©Operative Dentistry, 2013, **38-5**, 512-518

Dual-Cured Etch-and-Rinse Adhesive Systems Increase the Bond Durability of Direct Coronal Dentin Restorations

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Clinical Relevance

Bonding composite resin with dual-cured adhesive systems can promote increased bond durability to direct coronal dentin restorations.

SUMMARY

This study aimed to evaluate the bond durability of dentin restorations bonded with light- or dual-cured etch-and-rinse adhesive systems. A three-step adhesive system (Scotchbond Multipurpose Plus), an acetone-based two-step adhesive system (Prime & Bond 2.1), and an ethanol-based two-step adhesive system (Excite) were tested. Both the light- and the dual-cured versions were evaluated. High C-factor dentin cavities were prepared on 120 bovine incisors, which were then restored with resin composite (n=10). The samples were stored in water for 24 hours, and half of them were subjected to additional degradation with 10% NaOCl for five hours. The push-out bond

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DOI: 10.2341/12-246-L

strength test was performed in a universal testing machine until failure. Failure modes were evaluated by scanning electron microscopy. Data were analyzed by three-way analysis of variance and Tukey tests ($p<0.05$). The dual-cured adhesive system presented a higher immediate bond strength and durability than those that were light cured. The three-step adhesive system produced the highest values, whereas the acetone-based adhesive system produced the lowest result. Therefore, the use of dual-cured etch-and-rinse adhesive systems can induce increased bond durability to direct coronal dentin restorations.

INTRODUCTION

Successful adhesion to hard tissues is a fundamental requirement for dental materials. This is known to be directly dependent on the quality of the hybrid layer. Hence, any measure to increase the lifetime of adhesives might focus on improving the stability of the bonding interface between these biomaterials and tooth tissues.[1] The simplified two-step adhesives were developed from the traditional three-step etch-and-rinse adhesives. They contain the primer and the adhesive resin in one single solution. Despite being less technique sensitive, these simplified adhesives tend to perform more poorly when compared with their three-step counterparts, especially on dentin.[2]

Optimal monomer infiltration into the demineralized substrates and the achievement of a high degree of conversion (DC) are crucial factors in establishing long-lasting bonds.[3] Under clinical conditions, it seems likely that light may not be able to reach the entire coronal cavity with similar irradiance values during adhesive photoactivation. The curing-light tip is not placed directly toward the cavity wall in deeper areas. It has been shown that a long distance between the curing light tip and the material surface tends to generate loss of irradiance, which can negatively affect dentin bond strength.[4] These findings suggest that protocols that increase the DC of adhesive systems on deeper dentin surfaces of coronal cavities would contribute positively to the long-term bond durability of restorations. Although the DC of dual-cured etch-and-rinse adhesive systems has been investigated in the presence of light,[5] there is lack of evidence in the literature to confirm the increased bond durability of coronal dentin restorations bonded with dual-cured adhesive systems. This study aimed to evaluate the bond durability of direct coronal dentin restorations using light- or dual-cured three-step and two-step etch-and-rinse adhesive systems. The hypotheses tested were that 1) the use of dual-cured adhesive systems would increase the bond durability of dentin restorations and 2) the three-step adhesive system would perform better than the two-step adhesive system.

METHODS AND MATERIALS

The present study evaluated a three-step etch-and-rinse adhesive system (Scotchbond Multipurpose Plus, 3M ESPE, St Paul, MN, USA), an acetone-based two-step adhesive system (Prime & Bond 2.1, Dentsply/Caulk, Milford, DE, USA), and an ethanol-based two-step adhesive system (Excite, Ivoclar-Vivadent, Schaan, Liechtenstein). Both the light- and the dual-cured versions were tested. The Scotchbond Activator/Catalyst (3M ESPE) and the self-cure activator (Dentsply/Caulk) were used in the dual-cured versions of Scotchbond Multipurpose Plus and Prime & Bond 2.1, respectively. Excite DSC (Ivoclar-Vivadent) was used as the dual-cured version of Excite. Table 1 shows further details regarding the chemical components of the materials used in this study.

