

ORIGINAL ARTICLE

Shear bond strength of one etch-and-rinse and five self-etching dental adhesives when used by six operators

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Abstract

Objective. To test the hypothesis that some single-bottle self-etching adhesives bond as well to enamel and dentin as a typical two-bottle etch-and-rinse adhesive. **Material and methods.** Six operators used one two-bottle etch-and-rinse dentin adhesive (Scotchbond MP) and five all-in-one self-etching adhesives (iBond Gluma Inside, Clearfil S³ Bond, iBond Experimental, Xeno IV, and G-BOND). Each operator carried out six bondings to enamel and six bondings to dentin with each adhesive. After 24 h of storage in water at 37°C, bond strength was determined in shear. **Results.** The pooled results of all the adhesives revealed no significant difference ($p > 0.05$) in bond strength between dentin and enamel. However, there were significant differences ($p < 0.0001$) between the different adhesives. The etch-and-rinse adhesive did better than the self-etching adhesives when substrate was not an issue (pooled enamel and dentin results). On comparing the performance of the different adhesives, it became clear that there were significant interactions ($p < 0.0001$) between substrates and products. There were also significant differences ($p < 0.0001$) between operators, and the interaction between operators and products was significant ($p < 0.0002$). **Conclusions.** The tested etch-and-rinse adhesive did better than the tested self-etching adhesives. The shear bond strength results were also strongly affected by the operator as well as by the interaction between operator and used product. The pooled bond strength values of the different adhesives revealed no difference in bond strength to dentin versus enamel.

Key Words: Air-thinning, dentin bonding, enamel bonding, technique sensitivity

Introduction

Dentin adhesive research data published during the past 15 years reflect large differences in bond strength values between operators and research groups [1–5]. The different results produced by different investigators have been attributed to several factors, including differences in substrates [5], in load rates [5], in testing methods [6], and in technique sensitivity [7–9]. To decrease technique sensitivity, easier-to-use products have been developed in an attempt to decrease the number of steps where things can go wrong [7–10]. Unfortunately, technique sensitivity is not easy to define, because what one dentist perceives as being an easy to use product with good performance may not behave as well in the hands of another dentist. Technique sensitivity is simply a combination of different

clinical usage variables and how these interact during use and affect the final outcome [10,11].

It is reasonable to assume that by decreasing the variables introduced during usage, technique sensitivity decreases [10] and the clinical outcome can be improved. However, in a previous study in which completely inexperienced bonders participated, it was found that an etch-and-rinse adhesive with separate primer and adhesive still performed better than two self-etching all-in-one adhesives, even though the two self-etching adhesives were clearly easier to use [12]. That study also revealed that adhesion to dentin, at least for one of the self-etching adhesives, was greater than adhesion to enamel and was attributed to an inferior enamel etching ability of that self-etching adhesive. Similar findings reported by others [13] have been attributed to a more

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efficient enamel etching with 30–40% phosphoric acid than with self-etching adhesives.

During the past few years it has become clear that the removal of solvent from the adhesive is an important step in the bonding procedure [14–17]. Some manufacturers now recommend the use of high or maximal air pressure in order to remove the solvent components. However, some research data published regarding this topic are still not conclusive [14,15] and need to be further explored.

The objective of this study was to determine whether some new one-bottle self-etching adhesives, including some requiring the use of high or maximum air pressure, *in vitro* perform as well as a traditional etch-and-rinse system with separate primer and adhesive when used by different operators for both dentin and enamel bonding.

Material and methods

Materials

One two-bottle etch-and-rinse dentin adhesive [Scotchbond MP (SMP)] and five all-in-one self-etching adhesives [iBond Gluma Inside (IBGI), Clearfil S³ Bond (CSB), iBond Experimental (IEXP), Xeno IV (XIV), and G-BOND (GB)] were evaluated (Table I). In our study, a 40% phosphoric acid gel (Etch Gel 40%; Henry Schein Inc., Melville, NY, lot no. 032102) was used to etch enamel and dentin before SMP was placed. The one-bottle self-etching adhesives were used in their bottle forms. Product usage followed the instructions given by the manufacturers.

