

Tooth Discoloration after the Use of New Pozzolan Cement (Endocem) and Mineral Trioxide Aggregate and the Effects of Internal Bleaching

Ji-Hyun Jang, DDS, MSD,* Minji Kang, DDS,* Soyeon Abn,[†] Soyeon Kim, BS,[†] Wooksung Kim,[†] Yaelim Kim, BS,[†] and Euseong Kim, DDS, MSD, PhD*

Abstract

Introduction: The aim of this study was to evaluate tooth discoloration after the use of mineral trioxide aggregate (MTA) and to examine the effect of internal bleaching on discoloration associated with MTA.

Methods: Thirty-two teeth were endodontically treated. Three-millimeter plugs of MTA, ProRoot, Angelus, or Endocem were placed on the access cavities of 24 teeth. Eight teeth served as the control group. After 24 hours, the access cavities were restored, and the tooth color was recorded at baseline and at 1, 2, 4, 8, and 12 weeks.

After 12 weeks, the MTA materials were removed under a microscope, and an internal bleaching treatment was performed. After removal of the MTA materials and after a 1-week bleaching treatment, the color changes were measured, and the MTA-dentin interfaces were observed under a microscope.

Results: The ProRoot and Angelus groups displayed increasing discoloration during a period of 12 weeks. The discoloration associated with ProRoot and Angelus was observed at the MTA-dentin interface and on the interior surface of the dentin. However, the Endocem groups demonstrated no significant discoloration ($P < .05$). No marginal discoloration was observed around the material in the Endocem group. Removal of the discolored MTA was effective for resolving the discoloration in all of the experimental groups ($P < .05$). However, a subsequent internal bleaching treatment was not significantly effective compared with the removal of MTA. **Conclusions:** ProRoot and Angelus caused tooth discoloration. However, Endocem did not affect the contacting dentin surface. Removing the discolored MTA materials contributed more to resolving the tooth discoloration than post-treatment internal bleaching. (*J Endod* 2013;39:1598–1602)

Key Words

Internal bleaching, mineral trioxide aggregate, pozzolan cement, tooth discoloration

Mineral trioxide aggregate (MTA) is a biocompatible material with a high sealing ability and less cytotoxicity compared with conventional endodontic materials such as amalgam, Super EBA, and intermediate restorative material (1–4). MTA is a powder derived from Portland cement that consists of fine hydrophilic particles of tricalcium silicate, tricalcium aluminate, tricalcium oxide, and other mineral oxides. MTA is set in the presence of water, which results in the formation of a crystallized calcium silicate hydrate gel and calcium hydroxide (5). Setting also occurs in the presence of blood; however, no significant negative effect on the leakage of MTA was reported (6). On the basis of various experimental results, MTA is considered to be a reliable material for use in vital pulp therapy in dental traumatology and may replace calcium hydroxide (7–9).

The first developed MTA was gray (GMTA). GMTA has the potential to cause tooth discoloration. Discoloration occurred in 60% of treated cases when GMTA was used as a pulpotomy medicament in primary teeth (10, 11). Consequently, white MTA (WMTA) was developed, and WMTA displayed no significant difference in the pulp response compared with GMTA (12). The major difference in the chemical composition between WMTA and GMTA is the concentration of metal oxides such as Al_2O_3 , MgO, and FeO (13), which were assumed to be the main causes of discoloration. The WMTAs used in this study were the ProRoot tooth-colored MTA formula (Dentsply, Tulsa, OK) and Angelus MTA (Angelus, Londrina, PR, Brazil). Still, unexpected tooth discoloration has been reported after using WMTA for vital pulp therapy (14–16).

Recently, a new type of MTA derived from pozzolan cement (Endocem MTA; Maruchi, Wonju, Korea) has been introduced. Endocem is advantageous because of its rapid setting and manipulation properties. Furthermore, the biocompatibility and osteogenicity of Endocem are similar to those of conventional MTA (17).

