ORIGINAL RESEARCH

Evaluation of Microleakage and Shear Bond Strength of Two Different Calcium-based Silicate Indirect Pulp Capping Agents When Restored with Glass Ionomer Cement and Composite Resin Restoration: A Comparative *In Vitro* Study

Chhaya Patel[1], Megha Patel[2], Margi Panchal[3], Rohan Bhatt[4], Foram Patel[5], Disha Makwani[6]

Abstract

Background: Indirect pulp therapy (IPT) is a conservative treatment approach that leaves the deepest caries adjacent to the pulp undisturbed in an effort to avoid pulp exposure. The result of IPT is primarily dependent on the biocompatibility, sealing capacity, adaptability of the overlying restoration to underlying dentin, and strength of the liner material utilized.

Aim: To evaluate shear bond strength (SBS) and microleakage of mineral trioxide aggregate (e-MTA) and biodentine when restored with glass ionomer cement (GIC) and composite resin after their initial set time.

Materials and methods: Around 40 extracted caries-free permanent molars were randomly divided into two groups according to the liner material used (group I (e-MTA) and group II—biodentine). Each group was further divided into subgroups A1, A2, B1, and B2, where A1 and B1 received GIC and A2 and B2 received composite resin restoration after the initial set time. Half of the samples from each subgroup were allotted to two test groups of SBS and microleakage evaluation. The SBS was evaluated using a universal testing machine, and microleakage was assessed using a dye penetration test. One-way analysis of variance (ANOVA) was used for intergroup comparison, and the paired sample *t*-test was used for intragroup comparison. Categorical data were analyzed using a nonparametric test at the 5% level of significance using Statistical Package for the Social Sciences (SPSS) version 21 for Windows.

Results: A statistically significant difference ($p = 0.001$) was found in SBS between the group (e-MTA) + composite and biodentine + composite, where the latter group showed the highest SBS. Microleakage was highest in the group (e-MTA) + GIC (0.6 ± 0.894) and lowest in the group biodentine + composite (0.2 ± 0.447). However, no statistically significant difference was found in microleakage among all four groups.

Conclusion: Within the constraints of this study, biodentine can be suggested as the pulp capping agent to be restored with composite resin restoration after its initial set time.

Keywords: Biodentine, Dental pulp capping, Initial setting time, Mineral trioxide aggregate (e-MTA).

International Journal of Clinical Pediatric Dentistry (2024): 10.5005/jp-journals-10005-2887

Introduction

"Indirect pulp therapy (IPT) includes the removal of infected dental tissues while allowing the affected tissues comprising hard dentin to be remineralized by a biocompatible material. This will stimulate the creation of tertiary dentin, avoiding pulp exposure and consequently maintaining pulp vitality."[1]

As pulpotomy ensures that all the infectious portions of the pulp are removed, it was considered the best treatment modality. Nowadays, a paradigm shift has been observed toward a more conservative treatment modality. Thus, IPT has been used as an alternative to treat reversible pulpitis.[2] Additionally, it facilitates regular exfoliation time by maintaining pulp vitality. The prognosis of IPT is dependent on the marginal seal and SBS between the liner and restorative material, which reduces marginal leakage—a precursor to secondary caries, tooth discoloration, postoperative sensitivity, and pulpal pathology.

Calcium hydroxide has been suggested as a near-ideal IPT material. This is due to its exceptional ability to create tertiary dentin, which then encases the pulp with recently formed hard structures. However, calcium hydroxide has 1 weak ability to attach to dentin, making it mechanically unstable. The long-term prevention of microleakage is also compromised by calcium hydroxide's propensity to dissolve. The spread of secondary infection will, as a result, jeopardize the integrity of the pulp.[3]

The above limitations were overcome by the development of hydrophilic calcium silicate-based materials, also known as mineral trioxide aggregate (MTA) cement, which is primarily made of the silicate minerals dicalcium and tricalcium and falls under the

