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Bonding to Glass Ionomer Cements Using Resin-based Adhesives

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Clinical Relevance

The adhesion of self-etching and etch-and-rinse adhesive systems may be effective when bonding to conventional glass ionomer cements (GIC). These systems can be used reliably with the GIC-resin composite laminate technique for restoration of approximal cavities.

SUMMARY

Objective: This study compared the micro-shear bond strengths (MSBS) of four self-etching adhesives (Adper Scotchbond SE [SSE], Clearfil SE Bond [CSE], Clearfil S³ Bond [CS3] and One Coat 7.0 [OC]) and an etch-and-rinse adhesive (Adper Single Bond Plus [SB]) when bonded to two conventional glass ionomer cements (GICs) (Fuji IX GP EXTRA and Riva Self Cure). The null hypothesis tested was there is no difference in the adhesive ability of an etch-and-rinse adhesive and self-etching adhesives when bonded to GIC for up to 6 months.

Methods: The GICs were embedded in type III dental stone and wet ground with 1200-grit SiC paper. Twenty specimens were bonded for each adhesive according to manufacturers' instructions with a 1.5-mm bonding diameter. Specimens were stored at 100% humidity for 24 hours, 1 month, or 6 months. Microshear bond strengths were obtained using a crosshead speed of 1 mm/min. The results were calculated and analyzed using analysis of variance (ANOVA) and Tukey HSD test.

Results: SB had significantly lower MSBS than the four self-etching adhesives for all storage periods. MSBS at 6 months for SB was significantly lower than at 1 month. There were no significant differences in MSBS among the self-etching adhesives. Cohesive failure within GIC was the most common failure mode observed.

Conclusions: SB showed a lower bond strength than the self-etching adhesives when bonded to conventional GICs for all storage periods. This might be a result of the phosphoric acid

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etching. However, cohesive strength of GIC was a limiting factor for the MSBS outcomes.

INTRODUCTION

Glass ionomer cements (GIC) are frequently used as a lining or base material under resin composite restorations as a dentin replacement to seal the dentin with a material demonstrated to form a reliable bond.[1] The concern with conventional GIC, however, is its poor strength when subjected to load. Therefore, an overlay of resin composite is placed on a GIC to provide mechanical strength, wear resistance, and esthetics, while the GIC is able to seal the cavity, reduce microleakage, and provide fluoride release.[2] This laminate technique, as developed by McLean et al.,[3] is especially useful in deep approximal box cavities where radicular dentin is involved.

In the case of a conventional (self-cure) GIC, the bond between the GIC and resin composite filling material is micromechanical in nature and mediated by a resin-based enamel/dentin bonding system. There is currently very little research data on the topic of bonding of GIC and resin-based adhesives, even though this technique is widely recommended for the restoration of large approximal cavities. Few data are available investigating the bond to resin composite. Only two relevant studies, one more than 10 years old and based on materials that have been superseded, and another recent study that investigated only one conventional GIC, not commonly used for the laminate technique, have been published.[4,5] It would be useful to know how recent resin-based adhesives, especially the so-called "all-in-one" systems, interact with conventional GICs recommended for the laminate technique with regard to the quality and durability of adhesion.

Resin-based adhesives have changed rapidly in composition and application method in the last 10 years. The original etch-and-rinse systems used aggressive acid etching to form a micromechanical bond to tooth structure. The same micromechanical bond is also important to bond to a conventional GIC. In the case of resin-modified GICs, there is also a chemical bond between the resin component of the resin-modified GIC and resin of the enamel/dentin adhesive.[6] The use of phosphoric acid etching is declining with the introduction of self-etching priming adhesives, which use acidic resin monomers to etch enamel and dentin and eliminates the rinsing step. The self-etching approach reduces technique sensitivity and clinical time as demonstrated in laboratory studies[7,8] and clinical trials.[9,10] When compared to etch and rinse systems, equivalent adhesion to tooth structure can be achieved with systems such as the two-step self-etching materials. However, for the recent all-in-one systems the evidence shows their performance is more variable.[11] However, the bond of self-etch systems to GIC materials is unknown. Evidence is also mounting that some self-etching adhesives are able to form a chemical bond to the calcium of tooth structure.[12] For those materials that do bond to the calcium in tooth structure, there could possibly be a potential for bonding to the calcium or strontium in the glass of the GIC, which could lead to an improved bond.

