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 ($7 \times 3 \times 0.3$ 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 ($\alpha = 0.05$). **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).

Calcium silicate–based materials (CSMs) such as mineral trioxide aggregate (MTA) have largely replaced $Ca(OH)_2$ as endodontic repair materials because of their superior seal, biocompatibility, and regenerative capabilities (11–13). Their antibacterial properties are attributed to its release of $Ca(OH)_2$ on surface hydrolysis of the calcium silicate components (11, 14). When applied as a dentin substitute or during pulp capping, pulpotomy, and apexification, CSMs are left in place for the life of the tooth. There are also indications for obturating the entire canal with CSMs (15–18). An early study reported that prolonged contact of root dentin with $Ca(OH)_2$ or MTA resulted in similarly severe reductions (32% versus 33%) in dentin fracture resistance (6). The results of more recent studies are generally of the consensus that immature roots are less susceptible to fracture when $Ca(OH)_2$ is replaced by CSMs after prolonged contact with root dentin (19–22).

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

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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% NaN_3 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. 1*A*). A dentin beam (3 × 7 × 0.3 mm) was prepared from each tooth slice (Fig. 1*B*); the use of 160 teeth resulted in 160 dentin beams. Dentinal 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 10 minutes. MTA Plus was hand-mixed by using a 3:1 powder-liquid ratio to achieve a putty-like consistency, 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. 1*C*), 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.

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. 1*D*). 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 crosshead speed of 1 mm/min.

Flexural strength (megapascals [MPa]) was calculated by using the formula $3PL/2bd^2$. Flexural modulus (Gigapascal [GPa]) was calculated by using the formula $L^3m/4bd^3$, 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 $\alpha = 0.05$.

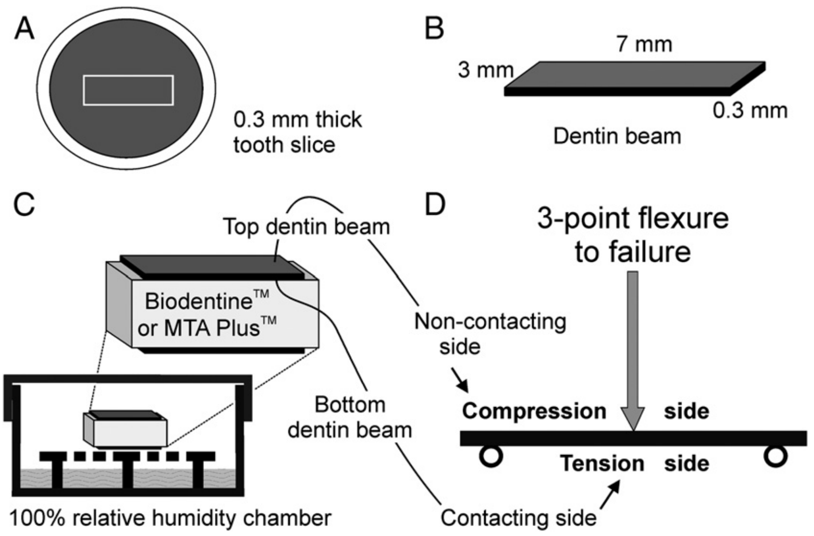

Figure 1. A schematic depicting (*A*) preparation of a tooth slice; (*B*) preparation of a dentin beam; (*C*) dentin beams in contact with set CSMs during aging in 100% relative humidity chamber; (*D*) flexing a beam to failure by using a uniaxial 3-point flexure design.