

## Microleakage Assessment of a Pozzolan Cement-based Mineral Trioxide Aggregate Root Canal Sealer

Mijun Kim, Howon Park, Juhyun Lee, Hyunwoo Seo

*Department of Pediatric Dentistry, Oral Science Research Center, College of Dentistry, Gangneung-Wonju National University*

### Abstract

This study aimed to assess microleakage of Endoseal MTA when it is used as a root canal sealer and a root canal filling material compared with conventional endodontic treatment materials. Forty-two mature human permanent teeth with a single root canal were divided randomly into three experimental groups ( $n = 10$ ) and two control groups ( $n = 6$ ). Group A was obturated with AH plus<sup>®</sup> and gutta-percha (GP). Group E1 was obturated with Endoseal MTA and GP. Group E2 was obturated with Endoseal MTA only. The positive control group was obturated with GP only and the negative control group was obturated in the same way as the experimental groups. The samples were kept in saline solution for 24 hours and were immersed in 0.2% rhodamine B dye solution for 24 hours. Then the samples were split longitudinally and the microleakage was assessed under a stereomicroscope. Complete microleakage was detected in all positive control group samples, whereas no microleakage was detected in the negative control group. There was no statistically significant difference between the experimental groups in the Kruskal-Wallis test. These results suggest that Endoseal MTA has potential use as a root canal sealer and a root canal filling material.

**Key words :** Pozzolan, Endoseal MTA, Mineral trioxide aggregate, Root canal sealer, Root canal filling material, Microleakage

### I . Introduction

Most endodontic treatment failures are caused by microleakage that occurs at the interface between the dentin and sealer or the core filling material and the sealer<sup>1-3)</sup>. Thus, good sealing ability of a root canal sealer and a root canal filling material is required for an ideal endodontic material.

Mineral trioxide aggregate (MTA) has been reported to have superior physical properties, biocompatibility, and sealing ability compared to other materials used during endodontic treatment<sup>4)</sup>. MTA was originally de-

veloped as a root-end filling material and has been used increasingly for pulp capping, root perforation repair, and apexogenesis due to its advantages. However, use of MTA as an orthograde root canal filling material is controversial and is related to decreased quality of the orthograde MTA seal caused by the difficulty delivering the material, the formation of voids, poor adaptation of MTA to the canal wall, and wash-out of MTA due to long setting time<sup>5-9)</sup>.

Some MTA-like products have been developed to overcome these problems. A new calcium silicate sealer (Endoseal MTA, Maruchi, Wonju, Korea) is based on

*Corresponding author : Hyunwoo Seo*

*Department of Pediatric Dentistry, College of Dentistry, Gangneung-Wonju National University, 7 Jukheon-gil, Gangneung, 25457, Korea*

*Tel: +82-33-640-2758 / Fax: +82-33-640-3113 / E-mail: hwseo@gwnu.ac.kr*

*Received March 9, 2016 / Revised June 14, 2016 / Accepted May 25, 2016*

*※ This study was supported by the research fund of Gangneung-Wonju National University Dental Hospital, 2015.*

MTA and is derived from pozzolan cement. Pozzolan is a siliceous material, which has few cementitious properties, but reacts with calcium hydroxide in the presence of water to form compounds with cementing properties<sup>10</sup>. According to the manufacturer's manual, Endoseal MTA is characterized by a fast setting time due to an extremely fine silica particle size in the pozzolan cement. MTA products derived from pozzolan cement show faster setting time and higher wash-out resistance than those of other MTA products<sup>11,12</sup>. Other studies about pozzolan cement derived-MTA have noted properties, such as low cytotoxicity, bioactivity inducing hard tissues, and minimal discoloration<sup>11-17</sup>. The Endoseal MTA manufacturer also suggests that it can be used as a root canal sealer and as a root canal filling material, as it allows complete obturation of the entire root canal, including accessory and lateral canals, regardless of whether it is used alone or with core material. These superior properties of Endoseal MTA, such as fast setting time, improved workability, and physical properties suitable for orthograde filling material, may improve the treatment prognosis and simplify the endodontic treatment procedure.

