

# Microtensile Bond Strengths to Cavity Floor Dentin in Indirect Composite Restorations using Resin Coating

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## ABSTRACT

**Purpose:** The aims of this study were (1) to evaluate the effect of a resin coating on the microtensile bond strengths ( $\mu$ -TBSs) of indirect composite restorations bonded to dentin with resin cement and (2) to compare the  $\mu$ -TBSs with that of a directly placed composite.

**Materials and Methods:** Class I cavities were prepared in extracted human molars. The specimens were divided into five groups: For the indirect restorations, the cavity surfaces of the control group were left uncoated (group 1), while the surfaces of the experimental groups were resin coated with a dentin bonding system, Clearfil Protect Bond (PB; groups 2 and 3), or with a combination of PB and a flowable resin composite, Protect Liner F (PLF; group 4). The cavities were temporized for 1 day. Indirect composite restorations (Estenia) were cemented with a resin cement (Panavia F). Pretreatment with ED Primer II was performed in the groups 1, 3, and 4. For the direct restorations, the cavities were restored with PB and a direct composite (Clearfil AP-X; group 5). After 24 hours of water storage,  $\mu$ -TBSs were measured at a crosshead speed of 1 mm/min. The data were analyzed with one-way analysis of variance and Sheffe's test ( $p < 0.05$ ). In addition, fracture modes were determined visually and by scanning electron microscopy.

**Results:** A combination of PB and PLF showed significantly higher bond strengths compared with the original bond strength of Panavia F and the single use of PB ( $p < 0.05$ ). However, the highest bond strengths were obtained when PB was used for direct composite restorations ( $p < 0.05$ ).

**Conclusion:** The application of a resin coating consisting of a self-etching primer dentin bonding system and a flowable resin composite significantly improved the  $\mu$ -TBS of indirect restorations bonded to dentin using resin cement.

## CLINICAL SIGNIFICANCE

A resin coating should be required to improve dentin bonding performance of Panavia F in indirect restorations. However, direct composite restorations still provide higher bond strength compared to indirect restorations.

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## INTRODUCTION

Recently, advancements in adhesive dentistry have brought significant changes in the treatment of caries. Direct composite restorations are preferred over indirect composite restorations when treating caries in posterior teeth because they require minimal intervention and cavity preparation.<sup>1-3</sup> However, indirect composite restorations are usually recommended when teeth require large restorations. As indirect restorations rely on cements to remain in place, the final outcome depends on the successful selection of the cement. However, the dentin bond strength of resin cement is less than those of resin adhesives.<sup>4</sup>

In the early 1990s, a resin-coating technique was developed in which a hybrid layer and a tight-sealing film are produced on the dentin surface with a dentin adhesive system and a low-viscosity microfilled resin.<sup>5,6</sup> It covers and protects the prepared dentin immediately after cavity preparation and enables good bonding of the resin cement<sup>7,8</sup> and adaptation of composite inlays,<sup>9</sup> if the proper combination of adhesive and low-viscosity microfilled resin is selected.<sup>10</sup> Therefore, this technique has the potential to minimize pulp irritation and postoperative sensitivity.<sup>11,12</sup>

Several reports<sup>13-15</sup> have shown that self-etching primer adhesive systems can be used for composite

restorations because of their ability to provide high bond strength to dentin and efficient marginal sealing. The two-step self-etching primer adhesive system, Clearfil Protect Bond (PB; Kuraray Medical, Tokyo, Japan), is composed of an antibacterial primer containing an antibacterial monomer (12-methacryloyloxydodecylpyridinium bromide [MDPB]) and a fluoride-releasing adhesive, which has shown good dentin bond durability and the potential for inhibiting secondary caries around composite restorations.<sup>16-19</sup>

The microtensile bond strengths ( $\mu$ TBSs) of self-etching primer adhesive systems to cavity floor dentin are lower than those to flat dentin surfaces. The bond strengths may be influenced by the cavity configuration factor,<sup>20</sup> the depth of the cavity, the burs selected for cavity preparation, the increment of composite, and the distance from the light source.<sup>21-24</sup>

However, there is little information on the bonding performance of indirect composite to cavity floor dentin using the resin-coating technique. Therefore, the purpose of this study was to evaluate the microtensile bond strengths of direct and indirect composite restorations to cavity floor dentin in Class I cavities. The null hypothesis of this study was that the microtensile bond strengths were

not affected by the bonding protocols and the restorative methods.