Currently, the most common method for the assessment of bond durability is long-term water storage lasting from a few months to several years or even longer. Most contemporary adhesives achieve high bond strengths immediately after polymerization of the resin.[13,14] In the long term, however, the bond strength of most adhesives decreases,[13,15] while others are more stable.[16,17] There have been anecdotal reports that some of these self-etching systems, particularly the all-in-one systems that incorporate etching, priming, and bonding into a single procedure, may not bond to GIC particularly well. At present, there is no evidence to support or deny the anecdote.

The aim of this study was to compare, over 6 months, the microshear bond strengths (MSBS) of two all-in-one adhesives, two two-step self-etching adhesives, and an etch-and-rinse (phosphoric acid etch–based) adhesive when bonded to two conventional glass ionomer cements. The null hypothesis was that there would be no difference in the adhesive ability of the etch-and-rinse adhesive and self-etching adhesives when bonded to GIC up to 6 months.

MATERIALS AND METHODS

Specimen Preparation

Two conventional glass ionomer cements (Table 1) were mechanically mixed (Ultramat 2, SDI Ltd, Bayswater, Victoria, Australia) according to the manufacturer's instructions and embedded in type III dental stone to form the bonding substrate.

All glass ionomer specimens were stored at 37°C in 100% relative humidity for 24 hours, after which they were wet ground with 1200-grit silicon carbide paper to form a flat surface. Twenty specimens of each GIC were prepared for each test group bonded per time period using one of the five bonding systems, ie, n=20 for each adhesive tested bonded

Table 1: *Glass Ionomer Cements and Resin Adhesives: Manufacturers, Batch Numbers and Composition*

Product	Code	Manufacturer	Batch No.	Composition
GC Fuji IX GP EXTRA	FJ	GC Corporation, Tokyo, Japan	810291	Polyacrylic acid 10%-15%, alumino-silicate glass 70%-80%, distilled water 10%-15%
Riva Self Cure	RV	SDI Limited, Bayswater, Victoria, Australia	A0802205	Compartment 1: polyacrylic acid 20%-30%, tartaric acid 10%-15% compartment 2: fluoro aluminosilicate glass 90%-95%, polyacrylic acid 5%-10%
Adper Single Bond Plus (etch-and-rinse adhesive) SDI acid etch gel	SB	3M ESPE, St Paul, MN, USA SDI Limited	6JM 610132	BisGMA, 2-hydroxyethl methacrylate (HEMA), dimethacrylates, silica nanofiller, ethanol, water, photoinitiator system, methacrylate functional copolymer of polyacrylic and polyitaconic acids; 37% phosphoric acid
Adper Scotchbond SE (two-step self-etch adhesive)	SSE	3M ESPE, St Paul, MN, USA	Liquid A: 7AH liquid B: 7AG	Liquid A: water, HEMA, surfactant, pink colorant liquid B: UDMA, TEGDMA, TMPTMA (hydrophobic trimethacrylate), HEMA phosphates, MHP (methacrylated phosphates), bonded zirconia nanofiller, initiator system based on camphorquinone
Clearfil SE Bond (two-step self-etch adhesive)	CSE	Kuraray Medical Inc, Tokyo, Japan	Primer: 00858A bond: 01260A	Primer: 10-methacryloyloxydecyl dihydrogen phosphate (MDP), HEMA, hydrophilic dimethacrylate, dl-camphorquinone, N,N-diethanol-p-toluidine, water bond: MDP, bis-phenol A diglycidylmethacrylate (Bis-GMA), HEMA, hydrophobic dimethacrylate, dl-camphorquinone, N,N-diethanol-p-toluidine, silanated colloidal silica
Clearfil S^3 Bond (all-in-one adhesive)	CS3	Kuraray Medical Inc, Tokyo, Japan	00117A	MDP, diglycidylmethacrylate, bis-GMA, HEMA, hydrophobic dimethacrylate, dl-camphorquinone, ethyl alcohol, water, silanated colloidal silica
One Coat 7.0 (all-in-one adhesive)	OC	Coltène/Whaledent AG, Altstätten, Switzerland	0149473	Methacrylates, photoinitiators, ethanol, water

to each GIC (600 specimens) per time period (Table 1).

