산업기술연구(강원대학교 산업기술연구소 논문집), 제31권 A호, 2011. Journal of Industrial Technology, Kangwon Natl. Univ., Korea, No. 31 A, 2011.

# The chelation of EDTA sodium salts solution for the root canal dentin treatment

상아질 치관 치료를 위한 EDTA Na염의 킬레이션

송 근 호· 장 화 익<sup>··</sup> 이 광 래<sup>···</sup> Song, Kun-Ho Chang, Hwa-Ik Lee, Kwang-Rae

# Abstract

The demineralizing effect of chelating agents(Maleic acid solution, EDTA salts solutions; disodium-, trisodium- and tetrasodium-EDTA) on hydroxyapatite(HA) for the root canal dentin treatment was investigated. Dissolution of Ca from HA was increased with increasing immersion time in chelating solutions. Among the EDTA salts solutions, tetrasodium-EDTA was least effective in dissolving Ca out from HA, and trisodium-EDTA was the most effective. Trisodium-EDTA solutionnight be more effective in removing the smear layer compared to tetrasodium-EDTA solution.

키워드 : 킬레이트제, 상아질, 스미어 층, EDTA Keywords : chelating agents, dentin, smear layer, EDTA

#### 1. Introduction

The key to endodontic success is to get rid of bacteria in the root canal and to provide a good seal of root canal filling materials, because the inflammatory and immunological reaction in the periapical area are caused by bacteria and their toxin, immunologic agents, tissue debris and products of tissue necrosis from the pulp. During canal preparation[1], dentin chips created by the action of endodontic instruments add to the remnants of organic material and irrigating solutions, forming a smear layer that adheres to the canal walls. Smear layer formed may harbor micro-organisms as well that might cause re-infection and, it may interfere in the sealing of root canal during obturation. It is desirable to have a chemical adjunct[1][2], which removes the smear layer and possesses antimicrobial activity. For endotoxins and smear layer removal, different agents such as citric acid[2][3], ethylenediaminetetraacetic acid (EDTA)[4] and tetracycline hydrocloride[5] have been employed. The decalcifying agents at a neutral pH[6], such as EDTA, have recently demonstrated that it not only preserves the vitality of the remaining periodontal cells close to the root surface, but also removes calcium ions selectively from the collagenous dentin matrix[7][8]. The chelating agent EDTA (ethylenediaminetetraacetic acid)

 <sup>\*</sup> 강원테크노파크 강원선도산업지원단, 공학박사
\*\* 강원지방중소기업청

<sup>\*\*\*</sup> 강원대학교 화학공학과 교수, 공학박사, 교신 저자

with the calcium the reacts ions in hydroxyapatite(HA) crystals. The chelation process can cause changes in the microstructure of the dentin and changes in the Ca:P ratio. The EDTA in different formulations is extensively used to remove the smear laver formed during the chemomechanical preparation of the root canal. However, there are few studies comparing the action of this chelating agent in its sodium salts.

Thus, the purpose of the present study was to evaluate the demineralizing ability of various sodium salts of EDTA (disodium-, trisodium-, and tetrasodium- EDTA) on the root canal dentin treatment. Hydroxyapatite(HA) was employed to compare the  $Ca^{+}$ -chelating power of various EDTA sodium salts.

## 2. Experimentals

The sodium salts of EDTA (disodium-, trisodium-, and tetrasodium -EDTA) were procured from Junsei Chemicals. The pH of the solutions was adjusted to pH8 by the HCl and NaOH. The hydroxyapatite(HA) was procured from SunKoo Ltd.(Korea) and used without further purification. The hydroxyapatite(HA) powder was pressed and molded as tablets(thickness : 5mm, radius : 13mm) at about 2,000psi with Laboratory Press (Carver). The tablet specimens were then subjected to the test solution ; HA specimens were immersed for 5, 10 and 15min into 5wt%, 10wt% and 15wt% of the EDTA sodium salt solutions. The concentration of Ca<sup>+</sup> ion dissolved out from HA specimen was analyzed with ICP(Inductively Coupled Plasma) Optical Emission Spectrometer (Leeman Profile, Leeman Labs Inc.)