There have been several recommendations on how to overcome the discoloration caused by MTA. Belobrov and Parashos (16) reported on a case in which the tooth was bleached by using sodium perborate mixed with saline to resolve discoloration that occurred 17 months after MTA pulp capping. Akbari et al (18) observed that applying a dentin bonding agent before MTA could prevent tooth discoloration. Although the incidence of tooth discoloration induced by MTA is frequent, there have been few published reports addressing this issue.

From the *Microscope Center, Department of Conservative Dentistry and Oral Science Research Center, College of Dentistry, Yonsei University, Seoul; and [†]College of Dentistry, Yonsei University, Seoul, Korea.

Supported by a faculty research grant of Yonsei University College of Dentistry for 2011 (6-2011-0042).

Address requests for reprints to Dr Euseong Kim, Microscope Center, Department of Conservative Dentistry and Oral Science Research Center, College of Dentistry, Yonsei University, 50 Yonsei-ro, Seodaemun-gu, Seoul 120-752, Korea. E-mail address: andyendo@hanmail.net
0099-2399/\$ - see front matter

Copyright © 2013 American Association of Endodontists.

<http://dx.doi.org/10.1016/j.joen.2013.08.035>

The aim of this study was to evaluate tooth discoloration after the use of MTA and to examine the effect of internal bleaching on MTA discoloration.

Materials and Methods

Sample Preparation

Thirty-two freshly extracted human single-rooted incisors were used in this study. The criteria for tooth selection included the tooth being free of caries, cracks, restorations, calcifications, and an absence of any signs of internal or external resorption. The external surfaces of the teeth were cleaned with curettes and stored in a physiological saline solution until use.

The access cavities were prepared, and the working lengths were determined by using stainless steel hand files (Dentsply Maillefer, Tulsa, OK) until the tips were observed at the apical foramen, which was calculated by subtracting 1 mm from the length. The canals were shaped and enlarged by using #2, #3, and #4 Gates Glidden drills and WaveOne Large reciprocating files (Dentsply Maillefer, Ballaigues, Switzerland). The canals were then irrigated by using a 2.5% sodium hypochlorite solution. The prepared root canals were filled with gutta-percha (GP) cones (Diadent, Seoul, Korea) and AH Plus sealer (Dentsply, Konstanz, Germany), and then the GP cones were cut off 3 mm below the cemento-enamel junction.

The endodontically treated teeth were randomly divided into 4 groups that each contained 8 teeth. The experimental groups consisted of 3-mm-thick MTA plugs that were represented by (1) ProRoot, (2) Angelus, and (3) Endocem, each of which was placed directly over the GP cones. Periapical radiographs were taken to ensure that there were no voids in the MTA materials and to determine the thickness of each filling. The access cavities were cleansed, and wet cotton pellets were placed over the MTA plugs. The access cavities were sealed with temporary restoration material (Cavition; GC Corp, Tokyo, Japan) for 24 hours and immersed in artificial saliva. After that delay, the temporary restorative materials were removed, and the setting states of the MTA materials were confirmed. The dentin adhesives (AdheSE; Ivoclar Vivadent, Schaan, Liechtenstein) were applied over MTA materials according to the manufacturer's instructions, followed by composite restoration by using Filtek Z350 (3M ESPE, St Paul, MN). The teeth in the control group were restored with a composite resin placed directly over the GP cones. All samples were stored in artificial saliva (Taliva; Hanlim Pharm Co, Seoul, Korea) at room temperature and replenished every 2 weeks.

