In vitro wear behavior of restorative resin composites against bovine enamel

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The purpose of this study was to clarify the effects of fabrication method of restorative resin-based composites on its wear using enamel as antagonist teeth. Wear evaluation was performed via two-body wear test using hemispherical samples of restorative resin-based composite (abrader specimen) fabricated through direct restoration method, indirect restoration method, and CAD/CAM, and bovine enamel (substrate specimen). As a result, there was a difference in wear volume between restorative composite and bovine enamel depending on the fabrication method. Resin composite used for indirect restoration method showed more wear in both the abrader and substrate specimens. Resin composite used for CAD/CAM crowns showed greater wear volume in the abrader specimen. In conclusion, results clarified that fabrication method of restorative resin-based composite has an influence on the wear of the resin composite itself and enamel as antagonist teeth.

Keywords: Resin composite, Enamel, Wear behavior, Hardness, Surface roughness

INTRODUCTION

Recently in dentistry, metal-based restorative materials with excellent strength and ductility have been replaced with ceramics and resin-based composite materials, also with high strength but with better aesthetic properties. The strength of resin-based composite materials has been improved by increasing content of inorganic fillers and degree of polymerization. This development in physical properties of resin-based composite materials has led to the diversification of fabrication methods.

Fabrication methods of resin-based composite materials used for dental restorations involve materials for direct restoration (D-CR) where resin material is directly filled to defective cavity, materials for indirect restoration (I-CR) where metal crown is faced with resin composite, and CAD/CAM blocks (C-CR). After molding into shape, resin-based composite material requires light irradiation or heat to cure via radicals generated by the action of tertiary amine. For direct restoration, the common method to cure resin composite is through light irradiation using lights of wavelengths of around 470 nm. For indirect restoration, light irradiation and heating are performed simultaneously using special devices. For CAD/CAM type restoration, blocks undergo pressurization and heating during the manufacture process. For direct and indirect restoration, crown form is filled and cured, whereas for CAD/CAM restoration, a prefabricated block is milled for fabrication. Therefore, it is presumed that degree of polymerization of the resin component of these materials differ depending on fabrication method and the influence of these methods on the mechanical properties of restorations is a concern.

Material and antagonist wear due to occlusion is a major concern regarding dental restorations. In clinical practice, cases have been reported where all-ceramic crowns or metal-ceramic crowns caused wear on antagonist teeth; others have reported that metal-based crowns were readily worn down by antagonist teeth. For resin-based composite materials, some studies have indicated that wear of antagonist teeth is small; however, others have indicated that wear is observed in both the material and the antagonist.

For ceramic restorations, in-vitro tests indicated that less wear occurs on ceramic materials compared to metals and resin-based composite materials, however significant wear occurs on antagonist teeth. In addition, the surface roughness and fabrication method affect the wear of antagonist teeth for ceramic materials such as porcelain and zirconia. The wear of antagonist teeth for resin-based composite is significantly influenced by filler size and shape. The wear behavior of resin-based composite is influenced by filler content, size, and shape; and also the type of matrix resin. Therefore, while resin-based composites with improved mechanical strength may have reduced material wear, there is a risk of increasing antagonist wear. However, there are very few studies that have investigated the wear behavior of antagonist teeth according to fabrication method and structure of resin-based composite materials.

The purpose of this study was to clarify the effects of resin composite fabrication method on the wear of restorative resin composite itself and enamel as the antagonist teeth. The fabrication method of resin-based composite materials included D-CR, where curing was done only through light irradiation, I-CR where curing was done using a special curing/heating unit, and C-CR where CAD/CAM system was used to mill blocks. Six types of materials were prepared for each method. In addition, hardness was evaluated by investigating the
microstructure and mechanical properties of the resin-based composite materials. Two-body wear test was performed with resin-based composite material against bovine enamel and wear volume of both materials were determined to identify possible relations. The null hypotheses are as follows: 1) fabrication method of restorative resin-based composite material has no effect on the wear of resin composites and 2) fabrication method of restorative resin-based composite material has no effect on the wear of antagonist teeth.

