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Bonding effectiveness of experimental one-step self-etch adhesives to sound and caries-affected dentin

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ABSTRACT

Experimental one-step, self-etch adhesives containing different contents of an acidic methacrylate monomer (GDMA-P) were formulated and their effectiveness in bonding to sound dentin (SoD) or caries-affected dentin (CAD) was investigated. The CAD was obtained using a microcosm biofilm model. HEMA-based adhesives were formulated with 5, 20, or 35 wt% of GDMA-P (AD5, AD20, AD35), with pH ranging between 1.05 ± 0.05 and 1.93 ± 0.15. Shear bond strength to dentin was assessed after storage for 24 h or 6 months. Morphology of the bonded interfaces was observed using SEM. The exposed collagen area at bonded interfaces was measured using a histological staining technique. Degree of C=C conversion within the hybrid layer, measured by micro-Raman spectroscopy, indicated that AD35 had lower *in situ* C=C conversion than the other adhesives. A more evident exposed collagen zone along the base of the hybrid layer was observed in CAD samples. The hybrid layer was generally thicker in CAD. AD20 had the highest *in situ* C=C conversion and yielded stable bond strengths that were generally independent of the dentin condition. Additionally, the bonding effectiveness was dependent on adhesive acidity, type of dentin bonding substrate, and water storage time.

1. Introduction

Dental caries is one of the most common oral diseases in humans [25]. Caries lesions extending to dentin are usually associated with the placement of restorations. Under the concepts of minimally invasive dentistry, decayed dental tissue located at the inner layer of the cavity may be only partially removed [13], thus the restorative procedure would include bonding to both sound dentin (SoD) and caries-affected dentin (CAD). CAD may still remain in the cavity even when a complete removal of the caries lesion is performed. Several studies indicate that bonding to CAD is more challenging than bonding to sound dentin [2,9,37,40] since the morphological and chemical alterations in CAD [3,22] may result in unfavorable conditions for effective adhesion [14,19].

Bonding to enamel and dentin can follow two different strategies, i.e. etch-and-rinse or self-etch approaches. Depending on the strategy, the resulting bonded substrate might present different characteristics. The etch-and-rinse strategy removes the dentin smear layer completely, leaving the tubules open for resin infiltration, and generates an up to 10 μm thick layer of demineralized collagen prone to hybridization. By contrast, the self-etch strategy only modifies the smear layer, resulting in a few micrometers thick layer of partially demineralized collagen infiltrated by resin. In teeth presenting CAD, self-etch adhesives would be incorporated to the bonding substrate. Previous studies reported that etch-and-rinse adhesives performed better than self-etch adhesives applied to CAD [2,40]. However, self-etch materials are increasingly popular in dentistry, especially due to their easier application and less sensitive bonding protocol.

One-step, self-etch adhesives have the simplest application protocol amongst all dental adhesives, but also the most complex composition. In one-step systems, however, all components are mixed together, including resin monomers (acidic, hydrophilic, hydrophobic), solvents, water, and photoinitiators. These adhesives are usually very hydrophilic and subject to hydrolysis over time, thus their long-term bonding performance is often contested [4]. Previous studies [11,18] show that the concentration of acidic monomers incorporated into two-step, self-etch adhesives might influence the immediate and long-term dentin bond strengths [11,18]. However, there are still few studies investigating the impact of formulation components on the bonding performance of one-step adhesives to CAD [24]. The aim of this study was to investigate bonding effectiveness of one-step, self-etch adhesives containing different contents of acidic monomer applied to SoD and

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Table 1
Components of the experimental one-step, self-etch adhesives tested (wt%).

Reagent	AD5			AD20			AD35		
	Bottle A	Bottle B	A + B	Bottle A	Bottle B	A + B	Bottle A	Bottle B	A + B
GDMA-P	10%	–	5%	40%	–	20%	70%	–	35%
HEMA	65%	15%	40%	35%	15%	25%	5%	15%	10%
Bis-GMA	10%	50%	30%	10%	50%	30%	10%	50%	30%
Water	–	20%	10%	–	20%	10%	–	20%	10%
Ethanol	15%	15%	15%	15%	15%	15%	15%	15%	15%
pH (mean ± SD)	1.93 ± 0.15^{A}			1.25 ± 0.04^{B}			1.05 ± 0.05^{C}		

Distinct letters indicate statistically significant differences in pH between the adhesives ($p < 0.05$).
Labels AD5, AD20, and AD35 refer to the concentrations of GDMA = P after mixing bottles A + B.

CAD. The study hypothesis was that CAD would be a more challenging bonding substrate than SoD irrespective of the acidic methacrylate concentration in the adhesive.

2. Materials and methods

2.1. Preparation of dentin discs

Bovine incisors were cleaned and stored in 0.5% chloramine-T solution for seven days. Standardized enamel-dentin discs with 2 mm in thickness and 6 mm in diameter were cut from the buccal surfaces of the teeth using a water-cooled trephine drill. The discs were wet-ground using 80-grit SiC abrasive papers until superficial dentin was visually exposed, then wet-polished with 600-grit SiC abrasive papers for 1 min to standardize the smear layer. All discs (n = 174) were inspected with 40× magnification stereomicroscope to ensure the absence of enamel. The dentin discs were randomly assigned to two group: SoD or CAD. The SoD discs were not subjected to any further treatment, whereas the CAD discs had all surfaces except the buccal coated with nail varnish. The buccal surface was left uncoated to undergo the cariogenic challenge detailed in subheading 2.2. All discs were sterilized using gamma radiation and kept at 4 °C in a humid atmosphere until use.

