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Fatigue resistance of dentin bonds prepared with two- vs. three-step adhesives: Effect of carbodiimide

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ABSTRACT

The application of a cross-linker to demineralized dentin is reportedly effective at extending the durability of dentin bonds.

Objective. To compare the effect of a cross-linker pretreatment on the fatigue crack growth resistance of resin–dentin bonds prepared with a two- vs. three-step adhesive system.

Methods. Bonded interface Compact Tension (CT) specimens were prepared using commercial two- and three-step etch-and-rinse adhesives and compatible hybrid resin-composite. For the treated groups, adhesive bonding was preceded by a 1 min application of an experimental carbodiimide (EDC) conditioner to the acid-etched dentin. The control groups received no such treatment. The fatigue crack growth resistance was examined after storage in artificial saliva at 37 °C for 0, 3 and 6 months.

Results. There was no significant difference in the immediate fatigue crack growth resistance the control and EDC-treated groups at 0 months for either adhesive system. After 3 and 6 months of storage, the EDC-treated groups exhibited significantly greater ($p \leq 0.05$) fatigue crack growth resistance than the controls. Although the EDC treatment was equally effective in deterring degradation for both adhesives, bonds prepared with the three-step system exhibited the lowest resistance to fatigue crack growth overall.

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Significance. An EDC treatment applied during dentin bonding could help maintain the durability of bonds prepared with two or three-step adhesive bonding systems.

1. Introduction

Over the past few decades the quality of adhesive bonds achieved to dentin and enamel has increased substantially. Although most commercial products are capable of achieving acceptable immediate bond strength to the tooth, adhesive bonds to dentin undergo a gradual reduction in durability over time [1–5]. The solution to this problem has been elusive as both the degree of degradation and the contributing mechanisms are not consistent for all adhesive systems [6–9]. Clinically relevant approaches for increasing the durability of adhesive bonds to the tooth are essential to the success of adhesive dentistry.

One of the primary forms of degradation to resin–dentin adhesive bonds is the exposure and activation of endogenous dentin proteases [10,11]. Contemporary bonding procedures involving either etch-and-rinse or self-etch adhesives cause activation of matrix-metalloproteinases (MMP)s and cysteine cathepsins (e.g., [11–14]). These are host-derived proteolytic enzymes that are bound to the dentin collagen matrix. When uncovered by etching, the MMPs slowly solubilize the collagen fibrils [10] and remain active even after resin-infiltration. This process triggers the gradual destruction of poorly infiltrated fibrils within the hybrid layers [15–17]. Resin tags that are not well anchored to collagen fibrils and/or poor collagen integrity are critical weak links of the bonded interface [18]. The degradation of collagen matrices resulting from endogenous protease activity can cause a decrease in bond strength over time and a reduction in the durability of resin–dentin bonds.

One promising approach to extending the durability of resin–dentin bonds is to treat acid-etched dentin with cross-linking agents that are applied just prior to the adhesive. Various cross-linking agents have been explored for this purpose such as glutaraldehyde, genipin, proanthrocyanidin, and carbodiimide [19–21]. Cross-linking of the fibrils serves to inactivate the catalytic site of proteases [22], which reduces the susceptibility of the collagen fibrils to enzymatic degradation by collagenases.

Carbodiimide (hereafter referred to as EDC) has recently been identified as one of the most promising cross-linkers for stabilizing dentin bonds. In comparison to other products it has the advantage of very low cytotoxicity, and an ability to preserve dentin bond strength within clinically acceptable treatment times [23–26]. The application of 0.5 M EDC for 1 min during dentin bonding reportedly reduced the degradation in fatigue crack growth resistance of bonds prepared with a three-step resin adhesive bonding system [27]. While promising, two-step adhesive systems generally exhibit lower durability and resistance to degradation with aging [15]. Indeed, EDC was found less effective in maintaining the microtensile strength of dentin bonds prepared with a two-step adhesive system when compared to results obtained with a comparable three-step system [26].

A comparison of the effectiveness of EDC treatment in preserving the fatigue crack growth resistance of dentin bonds prepared using two- and three-step adhesive systems has not been reported. Therefore, the primary objective of this investigation was to compare the effectiveness of an EDC treatment (consisting of 0.5 M and 1 min exposure) in maintaining the fatigue crack growth resistance of adhesive bonds to dentin prepared with a two-step vs. three-step adhesive resin after aging. The null hypotheses to be tested were that (1) there is no difference in the fatigue crack growth behavior of dentin bonds prepared with two- vs. three-step adhesive resins, and (2) there is no difference in the effectiveness of the EDC treatment on the fatigue crack growth resistance for the two resin systems up to 6 months of storage.

2. Materials and methods

The fatigue crack growth resistance of resin–dentin bonds was evaluated using specimens that were prepared from sections of mid-coronal dentin. Axial sections were obtained from caries-free human third molars, which were acquired with informed signed consent from participating clinics in Maryland with record of age ($18 \leq \text{age} \leq 30$ years) according to an approved protocol (#Y04DA23151). Each tooth was sectioned using a slicer/grinder (Chevalier Smart-H818II, Chevalier Machinery, Santa Fe Springs, CA, USA) with diamond abrasive slicing wheels (#320 mesh abrasives) and copious water coolant. Two mm thick sections were obtained from the mid coronal region as shown in Fig. 1a. The remaining materials used in the development of the specimens included a three-step etch-and-rinse adhesive (Scotchbond Multipurpose, SBMP, 3M ESPE) and a two-step etch-and-rinse adhesive (Sinlge Bond, SB, 3M ESPE), as well as a compatible resin composite (Z100, 3M ESPE).

Bonded interface Compact Tension (CT) specimens were prepared from the sections of dentin using a special molding technique described previously [18,28]. As noted in Fig. 1b, the dentin sections represented half of the completed CT specimen geometry. The outer dentin (edge oriented farthest from the pulp) was etched for 15 s with SB 37% phosphoric acid etchant and rinsed with water for 15 s. For SBMP, the primer and adhesive were applied to the etched surface according to the manufacturer's recommendations. Similarly, for the SB specimens the resin adhesive was applied and light-cured according to the manufacturer's instructions. Thereafter, the prepared sections were placed in a specially designed mold for incremental application of the resin composite. A thin Mylar sheet was placed at one end of the interface to introduce a molded notch as shown in Fig. 1b. The composite was

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