Although a study has reported bacterial leakage from Endoseal MTA used as a root canal sealer, no study has reported on microleakage of Endoseal MTA used as a root canal sealer or a root canal filling material. Thus, the aim of this study was to assess microleakage of Endoseal MTA when it is used as a root canal sealer and a root canal filling material compared with conventional endodontic treatment materials.

## II. Materials and methods

This study was conducted after approval from the Institutional Review Board (IRB) of Gangneung-Wonju National University Dental Hospital (IRB No. : 2015-04) and all experimental procedures were performed by a single operator.

### 1. Tooth selection and preparation

Forty-two mature human permanent teeth with a single root canal were collected from Gangneung-Wonju National University Dental Hospital. Periapical radiographs of all teeth were taken and teeth with a curved root, internal or external resorption, deep caries, or canal calcification were excluded. The soft tissues of the root surfaces were scraped off with a No. 15 surgical blade (Father Safety Razor Co., Ltd., Osaka, Japan) and were kept in saline solution replaced daily until use. The teeth were decoronated 14 mm from the apex using a low-speed diamond disc (Brasseler GmbH & Co. KG, Lemgo, Germany) under running tap water to standardize the length of the roots.

### 2. Root canal preparation

A size 10 K-file (Mani Inc., Tochigi, Japan) was inserted into the root canal until it was visible at the apical foramen, and the working length was 1 mm short of total root canal length. Root canals were prepared using S1, S2, F1, F2, and F3 ProTaper<sup>®</sup> Universal (Dentsply Maillefer, Ballaigues, Switzerland) nickel-titanium files and the WaveOne<sup>™</sup> endo motor (Dentsply Maillefer), according to the manufacturer's instructions. The root canals were irrigated with 3 mL of 2.5% sodium hypochlorite between each file and then a final irrigation was performed with 10 mL of saline. The root canals were dried with F3 Dia-ProT<sup>™</sup> paper points (Diadent Group International, Chungcheongbuk-do, Korea).

### 3. Root canal obturation

The compositions of the root canal sealers used in this study are presented in Table 1. The samples were divided randomly into three experimental and two control groups (Table 2).

**Table 1.** Compositions of the root canal sealers used in this study

Root canal sealer	Composition	Manufacturer
AH Plus <sup>®</sup>	Paste A: bisphenol-A epoxy resin, bisphenol-F epoxy resin, calcium tungstate, zirconium oxide, silica, iron oxide pigments Paste B: dibenzylidiamine, aminoadamantane, tricyclodecane-diamine, calcium tungstate, zirconium oxide, silica, silicone oil	Dentsply DeTrey, Konstanz, Germany
Endoseal MTA	Calcium silicates, calcium aluminates, calcium aluminoferrite, calcium sulfates, radiopacifier, thickening agent	Maruchi, Wonju, Korea

**Table 2.** Experimental and control group classification

Group		Root canal obturation	Root covering
A (n = 10)		AH Plus <sup>®</sup> + Dia-ProT plus <sup>™</sup> GP	Covering with two layers of nail varnish except apical foramen around 2mm
E1 (n = 10)		Endoseal MTA + Dia-ProT plus <sup>™</sup> GP	
E2 (n = 10)		Endoseal MTA	
Positive control (n = 6)		Dia-ProT plus <sup>™</sup> GP	
Negative control (n = 6)	N1 (n = 2)	AH Plus <sup>®</sup> + Dia-ProT plus <sup>™</sup> GP	Covering with two layers of nail varnish including apical foramen
	N2 (n = 2)	Endoseal MTA + Dia-ProT plus <sup>™</sup> GP	
	N3 (n = 3)	Endoseal MTA	

GP = gutta-percha

Group A was obturated with AH Plus<sup>®</sup> (Dentsply DeTrey, Konstanz, Germany) and F3 Dia-ProT plus<sup>™</sup> gutta-percha (GP) (Diadent Group International, Chungcheongbuk-do, Korea) using the single-cone obturation technique. AH Plus<sup>®</sup> was mixed according to the manufacturer's instructions and applied inside the canal using the lentulo spiral (Mani Inc.).