One etch-and-rinse adhesive, two two-step self-etching priming adhesives, and two all-in-one adhesives were selected as the resin-based adhesives. The resin-based adhesives were applied to the GIC surface in the same manner as conducted clinically and following manufacturers' instructions (Table 2). The bonding procedure used a microshear bond test method[8,18] as follows: translucent polyvinylchloride tubes of 1.50 mm internal diameter and less than 2 mm high, were firmly placed on the unpolymerized adhesive surface, which was light-polymerized according to the manufacturers' instructions using a light-emitting diode curing unit of 800 mW/cm^2 intensity (Bluephase C8, Ivoclar Vivadent, AG, Schaan, Liechtenstein). The bonded tube was then filled with resin composite (Gradia Direct Anterior A3, batch number: 802071, GC Corporation, Tokyo, Japan) and light-cured for 40 seconds. Microscopic examination (10×) of each specimen following bonding was performed to ensure no voids were present at the base of the tube. The bonded specimens were placed in distilled water and kept in an incubator at 37°C. The storage periods were set at 24 hours, 1 month, and 6 months.

Microshear Bond Strength Test

After the storage periods, specimens were removed from the incubator and tested in shear mode in a universal testing machine (Imperial 1000, Mecmesin, Slinfold, West Sussex, UK) using the corre-

Table 2: *Application of Bonding Systems*

Product	Application Instructions
Adper Single Bond Plus (SB)	Phosphoric acid etch for 15 seconds, immediately after etching, apply 2–3 consecutive coats of adhesive for 15 seconds with gentle agitation using a fully saturated applicator. Gently air thin for 5 seconds to evaporate solvent. Light-cure for 10 seconds.
Adper Scotchbond SE (SSE)	Apply liquid A. Scrub liquid B into the bonding surface for 20 seconds. Air dry for 10 seconds. Apply second coat of liquid B to the bonding surface. Lightly air thin adhesive layer. Light-cure for 10 seconds.
Clearfil SE Bond (CSE)	Apply primer for 20 seconds then dry with mild air flow. Apply bond then air flow gently. Light-cure for 10 seconds.
Clearfil S^3 Bond (CS3)	Apply Bond then dry with high-pressure air flow for more than 5 seconds. Light-cure for 10 seconds.
One Coat 7.0 (OC)	Shake the bottle well before dispensing. Massage One Coat 7.0 using a brush for 20 seconds onto the bonding surface. Gently air dry for 5 seconds. Light-cure for 10 seconds using a light source >800 mW/cm^2.

sponding computer software (Emperor v.01). A stainless steel wire loop (0.35 mm diameter) was positioned such that it contacted the junction between the GIC specimen and resin composite bonded assembly. The load was applied at a crosshead speed of 1 mm/min until bond failure and the load at failure were converted to microshear bond strength (MPa) by dividing the load by the surface area of the specimen. Mode of failure was assessed for each specimen under a light microscope at 40× magnification and classified into the predominant failure pattern, namely: A - cohesive within GIC; B - adhesive between GIC and bonding resin; C - adhesive between bonding resin and composite; D - mixed failure where more than 25% of both B and C occurred; and E - cohesive within resin composite.

Statistical Analysis

For each group, the mean value and standard deviation were calculated. The results were analyzed using an analysis of variance test (ANOVA). Mean values between the groups were analyzed using Tukey HSD test with the significance level set at $p<0.05$.

RESULTS

The results of the microshear bond test are shown in Table 3. The mean MSBS of SB were significantly lower than the mean MSBS of other adhesive systems for all storage periods ($p<0.05$). There were no significant differences in MSBS among the self-etching priming adhesives ($p>0.05$). The mean MSBS of SB at 6 months was significantly lower than its MSBS at 1 month ($p=0.02$). Small, but not statistically significant, decreases in MSBSs were also observed for all other adhesives ($p>0.05$).

The modes of failure for each test group are shown in Table 3. The failure mode showed little variation among the bonding systems tested, or the storage periods, with cohesive failure within GIC (type A) being the most common type of failure observed. An increase in failure pattern B was observed during the study, and it was most prevalent in SB. Failure patterns C and D were not recorded in any specimen.