MATERIALS AND METHODS

Two-body wear test was performed as shown in the schematic diagram Fig. 1 (a): restorative resin-based composite material was placed on top as the abrader specimen and bovine tooth enamel was placed on bottom as the substrate specimen. Six samples of resin-based composite were prepared with each fabrication method listed in Table 1.

Characterization of restorative resin-based composite materials

Specimens of restorative resin-based composite materials for microstructure observation and hardness measurements were prepared by the following methods. For D-CR, an acrylic ring with an inner diameter of 6 mm and height of 1 mm was filled with resin composite and cured with LED light-curing unit (Blue Shot, Shofu, Kyoto, Japan) for 20 s. For I-DR, similar to D-CR, an acrylic ring was filled with a resin composite and light cured for 2 min using a light-curing unit (Pearl Cure Light, Tokuyama Dental, Tokyo, Japan). For the 3 kinds of resin composites of light-heat cured type (PEE, EST, GRF), specimens were additionally heated at 100°C for 15 min in a heat curing unit (Pearl Cure Heat, Tokuyama Dental). For C-CR, each block was sectioned at thickness of 1 mm using cutting machine (Isomet 100, Buehler Japan, Tokyo, Japan). The cured or sectioned specimens were embedded with epoxy resin (Scandiplex, Fritsch Japan, Kanagawa, Japan) in 1-inch epoxy rings (EX-ring, Refine Tec, Kanagawa, Japan). After curing of epoxy resin, specimens were polished using abrasive paper 320–1200 grit with an automatic polishing machine (EcoMet 250 & AutoMet 250, Buehler Japan). Three specimens of each type of restorative resin-based composite materials were prepared.

Specimens were observed under a field emission scanning electron microscope (FE-SEM; SU 6600, Hitachi, Tokyo, Japan). Carbon coating was done prior to SEM observation. Vickers hardness was measured using a micro-Vickers hardness tester (MVF-G, Shimadzu, Kyoto, Japan) where a load of 490 mN was applied for 20 s.

Abrader specimen preparation

Abrader specimens of restorative resin-based composite materials for wear tests were designed to be hemispherical specimens with a radius of curvature of 5 mm. An impression of a 5 mm radius stainless steel ball was made with vinyl silicone rubber impression material (Examixfine regular type, GC, Tokyo, Japan) to prepare a hemispherical concave mold. Each hemispherical concave mold was filled with each D-CR material to about 1 to 1.5 mm and irradiated with a light irradiator (Blue Shot, Shofu) for 20 s. This process was repeated 3 to 4 times to obtain each hemispherical sample.

For I-CR, similar to D-CR, hemispherical concave mold was filled with resin-based composite material and cured with light-curing unit (Pearl Cure Light, Tokuyama Dental) and heat-curing unit (Pearl Cure Heat, Tokuyama Dental).

C-CR specimens were fabricated using the CAD/CAM system (CEREC 3, Dentsply Sirona, Tokyo, Japan). A hemispherical concave mold made of silicone rubber material was scanned and designed using CEREC 3; then specimens were milled from CAD/CAM resin composite blocks.

The spherical surface of specimens was polished using ultrafine diamond polishing tool (CompoMaster cup, Shofu) on a micro-motor hand piece at 5,000 rpm while maintaining spherical surface. Five specimens were prepared for each sample.

Substrate specimen preparation

Bovine tooth enamel was used as substrate specimen for wear test. Frozen bovine teeth were thawed and sectioned at the cement-enamel junction. The sectioned specimens were embedded with epoxy resin (Scandiplex, Fritsch Japan) in 1-inch epoxy rings (EX-ring, Refine Tec). After
Table 1  Resin-based composite materials used in this study