Group E1 was obturated with Endoseal MTA and F3 Dia-ProT plus<sup>™</sup> GP using the single-cone obturation technique. According to the manufacturer's instruction, a needle tip containing Endoseal MTA was inserted into the root canal no deeper than the coronal one-third, and the Endoseal MTA was applied smoothly. Immediately after the GP was inserted into the root canal at full working length, ultrasonic vibration was applied to the pincette holding the GP in order to make the material was applied to the apical portion.

Group E2 was obturated with Endoseal MTA only. A needle tip containing Endoseal MTA was inserted to reach the apical foramen, and the Endoseal MTA was applied gently while pulling the syringe back passively until the needle reached the orifice. A new needle tip containing Endoseal MTA was used for each tooth.

The positive control group was obturated with GP only without any other root canal sealer. The negative control group was obturated in the same way as the experimental groups.

Excess canal filling material was cut off and removed at the orifice level. The coronal portion was sealed with intermediate restorative material (Dentsply International, Caulk, Milford, DE, USA). Periapical radiographs were taken of all samples to confirm adequacy of root canal obturation. The three experimental groups and the positive group were covered with two layers of nail varnish, except the 2 mm area around the apical

foramen; the negative control group was covered entirely including the apical foramen. The samples were kept in saline solution for 24 hours in a 37°C incubator to set the canal filling materials.

#### 4. Microleakage test

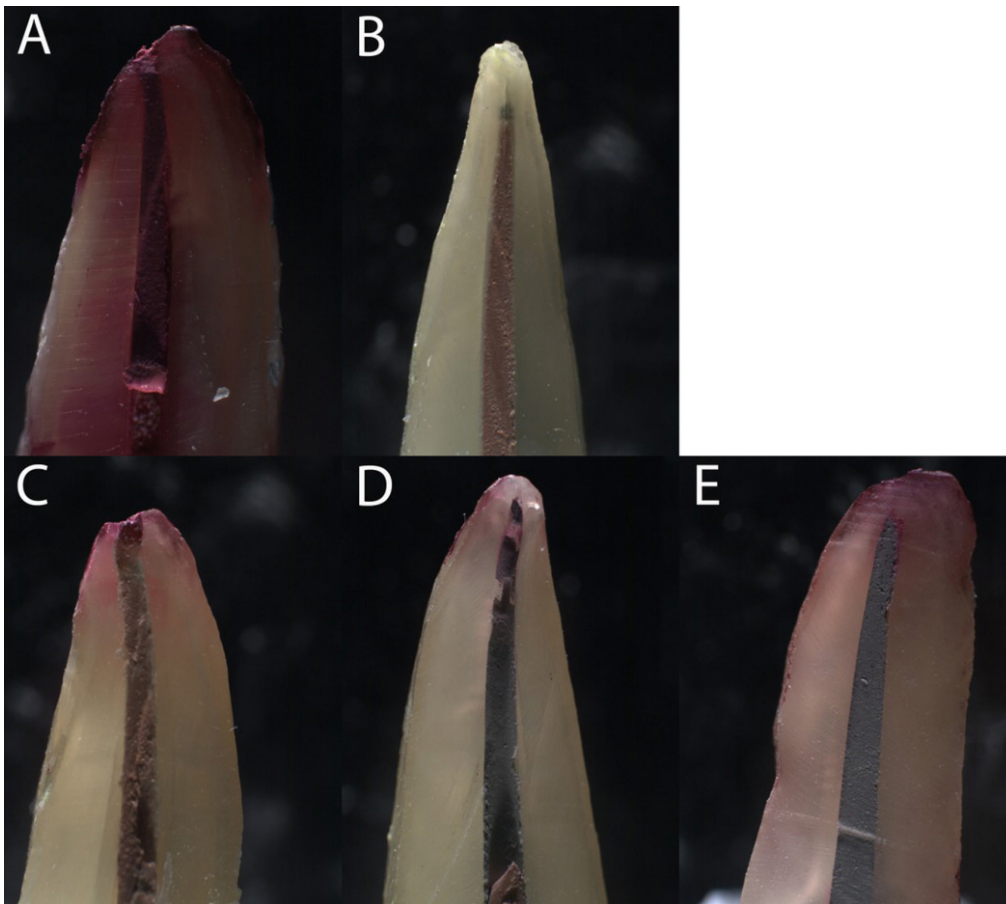
The apical one-third of the samples was immersed in 0.2% rhodamine B dye solution for 24 hours in a 37°C incubator, followed by rinsing under tap water. The nail varnish was removed with a No. 15 surgical blade and the samples were split longitudinally using a 0.2 mm low-speed diamond disc (Brasseler). Microleakage was observed and photographed under a Leica MZ125 stereomicroscope (Leica Microsystems, Heerbrugg, Switzerland) at ×16 magnification and measured using Leica application suite v2.7.0. The length (mm) of microleakage was calculated from the apex to the most deeply dyed portion. The larger value of the two measured microleakage length in each splitted sample was adopted.

