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The Effect of Simplified Adhesives on the Bond Strength to Dentin of Dual-cure Resin Cements

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Clinical Relevance

Unique initiator systems in resin cements may not sufficiently overcome incompatibility with simplified adhesives.

SUMMARY

The purpose of this study was to compare the shear bond strengths to dentin of two dual-cure resin cements, one with a unique initiator, NX3 (Kerr Corp), and the other with a traditional redox-initiator system, Calibra (Dentsply), when used in combination with simplified or nonsimplified adhesive agents. The two dual-cure resin cements, in either self- or dual-cure activation modes, were bonded to human dentin with four dental adhesives to create 16 subgroups of 10 specimens each. After 24 hours of storage in distilled water at 37°C, the specimens were tested in shear in a universal testing machine. With both NX3 and Calibra, bond strengths significantly increased when the specimens were dual cured. In addition, with either cement in either mode, the nonsimplified adhesives performed significantly better than did the simplified adhesive bonding agents. When used specifically with simplified adhesives in either cure mode, NX3 did not produce significantly higher bond strengths than did Calibra. In general, lower dentin bond strengths were found with simplified adhesives or self-cure activation with either resin cement.

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INTRODUCTION

Ever since Buonocore[1] described acid etching as a means to increase resin-enamel bond strengths over 50 years ago, we have been in constant pursuit of the ideal dental adhesive. The initial etch-and-rinse adhesives required three steps that included an acidic conditioner, primer, and adhesive monomer. Examples include Optibond FL (Kerr Corp, Orange, CA, USA) and Adper Scotchbond MultiPurpose (3M ESPE, St Paul, MN, USA).

Over the years, the trend has been to develop systems that are "simplified" or, in other words, that involve fewer steps with less procedure time.[2] A simplified adhesive is one in which the adhesive step

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is incorporated into the primer. Manufacturers began to combine the primer and resin monomer components to create a two-step or simplified etch-and-rinse agent. Examples include Optibond Solo Plus (Kerr Corp), One-Step (Bisco, Schaumburg, IL, USA), and Prime and Bond NT (Dentsply, Milford, DE, USA).

Self-etch adhesives have been an even more recent introduction in which the use of an acidified primer has eliminated the use of the conditioner. An example of a popular nonsimplified self-etch adhesive is Clearfil SE (Kuraray, Japan). Today, simplified versions of the self-etch adhesives on the market are one-step systems in which the acidified primer and adhesive monomer are mixed together and placed in a single step. Examples include Optibond All-in-One (Kerr Corp), All-Bond SE (Bisco), Xeno 4 (Dentsply), and Adper Prompt L Pop (3M ESPE).

Using restorative systems with simplified adhesives does not necessarily result in reduced bond strength to dentin.[3,4] However, clinicians began to report bonding failures when self-cured "build-up" composites were bonded with simplified adhesive systems.[5] They were alerted to potential incompatibilities between self-cured resins and certain adhesive systems.[6] Simplified systems used with light-cure resins were found to produce bond strengths that were considerably higher than those produced with self-cure resins.[7,8] The process of simplification involves incorporation of acidified resin monomers into the primer-adhesive combination, resulting in a more hydrophilic mix. The concentration of acidic resin monomers is even higher in simplified self-etch adhesives, which serve to etch through the smear layer and enable bonding to the underlying dentin.[7-9] The hydrophilic property improves the wetting of the demineralized collagen matrix.[10] However, this layer acts like a semipermeable membrane, enabling the transudation of water from the underlying dentin across an osmotic gradient toward the oxygen-inhibited adhesive agent–resin cement interface.[9] These are described as water trees and interfacial blisters under transmission electron microscopy and contribute to diminished bond strengths of self-cure composites when compared to their nonsimplified counterparts.[7,8]

More significantly, simplified adhesives can lead to the deactivation of the amine initiators in self- and dual-cure composites. Conventional self-cure composites utilize a binary redox initiator system that consists of benzoyl peroxide with aromatic tertiary amines.[11] The amines react with the acidified monomers present in the superficial oxygen-inhibited layer and are unavailable to initiate the self-cure. This ultimately results in incomplete polymerization and compromised bond strengths along the composite–bonding agent interface.[12-14]

As mentioned earlier, the oxygen-inhibited layer in simplified adhesives acts as a hypertonic medium that triggers osmotic fluid transport through the permeable adhesive layer. It is also a source of acidic resin monomers that deactivate tertiary amines.[15] The combination results in an incompatibility between self- and dual-cure composite resin materials when used with simplified adhesives, as evidenced by lower bond strengths and the presence of water blisters at the interface.[15] The adverse chemical interaction between catalytic components of self-cured composites and simplified adhesives is the major cause of bond strength reduction, whereas permeability of the adhesives to water causes only a minor reduction in bond strength.[7,8] When the permeability component was completely removed, as with the use of neat water-free resins, even low concentrations of acidic monomers were shown to deactivate tertiary amines in self-cured resins.[15]