[1–6]Department of Pediatric and Preventive Dentistry, Karnavati School of Dentistry, Karnavati University, Gandhinagar, Gujarat, India

Corresponding Author: Margi Panchal, Department of Pediatric and Preventive Dentistry, Karnavati School of Dentistry, Karnavati University, Gandhinagar, Gujarat, India, Phone: +91 9909977688, e-mail: margipanchal@yahoo.com

How to cite this article: Patel C, Patel M, Panchal M, *et al*. Evaluation of Microleakage and Shear Bond Strength of Two Different Calcium-based Silicate Indirect Pulp Capping Agents When Restored with Glass Ionomer Cement and Composite Resin Restoration: A Comparative *In Vitro* Study. Int J Clin Pediatr Dent 2024;17(9):1035–1040.

Source of support: Nil

Conflict of interest: None

category of hydraulic self-setting materials. These materials form a sticky, self-setting calcium-silicate-hydrate (CSH) gel.[4,5] They have superior sealing associated with expansion, the capacity to set in the presence of fluids, and better biological properties.[6] Despite this, many disadvantages were noted with MTA, such as delayed final restoration due to long setting time, increased duration of treatment, and risks of failure of the endodontic procedure.[7] A second visit is necessary for the final restoration, which might not be appropriate for pediatric patients.[8] Recently introduced new calcium silicate cements like biodentine and e-MTA overcome the disadvantages of traditional MTA. Biodentine has been introduced as the "first all-in-one, bioactive, and biocompatible material for damaged dentin replacement." Biodentine and e-MTA have an initial set time of 12 and 15 minutes, respectively, shorter than that of conventional MTA, which is 2 hours and 45 minutes.[9]

Thus, the current study was conducted to assess the microleakage and SBS of recently available pulp capping materials based on tricalcium silicate (e-MTA, biodentine) when restored with cement after their initial setting time. The null hypothesis proposed was that there would be no statistically significant difference in microleakage and SBS between e-MTA and biodentine when restored with glass ionomer cement (GIC) and composite resin.

Materials and Methods

Study Design, Sample Size, Randomization, and Allocation

The proposed study was conducted as an *in vitro* study with prior approval and consent obtained from the Institutional Ethics Committee. For this investigation, 40 freshly extracted permanent molars that were intact and free of caries were collected. Their surfaces were debrided using ultrasonic scalers, and they were then kept in distilled water at room temperature throughout the trial.

The samples were equally divided (n = 20) into two groups study—MTA and biodentine. Further subgroups (n = 10) were based on the type of restoration (GIC and composite) placed, and the tests performed (n = 5) (SBS and microleakage) (Fig. 1). The materials used in the present study are shown in Table 1.

Procedure

Sample Preparation for Shear Bond Strength

A flat cut made with a diamond disk at a high rate of speed, using water spray, exposed the mid-coronal dentin of the occlusal surfaces. Polishing was done with 600-grit sandpaper under water lubrication. The molars were then embedded into acrylic blocks. Thereafter, samples were stored in distilled water at room temperature. The area and volume of liners (1.5 × 1.5 mm) and restorative materials (4 × 4 mm) were standardized using polyethylene tubes.

For groups A1 and B1, GIC (3M ESPE Ketac Molar) was mixed according to the manufacturer's instructions, and the mold was filled with GIC after the initial set of their respective liner. Later, the polyethylene molds were removed with a sharp instrument.

For groups A2 and B2, after the initial set of the respective lining materials, both study groups were restored with composite. Dentin surfaces were etched with 37% phosphoric acid gel (3M ESPE Scotchbond™ Universal Etchant) for 15 seconds, rinsed, and dried for 10 seconds. Then, a single layer of dentin bonding agent (3M ESPE Adper™ Single Bond 2 Adhesive) was applied and rubbed over the pulp capping agent and dentinal surface for 15 seconds. It was then cured for 10 seconds by a light-curing unit (Waldent, 1200 mw/cm^2). The mold was placed over the pulp capping agent and filled with composite (3M Filtek Z250 XT) in two increments of 2 mm, with each increment cured for 20 seconds. The polyethylene molds were removed after the curing of the composite. The Instron® 3382 100 kN universal testing machine was used to determine the shear bond strength (SBS). Load was applied at a speed of 0.5 mm/minute until bond failure occurred, and values were noted.

