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Dentin bond optimization using the dimethyl sulfoxide-wet bonding strategy: A 2-year in vitro study

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ABSTRACT

Objective. This study evaluated a new approach, named dimethyl sulfoxide (DMSO)-wet bonding, to produce more desirable long-term prospects for the ultrafine interactions between synthetic polymeric biomaterials and the inherently hydrated dentin substrate.
Methods. Sound third molars were randomly restored with/without DMSO pretreatment using a total-etch (Scocthbond Multipurpose: SBMP) and a self-etch (Clearfil SE Bond: CF) adhesive systems. Restored teeth (n = 10)/group were sectioned into sticks and submitted to different analyses: micro-Raman determined the degree of conversion inside the hybrid layer (DC); resin–dentin microtensile bond strength and fracture pattern analysis at 24 h, 1 year and 2 years of aging; and nanoleakage evaluation at 24 h and 2 years.
Results. DMSO-wet bonding produced significantly higher 24 h bond strengths for SBMP that were sustained over the two-year period, with significantly less adhesive failures. Similarly, DMSO-treated CF samples presented significantly higher bond strength than untreated samples at two years. Both adhesives had significant less adhesive failures at 2 years with DMSO. DMSO had no effect on DC of SBMP, but significantly increased the DC of CF. DMSO-treated SBMP samples presented reduced silver uptake compared to untreated samples after aging.
Significance. Biomodification of the dentin substrate by the proposed strategy using DMSO is a suitable approach to produce more durable hybrid layers with superior ability to

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withstand hydrolytic degradation over time. Although the active role of DMSO on dentin bond improvement may vary according to monomer composition, its use seems to be effective on both self-etch and etch-and-rinse bonding mechanisms.

1. Introduction

Adhesion of resin materials to tooth structure has been a challenge in the history of adhesive dentistry. Currently, the issue of bond durability has attracted significant attention regarding resin–dentin bonding [1–3]. Despite improvements in dental adhesive technology and advances in bonding knowledge, resin–dentin bonding still shows limited durability for both etch-and-rinse and self-etch adhesive systems [1,4–6].

Resin–dentin bonding is a unique form of tissue engineering in which an ultrafine biopolymer, known as hybrid layer, links composites to the underlying mineralized dentin by two substantially different bonding mechanisms produced by the adhesive system used: *etch-and-rinse* or *self-etch* [7,8]. Nevertheless, resin–dentin bonds created by infiltration of hydrophilic resin monomers into demineralized [9,10] and mineralized dentin [10,11] are imperfect and unstable [6,12]. Inadequate polymerization reduces the quality of the hybrid layer [6] leading to lower dentin bond strengths and increased nanoleakage [13]. Moreover, high permeability of the bonded interface and phase separation during adhesive application contribute to hydrolytic degradation of the adhesive resin [6,12]. Insufficient resin impregnation of dentin [9] is associated with the collagenolysis of unprotected collagen fibrils by endogenous matrix metalloproteinases (MMPs) and cysteine cathepsins [3,14]. Irrespective of adhesive type, hydrolytic degradation of the adhesive resin and collagen matrix degradation occur concurrently, for resin elution from hydrolytically unstable polymeric hydrogels within the hybrid layers increases the exposure of unprotected collagen matrix over time.

Several adjunctive procedures have been suggested to prevent biodegradation of hybrid layers over time [3]. Although encouraging results have been produced, the current available techniques do not effectively address both hydrolytic degradation of the adhesive resin and collagen degradation concurrently. The possible exception is the ethanol-wet bonding aiming to remove water from the exposed dentin collagen and to replace it with more hydrophobic resin components [2,8]. Excluding water with high ethanol concentrations would reduce/eliminate the hydrolytic degradation of both the collagen and resin components of the hybrid layer [2,8,15]. Unfortunately, ethanol-wet bonding is clinically unfeasible due to technique sensitivity and increase in application steps and treatment time [3]. Thus, current strategies are at least partially limited in their true potential to optimize the durability of resin–dentin bonding.

Dimethyl sulfoxide (DMSO; $(CH_3)_2SO$) is a polar aprotic solvent with a highly polar S=O group and two hydrophobic CH_3 groups. Its ability to penetrate biological surfaces and tissues makes it the best penetration enhancer for medical purposes [16]. Recent studies have indicated that DMSO may improve the penetration of adhesive into the exposed collagen matrix [10], and improve both immediate [10,17] and long-term [17] dentin bond strength. However, the long-term efficacy has only been demonstrated with a two-step etch-and-rinse adhesive [17]. Therefore, this in vitro study evaluated the effect of DMSO-wet bonding on dentin bond durability, monomer conversion inside the hybrid layer and the quality of aged bonded interfaces of two-step self-etch and three-step etch-and-rinse adhesives after 1 and 2 year storage. The null hypotheses to be tested were that irrespective of adhesive type, the application of 50 vol% DMSO in water on dentin: (i) would not influence monomer conversion at the hybrid layer; (ii) would not affect immediate or long-term dentin bond strength and; (iii) would not improve the adhesive interface quality regarding the formation of nanoleakage channels.

2. Materials and methods

2.1. *Teeth selection and preparation*

Forty intact human third molars with complete root formation were extracted for surgical reasons with patients' (age 18–25 years) informed consent and approval by the local Ethical Committee under protocol number 110/2014. Teeth were cleaned, disinfected for one week in 0.5% chloramine-T solution at 4 °C, and stored in distilled water at 4 °C for up to one month before use. A flat coronal dentin surface was obtained by sectioning off the occlusal one-third of the crown (Isomet 1000 Precision Saw, Buehler, Lake Bluff, IL, USA). The surface roughness was standardized with 600-grit silicon carbide paper (CarbiMet, Buehler Ltd., Lake Bluff, IL, USA) for 60 s under water cooling and the specimens were randomly assigned to four groups ($n = 10$) according to the bonding protocols.

2.2. *Dentin bonding protocol*

Two commercially available unaltered adhesive systems were used (Table 1): a three-step etch-and-rinse adhesive system (Adper Scotchbond Multi-Purpose, 3M ESPE, St. Paul, MN, USA) (SBMP) and a two-step self-etch adhesive (Clearfil SE Bond, Kuraray, Osaka, Japan) (CF). Table 1 lists the mode of application, components and manufacturers of the adhesive systems.

Dentin bonding in control groups was performed according to Table 1. In experimental groups, the DMSO-wet bonding technique was employed, which consisted of light-pressure circular scrubbing movements of a 50 μL of water-based 50% (v/v) DMSO (dimethyl sulfoxide, Sigma–Aldrich, St. Louis, MO, USA) (pH 8.2) for 60 s, using a disposable cavity brush. In SBMP groups, DMSO was applied after dentin etching and water rinsing. In CF groups, DMSO was applied onto smear layer-covered