©Operative Dentistry, 2007, **32-2**, 133-137

Increases in Dentin-bond Strength If Doubling Application Time of an Acetone-containing One-step Adhesive

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Clinical Relevance

When treating dentin surfaces with Futurabond one-step self-etch bonding agent, in order to obtain higher microtensile bond strength, doubling the application time of the adhesive should be considered.

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DOI: 10.2341/06-32

SUMMARY

This study investigated the microtensile bond strength (μTBS) of a one-step self-etching adhesive to human dentin and bovine enamel following different bonding treatments. Occlusal portions of human molars and labial surfaces of bovine incisors were ground flat to provide uniform dentin and enamel surfaces, respectively. Futurabond was used following five different protocols: 1) according to the manufacturer's directions, 2) acid etched with 36% phosphoric acid (H_3PO_4) for 15 seconds, 3) 10% sodium hypochlorite (NaOCl) treated for two minutes after H_3PO_4-etching, 4) doubling the application time of the adhesive and 5) doubling the number of adhesive coats. Composite build-ups (6 mm in height) were constructed incrementally with Arabesk resin composite. The specimens were stored in 100% humidity for 24 hours at 37°C and sectioned into beams of 1.0 mm² cross-sectional area. Each beam was tested in tension in an Instron machine at 0.5 mm/minute, and mean μTBS data (MPa) were analyzed by one-way

ANOVA and post-hoc multiple comparisons tests (α=0.05). Doubling the application time of Futurabond attained the highest μTBS to dentin; whereas, no differences among all bonding application parameters evaluated could be detected when the adhesive was applied to enamel.

INTRODUCTION

Bonding to dentin represents a challenge to clinical scientists, as the substrate is an intrinsically wet organic tissue, penetrated by tubular structures that communicate with the pulp.[1] The dynamic nature of dentin as a bonding substrate is responsible for marginal leakage and inconsistent bond strengths, which occur with all resin-based adhesives.[2] Direct application of enamel etching technology to dentin was successful due to the chemical and morphological differences between the two substrates. The adhesion of resin composite materials to enamel has become a routine and reliable aspect in restorative treatment[3] since Buonocore proposed the use of phosphoric acid for differential etching of the enamel substrate.[4]

The application of self-etch adhesives[5] onto dentin and enamel has been a controversial issue.[6-8] In an effort to make these adhesives more user-friendly, there is a trend towards increasing the acidic nature of one-step self-etch adhesives.[8-10] For these adhesives to become more acidic, the formulations have been rendered extremely hydrophilic, thus making the hybrid layers more permeable and liable to water sorption.[11-13] Thus, the benefit of saving time with these simplified adhesives is realized at the expense of compromising bond integrity.[7,11,14] More stable bonds may be formed if primers and resins penetrate less deeply, but more uniformly, through dental substrates.[15]

The diffusion of acidic resin monomers through the smear layer is slow.[16] Smear layers reinforced by impregnated resin may be too weak to provide strong, durable mechanical properties.[6] The mineralized components of the smear layer are efficient buffers,[1] making the pH of acidic monomers too high to demineralize the underlying dentin.[10,15] This often necessitates a separate conditioning step[17] to improve the infiltration of self-etch adhesives through partially demineralized dentin.[18] Treatment of the demineralized collagen matrix with a proteolytic agent, such as sodium hypochlorite (NaOCl), may have an additional beneficial effect to these adhesives, facilitating infiltration[19] of the acidic resin monomers into the dentin substrate.[20-21] Alternative bonding strategies, such as multiple applications[22] or increased substrate contact time of the acidic primers,[23] may also be helpful in achieving a better link between the adhesives and dental substrates.

This study examined the effect of different bonding application parameters on the microtensile bond strength (μTBS) of a one-step self-etch bonding agent. The null hypothesis tested was that there are no differences in the use of alternative bonding strategies with Futurabond, when compared with the original manufacturer's instructions on the dentin/enamel microtensile bond strength.

METHODS AND MATERIALS

Ten caries-free extracted human third molars and 10 freshly extracted bovine incisors, stored in isotonic saline solution containing a few crystals of thymol at 4°C for no longer than three months, were used. Human specimens were obtained, with a protocol that was reviewed and approved by the Ethics Committee of the University of Granada. Occlusal portions of the molars and the labial surfaces of incisors were ground flat in a mechanical grinder with 180-grit SiC papers under running water, resulting in uniform smear-layer covered dentin and enamel surfaces.[24]

Table 1 displays the mode of application, components and manufacturer of the tested one-step self-etching system Futurabond (Voco GmbH, Cuxhaven, Germany). The adhesive was applied following five different bonding procedures: 1) according to the manufacturer's directions (Table 1), 2) after acid etching with 36% phosphoric acid (H_3PO_4) (Conditioner 36, Dentsply/DeTrey, Konstanz, Germany) for 15 seconds and water rinsing for 30 seconds, 3) H_3PO_4-etching, water rinsing for 30 seconds and 10% sodium hypochlorite (NaOCl) (Panreac Química SA, Barcelona, Spain) application under agitation for two minutes, 4) doubling the application time of the adhesive's first coat (60 seconds), 5) doubling the number of coats of adhesive before polymerization. Regarding bonding treatments 2 and 3, the surfaces were rinsed for 30 seconds with distilled water and gently air-dried before adhesive application.