Sample Preparation for Microleakage

A class I cavity was made 0.5–1 mm below the dentinoenamel junction (DEJ) and with a width of not more than one-fourth of the intercuspal distance.

Group I

e-MTA (Kids-e-Dental) was mixed according to the manufacturer's instructions. It was then placed into the previously formed class I cavity with an amalgam carrier and applied in a 1.5 mm thickness. After the initial set of the cement (i.e., 15 minutes), the class I cavity was filled with GIC and composite resin restoration in groups A1 and A2, respectively.

Group II

Biodentine™ (Septodont, Mumbai, India) capsule was mixed in an amalgamator for 30 seconds at 4000 rpm. After the mixture (wet sand consistency) was introduced into the previously formed class I cavity with an amalgam carrier in a 1.5 mm thickness. After the

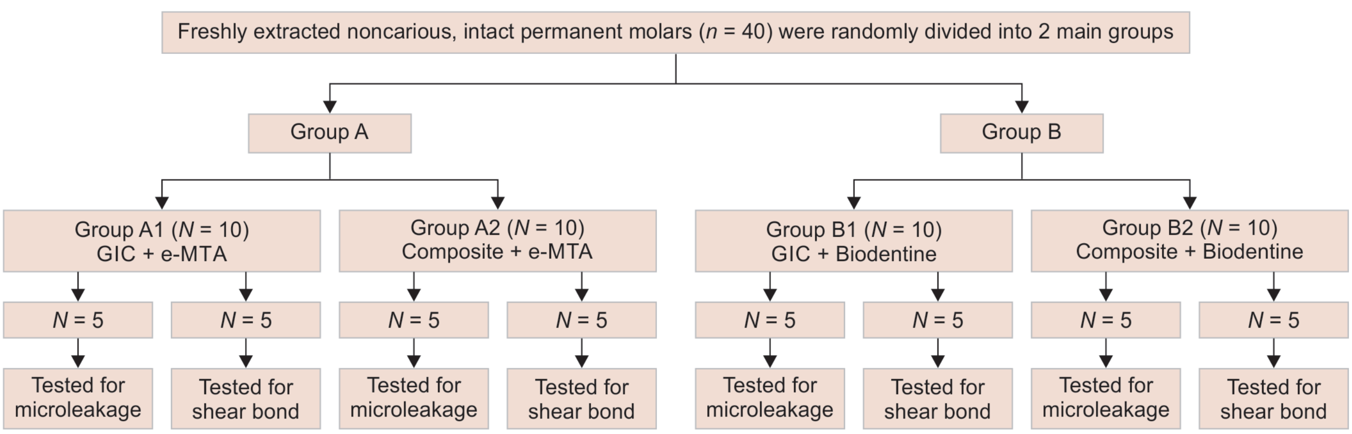

Fig. 1: Consolidated standards of reporting trials diagram showing sample size distribution

Table 1: List of materials

	Material	*Manufacturer*	*Composition*
	e-MTA	Kids-e-Dental, India	Powder: Tricalcium silicate, dicalcium silicate, tricalcium aluminate, bismuth oxide, gypsum Liquid: Distilled water
	Biodentine	Septodont (St. Maur-des-Fossés, France)	Powder: Tricalcium silicate, dicalcium silicate, calcium carbonate, iron oxide, zirconium oxide Liquid: Water, calcium chloride, hypersoluble polymer
	GIC	3M ESPE Ketac Molar	Fluorosilicate glass, Al-Ca copolymer (5% acrylic acid maleic acid)
	Composite	3M Filtek Z250 XT	Silane treated ceramic. (1-methylethylidene) bis[4,1-phenyleneoxy(2-hydroxy-3,1-propanediyl)] bismethacrylate
	Bonding agent	3M ESPE, St Paul, Minnesota, United States of America	Bis-GMA, HEMA, dimethacrylate, ethanol, water
	Etchant Scotchbond™ Universal etchant	3M ESPE, St Paul, Minnesota, United States of America	34% phosphoric acid with a pH of approximately 0.1