## MATERIALS AND METHODS

The materials used in this study are listed in Table 1. The method of specimen preparation is illustrated in Figure 1. Fifteen caries-free human molars were used in this study. Class I cavities with mesial-distal dimensions of ca  $4 \times 2 \times 3$ -mm depth were initially prepared with a diamond bur (ISO #109, GC, Tokyo, Japan). The cavity surfaces were then finished with a fine steel bur (ISO #012, GC) at a low speed of 5,000 rpm with water spray. Thereafter, the teeth were randomly divided into five groups of three teeth each.

For the indirect composite restorations, the cavity surfaces of the control group (group 1) were left without conditioning. For groups 2 and 3, the dentin bonding system PB was applied to the dentin surfaces and light-cured according to the manufacturer's instructions (see Table 1) for resin-coating of the cavity. For group 4, the PB was first applied to the cavity surfaces and immediately thereafter, a flowable resin composite, Protect Liner F (PLF; Kuraray Medical) was applied with a brush and light-cured for 20 seconds for the resin coating. The light source used in this study was Curing Light XL 3000 (3M-ESPE, Seefeld, Germany), with 600 mw/cm<sup>2</sup>.

TABLE 1. MATERIALS USED IN THIS STUDY

Material	Batch Number	Composition	Directions
DBS	Primer 000010	Primer: MDP, HEMA, MDPB,	Primer: apply 20 seconds,
Clearfil Protect Bond	Bond 000017	dimethacrylates, photoinitiator, water, ethanol	dry
		Bond: MDP, HEMA, dimethacrylates, photoinitiator, surface-treated NaF, microfiller	Bond: apply, dry, 20-second polymerization
Flowable Resin Composite	0052	Bis-GMA, TEGMA, microfillers, photo initiator	Apply on the cured DBS, 20-second polymerization
Protect Liner F			
Resin Cement	ED Primer II	ED Primer II A: MDP, HEMA, 5-NMSA, chemical initiator; B: 5-NMSA, water	Primer: apply 60 seconds, dry
Panavia F	A 00195A		
	B 00076A	A paste: quartz glass, microfiller, MDP, methacrylates, photoinitiator	Mix A + B paste, cement indirect composite inlays, polymerize for 60 seconds
	A paste 00073A		
	B paste 00036A	B paste: barium glass, NaF, methacrylates, chemical initiator	
Restorative Materials	01147A	Silanated barium glass, silica, colloidal silica, Bis-GMA, TEGDMA, photo initiator	Light-curing occlusal surface for 20 seconds
Direct Composite			
Clearfil AP-X			
Shade A3			
Indirect Composite	00209B	Hydrophobic methacrylates, 72 wt% microfiller, 16% superfine fillers	Polymerize in Alpha Light for 3 minutes, heat cure for 15 minutes
Estenia			
Shade DA3.5			

Bis-GMA = Bis phenol A glycidine dimethacrylate; DBS = dentin bonding systems; HEMA = hydroxyethyl methacrylate; MDP = 10-methacryloxydecyl dihydrogen phosphate; MDPB = 12-methacryloyloxydodecylpyridinium bromide; 5-NMSA = N-Methacryloyl 5 amino salicylic acid; NaF = sodium fluoride; TEGDMA = triethylene glycol dimethacrylate; TEGMA = triethylene glycol methacrylate.

The freshly prepared cavity surfaces (group 1) and the coated surfaces (groups 2, 3, and 4) were then temporized with a water-setting temporary filling material, Cavit-G (3M-ESPE), to simulate clinical practice.<sup>10</sup> After 24 hours of storage in distilled water at 37°C, the temporary filling material was removed with an excavator and the surfaces wiped with a cotton pellet soaked in ethanol for 10 seconds. The resin-coated surfaces were then treated with 37% phosphoric acid (K-etchant, Kuraray Medical) for

10 seconds and rinsed and dried to remove any debris. ED Primer II (Kuraray Medical) was applied to the cavity surfaces of the control teeth of group 1 for 30 seconds, and applied to the resin-coated cavities for 10 seconds in groups 3 and 4, respectively. On the other hand, ED Primer II was not used in group 2.