Bond Durability Analysis

Samples were prepared according to a previously described method for the push-out bond strength test.[6] A total of 120 bovine incisors, free from cracks and structural defects, were selected. The teeth were disinfected in a 0.1% aqueous solution of thymol at 40°C for no longer than one week. The roots were removed with a water-cooled diamond saw (South Bay Technology, San Clemente, CA, USA) coupled to a precision cutting machine (Isomet 1000, Buehler, Lake Forest, IL, USA). The buccal aspect of the crown was wet ground with 400- and 600-grit SiC abrasive papers in a polishing machine (Labopol-21, Struers, Copenhagen, Denmark) to obtain flat dentin surfaces.

Standardized conical cavities (2 mm top diameter $\times$ 1.5 mm bottom diameter $\times$ 2 mm height) were prepared with conical diamond burs (Komet Inc, Lemgo, Germany) at high speed under air-water cooling. A custom-made preparation device allowed the cavity dimensions to be standardized. The burs were replaced after every five preparations. To expose the bottom surface of the cavities, the lingual surfaces were ground in accordance to the procedure described for flattening the buccal aspects. In this manner, a cavity with a C-factor magnitude of 2.2 was obtained.

Table 1: *Materials Used in This Study*

Commercial Brand/Manufacturer	Composition	Batch No.
Scotchbond Etchant, 3M ESPE, St Paul, MN, USA	35% phosphoric acid	9NL
Adper Scotchbond Multipurpose Plus, 3M ESPE, St Paul, MN, USA	Primer: water, HEMA, copolymer of acrylic and itaconic acids	N124653
	Bonding agent: Bis-GMA, HEMA	N195685
	Activator: ethyl alcohol, sodium benzenesulfinate	N200988
	Catalyst: Bis-GMA, HEMA, benzoyl peroxide, triphenylphosphine	N253750
Excite, Ivoclar-Vivadent, Schaan, Liechtenstein	Dimethacrylates, alcohol, phosphonic acid acrylate, HEMA, SiO_2, initiators, stabilizers	N11163
Excite DSC, Ivoclar-Vivadent, Schaan, Liechtenstein	Dimethacrylates, alcohol, phosphonic acid acrylate, HEMA, SiO_2, initiators, stabilizers	N01061
Prime & Bond 2.1, Dentsply/Caulk, Milford, DE, USA	UDMA, PENTA, Bis-GMA, butylated hydroxytoluene, 4-ethyl dimethyl aminobenzoate, cetylamine hydrofluoride, initiator, acetone	L491546D
Self-Cure Activator, Dentsply/Caulk, Milford, DE, USA	Sodium p-toluenesulfinate, ethanol, acetone	L469025D
Brilliant NL (A2 shade), Coltène/Whaledent, Altstätten, Switzerland	Methacrylates, dental glass, amorphous silica, photoinitiator	0174947

Abbreviations: Bis-GMA, bisphenol A diglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; PENTA, dipentaerythritol pentacrylate monophosphate; UDMA, urethane dimethacrylate.

The prepared specimens were assigned to 12 groups (n=10) according to the factors under study (adhesive systems – 3 × curing modes – 2 × aging methods – 2). The adhesive systems were used according to the respective manufacturers' instructions, as follows:

- Adper Scotchbond Multipurpose Plus: Dentin was etched with 35% phosphoric acid (Scotchbond Etchant, 3M ESPE) for 15 seconds and washed thoroughly with water for 30 seconds. Excess water was blot dried with absorbent paper, leaving the dentin surface visibly moist (wet bonding). One coat of the primer was applied to dentin and air dried for 10 seconds at 20-cm with an air stream –2.6 bar (this distance was standardized using a millimeter ruler). One coat of the catalyst was applied to dentin and air dried for 10 seconds at 20 cm. One coat of the bonding agent was applied and light cured for 10 seconds with a Coltolux LED (Coltène/Whaledent, Altstätten, Switzerland) at 1264 mW/cm^2 (a radiometer was used to measure the light intensity; model 100, Demetron/Kerr, Danbury, CT, USA). Both the activator and the catalyst were applied only for the dual-cured version.
- Excite: Dentin was etched with 35% phosphoric acid for 15 seconds and washed thoroughly with water for 30 seconds. Excess water was blot dried with absorbent paper, leaving the dentin surface visibly moist (wet bonding). Two coats of the adhesive system were applied and agitated against the prepared surfaces for 10 seconds. The adhesive system was dispersed to a thin layer with a weak stream of air for 10 seconds at 20 cm and light cured for 10 seconds with a Coltolux LED. Before applying Excite DSC, the single-dose unit was placed on a firm surface, and the applicator