initial set of the cement (i.e., 12 minutes), groups B1 and B2 were restored with GIC and composite resin restoration, respectively.

Around 500 thermocycling cycles with dwell times of 60 seconds were performed on all samples. Next, two layers of nail polish were applied to each sample, stopping 1 mm from the restoration borders. Sticky wax was used to seal the apex of the tooth. After soaking the teeth in 5% methylene blue dye for 24 hours at room temperature, they were rinsed for 30 seconds under running water. Using a diamond disk and a high-speed handpiece with water as a coolant, the teeth were longitudinally sectioned into two halves mesiodistally. Under a stereomicroscope with 40× magnification, the extent of dye penetration was examined. The area of the sectioned tooth with the most microleakage received a score. According to International Standards Organization guidelines, the degree of microleakage using dye penetration was scored based on a four-grade scale:

0 = No dye penetration.

1 = Dye penetration into the enamel part of the cavity wall.

2 = Dye penetration into the dentin part of the cavity wall but not including the pulpal floor of the cavity.

3 = Dye penetration, including the pulpal floor of the cavity.[10]

Statistical Analysis

The collected data was entered into a 2019 Microsoft Excel spreadsheet. Data on continuous variables were presented as mean ± standard deviation (SD). One-way analysis of variance (ANOVA) was used between groups, and the paired sample *t*-test was applied within groups. Categorical data were analyzed using a nonparametric test at the 5% level of significance using Statistical Package for the Social Sciences (SPSS) version 21 for Windows (Armonk, New York: IBM Corp.).

Results

Shear Bond Strength Evaluation

In the present study, the maximum SBS was found in the group biodentine + composite (6.024 ± 0.520), followed by the group biodentine + GIC (5.094 ± 0.646), the group e-MTA + composite (4.430 ± 0.624), and the least was found in the group e-MTA + GIC (4.104 ± 0.597) (Fig. 2). There was no statistical significant difference found on intragroup comparison (Table 2) between group A1 and A2 ($p = 0.578$) and group B1 and B2 ($p = 0.106$). There was no statistically significant difference found on intergroup comparison (Table 3) between group A1 and B1 ($p = 0.845$), group A1 and B2 ($p = 0.386$), group A2 and B1 ($p = 0.079$); however, a highly statistically significant difference was observed between group A2 and B2 ($p = 0.001$).

Microleakage Evaluation

The maximum microleakage was observed in group e-MTA + GIC (0.6 ± 0.894), followed by group e-MTA + composite, group biodentine + GIC (0.4 ± 0.548), and the least microleakage was found in group biodentine + composite (0.2 ± 0.447) (Figs 3 and 4). There was no statistically significant difference in intragroup comparison (Table 4) between groups A1 and A2 (p = 0.958) and groups B1 and B2 (p = 0.958). Also, for intergroup comparison (Table 5), no statistically significant difference was reported.