Overall, the consequences are more acute in simplified self-etch adhesives than in simplified etch-and-rinse adhesives.[7,8] Furthermore, within the simplified etch-and-rinse adhesives, incompatibility was accentuated in some adhesives more than in others.[16] The decrease in tensile bond strengths of self-cure resins to dentin was shown to be inversely proportional to the acidity of the etch-and-rinse system.[17] In both of the above studies, the most acidic simplified etch-and-rinse agent, Prime and Bond NT by Dentsply (pH, 2.68), had the weakest bond strengths when compared to the least acidic, One-Step by Bisco (pH, 4.60).[16,17]

It should be noted that when a dual-cure cement is sufficiently light-cured, the incompatibility does not occur.[7,8] The bond strength to dentin is directly related to the amount of light energy to which it is exposed.[18] Manufacturers of dual- or self-cure cement systems accept this incompatibility and indicate their use exclusively with nonsimplified etch-and-rinse or self-etch adhesives.[19]

If a dual-cure cement is to be used with a simplified adhesive, adequate light-curing of the cement is emphasized. Some manufacturers recommend use of an additional dual-cure activator. However, it has been shown that the use of activators does not completely eliminate this incompatibility.[7,8] A recently released dual-cure resin cement (NX3, Kerr Corp) employs a unique redox

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initiator system that the manufacturer claims is acid resistant and that can initiate polymerization in the dark and in the presence of an acidic environment.[20,21] Consequently, the manufacturer proposes that this agent can be used with any adhesive system on the market without compromising bond strength.[22,23] To date, there are no published articles in the literature to verify this claim, and the few recent unpublished abstracts funded by other manufacturing companies that indirectly looked at this agent in their study models have not shown improved dentin bond strengths when employed with simplified agents in self-cure modes.[24-28]

The purpose of this study was to evaluate whether or not the proprietary initiators outlined above are able to circumvent the incompatibility issue with simplified dental adhesives in self-cure mode. If they are, do they exhibit dentin bond strengths similar to those achieved in the dual-cure mode?

It was the aim of this study to substantiate the manufacturer's claim so that clinicians can take advantage of the product's properties with the comfort of knowing it is supported by evidence-based dentistry. This study evaluated the shear bond strengths to dentin of two dual-cure resin cements, NX3 (new initiator system) and Calibra (standard system used as a control), either in self- or dual-cure modes when used in combination with simplified or nonsimplified adhesive agents. The study tested two specific null hypotheses, as follows:

1) There is no difference in the shear bond strength of NX3 or Calibra to dentin based on the choice of adhesive bonding agents, either simplified (Prompt L-Pop, Prime and Bond NT) or non-simplified (Optibond FL, Clearfil SE); and
2) There is no difference in the shear bond strength to dentin of dual-cure cements, NX3 or Calibra, based on the curing mode, either dual- or self-cure.

METHODS AND MATERIALS

The protocol was approved by the Institutional Review Board at Wilford Hall Ambulatory Surgical Center, Joint Base San Antonio - Lackland, Texas. The resin cements chosen for this study were NX3 (Kerr Corp) and Calibra (Dentsply).

Four dental adhesives were utilized, two non-simplified (Optibond FL [Kerr Corp] and Clearfil SE [Kuraray]) and two simplified (Prime and Bond NT [Dentsply] and Prompt L Pop [3M ESPE]).

Table 1: *Study Groupings*

Group No.	Adhesive	Subgroup No.	Resin Cement and Curing Mode
1	Prime and Bond NT	1	NX3 + self-cure mode
		2	NX3 + dual-cure mode
		3	Calibra + self-cure mode
		4	Calibra + dual-cure mode
2	Adper Prompt L-Pop	5	NX3 + self-cure mode
		6	NX3 + dual-cure mode
		7	Calibra + self-cure mode
		8	Calibra + dual-cure mode
3	Optibond FL	9	NX3 + self-cure mode
		10	NX3 + dual-cure mode
		11	Calibra + self-cure mode
		12	Calibra + dual-cure mode
4	Clearfil SE	13	NX3 + self-cure mode
		14	NX3 + dual-cure mode
		15	Calibra + self-cure mode
		16	Calibra + dual-cure mode

The following adhesive agent/resin cement combinations were used:

- Prime and Bond NT-NX3/Calibra;
- Prompt L-Pop-NX3/Calibra;
- Optibond FL- NX3/Calibra; and
- Clearfil SE-NX3/Calibra.

The following activation modes, in which each of the above groups were further subdivided based on cure mode, were used:

- Dual-cure and
- Self-cure.

A total of 16 subgroups were created (see Table 1). Ten samples were prepared per subgroup, resulting in 160 total samples. All 160 samples were prepared by a single provider to minimize interoperator differences and to ensure uniformity of fabrication.

