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Comparison of Enamel and Dentin Microshear Bond Strengths of a Two-step Self-etching Priming System with Five All-in-One Systems

MF Burrow • Y Kitasako
CD Thomas • J Tagami

Clinical Relevance

The newest all-in-one adhesives demonstrated bond strengths similar to other currently available enamel/dentin adhesives. Bonding to enamel showed lower bond strengths; however, clinical data are needed to determine the relevance.

SUMMARY

Data on the adhesive strength of new all-in-one adhesives are still relatively limited. This study compared the microshear bond strengths of five recent all-in-one self-etching priming systems (G-Bond, One-Up Bond-F Plus, Clearfil S^3 Bond, Adper Prompt L-Pop and Go!) with a widely used two-step self-etching priming system (Clearfil SE Bond).

Human molars were sectioned and finished with 600-grit SiC paper. Both enamel and dentin were bonded using adhesives with a 0.7 mm bonding diameter. Bond strengths were tested using a microshear bond test method at a crosshead speed of 1 mm/minute. The mean bond strengths and standard deviations were calculated and analyzed using ANOVA and the Tukey's HSD test.

Results showed the two-step self-etching system had significantly higher bond strengths to dentin. However, for enamel bond strength, Clearfil SE Bond showed no statistical difference to G-Bond and Go!; however, all of the other mate-

*Michael F Burrow, MDS, PhD, MEd, professor and clinical dean, School of Dental Science, The University of Melbourne, Victoria, Australia

Yuichi Kitasako, DDS, PhD, instructor, Department of Cariology and Operative Dentistry, Graduate School, Tokyo Medical and Dental University, Tokyo, Japan

C David Thomas, MEng (IT), research officer, School of Dental Science, The University of Melbourne, Victoria, Australia

Junji Tagami, DDS, PhD, professor and dean, Faculty of Dentistry, Department of Cariology and Operative Dentistry, Graduate School, Tokyo Medical and Dental University, Tokyo, Japan

*Reprint request: 720 Swanston Street, Victoria 3010, Australia; e-mail: mfburrow@unimelb.edu.au

DOI: 10.2341/07-125

rials were statistically lower. It is necessary to examine these new materials clinically to determine their efficacy.

INTRODUCTION

The advent of self-etching priming adhesives has simplified the use of resin-based bonding systems in clinical practice. The first versions were based on two-bottle systems with a separate etching-priming liquid, followed by the application of an adhesive resin. These systems have been shown to be less technique sensitive[1] and have the capability of bonding to various locations on teeth, for example, coronal, radicular and pulp chamber dentin.[2-3] Data from clinical trials have also indicated these systems are a good alternative to those systems that have a separate etch, primer and adhesive and have been shown to perform better than the "wet bonding" phosphoric acid etch-based systems.[4-5]

Self-etching systems act by dissolving the smear layer but leave remnants of the smear plugs and etch the superficial enamel and dentin at the same time. It is this simultaneous etch and infiltration of the resin that is believed to be conducive to forming a well-infiltrated hybrid layer, even to dentin approximating the pulp.[6]

Recently, manufacturers have further developed self-etching priming resin-based adhesives into a single solution, often referred to as "all-in-one" systems. These adhesives combine the etching, priming and adhesive steps into one process. Although very simple in technique, studies show that these systems may not perform as well as two-step self-etching priming systems. This is thought to be partially due to water in the adhesive, which is needed to maintain its acidity and also the smear layer being incorporated into the adhesive layer.[7-9] These systems have also been divided into low pH and high pH systems, and their subsequent mode of adhesion is also thought to vary.[6] Van Meerbeek and others[6] have stated that low pH systems tend to form a micromechanical bond to dentin; whereas, higher pH systems are believed to incorporate a chemical interaction with calcium in the dentin, in addition to the micromechanical bond.