Discussion

A significant factor limiting the success of IPT-treated teeth is ineffective sealing from the coronal restoration. The prognosis of IPT depends on the adhesive compatibility of the pulp capping agent with the overlaying restorative material in terms of SBS. It is the interfacial adhesion between the substrate and bonded material with the help of an adhesive layer. In clinical practice, fractures may occur either in the bonded material, the substrate, or both. Thus, SBS is measured as the force required to fracture the bond between the IPT agent and the final restoration.[11]

Microleakage is a major cause of failure for IPT agents. It is related to the material's adaptation to the prepared cavity walls, which determines its ability to seal the cavity and protect it from invasion by saliva and bacteria. Consequently, it can lead to hypersensitivity of the restored tooth, secondary caries, and pulp damage. The most commonly used method to detect microleakage is the penetration of dyes into sections of the tooth.[12] Various

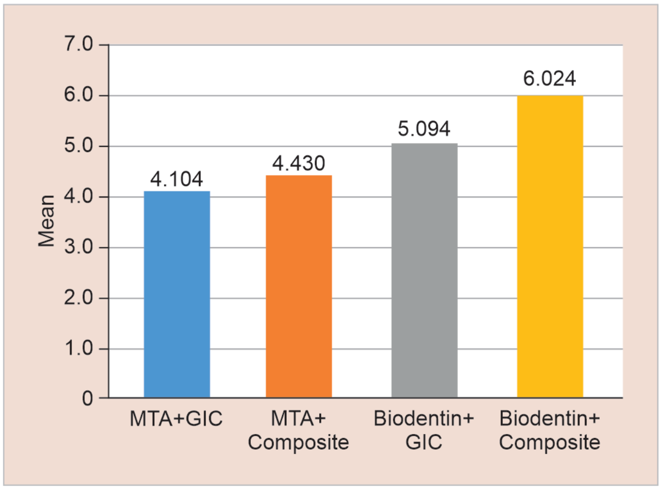

Fig. 2: Mean value for SBS

Table 2: Intragroup comparison of mean SBS

Groups			*p-value**	*Inference*
A1	VS	A2	0.578	Not significant
B1	VS	B2	0.106	Not significant

*One-way ANOVA

Table 3: Intergroup comparison of mean SBS

Groups			*p-value**	*Inference*
A1	VS	B1	0.845	Not significant
A1	VS	B2	0.386	Not significant
A2	VS	B1	0.079	Not significant
A2	VS	B2	0.001	Significant

*Tukey *post hoc* test

Table 4: Intragroup comparison of mean microleakage scores

Groups			*p-value**	*Inference*
A1	VS	A2	0.958	Not significant
B1	VS	B2	0.958	Not significant

*One-way ANOVA

Table 5: Intergroup comparison of mean microleakage scores

Groups			*p-value**	*Inference*
A1	VS	B1	0.958	Not significant
A1	VS	B2	0.400	Not significant
A2	VS	B1	1.00	Not significant
A2	VS	B2	0.958	Not significant

