Effects of Calcium Silicate–based Materials on the Flexural Properties of Dentin

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Abstract

Introduction: Prolonged exposure of root dentin to calcium hydroxide alters the fracture resistance of dentin. Calcium silicate–based materials (CSMs) used in endodontics release calcium hydroxide on setting. This study examined whether prolonged contact of dentin with CSMs adversely affects its mechanical properties. Methods: Dentin beams prepared from extracted human molars (7 × 3 × 0.3 mm) were divided into 3 groups on the basis of the material to which dentin was exposed (Biodentine, MTA Plus, and untreated control beams). Three-point flexure to failure was performed for each beam at designated exposure times (24 hours, 1, 2, and 3 months; n = 10). Data were analyzed with 2-factor repeated-measures analyses of variance to determine the effects of material and aging time on flexural modulus, flexural strength, and modulus of toughness (α = 0.05). Results: For flexural modulus, there was no significant difference for material (P = .947) or aging time (P = .064) when compared with baseline control. For flexural strength, significant differences were associated with aging time (P = .001) but not with material (P = .349). Flexural strength of dentin exposed to Biodentine decreased significantly after 2 and 3 months, whereas that exposed to MTA Plus decreased significantly after 3 months of aging (P < .05). For modulus of toughness, significant declines were observed for both material (P < .004) and aging time (P < .001). Conclusions: Both CSMs alter material toughness more than the strength and stiffness of dentin after aging in 100% relative humidity. Because dentin toughness is attributed to its collagen matrix, the amount of collagen extracted from mineralized dentin and changes in collagen ultrastructure should be further examined after exposure of dentin to CSMs. (J Endod 2012;38:680–683)

Key Words
Calcium silicate, dentin, flexural modulus, flexural strength, modulus of toughness

Calcium hydroxide (Ca(OH)₂) has been used for various endodontic procedures including interappointment antibacterial dressing, pulp capping, pulpotomy, and apexification (1–3). Previous studies have shown that Ca(OH)₂, on prolonged contact with dentin, adversely affects strength and fracture resistance (4–7). This is clinically relevant because endodontically treated teeth are generally thought to be weaker (8, 9). Moreover, teeth in need of apexification often have thin roots that are already prone to fracture (10).

As new CSMs become commercially available, it is important to identify how the mechanical properties of dentin are affected by these materials. Thus, the purpose of the present study was to examine whether prolonged contact of dentin with 2 recently introduced CSMs, Biodentine (Septodont, Saint-Maur-des-Fossés, France) and MTA Plus (Prevest-Denpro, Jammu City, India), adversely affects flexural properties. Specifically, flexural modulus, flexural strength, and modulus of toughness (MOT) of dentin were tested by using a 3-point flexure design. Biodentine is recommended for use as both an endodontic repair material and a dentin substitute under resin composite restorations. It contains tricalcium silicate, dicalcium silicate, calcium carbonate and oxide, iron oxide, and zirconium oxide as its powder components and calcium chloride and a water-soluble polymer as its liquid components (23). MTA Plus has a finer particle size than other commercially available MTA versions (50% of the particles finer than 1 µm) and uses a salt-free water-soluble polymer gel as the mixing vehicle to improve its washout resistance (24). The null hypothesis tested was that there are no changes in
flexural properties of dentin over time when the 2 CSMs are placed in direct contact with human dentin.

**Materials and Methods**

### Dentin Slabs and CSMs

One hundred sixty extracted caries-free, nonrestored third molars were obtained after receiving patients’ consent under a protocol approved by the Georgia Health Sciences University Human Assurance Committee (age range of patients, 18–33 years). These teeth were stored at 4°C in 0.9% NaCl containing 0.02% NaN₃ to prevent bacteria growth and used within 3 months after extraction. A 0.3-mm-thick tooth slice was obtained from the mid-coronal portion of each tooth by using a slow-speed saw (Isomet; Buehler Ltd, Lake Bluff, IL) under water cooling (Fig. 1A). A dentin beam (3 × 7 × 0.3 mm) was prepared from each tooth slice (Fig. 1B); the use of 160 teeth resulted in 160 dentin beams. Dental tubules in each prepared beam were oriented perpendicular to the 3 × 7 mm surface, the surface that was subsequently used for contacting the CSMs. Eighty beams were randomly assigned to 8 experimental groups (n = 10) to be placed in contact with the set CSMs for a designated time period. The other beams were used as controls for each of the 8 experimental groups (n = 10).

