s
HC	Hybrid Coat	Sun Medical, Moriyama, Japan	TE3	Liquid: 4-META, Monomethacrylate, Photoinitiators, Stabilizers, Polyfunctional acrylate, Acetone, Water Cata-brush: p-toluene sulfinate salt, Amine	Apply the coating material for 20 s Mild air-blow for 5 s and strong air-blow for 5 s Light-cure for 10 s

3D-SR monomer: three dimensional self-reinforcing monomer; HEMA: 2-hydroxyethyl methacrylate; Bis-GMA: Bisphenol-A-diglycidyl methacrylate; TEGDMA: triethyleneglycol dimethacrylate; 4-META: 4-methacryloyloxyethyl trimellitate anhydride

Fig. 1 Schematic illustration of sample preparation for toothbrush abrasion test in the study.

was rinsed under running water for 15 s. The specimen was then sonicated in deionized water for 5 min. A resin cement (Bistite II, Tokuyama Dental, Tokyo, Japan) was applied on the surface of the specimen in order to prevent wear the outer edge of the coating material during the polishing procedures. After curing of the resin cement, the specimens were embedded in epoxy resin (Epon 815, Nissin EM, Tokyo, Japan).

Each specimen was then sectioned perpendicular to the coating material-dentin interface using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under running water to yield three slices. The cross-sectional surfaces of the slices were polished consecutively up to #1200-grit SiC paper, followed by diamond pastes (6, 3, 1, and 0.25 μm ; DP-Paste, Struers A/S, Copenhagen, Denmark). After each step, the specimens were cleaned ultrasonically in distilled water for 2 min. Finally, the fine polished specimens were dried at room temperature for 24 h and gold sputter-coated for SEM (JSM-5310LV, JEOL, Tokyo, Japan) observations.

The remaining thickness of the coating material after toothbrush abrasion was measured using SEM. Two points selected from abrasion area were each 100 μm from the midpoints. Two points selected from the non-abrasion area were each 100 μm from the boundary between abrasion area and non-abrasion area (Fig. 2). The mean value of thickness of the coating layer was calculated for each cycle ($n=8$).

Nanoindentation test

Cross-sectional hardness of each coating material before abrasion was determined using a nanoindentation system (ENT-1100a, Elionix, Tokyo, Japan). Three additional specimens were prepared in the same manner as described for thickness measurement, with the exception of final dehydration and the sputter-coating step.

The coating materials were indented using a

Fig. 2 The remaining thickness of coating materials was measured using SEM at the two points, selected from non-abrasion area and abrasion area each (bold arrows).

Berkovich diamond indenter with an enclosed angle of 130° at a constant loading rate of 0.1 mN/s up to a maximum load of 1.5 mN. The temperature of the testing chamber was held constant at 28°C . Ten indentations were made at the midpoint of the coating layer for each specimen at 5 μm intervals ($n=30$).

Surface roughness analysis

Change in surface roughness of the coating materials before/after toothbrushing was evaluated. The specimen preparation was carried out in the same manner as described for the toothbrush abrasion test. The non-abraded specimens and the abraded specimens subjected to toothbrushing for 10,000 cycles were used for this purpose. In addition, bovine dentin specimens polished with #4000-grit SiC were used as a smooth dentin surface control for the roughness comparisons. Surface roughness of each specimen was analyzed along three lines by using a confocal laser scanning microscope (ILM21H/W, Lasertec, Yokohama, Japan) under $\times 200$ magnification. Three specimens were tested for each group ($n=9$).

Statistical analysis

All data were subjected to Kolmogorov-Smirnov test to confirm the normality required for the parametric comparisons. Data of thickness within each coating material were analyzed by two-way analysis of variance (ANOVA) to examine interaction between abrasion media and number of toothbrush abrasion cycles, followed by the *t*-test with Bonferroni correction. The relationship between the test cycles beyond the initial 1,000 cycles and the thickness of the coating layer under each abrasion media was analyzed by regression analysis. The slopes of linear regression were compared between the two coating materials by analysis of covariance (ANCOVA).

The data for nanoindentation hardness of the coating materials were analyzed by a Student's *t*-test. The data for surface roughness of the coating materials were analyzed by one-way analysis of variance (ANOVA) and the *t*-test with Bonferroni correction. All statistical procedures were performed in the SPSS software (SPSS, Chicago, IL, USA) at a 95% level of confidence ($\alpha=0.05$).

RESULTS

Thickness of the coating materials

Mean thickness of the coating materials was shown in Fig. 3. The typical SEM observations of the coating materials after 10,000 cycles of abrasion were revealed in Fig. 4. Since the coating in HC/S group was worn away before 50,000 cycles of toothbrush abrasion and dentin surface was exposed, thickness of the coating material could not be measured. Therefore, this group had to be excluded from the statistical analysis.