Operators

Two of the participants were residents and one was a fellow, all from the pediatric dentistry department (assigned as A1, A2, and A3), while the remaining three participants were faculty members with many years of clinical experience (assigned as B1, B2, and B3).

Teeth

The 216 lower 3rd molar teeth used for the study were collected primarily from Danish military recruits, suggesting that most of the teeth were from

male individuals 19–20 years old. All the teeth were first embedded in gypsum (Silky Rock) with their facial surfaces being slightly exposed. After the gypsum had set, the gypsum surfaces with the exposed enamel surfaces were ground flat under water with sandpaper (600 grit) until sufficiently large flat enamel surfaces, exceeding the surface areas needed to form the tooth-composite bond, were exposed. These 216 teeth were divided into 6 groups of 36 teeth each. The groups were then assigned to the six different operators participating in the study.

After the operators had completed their enamel bonding exercise on these surfaces and the teeth had been tested, the enamel surfaces were ground to expose flat dentin surfaces in the outer thirds of the dentin. The exposed dentin surfaces were ground flat under water with sandpaper (600 grit), and the teeth were assigned to the same operator that had performed enamel bonding on those teeth.

Bonding procedures

Before the bonding exercise started, the participating operator was shown how to perform a bonding and asked to read through the bonding instructions for the different products supplied by the different manufacturers. After reviewing the instructions, each operator was asked if any clarification was needed. None asked for any clarification. The operators were then told: to start the enamel bonding procedure with product SMP by preparing two specimens, to proceed with IBGI by preparing two specimens with that product, to continue with CSB, IEXP, XIV, and GB (two bonded specimens per material), and then to go back and repeat a second and third bond cycle in the same way as for the first bond cycle. By using such an approach, we hoped to minimize the learning effect that otherwise could skew the results.

The bonding agent was first placed and cured on the tooth surface, all in accordance with the manufacturer's instructions. A stainless steel metal disk (called "mold" in the text) with a central hole ($\varnothing = 3.0$ mm), and covered with adhesive tape on the mold surface in contact with the tooth surface, was then centered over the region where the composite would be bonded. The adhesive tape was placed there to prevent any bond formation between the

Table I. Evaluated materials.

Product	Lot no.	Symbol	Type
Scotchbond MP (3M Dental Products, St. Paul, Minn., USA)	Primer, Lot no. 5AU Adhesive, Lot no. 5NY	SMP	Etch-and-rinse
iBond Gluma Inside (Heraeus Kulzer, Hannau, Germany)	Lot no. 010065	IBGI	Self-etching
Clearfil S ³ Bond (Kuraray Medical Inc., Kurashiki, Okayama, Japan)	Lot no. 00002A	CSB	Self-etching
iBond Experimental (Heraeus Kulzer, Hannau, Germany)	Lot no. 000001	IEXP	Self-etching
Xeno IV (Dentsply Caulk, Milford, Del., USA)	Lot no. 050318	XIV	Self-etching
G-BOND (GC Corporation, Tokyo, Japan)	Lot no. 0501191	GB	Self-etching

adhesive and the steel mold. The outer diameter of the metal disk was 13.0 mm and it was 2.0 mm thick. The central hole served as the composite sample mold. The operators were made aware of the importance of holding the disk firmly during the entire bonding procedure to secure good bonding. After the metal disk had been properly positioned, the composite material was inserted and cured as recommended by the manufacturer of the adhesive. The same composite material (Venus, shade A2, lot no. 010118, Heraeus Kulzer) was used for all bonding materials. The light source used was an LED light source (Translux Power Blue, Heraeus Kulzer) with a light irradiance of $\sim 700\text{--}750\text{ mW/cm}^2$, measured with a Cure Rite radiometer (Model no. 644726, serial no. 7046; Dentsply Caulk, Milford, Del., USA). The light irradiance value was checked before and after each bond session.

When bonding had been completed, the metal molds were left on the bonded tooth surfaces and the bonded specimens were transferred to tap water at a temperature of 37°C . The specimens were then stored for 24 h at 37°C before they were tested.

