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Surface hardness of CEM cement and cold ceramic cement across different pH settings

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Abstract

Objective: This study aimed to assess the surface microhardness of calcium-enriched mixture cement (CEM) and cold ceramic (CC) cement after being cured in environments with different pH values.

Methods: Sixty maxillary canines were obtained. Their roots were cut below the cementoenamel junction (CEJ) to create root dentin blocks with a height of 3 mm. The internal space of the canal was then prepared with a Peeso reamer. The specimens were filled with either CEM (Group 1) or CC (Group 2) cement (n=30). The groups were further divided into three subgroups (n=10), each was wrapped in gauze soaked in one of the following solutions: A) distilled water (pH=7.4), B) buffered butyric acid (pH=4.4), or C) potassium hydroxide (pH=10.4). The samples were incubated at 37°C for one week. Afterwards, the Vickers surface microhardness of specimens was measured and compared between groups and subgroups using a two-way ANOVA (α=0.05).

Results: The microhardness values of both cements showed significant differences depending on the environmental conditions (P<0.05). The highest microhardness was observed in the alkaline environment, with CC cement measuring 60.32 ± 3.16 and CEM cement measuring 53.57 ± 2.09. The microhardness of both materials reduced significantly in the acidic environment (P<0.05). CC cement demonstrated significantly higher surface microhardness than CEM cement in neutral and alkaline environments (P=0.001 for both), but the two groups showed comparable microhardness in the acidic environment (P=0.51).

Conclusions: CC cement showed greater microhardness than CEM cement in neutral and alkaline environments. Both cements exhibit reduced hardness in the acidic condition.

Keywords: Bioceramics, Calcium-enriched mixture, Calcium Silicates, Cold ceramic, Dental cement, Hardness test

Introduction

Bioceramic materials are utilized in root canal treatments due to their excellent biocompatibility, ability to induce regeneration, antibacterial properties, non-toxicity, superior sealing capabilities, creation of a chemical bond with teeth, and appropriate radiopacity (1-3). The endodontic applications of these materials include retrograde filling, sealing perforations, serving as an apical barrier in open apices, pulp capping, apexogenesis, pulpotomy in permanent teeth, and managing internal and external root resorption (4).

Mineral trioxide aggregate (MTA) is the gold standard for various conservative and endodontic procedures (5). The application of MTA is associated with high clinical success because it provides adequate sealing ability and biocompatibility and promotes tissue regeneration (6, 7). However, MTA has some drawbacks such as challenging application, extended setting time, and tooth discoloration (4). Therefore, various materials have been explored as alternatives to MTA.

Calcium-enriched mixture cement (CEM) is a hydrophilic cement that, compared to MTA, has a chemical composition and mechanical properties more similar to dentin (8, 9). CEM cement exhibits excellent antibacterial properties primarily due to its alkaline pH, resulting from the release of calcium hydroxide during and after the setting process. A previous study indicated that the antibacterial effect of CEM cement is comparable to that of calcium hydroxide and superior to that of MTA (9). Its ability to seal the root canal in the presence of blood and moisture and its potential to induce hard tissue formation is comparable to MTA. However, it has a shorter setting time and a lower film thickness than MTA (9, 10).

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Cold ceramic (CC) cement is a calcium hydroxide-based cement and contains hydrophilic components (11). Its composition includes sulfur trioxide, barium oxide, silicon oxide, and calcium oxide (12). Upon mixing, the pH begins at 7.36 and increases to 11.6 after seven days due to the progressive creation of an alkaline environment (13). CC cement has a sealing ability comparable to MTA in the presence or absence of moisture; however, it exhibits superior sealing ability than MTA in the presence of blood contamination (14).

Investigating the physical, chemical, and antimicrobial capacities of bioceramics is important to estimate their clinical performance (15). The physical properties of interest include hardness, setting time, compressive strength, flexural strength, and setting expansion (4). Hardness is defined as a material's resistance to surface deformation when pressure is applied and is a key factor in evaluating the material’s overall strength (16). Factors including the environment's pH, material thickness, condensation pressure, the amount of entrapped air, moisture, and temperature affect the material hardness (17).

Acidic environments influence bioceramics' physical and chemical properties, sealing potential, surface microhardness, and setting processes (18, 19). It is assumed that reducing the pH would lower the microhardness of bioceramics by disrupting the formation of hydroxyapatite crystals and the hydration reaction (20). Tissue pH levels decrease in conditions like abscesses or pulp and periapical inflammation, likely due to the production of butyric acid, which is a byproduct of bacterial metabolism (21). Inflamed tissue and changes in pH levels are likely to occur where bioceramics are used (17, 18). This study aimed to examine and compare the surface hardness of CEM cement and CC cement in setting conditions with varying pH levels.

Materials and methods

Sample preparation

The experimental procedures were approved by the ethics committee of Shahid Sadoughi University of Medical Sciences (approval code: IR.SSU.DENTISTRY.REC.1399.116).

