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# Influence of Immediate Coronal Restoration on Microhardness of CEM Cement: An *In Vitro* Study

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**ABSTRACT**

**Introduction:** Coronal restoration could affect the setting reaction of the underlying CEM cement. The aim of the present study was to evaluate the effect of immediate coronal restoration placement on the subsurface microhardness of CEM cement. **Methods and Materials:** In 50 extracted human mandibular molars, access cavities were prepared and CEM cement was placed in the pulp chamber at a 3-mm thickness. Samples were divided into ten groups ($n$=5). CEM cement was placed and after 10 min, two groups were restored with Zonalin temporary restoration and eight groups were restored with glass ionomer cement (GIC), resin modified glass ionomer (RMGI), resin based composite and amalgam respectively. Vickers microhardness number (VHN) of CEM cement was measured in two time intervals (7- and 21-days). Data was analyzed with SPSS and two-way analysis of variance and Bonferroni tests. Level of significance was set at the 5%. **Results:** The mean VHN of CEM cement showed statistically significant differences only between Zonalin and amalgam groups ($P$=0.021). There were also significant differences considering the effect of time ($P$=0.042) and material ($P$=0.046). Although the effect of time-material on the microhardness values showed no statistically significant differences ($P$=0.636). **Conclusion:** Based on the results of the present study, immediate placement of final restorations affects the setting reaction in underlying CEM cement. Therefore, sufficient moist curing and hydration should be guaranteed before placement of the coronal restoration.

**Keywords:** Calcium-Enriched Mixture; CEM Cement; Dental Restoration; Setting Time; Vickers Microhardness

## Introduction

Vital pulp therapy (VPT) is a conservative treatment approach that aims to remove infected dentin and maintain pulpal vitality with application of a biocompatible material with a good sealing ability [1]. Bioceramic materials with excellent biocompatibility, antibacterial and osseo-conductive property, good sealing, insolubility in tissue fluids, chemical bond to the tooth structure and acceptable radio opacity are widely used in VPT cases [2]. Calcium silicate based cements (CSCs) such as Biodentine, EndoSequence and calcium-enriched mixture (CEM) cement have been introduced to overcome the limitations of mineral trioxide aggregate (MTA) in clinical conditions [3, 4]. During the clinical application of bioceramics, these materials are in direct contact with blood and body fluids in the radicular part and restorative materials in the coronal part of the teeth. In cases that bioceramics are exposed to dislocating forces such as condensational and masticatory forces, mechanical seal is also an important concern alongside the biologic seal [5]. Many factors such as bioceramic final hardness and material thickness could affect the mechanical seal of bioceramics [6].

CEM cement is a hydrophilic endodontic cement with similar pH and sealing ability and decreased working time and film thickness in comparison to MTA [7, 8]. The clinical application of this cement showed better results in pulp capping, pulpotomy of permanent molars, apexogenesis and internal root resorption [9, 10]. CEM cement comprises water soluble calcium and phosphate which forms hydroxyapatite crystals during and after setting [6]. CEM cement is a water-based cement and the presence of moisture could accelerate the setting process which lead to hermetic seal and proper final hardness [11, 12].

Following VPT, the coronal cavity over the bioactive materials should be restored with leakage free reparative materials. In these cases, restorative materials could interact with the setting process of inferior cement through condensation forces and hydration process [13, 14]. Forces resulting from the placement of the restorative materials may lead to dislodgment of bioceramic and hamper the mechanical seal [15].

Acid etching and formulation of solvent in the adhesives during the application of composite resins, hydrophilic nature of resin modified glass ionomer (RMGI) and glass ionomer cement (GIC) and finally condensation pressure at the time of amalgam restoration placement could affect the setting reaction and hardening process in the inferior cement and disrupt the hermetic seal [13].