Measuring Tooth Discoloration

The tooth color was recorded at baseline and at 1, 2, 4, 8, and 12 weeks by using a spectrophotometer (VITA Easyshade Advance; Vita Zahnfabrik, Bad Sackingen, Germany). The measurements were performed under constant laboratory illumination by positioning the spectrophotometer at the incisal, middle, and cervical areas of the teeth. The color measurements were repeated twice for each sample, and the records were reported by using the CIE L*a*b* system. The value of L* is the lightness ranging from 0 (black) to 100 (white). The values of a*

and b* are the chromaticity coordinates in the red-green axis and the yellow-blue axis, respectively. The comparison of the measured L*a*b* values obtained from the spectrophotometer is expressed as ΔE. In our study, ΔE describes the color difference between the baseline and each different measurement point, and it was calculated by using the following equation:

$$\Delta E = \{(L_2 - L_1)^2 + (a_2 - a_1)^2 + (b_2 - b_1)^2\}^{1/2} \text{ (19)}.$$

Effects of Internal Bleaching on Discoloration

After 12 weeks, the composite restorations and MTA materials were carefully removed with a carbide round bur #1 and ProUltra Endo tip #1 (Dentsply Tulsa, Tulsa, OK) by using an operating microscope (Zeiss OPMI pico; Carl Zeiss, Goettingen, Germany) at the range of ×7.5–15 magnification. A mixture of sodium perborate and 3% hydrogen peroxide solution was used as an internal bleaching agent. The bleaches were applied to the access cavities and sealed with intermediate restorative material for 1 week. The color changes were measured both after removing the MTA materials and after 1 week of application of the bleaching agent. The changes in the MTA-dentin interface were observed under a microscope at ×15 magnification to enhance visualization and to provide illumination.

Statistical Analysis

The time effects were analyzed at 3 different measuring levels and for 3 different materials. Repeated-measures analysis of variance was applied to examine the ΔE value as the dependent variable and time as a factor. The effects of the materials at each time point were analyzed by using a one-way analysis of variance. Scheffé and Bonferroni comparison tests were used to determine the statistical significance at a 95% confidence level. Statistical analysis was performed by using SAS 9.2 (SAS Inc, Cary, NC).

Results

Overall, the significant discoloration was observed only in the cervical area of the tooth samples, and the middle and incisal areas displayed no significant color changes. Table 1 displays the ΔE values from the tooth samples for the different groups, and it displays the time points for the measurements in the cervical area. The ProRoot and Angelus groups displayed an increasing discoloration pattern in the cervical area over time. Tooth samples from the Endocem group presented indistinct grayish color changes during the course of the 12 weeks. This group showed the largest ΔE value after the first week but the lowest ΔE change during the course of the remaining 11 weeks (Fig. 1).

After 12 weeks, the composite and MTA were removed, and the MTA-dentin interfaces were observed under a microscope. The ProRoot and Angelus groups revealed dark marginal discoloration that appeared to spread into the dentin in the interfacial layer. The Endocem group presented no marginal discoloration around the material (Fig. 2).

TABLE 1. Mean ΔE Values (standard deviation) of Tooth Samples for the Different Groups and Time Points Measured in Cervical Area

Groups	1 week	2 weeks	4 weeks	8 weeks	12 weeks
Control	3.95 (0.80) ^{aA}	3.61 (1.42) ^{aA}	3.98 (2.09) ^{aA}	3.49 (1.87) ^{aA}	3.59 (1.92) ^{aA}
ProRoot	4.31 (1.57) ^{aA}	4.69 (1.17) ^{aA}	4.69 (0.69) ^{aA}	7.60 (2.29) ^{abA}	14.85 (6.36) ^{bB}
Angelus	3.47 (1.13) ^{aA}	4.69 (2.03) ^{aA}	5.53 (2.55) ^{aAB}	7.71 (3.74) ^{abAB}	9.11 (4.07) ^{abB}
Endocem	6.18 (3.18) ^{bA}	6.87 (2.75) ^{aA}	6.85 (3.06) ^{aA}	8.30 (3.72) ^{bA}	8.46 (3.47) ^{aA}

Uppercase letters (in row) and lowercase letters (in column) indicate statistically homogenous subgroups (Scheffé and Bonferroni test, α = 0.05 was used for every column and row). The same uppercase letters (within one MTA material, in a row) and lowercase letters (within one time point, in a column) indicate statistically similar groups (Scheffé and Bonferroni test, α = 0.05).