2.2. Formation of artificially-induced CAD

The experimental setup used to induce the formation of CAD was described elsewhere [15] and it was approved by the local Research Ethics Committee (protocol 25/2013). Fresh saliva (20 mL) stimulated by paraffin film was collected from a healthy volunteer (a 48-year-old female) who had not been under antibiotic therapy for the past six months. The volunteer abstained from oral hygiene for 24 h and from food ingestion for 2 h before collection. No saliva volume was discarded before collection. A volume of 0.4 mL of this saliva was inoculated onto each dentin disc (n = 87) in a 24-microwell plate and remained for 1 h at 37 °C. The saliva was then gently aspirated from the bottom of each well and 1.8 mL of defined medium enriched with mucin (DMM) [35,36] containing 1% sucrose was added. The plates were incubated at 37 °C under an anaerobic atmosphere (5–10% CO_2, less than 1% O_2) [30]. After 4 h, the specimens were rinsed with 2 mL of sterile saline, placed into a new plate containing DMM without sucrose, and incubated for another 20 h under the same conditions. The biofilms were formed individually on the specimens in each well for 14 days, during which the same daily routine of alternate exposure to DMM supplemented or not with sucrose was followed. Previous experiments showed similar results when saliva from different donors were used in the same conditions [23]. A cross-sectional hardness test was performed to measure the integrated hardness loss (ΔS) and confirm the formation of artificially-induced CAD [15]. Briefly, four CAD specimens were longitudinally sectioned using a water-cooled diamond saw, embedded in PVC tubes using poly(methyl)methacrylate, and wet polished with 600-, 1200-, 1500-, and 2000-grit SiC abrasive papers, and with a 1 μm diamond suspension. Cross-sectional Knoop hardness measurements were performed using a microindenter (FM-700; FutureTech, Tokyo, Japan) under a load of 5 g and a dwell time of 5 s. Two columns each with eight indentations were performed in the specimens at distances of 10, 20, 30, 40, 50, 100, 150, and 200 μm from the surface. The ΔS was calculated by subtracting the hardness profile (Knoop hardness number, kgf/mm^2) of the CAD from the hardness values obtained in the sound substrate.

2.3. Formulation of experimental one-step, self-etch adhesives

Three 2-component, one-step self-etch adhesives were prepared by mixing the following componentes: bisphenol-A glycidyl dimethacrylate (Bis-GMA, MW = 512.6 g/mol) as hydrophobic monomer; 2-hydroxyethyl methacrylate (HEMA, MW = 130.1 g/mol) as hydrophilic monomer; 1,3-glycerol dimethacrylate phosphate (GDMA-P, MW = 413.3 g/mol) as acidic monomer; water and ethanol as solvents; and camphorquinone (0.4 wt%) and 4-ethyl-dimethylamino benzoate (0.8 wt%) as photoinitiators. All monomers were obtained from Esstech Inc. (Essington, PA, USA) except for GDMA-P, which was synthesized as previously described [11]. The concentration of HEMA and GDMA-P varied in the adhesives, as shown in Table 1. The adhesives were prepared using two distinct bottles (A and B), as detailed in Table 1. Before application of the adhesive, 5 μL from each bottle were dispensed into a mixing dish using a micropipette and mixed for 5 s. The final concentrations of acidic monomer after mixing the two bottles were 5 wt%, 20 wt%, and 35 wt%, thus the adhesives were labeled AD5, AD20, and AD35. The pH of the mixed adhesives (n = 3) was measured using a digital pHmeter (An2000; Analion, Ribeirão Preto, SP, Brazil).

2.4. Shear bond strength test and failure mode analysis

The dentin discs (60 SoD, 60 CAD) were cleaned with a toothbrush and distilled water and embedded in PVC tubes using poly(methyl) methacrylate [21]. The adhesives were vigorously applied to the dentin surfaces for 20 s using a microbrush, followed by air-drying for 10 s with a mild air stream. Elastomer molds with two cylindrical orifices (diameter 1.5 mm, thickness 0.5 mm) were placed at the center of the top dentin surfaces. The adhesive was photoactivated for 20 s using a light-emitting diode curing unit (Radii; SDI, Bayswater, Victoria, Australia) with 800 mW/cm^2 irradiance. The orifices were filled with composite resin (Filtek Z350 XT; 3 M ESPE, St. Paul, MN, USA), which were photoactivated for 20 s, to produce cylinder specimens with 1.77 mm^2 bonded area. The specimens were stored in distilled water at 37 °C for 24 h or 6 months, with renewal of the storage medium every month. For the shear bond strength test, a stainless steel wire (0.2 mm diameter) was looped around each cylinder and aligned with the bonded interface. The test was performed using a mechanical testing machine (DL500; EMIC, São José dos Pinhais, PR, Brazil) at a crosshead speed of 0.5 mm/min until failure. In total, 20 cylinder specimens were tested for each adhesive, substrate, and storage time combination. Fractured

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