Effect of lining with a flowable composite on internal adaptation of direct composite restorations using all-in-one adhesive systems

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The purpose of this study was to evaluate the effect of lining with a flowable composite on internal adaptation of composite restorations using three all-in-one adhesive systems; Bond Force (BF), G-Bond Plus (GP), and OptiBond All-in-one (OP), and a two-step self-etching adhesive system; Clearfil SE Bond (SE). They were applied to each cylindrical cavity prepared on the human dentin. The cavity surface was lined with/without a flowable resin composite prior to filling with a resin composite (FL/NL). After water storage for 24 h, the specimens were sectioned and polished, and internal adaptation of the restorations was assessed using a confocal laser scanning microscopy. For SE, a perfect cavity adaptation was recognized in both FL and NL. For BF, GP, and OP, cavity adaptation was material dependent in NL, whereas no gap formation was observed in FL. However, voids formation was observed at the composite-adhesive-dentin interface in every all-in-one adhesive system.

Keywords: Internal adaptation, All-in-one adhesive systems, Lining, Dentin, Direct restoration

INTRODUCTION

Direct composite restorations have become increasingly popular as tooth-colored restorations for extensive lesions in posterior teeth1-2. Adaptation of the restorative materials to cavity margins and internal cavity surfaces are crucial for long-term performance of the restorations3. Polymerization shrinkage of a resin composite creates contraction force that may disrupt the bond of the restoration to the cavity walls4. As the cavity configuration factor (C-factor)5, the ratio of the bonded surface area to the unbonded or free surface area, becomes larger, the competition between polymerization shrinkage and adhesion of the resin to dentin is increased, resulting in an increased risk of gap formation at the interface.

Many methods have been proposed to overcome the negative effects of the polymerization shrinkage, such as incremental layering technique6-7, different protocols of light-curing8 and use of semi-direct and indirect restorations.9 Several studies have shown that the incremental filling techniques ensure satisfactory polymerization of light-cured composite resins in deep preparations7,10,11). It was also reported that the use of a thin layer of a flowable composite resin as a lining beneath the bulk-filled hybrid composite could be an effective method for control of the stress and prevention of gap formation at the marginal area of the restoration12,13). In this regard, it was suggested that the lining with flowable composites might act as an elastic layer absorbing shrinkage stresses during polymerization shrinkage12).

Dental adhesive systems have been remarkably simplified and improved. Recently, all-in-one adhesive systems have been widely used in the clinics; these systems combine etching, priming and bonding procedures into a single application step. The all-in-one adhesives contain one or more functional monomers together with water and organic solvents, such as alcohol and acetone in the composition. Some all-in-one adhesives also contain 2-hydroxylethyl methacrylate (HEMA) which is a hydrophilic methacrylate monomer. HEMA may help to keep the polar and apolar ingredients together with water and organic solvents, such as alcohol and acetone in the composition. Since the simplified adhesives are more hydrophilic than the other adhesive systems which use a separate hydrophobic application step, the cured adhesive may act as a water permeable membrane15). It was reported that all-in-one adhesives have some morphological defects on the bonded interfaces, so called a “water-tree”16). In order to avoid these defects, it is important to remove water and the solvent by air blowing and to cure only adhesive resin layer in all-in-one adhesive systems17). However, the effect of air-drying on adhesive layer thickness has been rarely reported in all-in-one adhesive system.

In addition, there have been some arguments on the efficacy of the all-in-one adhesive systems on sealing capability of the restorations18,19). However, little information is available on internal adaptation of a composite restoration using all-in-one adhesive systems with a flowable composite lining. Therefore, the purpose of this study was to evaluate internal adaptation of
cylindrical-shaped composite restorations using three all-in-one adhesive systems and a two-step self-etch adhesive system. The null hypothesis of this study was that the internal adaptation of the restorations was not influenced by the types of adhesive systems or the use of flowable composite lining.

**MATERIALS AND METHODS**

*Adhesives used in this study*

The adhesive materials and their ingredients are listed in Table 1. Three all-in-one adhesive systems; Bond Force (BF, Tokuyama Dental, Tokyo, Japan), G-Bond Plus (GP, GC, Tokyo, Japan), and OptiBond All-in-one (OP, Kerr, Orange, CA, USA) were compared against a two-step self-etching adhesive system; Clearfil SE Bond (SE, Kuraray Medical, Tokyo, Japan) as the control in this study.

