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## Bonding to caries affected dentine

Naghmeh Meraji<sup>a</sup>, Mohammad H. Nekoofar<sup>a,b</sup>, Kazem Ashofteh Yazdi<sup>a</sup>, Mohammad Reza Sharifian<sup>a</sup>, Noushin Fakhari<sup>c</sup>, Josette Camilleri<sup>d,e,\*</sup>

<sup>a</sup> Department of Endodontics, School of Dentistry, Tehran University of Medical Sciences, Tehran, Iran,

<sup>b</sup> Endodontology Research Group, School of Dentistry, Cardiff University, Cardiff, Wales, UK,

<sup>c</sup> Department of Endodontics, School of Dentistry, Shahid Sadughi University of Medical Sciences, Yazd, Iran

<sup>d</sup> Department of Restorative Dentistry, Faculty of Dental Surgery, University of Malta, Msida, Malta,

<sup>e</sup> School of Dentistry, Institute of Clinical Sciences, College of Clinical and Medical Sciences, University of

Birmingham, Edgbaston, Birmingham, UK

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#### ABSTRACT

*Objectives.* Dentine replacement materials are often placed over caries affected dentine (CAD). The aim of this study was to compare the bonding characteristics and interactions of selected hydraulic calcium silicate-based dentine replacement materials to CAD and sound dentine.

Methods. Three hydraulic calcium silicate-based dentine replacement materials were assessed: Retro MTA, Biodentine and Theracal LC. Material characterization was done by scanning electron microscopy and X-ray diffraction analyses. Blocks of sound and CAD were prepared and standardized by Vickers microhardness testing. Half of the affected and sound dentine blocks were pretreated with 5.25% NaOCl prior to material placement. The materials were stored either for 1 week or 24 weeks in 37 °C in fully saturated conditions. Shear bond strength was assessed at both time periods. Radiopacity of the interfacial dentine was also evaluated to assess the remineralization potential of the dentine replacement materials.

Results. The reaction of Theracal was slower than that of the water-based materials. The bond strengths of different materials did not differ after 1 week (P > 0.05). The bond strength of Biodentine and Retro MTA increased over time but no change was observed for Theracal. NaOCl pre-treatment deteriorated the bond strength to sound dentine but improvement was observed in affected dentine. Radiopacity changes were observed after 24 weeks.

*Significance.* Biodentine and Retro MTA showed better bonding to CAD. Pretreatment with NaOCl improved the bond strength of dentine replacement materials to CAD.

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#### 1. Introduction

Carious dentin consists of two distinct layers: an outer bacterially infected layer of dentine, and an inner layer of affected dentine [1]. The caries-infected layer (CID) is highly demineralized, physiologically unremineralizable and contains irreversibly denatured collagen fibrils with a virtual disappearance of cross-linkages [2,3]. On the other hand, the caries-affected layer (CAD) is uninfected, partially deminer-

<sup>\*</sup> Corresponding author at: School of Dentistry, Institute of Clinical Sciences, College of Medical and Dental Sciences, The University of Birmingham, 5 Mill Pool Way, Edgbaston, Birmingham, B5 7EG, UK.

E-mail address: J.Camilleri@bham.ac.uk (J. Camilleri).

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alized and physiologically remineralizable [2–4]. High porosity and exposure of collagen fibers along with a decrease in the surface energy are seen in the inter-tubular CAD [2–4]. Collagen cross-linking remains intact which can function as a scaffold for remineralization of intertubular dentine [2–4]. Therefore, the caries-affected layer should be preserved during clinical treatments.

It has been shown that CAD exhibits lower bond strengths to restorative materials such as glass ionomer and composite resins than sound dentine [5,6]. The change in chemical and morphological characteristics of CAD may be a reason for this lower bond strength. As CAD is partially demineralized, an irregular and thicker hybrid layer enriched with organic components is created on it [7–9].

In an attempt to improve bond strength to CAD, studies have suggested pretreatment of dentine with sodium hypochlorite solution (NaOCl) [8]. NaOCl can effectively dissolve organic components. Taniguchi et al. [8] demonstrated that pretreatment with 6% NaOCl for 15 s could significantly improve the bond strengths of both 1-step and 2-step self-etch system to CAD. This finding was attributed to the dissolution of superficial organic components of smear layer by NaOCl.