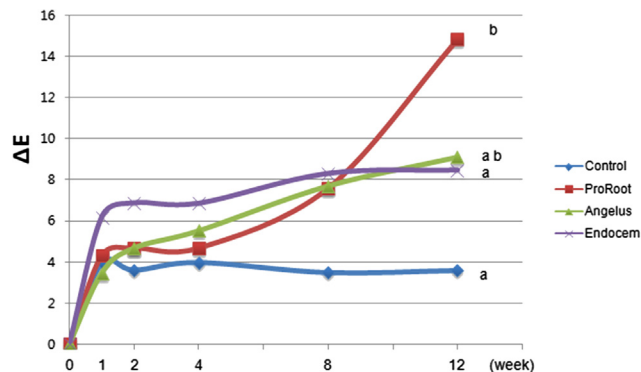


Figure 1. Change in the ΔE values during 12 weeks, measured at the cervical area. ΔE values indicate the differences in color, which were calculated by using the CIE $L^*a^*b^*$ values of 2 different measurements (L^* , luminosity; a^* , red-green parameter; b^* , yellow-blue parameter). Different letters indicate statistically significant differences between the groups at 12 weeks ($P < .05$).

The ΔE values were decreased significantly in all of the experimental groups after removal of the discolored MTA ($P < .05$). However, a sub-

sequent internal bleaching treatment did not significantly decrease the ΔE value compared with removal of MTA (Fig. 3).

Discussion

MTA satisfies many biological requisites for use as a vital pulp therapeutic material (6). However, tooth discoloration after MTA application is one of its main shortcomings, and this may be an esthetic concern when used on the anterior teeth. Several hypotheses have been proposed for the cause of MTA tooth discoloration (14, 15, 20); however, no report has clearly determined the mechanism.

In the present study, extracted teeth were used, and the teeth were endodontically treated so that we could apply the MTA materials over the GP cones. This pulpless tooth model is limited in its ability to appropriately reproduce the clinical and biological conditions of vital pulp therapy. However, this tooth model presented similar degrees and patterns of discoloration as did teeth treated with vital pulp therapy. To evaluate the tooth discoloration induced by endodontic materials, Lenherr et al (15) and Krastl et al (21) used extracted teeth and placed the endodontic materials within the dentin. Akbari et al (18) placed MTA materials over the GP cones in the dentinal root canals. These studies used extracted, nonvital teeth and reproduced significantly different tooth

