Effect of surface conditioning agents on the repair bond strength of an indirect hybrid composite

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Purpose: The objective of the current study was to evaluate the effect of silane primers and unfilled resin bonding agents when used for bonding between layers of a light-activated indirect composite material (Cesead II).

Materials and Methods: Disk specimens were prepared with the dentin portion of the composite material and abraded with a silicon-carbide rotary cutting instrument. The specimens were conditioned with varying combinations of silane primer (Add-on Primer, Clearfil Porcelain Bond Activator, Clearfil Porcelain Bond Activator + Clearfil Mega Bond-Primer, Porcelain Liner M, unprimed) and bonding agent (Clearfil Mega Bond-Bond, Modeling Liquid, Stain Diluent, no-bonding agent). After placement of the enamel portion of the same brand of composite material on each surface of the dentin material, the specimens were light-exposed, and either wet- or dry-stored at 37˚C for 24 hours. Shear bond strengths were determined and the results were analyzed by analysis of variances and *post-hoc* tests (p<0.05).

Results: Of the combinations tested, the Add-on Primer or Porcelain Liner M silane material combined with the Modeling liquid or Stain Diluent bonding agent showed the greatest bond strength. The groups with no bonding agent generally resulted in poor repair bond strength.

Conclusion: The use of specific combinations of silane primer and bonding agent is recommended to achieve reliable 24-hour bond strength between layers of the Cesead II composite.

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Clinical Significance: The results of the current study demonstrate that the ground surface of a hybrid composite material (Cesead II) can be repaired in dental laboratories by combined application of a silane primer and a light-activated bonding agent, if the composite material has not aged in the oral environment.

Key words: bonding, composite, repair, silane.

INTRODUCTION

Composite-to-composite bonding is required in varying clinical situations such as seating indirect composite restorations with a resin-based luting agent,[1-10] addition of a new composite material to an old

one,[11-13] and repair of fractured restorations.[14-15] Bonding techniques for composite materials are also used in dental laboratories. These procedures include bonding of artificial composite teeth to denture base resins, and layering of dentin and incisal portions of prosthodontic veneering composites.[16-17] The composition of composite materials varies considerably among manufacturers, curing modes and material indications, and bonding methods focus on organic matrices as well as inorganic fillers. Bonding between the two composite material layers is generally achieved in the presence of an oxygen-inhibited surface unpolymerized layer. It is difficult, however, to bond an additional material to an aged, ground or polished material surface. Both mechanical and chemical retention systems are being employed for composite-to-composite bonding. While a number of bond strength results have been reported for bonding composite materials in the clinic, only limited information is available on the bonding of laboratory cured prosthodontic composite materials, especially as related to laboratory repairing processes. In the current study, the repair bond strengths between dentin and incisal layers of an indirect composite were determined with the aim of evaluating bonding performance of the light-activated hybrid material treated with silane primers and bonding agents.

MATERIALS AND METHODS

A light-activated indirect composite material designed for veneering restorations and fixed partial dentures (Cesead II, Kuraray Medical, Tokyo, Japan) was selected as a substrate (shade DA3) and repair material (shade E1). This material is categorized as a hybrid composite, and it contains splintered inorganic fillers, inorganic micro fillers, and prepolymerized composite filler particles. Four silane primers and three bonding agents were used in the current study. The information on these materials is summarized in Table 1. The four primers contain a silane monomer but no initiator, whereas the three bonding agents contain a photo-initiator but no silane coupler. All materials except for the Porcelain Liner M two-liquid primer (Sun Medical. Moriyama, Japan) were supplied by Kuraray Medical (Tokyo, Japan).

The dentin portion of the Cesead II paste (DA3) was filled into a cylindrical plastic mold (10 mm in diameter by 2.5 mm height) and polymerized with a laboratory light-curing unit (Alpha-Light II, J. Morita Corp., Osaka, Japan) for 180 s. Of a total of 336 specimens, 320 specimens were cured between two pieces of glass plate, and the remaining 16 specimens (groups 1 and 2) were cured without shielding the top surfaces so as to produce an oxygen-inhibited surface unpolymerized layer. After light exposure, the 320 specimens were ground flat with 400 grit silicon-carbide abrasive paper, followed by abrasion with a silicon carbide rotary cutting instrument (HP13, Shofu Inc., Kyoto, Japan). The 16 specimens, each with an oxygen-inhibited layer, were left unground and were used as experimental controls. A piece of double coated tape with a circular hole 5 mm in diameter was positioned on the top surface of each of the 336 specimens.

