Effect of Various Bleaching Agents on the Surface Composition and Bond Strength of a Calcium Silicate-based Cement

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Clinical Relevance
We recommend the use of calcium silicate-based cement as a cervical barrier when performing intracoronal bleaching using a mixture of sodium perborate with water or 3% hydrogen peroxide.

ABSTRACT
This study aimed to evaluate the morphological and elemental changes that occur on the surface of calcium silicate-based cement (CSC) and to analyze the bond strength of composite resin to CSC after application of various bleaching agents. One hundred twenty-five CSC blocks (Biodentine) were prepared and randomly divided into five groups according to the bleaching agent applied over the material surface (n=25): SP-DW (sodium perborate–distilled water mixture), SP-HP (sodium perborate–3% hydrogen peroxide [H₂O₂] mixture), CP (37% carbamide peroxide gel), HP (35% H₂O₂ gel), and a control group (no bleaching agent). After 1 week, scanning electron microscopy provided an analysis of the surface morphology and elemental composition for 10 specimens from each group. Composite resin was placed at the center of each cement surface in the remaining specimens (n=15). A universal testing machine determined shear bond strength (SBS) and fracture patterns were identified with a dental operating microscope. Data were analyzed using one-way analysis of variance and Tukey HSD tests. The cement surface in the CP and HP groups presented changes in structure and elemental distribution compared with the remaining groups. The former groups exhibited a decrease in the calcium level and an increase in the silicon level and presented significantly fewer SBS values than the remaining groups (p<0.05). Most failures were adhesive in the CP and HP groups, while they were predominantly cohesive in the remaining groups. The bleaching agents with higher concentration induced deterioration of the cement surface.
and negatively affected the bond strength of the composite resin to CSC. The use of CSC is recommended as a cervical barrier when intracoronar bleaching is performed with a mixture of sodium perborate with water or 3% H$_2$O$_2$.

**INTRODUCTION**

Intracoronar bleaching is a conservative and cost-effective procedure to improve the esthetics of discolored endodontically treated teeth. It involves insertion of a bleaching agent into the pulp chamber, which is then sealed using a temporary restoration between visits.

Several bleaching agents are used for intracoronar bleaching, including a mixture of sodium perborate and water, carbamide peroxide, hydrogen peroxide (H$_2$O$_2$), or a combination of sodium perborate and H$_2$O$_2$. These agents typically accomplish bleaching through the release of H$_2$O$_2$ and diffusion of reactive oxygen through dentinal tubules, which then oxidizes the pigments and reverses the chromatic alteration of dental tissues. However, H$_2$O$_2$ has the capacity to denature dentin, which leads to a slightly acidic environment that may induce osteoclastic activity and invasive cervical resorption. To prevent such occurrences, the placement of a cervical barrier before intracoronar bleaching is recommended.

The cervical barrier should be impermeable in order to prevent intraradicular and extraradicular diffusion of the peroxides. Recently, mineral trioxide aggregate (MTA), a calcium silicate–based cement (CSC), has been evaluated as a cervical barrier during intracoronar bleaching procedures. Despite possessing several desirable properties such as superior sealing ability, good mechanical properties, and biocompatibility, MTA presents certain notable shortcomings such as a long setting time and tooth discoloration. In recent years, another CSC, Biodentine, has drawn attention because of its good mechanical properties, high biocompatibility and bioactivity. And owing to its color stability, this cement is recommended for use as a dentin substitute under composite resin restorations and in esthetically sensitive areas.

There is no available data on the use of Biodentine as a cervical barrier during intracoronar bleaching. The effects of bleaching agents on the Biodentine surface and adhesion between Biodentine and a composite resin restoration warrant further investigation. Therefore, the aim of this study was to evaluate the morphological and elemental changes that occur on the Biodentine surface and to analyze the bond strength of composite resin to Biodentine after application of various bleaching agents.

**METHODS AND MATERIALS**

**Specimen Preparation**

One hundred twenty-five acrylic blocks were prepared in silicone having an internal diameter of 5 mm and height of 7 mm. To obtain the blocks, autopolymerizing acrylic resin (Meliodent, Heraeus Kulzer, Armonk, NY, USA) was mixed according to the manufacturer's instructions and placed in the silicone tubes. After polymerization, a cavity with a diameter of 3 mm and height of 1.5 mm was prepared with a fissure bur (Dentsply Maillefer, Ballaigues, Switzerland) at the center of each acrylic block. Biodentine (Septodont, St-Maur-des-Fossés, France) was mixed according to the manufacturer's instructions and placed in the cavities using a spatula.