Another important factor in this regard is the time of coronal restoration placement. Immediate coronal seal with permanent restoration leads to less microleakage and promote the treatment success [16]. Limited studies with contradictory results are available according to the relation between immediate coronal restoration placement and setting reaction in MTA [17, 18], and no study is present according to CEM cement. The aim of the present study was to investigate the effect of immediate placement of four final restorative materials (Composite resin, GIC, RMGI and amalgam) in comparison with Zonalin temporary restoration on CEM cement microhardness.

## Materials and Methods

### *Sample preparation*

In this *in vitro* experimental study, 50 extracted human mandibular molars with minimally destroyed coronal structure and no coronal restoration, root resorption, canal calcification and endodontic treatment, were included. Teeth were stored in 0.5% Chloramine-T at room temperature before sample preparation. Access cavity was prepared and pulp chamber was rinsed with 5.25% sodium hypochlorite followed by normal saline. The root canals were filled with normal saline up to the orifices.

CEM cement (Bioniquedent Co., Tehran, Iran) powder and liquid parts were mixed according to the manufacturer's instruction. Powder was mixed with the liquid for 15-30 sec for hydration of all powder particles and turns the material into a thick consistency. Using a plastic instrument, CEM cement paste was placed in the pulp chamber at a 3 mm thickness. CEM cement mass was adapted to the cavity walls using gentle movements of a dry cotton pellet. A moistened cotton pellet was placed on CEM cement surface and teeth were randomly divided into ten groups ($n$=5) according to the coronal restoration:

Control groups (groups 1 and 2): A 2-mm layer of temporary filling material (Zonalin, Golchai, Iran) was placed on the wet cotton pellet after 10 min. Teeth were then maintained in an incubator at 37°C and 100% relative humidity for 7 and 21 days (groups 1 and 2, respectively).

Amalgam groups (groups 3 and 4): Cotton pellet was removed after 10 min and a 2-mm layer of amalgam filling material (SDI, GS 80, Bayswater, Victoria, Australia) was condensed over the CEM cement layer using an appropriate pear-shape condenser by gentle pressure. Teeth were then stored in an incubator at 37°C and 100% relative humidity for 7 and 21 days (groups 3 and 4, respectively).

RMGI groups (groups 5 and 6): Cotton pellet was removed after 10 min. Standard powder to liquid ratio of RMGI (GC FUJI II LC: Tokyo, Japan) was mixed according to the manufacturer's instruction and placed on CEM cement with a 2 mm thickness. RMGI was cured for 20 sec using a LED curing light (Demi TM Plus, Kerr, California, USA). Samples were then stored in an incubator at 37°C and 100% relative humidity for 7 and 21 days (groups 5 and 6, respectively).

GIC groups (groups 7 and 8): Cotton pellet was removed after 10 min. Standard powder to liquid ratio of GIC (GC Fuji IX, GC Corporations, Tokyo, Japan) were mixed correctly and placed on CEM cement at a 2 mm layer. After the completion of setting, samples were maintained in an incubator at 37°C and 100% relative humidity for 7 and 21 days (groups 7 and 8, respectively).

Resin composite groups (groups 9 and 10): Cotton pellet was removed after 10 min. The self-etch primer and bonding agent of Clearfil SE Bond (Kuraray, Okayama, Japan) were applied according to the manufacturer's instruction. Afterward flowable composite resin (Clearfil Majesty Flow A3 shade, Kuraray, Okayama, Japan) was placed on CEM cement at a 2 mm thickness and cured for 40 sec using a LED curing light. Teeth were then stored in an incubator at 37°C and 100% relative humidity for 7 and 21 days (groups 9 and 10, respectively).

### *Vickers microhardness (VMH) testing*

Following the maintenance period in the incubator (7 and 21 days), samples were mounted in a custom-made mold using self-cured acrylic resin (Asia Chemi Teb Co., Tehran, Iran). Teeth blocks were then sectioned longitudinally using a low-speed saw and polished with silicon carbide paper (300 to 1200 grit). VMH testing was performed using a Vickers microhardness tester (FM700 series, Future-Tech Corp., Tokyo, Japan) in three points at a 200 µm distance from CEM-filling material interface using a 5 gram-force load and a 5-sec dwell time. The angle between the opposite faces of diamond indenter was 136°. The diameter of indention was measured in each point and mean value of the three measurements was recorded as Vickers number of each sample.