| Code | Bland                | Component*1 | Lot No. | Manufacturer                     |
|------|----------------------|-------------|---------|----------------------------------|
| BEP  | Beautifil E posterior| Glass powder, Bis-GMA, Bis-MPEPP, TEGDMA, UDMA | 101202  | Shofu (Kyoto, Japan)             |
| CME  | Clearfil® majesty® ES-2 | Silanated glass filler, Pre-polymerized organic filler, Bis-GMA, Methacrylic acid-based monomer | 840024  | Kuraray Noritake Dental (Tokyo, Japan) |
| ESQ  | Estelite Σ quick     | Silica-zirconia filler, Prepolymerized filler, Bis-GMA, TEGDMA | 074023P | Tokuyama Dental (Tokyo, Japan)    |
| FSU  | FiltekTM supreme ultra | Inorganic filler, Bis-GMA, TEGCMA, UDMA | N580079 | 3M (Tokyo, Japan)                |
| KAL  | Kalore               | Fluoro alumino silicate, Bis-MEPP, UDMA | 1406031 | GC (Tokyo, Japan)                |
| MIG  | MI gracefil          | Barium glass filler, Bis-MEPP, UDMA | 1503021 | GC                               |

Indirect restoration method, I-DR

| Code | Bland                | Component                  | Lot No. | Manufacturer                     |
|------|----------------------|----------------------------|---------|----------------------------------|
| DIA  | Dia-na               | Fine silica powder, Strontium borate glass, UDMA | 1702035 | GC                               |
| EST  | Estenia® C&B         | Surface-treated powdered glass, Surface-treated alumina-based micro filler, Urethane-based methacryl monomer, Methacrylic acid-based monomer | 6E0029  | Kuraray Noritake Dental          |
| GRF  | Gradia forte         | Silica powder, Glass powder, Prepolymerized filler, UDMA | 1606141 | GC                               |
| LUW  | Luna-wing            | Inorganic filler, Methacrylic acid-based monomer | 01091606 | YAMAKIN (Osaka, Japan)           |
| PEE  | Pearlste             | Silica-zirconia filler, Bis-MPEPP, TEGDMA, UDMA | 1182    | Tokuyama Dental                  |
| SOL  | Solidex              | Organic filler, Silica power, UDMA | 071679  | Shofu                            |

CAD/CAM method, C-CR

| Code | Bland                | Component                  | Lot No. | Manufacturer                     |
|------|----------------------|----------------------------|---------|----------------------------------|
| CES  | Cerasmart            | Silica filler, Barium glass filler, UDMA | 1405211 | GC                               |
| ESB  | Estelite block       | Silica powder, Silica-zirconia filler, UDMA, TEGDMA | 001046  | Tokuyama Dental                  |
| KAB  | Katana® Avencia® block | Alumina/silicate filler, UDMA, TEGDMA, Methacrylic acid-based monomer | 000319  | Kuraray Noritake Dental          |
| KCH  | KZR-CAD-HR           | Inorganic filler, Methacrylic acid-based monomer | 01011622 | YAMAKIN                          |
| LAU  | Lava™ ultimate       | Inorganic filler, Methacrylate | N589864 | 3M                               |
| SHB  | Shofu block HC       | Micro fumed silica, Silica powder, zirconium silicate, UDMA, TEGDMA | 101401  | Shofu                            |

*1: Manufacturer’s instruction

curing of epoxy resin, specimens were polished using abrasive paper 320–1200 grit on an automatic polishing machine (EcoMet 250 & AutoMet 250, Buehler Japan). The exposed enamel was subjected to wear test.

**Evaluation for wear volume**

After wear test, the abrader and substrate specimens were observed under a 3D laser microscope (LEXT OLS 4000, Olympus, Tokyo, Japan). As shown in Figs. 1 (b) and (c), the worn surface of the abrader specimen was scanned using the semiconductor laser beam of 405 nm at a range of 1,280×1,278 μm; from the worn surface, the diameter was measured at 2 locations, horizontal and vertical, and the average radius value (c) was calculated. The wear volume of abrader specimen, V, was calculated from formulas (1) and (2).