DISCUSSION

The GIC was embedded in dental stone to assist with maintaining the GIC in a "moisture reservoir" to ensure the material remained hydrated during specimen preparation and bonding, as conventional GICs are known to rapidly dehydrate on exposure to air. In addition, the shear bond test was conducted such that the wire loop contacted the junction between the GIC specimen and the resin composite to ensure that the shearing force being applied to the adhesive interface was as true as possible.

The test results showed no significant differences in MSBSs among all the self-etching bonding systems when bonded to GIC. However, this must be viewed with caution because cohesive failure of the GIC occurred frequently, and the true interfacial bond strengths between the resin and the cement were unclear. This outcome is a commonly reported

Table 3: *The Mean Microshear Bond Strengths, Standard Deviation, and Mode of Failure of the Systems Tested (n=20)**

Group	Mean Microshear Bond Strength, MPa			Mode of Failure, %					
				Type A			Type B		
	24 h	1 mo	6 mo	24 h	1 mo	6 mo	24 h	1 mo	6 mo
SB/RV	10.6[a] (2.2)	10.3[a] (2.8)	8.7 (1.9)	90	80	80	10	20	20
SB/FJ	10.9[b] (2.2)	10.8[b] (2.8)	9.1 (2.1)	95	85	80	5	15	20
SSE/RV	12.1[c] (2.1)	12.0[c] (2.6)	11.2[c] (2.5)	95	90	90	5	10	10
SSE/FJ	12.3[c] (2.2)	12.2[c] (2.4)	11.2[c] (2.6)	95	95	90	5	5	10
CSE/RV	11.9[c] (2.1)	12.2[c] (2.6)	11.2[c] (2.4)	100	95	90	0	5	10
CSE/FJ	12.6[c] (2.3)	12.6[c] (2.6)	11.5[c] (2.5)	100	100	95	0	0	5
CS3/RV	11.9[c] (2.0)	12.1[c] (2.7)	11.0[c] (2.2)	100	90	90	0	10	10
CS3/FJ	12.2[c] (2.2)	12.3[c] (2.7)	11.4[c] (2.4)	100	95	90	0	5	10
OC/RV	12.2[c] (2.4)	12.2[c] (2.5)	11.2[c] (2.6)	100	95	90	0	5	10
OC/FJ	12.3[c] (2.2)	12.3[c] (2.5)	11.4[c] (2.5)	100	95	95	0	5	5

** Values are means (standard deviations). Results with same superscript letters are not significantly different ($p>0.05$).*
Abbreviations: SB, Single Bond Plus; SSE, Scotchbond SE; CSE, Clearfil SE Bond; CS3, Clearfil S3; OC, One Coat 7.0; RV, Riva GIC; FJ, Fuji IX GP Fast.

occurrence for adhesive testing of GICs. While some reports in the literature suggested that cohesive fracture within the substrate was indicative of higher bond strength,[19,20] other reports found no correlation between fracture mode and shear bond strength value.[21,22]

The cohesive failure pattern of the GIC was the predominant failure mode for all three storage periods, which is in accordance with other studies.[23,24] While the susceptibility of GIC to this type of failure is likely to be due to limitations of its physical properties, bond failure is a far more complex phenomenon. Processes that could induce cohesive failures within the substrate may include eccentric stress distribution during testing, microporosities within the cement itself acting as potential stress points,[19] differences in the setting reactions of the two materials,[6] and curing contraction of resin composite which could pull GIC off the margins.[7]

The cohesive strength of the GICs is a factor influencing the MSBS in this study and any changes in the GIC strength could therefore influence the success of a load bearing restoration, not only at the GIC-resin composite adhesive interface, but also in the bulk of the GIC itself. Changes within GIC over time are a complex phenomenon. In some instances, an improvement of mechanical strength and wear resistance due to the further setting of the cement and interaction with salivary ions may be observed.[25] However, there is also the possibility of simultaneous weakening of the cement occurring via erosion from plaque and other acids in association with the plasticizing effects of water. Most studies have reported that GIC gains most of its mechanical strength during the first day to 1 week after mixing and remains relatively stable over several weeks and months.[26,27] At 6 months, a decline in MSBS was detected for all adhesives, but this was only significant for Single Bond Plus. While this may possibly be attributed to the weakening of GIC by erosion and the plasticizing effects of water on the resin,[28] it may also relate to the lower MSBS values detected for adhesive failures at 6 months. Although no significant differences were observed, the Riva groups showed lower MSBS overall compared with the Fuji IX groups. These results may be attributed to the differences in the physical properties and overall strength between the two test GICs.