When treating deep carious lesions, the dentine replacement materials come in contact with CAD. Hydraulic calcium silicate cements have shown a lot of promise as dentine replacements due to their beneficial effect on the pulp. Biodentine (Septodont, Saint Maur des Fosses, France) [10] and Retro MTA (BioMTA, Seoul, Korea) [11] are fast setting dentine replacement materials exhibiting clinically suitable properties. Theracal LC (Bisco, Schaumburg, IL, USA) is a resinmodified light curable dentine replacement material used as a liner under composite restorations [12]. It contains a special hydrophilic resin matrix, which allows water penetration and ion release, resulting in apatite formation and sealing of the tooth [13]. The Biodentine and Retro MTA are waterbased and composed mostly of tricalcium silicate cement. The Theracal is resin based and contains fillers and other additives. Although previous research has reported the apatite formation and the ion release in solution [13], the calcium ion releasing ability and hydration of the tricalcium silicate component of the Theracal is disputed [14,15]. This will affect the material clinical performance.

Most research on the interactions between hydraulic calcium silicate-based dentine replacement materials and dentine is performed using sound dentine [16,17]. Clinically, when used over the pulp, these material are rarely placed on sound dentine. Therefore, the aim of this study was to compare the bonding characteristics and interactions of hydraulic calcium silicate based dentine replacement materials to CAD and sound dentine. The null hypothesis was that the bond of these material to CAD does not differ from sound dentine and pretreatment of dentine with NaOCl does affect their bond strength.

#### 2. Materials and methods

Three hydraulic calcium silicate-based materials were assessed. These included:

- Retro MTA (BioMTA, Seoul, Korea)
- Biodentine (Septodont, Saint Maur des Fosses, France)
- Theracal LC (Bisco, Schaumburg, IL, USA).

The materials were prepared according to manufacturer's instructions. Once set, they were placed in Hank's balanced salt solution (HBSS, H6648, Sigma Aldrich, St. Louis, MO, USA) for 1 week and 24 weeks time periods. At each time point the set material were removed from solution and were dried in a vacuum desiccator and tested.

#### 2.1. Material characterization

Cylindrical specimens 10 mm in diameter and 2 mm high were prepared. At each time point the specimens were removed from solution, dried and prepared for characterization. Three replicates were prepared for each test. The three materials were characterized by using scanning electron microscopy (SEM) and X-ray diffraction (XRD) analysis. For scanning electron microscopy the dried specimens were impregnated in resin (Epoxyfix, Struers GmbH, Ballerup, Denmark) under vacuum. The resin blocks were then ground with progressively finer diamond discs and pastes using an automatic polishing machine (Tegramin 20, Struers GmbH, Ballerup, Denmark). The specimens were mounted on aluminum stubs, carbon coated and viewed under the scanning electron microscope (Zeiss MERLIN Field Emission SEM, Carl Zeiss NTS GmbH, Oberkochen, Germany). Scanning electron micrographs of the different material microstructural components at different magnifications in back-scatter electron mode for the polished samples were captured and energy dispersive spectroscopy (EDS) was also performed.

For phase analysis the dried materials were crushed to a fine powder using an agate mortar and pestle. Phase analysis of the powders was performed using a Bruker D8 diffractometer (Bruker Corp., Billerica, MA, USA) with Co K $\alpha$  radiation (1.78 A°). The X-ray patterns were acquired in the 2 $\theta$  (15–45°) with a step of 0.02° and 0.6 s per step. Phase identification was accomplished using a search-match software utilizing ICDD database (International Centre for Diffraction Data, Newtown Square, PA, USA).

# 2.2. Assessment of bond strength of the materials to caries affected dentine

Human posterior teeth were used for this experiment. They were extracted for various reasons and were kept in water until use. Two types of dentine blocks (60 each) were prepared. One set was from sound dentine and the other from caries affected dentine. For sound dentine caries free teeth were selected, the enamel was trimmed from the surface of teeth with a gypsum model trimmer to achieve a flat dentine surface. For carious dentine specimens, molar teeth with class 1 occlusal carious lesions were selected. To expose the cariesaffected dentine the enamel portion was trimmed in a gypsum model trimmer to allow direct access to the carious dentine. The carious lesions in dentine were then removed with a manual excavator assisted by a caries detector dye (Seek, Ultradent, USA). Reaching the hard dentine layer was verified by dentine resistance to excavation, color and visual inspection. Both

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