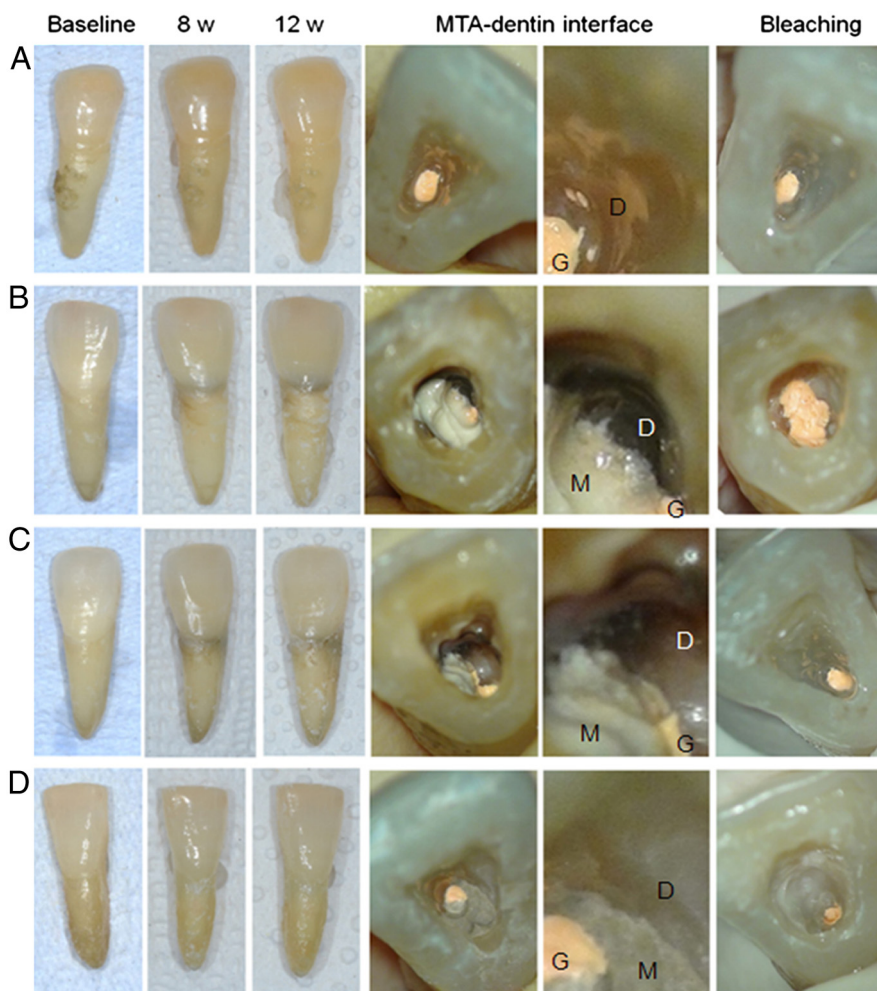


Figure 2. Photographs of discoloration at the time intervals after MTA application and microscopic images of the tooth sample after removal of MTA and bleaching treatment (original magnification, $\times 15$). (A) Control, (B) ProRoot, (C) Angelus. In the ProRoot and Angelus groups, the cervical areas of the tooth samples showed dark discoloration, and the MTA-dentin interface revealed evident dark marginal pigmentation that appeared to be spreading into the dentin. (D) Endocem. In this group, the marginal discoloration pattern was absent. Note the gray color of the Endocem material itself, which is different from the ProRoot and Angelus group. D, dentin; G, gutta-percha; M, MTA material.

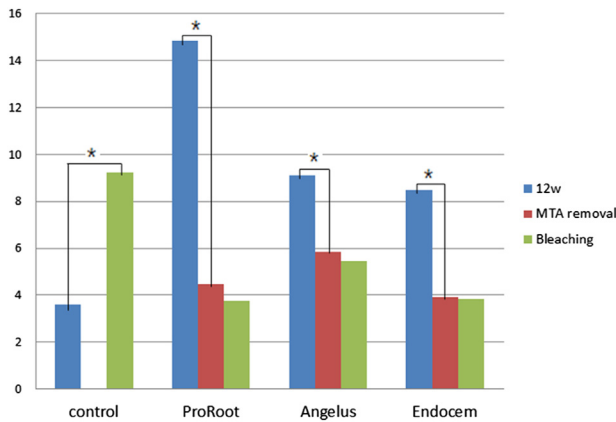


Figure 3. Change of the ΔE values after removal of MTA and bleaching. *Statistical significance ($P < .05$). ΔE values are coordinates of $L^*a^*b^*$ values between the baseline and each measuring point according to the following equation: $\Delta E = \{(L_2 - L_1)^2 + (a_2 - a_1)^2 + (b_2 - b_1)^2\}^{1/2}$. The control group presented an increase of the ΔE value after the bleaching treatment caused by the increase in the L^* value. Other experimental groups presented significant decreases in ΔE value after removal of MTA because it contributed to recovering the tooth color as similar to its original $L^*a^*b^*$ values.

discolorations that were based on different endodontic materials, and the results from our pilot study corroborated these findings.