The 320 ground and abraded specimens were divided into 40 groups (groups 3 to 42), which were then treated with the materials shown in Table 2. Each of the 40 groups consisted of eight identically treated specimens. In each group, the silane-primed surfaces and bonding-agent-treated surfaces were gently air-dried, while the surfaces to which the CMB Bond material was applied were light-exposed with the curing unit for 10 s. After surface preparation, a brass ring (6 mm inside diameter by 2 mm height and 1 mm

thick-wall) was placed so as to surround the 5 mm diameter area defined by the tape. The ring was filled with the enamel portion of the Cesead II material (E1) and light-cured for 180 s.

Thirty minutes after preparation, the specimens in the odd numbered groups were stored in a dry environment maintained by an incubator at 37˚C for 24 hours, and the specimens in the even numbered groups were immersed in 37˚C water for 24 hours. Shear bond strengths were then determined with a mechanical testing machine (AGS-10kNG, Shimazu Corp., Kyoto, Japan) at a crosshead speed of 0.5 mm/minute. Shear bond strength was defined as the force at failure divided by bonded surface area. For each condition, the average bond strength and standard deviation of eight replications were calculated. The bond strength results other than those in groups 1 and 2 were compared by analysis of variance (ANOVA), and a Duncan new multiple range test with the value of statistical significance set at the 0.05 level. Comparison between each group and the corresponding control group (group 1 or 2), and comparison between dry and wet conditions with an identical surface preparation were performed using mean comparison contrasts. The failure mode was categorized into five types as shown in Fig. 1.

**Table 1.** Materials assessed.

Materials/trade names	Abbreviation	Lot number	Functional monomer	Initiator
Indirect composite material				
Cesead II, Dentin, DA3		00542A, 00577A, 00588A, 00602A		Light
Cesead II, Enamel, E1		00055A, 00078A, 00086C, 00087A		Light
Silane primer				
Clearfil Porcelain Bond Activator	CPB Activator	00097A	Silane	None
Clearfil Porcelain Bond Activator	CPB Activator			
+ Clearfil Mega Bond, Primer	+ CMB Primer	0035A	Silane + MDP	None
Estenia Add-on Primer	Add-on Primer	00012E	Silane	None
Porcelain Liner M A liquid	Liner M	VE1	4-META	None
B liquid		VE1	Silane	None
Unfilled resin bonding agent				
Cesead Stain Diluent	Stain Diluent	00028D		Light
Clearfil Mega Bond, Bond	CMB Bond	00088A		Light
Estenia Modeling Liquid	Modeling Liquid	00017C		Light

4-META: 4-methacryloyloxyethyl trimellitate anhydride; MDP: Methacryloyloxydecyl dihydrogen phosphate.

RESULTS

Three-way ANOVA run on the shear testing results showed that the interaction among silane primer, bonding agent, and storage condition was not significant (p = 0.2699). The results were therefore analyzed by two-way ANOVAs. Two-way ANOVAs revealed a significant interaction between silane primer and

bonding agent in both dry- and wet-stored groups ($p<0.01$). The results were then analyzed separately by one-way ANOVAs and *post-hoc* Duncan new multiple range tests.
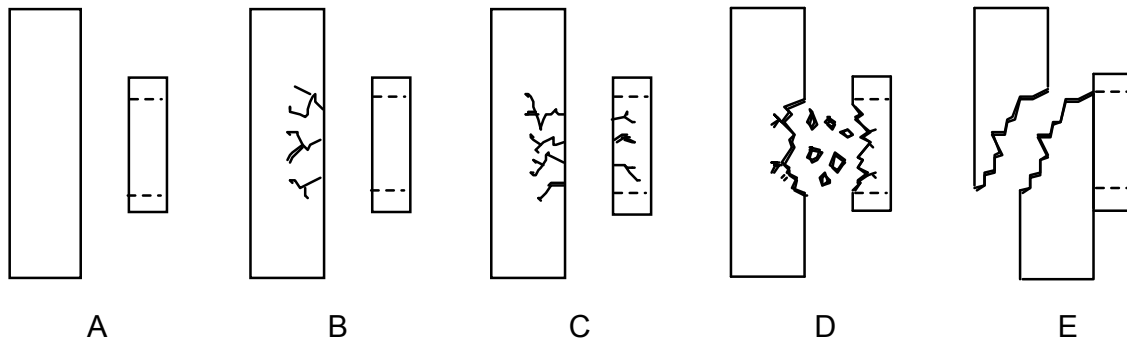
Fig. 1. Classification of failure modes.   A: composite-composite interface separation; B: composite-composite interface separation with crack propagation inside the substrate material; C: composite-composite interface separation with crack propagation inside both the substrate and added materials; D: cohesive failure of both sides of the composite material; and E: cohesive failure inside the substrate material.

