

# Effect of simulated pulpal pressure on dentin permeability and adhesion of self-etch adhesives

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# ABSTRACT

*Objectives*. Dentin bonds made with one-bottle etch-and-rinse and self-etch adhesives are affected by the formation of interfacial blisters, porosities and deterioration. The first objective of this study was to evaluate the fluid flow through resin-dentin interfaces created by self-etching adhesives applied to deep dentin using a replica technique and by directly measuring dentin permeability (*P*). The second objective was to examine the effect of intrapulpal pressure on the microtensile bond strength of these adhesives.

Methods. A fluid-transport model was used to measure the fluid permeability (%P) through different adhesives. Impressions of bonded dentin were taken with a polyvinylsiloxane impression material to monitor fluid transudation from the surface of the adhesive. Positive replicas were fabricated for SEM examination. Two groups of resin-bonded specimens (pulpal pressure versus no pulpal pressure) were created for microtensile bond strength evaluation. Adhesive application was performed under 0 cm H<sub>2</sub>O. Pulpal pressure group was submitted to 20 cm H<sub>2</sub>O of pulpal pressure during build-up procedures.

Results. Clearfil Protect Bond exhibited the lowest permeability and fewest numbers of fluid droplets over the surface of the bonded dentin. G-Bond and Clearfil-S3 Bond were more permeable than Clearfil Protect Bond. One Up Bond F was the most permeable adhesive. A highly significant correlation was observed between the relative permeability of these adhesives (%P) and the number of fluid droplets on the adhesive surfaces. The application of pulpal pressure significantly reduced bond strength.

Significance. Resin-dentin bonds created by contemporary self-etch adhesives are susceptible to fluid permeation induced by pulpal pressure. HEMA-based adhesives showed the largest reductions in bond strengths after pulpal pressure application.

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### 1. Introduction

Different classes of dentin–enamel bonding agents (DBAs) are now available to clinicians. Although simplified DBAs reduce the number of clinical steps involved in bonding [1,2], many of them are limited in their applications. In particular, most of these adhesives exhibited dramatic bond strength reductions after water storage [3,4].

Recent nanoleakage studies also demonstrated that simplified DBAs exhibited fairly severe water sorption [5], as manifested by the extensive silver tracer deposits seen within the hybrid and adhesive layers [6]. Water sorption is enhanced by the presence of hydrophilic and ionic resin or solvents. Water plasticizes polymer chains and lowers the mechanical properties of hydrophilic resins and promotes hydrolysis of resin and collagen fibrillar components [5,7,8]. Fluid movement within hybrid layers created by these DBAs has been demonstrated by the appearance of water droplets on the surface of crosssections of polished resin-dentin interfaces [9]. Transudation of fluid across polymerized adhesives bonded to dentin has also been observed in vitro and in vivo when resin composite build-ups were absent [10]. Water uptake and release were also evident from restorative margins of cavities bonded with many of these adhesives [11,12]. The outward movement of dentinal fluid under a slight positive pulpal pressure can permeate polymerized hydrophilic adhesives. This water may interfere with the subsequent coupling of resin composite to these adhesives under a stimulated pulpal pressure.

The aim of this study was to evaluate the dentin permeability (*P*) and bond strength to deep dentin bonded with different self-etching DBAs and subsequently submitted to simulated pulpal pressure. The extent of fluid transudation across resinbonded dentin was also examined with the use of an impression material replica technique to identify the relationship between adhesive permeability and its manifestation as fluid droplets on the adhesive surfaces. Two null hypotheses were tested: the first was that there is no correlation between the permeability of these DBAs and the quantity of fluid droplets identified on the surface of adhesives of bonded dentin. The second was that simulated pulpal pressure has no effect on the microtensile bond strength produced during the coupling of resin composite to self-etching adhesives.

# 2. Material and methods

# 2.1. Sample preparation

Extracted human molars (ages 20–40) were collected after informed consent had been obtained under a protocol approved by the Institutional Review Board the Department of Dental Sciences of the University of Bologna, Italy. The teeth were stored in  $4^{\circ}$ C water for no more than one month. Forty crown segments, each with a minimal remaining dentin thickness of 0.7–0.8 mm, were obtained by first removing the roots at 1mm beneath the cementoenamel junction (CEJ) using a slow-speed water-cooled diamond saw (Remet, Bologna, Italy). The occlusal enamel of each crown segment was subsequently removed with a parallel cut at 1.5 mm above the CEJ to expose the dentine. The exposed dentine was polished with 180 grit silicon carbide papers to create a standard bonding substrate in deep dentin. Pulpal tissue was removed from the exposed pulp chamber without altering the predentin surface. A pincer-type caliper was used for measurement of the remaining dentin thickness (RDT) that was between 0.7 and 0.8 mm. Each tooth section was attached to a Plexiglas platform ( $2 \text{ cm} \times 2 \text{ cm} \times 0.5 \text{ cm}$ ) that was perforated by an 18 gauge stainless steel tube using cyanocrylate adhesive (ROCKET<sup>TM</sup> Heavy DVA, Corona, CA, USA). Each specimen was connected to a hydraulic pressure device (Fig. 1) that delivered 20 cm water pressure [13] during the measurement of the dentine permeability (P).

### 2.2. Bonding procedures

Four DBAs were examined in this study. They included a two-step self-etching primer/adhesive system, Clearfil Protect Bond (Kuraray Medical Inc., Tokyo, Japan) and three one-step self-etch adhesive systems, G-Bond (GC Corp., Tokyo Japan); One Up Bond F Plus (Tokuyama Corp., Tokyo, Japan) and Clearfil S3-Bond (Kuraray Medical Inc., Tokyo, Japan). Their compositions and pH values are listed in Table 1. Each DBA was applied as per manufacturer's instruction (Table 2). Light activation of the DBAs was performed using a halogen light-curing unit (XL-2500, 3M ESPE, St. Paul, MN, USA) with an output power intensity of 600 mW/cm<sup>2</sup>, at a standardized distance of 5 mm from the bonding surface. All the tested DBAs were bonded while connected to the permeability device but in the absence of pulpal pressure application (Fig. 1).

#### 2.3. Permeability measurement

Ten crown segments were used for each DBA. A smear layer was created on dentine surface using 180 grit-paper for 30 s under water irrigation.

The smear layer was subsequently removed by treating the dentine surface with 0.5 M EDTA solution (pH 7.4) for 5 min to evaluate the maximum fluid filtration of each specimen, which was arbitrarily assigned a value of 100% permeability. After measuring the initial maximum permeability (P), a smear layer was re-created on dentin surface in the manner previously described, before the application of the self-etching DBAs. The permeability of each specimen after dentin bonding was expressed as a percentage of the fluid flow through the unbonded, EDTA-etched dentine of the same specimen using the following equation:

$$%P = \frac{\text{fluid filtration rate of resin-bonded dentine}}{\text{fluid filtration rate of unbounded EDTA-etched}} \times 100$$
  
dentine

This represents the permeability exhibited by the resinbonded dentine relative to its maximum EDTA-treated value, with each tooth serving as its own control.

Before measurements of fluid flow on the resin-bonded dentine, reference impressions of the adhesive surface were first taken before and after dentine perfusion at  $20 \text{ cm } \text{H}_2\text{O}$  pressure. The impression material employed was a low viscosity polyvinylsiloxane (President Light Body; Colténe AG,

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