The durability of the bond between GIC and resin composite is of significant importance for the longevity of laminate restorations. The bond strengths deteriorated slightly for all of the adhesives despite which GIC was bonded. Other studies have also shown bond strength deterioration when similar etch-and-rinse and all-in-one systems were bonded to enamel and dentin[29,30] While the proportion of adhesive failures remained relatively low across all time periods, a small decrease in adhesive strength was detected after an increased time period of water storage. At 24 hours, adhesive failures were only recorded in two of the five systems, namely, Single Bond Plus and Adper Scotchbond SE. After storage for 1 month, there was a 5%-10% increase in adhesive failures with the exception of Clearfil SE Bond. Further increases in adhesive failures were observed for some groups when the microshear bond tests were performed at 6 months. A number of mechanisms have been proposed to explain the deterioration in bonding effectiveness with the foremost being the degradation of the bonding resin and GIC at the interface by hydrolysis. Water sorption could also reduce the mechanical properties of the polymer matrix by swelling and reducing the frictional forces between the polymer chains, due to "plasticization."[31] In addition, residual components from the bonding procedure such as uncured monomers and break-down products, such as from resin hydrolysis, may also lead to weakening of the bond.[32]

Although enamel and dentin pretreatment before the application of bonding resin and restorative materials is well established in the literature,[33] the need for surface treatment of the GIC before the placement of laminate restorations remains debatable. While some studies have found that aggressive acid etching improved the bond strength of GIC to resin composite by forming a rough and porous surface to allow the infiltration of the bonding resin to form a GIC hybrid-like layer,[34,35] other studies have found no consistent bond improvement[36,37] and some reported a decrease.[38] In the present study the etching times recommended by the manufacturer were used to replicate the clinical scenario. However, it was observed that even with the short etch time for Single Bond Plus (15 seconds), the use of phosphoric acid (pH 0.8) etching resulted in a significantly lower MSBS than the four self-etching systems for all storage periods. It has been reported that aggressive etching undermines the cement surface by preferential dissolution of the filler particles.[37,39] The zone of weakened cement is therefore more likely to fail cohesively when debonded from resin, possibly resulting in lower bond strengths. In addition, the adhesive performance of the etch-and-rinse system, Single Bond Plus, could have also been affected by incomplete solvent evaporation after its application or by the intrinsic water present in the set GIC. Both factors may account for the slightly greater number of adhesive failures observed at 24 hours. The incorporation of a rinse and drying step tends to increase the sensitivity of the technique and may possibly further weaken the GIC surface by its desiccation and subsequent surface crack formation during drying. The experimental method differs from the clinical situation in that the GIC specimens were bonded after setting for 24 hours. This was done to standardize specimen production. Clinically, bonding occurs after the initial set of the GIC, usually within 7 minutes. It is possible the effect of the etching with phosphoric acid or self-etching primer may be different in this case because the GIC has not had time to completely mature.

The current study is one of the few studies to investigate the microshear bond strength of resin-based adhesives to GIC up to 6 months' duration. With cohesive failures in GIC being the limiting factor in this study, the MSBS recorded were not necessarily indicative of the true interfacial adhesion between the resin and the GIC. While conventional GICs have been the traditional materials used in the laminate technique, investigating the bond strengths of all-in-one adhesives to resin-modified GIC would be worthwhile.

CONCLUSIONS

Within the limitations of this study, the results suggest that when bonded to conventional GICs, the etch-and-rinse adhesive may not be as stable as all-in-one self-etching and two-step self-etching adhesives. Hence, the null hypothesis was rejected. It was also observed that water storage for 6 months significantly reduced the MSBS for the etch-and-rinse adhesive used in the study.

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