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Long-Term Bonding Efficacy of CAD/CAM Hybrid Restorative Materials and Universal Adhesives

ABSTRACT

Objectives: In-office and lab milled prostheses are the staple for indirect restorations. It is therefore critical to determine their long-term bonding durability. *Methods:* CAD/CAM blocks of two classes of restorative materials: 1) a nano-ceramic reinforced polymer matrix (NCPM) and, 2) a polymer-infiltrated ceramic network (PICN) were bonded using four different universal adhesives (UA) and silane systems. A lithium disilicate glass-ceramic (LDS) was used as a reference. The blocks were bisected and bonded with different UA/resin-cement pairs. Bonded blocks were then cut into 1.0x1.0x12.0 mm bar specimens for microtensile bond testing. Half the bars were subjected to bond strength testing immediately and the other half after aging by 50,000 thermal cycles between 5°C and 55°C. ANOVA and post-hoc tests were used to compare mean bond strength among groups. *Results:* NCPM presented consistently high bond strength regardless of bonding techniques, while the bond strength of PICN and LDS were lower when bonded with UA relative to traditional silanes. The more hydrophilic UA produced higher bond strengths. *Discussion:* Glass-ceramics exhibited lower bond strength with UA than the conventional etch-rinse-silane techniques. However, UAs preserved bonding interface in the long-term. *Significance:* NCPM displayed superior bond strength relative to PICN and LDS regardless of the type of adhesives and bonding techniques..

INTRODUCTION

Nowadays, due to the high demand for aesthetics and longevity, dental restorations are manufactured using ceramics. Materials with superior mechanical properties such as hardness, modulus, strength, fracture toughness, and wear resistance that are not properly polished, may lead to occlusal damage and excessive wear of the antagonist teeth. Therefore, novel materials have been developed that attempt to mechanically and physically match the properties of natural dentition.¹

Several hybrid resin-ceramic materials that mimic the mechanical and optical properties of natural teeth have been introduced. Polymer infiltrated ceramic networks (PICN; Enamic, Vita) and nano-ceramic reinforced polymer matrix (NCPM; Cerasmart, GC) are two hybrid resin-ceramic materials that are derived from varying manufacturing routes. PICN possesses a multiphase structure of both an inorganic, feldspathic ceramic phase (86 wt.% or 75 vol.%) and an organic, dimethacrylate polymer phase (UDMA & TEGDMA; 14 wt.% or 25 vol.%), produced by infiltrating a polymer into a pre-sintered porous ceramic matrix.² NCPM are produced as nano-clusters of ceramic embedded into a resin matrix that is highly cured at high temperature and pressure.³

Both the PICN and the NCPM are indicated for use as single crowns, inlays/onlays, and veneers. In terms of mechanical properties, studies have determined these hybrid resin-ceramic materials to be a favorable option when considering materials for restoration, based on their similarity to natural teeth and their resistance to chipping and fracture.^{4,5} When using these materials as per their indication, the restorations would have to be adhesively cemented to either an implant abutment, coping, or framework. However, the long-term bonding efficacy of these hybrid resin-ceramic materials has yet to be investigated.

Furthermore, to achieve high bond strength adhesive and silane systems are being used to condition the resin composite and tooth surface.

Common etch-and-rinse techniques involve the application of a silane coupling agent to promote the adhesion of dental restorative materials to the tooth surface. Hereby, the etching process is performed to create micro-retentive patterns on the substrate and enable mechanical interlocking and bonding of the silane. The second rinsing step is applied to remove contaminants and provide a clean surface for the silane molecules with both hydrophobic and hydrophilic functional groups. While the hydrophobic group interacts with the resin matrix of the restoration, the hydrophilic part binds with the tooth surface. Eighth generation universal adhesives (UA) are produced and advertised as an etchant, primer, and adhesive all in one bottle. The development of UA replaces the use of separate steps of hydrofluoric acid etch-and-rinse and silane coupling prior to cementation.^{6,7} In addition, the conventional etch and rinse techniques are considered as technique sensitive and not as time efficient. Any slight error or inaccuracy might result in low bond strength or marginal degradations of dental restorations *in vivo*.^{8,9} There have been several studies that demonstrated 8th generation adhesives to be equally effective as the traditional multi-step techniques.^{10,11} However, UAs have also been shown to have weaker bond strength to both dentin and enamel both immediately after cementation and in the long-term, when compared to multi-step adhesive techniques.^{12,13} This discrepancy has delayed the acceptance of 8th generation adhesives in the clinic, especially in the context of contemporary dental restorative materials.