**Specimen preparation**

The specimen preparation for cavity adaptation testing was illustrated in Fig. 1. Forty non-carious human third molars were collected after the individuals’ informed consent was obtained according to a protocol approved by the Human Research Ethics Committee, Tokyo Medical and Dental University, Japan. The teeth were stored in water at 4°C and used within 1 month after extraction.

The remaining oral tissues were manually removed using hand instruments. The molars were randomly distributed among four experimental groups of five teeth each according to the adhesive systems. The occlusal enamel was cut off with a model trimmer (Y-230, Yoshida, Tokyo, Japan) to expose superficial to mid coronal dentin surfaces, and ground with #600-grit silicon carbide paper under running water to flatten the surfaces. In order to obtain cavities with standard size and shape, the cylindrical cavities (3 mm diameter and 1 mm depth) were prepared using a milling machine (PFG200, Cendres & Metaux SA, Bienne, Switzerland) and a superfine diamond bur (SF114, Shofu, Kyoto, Japan) under water coolant. In the current study, removing of coronal enamel horizontally completely limited the cavity depth to only 1 mm. Following this, one of the four adhesive systems (BF, GP, OP and SE) was applied onto the cavity surface according to the manufacturers’ instructions. The adhesives were then light cured by a tungsten-halogen light curing unit (OPTILUX 500, Kerr, Orange, CA, USA) with a light output of 600 mW/cm². The specimens were then divided equally into two sub-groups. For one sub-group (FL), a flowable resin composite (Estelite Flow Quick, shade A2, Tokuyama Dental) was placed on one-third of the cavity depth and light-cured for 10 s. The amount of each increment was determined by measuring the height of

Table 1  Adhesive materials used in this study

<table>
<thead>
<tr>
<th>Adhesives</th>
<th>Code</th>
<th>pH</th>
<th>Composition</th>
<th>Lot No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clearfil SE Bond (Kuraray Medical, Tokyo, Japan)</td>
<td>SE</td>
<td>2.0</td>
<td>Primer: MDP, HEMA, hydrophilic dimethacrylate, water, photo initiator Bond: MDP, HEMA, hydrophilic dimethacrylate, photo initiators, Bis-GMA, microfiller</td>
<td>00863A</td>
</tr>
<tr>
<td>G-Bond Plus (GC, Tokyo, Japan)</td>
<td>GP</td>
<td>1.5</td>
<td>4-MET, phosphoric acid ester monomer, DMA, water, acetone, silica filler, photo-initiator</td>
<td>01266A</td>
</tr>
<tr>
<td>OptiBond All-in-one (Kerr, Orange, CA, USA)</td>
<td>OP</td>
<td>2.5</td>
<td>GPDM, GDM, HEMA, Bis-GMA, water, ethanol, acetone, silica filler, CQ, sodium hexafluorosilicate</td>
<td>0009</td>
</tr>
<tr>
<td>Bond Force (Tokuyama Dental, Tokyo, Japan)</td>
<td>BF</td>
<td>2.3</td>
<td>Isopropyl alcohol, water, 3D SR monomer, Bis-GMA, TEGDMA, HEMA, glass fillers, photo initiator</td>
<td>3075076</td>
</tr>
</tbody>
</table>

Abbreviations: MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate; Bis-GMA: bisphenol-A-diglycidyl methacrylate; 4-MET: 4-methacryloxyethyl trimellitic acid; DMA: dimethacrylate; GPDM: glycerol phosphate dimethacrylate; GDM: glycerol dimethacrylate; CQ: camphorquinone; 3D SR: 3D self-reinforcing; TEGDMA: triethyleneglycol dimethacrylate.
flowable resin composite. In FL groups, the floor surface of the cavities was completely covered with the flowable composite resin. For the other sub-group (NL), the cavity remained without lining.

Following this, a resin composite (Estelite Σ Quick, shade A2, Tokuyama Dental) was filled in the cavity in a single increment, and the top surface was covered with a plastic strip, pressed to flat and light-cured for 10 s. After the specimens were stored in 37°C distilled water for 24 h, they were sectioned at the center of the restoration parallel to the long axis of each tooth using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). They were then embedded in an epoxy resin (Epoxicure resin, Buehler) for 24 h. After curing of the epoxy resin, the specimens were polished with diamond pastes (DP-Paste, Struers, Ballerup, Denmark) with particle sizes down to 0.25 µm.