One hundred sixty extracted human third molars stored in 0.5% chloramine-T at 4°C were used within six months following extraction. The teeth were mounted in dental stone in polyvinyl chloride pipes with the crown exposed and accessible. A diamond saw (Isomet, Buehler, Lake Forest, IL, USA) was used to remove 2 mm or more of coronal tooth structure to ensure dentin exposure and the proper orientation of the surface relative to the direction of the applied shear force. Each sample was then examined under a stereomicroscope (SMZ-1B, Nikon, Melville, NY, USA) at 10× magnification to ensure complete exposure of the dentin surface with

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Table 2: *Application Methods*

Adhesive (Manufacturer)	Type	Manufacturer's Application Instructions
Prime and Bond NT (Dentsply)	Two-step, etch-and-rinse (simplified)	Etchant: apply and leave undisturbed (15 s)
		Water rinse
		Gently air-dry (5 s)
		Bond: apply and leave undisturbed (20 s); gently air-dry
		Light-cure (10 s)
Adper Prompt L-Pop (3M ESPE)	One-step, self-etch (simplified)	Mix the liquids in the red and yellow blister, brush the mixture onto tooth surface (15 s)
		Gently air-dry (5 s)
		Light-cure (10 s)
Optibond FL (Kerr Corp)	Three-step etch-and-rinse (nonsimplified)	Etchant: apply and leave undisturbed (15 s)
		Water rinse
		Gently air-dry (5 s)
		Primer: apply with light scrubbing motion (15 s)
		Gently air-dry (5 s)
		Bond: apply to a thin layer
		Light-cure (30 s)
Clearfil SE (Kuraray)	Two-step self-etch (nonsimplified)	Primer: apply and leave undisturbed (20 s)
		Gently air-dry (5 s)
		Bond: apply to a thin layer
		Light-cure (10 s)
NX3 (Kerr Corp)	Resin cement: dual-cure mode	Dual-cure paste
		2-mm increments
		Light-cure (20 s)
	Resin cement: self-cure mode	Dual-cure paste
		Bulk fill
Calibra (Dentsply)	Resin cement: dual-cure mode	Mix equal lengths of base and catalyst for 20 s
		2-mm increments
		Light-cure (20 s)
	Resin cement: self-cure mode	Mix equal lengths of base and catalyst for 20 s
		Bulk fill

no residual enamel. A uniform smear layer was created on the flat dentin surfaces using 10 passes on 600-grit carbide paper.

The 160 prepared teeth were randomly distributed to create four groups (40 specimens in each group) based on the four adhesive agents used. The adhesive agents were applied to the dentin surface according to the manufacturer's instructions (see Table 2).[19,29-33] The adhesive was cured as recommended by the manufacturer using the Bluephase 16i (Ivoclar, Amherst, NY, USA) light-curing unit. The irradiance of the curing light was monitored periodically with a radiometer (Bluephase Radiometer, Ivoclar) to verify that irradiance levels remained above 1200 mW/cm^2. Each of the four groups was further divided into four equal subgroups of 10 samples each. Each subgroup tested one of the two resin cements being evaluated in either self- or dual-cure activation mode. The prepared samples were placed in an Ultradent Jig and secured beneath the white, nonstick Delrin insert (Ultradent, South Jordan, UT, USA). The resin cement was then mixed and applied into the mold according to the manufacturer's instructions (see Table 2)[19,21] to a height of 3 mm. The bonding area was limited to the 2.4-mm-diameter circle determined by the Delrin insert. The self-cure subgroups were allowed to self-cure undisturbed for a period of 15 minutes in a light-proof container. The dual-cure subgroups were bulk–light-cured for 20 seconds to simulate the light penetration achieved in a clinical setting. Following the application of the resin cement with the designated curing method, all samples were stored for 24 hours in distilled water at 37°C. After 24 hours, the shear bond strengths of all samples were tested using a universal testing machine (Model 5943, Instron,

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Table 3: *Shear Bond Strength Data and Statistical Analysis Based on Adhesive Type*[a]

Cement	Shear Bond Strength (±SD), MPa											
	Adhesive											
	Nonsimplified						Simplified					
	Clearfil SE			Optibond FL			Prime and Bond NT			Prompt L-Pop		
	DC	SC	Two-way ANOVA	DC	SC	Two-way ANOVA	DC	SC	Two-way ANOVA	DC	SC	Two-way ANOVA
NX3	13.2 (4.8)	9.4 (3.2)	A	6.5 (1.6)	6.4 (1.7)	A	2.3 (1.8)	2.8 (2.0)	A	2.5 (2.4)	0.3 (0.3)	A
Calibra	9.5 (3.7)	6.6 (4.2)	A	4.0 (1.7)	2.7 (2.4)	B	3.8 (2.8)	1.1 (1.2)	A	1.1 (1.0)	0.08 (0.04)	A

Abbreviations: ANOVA, analysis of variance; DC, dual cure; SC, self cure; SD, standard deviation.
[a] *Groups with the same capital letter per column within a bond group are not significantly different ($p>0.008$).*

Norwood, MA, USA) at a crosshead speed of 1 mm/min using the notched blade at a 90° angle. Shear bond strength values in megapascals (MPa) were calculated from the peak load of failure (Newtons) divided by the sample surface area. The mean and standard deviation were determined for each group. The resultant data for the various groups were then analyzed to verify the two null hypotheses.