Two of these 8th generation UA are the more hydrophilic Scotchbond Universal (SBU; 3M) and the less hydrophilic All-Bond Universal (ABU; Bisco). Ideally, the use of hydrophilic components is meant to help with infiltration of the adhesive, while the hydrophobic components form a capsule to defend the bonding area from degradation.⁸ Silane on the other hand has been shown to increase wettability and improve the covalent bonds between restoration and the resin cement¹⁴ especially in the case of glass-based ceramics and composites. However, the new UA in the market have attempted to not only simplify the adhesion process for the clinician, but also to increase the bond capability with various components that were added to these mixtures.

10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), a dental adhesive component, has been shown to be an important agent when bonding to tooth structure and dental zirconia ceramics.⁹ It allows for an increase in chemical interactions between the phosphoric acid functional groups 10-MDP contains and the tooth structure or the surface of dental zirconia ceramics and metal alloys.¹⁵⁻¹⁷ 2-Hydroxyethyl methacrylate (HEMA) is another common component in dental adhesives that is hydrophilic and enhances the infiltration capability of adhesives into demineralized dentin.¹⁸⁻²¹ HEMA also increases the miscibility of the hydrophilic/hydrophobic components, allowing the adhesive to work most effectively.^{21,22} In order to increase bond strength, both SBU and ABU contain both of these components besides water. However, SBU has significantly more water in its components making it more hydrophilic. SBU also contains polyalkenoic acid copolymer (Vitrebond Copolymer) which has provided inconsistent results when coupled with 10-MDP.^{9,23,24}

Clinically, dental adhesion has become more focused on the use of UA. It is therefore imperative to assess the bond effectiveness of UA on contemporary hybrid ceramics relative to a widely used glass-ceramic material,²⁵ and to determine the long-term bonding capability and efficacy of these materials using various UAs and silane coupling agents. The significance of this study lies in the evaluation of the long-term bonding efficacy of contemporary hybrid restorative materials used in combination with UA and compared to traditional etch-and-rinse silane techniques. The long-term bonding efficacy is crucial for the clinical success of dental restorations. Accordingly, the following working hypotheses have been formulated: 1. The 8th generation UAs should be as effective as the traditional etch-rinse-silane technique of bonding the contemporary hybrid materials in PICN and NCPM. 2. The long-term thermocycling treatment would result in the degradation of bond strength in these hybrid materials regardless of the type of adhesives and techniques used.

MATERIALS AND METHODS

SAMPLE PREPARATION

CAD/CAM blocks (n = 24, C14, Shade A2 - HT) of two hybrid ceramics (NCPM, Cerasmart, GC, Tokyo, Japan) and polymer infiltrated ceramic network (PICN, Enamic, VITA, Bad Säckingen, Germany) and a lithium disilicate glass-ceramic (LDS, IPS e.max CAD, Ivoclar Vivadent, Schaan, Liechtenstein) (n = 24) were bisected in half using an Isomet Low Speed Saw (Beuhler, Otelfingen, Switzerland).

Prior to cementing the block halves back together, each cross-section underwent surface preparation. All halves had their surface roughened using a 45 µm bur in order to simulate an intaglio milled surface of a dental restoration. Cross-sections were then either etched with Bisco Porcelain Etch 4%

HF (LDS) or sandblasted with aluminum oxide powder (50 µm) at 2 bar for groups NCPM and PICN.

Cross-sections were then treated with one of 4 adhesive/silane treatments (All-Bond Universal (ABU, BISCO), Scotchbond Universal (SBU, 3M ESPE), Porcelain Primer/Bis-Silane (PPB, BISCO), RelyX Ceramic Primer (RCP, 3M ESPE) and then cemented with their respective resin cements in a CAD block-adhesive-CAD block model, as shown in Table 1. Two sets of 12 groups were prepared in order to test for immediate bond strength and long-term bond strength after aging. All surface treatment and cementation protocol followed the manufacturer’s recommendation for each of the adhesives, silanes, and resin cements.