**Wear test**

Abrader and substrate specimens were fixed on a testing machine and two-body wear test was performed in water. The abrader specimen was vertically loaded with a load of 10 N and reciprocated 30,000 times at a speed of 1.5 Hz and width of 3 mm.
\[ V = \frac{1}{6} \pi h (3c^2 + h^2) \]  
\[ h = r - \sqrt{r^2 - c^2} \]

\( r \): radius of the hemispherical specimen (5 mm), \( c \): radius of the circular worn surface, \( h \): worn height of the hemispherical specimen

For substrate specimens, the worn surface and polished surface were established as the lowest and highest position respectively, and an area of 2,560×2,556 \( \mu \)m was observed with a semiconductor laser beam in increments of 0.1 \( \mu \)m. Measurement software (LEXT OLS 4000, Olympus) was used to determine and construct three-dimensional images of worn area to calculate wear volume. For some specimens, the worn surface was observed with an FE-SEM (SU 6600, Hitachi), and surface composition was analyzed with an electron probe microanalyzer (EPMA; JXA-8200, JEOL, Tokyo, Japan).

Statistical analysis
Vickers hardness and wear volume of the abrader and substrate specimens were statistically analyzed by two-way analysis of variance (ANOVA), based on the fabrication method and sample of resin-based composite materials. Furthermore, Tukey’s multiple comparison tests were carried out after one-way ANOVA according to type of resin composite. Statistical analysis was conducted using statistical software (Bell Curve, SSRI, Tokyo, Japan) with a significance level of 5% (\( \alpha = 0.05 \)).

RESULTS

Characterization of resin composite materials
Figure 2 shows the SEM images of the resin-based composite materials. Most of the materials contained spherical fillers of various sizes, however relatively large irregular shaped fillers were also observed in parts of BEP of D-CR, LUW and EST of I-CR, and LAU of C-CR. Resin composites with spherical fillers also contained hybrid type fillers of various sizes and organic composite fillers. It was believed that there were no specific differences in microstructure among the fabrication methods.

Figure 3 shows the Vickers hardness (Hv) of the resin-based composite materials, and Table 2 shows results to the two-way ANOVA. Hv of D-CR was 50 to 97, I-CR was 50 to 196, and C-CR was 65 to 114. When samples of resin composites were compared, Hv of EST of I-CR was the largest at 196±7, and PEE and GRF of I-CR, LAU of C-CR and FSU of D-CR displayed relatively large values. As a result of the two-way ANOVA, significant differences were indicated between fabrication method and samples of abrader specimen (\( p < 0.05 \)), and significant differences were also observed in the interaction (\( p < 0.05 \)). From these results, hardness (Hv) of the resin based-composite material varied among samples of material and fabrication method.

Fig. 4 shows the wear volume of abrader specimens (resin-based composite material) after wear test, and Table 3 shows the results to the two-way ANOVA. The wear volume of abrader specimen ranged from 0.001 to 0.189 mm³. EST of I-CR demonstrated the most wear at 0.189±0.025 mm³. However, the wear volume of MIG and FSU of D-CR, DIA, LUW, and GRF of I-CR, and CES, SHB, LAU, and KAB of C-CR was less than 0.010 mm³. Two-way ANOVA showed significant differences in wear volumes of resin-based composite materials depending on fabrication method of the abrader specimen and samples of specimen (\( p < 0.05 \)); significant differences were also indicated in the interactions (\( p < 0.05 \)).

Fig. 5 shows the wear volume of substrate specimens (bovine enamel) after wear test, and Table 4 shows the results of the two-way ANOVA. The wear volume of enamel was in the range of 0.001 to 0.054 mm³. Wear volume was largest in BEP of D-CR at 0.054±0.028 mm³, which was the largest among the substrate specimens. When abrader specimen was MIG, FSU, ESQ, CME of D-CR, DIA, LUW, SOL, PEE, GRF of
I-CR, and CES, LAU, SHB, KAB of C-CR, wear volume of enamel was 0.010 mm$^3$ or less. As a result of two-way ANOVA, significant differences were observed in the wear volumes of enamel depending on fabrication method and sample of abrader specimen ($p<0.05$), and significant differences were indicated in the interaction ($p<0.05$).

