

# The effect of the elastic modulus of low-viscosity resins on the microleakage of Class V resin composite restorations under occlusal loading

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This study aimed to evaluate the effectiveness of various intermediate layers on the microleakage of Class V restorations under an occlusal load. Wedge-shaped cavities were prepared on the buccal surface of 72 extracted premolars, which were then treated with an adhesive system (One Up Bond F Plus), divided into three groups, and restored with: 1) Estelite Sigma resin composite, 2) a resin composite with Low Flow flowable composite, or 3) a resin composite with High Flow flowable composite. The specimens were subjected to a nano-indentation test to evaluate the elastic modulus of successive layers at the resin-dentine interface and were subjected to a microleakage test under either unloaded or loaded conditions. The elastic moduli were significantly different among substrates ( $p < 0.05$ ), except between the hybrid layer/Low Flow and the hybrid layer/High Flow. The elastic moduli of the Low Flow composite were higher than those of the High Flow composite. Occlusal force increased dentine leakage in the group that was restored without flowable composites.

**Keywords:** Elastic modulus, Elastic cavity wall, Microleakage

## INTRODUCTION

The demand for restoration of cervical defects, such as cervical erosion and root caries, has increased significantly in recent years<sup>1</sup>. Resin composite is one of the materials of choice for this purpose because of its aesthetic qualities.

The characteristic polymerization of resin composites is one of the main factors that affect the success of restoration using resin composites. Shrinkage due to polymerization likely causes marginal leakage, tooth fracture, resin composite fracture, and dislodgement of the restoration. Contraction stress from polymerization is related to the reduction in the flow capacity of the composite when it becomes constrained, to the amount of volumetric shrinkage, and to the stiffness of resin composites<sup>2,3</sup>.

Masticatory force is an additional factor that affects the durability of the composite material after the restoration process. The clinical success of composite restorations may be fundamentally dependent on effective and durable adhesion to enamel and dentine<sup>4-6</sup>, especially under occlusal loading. The occlusal force might also be partly responsible for the development of cervical lesions as well as the failure of Class V restorations. An elastic bonding area at the tooth-resin interface has been proposed as an inherent buffer to compensate for polymerization contraction stress and occlusal stress on the restorative resin<sup>7,8</sup>. A currently used practical technique for the creation of elastic bonding areas involves applying a layer with a relatively low modulus of elasticity as an intermediary between the composite and the tooth<sup>9,10</sup>. Van Meerbeek and colleagues<sup>8</sup> confirmed the effectiveness of flexible and low-viscosity intermediate layers as shock absorbers<sup>11,12</sup>. The relationship between the elastic

moduli of these layers is referred to as the “Elastic Cavity Wall Concept”<sup>9,10</sup>. Reduced marginal leakage as a result of the stress-relief function of these materials has been demonstrated<sup>8,13-15</sup>.

It is interesting to compare the effectiveness of various current flowable resin composites, all of which have different elastic moduli, as elastic cavity walls in class V resin composite restorations under mechanical loading. This research was conducted by measuring the elastic moduli of various substrates at the resin-dentine interfaces and investigating the marginal leakage at the enamel and dentine margins of Class V resin composite restorations.

The objectives of the study were: 1) to compare the moduli of elasticity of successive layers (including various flowable composites used as intermediate layers) at resin-dentin interfaces and 2) to compare the microleakage of Class V resin composite restorations with various intermediate layers with and without occlusal load.

## MATERIALS AND METHODS

### *Tooth specimen preparation*

Seventy-two freshly extracted, sound premolars were used in this study. The selected teeth were free from decay, cracks, and restorations. After extraction, the teeth were cleaned with pumice and stored in 0.1% thymol solution at 4°C. The 0.1% thymol solution was used for infection control of extracted teeth because it is a topical antiseptic and mold growth inhibitor<sup>16</sup>. It has been reported that thymol solution has no influence on bond strength when teeth are stored for up to 6 months<sup>17</sup>. The stored teeth in this study were used for testing within one month of preparation. The thymol solution was changed to normal saline one week before

use. Wedge-shaped cavities were introduced using a water-cooled, high-speed handpiece and fissure diamond burs (D8, Intensive, Lugano-Grancia, Switzerland) at the cemento-enamel junction on the buccal surface of the teeth. Each bur was used for the preparation of four cavities and was then replaced by a new bur. The cavity size was 2.0 mm high×4.0 mm wide. The depth of the cavity was approximately 2.0 mm, as determined by a periodontal probe. The occlusal margin of the cavity was located on the enamel, and the gingival margin was located on the cementum. The prepared teeth were divided randomly into three groups of 24 teeth and were kept in normal saline solution.