Sixty mature, intact, single-rooted maxillary canines were collected. The samples were cleaned using a scaler to eliminate calculus and tissue remnants. The surfaces of the teeth were then disinfected with a 2.5% hypochlorite solution for 20 minutes. The samples were stored in normal saline at 37°C until the experiment began.

Preparing dentin blocks

The roots of the teeth were cut 2 mm and 5 mm below the cementoenamel junction (CEJ) using a disk to create root dentin specimens with a height of 3 mm (Figure 1). The specimens were mounted in transparent acrylic resin to create dentin blocks. Subsequently, an internal space was prepared within the canal using a No. 5 Peeso reamer.

Sample allocation

Samples were randomly assigned to two groups (n=30) based on the bioceramic materials:

Group 1: CEM cement (Yekta Zist, Tehran, Iran).

Group 2: Cold Ceramic (CC) cement (SJM Co, Yazd, Iran).

The materials were prepared according to the manufacturer's instructions (1:2 liquid-to-powder ratio for CEM and CC cement). The liquid was gradually added to the powder and mixed with a spatula for 15-30 seconds. The working time for both mixed materials was approximately 5 minutes. Each bioceramic material was placed into the prepared space in the dental blocks using an MTA carrier.

The two groups were further divided into three subgroups (n=10), each was wrapped in gauze soaked in one of the following solutions:

A) Distilled water (pH=7.4)

B) Buffered butyric acid (pH=4.4)

C) Potassium hydroxide (pH=10.4).

All samples were incubated at 37°C for one week.

Measuring surface hardness

After seven days, the samples were removed, air-dried, and mounted in resin blocks. The surfaces were polished with minimal manual pressure at room temperature using 1000-grit silicon carbide papers. The Vickers hardness test was evaluated using a

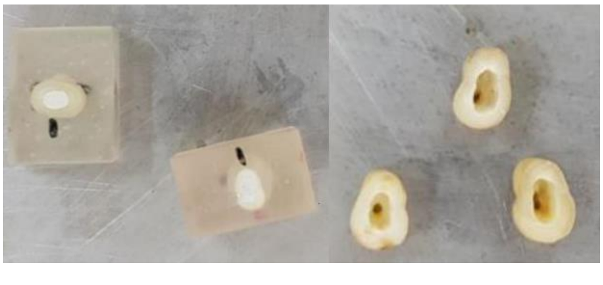

Figure 1. The prepared specimens (right) filled with the experimental materials (left)

Table 1. The mean± standard deviation (SD) of the hardness number of cold ceramic (CC) and CEM cement in setting environments with different pH

Group	pH			P value
	Distilled water (neutral)	Buffered butyric acid (acidic)	Potassium hydroxide (alkaline)	
	Mean± Std. Deviation	Mean± Std. Deviation	Mean± Std. Deviation	
CC cement	60.04 ± 7.28^{A}	33.72 ± 8.12^{B}	60.32 ± 3.16^{A}	0.004^{*}
CEM cement	49.80 ± 3.20^{B}	41.33 ± 8.12^{C}	53.57 ± 2.09^{A}	0.002*
P value	0.001*	0.51	0.001*	

The * represents a statistically significant difference at P<0.05.

Different uppercase letters indicate a significant difference in each row based on pairwise comparison according to the Tuckey HSD post-hoc test.

microhardness tester (MH4-COOPA, IRAN) with a 10 mg load and a 15-second dwell time. Hardness testing was conducted at three points on each sample, and the mean value was recorded.

Statistical analysis

The data were analyzed using a two-way ANOVA to evaluate the effect of environmental pH and material on surface hardness. Values less than 0.05 were considered statistically significant. The analysis was performed using SPSS 26.0 (IBM Corp, Armonk, NY, USA).

Results

The result of two-way ANOVA indicated a significant interaction between the pH and the bioceramic material (P<0.001). Therefore, the effect of these variables on surface hardness was evaluated separately.

The results of the present study are summarized in Table 1. The independent samples t-test indicated that the hardness of the CC cement was significantly higher than that of the CEM cement in neutral and alkaline environments (P=0.001; Table 1). However, in the acidic environment, no significant difference in hardness was observed between the two materials (P=0.51; Table 1).

One-way ANOVA indicated that the CC and CEM cement had significant differences in hardness at various pH levels (P<0.05; Table 1). Pairwise comparisons indicated that the CC samples had a comparable hardness value in alkaline and neutral environments (P>0.05). However, the hardness significantly decreased in the acidic condition compared to other environments (P<0.05; Table 1). On the other hand, CEM cement exhibited significantly higher hardness in the alkaline compared to neutral and acidic conditions (P<0.05). Furthermore, the hardness of CEM cement was significantly higher in the neutral than in the acidic environment (P<0.05; Table 1).