All cemented blocks were placed in water at 37°C for 5 days to allow for further polymerization of the resin cement. After 5 days of storage, samples were sectioned into ~1.0 × 1.0 × 12.0 mm³ bars using a low speed saw under water irrigation (Isomet, Beuhler, Otelfingen, Switzerland) in order to be tested for bond strength. A first set of the 12 experimental groups (n = 40) were used for baseline testing immediately after being sectioned into bars. Another set of the 12 groups (n = 40) were aged by thermo-cycling for 50,000 cycles between 5°C and 55°C water baths for 30 s each to test the long-term bond efficacy.

MICROTENSILE BOND STRENGTH TESTING

Both baseline and aged groups were tested for bond strength using a custom-made microtensile bond strength (µTBS) test jig. The test beams were mounted onto the test jig by glue (Loctite Instant Adhesive 404 Quick Set, Uline, WI). The jig, which consists of 2 aligned aluminum plates, was then clamp-mounted on a universal testing machine (Instron 5566, Norwood, MA). A cross-head speed of 0.5 mm/min and a load cell of 100 N were utilized in the µTBS test.

Bond Strength was calculated as follows:

$$\mu TBS = \frac{\text{Load at Failure (N)}}{\text{Bonding Area (mm}^2\text{)}}$$

DATA ANALYSIS

Preliminary analysis of the data showed heterogeneity in the size of the within cell variances under different combinations of the experimental conditions (Table 1). To homogenize these variances and adhere to the assumption of the ANOVA model, the data were log transformed [log₁₀(x + 1), where x refers to the raw value of µTBS] prior to ANOVA. Comparison of the resulting mean microtensile bond strengths, log₁₀(µTBS + 1), as a function of bond treatment, material, and aging was evaluated with a 3-way ANOVA, followed by post-hoc comparisons of means based on a pooled estimate of the standard error. All analysis used IBM SPSS (v.25, IBM Corp., Armonk, NY, USA). Exact values of statistical significance are shown, except when p < 0.001.

Table 1. Experimental groups.

Material	Surface Prep.	Surface Treatment	Resin Cement
LDS	Etch 4% HF	Bisco Porcelain Primer	Duo-Link Universal
		RelyX Ceramic Primer	RelyX Ultimate
		ALL-BOND Universal	Duo-Link Universal
		ScotchBond Universal	RelyX Ultimate
NCPM	Air Abrasion	Bisco Porcelain Primer	Duo-Link Universal
		RelyX Ceramic Primer	RelyX Ultimate
		ALL-BOND Universal	Duo-Link Universal
		ScotchBond Universal	RelyX Ultimate
PICN	Air Abrasion	Bisco Porcelain Primer	Duo-Link Universal
		RelyX Ceramic Primer	RelyX Ultimate
		ALL-BOND Universal	Duo-Link Universal
		ScotchBond Universal	RelyX Ultimate

LDS: lithium disilicate glass–ceramic (IPS e.max CAD);
 PICN: polymer infiltrated ceramic network (Vita Enamic);
 NCPM: Nano-ceramic reinforced polymer matrix (GC Cerasmart)

RESULTS

After thermocycling, samples with a bond strength of less than 2 MPa were excluded from the data set. These extremely low values of bond strength were less than the detection limit of the universal testing machine with a load cell of 100 N (1% – 2%), which therefore does not provide any valid information of the microtensile bond strength test. Additionally, low bond strength values can be caused by a misalignment of samples such that the stress would not be pure tension.

The mean µTBS values (standard deviation) for all 24 groups are listed in Table 2. The immediately tested CS samples using ABU adhesive as well as with the RelyX Ceramic Primer showed the highest mean µTBS values.

