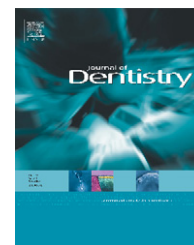


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Resistance of ten contemporary adhesives to resin–dentine bond degradation

Raquel Osorio^a, Jatyr Pisani-Proenca^a, Maria Carolina G. Erhardt^a,
Estrella Osorio^a, Fátima S. Aguilera^a, Franklin R. Tay^b, Manuel Toledano^{a,*}

^aDepartment of Dental Materials, Campus de Cartuja s/n, School of Dentistry, University of Granada, Granada, Spain

^bDepartment of Endodontics, School of Dentistry, Medical College of Georgia, Augusta, USA

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ABSTRACT

Objectives: To evaluate resin–dentine bond degradation after 1 year of water storage.

Methods: Human dentine surfaces were bonded with two etch-and-rinse self-priming adhesives (Single Bond/SB and Prime & Bond NT/PBNT), three 2-step self-etching adhesives (Clearfil SE Bond/SEB, Resulcin Aqua Prime/RES and Non-Rinse Conditioner with Prime & Bond NT/NRC-PBNT), and five 1-step self-etching adhesives (Etch & Prime 3.0/EP, Prompt L-Pop/PLP, Solist/SOL, Futurabond/FUT and AQ Bond/AQ). Adhesives were applied according to manufacturers' instructions. Composite build-ups were constructed and the bonded teeth were stored (24 h, 6 months, 1 year) in distilled water at 37 °C. After storage, the intact teeth were sectioned into beams and all specimens were tested for microtensile bond strengths (MTBS). ANOVA and multiple comparisons tests were applied at $\alpha = 0.05$. Fractographic analysis of debonded beams was performed using scanning electron microscopy.

Results: SB, PBNT and SEB attained the highest MTBS, regardless of the storage period. A significant decrease in MTBS was observed after 6 months for SOL. After 12 months the only groups that did not reduce bond strength were SB and SEB. Bonded specimens in NRC-PBNT, RES and FUT produced pre-testing failures after 12 months, and MTBS could not be measured.

Conclusions: The resistance of resin–dentine bonds to degradation is material-dependent. When the enamel–resin interface is preserved, the etch-and-rinse adhesives and the mild 2-step self-etch adhesive SEB exhibited the best resin–dentine bond durability. Those tested self-etching adhesives having a $\text{pH} \leq 1$ and using water or acetone as solvent attained catastrophic bond failure after 1 year of water storage.

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1. Introduction

The oral cavity is subject to temperature changes,¹ chewing loads² and chemical attacks by acids and enzymes³ that severely challenge the durability of resin–dentine bonds. Biodegradation of the collagen matrix and/or hydrophilic resin components within the hybrid layer is related to: (1) incomplete penetration/infiltration of resin into the dentine

substrate⁴; (2) heterogeneous distribution of resin monomers through the interdiffusion zone⁵; (3) suboptimal polymerization in the presence of water^{6,7}; (4) alterations of the organic matrix during preparatory procedures⁸; (5) hydrolysis of polymeric components^{9,10} or unprotected collagen.^{11,12}

Dentine bond strengths have been shown to decrease in vivo¹³ and after water storage.^{4,14} Some in vivo reports have suggested that degradation of the resin–dentine interface

* Corresponding author at: Av. de las Fuerzas Armadas 1, 1B, 18014 Granada, Spain. Tel.: +34 958243788; fax: +34 958240908.

E-mail address: toledano@ugr.es (M. Toledano).

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Table 1 – Description of tested adhesive systems

