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ElectroBond application may improve wetting characteristics of etched dentine

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ABSTRACT

Objective: To assess the efficacy of an electrically assisted application technique on dentine wettability (water contact angle measurements) and to determine the promoted surface roughness of dentine after applying 37% phosphoric acid etching, a self-etching primer or a self-etching adhesive system.

Methods: Wettability was assessed on 30 caries-free extracted human third molars. Specimens were sectioned parallel to the occlusal surface to expose moderately deep dentine and ground flat (water-wet 180-grit SiC) to provide uniform flat surfaces. 37% H_3PO_4, Clearfil SE Bond (CSEB) primer or Prompt-L-Pop (PLP) adhesive system were applied on dentine surfaces. In half of the specimens, the electric impulse-assisted application technique/ElectroBond-assisted application was followed. Contact angle measurements were performed to assess wettability using the Axisymmetric Drop Shape Analysis technique. Additional surfaces were conditioned for atomic force microscopy (AFM) analysis. Two-way ANOVA, Student's *t* and Student–Newman–Keuls tests were performed ($P < 0.05$).

Results: PLP-treated dentine showed the highest intertubular roughness and the lowest dentine wettability. ElectroBond application reduced water contact angles when dentine was treated with H_3PO_4 or Clearfil SE Bond primer, but not when dentine was treated with PLP.

Conclusions: The use of electric current improved wettability of dentine surface following application of phosphoric acid and a mild self-etch primer, but not self-etch adhesive.

1. Introduction

Dentine bonding relies on the proper impregnation of the substrate with an adhesive system forming the so-called hybrid layer, *i.e.* a mixture of dentine, residual smear layer and adhesive resin monomers.[1] The evolution of dental bonding agents ranged from adhesives that utilize multiple-step procedures (three step or two step etch-and-rinse; two step self-etch adhesive systems) to the development of ''easy-to-use'' single component systems (one step or all-in-one adhesive systems).[2,3] Reducing the steps of the adhesive procedure shortens the application time, and leads to a lower technique sensitivity.[2]

The establishing of the bond requires an intimate contact between the liquid adhesive and the solid adherent, the dentine.[4] Conditioning alters the inorganic phase of dentine

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Table 1 – Adhesives included in the study, manufacturers and their composition.

Materials and manufacturers	Chemical composition	Batch numbers
Clearfil SE Bond primer Kuraray Medical Inc., Kurashiki, Japan	10-Methacryloyloxydecyl dihydrogen phosphate 2-Hydroxyethyl methacrylate Hydrophilic dimethacrylate DL-Camphorquinone N,N-diethanol-p-toluidine Water	01352A
Prompt-L-Pop 3M ESPE, Seefeld, Germany	Water Methacrylated phosphoric acid–hydroxyethyl methacrylate esters BAPO initiator Stabilizer Fluoride complex Parabens	41926

and may also exert considerable changes in the surface morphology, which in turn induce modifications in its chemical and physical properties, including the wettability.[5] Wettability is one of the most important issues in adhesion and deals with surface-free energy of dentine[6] and the three dimensional arrangement of collagen fibrils during demineralization,[7] also depending on roughness and chemical composition.

The dynamic nature of dentine as a bonding substrate is responsible for marginal leakage and inconsistent bond strengths over time, which occur with all resin-based adhesives.[8–10] Different modifications to the application protocols of self-etch adhesives have been reported. They include preliminary etching of the dentine substrate with phosphoric acid or phosphoric acid plus sodium hypochlorite,[11] the use of multiple layers of adhesives,[12] additional non-solvated layer of hydrophobic monomers,[13,14] active or prolonged application of the bonding[15] and extended curing time.[16,17]

Recent studies revealed that a new electric device (ElectroBond; SETI, Rome, Italy) could be useful to improve resin infiltration of etch-and-rinse and self-etch adhesives.[18–21] The rationale for applying a dentine adhesive under an electric current is to improve adhesive infiltration of the demineralized dentine.[18] Incomplete infiltration by the adhesive resins through the demineralized zone may result in unprotected collagen and carbonated apatite crystallites that are vulnerable to enzymatic degradation and acid dissolution, respectively.[1,9,10,22,23] Nevertheless, despite the finding that the use of continuous electric current during adhesive application increased microtensile bond strength and reduced interfacial nanoleakage expression,[18–21] no conclusive hypotheses on the mechanisms sustaining the bonding increase were provided. It has been hypothesized that electricity may either affect surface wettability or directly influence adhesive monomer impregnation through iontophoretic forces that are superimposed to the polar monomers contained in the resin blends of the modern adhesive system.[18] Additionally, it has been speculated that resin infiltration improvement due to the electric-assisted application technique might be attained by altering the surface charges and hydrogen bonding potential of the dentine substrate.[18,24,25]

Contact angle measurement provides information about interfacial tension and thus a means of characterizing the wetting of the substrate by the fluid phase.[4] It is a useful tool to clarify the wettability modifications induced by electricity. Thus, the aim of this study was to investigate dentine wettability with or without the electrically assisted (ElectroBond) application assessing the contact angle. Nanoroughness and dentine morphology after testing dentine conditionings were also examined by AFM. The null hypothesis tested was that ElectroBond does not influence wettability of conditioned dentine.

2. Materials and methods

2.1. *Contact angle measurements*

Thirty caries-free extracted human third molars refrigerated at 4 °C in a solution of 0.5% chloramine for up to 1 month after extraction were used. Human specimens were obtained with the informed consent of donors, under a protocol that was reviewed and approved by the Institutional Ethics Committee. The teeth were cleaned of debris and mounted in phenolic rings with cold-cure acrylic resin leaving the occlusal two-third of the crown exposed. The specimens were sectioned below the dentine–enamel junction and ground flat through 180 grit SiC abrasive paper, under running water to provide uniform surfaces. The specimens were randomly assigned to three equal groups ($n = 10$) according to the following treatments of the dentine surfaces: (1) 37% phosphoric acid etching (Scotchbond Etchant, 3M ESPE, St. Paul, MN, USA) for 15 s, water rinsed for 60 s. (2) Clearfil SE Bond (CSEB, Kuraray Medical Inc., Kurashiki, Japan) primer was applied for 20 s. (3) Prompt-L-Pop (PLP, 3M ESPE, Seefeld, Germany) was applied for 15 s (Table 1). After application of self-etch solutions (groups 2 and 3), resin was removed by washing and ultrasonic agitation in ascending series of ethanol (50%, 60%, 70%, 80%, 90%, 96% and 100%) for 1 min each, and further sonicated in absolute ethanol for 1 min to dissolve the self-etching primer or uncured adhesive.[26]

Specimens were rehydrated in PBS solution (pH 7.0) for 24 h. Ten 0.3 μl drops of water were consecutively placed on the ground and wet (shiny but not pooled) dentine surface in each group. The measurements were made inside a thermostatic cell at 25 °C and close to 100% relative humidity.

In five of the specimens in each subgroup, the ElectroBond 3.0 (SETI, Roma, Italy) device was applied during contact angle measurements. To permit electrical conduction under *in vitro* conditions, the dentine surfaces to be tested were placed on a