Figure 1 shows the mean log₁₀(µTBS + 1) as a function of material, bonding technique, and time. The 3-way ANOVA model accounted for more than half of the crude variability in log₁₀(µTBS + 1). That is, less than half of the initial variance remained unexplained after fitting this model. In particular, analysis showed a significant 2-way interaction between

Table 2. Mean microtensile bond strength (standard deviation) in MPa.

Adhesive	Storage	LDS	PICN	NCPM
All bond	Immediate	5.12 (3.37)	8.07 (4.90)	41.98 (17.55)
	Aged	4.04 (1.79)	6.39 (4.44)	24.23 (13.27)
Scotchbond	Immediate	6.68 (3.01)	13.84 (9.57)	40.48 (19.08)
	Aged	6.87 (4.31)	10.35 (6.46)	31.21 (16.87)
Bisco Silane	Immediate	25.03 (16.77)	16.95 (10.41)	15.03 (10.30)
	Aged	21.09 (11.94)	24.25 (16.20)	35.18 (15.89)
3M Silane	Immediate	21.12 (14.05)	21.69 (12.97)	41.68 (13.67)
	Aged	9.79 (5.83)	24.89 (13.90)	29.81 (16.88)

LDS: lithium disilicate glass–ceramic (IPS e.max CAD); PICN: polymer infiltrated ceramic network (Vita Enamic); NCPM: Nano-ceramic reinforced polymer matrix (GC Cerasmart)

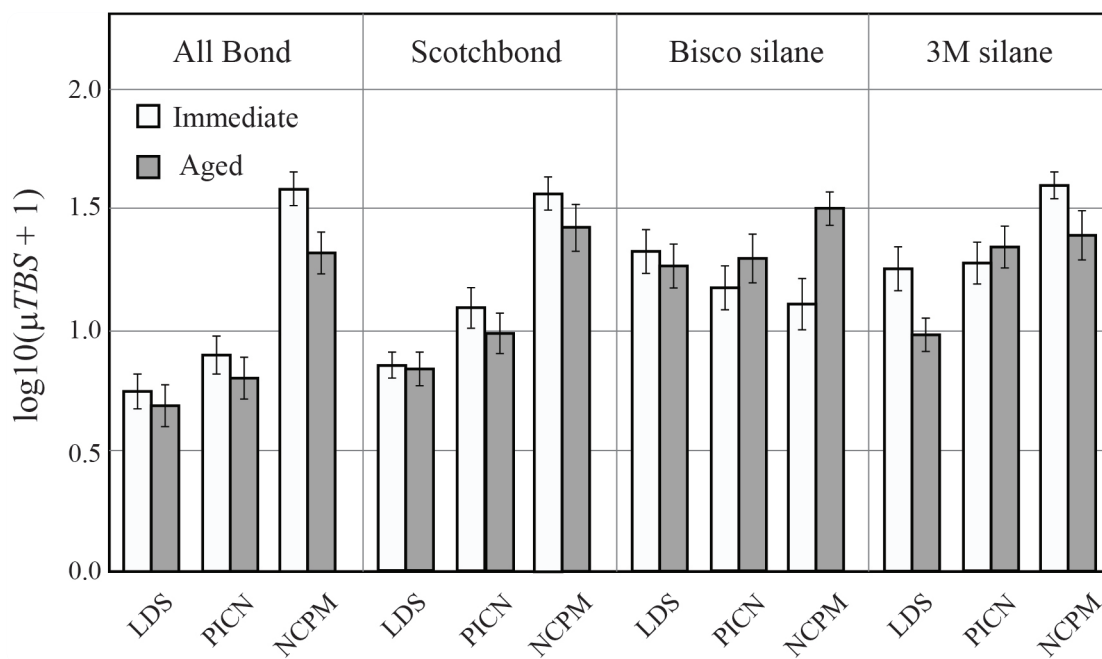


Figure 1: Mean $\log_{10}(\mu\text{TBS} + 1)$ as a function of material, bonding technique, and time. Note that the error bars represent a 95% confidence interval. LDS: lithium disilicate glass–ceramic (IPS e.max CAD). PICN: polymer infiltrated ceramic network (Vita Enamic). NCPM: Nano-ceramic reinforced polymer matrix (GC Cerasmart).

bonding techniques and material ($p < 0.001$), as well as a main effect of aging ($p < 0.001$) (Table 3, Figure 2). As for aging, all combinations of material and bonding technique had higher bond strength when tested immediately after construction than after aging [mean (SD) = 1.22 (0.37) vs. 1.19 (0.37) MPa; $p = 0.003$]. The material and bonding technique interaction is shown in Figure 2. The composite material NCPM displayed a high level of microtensile bond strength regardless of bonding

technique. On the other hand, LDS as well as PICN, a material with high levels of feldspathic ceramic content, were much stronger when paired with the silane bonding agents rather than with UAs.