#### Restorative procedures

The materials used in the present study and their compositions are described in Table 1. The prepared teeth in each group were restored according to the following conditions.

Group 1: The cavity was treated with One Up Bond F Plus following the manufacturer's instructions. The excess water on the prepared surface was removed by blowing air. One drop of Bonding Agent A and 1 drop of Bonding Agent B were mixed until the mixed bonding agents turned homogeneously pink. The mixture was applied to the prepared surfaces with a rubbing motion for 10 seconds and then gently air-dried. The bonded specimen was light-cured for 10 seconds with a halogen-curing unit (Eliper Trilight, 3M EPSE, Minnesota, USA). The pink color turned to a pale brown after light irradiation. A resin composite (Estelite Sigma) was placed into the cavity with a bulk

technique and cured for 40 seconds. The finishing and polishing steps were performed immediately after polymerization with abrasive disks (Soflex Disk, 3M ESPE, Minnesota, USA).

Group 2: The cavity was treated with One Up Bond F Plus and cured for 10 seconds. The cavity was lined with Pafique Estelite LV (Low Flow) flowable composite, gently air-blown to obtain a thin layer of material, and cured for 40 seconds. The specimen was then filled with the resin composite, cured for 40 seconds, and polished.

Group 3: The cavity was treated with One Up Bond F Plus and cured for 10 seconds. The cavity was lined with Pafique Estelite LV (High Flow) flowable composite and cured for 40 seconds. The specimen was then filled with the resin composite, cured for 40 seconds, and polished.

After complete restoration, the specimens were kept in distilled water at 37°C for 24 hours.

#### Evaluation of the modulus of elasticity

The four specimens in each group were sectioned buccolingually with a diamond saw (Microcutting instrument, Accutom-50, Struers, Copenhagen, Denmark) and embedded in epoxy resin (Epon 815, Nissin, Tokyo, Japan) in a polyvinyl chloride (PVC) ring. The embedded specimens were polished with wet sandpaper of 600, 800, 1000, and 1200 grit and with diamond paste with 6, 3, 1, and 0.25 μm grains. The polished specimens were attached into a nano-hardness testing system (ENT-1100, Elionix, Tokyo, Japan) for measurement of the elastic modulus under 5 gf loading on the dentine, hybrid layers, adhesive, and resin

Table 1 Compositions of the materials investigated in this study

Material	Type	Composition	Batch No.	Manufacturer
One Up Bond F Plus	self-etching adhesive systems	water, methyl methacrylate, hydroxyethyl methacrylate, coumerin dye, methacryloyloxyalkyl acid phosphate, methacryloxyundecane dicarboxylic acid (MAC-10), multifunctional methacrylic monomer, fluoroaluminosilicate glass, photoinitiator (aryl borate catalyst)	501M	Tokuyama Tokyo, Japan
Pafique Estelite LV (Low Flow)	flowable composite	mixture of silica-zirconia and silica-titania filler (65%) methacrylate (35%)	7051	Tokuyama Tokyo, Japan
Pafique Estelite LV (High Flow)	flowable composite	mixture of silica-zirconia and silica-titania filler (68%) methacrylate (32%)	305	Tokuyama Tokyo, Japan
Estelite Sigma	universal composite	silica-zirconia filler (82% by weight) bisphenol A diglycidyl ether dimethacrylate (Bis-GMA) triethylene glycol dimethacrylate photoinitiator	E5011	Tokuyama Tokyo, Japan

composite at the resin-dentine interfaces. The nano-hardness testing machine was a depth computer-controlled machine with a three-sided pyramidal diamond probe. The size of the imprint after the indentation test was approximately 1  $\mu\text{m}$  in diameter, which was small enough to evaluate the modulus of elasticity at specific areas such as the adhesive and hybrid layers at the resin-dentine interface<sup>18</sup>. Ten nano-indentations were performed for each successive layer of each specimen at 5  $\mu\text{m}$  intervals. Young's modulus of elasticity (MPa) was calculated and recorded with an attached computer.