The fabrication and pretreatment of the indirect composite inlays (Estenia, Kuraray Medical) were as follows: the composite inlays were

prepared and light-cured in a laboratory light-curing unit (Alpha Light-II, J. Morita Co., Kyoto, Japan) for 3 minutes followed by heat polymerization at 110°C for 15 minutes in an oven (KL-100, Kuraray Medical) according to the manufacturer's instructions. The internal surfaces of the polymerized composite inlays to be cemented were sandblasted and then treated with 37% phosphoric acid for 10 seconds, rinsed, dried, and silanized with a mixture of Clearfil Protect Bond Primer and Porcelain Bond

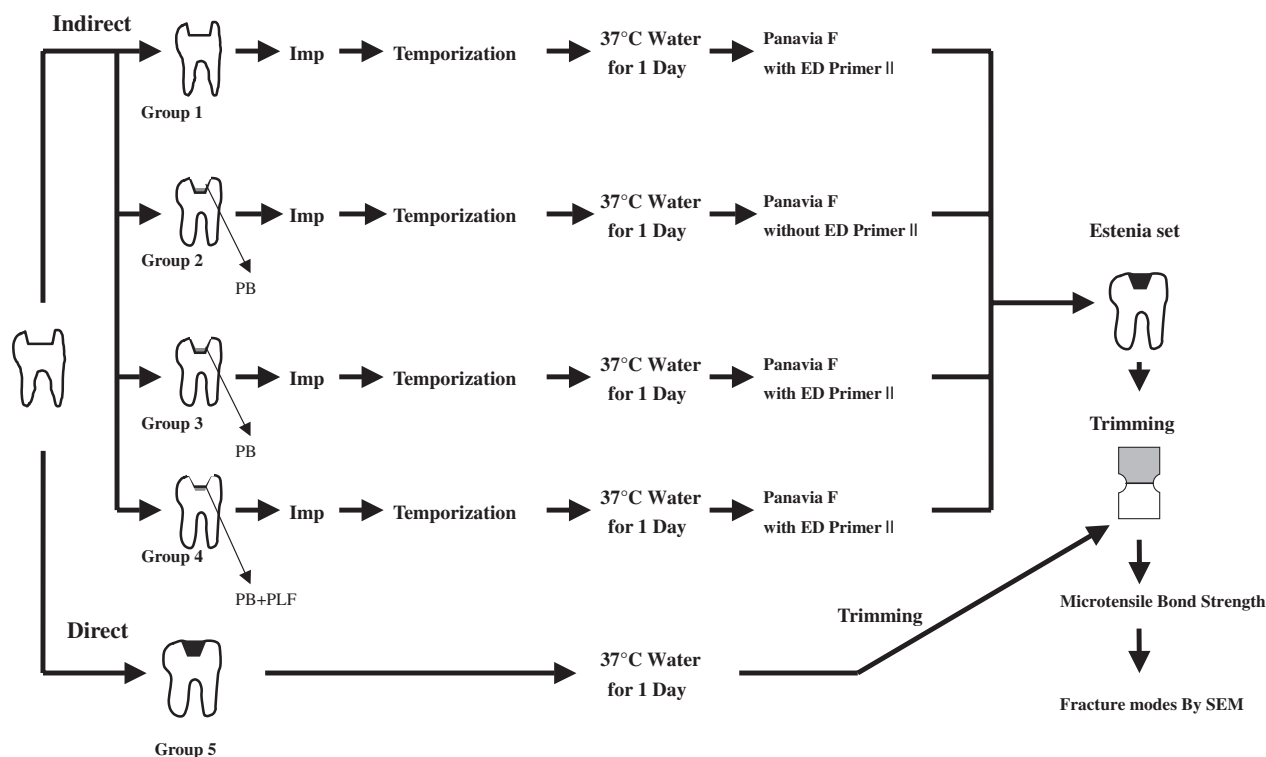


Figure 1. Illustration of specimen preparation. PB = Clearfil Protect Bond; PLF = Protect Liner F; SEM = scanning electron microscopy.

Activator (Kuraray Medical). The composite inlays were cemented with Panavia F (Kuraray Medical) and light-cured for 60 seconds. For the direct restorations (group 5), following an application of PB according to the manufacturer's instructions, the entire cavities were filled with a resin composite (Clearfil AP-X, Kuraray Medical) and light-cured from an occlusal direction for 20 seconds (Curing Light XL 3000).