The more hydrophilic SBU was associated with significantly higher bond strengths than the less hydrophilic ABU when bonding both LDS and PICN.

Table 3. Results of fitting a 3-way ANOVA model.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	60.751 ^a	23	2.641	40.668	0.000
Intercept	1197.351	1	1197.351	18435.303	0.000
Bond	12.126	3	4.042	62.232	0.000
Material	31.156	2	15.578	239.854	0.000
Time	0.557	1	0.557	8.575	0.003
Bond*Material	13.656	6	2.276	35.042	0.000
Bond*Time	3.304	3	1.101	16.959	0.000
Material*Time	0.357	2	0.178	2.745	0.065
Bond*Material*Time	3.400	6	0.567	8.724	0.000
Error	57.025	878	0.065		
Total	1432.720	902			
Corrected Total	117.776	901			

^aR Squared = 0.516 (Adjusted R Squared = 0.503)

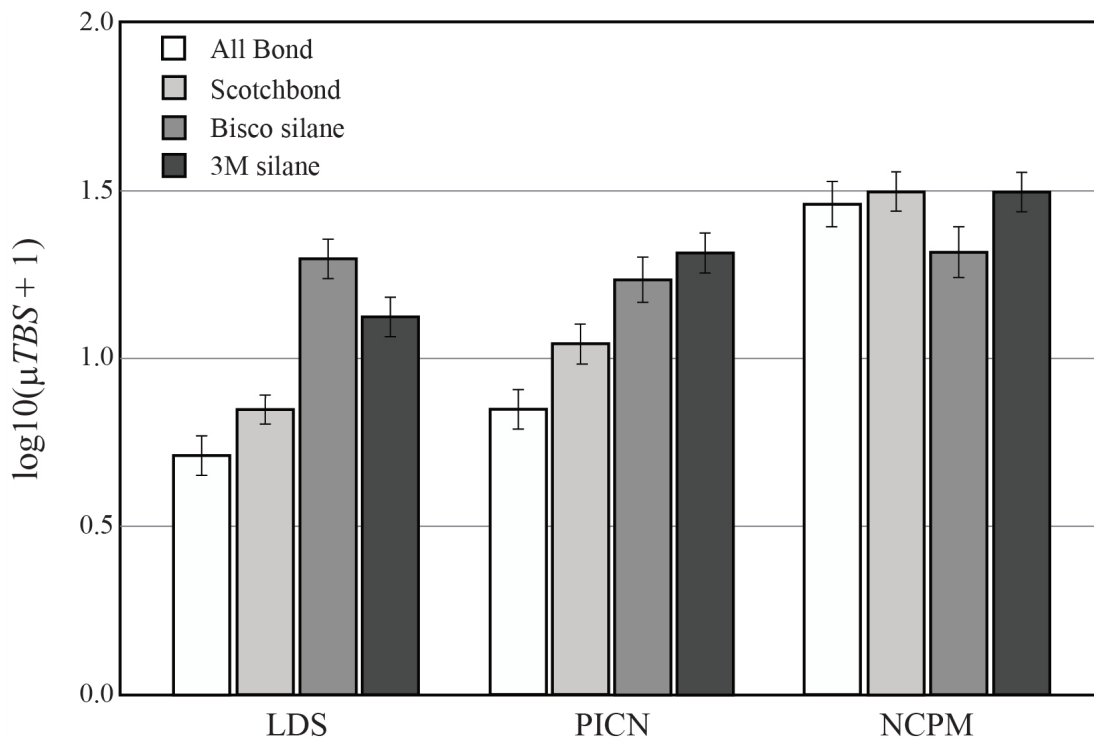


Figure 2: Average of $\log_{10}(\mu\text{TBS} + 1)$ before and after aging as a function of material and bonding technique. Note that the error bars represent a 95% confidence interval. LDS: lithium disilicate glass–ceramic (IPS e.max CAD). PICN: polymer infiltrated ceramic network (Vita Enamic). NCPM: Nano-ceramic reinforced polymer matrix (GC Cerasmart).