In our study, not only the gross observation of the teeth by the naked eye (Fig. 2) but also the ΔE values supported the discoloration caused by MTA materials. We used the CIE $L^*a^*b^*$ system to evaluate the color change. The mean color difference (ΔE) in the control group at 12 weeks was 3.59. The ΔE values for the experimental groups at 12 weeks ranged from 8.46–14.85 (Table 1). The CIE $L^*a^*b^*$ system is one of the most commonly used systems because it approximates uniform distances between the color coordinates, while entirely covering the visual color space (22, 23). In the previous studies it was shown that ΔE values over 2.0 and 3.7 were clinically detectable and could be obtained by calculating the $L^*a^*b^*$ values (23, 24). Our results for the color difference were coincident with those results, and we observed that all of the teeth in the experimental groups showed significant discoloration.

The ProRoot and Angelus (WMTA) groups demonstrated spontaneous increases in ΔE over time (Fig. 1), and the MTA-dentin interfaces revealed dark marginal discoloration that appeared to spread into the dentin (Fig. 2B and C). This observed tooth discoloration pattern was similar to the data provided in previous reports (14, 21). Recent *in vivo* studies demonstrated that the by-products of MTA hydration were deposited into the material surface or MTA-dentin interface and also into the intratubular dentin (25, 26). This has been reported to be the result of biomineralization, and some tag-like structures formed at the interfaces (26–28). Han and Okiji (29) noted that the width of the tag-like structure that formed along the MTA-dentin interface increased during a 90-day period. They suggested that the calcium ions released from the MTA reacted with phosphate ions that are available in the tissue fluid, and this reaction resulted in the precipitation of carbonated apatite. Although the methodology used in this study has limited ability to identify the mechanism of discoloration, we speculate that some component of the MTA may be bound to the phosphate ion or plasma protein in the dentinal fluid. After the chemical reaction between these components, the by-product might be oxidized, followed by transformation into a pigmented by-product. This hypothesis needs to be tested in future investigations.

In the results of the present study, the discoloration that spread into the interior of the dentin in the WMTA groups was absent in the

Endocem group (Fig. 2D). The evident increase in the ΔE value during the first week in the Endocem group might be due to the material itself, which has a grayish color. The Endocem specimens used in the pilot study also presented little or no discoloration regardless of the storage medium. Endocem is a newly developed, fast-setting MTA-derived cement. The chemical composition of Endocem is similar to that of ProRoot. The composition provided by the manufacturer is CaO (46.7%), SiO₂ (12.8%), Al₂O₃ (5.4%), and other metallic oxides, and Bi₂O₃ (11%) is used as a radiopacifier. This pozzolan-based cement material chemically reacts in the presence of water, which is known as the pozzolanic reaction. The pozzolanic reaction occurs between calcium hydroxide, which is the product of cement hydration, and pozzolan. The reaction progresses in a manner similar to an acid-base reaction with oxides (SiO₂ + Al₂O₃ + Fe₂O₃) of the pozzolan (30). The small particle size of Endocem increases the surface contact of the particles while mixing with sterilized water, resulting in fast setting and ease of manipulation (17, 31, 32). A potential influence of the particle size might be the increase in surface area and hence the potential increase in the reactivity of the calcium silicate particles to form calcium hydroxide and calcium silicate hydrate phases. A recent *in vitro* study (17) demonstrated the rapid setting time of Endocem compared with ProRoot MTA. The final setting time of Endocem was 4 minutes \pm 30 seconds, whereas that of ProRoot was 261 \pm 21 minutes. This study also reported the biological effect of Endocem on the formation of the mineralization matrix by using osteogenic differentiation markers. The results indicated that the mineralization potential of Endocem was comparable with that of ProRoot. Further experimental studies are required to demonstrate the effect of rapid-setting Endocem on the time-consuming biomineralization behavior of conventional MTA.