### 3. Results and Discussion

3.1 The effect of the concentration of EDTA sodium salt solution :

The effect of disodium-EDTA concentration on the dissolution of Ca ion (Ca<sup>+</sup>) from HA was investigated. For the disodium, trisodium- and tetrasodium- EDTA, Fig.1, Fig.2 and Fig.3 showed that the amount of dissolved Ca<sup>+</sup> from HA was not much dependent on the concentration of EDTA sodium salt ranging from 5~15wt% in aqueous solution. These experimental data means that it is not necessary to use above a certain concentration of EDTA solution; in this case, 5wt% of EDTA. However, the amount of dissolved Ca<sup>+</sup> was strongly dependent on the immersion time in aqueous solution of EDTA sodium salt . At the 5min immersion time in 5wt% EDTA aqueous solution, the amount of Ca<sup>+</sup> dissolved out from HA were 250ppm, 258ppm and 181ppm in di-, triand tetra-sodium EDTA solution, respectively. The dissolution of Ca<sup>+</sup> from HA by disodium- and trisodium-EDTA were nearly same, but was the least by tetrasodium-EDTA. Even though the ability of trisodium-EDTA for dissolving Ca<sup>+</sup> out from HA was similar to that of disodium-EDTA, the experimental data showed that trisodium-EDTA was rather preferable to disodium-EDTA for root canal treatment because pH of trisodium-EDTA solution was close to neutral.

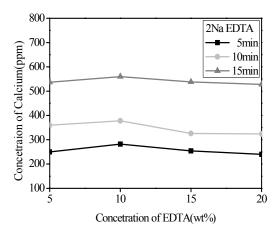


Fig.1. Dissolution of Ca from HA by disodium-EDTA solution

산업기술연구(강원대학교 산업기술연구소 논문집), 제31권 A호, 2011. The chelation of EDTA sodium salts solution for the root canal dentin treatment

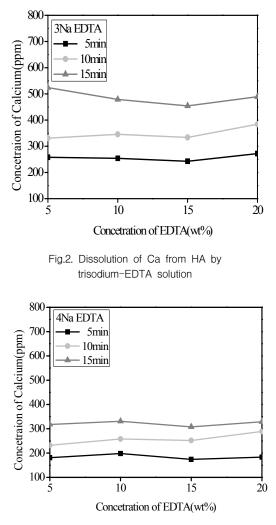
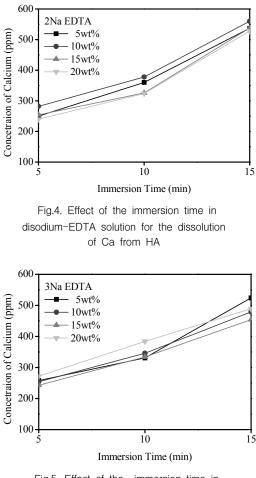


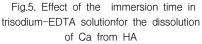
Fig.3. Dissolution of Ca from HA by tetrasodium-EDTA solution

# 3.2 The effect of immersion time of HA in EDTA solutions :

Fig.4, Fig.5 and Fig.6 showed the experimental data of the disodium-, trisodiumand tetrasodium-EDTA solutions. As can be expected, the amount of  $\mathrm{Ca}^{\scriptscriptstyle+}$  dissolved out from HA increased with longer immersed time of HA in EDTA solutions. However, the disodium- and trisodium EDTA solutions showed not so significant difference in ability to dissolve out Ca<sup>+</sup> from HA, but tetrasodium-EDTA solution showed the least ability. With increase of immersion time of HA in 5wt% disodium-EDTA

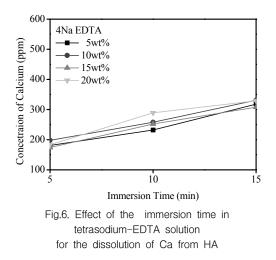
solution from 5min to 15min, the amount of Ca<sup>+</sup> dissolved out from HA increased from 250ppm to 537ppm; in 5wt% trisodium-EDTA, increased from 258ppm to 524ppm which is almost similar to the case of disodium-EDTA solution; in 5wt% tetrasodium-EDTA, increased from 181ppm to 318ppm which is lowest value.