#### 5. Statistical analysis

Data were analyzed using the Kruskal-Wallis test to confirm differences among the three experimental groups using IBM SPSS statistics ver. 21 software (IBM Corp., Armonk, NY, USA). The significance level was set at 5%.

### III. Results

Complete microleakage was detected in all positive control group samples; no microleakage was detected in the negative control group (Fig. 1). Table 3 shows the



**Fig. 1.** Representative stereomicroscope images of microleakage ( $\times 16$ ). (A) Positive control group shows complete microleakage along the walls; (B) negative control group shows absence of microleakage along the walls; (C) group A; (D) group E1; and (E) group E2 show similar microleakage length.

**Table 3.** Length of microleakage in the three experimental groups

Group	Length of Microleakage
	Mean $\pm$ SD (mm)
A (AH Plus <sup>®</sup> + GP)	1.082 $\pm$ 0.9023
E1 (Endoseal MTA + GP)	1.692 $\pm$ 1.1274
E2 (Endoseal MTA)	1.197 $\pm$ 0.9635

mean length and standard deviation of microleakage in the three experimental groups. Group A (1.082  $\pm$  0.9023 mm) showed the lowest value, followed by group E2 (1.197  $\pm$  0.9635 mm), and group E1 (1.692  $\pm$  1.1274 mm) had the largest value among the experimental groups. But, there was no significant difference in length of microleakage among the three experimental groups ( $p = 0.603$ , Table 3).

#### IV. Discussion

The sealing ability of materials used for endodontic treatment is a significant factor affecting treatment success. The dye penetration test is the most commonly used method to evaluate the sealing ability and is more exact than the bacterial leakage test because the particle size of the dye is extremely small<sup>18</sup>. Methylene blue is the dye used in many dye penetration tests, but it shows decoloration over time when contacting calcium hydroxide and MTA because its chemical structure is unstable with caustic alkali<sup>19</sup>. Rhodamine B used in this study has greater penetration and visualization ability than that of methylene blue and was noted that it is the most suitable dye to assess sealing ability of materials such as MTA<sup>20,21</sup>.

In this study, the single-cone obturation technique was used as the root canal obturation method. This technique shows the sealing ability of a root canal sealer more directly by excluding other factors that affect microleakage, such as the interface between accessory cones<sup>22</sup>. Several studies have compared the sealing ability of the single-cone technique with that of the lateral compaction technique and have found no differences between the two methods<sup>23,24</sup>.

AH Plus<sup>®</sup> is an epoxy resin-based sealer with superior properties, such as low solubility, little shrinkage after polymerization, and good adhesion to dentin<sup>25,26</sup>. Many studies have also shown its good sealing ability that is similar or superior to other root canal sealers<sup>27</sup>. Endodontic treatment with AH Plus<sup>®</sup> and GP is a conventional method used commonly by clinicians. Hwang et al.<sup>28</sup> reported that Endoseal MTA as a root canal sealer shows similar bacterial leakage with that of AH Plus<sup>®</sup> and GuttaCore<sup>®</sup> (Dentsply Tulsa Dental Specialties), which is consistent with our result.