Biodentine and MTA Plus were mixed according to manufacturer’s instructions and placed in 3 × 7 × 2 mm silicone molds inside a 100% relative humidity chamber until set. For Biodentine, liquid from the single-dose container was emptied into the powder-containing capsule and triturated by using a capsule mixer for 30 seconds, with a final setting time of 1.2 hours. Two dentin beams were placed in contact with a set CSM block, with only 1 side of each beam exposed to the CSM (Fig. 1C, to simulate contact of the material with crown/root dentin in a clinical scenario).

The dentin-CSM assemblies were aged at 37°C in 100% relative humidity chambers for 24 hours, 1 month, 2 months, or 3 months (ie, 2 materials × 4 aging times = 8 experimental groups). The control for each experimental group consisted of dentin beams that were aged similarly in the absence of CSMs (ie, 2 materials × 4 aging times = 8 control groups). At each designated aging time, the beams were copiously rinsed with deionized water and immediately tested.

Figure 1. A schematic depicting (A) preparation of a tooth slice; (B) preparation of a dentin beam; (C) dentin beams in contact with set CSMs during aging in 100% relative humidity chamber; (D) flexing a beam to failure by using a uniaxial 3-point flexure design.

### Three-point Flexure

Flexural testing was performed by using a miniature 3-point flexure device with a 5-mm support span (25). The side of the dentin beam that was in contact with the CSMs was subjected to tension, whereas the noncontacting side was subjected to compression during flexural testing (Fig. 1D). Each 7-mm-long beam was placed on top of the support span and loaded to fracture under water by using a universal testing machine (Vitrodyne V100, Burlington, VT) at a cross-head speed of 1 mm/min.

Flexural strength (megapascals [MPa]) was calculated by using the formula \( \frac{3PL}{2bd^2} \). Flexural modulus (Gigapascal [GPa]) was calculated by using the formula \( \frac{L^3m}{4bd^3} \), where \( P \) = load at fracture, \( L \) = length of support span, \( m \) = slope of the initial straight-line portion of the load-deflection curve, \( b \) = beam width, and \( d \) = beam thickness. MOT (MPa) was calculated by converting the load-deflection data to stress-strain data and integrating the area under the stress-strain curve from the origin to the strain-to-fracture (26).

Toughness of a material is its ability to absorb energy in the plastic range of the material.

### Statistical Analyses

For each variable (flexural modulus, flexural strength, and MOT), 1-way analysis of variance was first used to compare whether differences exist for the data obtained from the 8 control groups at the 4 designated aging times, after testing for the normality (Shapiro-Wilk test) and equal-variance assumptions (modified Levene test) of the data.

Because there was no statistically significant reductions in flexural properties of the control specimens after aging in 100% relative humidity for up to 3 months (data not shown), the 24-hour control data obtained from the 2 CSMs were used as baseline data for comparison with the data derived from the 8 experimental groups. Data for each testing parameter were analyzed separately with 2-factor repeated-measures analysis of variance to determine the effects of material and aging time and the interaction of those 2 factors on flexural modulus, flexural strength, and MOT. Because the equal-variance assumption of the data set for MOT was violated, logarithmic transformation of the data was performed before analysis. Statistical significance for all analyses was preset at \( \alpha = 0.05 \).
**Results**

For flexural modulus, there was no significant difference for material ($P = .640$) or aging time ($P = .064$) when compared with baseline control (Fig. 2A). The interaction of those 2 factors was also nonsignificant ($P = .947$). For flexural strength, significant differences were associated with aging time ($P < .001$) but not with material ($P = .349$) (Fig. 2B). The interaction of those 2 factors was nonsignificant ($P = .585$). Dentin flexural strength for dentin exposed to Biodentine decreased significantly after 2 and 3 months, whereas that exposed to MTA Plus decreased significantly after 3 months of aging ($P < .05$).

For MOT, significant differences were observed for both material ($P < .004$) and aging time ($P < .001$) (Fig. 3). The interaction of those 2 factors was significant ($P = .02$). For the factor “material,” MOT of Biodentine was significantly different from that of MTA Plus after 1 month and 2 months ($P < .05$), but not after 3 months. For the factor “aging time,” MOT of dentin was significantly reduced after it was in contact with Biodentine after 1 month ($P < .05$). By contrast, there was no significant reduction in MOT of dentin after it was in contact with MTA Plus for up to 3 months ($P > .05$).

