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Influence of degradation conditions on dentin bonding durability of three universal adhesives

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ABSTRACT

Objectives: This study aims to determine dentin bonding durability of universal adhesives using shear bond strength (SBS) tests under various degradation conditions.
Methods: G-Premio Bond (GP, GC), Scotchbond Universal (SU, 3 M ESPE) and All Bond Universal (AB, Bisco) were compared with conventional two-step self-etch adhesive Clearfil SE Bond (SE, Kuraray Noritake Dental). Bonded specimens were divided into three groups of ten, and SBSs with bovine dentin were determined after the following treatments: 1) Storage in distilled water at 37 °C for 24 h followed by 3000, 10,000, 20,000 or 30,000 thermal cycles (TC group), 2) Storage in distilled water at 37 °C for 3 months, 6 months or 1 year (water storage, WS group) and 3) Storage in distilled water at 37 °C for 24 h (control).
Results: SE bonded specimens showed significantly higher SBSs than universal adhesives, regardless of TC or storage periods, although AB specimens showed significantly increased SBSs after 30,000 thermal cycles. In comparisons of universal adhesives under control and degradation conditions, SBS was only reduced in SU after 1 year of WS.
Conclusion: Following exposure of various adhesive systems to degradation conditions of thermal cycling and long term storage, SBS values of adhesive systems varied primarily with degradation period.
Clinical significance: Although universal adhesives have lower SBSs than the two-step self-etch adhesive SE, the present data indicate that the dentin bonding durability of universal adhesives in self-etch mode is sufficient for clinical use.

1. Introduction

In order to assure the long-term stability of resin composite restorations, fillers, resin matrices, initiators of resin composite formation and various other adhesive technologies have been developed [1,2]. However, degradation of the bond remains inevitable due to biofilm attack, hydrolytic degradation of adhesives, enzymatic degradation by matrix metalloproteinases and adhesive fatigue [3–6]. Therefore, determinations of the durability of resin composite restorations under intraoral conditions are critical for clinical application.

Bonding performances of resin restorations have been investigated in long-term clinical trials [7,8]. However, the reported procedures were time-consuming and costly, and case selection, consistency of operators and examiners and numbers of patients are difficult to standardize. In contrast, simulated oral environment testing can be used to rapidly determine relative bonding durability of materials. Specifically, *in vitro* degradation of restored teeth can be simulated in long-term water storage and thermal cycling experiments, allowing standardization of conditions before and after storage, and assessing degradation using bond strength tests allows easy comparison [8,9].

Recently, developments of single-step self-etch adhesives have led to widespread clinical use. The most recently developed self-etch adhesives are referred to as 'universal' or 'multi-mode' because they can be used with various adherent substrates, including enamel, dentin, metal alloy and ceramics. [10–15]. Moreover, universal adhesive systems can be applied following phosphoric acid pre-etching using total-etch, selective-etch and self-etch approaches. In addition, *in vitro* investigations of universal adhesives indicate that initial bonding performances to enamel and dentin without phosphoric acid pre-etching are of

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similar quality to those of single-step self-etch adhesives. Furthermore, the total-etch approach for universal adhesives increases bond performance with enamel without affecting dentin bonding [16,17]. However, limited information is available on bond durability of universal adhesives and their reliability remains unclear.

The purpose of this study was to determine the dentin bonding durability of universal adhesives following simulated *in vitro* degradation. The null hypotheses to be tested were: (1) the dentin bonding durability of universal adhesive would not differ from that of a conventional two-step self-etch adhesive; and (2) different simulated degradation methods would not influence bond strength results.

2. Materials and methods

2.1. Study materials

Adhesive materials (Table 1) included the three universal adhesives G-Premio Bond (GP, GC Corp., Tokyo, Japan), Scotchbond Universal (SU, 3M ESPE, St Paul, MN, USA) and All Bond Universal (AB, Bisco, Schaumburg, IL, USA). The conventional two-step self-etch adhesive Clearfil SE Bond (SE, Kuraray Noritake Dental, Tokyo, Japan) was used as a control. Clearfil AP-X (Kuraray Noritake Dental, Tokyo, Japan) was used as a restorative material for bonding to dentin.