In our study, the mean ΔE values in the experimental groups significantly decreased after removing the MTA (Fig. 3). Removing the MTA resulted in an increase in the L^* value, and this caused the ΔE value to decrease significantly, which meant the tooth recovered its original color. After a subsequent bleaching treatment, the remnant pigments at the MTA-dentin interface were completely removed in all samples on microscopic observation. There were slight decreases in the ΔE values, but this did not statistically contribute to lightening the tooth. Our results were similar to those from the case report by Belobrov and Parashos (16).

Considering the indicated use of MTA, few clinical cases would allow for the removal of the entire MTA, followed by the application of a strongly acidic bleaching agent. Moreover, Tsujimoto et al (33) observed that the application of a bleaching agent over the MTA resulted in the structural deterioration of the MTA surface because of the bleaching agent's acidic pH. The use of a bleaching agent should be prescribed only in limited cases, and it may contribute a minor improvement in discoloration.

Keeping in mind the limitations of our study, we conclude that the MTA-derived Portland cement causes tooth discoloration during the course of 12 weeks. In contrast, MTA-derived pozzolan cement did not affect the contacting dentin surface. Tooth discoloration from MTA-derived Portland cement occurred at the MTA-dentin interface and in the interior of the dentin, whereas the discoloration of the MTA-derived pozzolan cement was caused by the color of the material itself. Removing the discolored MTA materials contributed more to improving tooth discoloration than post-treatment bleaching.

Acknowledgments

The authors deny any conflicts of interest related to this study.