3.3 The average dissolution rate of Ca+ from HA in EDTA salt solutions

The average dissolution rates of Ca<sup>+</sup>(ppm/min) from HA in EDTA salt solutions for the first 5min, next 5min and another 5min were calculated based on the experimental data; in case of the disodium-EDTA solution, 50.0ppm/min for the first 5min, 22.0ppm/min for 산업기술연구(강원대학교 산업기술연구소 논문집), 제31권 A호, 2011. 송 근 호, 장 화 익, 이 광 래



the next 5min and 35.4ppm/min for another 5min. In case of the trisodium-EDTA solution, 51.6ppm/min for the first 5min, 14.6ppm/min for the next 5min and 38.6ppm/min for another 5min. In case of the tetrasodium-EDTA solution, 36.2ppm/min for the first 5min, 10.2ppm/min for the next 5min and 17.2ppm/min for another 5min. The dissolution rates of Ca<sup>+</sup>(ppm/min) from HA in disodium- and trisodium-EDTA were very similar to each other, but in tetrasodium-EDTA it was much less than the others; for the initial 5 minutes, the dissolution rate of Ca<sup>+</sup>(ppm/min) in 5wt% disodium- and trisodium-EDTA solution were 50.0ppm/min and However, 51.6ppm/min, respectively. the dissolution rate of  $Ca^+$ in 5wt% tetrasodium-EDTA solution was only 36.2ppm/min which is about 70% of the rate in disodium- and trisodium-EDTA.

#### 4. Conclusion

EDTA salt(disodium-, trisodium- and tetrasodium-EDTA) aqueous solutions proved effective in the dissolution of Ca from HA, which meant the solutions were useful for removing the smear layer for the root canal dentin treatment. Among the sodium salt solutions of EDTA, trisodium-EDTA solution was most applicable to root canal treatment.

#### References

- Gulabivla K., B. Patel, G. Evans and Y.-L. -NG, "Effects of mechanical and chemical procedures on root canal surfaces", *Endodontic Topics*, 10, 103–122, 2005.
- [2] De-Deus G. and S. Paciornik, and M.H. Mauricio, "Evaluation of the effect of EDTA, EDTAC and citric acid on the microhardness of root canal", *International Endodontic J.*, 39, 401–407, 2006.
- [3] Lafferty TA, Gher ME, Gray JL. "Comparative SEM study on the effect of acid etching with tetracycline HCl or citric acid on instrumented periodontally-involved human root surfaces", J. Periodontol, 64, 689–93, 1993.
- [4] Bergenholtz A, Babay N, "Scanning electron microscopy of the root surface texture of extracted periodontally diseased teeth following various etching and chelating regimens", *Int. J. Periodontics Restorative Dent*, 18, 171–9, 1998.
- [5] Trombelli L, Scabbia A, Zangari F, Griselli A, Wikesjo UME, Calura G. "Effect of tetracycline on periodontally-affected human root surfaces", *J. Periodontol*, 66, 685–91, 1995.
- [6] Parmar G. and A. Chhatariya, "Demineralising effect of EDTA at different concentration and pH-A spectrophotometer study", *Endodontology*, 16, 54–57, 2004.
- [7] Blomlof J., L. Blomlof and S. Lindskog, "Effect of different concentrations of EDTA on smear removal and collagen exposure in periodontitis-affected root surfaces", *J. Clin. Periodontol*, 24, 534-537, 1997.
- [8] Blomlöf L, Bergman E, Forsgardh A, Foss L, Larsson A, Sjoberg B. "A clinical study of root surface conditioning with an EDTA gel. I. Non surgical periodontal treatment", *Int. J. Periodontics Restorative Dent*, 20, 561–5, 2000.
- [9] Prabhu, S.G., N. Rahim, K.S. Bhat and J. Mathew, "Comparison of removal of endodontic smear layer using NaOCl, EDTA, and different concentrations of maleic acid-A SEM study", *Endodontology*, 15, 20–25, 2003.