The average thicknesses of SF and HC before toothbrush abrasion testing were approximately 12 μm and 6 μm respectively. However, thickness of the coating

material decreased with increased cycles of toothbrushing ($p<0.05$). Wear resistance of the coating materials was significantly influenced by the abrasion media, being water or slurry with the toothpaste ($p<0.05$).

The tendencies for the decrease of thickness in the coating materials were divided into two patterns; 0–1,000 cycles and 1,000–50,000 cycles, which may be due to the initial presence of the oxygen-inhibited

Fig. 3 Relationship between the thickness of the coating materials and the cycles of toothbrushing.

The graph represents the mean and standard deviation of thickness, together with a simple linear regression of the four groups beyond 1,000 cycles ($n=8$)*. n.d.; HC/S was worn out and could not be measured at 50,000 cycles.

SF/NS; $y=-0.059x+11.1$, $R^2=0.99$ ($p<0.0001$);

SF/S; $y=-0.096x+10.5$, $R^2=0.96$ ($p<0.0001$);

HC/NS; $y=-0.038x+4.6$, $R^2=0.86$ ($p<0.0001$);

HC/S; $y=-0.184x+4.2$, $R^2=1.00$ ($p<0.0001$)

Fig. 4 Cross-sectional SEM micrographs showing the coating materials after 10,000 cycles of brushing ($\times 500$).

The dashed-lines indicate the border of the tape covering non-abrasion area as the original surface. At the boundary of abrasion area and non-abrasion area, the surface of coating material was worn in slope rather than a sharp step. The thickness of coating after abrasion (between two blank arrows) is visibly lower than that before abrasion (between two bold arrows). SF (Shield Force) consistently showed higher thickness compared to HC (Hybrid Coat). D: dentine, R: resin cement (for SEM preparation).

unpolymerized layer on the cured coating materials (Fig. 3). ANCOVA analysis of the regressions in Fig. 3 indicated that tendencies of the toothbrush wear of each coating material abraded under any of the abrasion media were not significantly different ($p>0.05$).

Nanoindentation test

The mean nanoindentation hardness values of the coating materials were listed in Table 2. The values for SF and HC were 295.4 and 283.9 MPa, respectively, in which there was no significant difference between the two coating materials ($p>0.05$).

Surface roughness

The mean surface roughness values of the coating materials and polished bovine dentin were summarized in Table 3. In the coating groups, one-way ANOVA and *t*-test with Bonferroni correction indicated that surface roughness of the coating surface after toothbrush abrasion for 10,000 cycles was significantly higher than that of the original surface before abrasion test ($p<0.05$). However, there was no significant difference in surface roughness between SF and HC either before or after abrasion ($p>0.05$). Surface roughness values of SF and HC after toothbrush abrasion were not significantly different from those of bovine dentin polished with #4000-grit SiC ($p>0.05$).

DISCUSSION

A clinical strategy to prevent root caries has not been established yet. The application of the coating material on root surface is a promising approach to seal and protect the exposed root dentin surfaces. Therefore, this study investigated the durability of these potentially

protective coatings.

A number of toothbrushing simulators have been proposed in the literatures with different principles in the fundamental design^{21,22}. However, a consensus about the design and parameter setting for predictable toothbrush abrasion in the oral environment has not been reached. The previous clinical studies reported that the mean brushing force values applied by subjects were 2.3 ± 0.7 N²³, 267 ± 73 g²⁴, 2.96 ± 0.8 N vertical force²⁵ and 330 ± 109 g²⁶; therefore, the 300 g vertical load used as the toothbrushing force in this study was clinically relevant.

Cementum is the first layer to be encountered when the root surface is exposed to the oral environment. However, the cementum formation exists as a superficial layer on the root and is easily peeled off by intensive root planing during the treatment of periodontal diseases or by toothbrushing²⁷. The vulnerability of the cementum layer causes the underlying dentin to be prone to exposure, hence increasing the risk of dentin hypersensitivity and root dentin caries formation. For this reason, the chief focus of this study was on surface coating of dentin with a view to protecting exposed root dentin.