Figure 6 shows the correlation between the wear volumes of abrader specimens (resin based-composite material) and substrate specimens (bovine enamel) after wear test and the regression line for each method of fabrication (D-CR, I-CR, and C-CR). The slope of the regression line of D-CR was 1.26, and the wear volume of abrader specimen was similar to that of the substrate.
specimen, indicating that wear occurred at the same time for both resin-based composite and enamel. The slope of the regression line of I-CR was 8.64, which is larger than D-CR and C-CR, and the wear of substrate specimen was small even though wear volume of abrader specimen was large. The slope of the regression line of C-CR was as small at 0.49, and the wear volume of the antagonist enamel tended to be greater than wear of abrader specimen.

 Observation of worn surface
 Figure 7 shows SEM images comparing the worn surfaces of CES and BEP as abrader specimen. CES with less wear volume showed slight scratch due to sliding-wear; whereas BEP showed clear wear marks where extensive wear of resin composite lead to partial detachment of fillers.

 Figure 8 shows the SEM image of the worn surface of the substrate specimen (bovine enamel) subjected to wear test with BEP and the EPMA mapping image of Si and Al. Scratches due to wear were observed on the enamel surface. When wear marks were enlarged in Fig. 8(b), Si and Al, which are filler components of resin composite, were detected in large amounts.

 DISCUSSION
 Wear test method
 In the oral cavity, strong forces such as attrition, bruxism, and mastication cause wear in restorations. Tooth brushing also has an effect on restoration wear. The wear behavior of crown restorative material is important when considering the durability of the material and several wear test methods have been proposed[16,18]. When testing the wear resistance of materials, there is the pin-on-disk method or the reciprocating sliding test, in which materials are brought into contact by rotation or gliding.

 Particularly during wear test of materials assuming occlusal stress, there are tests such as the reciprocating
Fig. 7 Representative SEM images of resin-based composite materials after wear test.
(a) and (c) less worn surface (CES), (b) and (d) larger worn surface (BEP), (c) is high magnification of (a), and (d) is also high magnification of (b). Less worn surface could not be observed clear wear mark, whereas larger worn surface was clear wear mark. Large worn surface is roughened comparison for less worn one.

Fig. 8 Representative SEM image (a)–(c), (d) Si mapping image and (e) Al mapping image on enamel after 30,000 cyclic wear testing. (b) is high magnification of (a), and (c) is also high magnification of (b). The Si and Al mapping images are corresponded to the SEM image (c).

slide test assuming attrition, a test method that takes into account impact and grinding motion during mastication, or the three-body wear test assuming wear due to friction of food and restoration. For simulation of wear in the oral cavity, the specimen shape was designed to be a cusp, the temperature corresponded to intraoral cavity (about 35°C), the vertical load was equivalent to occlusal force, and the occlusion cycle was taken into consideration. The two-body wear test is effective when investigating wear resistance of the materials since the direct restoration and antagonist teeth are contacted to examine wear behavior. In this study, we aimed to clarify the influence of fabrication methods.

In this study, the two-body wear test was carried out in a reciprocating sliding type using a resin-based composite material as the abrader specimen and bovine tooth enamel as the substrate specimen, assuming wear by direct occlusal contact.

Wear behavior for resin-based composite materials
In this study, the differences in wear volume of the abrader specimen and resin-based composite materials were observed according to fabrication method. The first null hypothesis was rejected according to results. The dental resin composite consists of a matrix resin and filler; strength and hardness of the resin composite increases as filler content increases. In addition, the silane-treated filler increases the strength of the composite resin by bonding with matrix resin. The wear of the resin composite is influenced by the filler particle shape and size, content, and degree of polymerization of the matrix resin.

