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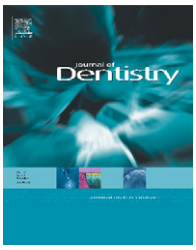

Hydrolytic degradation of the resin–dentine interface induced by the simulated pulpal pressure, direct and indirect water ageing

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ABSTRACT

Objectives: The aim of this study was to compare the hydrolytic effects induced by simulated pulpal pressure, direct or indirect water exposure within the resin–dentine interfaces created with three ''simplified'' resin bonding systems (RBSs).

Methods: A two-step/self-etching (CSE: Clearfil SE Bond), one-step/self-etching (S3: Clearfil S3) and etch-and-rinse/self-priming (SB: Single-bond 2) adhesives were applied onto dentine and submitted to three different prolonged (6 or 12 months) ageing strategies: (i) Simulated Pulpal Pressure (SPP); (ii) Indirect Water Exposure (IWE: intact bonded-teeth); (iii) Direct Water Exposure (DWE: resin–dentine sticks). Control and aged specimens were submitted to microtensile bond strength (μTBS) and nanoleakage evaluation. Water sorption (WS) survey was also performed on resin disks. Results were analysed with two-way ANOVA and Tukey's test ($p < 0.05$).

Results: The μTBS of CS3 and SB dropped significantly ($p < 0.05$) after 6 months of SPP and DWE. CSE showed a significant μTBS reduction only after 12 months of DWE ($p = 0.038$). IWE promoted no statistical change in μTBS ($p > 0.05$) and no evident change in nanoleakage. Conversely, SPP induced a clear formation of ''water-trees'' in CS3 and SB. WS outcomes were CS3 > SB = CSE.

Conclusion: The hydrolytic degradation of resin–dentine interfaces depend upon the type of the *in vitro* ageing strategy employed in the experimental design. Direct water exposure remains the quickest method to age the resin–dentine bonds. However, the use of SPP may better simulate the *in vivo* scenario. However, the application of a separate hydrophobic solvent-free adhesive layer may reduce the hydrolytic degradation and increase the longevity of resin–dentine interfaces created with simplified adhesives.

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1. Introduction

The resin–dentine interface is the most susceptible part of the adhesive-composite restorations to hydrolytic degradation[1] due to heterogeneity of the bonding structures and questionable stability of hydrophilic polymers contained within the composition of modern ''simplified'' resin bonding systems (RBSs).[2] Nevertheless, the *in vivo* durability of the resin–dentine interface may result superior to that estimated during *in vitro* assessments[3]; indeed, controversial outcomes are often observed in the scientific literature.[3–7] Several laboratory investigations presented remarkable degradation of resin–dentine bonds subsequent to a reasonably short-period of direct water ageing.[4,5] In contrast, further *in vivo* clinical studies performed on resin–dentine specimens created with the same RBSs previously tested *in vitro* showed a longevity of eight,[6] twelve[7] and twenty-two years.[8]

Although, many *in vitro* strategies have been employed to depict differences between adhesives and bonding techniques, some degradation regimens may submit bonds undergoing situations widely different from clinical conditions.[4,9]

The mainly accepted ageing strategy to challenge the durability of the resin–dentine bonds remains the direct exposure of match-stick or slabs in deionised water.[3,9] The water exposure of intact resin-bonded teeth, requires longer periods to contrast differences,[11] although it may resemble a more realistic clinical situation in terms of hydrolytic degradation. In contrast, the hydrolytic effect on smaller resin–dentine specimens directly exposed to water may be achieved in a relatively short period (*i.e.* 3–6 months).[10,12–14]

However, in a clinical situation, except for large class II and V cavities, resin–dentine interfaces are only partially in contact with environmental fluids, since outer resin-bonded enamel has been shown to prevent water uptake.[3,10] In such circumstances, these resin–dentine bonds may come in contact with fluids *in vivo* only *via* pulpal pressure through dentinal tubules.[15,16] Consequently, the use of the simulated pulpal pressure (20 cm H_2O) during the ageing period may be a suitable method for promoting hydrolytic degradation of resin-bonded dentine specimens *via* water seepage and polymer plasticisation.[17] Unfortunately, there is little information regarding the comparison of the hydrolytic effects induced by direct water exposure of tiny-specimens, indirect water exposure of intact bonded teeth and intact bonded-teeth submitted to simulated pulpal pressure.

This investigation aimed at comparing the influence of the simulated pulpal pressure, direct or indirect water exposure on the microtensile bond strengths (μTBS) and nanoleakage of resin–dentine specimens created using three representative simplified RBSs. The water sorption of the tested RBSs was also evaluated to discriminate the differences in the hydrolytic effects induced by the different ageing strategies.

Two null hypotheses were tested: (1) There is no difference between simulated pulpal pressure, direct and indirect water exposure in promoting hydrolytic degradation within the resin–dentine interface after a period of 6 or 12 months; (2) The three tested RBSs have similar attitude to water sorption.

2. Materials and methods

2.1. Sample preparation

One hundred five human third molars extracted for surgical reasons under approval of the institutional Ethics Committee (protocol 167/2009) were used in this study. The teeth were stored in 0.5% chloramine/water solution at 4 °C no longer than 2 months after extraction.

Deep dentine specimens with remaining tissue thickness of ~0.9 mm[18] were obtained by removing the roots 2 mm below cemento-enamel junction (CEJ) and the occlusal crown 2 mm above CEJ using a slow-speed water-cooled diamond saw (Isomet 1000; Buehler, Lake Bluff, IL, USA). The pulpal tissue was removed with small surgical tweezers without altering or scratching the pre-dentine surface along the walls of the pulpal chamber. The dentine surface of each specimen was wet-polished with a 600-grit SiC (CarbiMet 2; Buehler) paper for 30 s to create a standard smear-layer. The specimens were thoroughly rinsed using deionised water (5 s) and immediately bonded with the tested RBSs.

2.2. Experimental design

The dentine specimens were randomly divided into three principal groups ($n = 35$) based on the RBSs selected for this study: (i) self-etching/two-step adhesive (CSE – Clearfil SE Bond; Kuraray Medical, Tokyo, Japan); (ii) self-etching/one-step

Table 1 – Adhesives used, batches, chemical compositions and application protocols.

Materials	Composition	Application procedure	Batch
Clearfil S3 Bond	MDP, BisGMA, HEMA, dimethacrylates, photoinitator	Apply adhesive for 20 s. Air-dry for 5 s to evaporate solvent. Light cure for 10 s.	127A
Clearfil SE Bond	Primer: MDP, HEMA, water, photoinitator Bond: MDP, BisGMA, HEMA, TEGDMA, hydrophobics dimethacrylates, photoinitator	Apply primer for 20 s, gently air-dry; apply bond. Light cure for 10 s.	896A 1321A
Adper Singlebond 2	Etchant: 37% phosphoric acid Adhesive: HEMA, BisGMA, TEGDMA, polyalkenoic acid copolymer, dimethacrylates, ethanol, water and camphorquinone	Acid-etch for 15 s, rinse with water for 15 s leaving the dentine moist. Bond was applied in two coats and gently air-dried. Light cure for 10 s.	7KK 9WP

BisGMA: bisphenol-A-diglycidylmethacrylate; HEMA: hydroxyethylmethacrylate; MDP: 10-methacryloyloxi-decyl-phophate; TEGDMA: triethylene-glycol-dimethacrylate.

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