#### Application of cyclic loading

The remaining 20 teeth from each group were further divided into two subgroups. The first subgroup (10 teeth) was used as a control without mechanical loading. The second subgroup (10 teeth) was used as an experimental group with mechanical loading. In the mechanical loading group, the teeth were mounted onto the cyclic loading machine with a self-cured acrylic resin (Instant Tray Mix, Lang Dental Manufacturing Co, IL, USA). The mechanical stress was stimulated at a frequency of 1.5 Hz for 250,000 cycles at 50 N<sup>19-21</sup> in water using a cyclic loading machine. The loaded force was applied vertically parallel to the long axis of the tooth at occlusal surfaces using an aluminum steel rod indenter with a 2 mm diameter. The loaded specimens were then removed from the acrylic mounting resin.

#### Evaluation of microleakage

The specimens from both the control and experimental groups were sealed with two coats of nail varnish, leaving a 1 mm window around the restorations. The sealed specimens were then immersed in a 0.5%

methylene blue dye solution at room temperature for 3 hours. Methylene blue was used as a dye tracer because its particle size is about 0.68 nm and it has a molecular weight of 319 g/mol, which is smaller than both bacteria and bacterial toxins<sup>22</sup>. Additionally, methylene blue has a blue color that is easy to identify under the microscope. Furthermore, its pH is around 7.0, which might have no effect on the selected adhesive system (acid monomer).

According to the pilot study, a 5 sec immersion in methylene blue was not enough to identify the marginal leakage differences between the groups in our study. We found that the suitable immersion time for our study was 3 hours. Thus, the immersion time for dye tracer in this study was 3 hours.

After immersion, specimens were cleaned and sectioned bucco-lingually into three slabs of 0.7-mm thick specimens using a diamond saw. The dye penetration at the resin-tooth interface was observed and measured in millimeters under a measuring microscope (Nikon, Tokyo, Japan). The means of the dye penetration values from the three slabs of each tooth were calculated and recorded as representative data for each tooth. Thus, the thickness of the adhesive resin and flowable composites were observed and measured under the measuring microscope.

## RESULTS

#### Evaluation of the modulus of elasticity

The modulus of elasticity of each layer is shown in Figure 1. Analysis of variance (ANOVA) and Dunnett's *t*-test revealed that there were significant differences between the layers at the resin-dentine interfaces, with the exception of between the hybrid layer/Low Flow

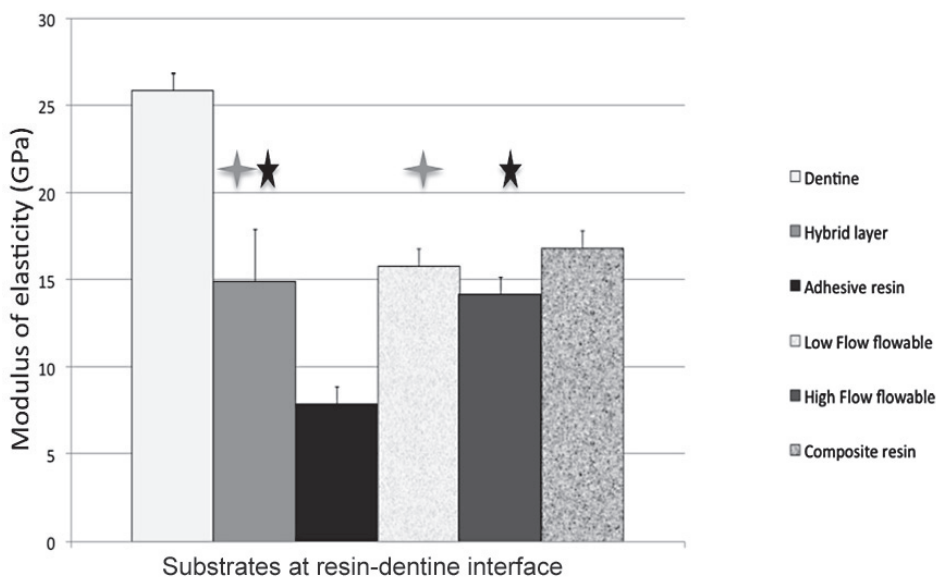


Fig. 1 Modulus of elasticity (GPa) of substrates at resin-dentine interfaces. Columns with the same symbol demonstrate no statistically significant difference.