Following testing, each specimen was examined using a 10× stereomicroscope to determine failure mode as 1) adhesive fracture at the cement/adhesive/dentin interface, 2) cohesive fracture in cement or dentin, or 3) mixed failure (combined adhesive and cohesive).

A mean and standard deviation were determined for each group. The study involved three independent variables—adhesive (four levels), cement (two levels), and cure method (two levels). Consequently, a three-way analysis of variance (ANOVA) was performed to identify differences at the three levels of variability. Alpha was set at 0.05. Though significant differences were detected at all three levels with the three-way ANOVA, global conclusions could not be made from the results as a result of the interactions between the individual parameters. Two-way ANOVAs were then performed, keeping one of the three variables constant each time. Tukey post hoc tests were used to determine differences between groups. The alpha value was adjusted to 0.008 with a Bonferroni correction because multiple comparisons were completed simultaneously.

RESULTS

The statistical analysis was reviewed and approved by the clinical research administrator, Clinical Research Division, JBSA-Lackland (Texas).

Four two-way ANOVA tests were performed for the four levels tested within the adhesives group (Table 3). Within all the samples bonded with simplified adhesives either Prime and Bond NT or Prompt L Pop, regardless of cure mode, no significant difference was noted between NX3 and Calibra. Within all the samples bonded with the nonsimplified Optibond FL, regardless of cure mode, bond strengths with NX3 were significantly higher than those obtained with Calibra.

Two ANOVA (two-way) tests were performed for the two levels tested within the cements group (Table 4). Dual-cure polymerization resulted in an overall higher bond strength than self-cure polymerization using NX3. Within all the groups cemented with NX3, regardless of cure mode, samples bonded with nonsimplified adhesives had significantly high-

Table 4: *Shear Bond Strength Data and Statistical Analysis Based on Cement Type*[a]

Cure Mode	Shear Bond Strength (SD), MPa									
	Cement									
	NX3					Calibra				
	CSE a	OFL b	PB c	PLP c	Two-way ANOVA	CSE a	OFL b	PB bc	PLP c	Two-way ANOVA
Dual cure	13.20 (4.78)	6.49 (1.61)	2.30 (1.76)	2.45 (2.34)	A	9.51 (3.74)	3.97 (1.68)	3.80 (2.85)	1.07 (1.05)	A
Self cure	9.43 (3.15)	6.36 (1.69)	2.81 (2.04)	0.32 (0.34)	B	6.58 (4.22)	2.71 (2.37)	1.13 (1.19)	0.08 (0.04)	B

Abbreviations: ANOVA, analysis of variance; CSE, Clearfil SE; OFL, Optibond FL; PB, Prime&Bond NT; PLP, Prompt L-Pop; SD, standard deviation.
[a] *Groups with the same capital letter per column or lowercase letter per row within a cement group are not significantly different ($p>0.008$).*

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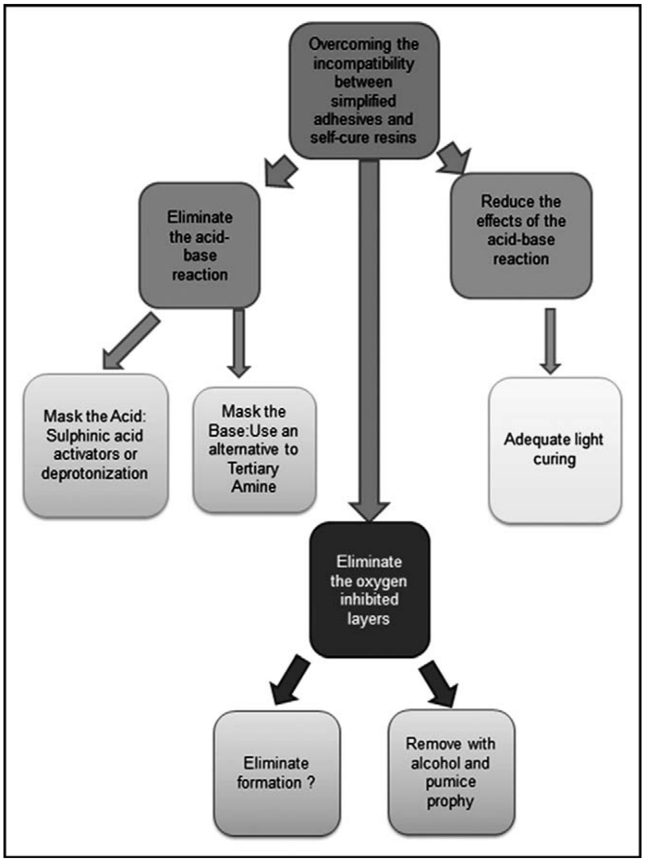

Figure 1. *Treatment strategies flow chart.*

er bond strengths than did those bonded with the simplified adhesives. Among the adhesives, the Clearfil SE samples exhibited the highest bond strengths. Within all the groups cemented with Calibra, regardless of adhesive used, the groups that were dual-cured showed higher bond strengths than did those that were self-cured. Within all the groups cemented with Calibra, regardless of cure mode, samples cemented with the nonsimplified adhesives showed higher bond strengths than did those cemented with the simplified adhesives. It must be noted as an exception that the values shown by Optibond FL were not significantly higher than those bonded with Prime and Bond NT. Samples bonded with Clearfil SE exhibited the highest bond strengths.