2.2. Specimen preparation

Mandibular bovine incisors were extracted from 2 to 3 year old cattle and were stored frozen for up to 2 weeks for use as human teeth. As a large number of samples were necessary, bovine dentin was used instead of human dentin. Previous research shows that this has little influence on the results [18,19]. Approximately two-thirds of the apical root structure of each tooth was removed using a diamond impregnated disk with a slow-speed saw (Isomet Low Speed Saw, Buehler, Lake Bluff, IL, USA). Pulp tissues were then removed and labial surfaces were ground with wet 240-grit silicon carbide (SiC) paper (Fuji Star Type DDC, Sankyo Rikagaku Co. Ltd., Saitama, Japan) to create flat dentin surfaces. Teeth were then mounted in self-curing acrylic resin (Tray Resin II, Shofu Inc., Kyoto, Japan) so that the flattened areas were exposed, and temperature rises due to exothermic polymerization reactions of the acrylic resin were moderated by placing specimens under running tap water. Dentin bonding surfaces were next ground flat using a water coolant and a sequence of SiC papers ending with 320-grit (Fuji Star Type DDC), and the dentin surfaces were finally dried with oil-free compressed air.

2.3. Storage conditions and shear bond strength tests

The experimental protocols for the bonding procedures are shown in Table 2. Ten specimens, each taken from a different tooth, were used. In total three hundred and twenty bovine teeth were used. Adhesives were applied to dentinsurfaces in accordance with the respective manufacturer's instructions, and specimens were then clamped in an Ultradent Bonding Jig (Ultradent Products Inc., South Jordan, UT, USA) with plastic molds of 2.38-mm internal diameter and 2.0-mm height [20]. Subsequently, resin composites were placed into the mold and were light irradiated for 30 s using a visible-light curing unit (Optilux 501, sds Kerr, Danbry, CT, USA) set at a light irradiance average of 600 mW/cm^2.

Specimens were subjected to thermal cycling (TC group) or storage in distilled water at 37 °C (water storage, WS group). Bonded specimens of the TC group were stored in distilled water at 37 °C for 24 h and were then treated with 3000, 10,000, 20,000 or 30,000 thermal cycles (TCs) between 5 and 60 °C with a dwell time of 30 s. Specimens of the WS group were stored in distilled water at 37 °C for 3 months, 6 months or 1 year before the shear bond strength (SBS) tests. The storage water was changed weekly, antibiotics were not used. Control specimens were stored in distilled water at 37 °C for 24 h before the SBS tests (control group).

SBSs were determined according to ISO 29022 [20]. Briefly, specimens were loaded to failure at 1.0 mm/min using a universal testing machine (Type 5500R, Instron Corp., Canton, MA, USA), and SBS Values (MPa) obtained. After testing, bonded tooth surfaces and resin composite cylinders were observed under an optical microscope (SZH-131, Olympus Ltd., Tokyo, Japan) at a magnification of ×10, and bond failures were recorded as 1) adhesive failure, 2) cohesive failure in composite, 3) cohesive failure in dentin, or 4) mixed failure [16,17,21].

2.4. Scanning electron microscopy (SEM) observations

Interfaces between restoratives and dentin and representative fracture sites after SBS tests were observed using field-emission electron scanning microscopy (SEM, ERA-8800FE, Elionix Ltd., Tokyo, Japan). Prior to ultrastructure observations of restorative–dentin interfaces, bonded specimens were embedded in epoxy resin (Epon 812, Nisshin EM, Tokyo, Japan) and were then longitudinally sectioned using a diamond saw (Isomet Low Speed Saw). Sectioned surfaces were then polished to a high gloss using

Table 1
Materials used in this study.

Code	Adhesive (Lot No.)	Main Components	Manufacturer
SU	Scotchbond Universal (41256)	MDP, HEMA, dimethacrylate resins, Vitrebond copolymer, filler, ethanol, water, initiators, silane	3M ESPE St. Paul, MN, USA
GP	G-Premio Bond (1501221)	MDP, 4-MET, MEPS, BHT, acetone,dimethacrylate resins, initiators,water	GC Corp Tokyo, Japan
AB	All-Bond Universal (1300008503)	MDP, bis-GMA, HEMA, ethanol, water, initiators	Bisco Inc. Schaumburg, IL, USA
SE	Clearfil SE Bond (011613)	Primer: MDP, HEMA, water, initiators Bond: MDP, HEMA, bis-GMA, initiators, microfiller	Kuraray Noritake Dental Tokyo, Japan
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Resin composite			
	Clearfil AP-X (CC0043)	bis-GMA, TEGDMA,silane barium glass filler, silane silica filler, silanated colloidal silica catalysts, accelerators, CQ, pigments, others Filler Load: 84.5% weight	Kuraray Noritake Dental

MDP: 10-methacryloyloxydecyl dihydrogen phosphate, HEMA: 2-hydroxyethyl methacrylate, 4-MET: 4-methacryloxyethyl trimellitate, MEPS: methacryloyloxyalkyl thiophosphate methylmethacrylate, BHT: butylated hydroxytoluene bis-GMA: 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy) phenyl) propane, TEGDMA: triethyleneglycol dimethacrylate, CQ: *dl*-camphorquinone.