All specimens were stored in distilled water at 37°C for 24 hours. Thereafter, each specimen was

sectioned perpendicular to the bonded interface to obtain four to five 1.0-mm-thick slabs. Each slab was then trimmed with a superfine diamond bur (V16ff, GC) to obtain an hourglass shape so that the narrowest portion at the adhesive interface had a surface area of  $1.0 \pm 0.2 \text{ mm}^2$ , in preparation for the  $\mu$ -TBS test. Thereafter, each specimen was attached to a Bencore-Multi T testing apparatus (Danville Engineering Co., San Ramen, CA, USA) with Zap-it cyanoacrylate adhesive (Dental Ventures of America, Corona, CA, USA) and placed in a universal testing machine (EZ Test,

Shimadzu Co., Kyoto, Japan) for  $\mu$ -TBS testing at a crosshead speed of 1.0 mm/min.

The data were analyzed using one-way analysis of variance and Scheffe's *F*-test at a 5% level of significance.

After  $\mu$ -TBS testing, fracture modes were inspected visually. The specimens were then fixed in 10% formalin for further observation using scanning electron microscopy (SEM). The fractured specimens were gold coated for SEM observation (JSM-5310 LV, Jeol, Tokyo, Japan). The mode of failure was

classified into one of four categories: A, complete or partial adhesive fracture; B, failure at the interface between coating and cement; C, completely cohesive failure in resin cement; and D, cohesive failure in adhesive. The percentage of each fracture pattern was calculated for each group.

## RESULTS

The bond strength data and failure modes are presented in Table 2. A mean bond strength of the original  $\mu$ -TBS of the resin cement to dentin (group 1) was 9.0 MPa. Application of a single coating of PB without pretreatment of ED Primer II (group 2) and with the pretreatment (group 3) resulted in mean bond strengths of 10.5 and 12.9 MPa, respectively. There was no significant difference in  $\mu$ -TBS among groups 1, 2, and 3. Application of a coating with the combination of PB and PLF (group 4) yielded a mean bond strength of

32.9 MPa, which was significantly higher than the bond strengths of groups 1, 2, and 3 ( $p < 0.05$ ). Group 5 exhibited the highest bond strengths when PB was used for direct composite restorations (47.7 MPa) ( $p < 0.05$ ).

## Fracture Patterns

SEM photomicrographs of typical fracture patterns are shown in Figure 2. In the noncoated group (group 1), all specimens showed complete or partial adhesive failure (A) (see Table 2). Specimens that received a resin coating showed failure at the interface between the coating material and resin cement (B). The other specimens showed completely cohesive failure in the resin cement (C). When direct composite restorations were bonded with PB, all specimens showed cohesive failure in adhesive (D).

## DISCUSSION

The bonding system, PB, is a two-step self-etching primer adhesive

system, which is composed of a self-etching primer containing the antibacterial monomer, MDPB, and a fluoride-releasing adhesive. The antibacterial monomer MDPB is a polymerizable biocide and has strong bactericidal activity against oral bacteria.<sup>25,26</sup> The antibacterial agent is immobilized in the polymer network by copolymerization of MDPB, and the cured resin containing MDPB exhibits inhibition of bacterial growth.<sup>27</sup> Therefore, a dentin bonding system incorporating MDPB can show antibacterial effects before and after the curing process.<sup>28–31</sup> In addition, fluoride ions released from the adhesive system may also inhibit secondary caries by the remineralization of the dentin around the restoration.<sup>18</sup>

When Class I cavities are prepared, both the cavity configuration and the effect of dentin depth may result in lower bond strengths to

**TABLE 2. MICROTENSILE BOND STRENGTHS TO CAVITY FLOOR DENTIN AND THE FRACTURE MODES IN DIRECT AND INDIRECT COMPOSITE RESTORATIONS**

	Microtensile Bond Strength	Fracture Modes (%)			
		A	B	C	D
Indirect Composite					
Group 1—Noncoating	9.0 ± 3.9*	100	0	0	0
Group 2—Coating with PB without ED Primer II	10.5 ± 3.0*	0	87	13	0
Group 3—Coating with PB with ED Primer II	12.9 ± 7.8*	0	80	20	0
Group 4—Coating with PB + PLF	32.9 ± 12.7	0	73	27	0
Direct Composite					
Group 5—PB	47.7 ± 9.1	0	0	0	100

PB = Clearfil Protect Bond; PLF = Protect Liner F; Fracture modes: A = complete or partial adhesive failure; B = failure at the interface between coating material and resin cement; C = completely cohesive failure in resin cement; D = cohesive failure in adhesive.  
Mean  $\pm$  SD; N = 15.