Table 1: *Adhesive System Used for Bonding in the Experimental Groups*

Material and Manufacturer	Composition	Mode/Steps of Application
Futurabond (Voco GmbH, Cuxhaven, Germany)	Bis-GMA, BHT, acetone, diurethanemethacrylate, HEMA, organic acids.	Mix equal amount of Liquid A and Liquid B for 5 seconds. Apply adhesive to tooth substrates (scrubbing) for 30 seconds. Air blow gently for 5 seconds. Light-activate for 20 seconds. Apply second layer of the adhesive. Air blow at least for 5 seconds. Leave the second layer uncured.

Abbreviations: Bis-GMA: bis-phenol A diglycidylmethacrylate; BHT= butylated hydroxy toluene; HEMA (2-hydroxyethyl methacrylate).

Resin composite build-ups, each 6 mm in height, were constructed incrementally (1.5 mm) with Arabesk (Voco GmbH, Cuxhaven, Germany) resin composite. Each layer of the composite was light activated for 40 seconds with a Translux EC halogen light-curing unit (Demetron Research Corporation, Danbury, CT, USA). Light intensity output was constantly monitored with a Demetron Curing Radiometer (Model 100, Kerr Demetron, Danbury, CT, USA) to be at least 600 mW/cm^2 at a temperature of 21°C and relative humidity of 60%.

After distilled water storage for 24 hours at 37°C, the bonded teeth were vertically sectioned into serial slabs and further into beams with cross-sectional areas of 1 mm^2, following the method described by Shono and others.[25] This procedure created a total of 30 beams per group. The beams were attached to a modified Bencor Multi-T testing apparatus (Danville Engineering Co, Danville, CA, USA) with a cyanoacrylate adhesive (Zapit, Dental Ventures of America Inc, Corona, CA, USA) and stressed to failure in tension using an Instron testing machine (Instron Inc, Canton, MA, USA) at a crosshead speed of 0.5 mm/minute. The fractured beams were carefully removed from the apparatus, and the cross-sectional area at the site of failure was measured to the nearest 0.01 mm with a pair of digital calipers (Sylvae Ultra-Call Ii, Fowler Inc, Newton, MA, USA). The fractured specimens were examined with a stereomicroscope (Olympus/DeTrey, Konstanz, Germany) at 40x magnification to determine the mode of failure. Failure modes were classified as adhesive or mixed.

The pH of the evaluated self-etch adhesive was assessed using pH indicator strips (Merck KgaA, Darmstadt, Germany). The self-etch adhesive solution was dispensed in a Dappen dish, and a strip was dipped into the adhesive until there was no additional color change. The color evaluation was carried out using the proprietary pH scale.

Data from human dentin and bovine enamel were evaluated separately. The bond strength values were calculated in MPa and analyzed by one-way ANOVA to examine the effect of the variable bonding treatment. The Student Newman Keuls multiple comparisons test was performed at α=0.05.

RESULTS

The mean μTBS values (MPa) obtained on bovine enamel and human dentin are shown in Table 2. Statistical analysis of the μTBS data revealed that only bond strength to dentin was affected by the bonding procedure (dentin: F=25.59, p<0.001; enamel: F=2.01, p=0.12).

When bonded to dentin, doubling the application time attained the highest bond strength; whereas, comparisons among former bonding procedures (as manufacturers, H_3PO_4, H_3PO_4 + NaOCl and doubling the number of adhesive coats) showed no significant differences. With regard to enamel, differences among bonding application procedures could not be detected. Futurabond recorded an acidic pH (1.0).

Table 2 also summarizes the percentage failure modes of the debonded specimens according to substrate and bonding application procedures. No cohesive failures were observed. Most of the encountered failures were adhesive, which is normally associated with lower bond strength values.

DISCUSSION

Trends toward simplification of bonding procedures have led to the introduction of self-etch adhesives.[2] However, research has demonstrated that these adhesive systems do not improve bonding effectiveness to both enamel and dentin[7] in spite of their purported reduction in technique sensitivity.[11,14,26] To offset these limitations, alterations on self-etch bonding protocols that increase resin-dentin bond quality are required.[19-20,22] In this study, even though increasing the diffusion time of adhesive yielded higher μTBS values to dentin, bond strength to enamel was not influenced by the several bonding application procedures evaluated. Therefore, the hypothesis that using different bonding techniques with a one-step self-etch system improves its bonding effectiveness should be accepted with respect to dentin bond strength but rejected for enamel evaluation.