Comparative data on adhesive ability for the recent all-in-one systems is still limited. The aim of this research was to compare the micro-shear bond strengths of one two-step self-etching adhesive with five commercially available all-in-one systems. The null hypothesis is that there is no difference among bond strengths of the systems tested.

METHODS AND MATERIALS

Human molars, which were used within three months after extraction, were stored in water containing Thymol and maintained at 4°C. The teeth were extracted as part of a comprehensive treatment plan and collected after obtaining verbal consent from the patient to retain the teeth for research purposes. Ten teeth were used for the study. The roots were removed from the teeth at the cemento-enamel junction, then the crowns were sectioned bucco-lingually using a diamond blade and stored in water prior to embedding in Type III dental stone. The sectioned surfaces comprised enamel and dentin, which were finished with wet 600-grit silicon carbide paper for approximately 10 strokes to ensure a flat surface and uniform smear layer.

The adhesive systems used are listed in Table 1. The enamel and dentin were bonded according to the manufacturers' instructions. The bonded area was delineated by placing a piece of polyethylene tube 0.7 mm in internal diameter and approximately 1.5 mm high on the uncured resin bonded surface and light cured. The tube was filled with Clearfil APX resin composite (Kuraray Medical, Tokyo, Japan, Batch #1079AA) and cured for 20 seconds. Each of approximately five tubes was bonded to the enamel and dentin of each tooth section. The bonded specimens were stored in water at 37°C for 24 hours.

The specimens were placed in a jig attached to a universal testing machine, and a loop of stainless steel wire was placed around the resin composite and loaded at a rate of 1 mm/minute until bond failure occurred. The load at failure was recorded, then converted to MPa. Fifteen specimens were tested for each resin-based adhesive for enamel and dentin.

The fracture mode for each of the bonded tubes was also recorded. The fracture pattern was divided into one of three types: a) partial adhesive failure, where remnants of bonding resin remained on the enamel or dentin; b) adhesive failure at the resin-enamel or resin-dentin interface; c) mixed adhesive failure and cohesive failure in enamel, dentin or resin composite.

The mean bond strengths were calculated then analyzed statistically using univariate analysis of variance (ANOVA) followed by multiple comparisons between groups using the Tukey's HSD test with the significance level set at $p<0.05$.

RESULTS

The mean bond strengths to enamel and dentin are listed in Table 2. The failure modes are listed in Table 3. All systems showed the bond strengths to dentin were greater than to enamel.

Results of the ANOVA showed that both the substrate (enamel or dentin) and material significantly affected bond strengths $p<0.05$. For materials bonded to enamel, the two-step self-etching-priming material SE Bond was significantly stronger than S[3], Adper Prompt L-Pop and One-Up Bond F ($p<0.05$). One-Up Bond F was significantly weaker than all the other test materials.

Table 1: *Materials and Manufacturers Used*

Material	Manufacturer	Batch #	Composition
All-in-One Systems			
Go!	Southern Dental Industries, Bayswater, Australia	Experimental	Phosphoric methacrylate monomer, TEG-DMA, UDMA, acetone, water, photoinitiators, SiO_2 nanofiller
S^3-Bond	Kuraray Medical, Tokyo, Japan	00001A	HEMA, Bis-GMA, 10-MDP, silanated colloidal silica, CQ, ethyl alcohol, water
G-Bond	GC International, Tokyo, Japan	504011	4-MET, UDMA, Dimethacylate component, phosphoric ester monomer, acetone, water
Adper Prompt L-Pop	3M-ESPE, St Paul, MN, USA	198523	Liquid 1: Methacrylated phosphoric esters, Bis-GMA, CQ, stabilizers Liquid 2: water, HEMA, polyalkenioc acid, stabilizers
One-Up Bond F Plus	Tokuyama Dental, Tokyo, Japan	Liquid A: 012M Liquid B: 511M	Liquid A: Methacryloyloxyalkyl acid phosphate, MAC-10, MMA, Bisphenol A polyethoxy methacrylate Liquid B: HEMA, MMA, Fluoroaluminosilicate glass, borate catalyst, water
2-step Self-etching Primer			
Clearfil SE Bond	Kuraray Medical	1079AA	Primer: 10-MDP, HEMA, hydrophilic dimethacrylate, photoinitiator, water Bond: 10-MDP, Bis-GMA, HEMA, dimethacrylate, microfiller

