Effect of filler content of flowable composites on resin-cavity interface

Ichiro IKEDA¹, Masayuki OTSUKI¹, Alireza SADR¹,², Tomomasa NOMURA¹, Ryuzo KISHIKAWA¹ and Junji TAGAMI¹,²

¹Cariology and Operative Dentistry, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan
²Global Center of Excellence 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, Ichiro IKEDA; E-mail: ikeda-ichiro@ikeda-shika.jp

The purpose of this in vitro study was to evaluate marginal integrity and wall adaptation in 1- and 2-mm-deep cavities restored with a high filler-loaded flowable composite in comparison to a flowable composite with lower filler content and a conventional hybrid composite. 1-mm-deep dentin and 2-mm-deep enamel-dentin Class I cavities were prepared and restored with a self-etch adhesive and with one of the composites. Samples were crosscut and evaluated for marginal integrity and gap formation using a digital microscope. Selected samples were also observed using a scanning electron microscope. For 1-mm-deep cavities, no differences in marginal integrity and wall adaptation were observed among the three composites. For 2-mm-deep cavities, those restored with the bulk technique and with the higher filler-loaded flowable composite demonstrated a similar outcome as that of the conventional hybrid composite. On the effect of restorative techniques, cohesive enamel defects were observed in bulk-filled 2-mm-deep cavities. However, when the incremental technique was used in conjunction with the higher filler-loaded flowable composite and the conventional hybrid composite for 2-mm-deep cavities, superior results were obtained.

Keywords: Flowable composite resin, Filler content, Resin-cavity interface

INTRODUCTION

Indications for composite resin restorations are expanding steadily as the properties of composite materials improve and the bond strength of resin adhesives to dental substrates increase. On the longevity of composite resin restorations, clinical trials have shown that they are acceptable for long-term use¹⁰. The longevity of a resin restoration is influenced by myriad factors: not only by the various properties of composite resins and adhesives, but also by the restorative technique. These factors have a significant impact on the marginal integrity and adaptation of the resin-cavity interface. Excellent marginal adaptation extends the longevity of restorations; on the contrary, inadequate adaptation results in interfacial gap formation and marginal microleakage, which in turn induces recurrent caries.

The “first generation” of flowable composites was introduced in late 1996⁷. Flowability is regarded as a desirable handling property which allows the material to be injected through small-gage dispensers, thus simplifying the placement procedure and amplifying the range of clinical applications⁸. The flowable composites can be easily inserted into small cavities and are expected to exhibit better adaptation to the internal cavity wall compared to the conventional restorative composites which are more viscous.

Flowable composites are characterized in a lower filler loading and a greater proportion of diluent monomers in the formulation⁹. These composites were traditionally created by retaining the same small particle size of the conventional hybrid composites, but reducing the filler content and allowing the increased resin to reduce the viscosity of the mixture¹⁰. However, their various mechanical properties such as flexural strength and wear resistance have been reported to be generally inferior compared to those of the conventional composites⁷,⁸. For this reason, flowable composites have been suggested to be filling materials for low-stress applications and in situations with difficult access or those requiring good penetration such as amalgam, composite or crown margin repairs; pit and fissure sealing; preventive resin restorations; air abrasion cavity preparations; cavity lining; porcelain repairs; enamel defects; incisal edge repairs in anterior sites; and for small Class III and Class V restorations⁷.

Recently, flowable composite resins of high filler loading have been introduced. According to the manufacturers, the filler content and polymerization shrinkage of the new materials are comparable to those of the conventional hybrid composite resins but with the same flow behavior. The application range for the newly introduced flowable composites is expected to include larger or deeper cavities and in higher thicknesses, similar to the conventional composites. However, reports are scarce concerning the applicability of flowable composites with high filler loading for larger or deeper cavities. The purpose of this in vitro study, therefore, was to evaluate the marginal integrity and wall adaptation of 1- and 2-mm-deep cavities restored with a high filler-loaded flowable composite, in comparison to a flowable composite with lower filler content and a conventional hybrid composite.
MATERIALS AND METHODS

Materials used

Materials used in this study are listed in Table 1. Two flowable composite resins and one conventional hybrid composite were used in this study. Clearfil Majesty LV (MJ; Kuraray Medical, Tokyo, Japan) is a high filler-loaded flowable composite and Clearfil Flow FX (Fx; Kuraray Medical) is a “first generation” flowable composite. A conventional hybrid composite, Clearfil AP-X (APX; Kuraray Medical) was used as a control. Clearfil SE Bond (Kuraray Medical), a two-step selfetching primer adhesive system, was used in conjunction with one of the composites resins.

