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Original Article

Biomineralization reaction from nanosized calcium silicate: A new method for reducing dentin hypersensitivity

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Hydroxyapatite

Abstract *Background/purpose:* This study assessed the ability of experimental materials consisting of dicalcium silicate (DCS) and tricalcium silicate (TCS) with nanosized particles to form intratubular crystals under phosphate-buffered saline (PBS) and the effect on dentin permeability reduction.
Materials and methods: By isolating the cervical part of the extracted premolars, 195 specimens were obtained. Two experimental materials (DCS/TCS and TCS) were applied to the dentin surface by brushing and stored in PBS (n = 65). Another 65 specimens were not treated. Each group was randomly divided into five subgroups based on the PBS immersion period (1, 15, 30, 60, and 90 days, n = 10). The dentin permeability was measured, and the hydraulic conductance, Lp (%), was calculated. After acid challenge with 1 M acetic acid, Lp (%) was re-measured. Data were analyzed using two-way analysis of variance and Fisher's least significant difference test ($\alpha = 0.05$). Three specimens of each subgroup were longitudinally sectioned

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and examined using scanning electron microscopy and a field emission-electron probe micro analyzer.
Results: The Lp (%) of the experimental groups gradually decreased over time ($P < 0.05$). The hydroxyapatite-like crystals that grew were observed and found to have a Ca/P ratio similar to that of hydroxyapatite. The crystals remained after the acid challenge, and the Lp (%) was not significantly different from that before acid treatment.
Conclusion: Intratubular crystals formed from the experimental materials consisted of DCS and TCS and were resistant to acid. These crystals significantly reduced dentin permeability.

Introduction

Dentin hypersensitivity (DH) results from exposed dentin, mainly resulting from gingival recession or loss of dental structure.[1,2] According to the most widely accepted hydrodynamic theory among the mechanisms for explaining DH,[3] the dentinal tubules can serve as a channel for stimulation transmission by moving fluids within the tubules.[4,5] Thus, the occlusion of exposed dentinal tubules is a crucial strategy for preventing invasion of irritants, reducing the permeability of dentinal tubules.[6] Many desensitizers attempt to treat DH by blocking the exposed dentinal tubules.[7,8]

However, most desensitizing agents on the market do not have long enough life spans. The limitations of the available desensitizers are related to the lack of intratubular occlusion due to the large particle size and high solubility of the occluding materials and the short duration time due to poor resistance to acid attacks.[9] To effectively manage hypersensitivity of the dentine, new materials are required that can penetrate deep enough into dentinal tubules, last a long time, and not easily wash out at low-pH conditions.[10]

Occluding dentinal tubules with a material that has a composition similar to that of the material constituting dentin is a biomimetic method that satisfies the conditions of the ideal desensitizer. Dentin is a hydroxyapatite with a variety of compositions. However, in a previous study,[1] a method of dentinal tubule occlusion that delivered nano-sized hydroxyapatite did not show very good performance as compared with existing materials.

Biomaterials containing silicone, such as calcium silicate, commonly form hydroxyapatite-like precipitates when coming into contact with physiological fluids containing phosphate.[11,12] In a previous study,[13] researchers observed intratubular crystal formation in a phosphate-buffered saline (PBS) environment. Intratubular crystals tend to fill the dentinal tubules densely as the crystals grow, unlike other materials that are less effective over time. To assess the usability of the desensitizer, it is necessary to evaluate whether such growing intratubular crystals can reduce fluid through the dentinal tubules.

The aim of this study was to assess the ability of two types of experimental materials consisting of dicalcium silicate (DCS) and tricalcium silicate (TCS) with nanosized particles to form intratubular crystals under PBS and to assess their effect on the reduction of dentin permeability.

Materials and methods

Specimen preparation

This study was approved by the institutional review board (IRB) of a dental hospital (No. S-D20190010). In total, 195 human premolars extracted for orthodontic treatment with intact coronal and root surfaces were prepared and stored in 0.1% thymol (T0501; Sigma—Aldrich, St. Louis, MO, USA) solution for no longer than 3 months prior to use.