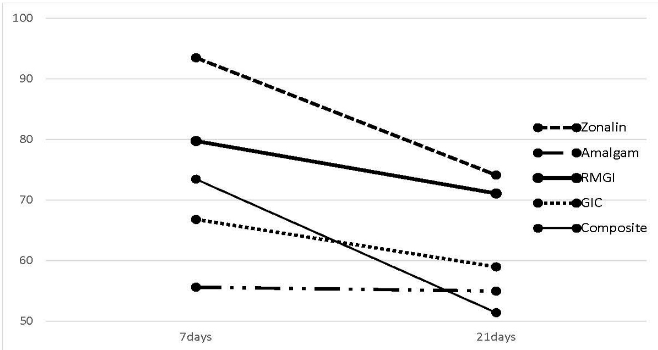


***Figure 1.***Vickers microhardness test results for each group at the two time intervals

***Statistical analysis***

Data were analyzed using SPSS Version 20 (SPSS Inc, IL, USA). The effect of filling material and time on CEM cement microhardness were evaluated through two-way ANOVA and Bonferroni tests. The significant level was set at 0.05.

## Results

The results of the present study are presented in Table 1 and Figure 1. Considering the effect of time (*P*=0.042) and material (*P*=0.046), there were significant differences between the groups. Although the effect of time-material on CEM cement microhardness showed no statistically significant differences (*P*=0.636). Based on Bonferroni analysis, the mean VHN of CEM cement showed statistically significant differences only between Zonalin and amalgam groups (*P*=0.007). The VHN measurements showed significant reduction from 7 days to 21 days.

The highest reduction of VHN values during the experimental period was recorded for resin composite group, followed by Zonalin, RMGI, GIC and amalgam respectively.

## Discussion

In the clinical conditions that biomaterials like CEM cement are placed in direct contact with coronal restoration, the setting reactions of the restorative material could interfere with the hardening process of the underlying layer of CEM cement, especially in the cases with immediate placement of coronal restoration [17, 19]. Any factor that affects the setting process of CEM cement could hamper CEM cement microhardness and physical seal [20]. In the cases of immediate coronal restoration placement, clinical procedures such as condensation pressure, etching, rinsing and priming all could affect the setting process of CEM cement. The effect of coronal restoration timing on MTA surface microhardness has been assessed in a few studies [17, 19, 21, 22].

Tsujimoto *et al.* [19] evaluated the relationship between time of resin composite placement and MTA microhardness. Flowable composite resin was placed over MTA after 10 min, 1 day and 7 days. Based on the results of the study, Vickers microhardness values were significantly lower in the 1-day group and the highest VHN was recorded for 10-min group.

In comparison to MTA, the setting time of CEM cement is shorter (4 h *vs.* 1 h) [23]. Therefore, immediate final restoration placement may not have effect on the setting process of the inferior CEM cement. Since the impact of immediate coronal restoration placement on CEM cement microhardness have not been evaluated up to the present, this survey was conducted to assess the effect of four final restorative materials in comparison with Zonalin temporary restoration on CEM cement microhardness.

Based on the results of the present study, the microhardness values showed significant reduction from 7 days to 21-day time intervals. Considering the effect of material, there were significant differences in hardness values only between Zonalin and amalgam groups.