Cavity preparation and restoration according to experimental groups

The research design adopted in this study, in particular on the use of extracted human teeth, was subjected to the guideline of the Ethics Committee of the Faculty of Dentistry, Tokyo Medical and Dental University.

Eighty-four extracted intact human third molars were employed in this study. After cleaning with a scaler, the roots were cut away. The occlusal surfaces of 36 teeth were ground using a model trimmer to obtain flat dentin surfaces, and then polished with a 600-grit silicon carbide paper (Sankyo Rikagaku Co. Ltd., Saitama, Japan). A Class I dentin cavity, 1 mm in depth (2 × 1 × 1 mm), was prepared in each tooth using a flat-end tapered diamond bur (SB2, GC, Tokyo, Japan) with an air turbine handpiece under water.

Table 1 Materials used in this study

<table>
<thead>
<tr>
<th>Material</th>
<th>Ingredients</th>
<th>Lot No</th>
<th>Composite filler content (wt%)*</th>
<th>Polymerization shrinkage (lin%)**</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clearfil Majesty LV (Kuraray Medical)MJ</td>
<td>Silanated barium glass powder, Silanated colloidal silica, TEGDMA, Hydrophobic aromatic dimethacrylate, other</td>
<td>060512D13</td>
<td>81</td>
<td>1.88</td>
</tr>
<tr>
<td>Clearfil Flow FX (Kuraray Medical)FX</td>
<td>Bis-GMA, TEGDMA, Barium Glass Filler, Lanthanoid fluoride, other</td>
<td>00006A</td>
<td>64</td>
<td>3.24</td>
</tr>
<tr>
<td>Clearfil AP-X (Kuraray Medical)APX</td>
<td>Silanated barium glass, Silanated colloidal silica, Silanated silica, Bis-GMA, TEGDMA, other</td>
<td>01084A</td>
<td>85</td>
<td>1.24</td>
</tr>
<tr>
<td>Clearfil SE Bond (Kuraray Medical)</td>
<td>Primer MDP, HEMA, di-camphor quinine, N,N-Dienthanol-P-toluidine, Water Bond MDP, Bis-GMA, HEMA, Hydorophobicdimethacrylate, di-camphor quinine N,N-Dienthanol-P-toluidine Silanated colloidal silica</td>
<td>00649A</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>00922A</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*, **: Data as disclosed by the manufacturer

Table 2 Experimental groups

<table>
<thead>
<tr>
<th>Group</th>
<th>Cavity depth</th>
<th>Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-MJ</td>
<td>1 mm</td>
<td>MJ</td>
</tr>
<tr>
<td>1-FX</td>
<td>1 mm</td>
<td>FX</td>
</tr>
<tr>
<td>1-APX</td>
<td>1 mm</td>
<td>APX</td>
</tr>
<tr>
<td>2-MJ</td>
<td>2 mm</td>
<td>MJ</td>
</tr>
<tr>
<td>2-FX</td>
<td>2 mm</td>
<td>FX</td>
</tr>
<tr>
<td>2-APX</td>
<td>2 mm</td>
<td>APX</td>
</tr>
<tr>
<td>2-MJ-APX</td>
<td>2 mm</td>
<td>MJ, APX (incremental)</td>
</tr>
</tbody>
</table>
spray coolant. The occlusal surfaces of the remaining 48 teeth were slightly ground to obtain flat enamel surfaces and then polished down to 1,500-grit silicon carbide paper. Cavities of 2 mm depth \((2 \times 1 \times 2 \text{ mm})\) were then prepared in this manner: all cavosurface margins were located within enamel and that each cavity wall consisted of approximately 1 mm enamel at the top and 1 mm dentin in the bottom, with the cavity floor in dentin.