TEG-DMA: triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate; HEMA: 2-hydroxy ethylmethacrylate; Bis-GMA: Bisphenol A diglycidylmethacrylate; CQ: camphorquinone; 4-MET: 4-methacryloxy ethyltrimerllitic acid; MAC-10: 11-methacryloxy-1, 1-undecanedicarboxylic acid; MMA: methyl methacrylate.

Table 2: *Mean Microshear Bond Strengths to Enamel and Dentin MPa (±SD); n=15*

All-in-One Systems						**2-step SE System**
	Go!	**S^3-Bond**	**G-Bond**	**Adper Prompt L-Pop**	**One-Up Bond F Plus**	**Clearfil SE Bond**
Enamel	27.7 (6.3)[a,b]	22.2 (5.2)[a]	27.1 (4.0)[a,b]	23.0 (7.1)[a]	12.9 (2.3)	30.2(5.0)[b]
Dentin	35.2 (5.4)[c]	33.5 (4.9)[c]	33.2 (4.5)[c]	32.0 (12.6)[c]	17.7 (3.4)	43.6(5.1)

Figures with the same superscript numbers are not statistically significantly different (p>0.05).

Table 3: *Fracture Modes to Enamel and Dentin*

	All-in-One Systems																**2-step SE System**	
	Go!			**S^3-Bond**			**G-Bond**			**Adper Prompt L-Pop**			**One-Up Bond F Plus**			**Clearfil SE Bond**		
	a	b	c	a	b	c	a	b	c	a	b	c	a	b	c	a	b	c
Enamel	0	13	2	1	12	2	0	12	3	0	12	3	0	15	0	0	12	3
Dentin	0	14	1	0	12	3	0	12	3	1	12	2	0	15	0	0	11	4

Modes of failure: a—partial adhesive failure, where remnants of bonding resin remained on the enamel or dentin; b—adhesive failure at the resin-enamel or resin-dentin interface; c—mixed adhesive failure and cohesive failure in enamel, dentin or resin composite.

When bonded to dentin, SE Bond was significantly stronger than all the other materials tested ($p<0.01$), and One-Up Bond F was significantly weaker than all the other test materials ($p<0.01$).

Go! was significantly stronger than S^3 and One-Up Bond F Plus ($p<0.05$) when bonded to enamel. The bond strength to dentin for Go! was not significantly different from S^3, G-Bond and Adper Prompt L-Pop.

The failure mode showed little variation among the materials tested, with adhesive failure being the most common type of failure observed.

DISCUSSION

Simplification of the self-etching priming systems has not led to an improvement in bond strengths. The results from this study indicate that the two-step self-etching priming adhesive provided consistently stronger bonds to both substrates, as has been previously reported.[8] However, with the exception of One-Up Bond F Plus, the all-in-one adhesives produced remarkably similar bond strengths to both enamel and dentin. Adper Prompt L-Pop, however, showed greater variability when bonded to dentin, which may indicate slightly greater technique sensitivity compared with the other materials tested. This is in variance to the recent study by Perdigão and others,[8] who showed Adper Prompt L-Pop bonded well to enamel and dentin; whereas, the current study showed Adper Prompt L-Pop bonded to a lesser extent to enamel than dentin. This may be due to the different methods used. The recent study by Grégoire and Millas[10] classified self-etching systems into three broad groups, depending on the pH of the adhesive. The most acidic materials exhibited a pH of less than 1, an intermediate group, of which One-Up Bond F Plus fits and has a pH of 1-1.5 and a third group, with a pH of greater than 1.5, of which Clearfil SE Bond belongs. The pH of G-Bond—2.3 and Clearfil S^3—2.4 was reported by Nishitani and others.[11] The pH of the latter two materials is much higher than the other systems tested; however, bond strengths were not different from the other materials tested. Therefore, pH alone is not the sole parameter for achieving a good bond.