Following debonding, all specimens were viewed under a 10× stereomicroscope to determine failure mode. The majority of the failures were adhesive. No cohesive failures were noted. Mixed failures were noted most in Clearfil SE Bond subgroups. More mixed failures were associated with dual-curing.

DISCUSSION

This study showed that dentin bond strengths obtained with NX3 and simplified dental adhesives, regardless of cure mode, are not significantly higher than those obtained with Calibra. This suggests that the proprietary redox initiator in NX3 is not able to sufficiently overcome the incompatibility issues encountered when a simplified dental adhesive is combined with a self-activated dual-cure resin cement. Therefore, like every other dual-cure resin cement, NX3 should be used with a nonsimplified adhesive for optimal results.

The goal of patient-centered care is to provide excellent treatment in an efficient manner at minimum cost. With the development of stronger, yet esthetic, resin materials and adhesives, we are getting closer to achieving this goal. It is no surprise that resin restorative materials and simplified adhesives are major players in the world of restorative dentistry. That being said, it is noteworthy that self- or dual-cure resin cements still maintain their place in any dentist's inventory. They are the norm in limited-light situations such as those involving composite-core buildups and cementation of posts and indirectly fabricated resin or ceramic crowns, inlays, and onlays. However, incompatibilities between simplified dental adhesives and self-cure resins were recognized as early as 1999.[5] Efforts at eliminating this problem can be targeted at three different levels (Figure 1), as follows: 1) Eliminate the acid-base reaction; 2) Reduce the effects of the acid-base reaction; or 3) Eliminate the oxygen-inhibited layer.

The ideal solution would be one that permits continued use of the time-saving simplified dental adhesive with any resin cement without compromising efficiency or bond strength. The remainder of the discussion will explore these levels in detail and correlate them to the results of this study.

Strategy 1—Eliminate the Acid-base Reaction

Elimination of the acid-base reaction can be achieved in two ways: eliminating the acidic component or eliminating the base.

Eliminating the Acidic Component—One could avoid the acid component altogether by exclusive use of nonacidic nonsimplified adhesives. This was reconfirmed by the results of our study, in which we found that with either cement in either cure mode, the nonsimplified adhesives performed the best (Figures 2 and 3).

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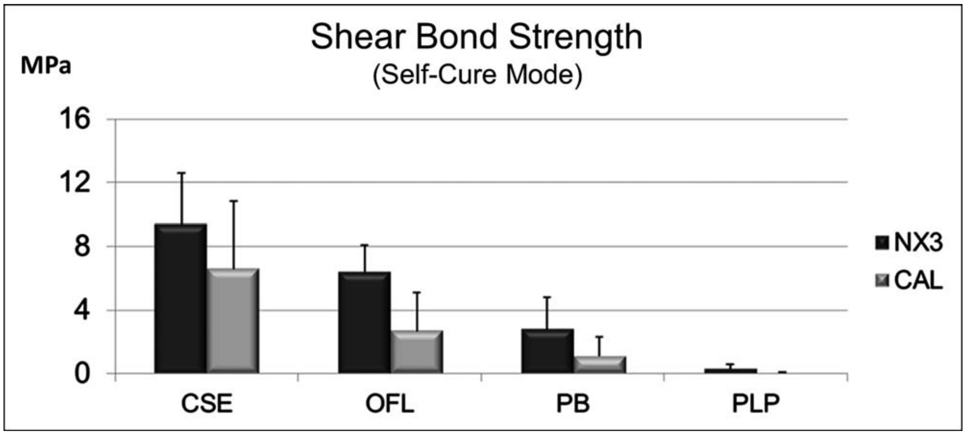

Figure 2. *Shear bond strengths of resin cements (MPa) to dentin in self-cure mode. Error bars represent 1 standard deviation.*

The alternative would be to mask the acid. This has been suggested by use of activators with the simplified adhesives. One commonly used product is the sodium salt of aryl sulfinic acid. This reacts with acidic resin monomers to produce phenyl or benzenesulfonyl free radicals that would then serve to initiate the polymerization of self-cured composites. However, the dentin bond strengths obtained with these activators continue to be suboptimal.[7,8] This is probably because the hydrophilicity of the acidic monomers is not overcome, and osmotic blistering continues to be an issue.[7,8] There is one study[34] that examines the possibility of deprotonization of the acidic adhesive with an anion exchange compound with good results.