One-step adhesives contain highly hydrophilic monomers and lack additional solvent-free hydrophobic resin for coupling to the primed dentin, which may decrease the stability of hybridized dentin.[6,9-11] When aggressive versions of these adhesives (pH=1) are applied, water transudation from the underlying dentin[10,21] occurs, resulting in dilution of the adhesive

Table 2: *Mean Microtensile Bond Strength (MPa) and Standard Deviation (SD) Obtained for Each Tested Group (n=30) and Distribution of Failure Modes (%)*

Substrate	Bonding Approach														
	Self-etch	A	M	H_3PO_4	A	M	H_3PO_4 + NaOCl	A	M	Double Time	A	M	Double Layer	A	M
Dentin	16.3 (9.7)c	81	19	18.4 (11.0)bc	58	42	17.3 (8.8)c	75	25	32.4 (8.2)a	37	63	20.5 (8.0)bc	51	49
Enamel	22.4 (8.2)A	59	41	25.6 (8.2)A	53	47	21.0 (8.9)A	52	48	19.7 (8.1)A	62	38	22.7 (7.9)A	60	40

(A: adhesive; M: mixed).
**Within the same row, identical letters indicate no significant differences (p<0.05).*

solution.[10] There is a reduction in the tendency of hydrophilic and hydrophobic monomers to form polymer blends[27] at the dentin interface, which may be caused by: 1) phase separation of these monomers within the adhesive,[6,9] and 2) differences in molecular weight or affinity to the tooth substrates of these monomers. Diffusion of bis-phenol A diglycidyl-methacrylate (Bis-GMA) within the layer of partially demineralized dentin is restricted due to its high molecular weight. Water entrapment within the hydroxyethyl methacrylate (HEMA)-rich hybrid layer[12] will also adversely affect Bis-GMA polymerization and its copolymerization with HEMA, resulting in bond strength reduction after resin composite placement.

Futurabond is a one-step self-etching adhesive consisting of organic acids combined with hydrophobic monomers and HEMA, all dissolved in acetone. The organic acids act as an etchant, while HEMA may behave as a priming agent.[29] However, Futurabond does not contain water, which is required to dissociate the weak acids into ionized forms for permeation of the smear layer and demineralization of the underlying intact tooth substrates.[10,29] Moreover, the presence of high concentrations of water-soluble ionic monomers from self-etch adhesive induces an osmotic water flux from deep dentin.[30] Thus, prolonging the time between adhesive application and drying may result in more optimal water permeation within the adhesive, contributing to a more complete dissociation of the acid functional groups[31] and enhancement of the resin monomer infiltration.[32] Both residual water and acetone have to be completely eliminated from dentin before resin polymerization.[33] This is crucial with the use of simplified adhesives.[34] Prolonged air-drying may also facilitate solvent evaporation.[35] However, excessive air-drying may over-thin the adhesive layer, resulting in inhibition of the polymerization reaction by oxygen to form peroxy radicals.[36-37] Acetone is a volatile solvent[38] for many methacrylates.[39] Rapid evaporation of acetone from the adhesive blend promotes a decrease in solvent-resin affinity and formation of a monomer rich-phase, which may promote cross-linking.[40] With acetone evaporation exceeding that of water,[39] the aqueous fraction accumulated in adhesive film tends to increase. The recommended application time may not be sufficient to allow water to be removed from the partially polymerized adhesive layer.[35]

The results obtained with Futurabond in this study do not support the hypothesis that the application of additional adhesive coats is required to ensure a sufficiently thick resin film that increases bond strength.[41-42] This confirms the work of Nakabayashi and Saimi,[16] who reported that hybrid layers do not have to be thick to be strong, but they have to contain enough resinous material to produce hybrid layers that are devoid of incompletely infiltrated collagen fibrils.[23,43-44]

CONCLUSIONS

Within the limits of this study, when dentin and enamel surfaces are treated with an acetone-containing one-step self-etch bonding agent, doubling the adhesive application time should be considered in order to increase dentin bond strength. Further long-term investigations and clinical trials are desirable in order to improve adhesive dentistry not only by producing faster, more user-friendly adhesives, but also toward enhancing the quality and long-lasting durability of resin bonds created in dental substrates.

Acknowledgements

This research project was supported by grants CICYT/FEDER MAT2004-06872-C03-02 and RED CYTED VIII. J.

(Received 21 February 2006)

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