



Single-bottle adhesives behave as permeable membranes after polymerisation. II. Differential permeability reduction with an oxalate desensitiser

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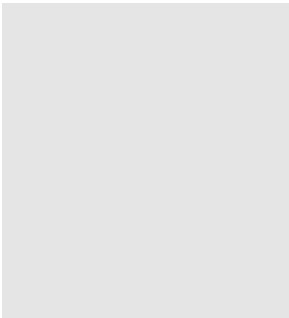
Summary *Objectives:* This study examined the changes in hydraulic conductance and ultrastructure of dentine bonded with simplified etch-and-rinse adhesives to oxalate desensitiser pre-treated acid-etched dentine.

Methods: Human dentine disks were acid-etched, treated with an oxalate desensitiser (BisBlock, Bisco), and bonded with One-Step (OS), Single Bond (SB), OptiBond Solo Plus (OB) or Prime and Bond NT (PB). Similar disks from each group were acid-etched, and bonded without oxalate pre-treatment. Hydraulic conductance of the specimens was measured at 20 cm of water pressure and analysed with nonparametric statistical methods. Epoxy resin replicas of the smear layer-covered dentine and bonded dentine were examined with SEM for the extent of fluid transudation. Specimens bonded under perfusion were examined with TEM after tracer immersion.

Results: OB and PB exhibited a highly significant reversal of the reduced hydraulic conductance obtained with BisBlock on unbonded acid-etched dentine. Profuse water transudation across the bonded dentine was observed from the replicas. Adhesive interfaces were covered with spherical globules that interfered with dentine hybridization. Conversely, no significant difference in hydraulic conductance

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was observed in SB, between Bisblock pre-treated, unbonded and bonded acid-etched dentine. Significantly lower hydraulic conductance was shown on application of OS to Bisblock-treated acid-etched dentine. Water transudation was sparse, interfering surface globular structures were absent, and only angular subsurface crystals were seen in the dentinal tubules.

Conclusions: Convective water fluxes through dentine may be reduced by applying Bisblock to acid-etched dentine before bonding with One-Step or Single Bond. However, reducing adhesive permeability with the use of oxalate desensitiser is not applicable to low acidity adhesives such as OptiBond Solo Plus and Prime and Bond NT.

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Introduction

It has been shown in Part I of this study that single-bottle adhesives remained highly permeable after polymerisation due to the lack of hydrophobic resin coatings and permit fluid transudation across the bonded dentine *in vivo*.¹ The permeability of these simplified etch-and-rinse adhesives is further aggravated by the incorporation of hydrophilic and ionic resin monomers to facilitate bonding to intrinsically wet dentine substrates.^{2,3} Incomplete sealing and continuous fluid movement through the adhesive layer to the adhesive-composite interface^{4,5} contribute to the incompatibility between simplified adhesives and dual/auto-cured composites.^{6,7}

One way of reducing the increased adhesive permeability is the adjunctive use of oxalate desensitisers on acid-etched dentine prior to adhesive application.^{8,9} Depletion of calcium ions from the surface dentine forces the oxalate ions to diffuse further down into the dentinal tubules until calcium ions are encountered for reaction. The calcium oxalate crystals that are formed result in subsurface tubular occlusion and reduction in the hydraulic conductance of dentine. However, the solubility of calcium oxalate is affected by pH, as the anion is the conjugate base of a weak acid.¹⁰ Preliminary screening of the compatibility of oxalate desensitisers with simplified etch-and-rinse adhesives demonstrated optimal compatibility of two different oxalate solutions with One-Step (Bisco Inc., Schaumburg, IL, USA) and Single Bond (3M ESPE, St Paul, MN, USA), but poor compatibility with PrimeandBond NT (Dentsply DeTrey, Konstanz, Germany) and OptiBond Solo Plus (Sybron-Kerr, Orange CA).¹¹ It was hypothesised that the acidity of the single-bottle adhesives may influence the permeability of oxalate desensitiser pre-treated acid-etched dentine bonded with these adhesives.

Thus, the objectives of this study were: (1) to compare the reduction in hydraulic conductance of the bonded interface formed by the four simplified etch-and-rinse adhesives of different acidity to oxalate desensitiser pre-treated acid-etched

dentine; (2) to examine, with the use of scanning electron microscopy and transmission electron microscopy, the ultrastructure of the bonded interfaces. The null hypothesis tested was that there is no difference in the ability of simplified etch-and-rinse adhesives of different acidity in reducing dentine permeability when they are bonded to oxalate desensitiser pre-treated acid-etched dentine.

Materials and methods

Dentine disk preparation

Seventy-two non-carious human third molars that were stored in a 0.5% chloramine T solution at 4 °C were used within one month after extraction. The teeth were collected after the patients' informed consent had been obtained under a protocol reviewed and approved by the Institutional Review Board of The University of Hong Kong. The crowns of these teeth were decoronated perpendicular to the longitudinal axis of the roots by a slow-speed saw with a diamond-impregnated disk (Isomet, Buehler Ltd, Lake Bluff, IL, USA) under water cooling. Dentine disks, approximately 0.5 mm thick, were prepared and carefully inspected to ensure that they were free of coronal enamel or pulpal exposures. A 180-grit silicon carbide paper was used under running water for 30 s to create clinically relevant smear layers on the dentine surfaces. The pulpal sides of the disks were acid-etched with 32% phosphoric acid gel (Uni-Etch, Bisco Inc.) for 15 s to render the pulpal ends of the dentinal tubules patent. The smear layers on the coronal side of these disks were left intact. Fifty-six dentine disks were prepared and were randomly assigned to each of the four groups ($N=14$). Four simplified etch-and-rinse adhesives, One-Step (OS), Single Bond (SB), OptiBond Solo Plus (OB) and Prime and Bond NT (PB), and an oxalate desensitiser BisBlock (BB; Bisco Inc.) were investigated. These materials were used according to the manufacturers' instructions.

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