pressed down. The brush and the initiators came in contact with the adhesive solution.

- Prime & Bond 2.1: Dentin was etched with 35% phosphoric acid for 15 seconds and washed thoroughly with water for 30 seconds. Excess water was blot dried with absorbent paper, leaving the dentin surface visibly moist (wet bonding). Two coats of the adhesive system were applied. The solvent was allowed to evaporate for 10 seconds at 20 cm and light cured for 10 seconds with a Coltolux LED. For the dual-cured version, Prime & Bond 2.1 was mixed to the self-cure activator in a 1:1 ratio.

After applying the adhesive systems, the specimens were placed onto a glass slab. The restorative procedures were performed with a resin composite (Brilliant NL, Coltène/Whaledent) that was bulk inserted into the cavity from its wider side. Photoactivation was performed with the Coltolux LED for 20 seconds. The light tip was positioned directly over the restoration, which had been previously covered with a Mylar strip. The top and bottom surfaces of all restorations were finished with #1200 abrasive papers (Buehler, Lake Bluff, IL, USA) coupled with a polishing machine (Labopol-21, Struers, Copenhagen, Denmark). The samples were stored in distilled water at 37°C for 24 hours, and half of them were subjected to further chemical degradation with 10% sodium hypochlorite (NaOCl) for five hours.[7]

The push-out test was performed in a universal testing machine (Instron model 4411, Canton, MA, USA). An acrylic device with a central orifice was adapted to the base of the machine. Each specimen was placed in the device with the top of its cavity against the acrylic surface. The bottom surface of the restoration was loaded with a 1-mm-diameter cylindrical plunger at a cross-head speed of 0.5 mm/min until failure of the tooth-composite bond at the lateral walls of the cavity. The plunger tip was positioned in a way to touch only the filling material, thereby creating no stress at the surrounding walls. The load required for failure was recorded by the testing machine and later converted into MPa values. The data were analyzed by three-way analysis of variance and the post hoc Tukey test. Statistical significance was established at α=0.05.

The fractured specimens were cut in half with a water-cooled low-speed diamond saw (Isomet 1000, Buehler) in order to obtain two specimens. Both specimens were fixed to aluminum stubs with the fractured interfaces upward. Specimens were sputter coated with gold (SDC 050 Sputter Coater, Baltec, Balzers, Liechtenstein) and evaluated by scanning electronic microscopy (JSM 5600LV, JEOL, Tokyo, Japan) to determine the failure mode. The failure modes were defined as adhesive failure, cohesive failure in composite, cohesive failure in dentin, and mixed failure (adhesive and cohesive).

RESULTS

Bond Durability

Intergroup comparisons are shown in Table 2. Regardless of curing mode, the tested adhesive systems yielded a lower bond strength following the NaOCl aging method. Despite this, the dual-cured materials were found to produce higher immediate bond strength and durability than light-cured materials. The three-step adhesive system (Scotchbond Multipurpose Plus) produced the highest values, whereas the acetone-based simplified system (Prime & Bond 2.1) led to the lowest results.

Failure Modes

The frequency of each failure mode is shown in Table 3. Samples bonded with dual-cured adhesive systems showed a lower number of adhesive failure episodes. Samples subjected to the additional NaOCl aging method showed a higher number of adhesive failures.

