

## Effect of various types of glass ionomers on the microhardness of CEM Cement

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### Abstract

**Objective:** This study aimed to compare the effects of different types of glass ionomer cement (GICs), including conventional GIC, resin-modified GIC (RMGIC), and syringe-applied RMGIC (Ionoseal), on the microhardness of calcium-enriched mixture (CEM) cement.

**Methods:** Forty CEM cement samples were prepared. The study included eight groups (n=5), based on the type of GIC applied over CEM (conventional GIC, RMGIC, Ionoseal, or no coating) and the timing of application (immediate or after a 5-minute moisture application using a wet cotton pellet). Samples were incubated at 37°C for 7 days, then subjected to Vickers microhardness testing. Two-way ANOVA was used for the data analysis ( $\alpha=0.05$ ).

**Results:** There was no significant difference in CEM cement microhardness among the immediate restoration groups ( $P=0.4$ ). After exposure to wet cotton pellet, all GIC groups exhibited significantly higher microhardness than the control group ( $P<0.05$ ). Furthermore, samples coated with conventional GIC showed significantly greater microhardness than those coated with RMGIC and Ionoseal ( $P<0.05$ ). Delayed restoration significantly increased microhardness in the conventional GIC group ( $P = 0.001$ ), while no significant changes were detected in the RMGIC or Ionoseal groups ( $P>0.05$ ).

**Conclusions:** In the case of immediate restoration, all types of GIC support CEM cement setting without compromising its microhardness. For delayed restorations, conventional GIC offers superior results in enhancing CEM cement microhardness. If RMGIC or Ionoseal is used for coating, placing a wet cotton pellet on CEM for 5 minutes is not recommended, as it does not improve CEM cement microhardness.

**Keywords:** CEM cement, Glass ionomer, Ionoseal, Microhardness, Regenerative pulp treatment, Resin-modified glass ionomer

### Introduction

Vital pulp therapy (VPT) is a reliable approach for preserving and maintaining pulp tissue that has been compromised, but not destroyed, by extensive caries, trauma, restorative procedures, or iatrogenic factors (1). Reported success rates for VPT range from 81% to 90% in permanent teeth with symptomatic irreversible pulpitis (2), while in primary teeth, success rates range from 82.6% to 94% (3). Mineral trioxide aggregate (MTA) is considered the gold standard material for pulp capping, pulpotomy, surgical root-end filling, root

perforations, and revascularization procedures (4). MTA is biocompatible and demonstrates both inductive and conductive properties for hard tissue formation (5). However, its clinical use is limited by several drawbacks, including high cost, difficult handling, long setting time, and potential for tooth discoloration (6).

Calcium-enriched mixture (CEM) cement is a newer bioactive material that offers favorable handling, chemical stability, high alkalinity, antimicrobial activity, color stability, and bio-sealing ability (7). Its main components include calcium oxide (51.75%), sulfur trioxide (9.53%), phosphorus pentoxide (8.49%), and silica (6.32%), along with trace amounts of other elements (8). When mixed with water, CEM generates an environment rich in calcium and phosphate ions. It is applied in various endodontic procedures, including root-end surgery, perforation repair, and vital pulp therapy (9). CEM sets in less than one hour, has greater flow and lower film thickness than MTA, and promotes hydroxyapatite formation in saline (8, 10).

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The accepted clinical method regarding restoration timing over CEM cement varies. Manufacturers recommend placing a moist cotton pellet over CEM after its application, followed by temporary cavity restoration. This traditional protocol delays the final coronal restoration to allow CEM cement to hydrate and gain maximum mechanical strength, which can extend up to 24 hours or even longer for optimal properties (11).

