Bonding over Dentin Replacement Materials

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Abstract

Introduction: Dentin replacement materials are necessary in large cavities to protect the pulp and reduce the bulk of filling material. These materials are layered with a composite resin restorative material. Microleakage caused by poor bonding of composite resin to underlying dentin replacement material will result in pulp damage. The aim of this study was to characterize the interface between dentin replacement materials and composite resin and to measure the shear bond strength after dynamic aging. **Methods:** Biodentine (Septodont, Saint Maur-des-Fosses, France), Theracal LC (Bisco, Schaumburg, IL), and Fuji IX (GC, Tokyo, Japan) were used as dentin replacement materials. They were then overlaid with a total-etch and bonding agent or a self-etch primer and composite resin or a glass ionomer cement. All combinations were thermocycled for 3000 cycles. The interface was characterized using scanning electron microscopy and elemental mapping. Furthermore, the shear bond strength was assessed. **Results:** The Biodentine surface was modified by etching. The Theracal LC and Fuji IX microstructure was unchanged upon the application of acid etch. The Biodentine and glass ionomer interface showed an evident wide open space, and glass particles from the glass ionomer adhered to the Biodentine surface. Elemental migration was shown with aluminum, barium, fluorine, and ytterbium present in Biodentine from the overlying composite resin. Calcium was more stable. The bond strength between Theracal LC and composite using a total-etch technique followed by self-etch primer achieved the best bond strength values. Biodentine exhibited the weakest bond with complete failure of bonding shown after demolding and thermocycling. **Conclusions:** Dynamic aging is necessary to have clinically valid data. Bonding composite resin to water-based dentin replacement materials is still challenging, and further alternatives for restoration of teeth using such materials need to be developed. *(J Endod 2017;■:1–7)*

Key Words

Biodentine, characterization, dentin replacement materials, dynamic testing, elemental mapping, glass ionomer cement, shear bond strength, Theracal LC

Significance

In vitro testing without the necessary aging may have limited clinical significance because the material performance is not mimicked adequately.

Pulp capping materials are used to protect the pulp from thermal, chemical, and other noxious stimuli. Tricalcium silicate–based materials such as mineral trioxide aggregate (MTA) are commonly used for this purpose. MTA exhibits many disadvantages such as difficult handling, long setting time (1), induction of tooth discoloration (2–5), and incompatibility with other dental materials when layered (6, 7). Second-generation tricalcium silicate–based materials exhibiting improved physical properties were introduced in an attempt to overcome the disadvantages presented by MTA.

Two such materials that are used as dentin replacement materials include Biodentine (Septodont, Saint Maur-des-Fosses, France) and Theracal LC (Bisco, Schaumburg, IL). Both materials are tricalcium silicate based but are rather diverse. Biodentine is a water-based cement presented as a powder in a capsule composed of tricalcium silicate cement, zirconium oxide, and calcium carbonate. The liquid in the ampule is composed of water, calcium chloride, and a water-based polymer (8, 9). Biodentine exhibits a short and clinically suitable setting time and was introduced as an alternative to MTA to be used as dentin replacement material. On the other hand, Theracal LC is a resin-modified light curable material used as a liner under composite restorations. The cement component is Portland type, and it includes a radiopacifying material and a silicon oxide additive in a resin matrix (10). Theracal LC does not include water for material hydration, and thus it depends on the water taken up from the environment and its diffusion within the material. The hydration of this material may be compromised when compared with water-based materials such as Biodentine (8). The manufacturer suggests placement of permanent restoration immediately after curing.

The bond strength between restorative and pulp capping materials is important for the success of restorations (11). Etch-and-rinse adhesive systems exhibit stronger bonding than self-etch adhesive systems (12). Bonding composite resins to novel dentin replacement materials requires adequate research on the material interactions and interfacial characteristics. Etching of Biodentine has been shown to result in destruction of the material microstructure and enhanced leakage through the Biodentine composite interface (13). Furthermore, layering with composite resin has been investigated (14–19), and Biodentine was shown to be weak. It has been suggested that the final restoration with composite resin should be placed after at least 6 weeks using either a self-etch or total-etch bonding system (18). Theracal LC had higher bond strength values than Biodentine when layered with either composite or glass ionomer cement. The glass ionomer

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cement overlayering resulted in lower bond strengths than composite resin. Composite resins applied with self-etching adhesives increased the bond strength of Theracal LC. However, the application of etch-and-rinse adhesives improved the adhesion of composite resins to Biodentine (17). All the studies investigated interfaces of aged static systems, and no characterization was performed.

The aim of the current study was to characterize and evaluate the adequacy of different bonding systems to layer composite or glass ionomer cement over 3 dentin replacement materials (Biodentine, Theracal LC, and Fuji IX glass ionomer [GC, Tokyo, Japan]) using dynamic aging.