*Tukey *post hoc* test

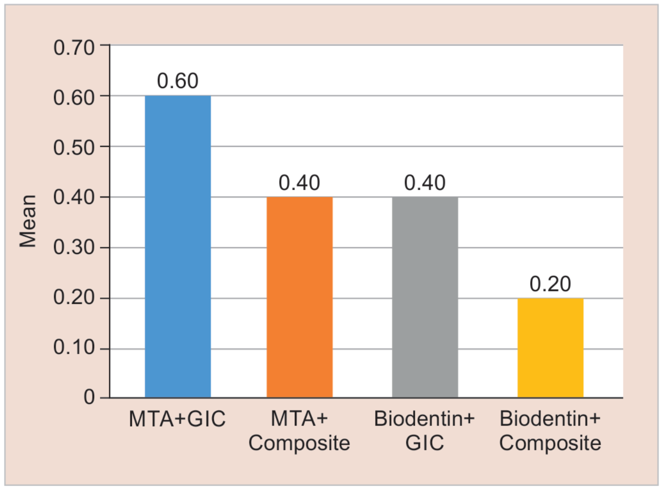

Fig. 3: Mean value for microleakage

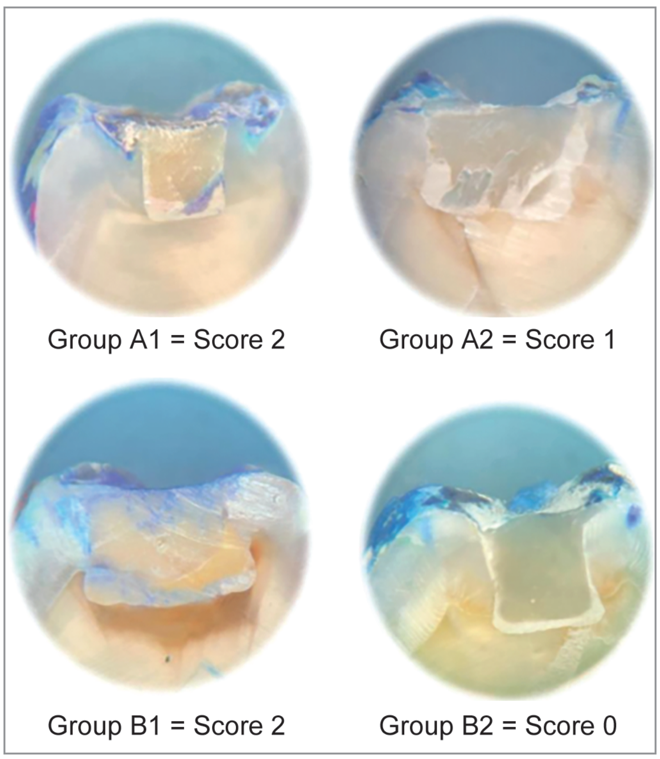

Fig. 4: Stereomicroscopic images of microleakage scoring

dyes can be used, such as methylene blue, basic fuchsine, Indian ink, etc. In this study, methylene blue dye was used to measure microleakage.

There has always been controversy regarding the timing of permanent coronal restoration after the initial or final setting time of the pulp capping agent. GIC has traditionally been used as an intermediate restoration, which is replaced by composite restoration during a follow-up visit after the final setting of the pulp capping agent, especially MTA. The purpose of delaying resin composite insertion is to allow calcium silicate cements enough time to mature completely and acquire all of their physical properties. According to Davidson et al., a bond strength value of 17–20 MPa between calcium silicate cements and resin composites is needed to resist the contraction of resin composite.[13] The second visit for final restoration is not practical for both patients and practitioners due to the increased cost and risk of IPT failure. Thus, immediate placement of the final restoration on the calcium silicate cement layer may be considered an alternative. An IPT agent with a shorter time setting is required to place the final restoration at the same appointment. The present study was conducted to compare the SBS and microleakage of pulp capping agents biodentine and e-MTA, which have shorter initial setting times when restored with GIC and composite resin restoration.

In the present study, the group biodentine + composite showed the highest SBS. A statistically significant difference was found between the biodentine + composite and e-MTA + composite groups, which implies that biodentine is a better pulp-capping agent to use with composite resin restoration after its initial set. Thus, the null hypothesis was rejected. Our results are in accordance with a study by Palma et al., where biodentine made it possible for quick restoration, whereas bonding treatments directly on top of MTA were reported to be preferred at a later period.[11] However, contradictory findings were observed by Tsujimoto et al.[14] and Hashem et al.,[15] where biodentine performed better when restored after 2 weeks while MTA at the same appointment.