Each cavity was treated with Clearfil SE Bond according to the manufacturer’s instructions. The self-etching primer was applied for 20 seconds and then dried with air. One coat of the bonding agent was applied on the cavity surface and irradiated with a halogen light curing unit (Optilux 501, Kerr, CA, USA; 800 mW/cm\(^2\)) for 10 seconds. The teeth with 1-mm-deep cavities were divided into three groups and those with 2-mm-deep cavities were divided into four groups, with 12 cavities in each group. Cavities in all the 1-mm-deep groups and in three of the 2-mm-deep groups were filled with one of the three composite resins in bulk, and then light-cured for 40 seconds to make up 1-MJ, 1-FX, 1-APX, 2-MJ, 2-FX, and 2-APX groups with respect to the cavity depth and composite resin type. The remaining 2-mm-deep cavities were restored in two increments, with MJ in the deeper layer and APX in the surface layer, to make up the 2-MJ-APX group. MJ was photocured for 20 seconds and APX was irradiated for 40 seconds. The experimental groups are summarized in Table 2.

**Gap formation evaluation**

The restored teeth were stored in tap water for 24 hours at room temperature. After removal from water storage, each tooth was buccolingually crosscut at the center of cavity using a low-speed diamond saw (Isomet, Buehler, IL, USA) with copious water. The crosscut surfaces were polished down to 1,500-grit silicon carbide paper. The polished surfaces were observed and evaluated for marginal gap formation using a digital microscope (VHX-500, Keyence, Osaka, Japan) at \(\times50\) to \(\times200\) magnifications. The frequency of defects detected at resin-cavity interface in each group was recorded, and statistical analysis of the results was performed using Mann–Whitney U-test (significance level at \(p<0.05\)). After evaluation with a digital microscope, the samples were trimmed, dried, gold-sputtered, and then observed with a scanning electron microscope (SEM).

**RESULTS**

Figures 1 to 6 show the typical images obtained by a digital microscope for each group, and the results are shown in Table 3.

For 1-mm-deep groups, there was no gap formation at the cavity floor and no marginal defects were detected in any of the samples.

---

**Fig. 1** Microscopic images of the crosscut surfaces of 1-mm-deep groups (original magnification \(\times100\)): (a) 1-MJ; (b) 1-FX; (c) 1-APX. Marginal fracture and gap formation were not observed.

**Fig. 2** Microscopic images of the crosscut surfaces of 2-MJ at different magnifications: (a) low magnification \(\times50\); (b) high magnification \(\times200\) of the cavosurface margin; (c) high magnification \(\times200\) of the cavity floor. R: resin, E: enamel, D: dentin.
For 2-mm-deep cavities, five samples in 2-FX group showed resin-dentin gap formation at the cavity floor. Moreover, cracks or fracture lines parallel or diagonal to the cavity wall were detected within enamel in 10 samples of 2-FX group. While there was no interfacial gap formation in other groups, five samples in each of the 2-APX and 2-MJ groups showed cohesive fracture or enamel cracking. 2-MJ-APX group did not show any gap formation or enamel damage. These damages were confirmed by SEM images.

According to Man–Whitney U-test, the frequency of gap formation at the cavity wall was significantly higher in 2-FX group than in the other 2-mm-deep groups. Fracture within enamel wall was also more frequently observed in 2-FX group as compared to the other groups. There were no statistical differences in gap formation or cohesive enamel fracture among the other groups.

Fig. 3 Microscopic images of the crosscut surfaces of 2-FX at different magnifications: (a) low magnification ×50; (b) high magnification ×200 of the cavosurface margin, where the arrows show the marginal enamel crack; (c) high magnification ×200 of the cavity floor, where the arrows show gap formation at the cavity floor. R: resin, E: enamel, D: dentin.