CLSM observation
Internal adaptation of the restorations was assessed using a confocal laser scanning microscope (CLSM) (1LM21, Lasertec, Yokohama, Japan) under ×1,250 magnification. Cavity adaptation rate (%) was then calculated using the following equation:

Cavity adaptation rate (%) = (sum of adaptation length/total length of cavity wall and floor) × 100

SEM observation
After the CLSM observations, the specimens were subjected to an argon-ion-beam etching (100 mA and 2.0 V) for 6 min and then gold sputter-coated. The thickness of the adhesive layer in each restoration was measured using a scanning electron microscopy (SEM) (JSM-5310 LV, JEOL, Tokyo, Japan) at ×2,000 magnification. Thickness of the bonding layer at the cavity wall for each specimen was determined by averaging the values measured at three points; the center and at a distance of 20 µm from the center on either side. The mean value of these three measurement points was calculated for each group (n=5). Thickness of the bonding layer of the corner and the floor of the cavity was also determined in the same manner. Furthermore, the morphology of the adhesive-dentin interface was observed by the SEM.

Statistical analysis
The results in cavity adaptation rate obtained from the CLSM study were statistically analyzed and compared by Wilcoxon rank sum test with Bonferroni correction (α=0.05). Thickness of the bonding layer of each composite restoration was statistically analyzed by two-way ANOVA and t-test with Bonferroni correction, with adhesive type and cavity location as factors (α=0.05).

RESULTS

Cavity adaptation rate
The results of the cavity adaptation rate (%) in each group were summarized in Table 2. A perfect internal adaptation of restoration (100%) was shown for all groups. However, the cavity adaptation rates were adhesive material dependent in the NL groups, where perfect adaptation was only obtained for SE. Gaps were observed with BF, GP and OP adhesives, and there were significant differences among the three all-in-one adhesives. GP and OP showed significantly lower internal adaptation rates compared to BF (p<0.05). BF showed the lowest cavity adaptation rate without lining (p<0.05).

CLSM images
Figure 2 showed typical CLSM images of the internal adaptation test of the composite restorations at the whole cavity (Fig. 2a) and at the cavity floor (Figs. 2b and 2c) and the corners (Figs. 2d and 2e). In the all-in-one adhesive (BF) without flowable lining (Fig. 2b), gap formation was observed at the interface between the adhesive and the resin composite at the cavity floor, while no gap was formed in the lining group (Fig. 2c). The adhesive layer was thin and uniform in this all-in-one system at the cavity corners (Fig. 2d). The layer of SE was thicker at the corners (Fig. 2e).

In GP without lining, the gaps were observed both at the cavity floor and the corners. However, only few gaps were observed at the cavity wall. OP showed gaps at the cavity floor, corner and wall. In BF, gaps were rarely observed at the interface of the cavity walls; they mainly appeared at the cavity floor.

Table 3 shows mean thickness of the adhesive layer at the cavity walls, floor and corners, respectively. Thickness of the adhesive layer was significantly affected by type of adhesive and location in the cavity (p<0.05). SE showed a significantly thicker adhesive layer than GP, OP and BF in each location (p<0.05). The thickness of the adhesive layer was also significantly affected by the location (p<0.05); adhesive layer of SE was significantly thicker at the corner compared to the cavity wall and floor (p<0.05). However, there was no regional difference in thickness of the adhesive layer among the all-in-one adhesive systems (p>0.05). Moreover, there was no significant interaction between the two factors (p>0.05)

SEM images
SEM pictures at the adhesive-dentin interface at the cavity corner and the cavity floor of the restorations in
Fig. 2 Typical CLSM images of the internal adaptation test of the cylindrical-shaped composite restorations. a: NL-GP at the whole cavity (×125), b: NL-BF at the cavity floor (×1,250), c: FL-BF at the cavity floor (×1,250), d: FL-GP at the cavity corner (×250) and e: FL-SE at the cavity corner (×250). CR: resin composite; FR: flowable composite; B: bonding; D: dentin.

Gap formation (indicated by blank arrows) was observed in the no lining group (b). Adhesive layer thickness (between arrow heads) was affected by the adhesive type and the location. The adhesive layer at the cavity corner of the all-in-one adhesive (d) was significantly thinner than that of SE (e).