DISCUSSION

Because the dual-cured version of all adhesive systems was found to promote an increased bond durability to direct coronal dentin restorations, the first hypothesis tested in this study could be validated. In fact, dual-cured adhesive systems have been traditionally recommended when either bonding fiber posts to the dentin root surface or when performing indirect coronal restorations. The rationale for such a clinical protocol is based on the fact that light tends to become attenuated at the deeper aspect of the root as well as underneath indirect restorations. This also happens because of the incompatibility of light-cured simplified adhesive systems with both self- and dual-cured resin-based luting cements.[5,8-10] However, our results confirmed the benefits from using dual-cured etch-and-rinse adhesive systems for direct coronal dentin composite restorations.

The dual-cured etch-and-rinse adhesive systems, which were tested in the present study, contain different self-curing promoters. Whereas the three-step etch-and-rinse adhesive system (Adper Scotchbond Multipurpose Plus) requires the use of an

Table 2: *Bond Strength Means (Standard Deviations) for Each Adhesive System According to Curing Mode and Aging Method*

Adhesive System	Aging Method	Curing Mode	
		Light	Dual[a]
Adper Scotchbond Multipurpose Plus	Water	45.9 (8.8) Ba	64.3 (7.7) Aa
Excite		30.2 (4.9) Bb	37.2 (2.7) Ab
Prime & Bond 2.1		19.4 (4.8) Bc	24.9 (2.6) Ac
Adper Scotchbond Multipurpose Plus	Water and NaOCl	39.1 (8.5) Ba[b]	57.0 (6.3) Aa[b]
Excite		25.3 (3.8) Bb[b]	31.5 (2.8) Ab[b]
Prime & Bond 2.1		15.5 (1.9) Bc[b]	20.9 (1.8) Ac[b]

[a] *Excite DSC was used as the dual-cured version of Excite. Different uppercase letters in rows and lowercase letters in columns indicate statistically significant differences for each aging method ($p<0.05$).*
[b] *Statistically significant differences between aging methods for the same adhesive system and curing mode.*

activator solution before priming and a catalyst liquid after priming, both the simplified systems (Prime & Bond 2.1 and Excite) contain only one activator component, which is mixed with the adhesive system before usage. All of the activator components are derived from sulfinate salts, which react with acidic resin monomers to produce either phenyl or benzenesulfonyl free radicals that initiate the self-curing polymerization reaction of the adhesive system.[11] This is thought to increase the degree of conversion of the adhesive system, thereby contributing to long-lasting bonds. Moreover, the catalyst liquid that is used after the application of the primer (Adper Scotchbond Multipurpose Plus)

Table 3: *Frequency of Failure Mode*

Adhesive System	Aging Method	Curing Mode/Failure Mode									
		Light					Dual[a]				
		A	CA	CC	CD	M	A	CA	CC	CD	M
Adper Scotchbond	Water	—	—	6	—	4	—	—	5	5	—
Multipurpose Plus	Water and NaOCl	4	—	2	2	2	2	—	4	2	2
Excite	Water	—	5	1	—	4	—	5	—	1	4
	Water and NaOCl	5	—	1	2	2	3	—	1	3	3
Prime & Bond 2.1	Water	6	—	—	2	2	1	—	2	2	5
	Water and NaOCl	7	—	—	—	3	5	—	—	—	5

Abbreviations: A. adhesive failure; CA, cohesive failure in adhesive; CC, cohesive failure in composite; CD, cohesive failure in dentin; M, mixed (adhesive and cohesive).
[a] *Excite DSC was used as the dual-cured version of Excite.*

contains benzoyl peroxide, which has been reported to initiate a self-curing process of the monomers.[12] The self-cure polymerization is triggered by a reaction between benzoyl peroxide and a tertiary amine, a co-initiator that is often present in resin-based light-cured dental materials. Regardless of exposure to light, the co-initiator releases free radicals to promote monomer conversion.[13] It is likely that an additional polymerization occurs in the bonding agent of Adper Scotchbond Multipurpose Plus when the benzoyl peroxide–based catalyst is applied and the material is later exposed to light. Although the manufacturer claims that it is unnecessary to use the bonding agent after applying the catalyst, we did not follow its instructions in the face of previous positive results with regard to degree of conversion when mixing the bonding agent with the catalyst liquid.[5] However, further analysis should be encouraged to investigate whether an increased degree of conversion could be obtained when other adhesive systems, herein tested, are mixed with activator solutions and later exposed to light.