Adhesive category	Components	Principal ingredients (according to manufacturers)	Mode/steps of application
2-Step self-etch	Clearfil SE bond (pH = 2.1)		
	Primer	10-MDP; HEMA; hydrophilic dimethacrylate; dl-camphorquinone; N,N-diethanol-p-touidine; water	Apply primer 20 s. Mild air stream. Apply bond. Gentle air stream. Light cure 10 s
	Bond	10-MDP; bis-GMA; HEMA; hydrophobic dimethacrylate; di-camphorquinone; N,N-diethanol-p-toluidine; silanated colloidal silica	
	Resulcin AquaPrime + Monobond (pH = 1)		
	AquaPrime	2-Methacryloyloxyethyl-dihydrogen-phosphate	Mix AquaPrime with water (1:1). Scrub into dentine surface 30 s. Gently air dry. Apply Monobond. Air blow. Light cure 20 s
	Monobond	Bis-GMA, TEGDMA, polymethacryl-oligomaleic acid	
	NRC, non rinse conditioner (pH = 1)		
1-Step self-etch	NRC	Organic acids/monomers in aqueous solution	Apply NRC. Leave undisturbed 20 s. Remove excess by blowing gently with air syringe. Do not desiccate the dentine
	Prime & Bond NT	PENTA, UDMA resin, Resin R5-62-1, T-resin, D-resin, nanofiller, initiators, stabilizer, cetylamine hydrofluoride, acetone	Apply ample amounts of Prime & Bond NT. Leave the surface undisturbed 20 s. Air blow at least 5 s. Light cure 10 s
	Etch & Prime 3.0 (pH = 0.76)		
	Universal	HEMA, water, ethanol, stabilizer	Mix Universal and Catalyst.
	Catalyst	HEMA, initiators, stabilizers, tetra-methacryloyloxyethylpyrophosphate	Apply 30 s. Air blow gently. Light cure 10 s. Repeat previously mentioned steps
	Prompt L Pop (pH = 0.7)		
	Red blister	Bis-GMA, methacrylated phosphoric esters, CQ, stabilizers	Pack activating. Scrub first coat 15 s. Gently air dry. Light-cure 10 s. Second coat application (scrubbing) 15 s. Gently air dry. Light-cure 10 s
	Yellow blister	Water, zinc-fluoride complex, stabilizers	
	Solist (pH = 1.8)		
	–	HEMA, TEGDMA, acetone, catalyst/stabilizer, elastomers	Apply adhesive 30 s. Air blow gently. Light cure 10 s. Apply a second coat 5 s. Air blow gently. Light cure 10 s
	Futurabond (pH = 1)		
	–	Bis-GMA, diurethanedimethacrylate, hydroxyethylmethacrylate, BHT, acetone, organic acids	Apply adhesive (scrubbing) 30 s. Air blow. Light cure 20 s. Apply adhesive. Air blow gently. Leave second coat uncured
	AQ Bond (pH = 2.1)		
	Base liquid	Water, acetone, 4-META, HEMA, MMA, UDMA, photoinitiator	Soak AQ Sponge on Base liquid; saturate dentine surface with ample amounts of the adhesive. Air blow gently 5 s. Apply second coat of the adhesive. Air blow 5 s. Light cure 10 s
Etch-and-rinse	AQ Sponge	Polyurethane foam, Initiator (p-TSNa).	
	Prime & Bond NT (pH = 2.7)		
	–	PENTA, UDMA resin, Resin R5-62-1, T-resin, D-resin, nanofiller, initiators, stabilizer, cetylamine hydrofluoride, acetone.	H ₃ PO ₄ etching 15 s. Rinse with water spray 15 s. Leave a moist surface with a soft blow of air. Saturate the surface with ample amounts of the adhesive, reapply if necessary. Leave the surface undisturbed 20 s. Air blow gently 5 s. Light cure 10 s
	Single Bond (pH = 4.3)		
	–	HEMA, water, ethanol, amines, Bis-GMA, methacrylate-functional, copolymer of polyacrylic and polyitaconic acids, dimethacrylates.	H ₃ PO ₄ conditioning 15 s. Rinse with water spray 10 s, leaving tooth moist. Apply two consecutive coats of the adhesive with a fully saturated brush tip. Dry gently 5 s. Light cure 10 s

Abbreviations: 10-MDP, 10-methacryloxydecyl dihydrogen phosphate; HEMA, 2-hydroxyethyl methacrylate; Bis-GMA, bis-phenol A diglycidylmethacrylate; TEGDMA, triethylene glycol-dimethacrylate; MAC-10, methacryloyloxyalkyl acid phosphate; CQ, camphoroquinone; BHT, butylated hydroxy toluene; 4-META, 4-methacryloxyethyltrimellitic acid anhydride; MMA, methyl methacrylate; UDMA, urethane dimethacrylate; p-TSNa, sodium p-toluenesulfinate; PENTA, penta-acrylate ester; H₃PO₄, phosphoric acid.

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