<table>
<thead>
<tr>
<th>Adhesives</th>
<th>Cavity floor</th>
<th>Cavity wall</th>
<th>Cavity corner</th>
</tr>
</thead>
<tbody>
<tr>
<td>SE</td>
<td>13.4 (9.0)</td>
<td>22.7 (14.7)</td>
<td>70.2 (50.1)</td>
</tr>
<tr>
<td>GP</td>
<td>3.1 (0.5)</td>
<td>3.6 (1.1)</td>
<td>3.3 (0.8)</td>
</tr>
<tr>
<td>OP</td>
<td>5.9 (1.2)</td>
<td>13.5 (4.7)</td>
<td>11.1 (7.5)</td>
</tr>
<tr>
<td>BF</td>
<td>4.0 (2.5)</td>
<td>4.1 (1.6)</td>
<td>7.2 (5.5)</td>
</tr>
</tbody>
</table>

Values are mean±S.D. (n=5). Same superscript letters indicate statistically significant difference (p<0.05).
Fig. 3  SEM images at the adhesive-dentin interface at the cavity corner (left) and the cavity floor (right) of the restorations in the flowable lining group (×2,000) for different adhesives. SE (a and b), GP (c and d), OP (e and f) and BF (g and h). CR: resin composite; FR: flowable composite; B: bonding; D: dentin.

The adhesive layer is marked between two arrow heads. Void formation was observed in the all-in-one adhesive groups (white bold arrows). However, the size and location of the voids were adhesive material dependent. The gap formation between the adhesive layer and the dentin at the cavity floor (d, f and h) were observed (blank arrows), which may be due to high vacuum for SEM observation.
the FL groups were shown in Fig. 3. For SE, no gap formation was observed at the interface both at the cavity corner and floor (Figs. 3a and 3b). A relatively thicker adhesive, compared with the all-in-one adhesives, was observed at the cavity corner and floor. The adhesive layer appeared to be homogeneous in texture.

In GP (Figs. 3c and 3d), a thin adhesive layer (less than 5 µm thickness) was observed with some void formation within the adhesive close to the interface between dentin and the bonding layer (Fig. 3c). Gap formation between the adhesive layer and the dentin was often observed at the cavity floor (Fig. 3d).

In OP (Figs. 3e and 3f), voids with different sizes were observed within the adhesive. The location of the void formation was close to the adhesive-flowable composite interface (Fig. 3e). The bonding resin layer was separated at the adhesive-dentin interface at the cavity floor (Fig. 3f). In BF (Figs. 3g and 3h), the thin adhesive, less than 5 µm, was observed at the interface. However, voids with different sizes were formed between the composite and the adhesive layer. Gap formation partially took place between the composite and the adhesive layer at the cavity floor (Fig. 3h).

**DISCUSSION**

Since the gaps between the adhesive and dentin would lead to bacterial infection, sensitivity, secondary caries and pulp damage, they should be avoided from the viewpoint of clinical implication.

Internal adaptation of the cylindrical-shaped composite restorations was evaluated by using a CLSM. The CLSM observation requires only simple preparation of the specimens without desiccation and high vacuum. Therefore, the specimens can be observed under ambient conditions.

Generation of shrinkage stress during curing of the methacrylate-based light-cured composites is inevitable in a cavity, and its magnitude is strongly related to the C-factor. The larger the C-factor, the greater shrinkage stress is generated, resulting in increase of risk of gap formation at the interface. The cylindrical-shaped dentin cavity was prepared in this study after cutting the coronal enamel, to standardize the cavity size (depth and diameter). Therefore, depth of the cavity used in this study had to be limited to 1 mm. However, almost the same C-factor could be obtained for each specimen (approximately 2.3).

It is well known that an incremental technique could be a viable approach to reducing contraction stress in the situation where the first increment was effective in reducing the C-factor value. The flowable composite lining must be in effect similar to the first increment of the incremental filling technique. The low-elasticity of the flowable composite is more effective to reduce shrinkage stress of the overlying composites which is an advantage in clinic.

The two-step self-etching system of SE and the three all-in-one adhesive systems of GP, OP and BF were used in the current study. SE showed good internal adaptation to the whole cavity in both the FL and the NL groups. According to the SEM observations, SE created a homogeneous bonding layer. This was thought to be due to the adhesive resin of SE being Bis-GMA based and water/solvent free. In line with these findings, a previous study also reported that the composite restoration with SE exhibited excellent marginal integrity and no gap formation at the resin-cavity interface for enamel-dentin class I cavity. On the other hand, internal adaptation of the composite restorations depended on adhesive material and filling strategy for the all-in-one adhesives. Lining with the flowable composite improved the internal adaptation in all-in-one adhesives. However, there were some gaps formed at the interface with these adhesives, as confirmed by SEM observation. In some SEM specimens, gap formation was observed at the adhesive-dentin interface in the all-in-one adhesive systems, which may be due to weak bonding, and also to artifact from high vacuum during the specimen preparation for SEM observation.