The specimens were stored for 72 hours at 37°C and 100% humidity. All prepared surfaces were polished with a 400-grit silicone carbide abrasive disc (Buehler, Lake Bluff, IL, USA) using a polishing device (Mecapol P230, Presi, France) for 60 seconds to create a standard layer. The specimens were randomly divided into five groups (n=25) according to the bleaching agent, applied to the surface of the cement as follows (from lower to higher H$_2$O$_2$ release):

- **Control group**—No treatment
- **SP-DW group**—Sodium perborate powder mixed with distilled water in a ratio of 2:1 g/mL
- **SP-HP group**—Sodium perborate powder mixed with 3% H$_2$O$_2$ solution in a ratio of 2:1 g/mL
- **CP group**—37% carbamide peroxide gel (Whiteness Super-Endo, FGM Products, Joinville, SC, Brazil)
- **HP Group**—35% H$_2$O$_2$ gel (Opalescence Endo, Ultradent Products, South Jordan, UT, USA)

Following the preparation, the bleaching agents were placed in silicone tubes with an internal diameter of 5 mm and height of 2 mm. Then, the tubes with the bleaching agents were placed on the cement surfaces. The top surface of each bleaching agent was covered with a cotton pellet and a temporary filling material (Cavit-W, 3M ESPE, Seefeld, Germany). Each tube with the bleaching agent and block with CSC were taped together to prevent separation during storage. All specimens were stored for 1 week at 37°C and 100% humidity.
Thereafter, each tube with the bleaching agent was removed and the cement surface was cleaned with distilled water for 1 minute and air-dried.

**Scanning Electron Microscopy Analysis**

Ten specimens from each group were coated with gold using a vacuum evaporator (JEE-400; JEOL Ltd, Tokyo, Japan) and examined with a scanning electron microscope (SEM) (JEOL 6400, JEOL Corp, Tokyo, Japan) fitted with an energy dispersive spectroscopy (EDS) system (EMAX-7000 Type S; Horiba Ltd, Kyoto, Japan) to characterize the microstructural surface morphology and to calculate the weight percentages of the elements Ca, Si, Mg, Na, K, Al, Zr, and P. To standardize the examined area of each sample, the central beam of the SEM was directed to the center of the specimen under 10× magnification, which was gradually increased to 1000×.

**Placement of Restorative Material**

A composite resin system (Ice, SDI, Bayswater, Victoria, Australia) was applied over the remaining samples (n=15) according to the manufacturer’s instructions. Initially, each cement surface was etched with 37% phosphoric acid (Super Etch, SDI) for 20 seconds and washed with water for 15 seconds. Then, the surfaces were dried with an oil-free air spray to remove excess water. The bonding agent (Stae, SDI) was applied with a microbrush to the surfaces, which were then air-dried for 5 seconds. The bonding agent was cured using a light-emitting diode light-curing unit (Mini LED, Acteon, Mount Laurel, NJ, USA) with an output of 1000 mW/cm² for 10 seconds. Cylindrically shaped plastic tubes, with a 2-mm internal diameter and 2-mm height, were placed at the center of the cement surface, and composite resin was condensed into each tube and polymerized for 40 seconds with the light-curing unit. After the polymerization process, the tubes were cut with a scalpel and carefully removed. The specimens were stored at 37°C in 100% humidity for 24 hours.

In this study, a single operator performed the preparation and placement of the CSC and bleaching agents; a second operator, who was blinded to the groups, placed the restorative material.

**Shear Bond Strength Test**

The shear bond strength (SBS) test was performed using a knife-edge blade in a universal testing machine (Instron, Model 1334, Instron, Canton, MA, USA) with a crosshead speed of 1 mm/min. The load at failure was recorded in newtons, and the bond strength was calculated in megapascals (MPa) by dividing the load at failure by the adhesive surface area. Failure modes were evaluated under a dental operating microscope at 20× magnification and categorized into one of three failure types: adhesive failure at the CSC and composite resin interface, cohesive failure within the CSC or composite resin, or mixed failure composed of adhesive and cohesive failure of the materials (Figure 1a-c).

**Statistical Analysis**

Data were analyzed using one-way analysis of variance and Tukey HSD tests. The level of significance was set at α=0.05.

**RESULTS**

In the SEM examination, platelike, globular, and rodlike structures were observed on the surface of the control group (Figure 2a). The SP-DW group showed clusters of globular and cubic crystals with air bubbles in some places (Figure 2b). In the SP-HP group, globular and rodlike structures were observed with air bubbles throughout the surface (Figure 2c). There were numerous crack lines and “woodpecker holes” on the cement surface in the CP (Figure 2d) and HP groups (Figure 2e).

Table 1 shows the results of element analysis detected in each group. The Ca and Si values of the SP-DW and SP-HP groups were similar to those of the control group (p>0.05). The Ca values of the CP and HP groups were significantly lower, while the Si values were higher than were the remaining groups (p<0.05).

The mean (±SDs) of the SBS values of the groups and the modes of failure of the specimens after the SBS test are presented in Table 2. There was no statistically significant difference between the CP and HP groups (p>0.05), while they showed significantly lower SBS values than did the remaining groups (p<0.05). The SP-DW, SP-HP, and control groups presented similar SBS values (p>0.05). Inspection of the samples revealed the bond failure to be predominantly cohesive for the SP-DW, SP-HP, and control groups and adhesive for the CP and HP groups.