Even though the light source tip was placed very close to the superficial tridimensional cavity margins, it must be assumed that the light gets attenuated in deeper areas. This occurs in most clinical situations and compromises the degree of conversion of the adhesive system. Because of such incomplete monomer conversion, the acidic monomers of simplified systems might become capable of inactivating the tertiary amine co-initiator, which may be present in the composite resin, thereby affecting negatively both the composite conversion and the bond durability.

Our dentin bond durability results are in agreement with previous systematic data as the three-step etch-and-rinse adhesive systems remain the gold standard in terms of bond durability.[2,14] Consequently, the second hypothesis tested was also validated. In fact, by blending primer and bonding components in a single solution, simplified etch-and-rinse adhesives show a reduced ability to infiltrate the demineralized dentin substrate, thereby producing suboptimal hybridization.[14] In addition, the hydrophilic nature of such adhesives render them more prone to water sorption and consequently more susceptible to the effects of hydrolytic degradation. The solvent present in such adhesives is also more difficult to evaporate, frequently remaining entrapped within the adhesive layer after polymerization.[15]

The influence of the adhesive solvent component on dentin bond strength and durability according to dentin moisture conditions has been observed. For instance, an acetone-based adhesive system was found to perform better than a water/ethanol material only on very wet dentin surfaces.[16] In the present study, because dentin moisture conditions were standardized according to the manufacturers' instructions, it is likely that the lower bond strength values produced by the acetone-based adhesive system, in comparison with the ethanol-based adhesive system, would be attributed not only to the differences between the solvent components. Probably, for both materials, differences between chemical components also justify distinctiveness in bonding performance.

In this study, an accelerated aging test was used to stimulate dentin bond degradation by exposing samples to a 10% NaOCl solution for five hours.[7] Although this method has been frequently used to accelerate bond degradation in beams, it decreased bond strength in the tridimensional cavities evaluated. In fact, 10% NaOCl can induce oxidation, leading to the fragmentation of resin-unprotected collagen fibrils and affecting bond integrity.[17] Such a solution can also promote resin dissolution at the hybrid layer of adhesive systems,[18] thereby decreasing bond integrity. The high number of adhesive failures in samples subjected to the NaOCl aging method can be justified by the lower integrity of the hybrid layer caused by NaOCl.

Different methods, such as the shear and tensile bond strengths, have been used to measure dentin bond integrity. One disadvantage of these methodologies is that they are generally performed on flat surfaces. In such situations, the C-factor is very low, and the shrinkage stress is not directed at the bonding interface.[6] For these reasons, the push-out bond strength test was performed in the present study. The advantage of using the push-out test is that the bond strength can be evaluated in a high C-factor cavity (2.2), such as Class I and inlay cavities, with the high-stress generation directed at the bonding area. The method provides a better estimation of the bond strength than does the conventional shear test, because fracture occurs in parallel (not transverse) with the dentin-bonding interface, thereby simulating the clinical condition.[19] Thus, since dual-adhesive systems produced a higher bond durability than light-cured adhesive systems in tridimensional dentin cavities, they might then be able to improve the clinical performance of composite coronal restorations. On the other hand, clinicians should be prepared to increase working time due to a high number of steps. Further clinical trials are still necessary to confirm this assumption.

CONCLUSION

The use of dual-cured etch-and-rinse adhesive systems can provide increased bond durability to direct coronal dentin restorations.

Acknowledgements

The authors are grateful to Ivoclar-Vivadent, and Dentsply/Caulk for providing them with their adhesive systems. The authors also acknowledge the invaluable contribution from Coltène/Whaledent for donating the composite resin and the curing unit used in this study.

(Accepted 8 November 2012)

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