Kazemipoor *et al.* [17] in a similar study have evaluated the effect of immediate coronal restoration placement on MTA microhardness. They have concluded that time didn't significantly affect the MTA microhardness. In contrast, the effect of material on microhardness was recorded significant. The highest and the lowest mean microhardness values were recorded for Zonalin-RMGI and resin composite, respectively. The differences between the two surveys may be attributed to the setting reactions occurred in the two bioceramics. The differences in the chemical composition, shape, size and distribution of

***Table1.*** Mean (SD) of CEM cement microhardness values at the two time intervals. († One-way analysis of variance)

| | **7 days** | **21 days** |
|---|---|---|
| **Zonalin** | 93.44 (21.44) | 74.08 (9.33) |
| **Amalgam** | 55.56 (29.20) | 54.94 (11.64) |
| **RMGI*** | 79.70 (17.31) | 71.04 (19.75) |
| **GIC**** | 66.76 (20.67) | 58.94 (10.53) |
| **Flowable resin composite** | 73.42 (9.75) | 51.36 (13.78) |
| ***P*-value†** | 0.089 | 0.058 |

**Resin modified glass ionomer; **Glass ionomer cement* hydroxyapatite crystals that formed following setting reactions may explain the different hardness values obtained in two studies [23]. High percentage of small particles (0.5-2.5 µm) in CEM cement and formation of hydroxyapatite crystals at the surface of the filling area, alongside the different loading forces and dwell time, are some reasons for lower hardness values recorded in the study by Kazemipoor *et al.* [17] for MTA in comparison with CEM cement [24, 25].

Various factors could affect the setting reaction in CEM cement. Although increased thickness of bioceramics resulted in better bacterial resistance and seal, the moisture supplies for setting reaction remains a concern [26]. Rahimi *et al.* [6] have concluded that 3-mm thickness of CEM cement (as applied in the present study) had the most effective sealing ability in the conditions of one-sided moisture exposure.

Another important factor is the pH value of the environment. The formation and growing of needle-like crystals between the cubic crystals increase the final microhardness of MTA [27]. Acidic environment prevents the formation of these needle-like crystals that leads to a decrease in MTA microhardness [27]. Lower pH values also could affect the final microhardness of CEM cement *via* two mechanisms.

CEM cement contains calcium silicate and calcium carbonate crystals that undergo dissolution in an acidic environment [28]. Also CEM has an endogenous source of calcium and phosphate to produce apatite crystals that could affect both the material seal and hardness [24]. An acidic condition may decline this internal source of phosphate ions that interfere with the formation of apatite crystals and final material microhardness. In the present study we have applied a non-phosphate fluid (normal saline) instead of PBS, because CEM cement has an endogenous source of phosphate.

Concerning the results of the present study, condensing forces, pH and hydration supply play important roles in setting reaction and final strength of CEM cement.

It seems that in the early phase of setting reaction, the impact of condensation forces is higher than pH and hydration supply because the lowest CEM cement hardness belonged to amalgam restoration group. Condensation forces decrease the remaining spaces between cement particles and water molecules that could interfere with the hydration reaction [29]. Also, it makes changes in the CEM cement powder to liquid ratio, decrease the porosities and micro canals that is responsible for the hydration process, setting reaction and compressive strength of the material [30].

Loxely *et al.* [31] indicated that immersion of MTA in saline solution, for seven days may lead to solidification of remaining unreacted mineral oxides after additional hydration supply. Based on the results of the present study in the early phase of the setting reaction, hydration of the material is of greater importance in comparison with pH factor. Therefore, between 7 days groups Zonalin showed the highest VHN values, followed by RMGI, resin composite with hydrophilic primer and GIC. GIC showed the highest water absorption during the first day of setting process [32].

In 21-days resin composite groups showed the lower VHN values in comparison to GIC. Also the presence of phosphate functional monomer (GPDM) in the self-adhesive flowable composites may interact with the calcium ions in CEM cement and affect the setting process [33].

## Conclusion

Since the immediate placement of the coronal restoration could interfere with the setting reaction and final hardness of CEM cement, it is better to postpone the final restoration after the completion of the setting process of the underlying CEM cement.

In the clinical conditions that coronal seal with a permanent restoration is recommended, RMGI restorative material with the least interaction to CEM cement setting process may be a good choice.

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Conflict of Interest: 'None declared'.

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