Recent studies suggest that a minimum delay period of around 5 to 15 minutes provides significant improvement in CEM cement's surface properties before placing glass ionomer or resin-modified glass ionomer restorations (12). Longer delays (e.g., 30 minutes to several hours) may further enhance the setting and bond strength, but are less practical for typical clinical workflow. A one-session approach where final restoration is placed immediately after CEM application is attractive for reducing chair time and improving patient comfort, especially in pediatric cases (13). However, this may compromise the cement's physical properties unless moisture conditions are carefully controlled or specific restorative materials are used.

GICs are commonly applied in clinical practice as liners, bases, and even as definitive restorative materials due to their physical properties and low thermal expansion coefficient, which closely resemble those of dentin materials (14, 15). They form a chemical bond with dentin and release fluoride ions that aid in hard tissue remineralization (16). To enhance their mechanical strength and control the setting process, resin-modified glass ionomer cements (RMGICs) were developed by adding organic monomers and photoinitiators to the traditional GIC formulation (16).

Ionoseal is a specific type of RMGIC designed for ease of use, especially in hard-to-reach areas. It is supplied with a Non-Dripping Technology (NDT) delivery system, which helps reduce material waste during application (17).

The setting quality of calcium-silicate-based biomaterials, such as CEM cement, can be evaluated by measuring surface microhardness. Previous studies have shown that applying conventional GIC or resin-modified GIC (RMGIC) immediately after CEM does not negatively affect the long-term microhardness of either material (12). However, limited evidence exists regarding the performance of newer glass ionomer types and the optimal timing for their application. Therefore, this in vitro study aimed to compare the effects of placing conventional GIC, RMGIC, and Ionoseal

either immediately or after a delayed period following CEM cement application on the surface microhardness of CEM cement.

## Materials and Methods

This study was approved by the ethics committee of Mashhad University of Medical Sciences (Ethics Code: IR.MUMS.DENTISTRY.REC.1402.034). The sample size for each group was calculated as  $n=5$ , considering  $\alpha = 0.05$ ,  $\beta = 0.2$ , and data from a previous study (12).

### Sample Preparation

Custom wooden plates were prepared, each containing 5 molds measuring 4 mm in height and 4 mm in diameter (Figure 1). The molds were moistened with distilled water before placing the CEM cement to prevent material dehydration and to simulate clinical humidity.

A total of 40 CEM cement samples (Bionique Co., Tehran, Iran) were prepared according to the manufacturer's instructions, using a powder-to-liquid ratio of 1:2. The liquid was slowly added to the powder and mixed with a spatula for 30 seconds. The mixture was then placed into molds to a depth of 2 mm and condensed. Excess material on the surface was removed using a wet cotton pellet after condensation.

### Study Groups

The study included eight groups ( $n = 5$  per group), based on the type of glass ionomer cement (GIC) applied over the CEM cement (conventional GIC, RMGIC, Ionoseal, or no coating) and the timing of GIC placement (immediately or after a 5-minute application of a wet cotton pellet for external moisture). The study groups were as follows:

*Groups 1 (Conventional GIC, immediate restoration):* Conventional GIC (GC Fuji II, GC Corporation, Tokyo, Japan) was mixed at the standard powder-to-liquid ratio



**Figure 1.** Custom wooden molds for sample preparation

and applied as a 2 mm layer immediately after placing the CEM cement.

*Group 2 (RMGIC, immediate restoration):* Light-cure RMGIC (GC Fuji II LC, GC Corporation, Tokyo, Japan) was prepared according to the manufacturer's instructions and applied as a 2 mm layer immediately after CEM cement placement.

*Groups 3 (Ionoseal, immediate restoration):* Syringe-applied RMGIC (Ionoseal) was placed directly over CEM cement at a thickness of 2 mm immediately after CEM cement insertion, then light-cured for 20 seconds using an LED curing unit (Demi TM Plus, Kerr, California, USA).

*Group 4 (Control, immediate):* No restorative material was applied over the CEM cement.

*Group 4 (Control, immediate):* No restorative material was applied over the CEM cement.

*Group 5 (Conventional GIC, delayed restoration):* After placing the CEM cement, a wet cotton pellet was applied for 5 minutes, then conventional GIC was applied as in Group 1.