**DISCUSSION**

Numerous clinical reports using several bleaching agents have revealed that there is no ideal protocol for intracoronal bleaching of endodontically treated
However, it is clear that the aim should be to maximize bleaching efficacy while minimizing damage to adjacent tissues. Sodium perborate with water has been reported as the safest bleaching agent owing to its low hydroxyl radical diffusion. However, in case of severe discoloration, the use of bleaching agents that generate greater hydroxyl radical penetration may be indicated. In this regard, a balance might be achieved by decreasing the change frequency or application time of the bleaching agent while increasing the concentration. Considering that the use of each agent may have a clinical indication, bleaching agents that release H₂O₂ at varying concentrations were tested in the present study.

SEM images obtained in the present study indicate that the effects of a bleaching agent on the surface morphology of CSC were concentration dependent. The cement surface presented various defects such as cracks and holes after application of 37% carbamide peroxide and 35% H₂O₂. On the other hand, the cement surface in the SP-DW and SP-HP groups presented mainly platelike, globular, cubic, and rodlike structures as in the control group. These findings are consistent with those of a previous study wherein MTA was used as a cervical barrier. H₂O₂ may produce bubbling when in contact with the surface of CSC; this oxygen bubbling could be the reason for the more porous surface in the SP-HP group compared with the control and SP-DW groups. When in contact with moisture, carbamide peroxide decomposes into one-third H₂O₂ and two-thirds urea. Accordingly, approximately 10%-15% H₂O₂ was released in the CP group. With the increase in H₂O₂ release, the cement surface seemed to be disturbed more because larger defects were evident on the surfaces of the CP and HP groups. Of note, cracks and holes on the disturbed surface may interfere with the mechanical and adhesive properties of the material.

Reportedly, the pH of 35% H₂O₂ gel and 35% carbamide peroxide gel is 3.7 and 6.5, respectively, while mixtures of sodium perborate with water or H₂O₂ have a pH of >7.4 when prepared in a 2-g/mL ratio. According to the EDS analysis conducted in the present study, the Ca/Si ratio of the cement surface decreased in the CP and HP groups compared with the control group, while it remained stable in the SP-DW and SP-HP groups. The acidic condition of the bleaching agents in the CP and HP groups may induce the release of Ca ions and lead to an increase in the relative concentration of Si. In line with the present findings, Tsujimoto and others reported that the acidic conditions induced by bleaching agents resulted in deterioration of the MTA surface and caused a marked decrease in the Ca/Si ratio. It is worth mentioning that the pH of bleaching agents increases with time, probably because of the decomposing of acidic H₂O₂ radicals into oxygen and water. However, the initial pH levels of the bleaching agents seem to affect the
micromorphology and elemental distribution of the cement surface. Other elements detected by the EDS analysis could be from residue of the bleaching agents or the material itself after exposure to the agents.

The bond strength between restorative and underlying materials is important for optimal quality of the filling as well as the success of restorations. Several studies have evaluated the bond strength between Biodentine and composite resin materials. However, the bond strength between Biodentine and composite resin after application of various bleaching agents remains unclear. In the present study, the application of a mixture of sodium perborate with either water or 3% H₂O₂ had no significant effect on the bond strength of composite resin to Biodentine. However, adhesion between the two materials was negatively affected after the placement of either 37% carbamide peroxide or 35% H₂O₂ gel over the cement surface. One might speculate that the bond strength between CSC and composite resin is affected by morphological changes in the surface after application of bleaching agents at high concentrations. The deteriorated surface observed in the CP and HP groups could interfere with micromechanical attachment, thereby resulting in decreased bond strength to composite resin. Furthermore, breakdown of the chemical union could be another reason for the poor bond strength results. Although it is unknown whether chemical adhesion exists between CSC and composite resin, it is possible that resin monomers bind chemically to Ca in CSC. Therefore, the distinctly lower Ca levels in the CP and HP groups could be another explanation for the negative impact on the bond strength.

Bond failures observed in the CP and HP groups were predominantly adhesive, while these were predominantly cohesive in the remaining groups. Interestingly, in a recent study, no adhesive failure was observed at the CSC or composite resin interface. Based on the present findings, the application of bleaching agents at high concentrations may affect the bond strength between CSC and composite resin as well as the failure type. Clinically, this finding should be viewed with caution as the bond could be more acceptable when fracture occurs inside each material rather than in the bonded interface.
CONCLUSIONS

Bleaching agents applied at higher concentrations negatively affected the micromorphology of the CSC surface and the bond strength between CSC and composite resin. Nevertheless, the use of CSC can be recommended as a cervical barrier when intracoronal bleaching is performed with a mixture of sodium perborate with water or 3% H₂O₂.

Conflict of Interest

The authors deny any conflicts of interest related to this study and certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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