Bond strength is significantly influenced by the chemical composition of the materials. The chemical composition of Biodentine powder primarily includes tricalcium silicate, dicalcium silicate, and calcium oxide, while the liquid contains water, calcium chloride, which plays a significant role in the faster setting of Biodentine (within 12 minutes) and a carboxylate-based hydrosoluble polymer. The polymer acts as a water-reducing agent, decreasing the amount of water without affecting the workability (i.e., the flow of the material) and improving the strength of the cement.[16] Powders of e-MTA consist of calcium oxide, silicon dioxide, aluminum oxide, magnesium oxide, ferrous oxide, and zirconium oxide, while the liquids consist of 100% distilled water (just like MTA). Thus, due to the presence of water, e-MTA may not set as quickly as biodentine. According to Bodanezi et al.,[17] Vanderweele et al.,[18] Sluyk et al.,[19] MTA shouldn't be restored for 72 hours as it had a high degree of solubility over the first 72 hours after mixing. GIC has tendency to absorb water from newly mixed calcium silicate-based cement, which causes incomplete hydration and porosities in MTA. A portion of the MTA remains bonded to the GIC as the two materials begin to separate from one another. It leads to microcracking and lower bond strength.[16] Another reason for the weak SBS of e-MTA is its large particle size (3–30 µm) compared to biodentine (1–10 µm). Biodentine forms tag-like structures after penetrating the dentinal tubules, resulting in better micromechanical retention. Additionally, the GIC setting process creates an acidic pH between 1.9 and 2.2. An acidic atmosphere may impact calcium silicate-based materials, diminishing their hardness and strength, as well as slowing down the setting process and increasing solubility, which results in significant leakage. This provides a possible explanation for both lower SBS and microleakage in the e-MTA + GIC group and biodentine + GIC group. Our results were in accordance with a study conducted by Cengiz and Ulusoy,[20] Meraji and Camilleri,[21] and Niranjan et al.,[22] where it was shown that SBS between composite and biodentine is superior to GIC and biodentine after the initial set time of this cement. According to Koubi et al., biodentine is a suitable IPT agent that can be used for posterior restorations under a composite.[23]

The scope of our investigation did not include the failure modes (adhesive/cohesive) between the restoration and pulp capping agents. As no statistically significant difference was found in microleakage between e-MTA and biodentine, further *in vitro* and *in vivo* studies with a larger sample size are required to investigate the performance of e-MTA with restorative materials and validate our observations.

Conclusion

Enhanced e-MTA and biodentine can be used successfully as an IPT agent with GIC restoration at the same visit. Biodentine can be suggested as an ideal IPT agent for final restoration of a tooth with composite at the same visit, as it showed the highest SBS.

Author Contributions

Patel Chhaya: Conception of idea and data analysis; Patel Megha: Adding scientific content; Panchal Margi: Conducted the study and preparation of the manuscript; Bhatt Rohan: Review of manuscript; Patel Foram: Compilation of data; Makwani Disha: Record keeping.

References

1. Seale NS. Indirect pulp therapy: an alternative to pulpotomy in primary teeth. Tex Dent J 2010;127(11):1175–1183.
2. American Academy of Pediatric Dentistry. Pulp therapy for primary and immature permanent teeth. The Reference Manual of Pediatric Dentistry. Chicago, Ill.: American Academy of Pediatric Dentistry; 2022:415–423.
3. Bhatt RA, Patel MC, Bhatt R, et al. A comparative evaluation of light cure calcium silicate and resin-modified glass ionomer as indirect pulp capping agent in primary molars: a randomized clinical trial. Dent Res J 2023;20:18. DOI: 10.4103/1735-3327.369620
4. Gandolfi MG, Van Landuyt K, Taddei P, et al. Environmental scanning electron microscopy connected with energy dispersive X-ray analysis and Raman techniques to study ProRoot mineral trioxide aggregate and calcium silicate cements in wet conditions and in real time. J Endod 2010;36(5):851–857. DOI: 10.1016/j.joen.2009.12.007
5. Camilleri J, Gandolfi MG. Evaluation of the radiopacity of calcium silicate cements containing different radiopacifiers. Int Endod J 2010;43(1):21–30. DOI: 10.1111/j.1365-2591.2009.01621.x
6. Gandolfi MG, Siboni F, Botero T, et al. Calcium silicate and calcium hydroxide materials for pulp capping: biointeractivity, porosity, solubility and bioactivity of current formulations. J Appl Biomater Funct Mater 2015;13(1):43–60. DOI: 10.5301/jabfm.5000201
7. Mente J, Geletneky B, Ohle M, et al. Mineral trioxide aggregate or calcium hydroxide direct pulp capping: an analysis of the clinical treatment outcome. J Endod 2010;36(5):806–813. DOI: 10.1016/j.joen.2010.02.024
8. Dawood AE, Manton DJ, Parashos P, et al. The effect of working time on the displacement of Biodentine beneath prefabricated stainless steel crown: a laboratory study. J Investig Clin Dent 2016;7(4):391–395. DOI: 10.1111/jicd.12162