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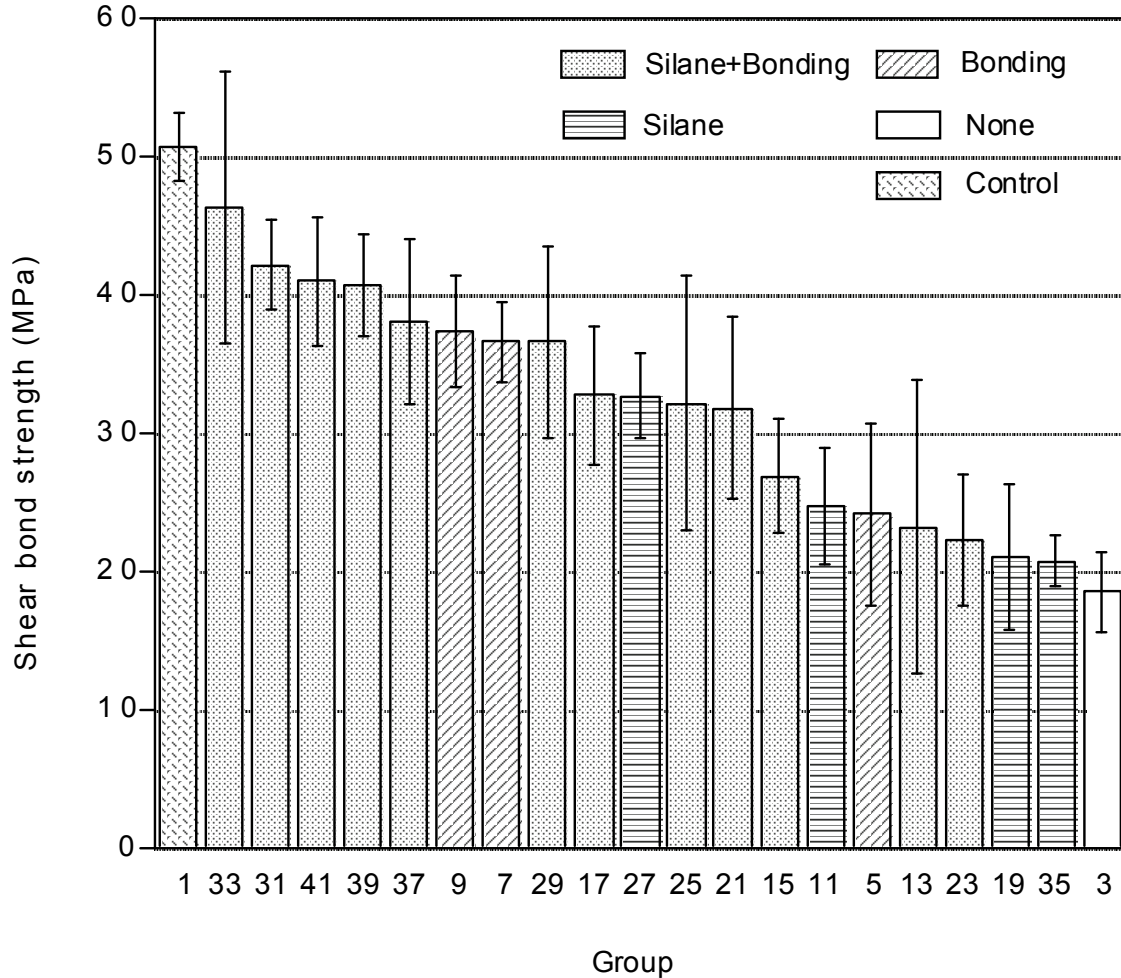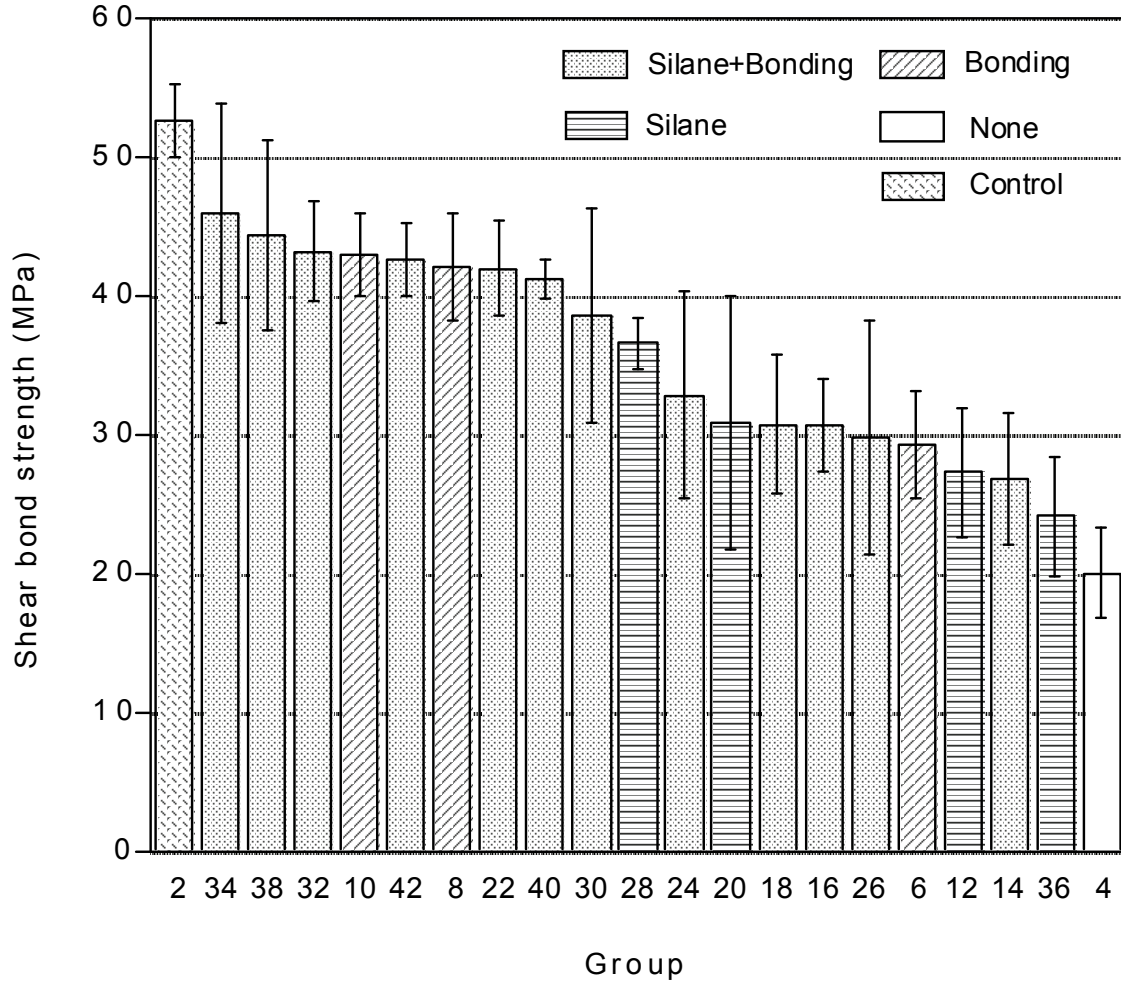