**Discussion**

Because there were significant reductions in the flexural strength and MOT of dentin after specimen beams were aged in direct contact with CSMs, the null hypothesis that there are no changes in flexural properties over time when the 2 CSMs are placed in direct contact with human dentin has to be rejected. The rationale for subjecting the CSM-contacting side of the dentin specimens to tensile stresses is that brittle materials are much weaker in tension than in compression (27). The advantage of a uniaxial flexure design is that a state of pure tension might be achieved on the lower side of the specimen, which is usually responsible for crack initiation in brittle materials (28). The values for strength and MOT reported in the present study are large with respect to values reported in the literature on flexure. This difference is due to (1) the 3-point load configuration and (2) the fact that the beam cross section (rectangular with large width-to-thickness ratio, versus square cross section) resulted in plane-strain stress distribution instead of pure uniaxial stress. Thus, the results generated from the present study are not directly comparable with those obtained from 3- or 4-point bending of specimens with square cross sections or those generated by using a biaxial flexural design.

Grigoratos et al. (4) previously reported that contact of dentin with saturated Ca(OH)$_2$ reduced the flexural strength but not the modulus of elasticity of dentin. Similar trends were observed in the present study when dentin was placed in contact with the CSMs. Although we had evaluated the effect of Ca(OH)$_2$ on dentin flexural properties, those results were not directly comparable with the data in this work and were not reported. This is because when the same experimental design was used, the fluidity of a Ca(OH)$_2$ paste (Calexpt; Nordiska Dental AB, Angelholm, Sweden) caused sink-in of the dentin beams, producing double-sided instead of single-sided contact.
Human dentin contains 70% mineral, 20% organic materials, and 10% water. Carboxylated apatite is the inorganic component of dentin, whereas the organic phase is predominantly type I collagen fibrils. This composition makes dentin more compliant than enamel, with a typical modulus of elasticity of 11–20 GPa versus 86 GPa for enamel (29, 30). The inorganic phase provides strength, whereas the organic phase is responsible for the toughness of dentin (31, 32). Overall, MOT of dentin was altered more (37.3% reduction for Biodentine, 22.3% reduction for MTA Plus) than its flexural strength (17.6% reduction for Biodentine, 14.0% reduction for MTA Plus). MOT should not be confused with fracture toughness. Fracture toughness is a property that describes the ability of a material containing a crack to resist fracture and is usually determined by subjecting compact tension specimens with a crack to a quasi-static tensile load (33, 34). Conversely, MOT is defined as the amount of energy per volume that a material can absorb before rupturing. The larger the area under a stress-strain curve, the tougher the material. A high MOT is important when a material is subject to stresses that exceed the elastic limit or during impact loads (26).

On the basis of the results, it might be concluded that the CSMs investigated in the present study reduce dentin’s ability to resist deformation (strength) and to absorb energy without fracturing (toughness). This is clinically significant because CSMs such as MTA were found to reduce the risk of root fractures by avoiding long-term Ca(OH)2 treatment. Placement of these CSMs will probably not adversely affect the fracture resistance of roots with a short apical CSM plug or a thin layer of CSM as pulp-capping material. However, the practice of completely obturating root canals with these new CSMs or their use as dentin substitutes should be carefully considered. Hatibovic-Kofman et al (21) attributed the improved fracture resistance of extracted immature sheep teeth treated with MTA over Ca(OH)2 to the ability of MTA to induce expression of tissue inhibitor of metalloproteinase-2 (TIMP-2). The latter prevents destruction of the collagen matrix by matrix metalloproteinases MMP-2 and MMP-14. However, such an explanation is questionable because it is difficult to justify how TIMP-2 expression could be altered in extracted sheep teeth in the absence of live cellular components. It is known that MTA-like materials can dissolve bioactive matrix components from mineralized dentin because of its high pH after setting (35). This could also have resulted in dissolution of collagen from the surface of the mineralized dentin. Because dentin toughness is attributed to its collagen matrix, the amount of collagen extracted from mineralized dentin and changes in collagen ultrastructure should be further examined after exposure of these materials to dentin.

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References