Testing procedure

After 24 h of storage in water, the samples were attached to a shear bond strength testing device and placed in a universal testing machine (Instron Model 1125; Canton, Mass., USA). Shear testing was conducted at a cross-head speed of 0.5 mm/min until failure occurred. The shear strength was determined by dividing the load at failure with the cross-sectional area of the bonded sample. On completion of the enamel testing, the samples were refurbished and prepared for dentin bonding and testing.

Statistical evaluation

The statistical analysis was conducted using a three-way PROC GLM (SAS Institute Inc, Cary, N.C., USA), where GLM stands for General Linear Model. The three variables were products, substrates, and operators, as well as interactions of these variables. All evaluations were conducted at the 95% significance level.

Results

The results of the GLM analysis (given in Table II) indicate that the most important variable was the product used. Of the six tested products, SMP did best (Table III, under "Pooled ($n=72$)"). There were significant differences between the different products. SMP belonged to statistical group "A", IBGI to group "C", CSB to group "B", IEXP to group "B", XIV to group "D", and GB to groups "B" and "C".

The second most important factor was the product*substrate interaction (Tables II and III). When the dentin and enamel bond strength values were compared independently of the adhesive system used, there was no difference in bond strength (Table III, under "pooled dentin ($n=216$)" and "pooled enamel ($n=216$)"). These results, however, need to be compared to the individual dentin and enamel values of the different products given in Table III. Comparison reveals that SMP and XIV performed better on enamel than on dentin, while IBGI and IEXP bonded better to dentin. CSB and GB had basically the same bond strength to dentin and enamel.

Comparing the different operator groups (Figure 1) revealed that the resident/fellow group did better than the experienced B group. On comparing the results generated by the different operators, it became obvious ($p<0.0001$) that bond results are strongly affected by the operator (Figure 1). Operator A3 had the significantly highest bond strength value, while operators B1 and B3 had the lowest.

By looking at the interactions between operator*product (Table II), some other interesting results are revealed (Figure 2). According to Figure 2, all three B operators and one of the A operators (A3) succeeded best with SMP. Another trend was that XIV performed worst among all operators except operator A2.

The interaction becomes even more complex when we consider interactions between product*substrate*operator (Table IV). However, the tendency that SMP and XIV performed better on enamel than dentin and that IBGI and IEXP performed better on dentin becomes even more obvious.

Table II. Results generated by PROC GLM.

Source	DF	Type I SS	Mean square	F value	Pr > F
PROD	5	6299.701435	1259.940287	27.24	<.0001
SUB	1	19.182566	19.182566	0.41	0.5200
OPERATOR	5	2044.887931	408.977586	8.84	<0.001
PROD*SUB	5	2415.623685	483.124737	10.44	<.0001
PROD*OPERATOR	25	2820.349271	112.813971	2.44	0.0002
SUB*OPERATOR	5	187.589477	37.517895	0.81	0.5423
PROD*SUB*OPERATOR	25	1075.781196	43.031248	0.93	0.5631

Table III. Mean bond strength values with their standard deviations of the different products. The values are the means for all six operators and are expressed in MPa.

Adhesive	Dentin (<i>n</i> = 36)	Enamel (<i>n</i> = 36)	Pooled enamel and dentin (<i>n</i> = 72)	Pooled dentin (<i>n</i> = 216)	Pooled enamel (<i>n</i> = 216)
SMP	20.4 (11.3)	23.2 (12.2)	21.8 (11.8)	—	—
IBGI	14.4 (5.3)	11.2 (3.9)	12.8 (4.9)	—	—
CSB	15.5 (7.0)	15.4 (8.0)	15.4 (7.5)	—	—
IEXP	18.4 (6.7)	11.9 (4.7)	15.0 (6.6)	—	—
XIV	4.6 (5.3)	13.3 (7.0)	9.0 (7.6)	—	—
GB	14.0 (6.9)	14.9 (5.0)	14.4 (6.0)	—	—
All (pooled)	—	—	—	14.6 (8.8)	15.0 (8.3)

The shear bond test used in this study, in contrast to many other shear bond tests previously used in our laboratory [18], resulted in so-called “adhesive” mode failures only. It is important to report this finding because other shear bond tests often result in cohesive failures in the tooth substrate.