Fig. 4 Microscopic images of the crosscut surfaces of 2-APX at different magnifications: (a) low magnification ×50; (b) high magnification ×200 of the cavosurface margin; (c) high magnification ×200 of the cavity floor. R: resin, E: enamel, D: dentin.

Fig. 5 Microscopic images of the crosscut surfaces of 2-MJ-APX at different magnifications: (a) low magnification ×50; (b) high magnification ×200 of the cavosurface margin; (c) high magnification ×200 of the cavity floor. R: resin, E: enamel, D: dentin.
Fig. 6 Typical SEM images of the resin-cavity interfaces of 2-mm-deep groups: (a) Cavosurface margin of 2-MJ (×200), where no gap formation and enamel cracks were observed; (b) resin-cavity interface of 2-MJ (×200), where no gap formation was observed; (c) cavosurface margin of 2-FX (×200), where arrows show the enamel crack; (d) resin-cavity interface of 2-FX (×200), where arrow shows gap formation. R: resin, E: enamel, D: dentin.

Table 3 Gap formation and marginal fracture results

<table>
<thead>
<tr>
<th>Group</th>
<th>Gap formation at cavity wall</th>
<th>Cohesive fracture in enamel</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-MJ</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>1-FX</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>1-APX</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2-MJ</td>
<td>0</td>
<td>5</td>
</tr>
<tr>
<td>2-FX</td>
<td>5</td>
<td>10</td>
</tr>
<tr>
<td>2-APX</td>
<td>0</td>
<td>5</td>
</tr>
<tr>
<td>2-MJ-APX</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

The vertical bars show statistical differences (p<0.05).

DISCUSSION

For in vitro evaluation of a restorative material or technique, investigations typically focus on marginal adaptation at the resin-cavity interface and bond strength. For interfacial analysis, different methods have been employed: three-dimensional analysis, dye penetration test, and SEM observation. Although microleakage evaluation is one of the most common methods for assessing the sealing efficiency of a restorative material, a gold standard has not been established for this evaluation method yet. It is also noteworthy that even a minor microleakage might induce recurrent caries, which implies that it may not be appropriate to evaluate resin-cavity interfaces based on microleakage rankings or scores. In the present study, the crosscut surfaces of restorations were observed using a high-resolution digital microscope, and then confirmed by SEM observation. While SEM observation requires low atmospheric pressure and sample desiccation, which might cause artifacts, these two requirements do not apply to digital microscopic observations.

On the clinical effectiveness of two-step self-etching adhesives, it has been resoundingly established in published literature. In this study, a two-step self-etching adhesive system Clearfil SE Bond was used. This material has been shown to provide acceptable marginal sealing and high bond strength.

On light curing method, each composite was photocured for 40 seconds using a halogen light unit for all experimental groups except 2-MJ-APX group. It has been suggested that the photoirradiation method has an effect on contraction stress, and therefore alternative strategies such as soft-start mode of curing have been introduced. The soft-start polymerization technique is characterized by a low initial light intensity followed by high-intensity irradiation. It has been reported that the soft-start polymerization method provided twofold advantages: an increase in resin-dentin bond strength in box-shaped cavities and reduced polymerization shrinkage stress without compromising the degree of conversion into a polymer network. However, Class I and II composite restorations clinically placed with a soft-start technique showed no significant improvements in terms of postoperative sensitivity to cold or reduction in marginal stress.

Resin composites vary widely in shrinkage-strain magnitude, and the inverse relationship between filler loading and shrinkage-strain is explained by the corresponding volume fraction of monomers present to undergo polymerization. In general, the higher fluidity of flowable composites is achieved by increasing the proportion of monomers in the formulation of the composite paste. Due to their lower filler contents, the traditional flowable composites shrink more upon polymerization and are hence less rigid than the conventional composites. Due to their reduced rigidity, traditional flowable composites may be successfully used in micro-conservative occlusal cavities, since their polymerization shrinkage would be low because of the limited volume of the material used. Indeed, this suggestion was corroborated by the results observed for 1-mm-deep cavities in this study. Then, when the cavity size increased, the traditional low-filler-content flowable composite (2-FX) showed frequent resin-dentin gap formation and enamel fractures as compared to other 2-mm-depth groups (i.e., 2-MJ and 2-APX), thereby confirming the effect of filler content on polymerization shrinkage. Additionally, the different degrees of expansion caused...
by the varying water absorption capacities of each material during the 1-day storage could affect the results.