Eliminating the Basic Component—Tertiary amines are present in both light- and self-cure resin formulations. In light-cure resins, the light activates the camphorquinone initiator, which is then transformed to its exciplex state by a tertiary amine accelerator. Self-cure systems employ a binary redox catalytic system composed of peroxide and a tertiary amine. However, there is a difference. The tertiary amine in the light-cure formulations is present in far smaller concentrations and is much less nucleophilic than that in self-cure formulations. This, combined with the fact that the light-cure reaction takes place too quickly to allow an acid-base reaction, accounts for the fact that the incompatibilities are restricted to self-cure groups.

The results of our study show that although overall dentin bond strengths with NX3 appear superior to those obtained with Calibra, when we looked at these bond strengths specifically within the realm of simplified adhesives, the bond strengths with NX3 are not statistically higher than those obtained with Calibra. Details on the proprietary redox system are not available in the NX3 Material Safety Data Sheets. Whatever the composition, it is apparent that it does not completely overcome the

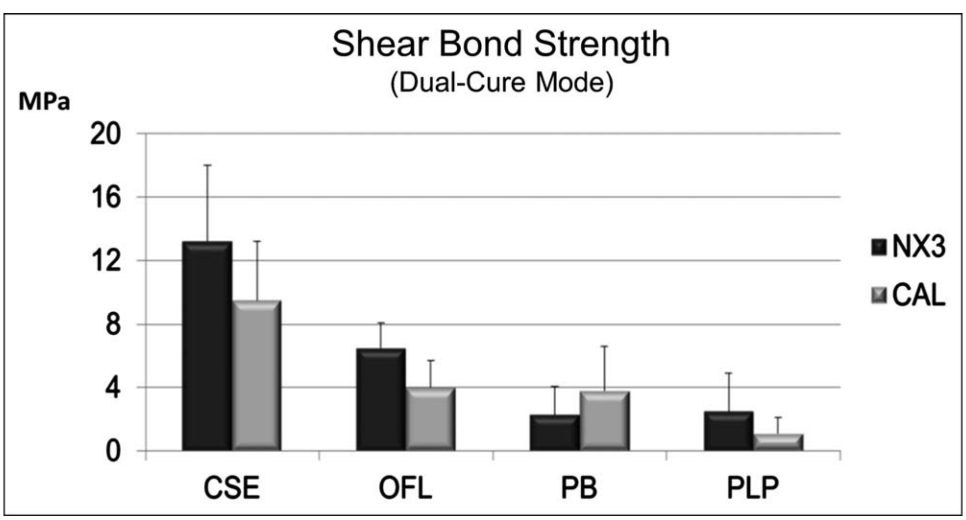

Figure 3. *Shear bond strengths of resin cements (MPa) to dentin in dual-cure mode. Error bars represent 1 standard deviation.*

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incompatibilities. NX3 appears no different from other dual-cure resin cements on the market. It is most likely that the numeric bond strength values of NX3 are higher than those of Calibra because the absence of tertiary amines allows for more complete polymerization of the resin. However, the presence of residual uncured hydrophilic acidic monomers at the oxygen-inhibited interface could continue to contribute to interfacial stresses and less-than-optimal bond strengths. Despite manufacturer's claims, we failed to reject the first null hypothesis.

Strategy 2—Reduce the Effects of the Acid-base Reaction: Light-Cure Adequately

When using a dual-cure composite, if clinical conditions allow, adequate light-curing of the cement is sufficient to overcome the acid-base interactions.[7,8] The bond strength to dentin is directly related to the amount of light energy to which the dentin is exposed.[18] Photo-polymerization results in rapid setting of the resin matrix, allowing no time for adverse acid-base reactions to occur. The light energy should also be able to successfully and rapidly cure the acidic monomers in the oxygen-inhibited layer with remnants of the photo-initiator.[35] Results of the two-way ANOVA confirmed that with either resin cement NX3 or Calibra, the dentin bond strengths were significantly higher when the cement is dual-cured. The second null hypothesis was therefore rejected.

Strategy 3—Eliminate the Oxygen-inhibited Layer

Light- and chemically cured dental composite resins leave a soft, sticky superficial layer upon polymerization. When oxygen diffuses through the superficial layer of resin, it forms peroxide radicals with the monomer. The peroxide radicals are poorly reactive and do not participate in the polymerization reaction, creating the sticky oxygen-inhibited layer. This layer has the same composition as the uncured resin except that it has less of the photo-initiator.[36] Additionally, this layer in simplified adhesives serves both as a source of acidic resin monomers that deactivate the tertiary amines and as a hypertonic medium that triggers osmotic fluid transport through the adhesive layer.[15]

How can we eliminate this layer? The answer is twofold. The oxygen-inhibited layer can be prevented or it can be removed after formation. Preventing formation of the oxygen-inhibited layer can be achieved by polymerization in an inert nitrogen environment (not clinically feasible) or by coating the adhesive with glycerol prior to activation. It has been suggested[37] that one can remove the oxygen-inhibited layer by wiping/rubbing with isopropyl alcohol or prophylaxis with a prophy cup with a mixture of flour of pumice and rubbing alcohol. Since dentin adhesive agents are applied in thin layers, unlike restorative composites, the use of the latter technique could remove too much of the bonding agent, creating direct contact of resin with the hybridized dentin.[15] It is noteworthy that contrary to common perception, presence of an oxygen-inhibited layer is not required for higher bond strengths to additional increments of composite.[36,38,39]

In spite of the sound theory behind elimination of the oxygen-inhibited layer, a study[34] evaluating its efficacy in improving bond incompatibilities of self-etch adhesives on self-cure resins showed persistence of low bond strengths regardless of its presence. This study was very limited in sample size (one sample per group) and perhaps warrants additional research.