DISCUSSION

This study evaluated the effectiveness of universal adhesives on hybrid materials, compared with the conventional etch-and-rinse techniques coupled with silane. Much of the bonding success of UA depends on the class of material that is being used for the restoration. It worked as well as the traditional treatments when bonded to a classical composite material NCPM, but was less effective with materials containing high content of glass–ceramic LDS and PICN. Thus, our first working hypothesis has been rejected.

Our findings confirmed the efficacy of universal adhesives for use with classical composite material systems, in both the short and the long term, but the same could not be said for PICN and LDS. NCPM, a classical composite material, displayed similar levels of bond strength, regardless of the bonding technique that was used in both the short term and the long term. The indications to successfully bond composites does not specify the need for silane and these results confirm the effectiveness of universal adhesives on composite materials, working just as well as the traditional silane methods. However, the lower immediate bond strength when compared to aged samples of NCPM when paired with Bisco Silane defies rational explanation and appears driven by several low-level outliers in that group.

An important factor when bonding the resin cement with glass–ceramics is the necessity of silane to react with the glass following a proper etching treatment.⁹ Therefore, both LDS (glass–ceramic) and PICN (high feldspathic ceramic content) showed good results when using traditional silanes, while the bond strength was exceptionally low for both materials when bonded with universal adhesives. This could be due to the separate steps of etching (properly cleaning and preparing the material) followed by priming (with silane) that ensured a secure bond with the ceramic, rather than using the one-step bottle, where these important factors cannot be properly monitored and controlled.¹⁰

When comparing the effect of the two universal adhesive systems on the glass-containing materials, there was a significantly higher bond strength when using SBU as opposed to ABU. The higher initial hydrophilicity/acidity of SBU provided better etching to the materials and consequently increasing the chemical and mechanical interactions holding the bond together, could be the factor of better bond strength results in glass-containing materials.⁸

The effect of thermocycling on bond strength seemed to depend both on the bonding technique and the material (Figure 1). Although silanes may show more of a reduction in bond strength over time than universal adhesives, even the lowest values of bond strength for the silane groups were comparable to the highest bond strength values that the universal adhesives displayed with both LDS and PICN. This demonstrates that although there were promising factors in securing the

bond interface when using universal adhesives, the conventional silane method still ensured a strong bond in both the short-term and the long-term. Thus, the second working hypothesis has also been rejected.

A limitation of this study concerned the aging procedure that does not perfectly mimic the oral environment. The oral environment contains more than just an aqueous solution. It is involved with saliva, enzymes, and bacteria that could penetrate the affected margins and degrade the bonding interface and resins that are present.^{26–28} Furthermore, the material-dependent nature of the bond effectiveness of UA and the impact of silane should be further investigated in an artificial oral environment to better understand their long-term efficacy *in vivo*.

CONCLUSIONS

It was found that NCPM displayed high bond strength values regardless of the bonding techniques. LDS and PICN exhibited significantly lower bond strength with universal adhesives relative to traditional silanes. In addition, the more hydrophilic Scotchbond Universal yielded significantly higher bond strengths than the less hydrophilic All-Bond Universal when bonding both LDS glass–ceramic and PICN.

CLINICAL RELEVANCE

The nano-ceramic reinforced polymer matrix displays superior bond strength regardless of the type of the adhesives and bonding techniques used. Lithium disilicate glass-ceramics and the polymer infiltrated ceramic network exhibited higher bond strengths with the traditional etch-rinse-silane techniques relative to the 8th generation universal adhesives. However, one advantage of the universal adhesives, regardless of their hydrophilicities, is their ability to preserve the bonding interface in the long-term.

DISCLOSURE

The authors declare that they have no conflict of interest.

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