According to the manufacturer, the newly introduced flowable composite evaluated in this study, MJ, had a high filler loading (81 wt%) and that its polymerization shrinkage was only 1.88 % (Table 1) — which was close to that of conventional hybrid composites. It has been reported that MJ exhibited mechanical properties and wear resistance similar to those of universal resin composites. Indeed, this report was corroborated by the results of this study where the marginal integrity and gap formation at the resin-cavity interface of 2-MJ group were similar to those of 2-APX group.

Fractures along the cavosurface enamel margin and cracks along the enamel wall were detected in 2-mm-deep bulk-filled cavities. It is known that stress, developed as a result of polymerization contraction that accompanies curing, might be present within the composite and transferred to the bonded margins of the restoration. When the bonding is strong enough, the shrinkage stress may lead to crack initiation and propagation within the bonded substrate. This is particularly the case for large bulk-filled cavities with high C-factors where the cavosurface margins are located in enamel, which is of a brittle nature. Tooth fracture is still a frequently occurring problem caused by induced contraction stresses when polymerization shrinkage takes place under constrained conditions with the composite bonded between cavity walls.

In this study, 2-MJ-APX group which employed an incremental filling technique showed improved marginal integration with no enamel fracture. It has been suggested in previous studies that the use of an incremental technique resulted in significantly less microleakage compared to the use of a bulk technique — which meant that the incremental restorative technique was effective in improving marginal sealing. For incremental build-ups of resin composites, 2-mm-thick layers are typically recommended. However, for some types or shades of resin composites, a high degree of cure might not be achieved adequately throughout the entire 2 mm thickness of the composite resin. Therefore, the incremental filling technique may also be advantageous to achieving a thorough polymerization and a proper contour of the restoration.

Flowable composites have been used as intermediate materials or liners between the adhesive layer and higher-viscosity composite. The low stiffness of flowable composites might compensate for the polymerization contraction of restorative composites with a high modulus of elasticity. The liner may act as a buffer for the contraction stress of the composite resin under polymerization, thereby reducing stress at the resin-cavity interface. The use of a flowable composite as a liner probably relieves the stress on the adhesive interface generated by thermal changes and occlusal forces, and may lead to an improvement in the durability of marginal sealing. However, some studies showed no major improvements in marginal sealing and clinical performance for restorations lined with flowable composites. The edge-fracture resistance of flowable composites is lower towards the margins than towards the center of restoration, resulting in cracking or chipping of the material at the restoration margins. Moreover, their lower elastic moduli, some flowable composites generated polymerization contraction stress values similar to or even higher than those produced by non-flowable composites.

Based on the results obtained, the high filler-loaded flowable composite used in this study might be applicable for filling relatively larger cavities or at a higher thickness, while maintaining excellent wall adaptation. On the influence of restorative techniques, the use of an incremental filling technique seemed to be beneficial for large cavities to reduce the effect of contraction stress.

CONCLUSIONS

With the smaller 1-mm-deep cavities, no differences in marginal integrity and wall adaptation were observed among the flowable composites with high and low filler contents and the hybrid composite. With bulk-filled, 2-mm-deep Class I cavities, restoration using the low-filler-content flowable composite showed inferior results. On the influence of restorative techniques, the incremental restorative technique emerged to be the superior one when used in conjunction with either the high filler-loaded flowable composite or the conventional hybrid composite for 2-mm-deep cavities.

ACKNOWLEDGMENTS

This work was partially supported by a grant from the Japanese Ministry of Education, Global Center of Excellence (GCOE) Program, “International Research Center for Molecular Science in Tooth and Bone Diseases”.

REFERENCES

6) Chan DC, Browning WD, Frazier KB, Brackett MG.


24) Chi HH. A posterior composite case utilizing the incremental and stratified layering technique. Oper Dent 2006; 31: 512-516.