Discussion

As can be deduced from the results (presented in Tables III and IV and Figures 1 and 2), every group is associated with a large standard deviation, i.e. deviations primarily due to failures occurring at low stress levels, and in some cases (particularly product XIV) even to failures that occurred before or during insertion of the specimens into the testing machine. The large standard deviations are typical of strength tests of dental adhesives and can be attributed to differences in substrate [5] and to the fact that the bonding procedures include many details/steps [10]

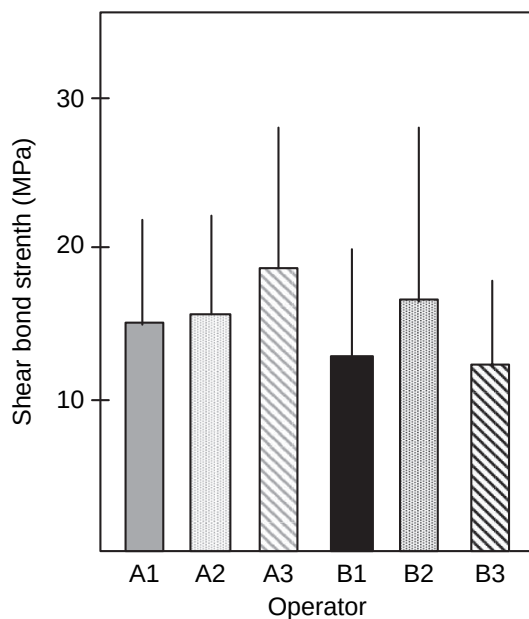


Figure 1. Bars showing mean values and standard deviations for the six operators. Each bar (*n* = 72) represents the combined mean shear bond strength of the six bonding agents when used on enamel (6 specimens per material) or dentin (6 specimens per material) by a particular operator. Operators with prefix “A” are the residents/fellow group and those with “B” the experienced clinicians.

which might not have been followed in exactly the same way by the different operators. Even though each operator tries to standardize the bonding procedures as much as possible, minor variations in different bonding steps can influence the number of defects introduced in the adhesive joint during the bonding procedure. Such defects will act as local stress concentrators, and, when a critical stress level is reached in the defect region, the flaw will grow and cause a failure [19,20]. Thus, the localized stress level causing a flaw to grow could be significantly higher than the averaged stress levels [19,20] reported in most dental research articles. Considering that aspect, it seems as if traditional shear and tensile strength tests used in dentistry to study adhesion may not be the ideal way to study adhesion [6]. In fact, such a difference between true localized stress level at a flaw tip and an averaged strength value as those presented in this study, as well as in others, may explain why, in two recent meta-analyses, no reliable correlation between *in vitro* bond strength tests, laboratory marginal test and clinical performance was found [21,22]. In a recent publication [23], the micro-tensile bond strength of G-Bond was found to be 4–6 times higher than the shear bond strength values reported for the same product in this study. Even though micro-tensile and shear strength values are compared in this example, the differences are remarkably high. Such large differences may explain why no reliable correlations could be found between *in vitro* bond strength tests, laboratory marginal tests and clinical performance [21,22].

Despite the large standard deviations, SMP clearly outperformed the five self-etching bonding agents (Tables II and III), thus supporting the findings of others [10,12]. The superiority of the etch-and-rinse bonding agent may be related to differences in etching and resin infiltration mechanisms among the adhesives. In the case of the etch-and-rinse bonding agent, a separate etching procedure is conducted followed by water rinse and removal of precipitates that might have formed during etching. After the etching and rinsing procedure, a fairly neutral monomer is used as a primer to infiltrate the etched and somewhat moist surface. Because of these conditions, diffusion of the primer is facilitated and enhances micro mechanical retention. In the