*Group 6 (RMGIC, delayed restoration):* A wet cotton pellet was placed over CEM cement for 5 minutes, followed by RMGIC application as described in Group 2.

*Groups 7 (Ionoseal, delayed restoration):* A wet cotton pellet was placed over CEM cement for 5 minutes, followed by Ionoseal application, as described in Group 3.

*Group 8 (Control, delayed):* A wet cotton pellet was placed over the CEM cement for 5 minutes, but no glass ionomer was applied.

Following these treatments, all samples were incubated at 37°C in 100% humidity for one week.

### Vickers Microhardness Assessment

After the 7-day incubation period, samples were removed from the wooden molds with a 2-mm safety margin to preserve the specimens and their interfaces. The sectioned samples were then embedded in molds using acrylic resin (Asia Chemi Teb Co., Tehran, Iran). The acrylic resin was trimmed to expose the interface between the CEM cement and the glass ionomer cement (GIC) layers. This interface was polished sequentially using abrasive papers with grits of 1000, 2000, 3000, and 5000 before microhardness testing (Figure 2).

Due to the white and porous nature of CEM cement, which makes it difficult to locate the exact point for Vickers hardness testing, the CEM samples were colored to improve visibility.

Surface microhardness of the CEM cement was measured at 160 µm from the GIC-CEM interface using a Vickers hardness tester (MH3 series, Koopa Corp., Iran)

(Figure 3), with a 200 g load applied for 20 seconds. Three measurements were taken per sample, and the average value was recorded.

### Statistical Analysis

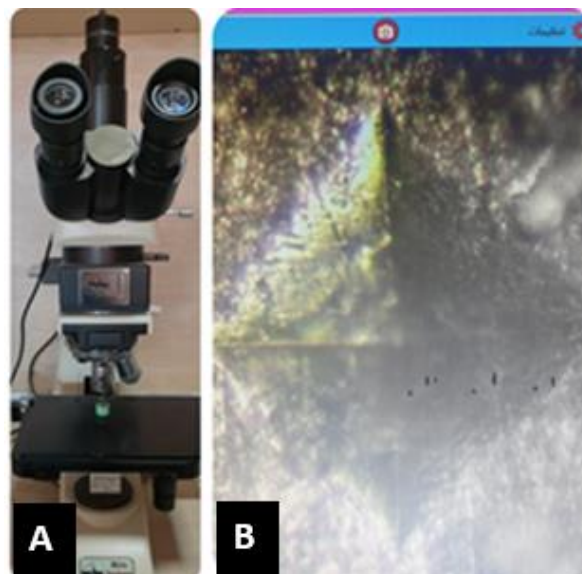
The Shapiro-Wilk test confirmed the normality of data distribution ( $P > 0.05$ ). Two-way ANOVA was performed to evaluate the effects of the variables. Data were analyzed using SPSS version 26.0 (IBM Corp., Armonk, NY, USA) and a  $p$ -value  $< 0.05$  was considered statistically significant.

### Results

The two-way ANOVA revealed that external moisture application ( $P = 0.011$ ), group type ( $P < 0.001$ ), and their interaction ( $P = 0.017$ ) all had a significant effect on microhardness values. Therefore, the effects of each variable were analyzed separately.



**Figure 2.** Samples prepared for Vickers microhardness testing



**Figure 3.** A. The Vickers hardness tester used in this study; B. Indentation produced at a depth of 160 µm to measure the microhardness of CEM cement

Table 1 presents the mean and standard deviation of microhardness values for each study group. In the immediate restoration groups, microhardness was slightly higher in samples coated with different types of glass ionomers compared to the control; however, no significant difference in microhardness was found among the immediate restoration groups ( $P = 0.4$ ).