\*No significant difference ( $p > 0.05$ ).



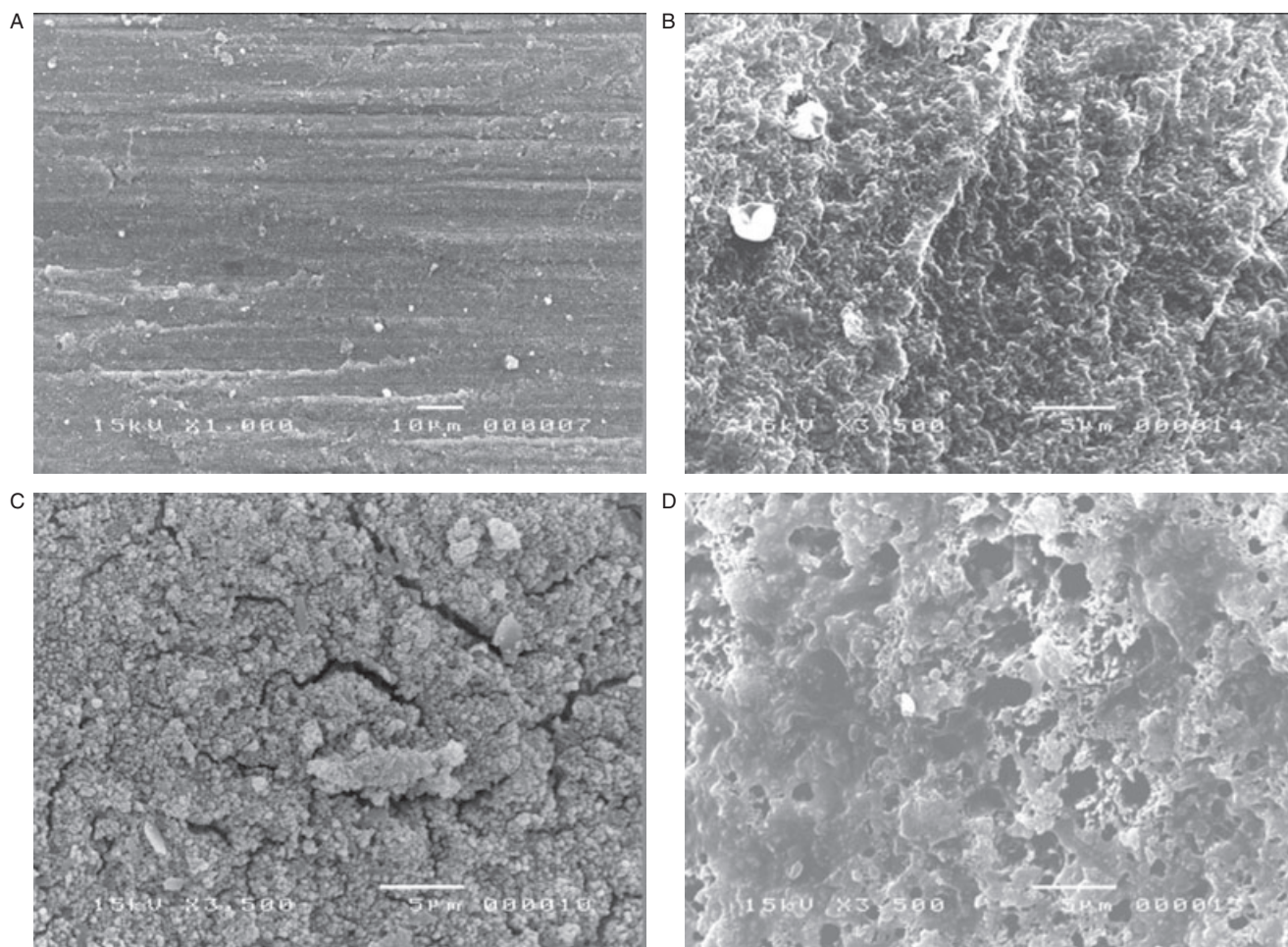


Figure 2. Scanning electron photomicrograph to illustrate the type A to D fracture pattern. A, Complete or partial adhesive failure. B, Failure at the interface between coating and cement. C, Complete cohesive failure in resin cement. D, Cohesive failure in adhesive.