Materials and Methods

Three dentin replacement materials were used in this study: Biodentine, Theracal LC, and Fuji IX. Mandibular molars extracted for periodontal reasons were used in this study. Twenty-four teeth are selected to enable 3 teeth per dentin replacement/restorative material combination. The teeth were caries free and without restorations present. They were checked under a stereomicroscope to make sure that there were no microcracks visible. The roots were embedded in cold-cured epoxy resin (EpoxyFix; Struers, Ballerup, Denmark) to avoid interaction of any fluids seeping in from the apices through the root canal and into the pulp chamber, thus affecting the materials. Occlusal cavities 6 mm × 4 mm and 4 mm deep. The pulpal floors were covered with Biodentine, Theracal LC, or Fuji IX. Biodentine was mixed according to the manufacturer's instructions, was placed over the cavity floor, and was allowed to set for 15 minutes. Theracal LC was syringed on the cavity floor and light cured for 20 seconds at 600 mW/cm^2 using an light-emitting diode light source. Fuji IX was mixed according to the manufacturer's instructions and placed over the cavity floor without prior cavity conditioning. A 2-mm layer was placed for each material, leaving a 2-mm cavity to be filled with restorative material. The height was checked with a vernier caliper.

The adequacy of layering with composite using different adhesive systems was assessed by using 2 different bonding systems.

1. Total etch and bond (Excite F; Ivoclar, Schaan, Lichtenstein) preceded by etching with 37% phosphoric acid for 30 seconds
2. Self-etch adhesive (AdheSe One F, Ivoclar)

The bonding systems were applied and cured for 10 seconds according to the manufacturer's instructions. After application of the bonding system, the pulp capping materials were layered with light-curing composite resin (Evetric, Ivoclar) for 20 seconds for each layer. Furthermore, layering with a glass ionomer cement (Fuji IX) was also performed. No material preparation was necessary when layering with glass ionomer cement. The teeth were thermocycled for 3000 cycles at 5°C and 55°C. The dwell time in each bath was 20 seconds, and the transfer time between the 2 baths was 10 seconds following ISO 11405:2015 (20) instructions.

Scanning Electron Microscopy and Elemental Mapping

After thermocycling, the teeth were surface dried, sectioned longitudinally, and polished with progressively finer grits of diamond discs and abrasive solution (Tegramin 20; Struers, Ballerup, Denmark). The specimens were then mounted on an aluminum stub, carbon coated, and viewed under a scanning electron microscope (Zeiss MERLIN Field Emission SEM; Carl Zeiss NTS GmbH, Oberkochen, Germany). Scanning electron micrographs of the material interfaces were captured at ×1000 magnification; energy-dispersive spectroscopy was performed followed by elemental mapping of the materials and the interface, thus determining if elemental migration between the dentin replacement material and the overlying composite occurred.

Shear Bond Strength

Plexiglass blocks (n = 120) containing a central hole 4 mm in internal diameter and 2 mm in height were prepared by computer numeric control (CNC) laser cutting (LaserProI; GCC, New Taipei City, Taiwan) and filled with 1 of the dentin replacement materials evaluated in this study (ie, Biodentine, Theracal LC, or Fuji IX [n = 45 each]). Split plexiglass blocks with a central hole 2 mm in internal diameter and 2 mm in height were also prepared with CNC laser cutting and placed and fixed on the filled blocks in a way that the smaller 2-mm diameter hole was positioned in the center of the 4-mm diameter hole filled with dentin replacement materials. Then, the specimens of each main experimental group were divided into 3 subgroups according to the filling protocol used: Excite F total etch and bond preceded by etching with 37% phosphoric acid, AdheSe One F self-etch adhesive, and Fuji IX.

Subsequently, the split molds were removed, and specimens were thermocycled as indicated previously. The specimens were tested for shear bond strength using a universal testing machine (Z050; Zwick/Roell, Ulm, Germany) with a chisel-edge plunger aimed at the dentin replacement material/adhesive interface with a speed of 0.5 mm/min. Shear bond strength in Nmm^{-2} was calculated by dividing the peak load at failure by the specimen surface area. Failure modes were evaluated by a single operator under a stereomicroscope (Olympus SZ30; Olympus, Tokyo, Japan) at ×20 magnification and categorized into 1 of 3 types:

1. Adhesive: 100% adhesive failure between the dentin replacement materials and the filling materials
2. Cohesive: 100% cohesive failure within the dentin replacement materials or filling materials
3. Mixed: Mixed failure composed of adhesive and cohesive failure of the dentin replacement materials or the filling materials. For this category, a descriptive evaluation was also included.

Statistical Analyses

Data were evaluated using SPSS software (PASW Statistics 18; SPSS Inc, Chicago, IL). Parametric tests performed as Kolmogorov-Smirnov tests on the results indicated that the data were normally distributed. Analysis of variance with P = .05 and the Tukey post hoc test were used to perform multiple comparison tests.

Results

Scanning Electron Microscopy and Elemental Mapping

The scanning electron micrographs showing the material interfaces of each dentin replacement material are shown in Figure 1*A*. Some cracks were visible in the materials, and this was not considered as an important finding because the materials were subjected to vacuum conditions.

Biodentine exhibited changes in the material microstructure, particularly when etched. In the total-etch method, the materials were well compacted together (Fig. 1*A*). With the self-etch adhesive, some areas seemed to have separated. The glass ionomer became completely dissociated from the Biodentine, leaving an uneven surface in Biodentine. Theracal LC exhibited a more uniform interfacial region. The materials contacted each other well. The same was observed for the glass ionomer and composite combination. The self-etch adhesive exhibited a better interface than the total-etch adhesive. The total-etch adhesive resulted in pulling away of the glass ionomer from the composite surface with remnants of glass particles adhering to the composite at the interface.