CONCLUSIONS

In conclusion, within the limitations of this study, the following recommendations can be made. When used specifically with simplified dental adhesives in either cure mode, NX3 does not produce significantly higher bond strengths than does Calibra. In general, lower bond strengths continue to be observed when simplified adhesives are used with the resin cements in self-cure mode. Clinicians should be cautioned to continue to restrict this usage with limited light-cure situations. Dentists should periodically evaluate their curing lights so they consistently provide adequate output to polymerize these dual-cure cements and combat compromised bond strengths. Dentin bond strengths can be maximized when the resin cement is dual-cured. Clearfil SE Bond shows superior bond strengths with the resin cements in any cure mode.

Disclosure

The views expressed in this study are those of the authors and do not reflect the official policy of the US Air Force, the Department of Defense, or the US Government.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

1. Buonocore MG (1955) A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces *Journal of Dental Research* **34(6)** 849-853.
2. Tay FR, & Pashley DH (2002) Dental adhesives of the future *Journal of Adhesive Dentistry* **4(2)** 91-103.
3. Moll K, Park HJ, & Haller B (2002) Bond strength of adhesive/composite combinations to dentin involving total and self-etch adhesives *Journal of Adhesive Dentistry* **4(3)** 171-180.
4. Armstrong SR, Vargas MA, Fang Q, & Laffoon JE (2003) Microtensile bond strength of a total etch 3-step, total etch 2-step, self-etch 2-step and a self-etch 1-step dentin bonding system through 15-month water storage *Journal of Adhesive Dentistry* **5(1)** 47-56.
5. Swift EJ Jr (1999) Ask the experts: Self-cured composites *Journal of Esthetic Dentistry* **11(2)** 122.
6. Hagge MS, & Lindemuth JS (2001) Shear bond strength of an autopolymerizing core build up composite to dentin with 9 dentin adhesive systems *Journal of Prosthetic Dentistry* **86(6)** 620-623.
7. Tay FR, Pashley DH, Yiu CKY, Sanares AM, & Wei SH (2003) Factors contributing to the incompatibility between simplified-step adhesives and chemically-cured or dual–cured composites: Part 1: Single-step self-etching adhesive *Journal of Adhesive Dentistry* **5(1)** 27-40.
8. Tay FR, Suh BI, Pashley DH, Prati C, Chuang SF, & Li F (2003) Factors contributing to the incompatibility between simplified–step adhesives and self-cured or dual-cured composites: Part 2: Single-bottle, total-etch adhesive *Journal of Adhesive Dentistry* **5(2)** 91-105.
9. Tay FR, Frankenberger R, Krejci I, Bouillaguet S, Pashley DH, Carvalho RM, & Lai CN (2004) Single-bottle adhesives behave as permeable membranes after polymerization *Journal of Dentistry* **32(8)** 611-621.
10. Eick JD, Gwinnett AJ, Pashley DH, & Robinson SJ (1997) Current concepts on adhesion to dentin *Critical Review of Oral Biology in Medicine* **8(3)** 306-335.
11. Reyter IE (1985) *Monomer Systems and Polymerization: Posterior Composite Resin Dental Restorative Materials* Peter Szulc Publishing Co, Utrecht, The Netherlands.
12. Bowen RL, Cobb EN, & Rapson JE (1982) Adhesive bonding of various materials to hard tooth tissues: Improvement in bond strength to dentin *Journal of Dental Research* **61(9)** 1070-1076.
13. Nakamura M (1985) Adhesive self-curing acrylic resin: Composition of 4-meta bonding agent. *Japanese Journal of Dental Materials* **4(2)** 672-691.
14. Ikemura K, & Endo T (1999) Effect on adhesion of new polymerization initiator systems comprising 5-monosubstituted barbituric acids, aromatic sulphonate amides and tert-butyl peroxymaleic acid in dental adhesive resin *Journal of Polymer Science* **72(1)** 1655-1668.
15. Suh BI, Feng L, Pashley DH, & Tay FR (2003) Factors contributing to the incompatibility between simplified-step adhesives and chemically-cured or dual-cured composites: Part 3: Effect of acidic resin monomers *Journal of Adhesive Dentistry* **5(4)** 267-282.
16. Swift EJ, Perdigao J, Combe EC, Simpson CH, & Nunes MF (2001) Effects of restorative and adhesive curing methods on dentin bond strengths *American Journal of Dentistry* **14(3)** 137-140.
17. Sanares AME, Itthagarun A, King NM, Tay FR, & Pashley DH (2001) Adverse surface interactions between one-bottle light-cured adhesives and chemical-cured composites *Dental Materials* **17(6)** 542-556.
18. Takahashi R, Nikaido T, Ariyoshi M, Foxton RM, & Tagami J. Microtensile bond strengths of a dual-cure resin cement to dentin resin coated with an all-in-one adhesive system using two light cure modes *Dental Materials Journal* **29(3)** 268-276.
19. Dentsply Caulk (2008) Calibra Esthetic Resin Cement: Directions for Use. Retrieved online July 1, 2011 from: http://www.dentsply.es/DFU/eng/CalibraDFU_English.pdf
20. Kerr Dental Products (2007) NX3 Nexus Third Generation Advancements–The Redox Advantage: The Powerful Chemistry Behind Maxcem Elite and NX3. Retrieved online July 1, 2011 from: http://www.kerrdental.com/kerrdental-cements-nx3-advancements-2
21. Kerr Dental Products (2007) NX3 Technical Bulletin. Retrieved online July 1, 2011 from: http://www.kerrdental.com/cms-filesystem-action/KerrDental-Products-TechSpecs/nx3-technicalbulletin-082008web.pdf
22. Kerr Dental Products (2011) OptiBond All-in-One Technical Bulletin. Retrieved online July 1, 2011 from: http://www.kerrdental.com/cms-filesystem-action?file=KerrDental-Products-TechSpecs/optibond-aio-techbulletin-july.pdf
23. Kerr Dental Products (2007) Defining a New Generation in Permanent Cements. Retrieved online June 26, 2011 from: http://www.kerrdental.com/kerrdental-cements-nx3-pressrelease-2
24. Bui H, Nguyen TT, Qian X, & Tobia D (2007) A new resin-cement with improved compatibility with one-bottle adhesives *Journal of Dental Research* **85(Special Issue B)** Abstract #1550.
25. Bui H, Qian X, Chen X, & Tobia D (2008) Bond compatibility of NX3 resin-cement with 7th generation adhesives *Journal of Dental Research* **86(Special Issue B)** Abstract #0443.
26. Lui H, Hayes L, & Waller M (2010) Compatibility of self-adhesive cement with bonding agents *Journal of Dental Research* **88(Special Issue B)** Abstract #234.
27. Hirata K, Armstrong S, & Qian F (2012) Material compatibility of self-etching self-cure 1-step adhesive when dentin bonding *Journal of Dental Research* **90(Special Issue B)** Abstract #225.
28. Chen L, Suh B, Shah M, & Shen H (2013) Dentin bonding of universal adhesives with self-cured or delayed-cured