From the SEM observations, the formation of multiple voids was observed in the three all-in-one adhesives, and the location and size of the voids were different for each adhesive system. On the mechanism of the formation of the voids in the all-in-one adhesives, it has been recently suggested that these systems may exhibit voids from two different origins: those arising from phase separation and those from water attraction and osmosis through the cured adhesive layer. These two kinds of voids can be differentiated by their location with reference to the adhesive layer, their size, and their behavior. The voids originating from a phase-separation reaction are located throughout the adhesive layer and are generally larger than osmosis droplets. The latter are not only smaller, but can also be found only at the transition between adhesive layer and lining composite, which corresponds to the oxygen-inhibition layer, a zone with uncured monomers.

The formation of voids has been frequently discussed in relation to the HEMA content of adhesives; GP is a HEMA-free adhesive, and OP and BF are HEMA-containing adhesives. HEMA is a water-soluble methacrylate monomer, which is frequently used as one of the ingredients. Recently, HEMA was shown to play an important role in preventing phase-separation reactions in one-component all-in-one adhesive systems. In these ‘one-bottle’ solutions, polar and apolar ingredients are blended together with a solvent such as water, acetone and ethanol. The solvent keeps the ingredients in solution, but once dispensed subsequent evaporation of the solvent can trigger a phase-separation reaction with the formation of multiple voids. HEMA can prevent such a phase-separation reaction by replacing the solvent and keeping the ingredients in solution, whereas the water molecules in a hydrophilic adhesive will remain hydrogen-bonded to the HEMA monomers.

For GP, voids were observed within the adhesive close to the interface between the adhesive and dentin. GP has an increased hydrophobicity and its potential for
“water-tree” affected by osmosis should be reduced because it is a HEMA-free adhesive. However, lack of HEMA may induce phase separation, and together with rapid evaporation of acetone solvent could result in the formation of voids and droplets throughout the adhesive layer. Moreover, nanoleakage within the hybrid layer has been observed even in HEMA-free self-etch adhesives, which is caused by the water movement across the resin-dentin interface from dentinal tubules, located along the interface and extending towards the adhesive layer during the bonding procedure.

On the other hand, in HEMA-containing OP, voids with different sizes were formed within the adhesive, rather closer to the adhesive-composite interface. Also, some voids were observed at the interface between the adhesive and composite. OP has two solvents of ethanol and acetone, with different vapor pressures, which also might increase the void formation within the adhesive resin. Moreover, it has been suggested that uncured small HEMA-molecules must be important components of the oxygen-inhibition layer. The strong hydrophilic character and the small dimensions of this monomer explain fast and easy osmosis leading to adhesive-composite interfacial voids in these adhesives.

In BF, the adhesive layer was thin and very homogeneous. However, the separation took place at the adhesive-composite interface, which may be due to the oxygen-inhibition layer as mentioned above. In addition, residual water/solvents might be present within the uncured layer, which would impair co-polymerization between the hydrophilic uncured resin and hydrophobic resin composite, because the presence of residual water/solvents causes a reduction in the degree of conversion.

Observation of the voids at the interface between an all-in-one adhesive and composite has also been discussed in relation to the placement pressure; with the viscous composite less voids are observed within the oxygen-inhibition layer, while with flowable resins, voids are more frequently observed.

The previous study reported that air-drying was shown to have a significant effect on the removal of the solvent and on the mechanical properties of the all-in-one adhesive systems. Moreover, the air blowing method of the adhesive may influence thickness of the bonding layer. The thickness of the adhesive layer was also influenced by shape of the cavity. In this study, the adhesive layer was thicker at the cavity corner than those of the cavity wall and cavity floor. The thickest adhesive layer was obtained at the corner of SE, because the SE bonding resin is more viscous than the all-in-one adhesive, and also mild air-blowing is recommended according to the manufacturer’s instructions to spread the adhesive. On the other hand, the manufacturers instructed to apply strong air blow to all-in-one adhesive systems. As a result, strong air-blowing made a thin adhesive layer through the whole cavity. It should be noted that the thin adhesive layers might be associated with reduced physico-mechanical strength due to oxygen inhibition of the polymerization. In addition, it was suggested that water is able to reach the oxygen inhibition layer in areas where the adhesive resin is very thin.

The current results demonstrated that the type of the adhesives and the flowable composite lining influenced the internal adaptation rates. Therefore, the null hypothesis of this study was rejected.

In conclusion, the two-step adhesive system showed a better cavity adaptation in the cylindrical shaped cavity. Lining with the flowable composite improved the internal adaptation of the composite restorations placed with the all-in-one adhesive systems. However, void formation was observed in all of the all-in-one adhesive systems at various locations. Further improvement in these all-in-one adhesive systems is desired to prevent such interfacial defects.

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