the cavity floor.<sup>21,32,33</sup> The C-factor is the ratio of the bonded surface area to the unbonded or free surface area.<sup>20</sup> As the ratio is the largest in Class I cavities, the competition between polymerization shrinkage and adhesion between the resin and dentin is maximized, when placing photocured resin composite into a Class I cavity using a bulk-filling technique.<sup>20</sup>

The microtensile bond strength of the PB to cavity floor dentin in direct restorations demonstrated the highest bond strength in all groups in this study. The  $\mu$ -TBS of PB in the Class I cavities was higher compared with those of previous experiments.<sup>34,35</sup>

The resin-coating technique enables coverage and protection of the

prepared dentin immediately after cavity preparation. It also improves marginal and interfacial adaptation of the indirect composite restorations. Application of a resin coating consisting of the PB and PLF resulted in significantly higher dentin bond strengths of Panavia F than its original bond strength and that of the single coating using PB. It has been reported that the

selection of the materials for impression making<sup>36,37</sup> and the provisional restoration<sup>38</sup> after application of the resin-coating technique are important for success of the final restoration. A water-setting material, Cavit-G, was used for temporization in this study because the material does not influence the bond strength to the resin-coated cavity. The use of a resin-based material is not available for resin-coated dentin because the material glues to the coating material. Also, eugenol-based temporary cement is not recommended because of the possibility of polymerization inhibition of the resin cement.

Contrary to our expectations, there was no significant difference in the bond strength between without and with the use of ED Primer II in groups 2 and 3. ED Primer II is a self-etching primer containing an aromatic sulfinic salt, which is believed to accelerate the interfacial polymerization between the resin coating surface and the resin cement.<sup>39</sup> This result indicates that the pretreatment of ED Primer II did not affect the dentin bond strength in the Class I cavities of this study.

Applying a flowable resin composite such as PLF on the cured adhesive can polymerize the oxygen inhibition layer, which contains uncured resin. The uncured resin of

the oxygen inhibition layer may subsequently polymerize with the diffusion of free radicals from the flowable resin. In addition, the flowable resin composite may protect the adhesive from being torn at the time of removal of the temporary cement. Furthermore, the flowable resin composite can function as a stress breaker. The moduli of elasticity are 3 to 4 GPa in unfilled adhesive, 6 to 10 GPa in flowable resin, and 15 to 20 GPa in heavily filled direct or indirect composite.<sup>40–42</sup> Thus, the resin coating, a combination of a dentin bonding system and a low viscosity resin composite, creates a thick sealing film.<sup>9</sup>

As the bond strengths improved, the failures shifted from adhesive to failures within the cement or between the coating and cement. This suggests that the cement may be the weakest link in these complex bonded assemblies.

A single application of the adhesive system on the prepared cavity is claimed to protect the exposed dentin and prevent postoperative sensitivity. However, previous studies have shown that application of a flowable resin composite to the cured adhesive provided perfect sealing of the dentinal margins in direct composite restorations.<sup>8,9,37,43</sup> However, the bond strength of direct composites was significantly

higher than those of indirect restorations even with the resin-coating technique.

The resin coating has been shown to improve dentin bonding of resin cement, whereas no effect was observed on enamel bonding.<sup>44</sup> The bonding performance of indirect restorations to enamel was clinically acceptable, which is almost identical to direct resin composite. Also, the enamel surfaces did not demonstrate any statistical difference in bond strengths with or without the use of a low-viscosity microfilled resin.

As direct restorations are less invasive than indirect restorations, they should be the first choice for posterior restorations. However, in certain situations, such as when large restorations are required, indirect restorations are indicated. If indirect restorations are selected, a resin coating consisting of a dentin bonding system and a flowable resin composite should be applied to the dentin surface to improve the bond strengths of resin cement to dentin.

## CONCLUSIONS

Within the limitations of this study, the following conclusions were drawn:

1. The cement bond strength was significantly increased by a resin coating of PB and PLF.

2. Resin coating with PB could not enhance the original resin cement bond strength.
3. The dentin bond strength of resin cement is still lower than that of a direct resin composite, even with the resin coating.

#### DISCLOSURE AND ACKNOWLEDGMENTS

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