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material *Journal of Dental Research* **91(Special Issue B)** Abstract #576.

29. Dentsply Caulk (2001) Prime and Bond NT: Directions for Use. Retrieved online July 1, 2011 from: http://www.dentsply.com.au/www/770/files/dfu-primeandbondnt.pdf

30. 3M ESPE (2001) Adper Prompt L Pop: Technical Product Profile. Retrieved online July 1, 2011 from: http://multimedia.3m.com/mws/mediawebserver?mwsId=66666UF6EVsSyXTtlxf2NXfXEVtQEVs6EVs6EVs6E666666–

31. Kerr Dental Products (2003) Optibond FL: Technique Summary. Retrieved online July 1, 2011 from: http://www.kerrdental.com/cms-filesystem-action/KerrDental-Products-TechSpecs/OptiBond_FL_80465_Rev1.pdf

32. Kuraray Dental (2004) Clearfil SE Bond: Instructions for Use. Retrieved online July 1, 2011 from: http://www.kuraraydental.com/product/bonding-agents/clearfil-se-bond

33. Kerr Dental Products (2007) NX3 Technique Guide. Retrieved online July 1, 2011 from: http://www.kerrdental.eu/media/Products/ProductFamily/133/en-US/Documents/81878/NX3_Technical%20Guide.pdf

34. Endo T, Finger WJ, Hoffman M, Kanehira M, & Komatsu M (2007) The role of oxygen inhibition of a self-etch adhesive on self-cure resin composite bonding *American Journal of Dentistry* **20(3)** 157-160.

35. Suh BI (2004) Oxygen-inhibited layer in adhesion dentistry *Journal of Esthetic and Restorative Dentistry* **16(5)** 316-323.

36. Eliades GC, & Caputo AA (1989) The strength of layering technique in visible light-cured composites *Journal of Prosthetic Dentistry* **61(1)** 31-38.

37. Brady LA (2011) Removing the air inhibited layer-restorative dentistry; Retrieved online July 1, 2011 from: http://leeannbrady.com/restorative-dentistry/removing-the-air-inhibited-layer

38. Rueggeberg FA, & Margeson DH (1990) The effect of oxygen inhibition on an unfilled/filled composite system *Journal of Dental Research* **69(10)** 1652-1658.

39. Ghivari S, Chandak M, & Manvar N (2010) Role of oxygen inhibited layer on shear bond strengths of composites *Journal of Conservative Dentistry* **13(1)** 39-41.

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