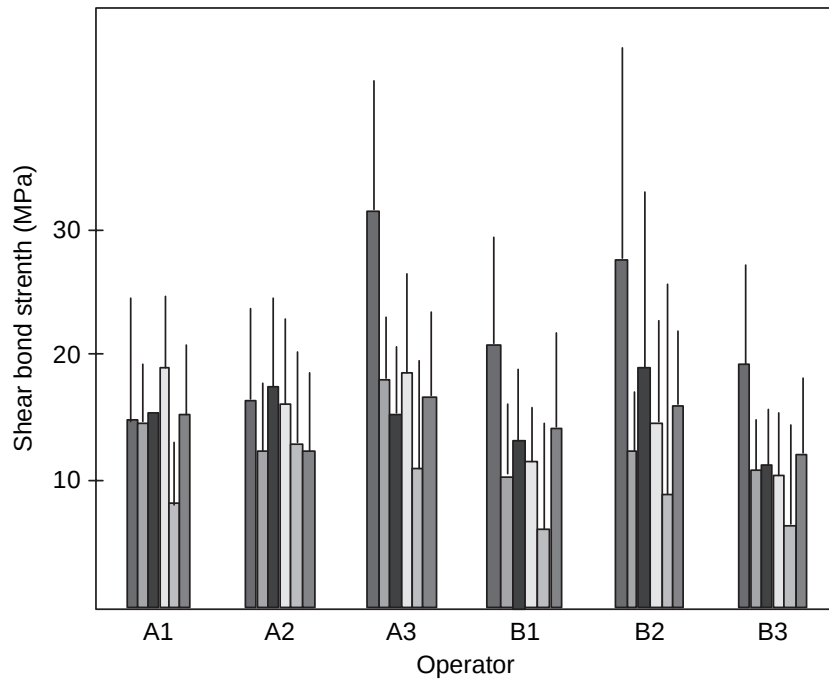


Figure 2. Bars showing mean values and standard deviations expressed in MPa for each operator and product. Each bar ($n = 12$) represents pooled enamel and dentin bond strength values for the operator. The six bars within each operator are, from left to right, SMP, IBGI, CSB, IEXP, XIV, and GB.

case of the self-etching bonding agents used in this study, the self-etching resin systems are placed on dry tooth surfaces. When the acidic monomer starts etching the surface, the protons of the acidic monomer react with the OH groups of the hydroxyapatite, and the remaining part of the acidic monomer becomes negatively charged. These negatively charged chain sites are likely to react with positively charged sites in their nearest neighborhood, which may prevent the now negatively charged molecules from diffusing deeper into the tooth structure. At the same time, these negative charge sites may also form a bond to calcium ions present in the tooth surface [11]. By increasing the molecular weight of the acidic molecule and introducing more acidic groups along the backbone structure of the molecule, more molecular/tooth bonds form per molecule. At the same time, by increasing the molecular weight, diffusion of these molecules may decrease as a result of strong early surface interactions with positive surface bond sites.

The one-bottle self-etching adhesives that performed best (CSB, IEXP, and GB) all required the use of high air pressure. According to the instruction, after 20 s of undisturbed etching, CSB was blown with high-pressure oil-free air for more than 5 s while spreading the bond layer thinly. IEXP, on the other hand, has to be placed and agitated for 20 s before the solvent is evaporated. The evaporation step should begin with a gentle air-stream and end with a strong one (could last 10 s or longer). The third of these adhesives, GB, should be left for 10 s undisturbed, and at this stage dried thoroughly for

5 s in oil-free air under maximum pressure. Whether the good performance of these three adhesives is mainly related to the air pressure or to the composition of the adhesives requires further investigation. It should be noted that other research articles have pointed in the same direction; namely, that removal of the solvent may be one key factor when it comes to the performance of an adhesive [14–17]. On the other hand, it is also important to consider that oxygen present in the air causes oxygen inhibition [24]. Too intense usage of air thinning could therefore drive the resin towards a thin and poorly cured film. Deviations from a happy balance between solvent removal and the formation of too thin an oxygen inhibited film could explain why bond results tend to differ even for the same operator and product [14].

In contrast to our previous study [12], there was no significant difference in bond strength to dentin compared to bond strength to enamel (Table III) – a finding that relates to the strong interaction between substrate and products. Products SMP and XIV performed better on enamel than on dentin. In the case of XIV, dentin bonding was so poor (Table IV) in some cases that the overall average value for dentin bonding suffered substantially, explaining why dentin bonding did not prove to be higher in this study in contrast to our previous study.

Of the different operator groups, the resident/fellow group performed better than the more experienced group, except in their use of SMP. A possible explanation could simply be that group A was

Table IV. Individual performance of the six operators for the different products when adhesives were used on dentin and enamel. The mean values and their standard deviations are expressed in MPa.