Table 2 Means and standard deviations of enamel microleakage in millimeters ( $n=10$ )

	Group 1	Group 2	Group 3
Unload	0.3696 (0.2908) <sup>a</sup>	0.3028 (0.1104) <sup>a</sup>	0.2763 (0.1603) <sup>a</sup>
Load	0.4634 (0.7565) <sup>a</sup>	0.3315 (0.1313) <sup>a</sup>	0.3825 (0.1124) <sup>a</sup>

The numbers in parentheses represent the standard deviations. Data with the same superscript demonstrate no statistically significant difference.

Table 3 Means and standard deviations of dentine microleakage in millimeters ( $n=10$ )

	Group 1	Group 2	Group 3
Unload	0.3806 (0.1301) <sup>a</sup>	0.3091 (0.1285) <sup>a</sup>	0.3098 (0.2426) <sup>a</sup>
Load	2.0015 (0.4405) <sup>b</sup>	0.4773 (0.6979) <sup>a</sup>	0.6209 (0.7279) <sup>a</sup>

The numbers in parentheses represent the standard deviations. Data with the same superscript demonstrate no statistically significant difference.

flowable composite and hybrid layer/High Flow flowable composite. The moduli of elasticity of the dentine, hybrid layer, adhesive resin, Low Flow flowable composite, High Flow flowable composite, and resin composite were  $25.83 \pm 1.11$ ,  $14.89 \pm 2.88$ ,  $7.88 \pm 0.50$ ,  $15.78 \pm 0.46$ ,  $14.14 \pm 1.05$ , and  $16.81 \pm 0.81$  GPa, respectively.

#### Evaluation of microleakage

A two-way ANOVA revealed that the two factors (occlusal force loading ( $p=0.399$ ) and flowable composites ( $p=0.618$ )) had no significant effect on enamel microleakage values. The means and standard deviations of the enamel microleakage values are shown in Table 2. No statistically significant differences were found among the groups.

The two-way ANOVA examining dentine microleakage revealed a significant effect of two factors, the occlusal force loading ( $p<0.01$ ) and the flowable composites ( $p<0.01$ ). The means and standard deviations of the dentine microleakage values are given in Table 3. No significant differences between the three groups were found for the unloaded condition. Occlusal loading significantly increased dentine leakage only in the group restored without flowable composites.

The thickness of adhesive layers, High Flow flowable composite layers, and Low Flow flowable composite layers for all groups were  $0.03 \pm 0.01$  mm,  $0.14 \pm 0.02$  mm, and  $0.18 \pm 0.02$  mm, respectively. The statistical analysis revealed that there were significant differences ( $p<0.01$ ) among the three layers.

## DISCUSSION

The elastic cavity wall created by the application of a low viscosity resin<sup>9,11,23</sup> acts as an inherent buffer to compensate for the polymerization contraction of resin composites<sup>9</sup> and transfers occlusal stress to the underlying tooth structure<sup>8</sup>. Therefore, these walls reduce the composite restoration failure rate *in vitro*<sup>24</sup>. Two testing conditions were examined in this study. First, the unloaded condition was used as a control to

evaluate the effect of resin composite contraction stress on microleakage. Second, to simulate a clinical situation, a loading condition was used to evaluate the effect of occlusal loading on microleakage. In this study, a 50 N occlusal force was applied parallel to the long axis of the tooth for 250,000 cycles under 100% humidity. This loading condition has been shown to model one year of clinical wear<sup>25,26</sup>.