From the current results, thickness of the coating material decreased in both SF and HC in proportion to the frequency of toothbrush abrasion cycles. However, no significant difference was observed in abrasion regression slopes between the two materials under each test condition, which confirmed similar wear tendencies for both coatings. Generally, in the resin-based materials with additional filler particles, the filler particles in close proximity protect the softer resin-matrix from abrasion and wear²⁸. Therefore, a higher toothbrush wear resistance was expected in SF than in HC, because SF contains glass fillers, whereas HC does not contain filler particles. On the other hand, another study reported that coating materials with filler particles were considered to show complicated abrasion patterns because abrasion occurred in the resin matrix²⁹. Furthermore, similar hardness values were found between SF and HC in this study. While the presence of fillers is expected to contribute to the hardness of a thin-film, it should be noted that the nanoindentation test conducted at the small load of 1.5 mN projected a small area (indentation depth 500–600 nm) on the resin

Table 2 Nanoindentation hardness of the coating materials

Coating material	Hardness (MPa)
SF	295.4 $\pm$ 16.4*
HC	283.9 $\pm$ 21.0*

Values are mean $\pm$ SD; $n=30$. * indicates no significant difference ($p>0.05$).

Table 3 Surface roughness of the coating materials and bovine dentin (Ra)

	Surface roughness (μ m)	
	Before toothbrushing	After 10,000 cycle with the slurry
SF	1.28 $\pm$ 0.29 ^a	5.09 $\pm$ 1.14 ^b
HC	1.17 $\pm$ 0.31 ^a	4.62 $\pm$ 1.71 ^b
Bovine dentin (#4000-grit SiC)		4.86 $\pm$ 1.71 ^b

Values are mean $\pm$ SD; $n=9$.

Values marked by different superscript letters are significantly different ($p<0.05$).

matrix, and the resulting values could greatly depend on compositional factors affecting the quality of the resin matrix³⁰. The hardness values obtained for both coating materials were in close agreement with those reported previously for dental adhesives^{30,31}. Interestingly, there was an abrupt initial loss of coating thickness, especially in the toothpaste slurry groups, at the 0–1,000 cycles for both materials, which should be attributed to the presence of an oxygen-inhibited unpolymerized layer on the cured coating materials. Methacrylate monomers polymerize following a free-radical-induced reaction, which is strongly inhibited by oxygen diffusing from the atmosphere into curing resin. The thickness of this layer can be up to a few μm from the surface, and the degree of inhibition depends on the compositional factors such as presence of fillers and viscosity of the resin, which will in turn affect the mechanical properties of the polymer³².

The thickness of SF at the baseline was higher than that of HC. Perhaps filler particles in SF could increase the viscosity of the coating material, which may secure the film thickness of the coating material. Therefore, SF could remain on the dentin surface after 50,000 cycles of toothbrushing. On the other hand, a single coat of HC created an approximately 6 μm -thick layer, which could be easily worn away before 50,000 cycles. In addition, wear resistance of the coating materials was significantly influenced by the abrasion media (water or slurry with the toothpaste). The toothpaste contains CaCO_3 particles as an abrasive for stain removal, which enhanced friction and accelerated wear of the resin matrix of the coating materials in both SF and HC.

In the current study, dentin surface coating with SF or HC before toothbrush abrasion created significantly smoother surfaces than the dentin surfaces polished with #4000-grit SiC, which was considered to be far smoother than dentin surface achieved clinically by any root surface planing method³³. The coating after slurry abrasion did not show significantly rougher surface compared to the highly polished dentin surfaces, indicating the potential benefit of dentin surface coating in terms of surface smoothness. It has been reported that salivary pellicle absorption and biofilm formation are strongly influenced by some peculiar characteristics of the material surface such as surface roughness, surface electric charge and chemical composition^{34,35}. Increased roughness has been correlated with increased accumulation of dental plaque. Some *in vivo* studies suggested a threshold surface roughness for bacterial retention below which no further reduction in bacterial accumulation could be expected^{36,37}. Furthermore, Daneshmehr *et al.* reported that root surface coated with the all-in-one adhesive materials created smoother surfaces than ground dentin surfaces, and reduced biofilm attachment on the surfaces compared to the uncoated dentin surfaces¹⁴. Therefore, both of SF and HC appear to be effective root surface coating materials that can to some extent prevent biofilm formation.

The coating materials remained after 10,000 cycles abrasion in both of SF and HC, which is assumed to be simulating approximately one year of clinical toothbrush

wear^{19,22}. However, several other factors can adversely affect the clinical retention of coating materials on the root surface. It was reported that bonded interfaces produced by adhesive systems degraded for long time *in vivo* due to hydrolysis or enzymatic degradation of dentin organic phase³⁸. Therefore, further studies should be carried out to evaluate the long-term reliability of the root surface coating materials under the clinical setting.

CONCLUSIONS

The coating materials, SF and HC, wore at a similar pace under toothbrush abrasion, and remained after 10,000 cycles abrasion. SF has a thicker coat and therefore could protect dentin longer, up to 50,000 cycles.

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