Operator	SMP			IBGI			CSB			IEXP			XIV			GB		
	Dentin	Enamel		Dentin	Enamel		Dentin	Enamel		Dentin	Enamel		Dentin	Enamel		Dentin	Enamel	
A1	14.7 (11.1)	14.0 (9.1)		15.9 (5.1)	12.0 (1.8)		14.8 (7.6)	15.7 (2.3)		20.6 (5.1)	18.1 (4.4)		0.6 (0.8)	14.9 (6.6)		13.2 (5.4)	17.1 (4.7)	
A2	16.2 (7.9)	17.4 (7.1)		12.9 (5.0)	11.0 (5.4)		18.3 (9.3)	17.6 (4.8)		19.8 (8.0)	13.4 (4.0)		8.3 (5.7)	16.8 (8.5)		7.3 (4.3)	16.8 (3.8)	
A3	27.8 (8.7)	35.5 (10.5)		19.7 (5.4)	15.8 (1.4)		15.6 (5.2)	15.6 (4.6)		24.1 (5.5)	12.5 (4.3)		5.5 (5.6)	16.3 (8.2)		16.9 (7.5)	16.6 (4.8)	
B1	22.1 (9.8)	20.1 (7.4)		11.5 (5.8)	9.0 (4.9)		13.1 (5.7)	13.5 (5.1)		12.6 (4.1)	11.1 (1.7)		1.8 (1.7)	12.1 (5.6)		14.2 (8.7)	14.3 (5.1)	
B2	24.8 (17.8)	30.3 (16.8)		14.5 (4.0)	9.5 (2.0)		18.7 (9.7)	19.7 (16.8)		20.1 (6.6)	8.2 (1.8)		8.3 (7.8)	10.0 (7.8)		17.4 (7.4)	14.7 (4.2)	
B3	16.9 (7.5)	21.9 (7.4)		12.1 (3.9)	9.7 (2.4)		12.2 (2.1)	10.0 (5.0)		12.9 (2.9)	8.5 (3.8)		3.3 (2.6)	9.8 (1.3)		15.2 (4.0)	9.6 (4.6)	

less used to etch-and-rinse adhesives than group B was, while group A was at least as experienced with self-etching products as group B. That explanation is in part supported by the good performance of operator A3, who happened to be the one of the three A group participants who was most experienced with etch-and-rinse adhesives.

There were significant differences among operators (Figure 1). The variables that caused those differences are not known at present, but they could be attributed to variations in water rinse pressure during removal of the acid gel used for the etch-and-rinse adhesive, air bubble incorporation during adhesive application, differences in air drying, light cure angles, or composite insertion techniques. For example, it is important first to disperse the acidic primer and the solvent and after completed surface interaction to remove the solvent. Differences in air pressure and air drying can result in differences in solvent removal. There is also a risk that too much solvent removal achieved by too long air drying results in oxygen inhibition. Figure 2 and Table IV show that there is a clear interaction among operator, bonding agent and substrate, making it impossible to identify a universal bonding agent. At the present time we do not know why such interactions occur, only that it is important to decrease the technique variability of the bonding procedure. Whether the unpredictable link is the bonding agent or the insertion/adaptation procedure needs to be investigated further.

From the results presented here and in our previous article [12], in which we compared an etch-and-rinse adhesive with two self-etching adhesives, there is no evidence supporting claims that the self-etching bonding agents perform better than the etch-and-rinse adhesive. On the contrary, the tested etch-and-rinse adhesive is likely to perform better than most self-etching adhesives available at the present time, at least *in vitro*. However, having said this, newer developments of self-etching adhesives have resulted in improvements within the self-etching group (compare IBGI with IEXP). IEXP, like IBGI, contained glutaraldehyde and was similar to the original iBond with only minor modifications compared to original iBond. This is important to note, because IEXP is not the same product as the iBond Self Etch that is now available on the market.

Our results clearly show that the individual operator affects the results and that there are significant interactions between products, substrates, and operators. Consequently, it is impossible to recommend one particular product over another as being the most efficacious.

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