The elastic modulus of dentine was 25.83 GPa MPa. This value is in the line with results found in studies by Urabe and colleagues<sup>27</sup> and Senawongse and colleagues<sup>28</sup>. The elastic moduli of the dentin were greater than those of the hybrid layer, adhesive layer, low viscosity resins, and resin composite. This finding is similar to results reported in a previous study<sup>8</sup>. The Low Flow flowable composite demonstrated a greater elastic modulus than High Flow flowable composite. Because the filler content of Low Flow flowable composite (68%) is higher than that of High Flow flowable composite (65%), the differences in compositions might be responsible for this disparity. A progressive gradient of elasticity was observed at the resin-dentine interface, ranging from the stiff dentine to the more elastic hybrid layer, adhesive layer, and low viscosity resin to the resin composite restoration. This gradient in the elastic bonding area may indicate a strain capacity capable of relieving stresses due to polymerization shrinkage and occlusal loading between the composite restoration and rigid dentine substrate<sup>8</sup>.

For Class V restorations, the thicker layers of the relatively low-modulus resin or adhesive can significantly reduce the contraction stress of resin composites and consequently reduce the overall degree of marginal leakage<sup>13-15</sup>. The thicknesses of these layers were measured and analyzed statistically. The statistical analysis confirmed the significant differences in thickness ( $0.14 \pm 0.02$  mm for High Flow flowable composite and  $0.18 \pm 0.02$  mm for Low Flow flowable composite). However, an effect due to the differential thickness of the low viscosity resin layers on the marginal leakage could not be observed in this study

because no significant differences in microleakage were found between the groups lined with different flowable composites.

The marginal seal is one of the most important factors influencing the success of a restoration. Many studies have shown that bonding of restorative materials to enamel with total-etching adhesive systems is adequate to resist contraction stress<sup>29-31</sup>. Thus, several studies have demonstrated more microleakage at the enamel margin when self-etching adhesive systems were used<sup>32,33</sup>. Low amounts of microleakage occurring along the enamel margins were shown. The low-viscosity resin layer exhibited no ability to reduce enamel microleakage in this study, because no significant differences in microleakage were found among groups. These results are in agreement with those from a previous study<sup>34</sup>.

Various degrees of microleakage occurred along the gingival margins placed on the dentine. The low-viscosity resin layer did not reduce microleakage at the dentine margins in the control group in this study. This finding indicated that the application of a flowable resin composite did not produce any benefit in reducing the microleakage caused by polymerization contraction of the resin composite. This finding stands in contrast to those of several other studies, which reported encouraging results in the reduction of microleakage with the use of flowable composite restorative materials<sup>35,36</sup>. This result might be caused by the slight difference between the elastic modulus obtained from the flowable composite and that of Esthelite Sigma. Occlusal loading increased the microleakage when Class V cavities were restored without flowable composites. There was a significant difference between the group restored without flowable composites and the groups restored in combination with either High Flow flowable composites or Low Flow flowable composites under the occlusal loading condition. The application of flowable composites may relieve occlusal stress and result in reducing marginal leakage.

No effect due to the use of flowable composites with different elastic moduli was observed in this study because there was no significant difference in leakage when Low and High Flow flowable composites were used in the unloaded and loaded conditions. For the loaded condition, however, the leakage in groups with a flowable composite was significantly less than that in groups without a flowable composite. The elastic modulus of the adhesive (7.88±0.50 GPa) might not be sufficient to withstand the occlusal force and prevent microleakage, especially at the dentine margin. It has been reported that the incompatibility of one-step self-etching adhesive with resin composite<sup>37</sup> and the lack of hydrophobic resins for hybrid layer formation of this adhesive compromised the dentine bond<sup>38</sup>. Furthermore, an additional intermediate flowable composite was used and reported to increase the adhesion<sup>39</sup>. According to that result, marginal sealing may be improved by the application of the flowable composite.

The results of this study suggested that the use of a self-etching adhesive resin in combination with the flowable composites may be more advantageous than self-etching adhesive resin alone to compensate for occlusal stress.

## CONCLUSION

Within the limitations of this study, our findings suggest that elastic moduli of flowable composites ranging from 14.14 to 15.78 GPa might be sufficient to compensate for stress generated by the occlusal forces. The application of these composites reduced marginal leakage at the dentine margin of wedge-shaped composite restorations.

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