The microstructure and hardness of resin-based composite materials as factors affecting wear resistance was investigated in this study. A significant difference was observed in the Vickers hardness of resin composite depending on the fabrication method. The difference in hardness due to fabrication method is most likely caused by degree of polymerization due to differences in the polymerization method. Polymerization was carried out with localized light irradiation for D-CR, overall light irradiation and heat for I-CR, and pressurization and heat or light irradiation for C-CR. Hardness of resin-based composite may increase as degree of polymerization increases. However, hardness of resin-based composite materials depends not only on the degree of polymerization of the resin matrix but also on filler content. The relationship between the hardness of the resin-based composite material and the wear volume of resin composite and enamel is shown in Fig. 9. As the hardness of resin composite increases, the wear volume of the resin composite itself increases. Since the correlation coefficient is $r=0.753$, the increase in the hardness of the resin composite increases the wear volume of resin composite itself when enamel is the antagonist.

Resin-based composite material with smaller wear volume contained more spherical filler particles with smaller diameters. Bayne et al. reported that resin composite containing small fillers show minimal wear. In addition, Jaarda et al. reported that even nano-sized
filers act as organic composite fillers and affect the wear behavior of resin composites. Lim et al. reported that silanization reduce the wear of resin composite. Therefore, spherical filler shape, appropriate sized filler, and a strong bond between resin matrix and filler can reduce the wear of resin composites.

**Wear behavior of bovine enamel**

Studies have indicated that the type of crown restorative material, hardness, and surface roughness of restorations all influence enamel wear. Regarding the effect of resin composite on enamel wear, Suzuki and Leinfelder reported that resin composite containing organic composite fillers showed less wear volume of enamel than those containing silica filler. In this study, difference in wear volume was observed in enamel depending on fabrication method of resin-based composite material. Therefore, the second null hypothesis was rejected. The Vickers hardness of bovine enamel used in this study was approximately 300–340, which is larger than the hardness of resin-based composites. Figure 9 demonstrates the correlation between hardness of resin composite and enamel wear; both the slope and correlation coefficient is small indicating that hardness of resin-based composite as abrader specimen did not affect the wear of enamel. The BEP in I-CR was the greatest wear of enamel. The microstructure of BEP showed that filler shape was irregular and sharp and diameter of filler particle was large at 10 μm. Filler exposure was observed on the wear surface of BEP (Fig. 7). EPMA analysis of enamel surface after wear test with BEP indicated that deposits were derived from filler particles (Fig. 8). These observations suggested that enamel wear occurred due to exposure and abrasive effects of detached filler particles, which were sharp as a result of getting crushed.

**Clinical significance**

Resin composite is said to have higher wear resistance compared to metal-ceramic and full-metal type crown materials. However, it has been reported that wear was observed in about half of the restorations over a 4-year period in class II cavity resin composite restorations which used resin composites containing 2 types of micro fillers. In a clinical setting, it is ideal that the restoration maintains occlusal function while undergoing the same changes over time as the adjacent tooth.

In this study, two-body wear test was carried out using resin-based composite materials of various fabrication methods and bovine enamel. As a result, wear occurred more in resin composite than enamel for I-CR, and wear occurred more in enamel than resin composite for C-CR. Degree of wear for both resin composite and antagonist enamel were the same for D-CR. This indicates that wear of enamel and resin composite is influenced by the fabrication method of the restoration material.

**CONCLUSIONS**

In this study, the microstructure and hardness of resin-based composite materials with different fabrication methods were investigated, and two-body wear test of resin-based composite material and bovine enamel was conducted, and the following conclusions were obtained.

1) The hardness of resin-based composite was influenced by both the fabrication method and sample. The hardness of resin composite containing hybrid-type filler was large, independent of fabrication method.

2) In two-body wear test, resin-based composite fabricated by different methods influenced both the wear of the resin-based composite itself and antagonist enamel. The wear of resin-based composite fabricated by indirect method was larger than those by direct method and CAD/CAM method, whereas the wear of enamel was larger in the hybrid resin of CAD/CAM crowns. Resin composite for direct restoration showed similar wear of resin composite and enamel.

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**CONFLICTS OF INTEREST**

The authors declare no conflicts of interest.
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