Fig. 2. Shear testing results for the dry-stored groups.

Table 2 and Figs. 2 and 3 summarize the shear testing results, statistical groupings, and failure modes. Except for the controls, average bond strength varied from 46.3 MPa (group 33, Add-on Primer and Stain Diluent) to 18.6 MPa (group 3, no silane and no bonding agent) in the dry-stored groups, whereas the average values ranged from 46.0 MPa (group 34, Add-on Primer and Stain Diluent) to 20.1 MPa (group 4, no silane and no bonding agent). The combination of the Add-on Primer silane material and the StainDiluent bonding agent was categorized into the greatest bond strength groups (categories f and m), and the untreated group was categorized into the lowest bond strength groups (categories a and g) for both dry and wet conditions (p<0.05). Bond strengths of the control groups were 50.7 MPa for the dry-stored group, and 52.7 MPa for the wet-stored group respectively. Of the surface-ground groups (groups 3 through 42), only group 33 showed bond strength statistically comparable to the control (group 1, p = 0.1074).

Among the 20 dry-stored groups with the exception of group 1, four combinations consisting of one of the two silane primers (Add-on Primer and Liner M) and one of the two bonding agents (Modeling Liquid and Stain Diluent) showed the greatest bond strength (category f). Another four groups (groups 3, 11, 19, and 35),

in which no bonding agent was used, were categorized into the lowest bond strength category (category a). Bond strength ranking somewhat changed for the wet-stored groups, although the combination of the Add-on primer and the Stain Diluent again showed the greatest bond strength (category m).

Fig. 3. Shear testing results for the wet-stored groups.

Bond strengths were statistically evaluated in the dry-stored and wet-stored groups by using mean comparison contrasts. The results are summarized in Table 3. Of a total of 21 combinations, bond strength was significantly greater in the six wet-stored groups than in the corresponding dry-stored groups. No significant difference in bond strength was found between dry- and wet-stored groups for the remaining 15 combinations.

Evaluation of debonded surfaces after shear testing indicate that high-bond strength groups tend to show cohesive failure inside the composite material rater than at the adhesive interface, and that low-bond strength groups tend to exhibit adhesive failure (Table 2). All the specimens in groups 3, 4, 11, and 12, in which no

bonding agent was used, resulted in type A adhesive failure.

Table 2. Shear testing results, statistical categories, and failure modes.

Group	Silane primer	Bonding agent	Mean (MPa)	SD (MPa)	Statistical category	A	B	C	D	E
1	(Control, dry-stored)		50.7	2.5		0	0	0	8	0
2	(Control, wet-stored)		52.7	2.6		0	0	0	8	0
Dry-stored groups										
3	None	None	18.6	2.9	a	8	0	0	0	0
5	None	CMB Bond	24.2	6.6	a, b	1	7	0	0	0
7	None	Modeling Liquid	36.6	2.9	d, e	0	1	7	0	0
9	None	Stain Diluent	37.4	4.0	d, e	0	0	4	2	2
11	CPB Activator	None	24.8	4.2	a, b	8	0	0	0	0
13	CPB Activator	CMB Bond	23.2	10.6	a, b	5	0	3	0	0
15	CPB Activator	Modeling Liquid	26.9	4.1	b, c	5	2	1	0	0
17	CPB Activator	Stain Diluent	32.8	5.0	c, d	6	2	0	0	0
19	CPB Activator + CMB Primer	None	21.1	5.3	a, b	5	1	2	0	0
21	CPB Activator + CMB Primer	CMB Bond	31.8	6.6	c, d	1	2	5	0	0
23	CPB Activator + CMB Primer	Modeling Liquid	22.3	4.8	a, b	4	1	3	0	0
25	CPB Activator + CMB Primer	Stain Diluent	32.2	9.2	c, d	1	3	4	0	0
27	Add-on Primer	None	32.7	3.1	c, d	0	0	7	1	0
29	Add-on Primer	CMB Bond	36.6	7.0	d, e	0	1	7	0	0
31	Add-on Primer	Modeling Liquid	42.2	3.2	e, f	0	0	6	2	0
33	Add-on Primer	Stain Diluent	46.3	9.8	f	1	3	4	0	0
35	Liner M	None	20.8	1.9	a, b	4	3	1	0	0
37	Liner M	CMB Bond	38.1	6.0	d, e	0	2	6	0	0
39	Liner M	Modeling Liquid	40.7	3.7	e, f	0	0	5	3	0
41	Liner M	Stain Diluent	41.0	4.7	e, f	0	0	4	3	1
Wet-stored groups										
4	None	None	20.1	3.2	g	8	0	0	0	0
6	None	CMB Bond	29.3	3.8	h, i	1	7	0	0	0
8	None	Modeling Liquid	42.1	3.9	k, l, m	0	0	8	0	0
10	None	Stain Diluent	43.0	2.9	l, m	0	0	5	1	2
12	CPB Activator	None	27.3	4.6	h, i	8	0	0	0	0
14	CPB Activator	CMB Bond	26.9	4.7	h, i	4	1	3	0	0
16	CPB Activator	Modeling Liquid	30.7	3.4	i	4	3	1	0	0
18	CPB Activator	Stain Diluent	30.8	5.0	i	6	2	0	0	0
20	CPB Activator + CMB Primer	None	30.9	9.1	i	4	1	3	0	0
22	CPB Activator + CMB Primer	CMB Bond	42.0	3.4	k, l, m	0	1	7	0	0
24	CPB Activator + CMB Primer	Modeling Liquid	32.9	7.4	i, j	2	1	5	0	0
26	CPB Activator + CMB Primer	Stain Diluent	29.8	8.4	h, i	1	5	2	0	0
28	Add-on Primer	None	36.6	1.9	j, k	0	0	8	0	0
30	Add-on Primer	CMB Bond	38.6	7.7	k, l	0	2	6	0	0
32	Add-on Primer	Modeling Liquid	43.2	3.6	l, m	0	0	7	1	0
34	Add-on Primer	Stain Diluent	46.0	7.9	m	0	1	7	0	0
36	Liner M	None	24.2	4.3	g, h	3	4	1	0	0
38	Liner M	CMB Bond	44.4	6.8	l, m	0	2	6	0	0
40	Liner M	Modeling Liquid	41.2	1.4	k, l, m	0	0	5	3	0
42	Liner M	Stain Diluent	42.6	2.6	l, m	0	0	2	4	2