ANOVA indicated a significant difference in microhardness among groups exposed to external moisture application ( $P < 0.001$ ). According to the Tukey post-hoc test, all glass ionomer groups had significantly higher microhardness values than the control group ( $P < 0.05$ ). Moreover, samples coated with self-cured GIC exhibited significantly higher microhardness than those coated with RMGIC and Ionoseal ( $P < 0.05$ ; Table 1).

When comparing the immediate application of glass ionomer to application after a 5-minute delay, only the self-cured glass ionomer group showed a significant increase in microhardness ( $P = 0.001$ ), while no significant improvement was observed in the other groups.

## Discussion

This in vitro study investigated the effect of glass ionomer application on the physical properties of CEM cement by analyzing its microhardness. Microhardness testing is a non-destructive and quantitative technique that provides valuable information about the degree of material setting and its mechanical integrity, both of which are critical for predicting clinical performance. This method measures localized resistance to deformation, identifies structural changes, and provides reproducible, depth-specific results (18, 19).

The microhardness of CEM cement is influenced by several factors, including the water-to-powder ratio, temperature, humidity, pH of the setting environment, and the level of compression or pressure applied during setting. Under clinical conditions, biomaterials such as CEM cement are placed in direct contact with coronal

restorations. Consequently, the setting reactions of restorative materials may interfere with the hardening process of the underlying CEM cement, especially when the restoration is placed immediately (20, 21).

In the present study, restorative materials were applied either immediately or five minutes after placing a wet cotton pellet to provide external moisture for CEM cement setting. The results indicated that in the immediate restoration groups, microhardness values were slightly higher in samples coated with different types of glass ionomer compared to the control group (no restoration), although the differences were not statistically significant. This finding suggests that when restorations are placed immediately, there is no significant difference between conventional GIC, RMGIC, and Ionoseal concerning the microhardness of the underlying CEM cement.

After five minutes of moisture application with a wet cotton pellet, all glass ionomer groups showed significantly higher microhardness values than the control group. Among the tested materials, samples coated with conventional GIC exhibited significantly greater microhardness than those coated with RMGIC and Ionoseal. These results suggest that different types of GIC positively affect the microhardness of CEM cement compared to no restoration (control). The superior performance of conventional GIC compared to RMGIC and Ionoseal can be attributed to their different setting mechanisms and chemical interactions. Conventional GICs release ions such as calcium and fluoride, which may enhance the maturation and hardening of the underlying CEM cement when the cement is allowed to set initially. In contrast, RMGIC and Ionoseal, due to their resin components and slower acid-base reaction, may interfere with optimal ion release and water balance, resulting in lower microhardness compared with conventional self-cured GIC.

Comparison between immediate and delayed restoration placement revealed that delayed restoration

**Table1.** Comparison of mean and standard deviation (SD) of the hardness number of CEM cement between the experimental groups

Group	Type of GICs	Immediate restoration	Delayed restoration	P-value*
		Mean± SD	Mean± SD	
1	CGIC	13.52 ± 1.85	19.24 ± 1.85 <sup>a</sup>	0.001
2	RMGI	13.99 ± 3.00	15.34 ± 1.48 <sup>a,b</sup>	0.39
3	Ionoseal	14.01 ± 2.80	15.38 ± 1.19 <sup>a,b</sup>	0.34
4	control	10.76 ± 2.87	11.67 ± 1.92 <sup>b</sup>	0.57
P-value**		0.40	<0.001	

\*Values less than 0.05 represent a significant difference according to independent samples t-test.