Discussion

The present study examined the surface hardness of CEM cement and CC cement in environments with alkanoic, acidic, and neutral pH. Cement's microhardness depends on several factors including particle size, water-to-powder ratio, temperature, humidity, the amount of entrapped air, and the pH of the setting environment (22).

In the present study, samples were immersed in pieces of moist gauze soaked in neutral (distilled water), acidic (butyric acid), or alkaline (KOH) solutions, similar to the studies of Namazikhah et al. (16) and Saghiri et al. (23). The specimens were not immersed in the solutions to control the washout procedure and simulate the clinical condition. Butyric acid was used for acid exposure since it is a product of anaerobic bacteria that cause peri-radicular infection in clinical situations (21).

The findings of this study indicated that the environmental pH significantly affected the microhardness value of CC and CEM cement. In an acidic environment, the microhardness values of both types of cement decreased significantly. The CC cement showed comparable microhardness in neutral and alkaline conditions, but the microhardness of CEM cement was significantly higher in the alkaline than in the neutral environments. Different bioceramic materials may behave differently against pH due to the different components and setting mechanisms (24). Our previous study indicated that CC cement had better structural integrity in neutral and alkaline pH than in acidic conditions in which CC cement exhibited more amorphous microstructures (25).

The pH of the setting environment affects early hydration, hydrate phase development, and particle reactions of the bioceramic materials (19, 26). Scanning electron micrographs (SEM) of hydrated bioceramics in neutral pH or distilled water showed the formation of cubic and needle-like crystals (17). However, needle-like crystals are not formed in an acidic pH; a significant portion would be cubic (17, 27). The needle-like crystals play a significant role in material consolidation, and their absence would reduce material surface hardness (17). Infected root canals often exhibit acidic pH levels due to bacteria's metabolic byproducts, leading to a pH as low as 5.4. Such low pH values can reduce the cement's bonding strength and dislodgement resistance (28, 29).

The outcomes of this study are in agreement with Mohebbi and Asgari (30), who reported that the CEM cement microhardness decreased significantly in acidic environments (pH 5.4) compared to neutral (pH 7.4) and alkaline (pH 9.4) conditions. They also reported that extending the CEM cement setting time in acidic conditions correlates with reduced microhardness. Other studies have also indicated that the hardness values of bioceramic materials decrease in acidic environments (16, 17, 31). Torabinejad and Chivian (18) observed the deteriorating effect of acidic pH on the setting reaction of MTA. Deepthi et al. (32) also reported that the microhardness of bioceramic materials, including TheraCal LC, Biodentine, Endosequence, and MTA, was reduced in an acidic environment, which resulted in these materials having more porous and less crystalline microstructures.

High alkalinity could occur after placing and removing an intracanal medicament like calcium hydroxide (33), which is commonly employed as an intracanal medicament. It has been shown that calcium hydroxide significantly raises the pH of the surrounding medium, reaching levels as high as 12 to promote healing and inhibit bacterial growth (34). The present findings indicated that the highest microhardness of CEM and CC cement was observed in alkaline conditions, indicating their suitability for use in root canals treated with calcium hydroxide. Xu and Stark (26) studied an alkaline accelerator's effect on the setting reaction of Portland cement. They reported that high pH accelerates the reaction of the cement ingredients and enhances the early hydration phase development.

In the alkaline and neutral pH, the surface hardness of CC cement was higher than that of CEM cement. However, in acidic pH, the microhardness of both cements was statistically comparable. Therefore, CC cement may outperform CEM cement in neutral and alkaline environments. However, our previous study indicated that the time required to reach a neutral pH from acidic pH was longer for the CC samples (4 days) compared to the CEM cement samples (3 days) (25). These findings suggest that CEM cement reaches a neutral pH more quickly, likely due to the differences in its composition and reaction kinetics compared to CC cement. This characteristic may enhance the setting properties of CEM cement as compared to CC cement.

The present study had some limitations. The observation period was limited to one week. Additionally, only a specific range of pH values was tested, and other potential influencing factors, such as temperature variations and microbial interactions, were not considered. Future studies should aim to extend the observation period to several weeks or months. Investigating the effects of a broader range of pH values and other environmental factors would also provide a more comprehensive understanding of the materials' behavior in various conditions.

Conclusions

Within the limitations of an in vitro study, the following statements are concluded:

- CC cement showed greater surface hardness in neutral and alkaline environments than CEM cement. However, both cements exhibited similar surface hardness values in acidic conditions.
- The surface hardness values of CEM and CC cement in acidic environments were significantly lower than those found in alkaline and neutral environments.

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Conflicts of Interest

The authors declare that they have no conflict of interest.

Ethical Considerations

This study was approved by the ethics committee of Shahid Sadoughi University of Medical Sciences (code: IR.SSU.REC.1399.116).

Author Contributions

M.K. contributed to the design and conceptualization of the study, data analysis, and manuscript preparation. R.H. contributed to data collection and manuscript preparation. All authors approved the final manuscript.

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