The powder/liquid (P/L) ratio, setting time, setting expansion, and solubility are associated with the sealing ability of a root canal sealer. Endoseal MTA is a pre-mixed single paste that includes hydraulic cement, non-aqueous liquid, and the radiopacifier. Therefore, a constant P/L ratio can be maintained when applying it. Endoseal MTA is also characterized by fast setting time, as pozzolan cement-based MTA has extremely fine silica particles added as hydraulic cement. Endoseal MTA shows larger expansion than that of AH Plus<sup>®</sup>, and appropriate setting expansion may contribute to good sealing ability<sup>29,30</sup>. Despite these favorable properties, Endoseal MTA showed similar sealing ability to that of AH Plus<sup>®</sup> in our study, which may be associated with the higher solubility of Endoseal MTA than that of the AH Plus<sup>®</sup>. According to the Endoseal MTA manufacturers, the solubility of Endoseal MTA is 0.7% and that of AH Plus<sup>®</sup> is 0.06%. Lim et al.<sup>30</sup> verified that Endoseal MTA is more soluble than AH Plus<sup>®</sup>, although the exact value was not presented. However, the solubility rate of MTA decreased over 78 days<sup>31</sup>. Therefore, further studies are needed to evaluate the long-term sealing ability of Endoseal MTA.

In studies of other root canal sealers based on MTA, it showed diverse results depending on the composition and physical properties of the products, study methods, or experimental conditions.

From the study of Gomes-Filho et al.<sup>22</sup>, MTA Fillapex

(Angelus, Paraná, Brazil) had more superior sealing ability than Endo-CPM<sup>®</sup> sealer (EGEO S.R.L., Buenos Aires, Argentina). Sonmez et al.<sup>32</sup> reported that MTA Fillapex showed more leakage than AH plus; the result corresponded with the finding of Ehsani et al.<sup>33</sup>. MTA Fillapex is MTA-based sealer with additional natural resin and Endo-CPM<sup>®</sup> sealer is composed of MTA-like material and calcium carbonate. Maybe, the different physical properties which are derived from the differences in compositions of each product could explain these consequences.

According to Parwar et al.<sup>34</sup>, the Endosequence<sup>®</sup> BC sealer (Brasseler), which is a calcium silicate based sealer, leaked less than AH Plus<sup>®</sup>. It was assumed that his result was obtained through the 0.2% setting expansion of Endosequence<sup>®</sup> BC sealer. This finding differed from ours, perhaps due to decoloration of methylene blue.

MTA has been not recommended as an orthograde root canal filling material for permanent teeth with a closed apex due to long setting time, poor workability, and removal difficulty. However, the manufacturer suggests that Endoseal MTA can be used alone as an orthograde root canal filling material due to fast setting time, improved workability, and the needle-like crystal structure having a low strength. It is also suggested that the teeth can be re-treated by conventional re-endodontic technique in case of poor prognosis observed. However, further studies are needed to verify the relevant facts. Until now, no study has reported sealing ability of Endoseal MTA used as a root canal filling material. We verified that Endoseal MTA used alone as a canal filling material had similar sealing ability to that of GP with AH Plus<sup>®</sup>, which is a conventional filling material. This result indicates that Endoseal MTA will be valuable as a root canal filling material after considering sealing ability and better workability.

## V. Conclusion

From the results, Endoseal MTA has sealing ability similar to that of AH Plus<sup>®</sup> when used as a root canal sealer and for obturating with AH Plus<sup>®</sup> and GP using the single-cone obturation technique when used as a root canal filling material. Endoseal MTA also showed relatively constant sealing ability, regardless of whether it was used alone or with GP. These results suggest that Endoseal MTA has potential use as a root canal sealer and an orthograde root canal filling material when seal-

ing ability is evaluated.