\*\*Values less than 0.05 represent a significant difference according to one-way ANOVA

Groups labeled with different lowercase letters indicate significant differences at  $P < 0.05$ , whereas those sharing the same

generally increased the microhardness of CEM cement. However, this increase was statistically significant only in the conventional GIC group, while the increases in the Ionoseal and RMGIC groups were not significant. Therefore, in clinical practice, allowing a short delay before placing the restoration may promote better hydration and early maturation of CEM cement when conventional GIC is used for restoration. This delay allows the CEM cement to achieve initial hydration and hardening before exposure to the acidic and ionic environment of the restorative material. If RMGIC or Ionoseal are used for restoration, placing a wet cotton pellet on CEM cement for 5 minutes before applying the glass ionomer is not recommended, as it does not provide any beneficial effect on the microhardness of the underlying CEM cement. Ansari et al. (12) also recommended light-cure glass ionomer for immediate placement on CEM cement. Kazemipour and Tamizi (22) concluded that RMGI interferes least with the setting of CEM cement and is the best choice for restorations using this cement.

In contrast to the outcomes of this study, Tabrizzadeh et al. (23) reported reduced microhardness of CEM cement in a humid environment compared to a dry one. In another study, Tabrizzadeh (24) found that creating a humid environment likely does not influence the microhardness of MTA or CEM apical plugs. They assumed that the optimal level of moisture required for maximum mechanical properties remains unclear, and thus, it is possible that excessive moisture absorption after setting may cause material degradation and reduced MTA or CEM strength.

In clinical practice, moisture for setting calcium-based cements like CEM may come from natural sources within the tooth structure. Previous studies on similar materials, such as mineral trioxide aggregate (MTA), have shown that intrinsic moisture from the dentinal tubules or surrounding tissues can be sufficient for complete setting. Budig and Eleazer (25) demonstrated that MTA packed inside roots can fully set by absorbing moisture from saline-soaked roots without additional external moisture. Similarly, Shokouhinejad et al. (26) found that moisture from the apical side alone is sufficient for setting a 2-mm thick MTA plug, and bilateral moisture is unnecessary. DeAngelis et al. (27) reported that using wet cotton is not required for 4-mm-thick MTA Angelus plugs when the apical perforation diameter exceeds 1 mm, allowing sufficient fluid exchange. Lee et al. (28) noted that dentinal tubules of the axial walls, especially in young teeth, provide an adequate water source for cement setting. These

findings suggest that in vivo, natural moisture may support the setting of CEM cement, potentially reducing the need for externally applied moisture during restoration procedures.

This study was conducted under laboratory conditions, which do not fully replicate the clinical environment. In the mouth, teeth experience varying forces and constant changes in temperature and humidity. Additionally, the microhardness test measures only one physical property of CEM cement. For broader clinical use, other physical and mechanical properties should be evaluated in future research. It is recommended that further studies explore a wider range of materials and environmental conditions over a longer period to identify the best option for immediate application on CEM cement with minimal side effects.

## Conclusions

Under the conditions used in this study, it can be concluded that:

- 1- In the case of immediate restoration, different types of glass ionomers provide favorable conditions for CEM cement setting without adversely affecting its microhardness.
- 2- When a wet cotton pellet was applied for five minutes after CM application, all glass ionomer groups showed significantly higher microhardness values than the control group (no restoration). Furthermore, samples coated with conventional GIC exhibited significantly greater microhardness than those coated with RMGIC and Ionoseal.
- 3- Comparison between immediate and delayed restoration placement revealed that delayed restoration significantly increased the microhardness of CEM cement in the conventional GIC group, while the increases in the Ionoseal and RMGIC groups were not significant.
- 4- If RMGIC or Ionoseal are used for restoration, placing a wet cotton pellet on CEM cement for 5 minutes before applying the glass ionomer is not recommended, as it does not provide any beneficial effect on the microhardness of the underlying CEM cement.

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## Conflict of interest

The authors declare that they have no conflict of interest.

## Author contributions

I.P. and F.M. contributed to the research design and implementation; M.G. contributed to the research implementation, data collection, and writing of the manuscript; H.B. contributed to the research implementation and data analysis; and M.M. contributed to the data interpretation and writing of the manuscript. All authors have read and approved the final manuscript.

## Ethical approval

This study was approved by the ethics committee of Mashhad University of Medical Sciences (Ethics Code: IR.MUMS.DENTISTRY.REC.1402.034).

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