In their study, Sidhu and others[12] showed that G-Bond did not perform as well as the other all-in-one systems they tested. The current study showed that all-in-one systems varied little, other than One-Up Bond-F Plus. However, both the current study and the one conducted by Sidhu and others[12] showed the two-step system performed better than the all-in-one systems.

The current study is one of the few studies to investigate the microshear bond strength of all-in-one systems to enamel and dentin. Most other studies have used the microtensile bond test method, which is more complicated and labor intensive for specimen production. In addition, the production of specimens has the potential to induce microcracks at the interface, which can affect bond failure, particularly in enamel.[13] When enamel bonding is evaluated, the microshear bond strength test method is considered the more suitable method compared with the microtensile test method. The microshear test is able to bond a number of tubes to the same tooth simultaneously. It is very important, however, to ensure that the wire loop is pushed against the tooth to ensure the shear stress is applied to the adhesive interface between the tooth and resin composite.[14] Embedding the teeth in dental stone maintained a moisture reservoir to ensure the tooth sections remain hydrated during specimen preparation and debonding. Orientation of the tooth sections was also different from microtensile bond studies in that the teeth were sectioned bucco-lingually, which is equivalent to bonding to the sides of cavity walls/margins, rather than the occlusal surface, which is typically done for the microtensile test. This orientation may have had some influence on bond strengths, as several studies indicate that dentin tubule and enamel prism orientation can affect bond strengths.[15-18] Care was taken to ensure that the samples bonded to dentin were as close as possible to the dentino-enamel junction to avoid the influence of regional differences in the dentin, such that a close approximation to pulp horns could have increased variation in the results.

Although One-Up Bond F Plus showed lower bond strengths, a recent clinical study using the previous version (One-Up Bond F) showed good results over five years.[19] Therefore, attaining very high bond strengths is not necessarily an indicator of clinical success. It would appear that other parameters, such as chemical interaction with the tooth surface and bond stability over the long-term, may be important for the clinical success of bonded restorations.

It is interesting to note that, although a system, such as Adper Prompt L-Pop, has a low pH, it showed no difference in bond strengths to ground enamel compared with the milder etching systems. It would have been anticipated that this system should have etched and bonded well to the enamel surface, but it produced bond strengths equivalent to S^3 and was significantly less than the two-bottle system SE Bond. The pH of the adhesive is not the only factor to determine the bond. Functional monomers, the catalyst used, as well as other components in the bonding system, vary among products. All of these factors will influence bonding. However, all systems tested showed better bond strengths to the dentin surface compared with enamel. This could lead to potential problems during curing of the resin composite, as curing contraction could pull the resin composite off the enamel margin, leaving it susceptible to leakage, staining and caries. To overcome this possible problem, particularly to uncut enamel where it has been shown that bond strengths are reduced, the GC Corp for G-Bond recommends the use of phosphoric acid to ensure a good enamel bond.[20] Further investigation should be carried out to determine whether the etching of enamel prior to application of self-etching solutions will provide any clinical benefit to retention rates and reduced marginal staining around restorations.

CONCLUSIONS

It seems that bond strengths, which can be attained by all-in-one systems, may provide satisfactory results clinically, although far more data are needed to confirm this. The null hypothesis was partially rejected, since the two-step system demonstrated superior results when bonding to enamel in this laboratory study.

However, it is necessary to conduct clinical trials to clearly ascertain performance of the new, all-in-one systems when used to restore teeth.

(Received 30 July 2007)

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