## References

1. Hoen MM, Pink FE : Contemporary endodontic retreatments: an analysis based on clinical treatment findings. *J Endod*, 28:834-836, 2002.
2. Siqueira JF, Jr., Rocas IN : Clinical implications and microbiology of bacterial persistence after treatment procedures. *J Endod*, 34:1291-1301 e1293, 2008.
3. Ng YL, Mann V, Rahbaran S, *et al.* : Outcome of primary root canal treatment: systematic review of the literature — Part 2. Influence of clinical factors. *Int Endod J*, 41:6-31, 2008.
4. Torabinejad M, Chivian N : Clinical applications of mineral trioxide aggregate. *J Endod*, 25:197-205, 1999.
5. Hachmeister DR, Schindler WG, Walker WA, Thomas DD : The sealing ability and retention characteristics of mineral trioxide aggregate in a model of apexification. *J Endod*, 28:386-390, 2002.
6. Vizgirda PJ, Liewehr FR, Patton WR, *et al.* : A comparison of laterally condensed gutta-percha, thermoplasticized gutta-percha, and mineral trioxide aggregate as root canal filling materials. *J Endod*, 30:103-106, 2004.
7. Aminoshariae A, Hartwell GR, Moon PC : Placement of mineral trioxide aggregate using two different techniques. *J Endod*, 29:679-682, 2003.
8. Lawley GR, Schindler WG, Walker WA, Kolodrubetz D : Evaluation of ultrasonically placed MTA and fracture resistance with intracanal composite resin in a model of apexification. *J Endod*, 30:167-172, 2004.
9. Yeung P, Liewehr FR, Moon PC : A quantitative comparison of the fill density of MTA produced by two placement techniques. *J Endod*, 32:456-459, 2006.
10. Malhotra VM, Mehta PK : Pozzolan and cementitious materials. Amsterdam, The Netherlands: Gordon and Breach; 1996.
11. Choi Y, Park SJ, Lee SH, *et al.* : Biological effects and washout resistance of a newly developed fast-setting pozzolan cement. *J Endod*, 39:467-472, 2013.
12. Jang GY, Park SJ, Heo SM, *et al.* : Washout resistance of fast-setting pozzolan cement under various root canal irrigants. *Restor Dent Endod*, 38:248-252, 2013.
13. Kim M, Yang W, Kim H, Ko H : Comparison of the biological properties of ProRoot MTA, OrthoMTA, and Endocem MTA cements. *J Endod*, 40:1649-1653, 2014.
14. Song M, Yoon TS, Kim SY, Kim E : Cytotoxicity of newly developed pozzolan cement and other root-end filling materials on human periodontal ligament cell. *Restor Dent Endod*, 39:39-44, 2014.
15. Han L, Kodama S, Okiji T : Evaluation of calcium-releasing and apatite-forming abilities of fast-setting calcium silicate-based endodontic materials. *Int Endod J*, 48:124-130, 2015.
16. Park SJ, Heo SM, Hong SO, *et al.* : Odontogenic effect of a fast-setting pozzolan-based pulp capping material. *J Endod*, 40:1124-1131, 2014.
17. Jang JH, Kang M, Ahn S, *et al.* : Tooth discoloration after the use of new pozzolan cement (Endocem) and mineral trioxide aggregate and the effects of internal bleaching. *J Endod*, 39:1598-1602, 2013.
18. Oppenheimer S, Rosenberg PA : Effect of temperature change on the sealing properties of Cavit and Cavit G. *Oral Surg Oral Med Oral Pathol*, 48:250-253, 1979.
19. Wu MK, Kontakiotis EG, Wesselink PR : Decoloration of 1% methylene blue solution in contact with dental filling materials. *J Dent*, 26:585-589, 1998.
20. Vogt BF, Xavier CB, Demarco FF, Padilha MS : Dentin penetrability evaluation of three different dyes in root-end cavities filled with mineral trioxide aggregate (MTA). *Braz Oral Res*, 20:132-136, 2006.
21. Tanomaru Filho M, Figueiredo FA, Tanomaru JM : Effect of different dye solutions on the evaluation of the sealing ability of Mineral Trioxide Aggregate. *Braz Oral Res*, 19:119-122, 2005.
22. Gomes-Filho JE, Moreira JV, Watanabe S, *et al.* : Sealability of MTA and calcium hydroxide-containing sealers. *J Appl Oral Sci*, 20:347-351, 2012.
23. Samiei M, Aghazade M, Farhadi F, *et al.* : Sealing efficacy of single-cone obturation technique with MTA and CEM cement: An in vitro bacterial leakage study. *J Dent Res Dent Clin Dent Prospects*, 8:77-83, 2014.
24. Antonopoulos KG, Attin T, Hellwig E : Evaluation of the apical seal of root canal fillings with different