References

1. Lee SJ, Monsef M, Torabinejad M. Sealing ability of a mineral trioxide aggregate for repair of lateral root perforations. *J Endod* 1993;19:541–4.
2. Tsatsas DV, Meliou HA, Kerezoudis NP. Sealing effectiveness of materials used in furcation perforation *in vitro*. *Int Dent J* 2005;55:133–41.
3. Vajrabhaya LO, Korsuwannawong S, Jantararat J, Korre S. Biocompatibility of furcal perforation repair material using cell culture technique: Ketac Molar versus Pro-Root MTA. *Oral Surg Oral Med Oral Pathol Endod* 2006;102:e48–50.
4. Weldon JK Jr, Pashley DH, Loushine RJ, et al. Sealing ability of mineral trioxide aggregate and super-EBA when used as furcation repair materials: a longitudinal study. *J Endod* 2002;28:467–70.
5. Torabinejad M, Hong CU, McDonald F, Pitt Ford TR. Physical and chemical properties of a new root-end filling material. *J Endod* 1995;21:349–53.
6. Torabinejad M, Higa RK, McKendry DJ, Pitt Ford TR. Dye leakage of four root end filling materials: effects of blood contamination. *J Endod* 1994;20:159–63.
7. Parirokh M, Asgary S, Eghbal MJ, et al. A comparative study of white and grey mineral trioxide aggregate as pulp capping agents in dog's teeth. *Dent Traumatol* 2005; 21:150–4.
8. Karabucak B, Li D, Lim J, Iqbal M. Vital pulp therapy with mineral trioxide aggregate. *Dent Traumatol* 2005;21:240–3.
9. Bakland LK, Andreasen JO. Will mineral trioxide aggregate replace calcium hydroxide in treating pulp and periodontal healing complications subsequent to dental trauma? a review. *Dent Traumatol* 2012;28:25–32.
10. Maroto M, Barberia E, Planells P, Garcia Godoy F. Dentin bridge formation after mineral trioxide aggregate (MTA) pulpotomies in primary teeth. *Am J Dent* 2005;18:151–4.
11. Naik S, Hegde AH. Mineral trioxide aggregate as a pulpotomy agent in primary molars: an *in vivo* study. *J Indian Soc Pedod Prev Dent* 2005;23:13–6.
12. Holland R, de Souza V, Nery MJ, et al. Reaction of rat connective tissue to implanted dentin tube filled with mineral trioxide aggregate, Portland cement or calcium hydroxide. *Braz Dent J* 2001;12:3–8.
13. Asgary S, Parirokh M, Eghbal MJ, Brink F. Chemical differences between white and gray mineral trioxide aggregate. *J Endod* 2005;31:101–3.
14. Boutsioukis C, Noula G, Lambrianidis T. *Ex vivo* study of the efficiency of two techniques for the removal of mineral trioxide aggregate used as a root canal filling material. *J Endod* 2008;34:1239–42.
15. Lenherr P, Allgayer N, Weiger R, et al. Tooth discoloration induced by endodontic materials: a laboratory study. *Int Endod J* 2012;45:942–9.
16. Belobrov I, Parashos P. Treatment of tooth discoloration after the use of white mineral trioxide aggregate. *J Endod* 2011;37:1017–20.
17. Choi Y, Park SJ, Lee SH, et al. Biological effects and washout resistance of a newly developed fast setting pozzolan cement. *J Endod* 2013;39:467–72.
18. Akbari M, Rouhani A, Samiee S, Jafarzadeh H. Effect of dentin bonding agent on the prevention of tooth discoloration produced by mineral trioxide aggregate. *Int J Dent* 2012;2012:563203.
19. International Commission on Illumination. *Recommendations on Uniform Color Spaces, Color-difference Equations, Psychometric Color Terms*. Bureau Central de la CIE 1978;15:13.
20. Valles M, Mercade M, Duran-Sindreu F, et al. Color stability of white mineral trioxide aggregate. *Clin Oral Investig* 2013;17:1155–9.
21. Krastl G, Allgayer N, Lenherr P, et al. Tooth discoloration induced by endodontic materials: a literature review. *Dent Traumatol* 2013;29:2–7.
22. Seghi RR. Effects of instrument-measuring geometry on colorimetric assessments of dental porcelains. *J Dent Res* 1990;69:1180–3.
23. Johnston WM, Kao EC. Assessment of appearance match by visual observation and clinical colorimetry. *J Dent Res* 1989;68:819–22.
24. O'Brien WJ, Groh CL, Boenke KM. A new, small-color-difference equation for dental shades. *J Dent Res* 1990;69:1762–4.
25. Bird DC, Komabayashi T, Guo L, et al. *In vitro* evaluation of dentinal tubule penetration and biomineralization ability of a new root-end filling material. *J Endod* 2012;38:1093–6.
26. Dreger LA, Felipe WT, Reyes-Carmona JF, et al. Mineral trioxide aggregate and Portland cement promote biomineralization *in vivo*. *J Endod* 2012;38:324–9.
27. Reyes-Carmona JF, Felipe MS, Felipe WT. Piomineralization ability and interaction of mineral trioxide aggregate and white Portland cement with dentin in a phosphate-containing fluid. *J Endod* 2009;35:731–6.
28. Chang SW. Chemical characteristics of mineral trioxide aggregate and its hydration reaction. *Restor Dent Endod* 2012;37:188–93.
29. Han L, Okiji T. Uptake of calcium and silicon released from calcium silicate-based endodontic materials into root canal dentine. *Int Endod J* 2011;44:1081–7.
30. Askarinejad A, Pourkhorshidi AR, Parhizkar T. Evaluation the pozzolanic reactivity of sonochemically fabricated nano natural pozzolan. *Ultrason Sonochem* 2012;19: 119–24.
31. Parirokh M, Torabinejad M. Mineral trioxide aggregate: a comprehensive literature review—part I: chemical, physical, and antibacterial properties. *J Endod* 2010;36:16–27.
32. Byun SH, Kim HC, Kim JY, et al. Effect of cement particle size on properties of ordinary Portland cement. *J Kor Ceram Soc* 2010;47:394–400.
33. Tsujimoto M, Ookubo A, Wada Y, et al. Surface changes of mineral trioxide aggregate after the application of bleaching agents: electron microscopy and an energy-dispersive X-ray microanalysis. *J Endod* 2011;37:231–4.