9. Chaipattanawan N, Manmontri C, Fanchiang JC, et al. Delayed versus immediate placement of direct resin composite restorations following vital pulp therapy with ProRoot® mineral trioxide aggregate or Biodentine™: a review of the literature. CM Dent J 2021;42(2):44–58.
10. International Standards Organization. Dental Materials: Testing of Adhesion to Tooth Structure, 2nd edition. Geneva: ISO/TS; 2003.
11. Palma PJ, Marques JA, Falacho RI, et al. Does delayed restoration improve shear bond strength of different restorative protocols to calcium silicate-based cements? Materials (Basel) 2018;11(11):2216. DOI: 10.3390/ma11112216
12. Abd El Halim S, Zaki D. Comparative evaluation of microleakage among three different glass ionomer types. Operat Dent 2011;36(1):36–42. DOI: 10.2341/10-123-LR
13. Davidson CL, de Gee AJ, Feilzer A. The competition between the composite-dentin bond strength and the polymerization contraction stress. J Dent Res 1984;63(12):1396–1399. DOI: 10.1177/00220345840630121101
14. Tsujimoto M, Tsujimoto Y, Ookubo A, et al. Timing for composite resin placement on mineral trioxide aggregate. J Endod 2013;39(9):1167–1170. DOI: 10.1016/j.joen.2013.06.009
15. Hashem DF, Foxton R, Manoharan A, et al. The physical characteristics of resin composite-calcium silicate interface as part of a layered/laminate adhesive restoration. Dent Mater 2014;30(3):343–349. DOI: 10.1016/j.dental.2013.12.010
16. Camilleri J. Mineral trioxide aggregate: from preparation to application. Berlin Springer 2014. DOI: 10.1007/978-3-642-55157-4
17. Bodanezi A, Carvalho N, Silva D, et al. Immediate and delayed solubility of mineral trioxide aggregate and Portland cement. J Appl Oral Sci 2008;16(2):127–131. DOI: 10.1590/s1678-77572008000200009
18. Vanderweele RA, Schwartz SA, Beeson TJ. Effect of blood contamination on retention characteristics of MTA when mixed with different liquids. J Endod 2006;32(5):421–424. DOI: 10.1016/j.joen.2005.09.007
19. Sluyk SR, Moon PC, Hartwell GR. Evaluation of setting properties and retention characteristics of mineral trioxide aggregate when used as a furcation perforation repair material. J Endod 1998;24(11):768–771. DOI: 10.1016/S0099-2399(98)80171-4
20. Cengiz E, Ulusoy N. Microshear bond strength of tri-calcium silicate-based cements to different restorative materials. J Adhes Dent 2016;18(3):231–237.
21. Meraji N, Camilleri J. Bonding over dentin replacement materials. J Endod 2017;43(8):343–1349. DOI: 10.1016/j.joen.2017.03.025
22. Niranjan B, Shashikiran ND, Singla S, et al. A comparative microleakage evaluation of three different base materials in class I cavity in deciduous molars in sandwich technique using dye penetration and dentin surface interface by scanning electron microscope. J Indian Soc Pedod Prev Dent 2016;34(4):324–330. DOI: 10.4103/0970-4388.191410
23. Koubi S, Elmerini H, Koubi G, et al. Quantitative evaluation by glucose diffusion of microleakage in aged calcium silicate-based open-sandwich restorations. Int J Dent 2012;2012:105863. DOI: 10.1155/2012/105863