- methods. *J Endod*, 24:655-658, 1998.
25. McMichen FR, Pearson G, Rahbaran S, Gulabivala K : A comparative study of selected physical properties of five root-canal sealers. *Int Endod J*, 36:629-635, 2003.
  26. Gogos C, Economides N, Stavrianos C, *et al.* : Adhesion of a new methacrylate resin-based sealer to human dentin. *J Endod*, 30:238-240, 2004.
  27. Erdemir A, Adanir N, Belli S : In vitro evaluation of the dissolving effect of solvents on root canal sealers. *J Oral Sci*, 45:123-126, 2003.
  28. Hwang JH, Chung J, Na HS, *et al.* : Comparison of bacterial leakage resistance of various root canal filling materials and methods: Confocal laser-scanning microscope study. *Scanning*, 37:422-428, 2015.
  29. Donnelly A, Sword J, Nishitani Y, *et al.* : Water sorption and solubility of methacrylate resin-based root canal sealers. *J Endod*, 33:990-994, 2007.
  30. Lim ES, Park YB, Kwon YS, *et al.* : Physical properties and biocompatibility of an injectable calcium-silicate-based root canal sealer: in vitro and in vivo study. *BMC Oral Health*, 15:129, 2015.
  31. Fridland M, Rosado R : Mineral trioxide aggregate (MTA) solubility and porosity with different water-to-powder ratios. *J Endod*, 29:814-817, 2003.
  32. Sonmez IS, Oba AA, Sonmez D, Almaz ME : In vitro evaluation of apical microleakage of a new MTA-based sealer. *Eur Arch Paediatr Dent*, 13:252-255, 2012.
  33. Ehsani M, Dehghani A, Abesi F, *et al.* : Evaluation of Apical Micro-leakage of Different Endodontic Sealers in the Presence and Absence of Moisture. *J Dent Res Dent Clin Dent Prospects*, 8:125-129, 2014.
  34. Pawar SS, Pujar MA, Makandar SD : Evaluation of the apical sealing ability of bioceramic sealer, AH plus & epiphany: An in vitro study. *J Conserv Dent*, 17:579-582, 2014.

국문초록

## 포졸란 시멘트를 기반으로 하는 근관전색제의 치근단부 미세누출 평가

김미준 · 박호원 · 이주현 · 서현우

강릉원주대학교 치과대학 소아치과학교실 및 구강과학연구소

본 연구는 포졸란 시멘트를 기반으로 하는 근관전색제인 Endoseal MTA (Maruchi, Wonju, Korea)를 근관전색제와 근관충전제로 사용하였을 때 나타나는 치근단부 미세누출을 기존의 근관전색제인 AH Plus® (Dentsply DeTrey, Konstanz, Germany)와 비교하여 평가하고자 하였다.

만곡되지 않은 영구 전치를 3개의 실험군(A군, E1군, E2군)으로 나누고 A군은 AH Plus®와 gutta-percha (GP), E1군은 Endoseal MTA와 GP, E2군은 Endoseal MTA 단독으로 충전하였다.

충전이 완료된 치아는 완전한 경화를 위해 24시간동안 멸균 증류수에 보관하였고 염색액 누출을 위해 치근단 부위를 0.2% 로다민 B 염색 용액에 24시간 동안 노출시켰다.

입체 현미경을 이용하여 미세누출 정도를 평가하였으며 3개의 실험군 사이에 유의한 차이가 관찰되지 않았다.

결론적으로 Endoseal MTA는 GP의 사용에 관계 없이 AH Plus®와 유사한 밀폐력을 보였으며 이러한 결과는 Endoseal MTA의 근관전색제 및 근관 충전제로써 유용한 사용에 대한 가능성을 제시한다.

**주요어:** 포졸란, Endoseal MTA, Mineral trioxide aggregate, 근관 전색제, 근관 충전 재료, 미세누출