Table 3. Means comparison contrast results between dry- and wet-stored groups.

Silane primer	Bonding agent	Dry	Wet	S/NS	P-value
Control	Control	1	2	NS	0.4564
None	None	3	4	NS	0.5816
None	CMB Bond	5	6	NS	0.0616
None	Modeling Liquid	7	8	S	0.0436
None	Stain Diluent	9	10	S	0.0398
CPB Activator	None	11	12	NS	0.3576
CPB Activator	CMB Bond	13	14	NS	0.1736
CPB Activator	Modeling Liquid	15	16	NS	0.1720
CPB Activator	Stain Diluent	17	18	NS	0.4656
CPB Activator + CMB Primer	None	19	20	S	0.0004
CPB Activator + CMB Primer	CMB Bond	21	22	S	0.0002
CPB Activator + CMB Primer	Modeling Liquid	23	24	S	0.0001
CPB Activator + CMB Primer	Stain Diluent	25	26	NS	0.3808
Add-on Primer	None	27	28	NS	0.1499
Add-on Primer	CMB Bond	29	30	NS	0.4506
Add-on Primer	Modeling Liquid	31	32	NS	0.7238
Add-on Primer	Stain Diluent	33	34	NS	0.9184
Liner M	None	35	36	NS	0.2088
Liner M	CMB Bond	37	38	S	0.0204
Liner M	Modeling Liquid	39	40	NS	0.8462
Liner M	Stain Diluent	41	42	NS	0.5576

S: Significant ($p < 0.05$); NS: Not significant ($p > 0.05$).

DISCUSSION

The CPB Activator material and the CMB Primer material was combined with the aim of activating the silane monomer contained in the former liquid with the acidic MDP monomer in the latter liquid. This concept has been established for porcelain bonding.[18] The current results, however, did not show any apparent silane-acid combination effect except for the one case in which it was used together with the CMB Bond material. Since the combination of the CMB Primer and the CMB Bond materials was originally introduced as a self-etching bonding system for tooth substrates, the bonding performance to composite materials in the CMB system should be evaluated further with the use of three-liquid systems consisting of the CMB Primer, CMB Bond, and one of the single-liquid silane agents.

The effect of the Liner M material on bond strength varied considerably with and without the use of

bonding agents. Both monomer liquids of the Liner M material contain volatile methyl methacrylate (MMA) monomer as the solvent. As a result, the composite surface primed with the Liner M material is coated with a thin layer of silane and 4-META monomers. Bonding of an additional composite to the primed surface is expected to be difficult in the absence of a flowable bonding agent, and indeed the results of groups 35 and 36 support this speculation. As shown in the statistical categories, bond strengths generated with the no-bonding agent groups were not particularly good, and the results suggest that the use of an unfilled resin bonding agent helps the Cesead II composite to achieve adequate 24-hour bond strength.

In the comparison of dry- and wet-stored groups, the bond strength of dry-stored groups was either comparable to or less than that of the corresponding wet-stored groups. Specimens were stored at an identical temperature in both dry- and wet-conditions. Thus the only difference between the two conditions was the presence/absence of water. The difference in coefficient of thermal conductivity between air and water, or the difference in concentration of oxygen around previously laboratory-cured specimens, may affect the post-curing properties of bonded specimens. Further evaluation including aging testing is necessary to determine the exact influence of water on composite-to-composite interfacial bonding.

Of the various combinations evaluated in the current study, the combined use of a silane primer and an unfilled resin bonding agent generally exhibited high bond strengths. The results suggest that both an appropriate functional monomer and reproduction of an oxygen-inhibited layer is indispensable to proper interfacial bonding between layers of the laboratory-processed hybrid composite.

ACKNOWLEDGMENT

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