Using a low-speed diamond saw (Isomet™, Buehler, Lake Bluff, IL, USA) under water cooling, 8 mm height specimens centered on the cementoenamel junction were fabricated and fresh dentin was exposed on the top without perforation at the pulp horn area. The sectioned tooth was then mounted in a ring-shaped acrylic mold using self-cured blue resin (Keystone Industries GmbH, Singen, Germany; Fig. 1).

The exposed upper dentin surface of each specimen was treated with 17% ethylenediaminetetraacetic acid (MD Cleanser; Meta Biomed, Chungju, Korea) for 1 min. To open the dentinal tubules and remove the smear layer, 2.5 mL of 5.25% sodium hypochlorite (Samchun Chemical, Seoul, Korea) was subsequently applied to the exposed dentinal surface. The specimen was then rinsed twice with 10 mL of distilled water.

Experimental material preparation

The experimental material consisting of DCS/TCS was prepared with the sol-gel method[14,15] using calcium carbonate [$CaCO_3$] (239216; Sigma—Aldrich), silicon dioxide [SiO_2] (342890; Sigma—Aldrich), and aluminum oxide [Al_2O_3] (414069; Sigma—Aldrich) as the raw materials, while nitric acid [HNO_3] (438073; Sigma—Aldrich) served as a solvent. The obtained material was calcined at 1450 °C for 6 h, and the resultant powders were ground at 300 rpm using a disk mill (KM Tech, Icheon, Republic of Korea) and then at 200 rpm using a ball mill (BML-2, Daihan Scientific Group, Wonju, Republic of Korea) for 48 h.

The second experimental material consisted of single-phase and high-purity TCS. Calcium carbonate [$CaCO_3$]

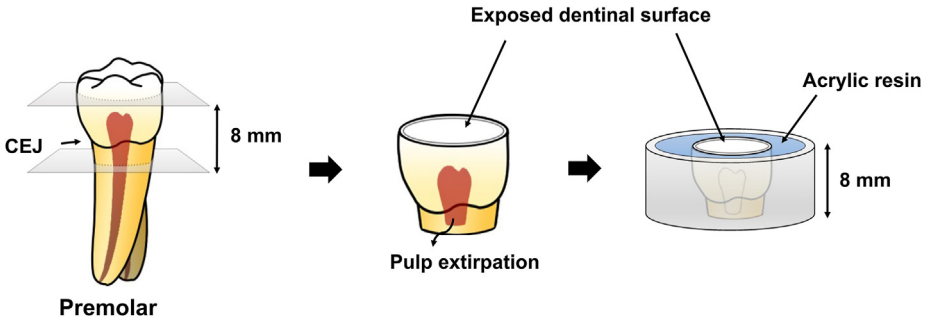

Figure 1 Schematic illustrating the preparation of the specimens. The occlusal and radicular portions of the teeth were removed using a low-speed diamond saw, and the fresh dentin surface was exposed without pulp horn involvement. A total of 8 mm of the cervical part centered at the cementoenamel junction was isolated. The remaining pulp tissue was removed carefully with small forceps without touching the inner part of the pulpal space. The sectioned tooth was then mounted in a ring-shaped acrylic mold with self-curing blue resin.

(239216; Sigma—Aldrich) and silicon dioxide [SiO_2] (342890; Sigma—Aldrich) were mixed completely using a mixer at a molar ratio of approximately 3:1. The mixed CaO—SiO_2-based compound was compressed into pellet form to use the high-temperature solid-state method.[16] The compressed CaO—SiO_2-based compound pellets were placed in an alumina crucible for solid chemical reaction synthesis and calcined in an electric furnace. During calcination, the temperature was fired at 1350 °C for 18 h and then cooled. The material was cooled and then completely ground again. The process of pelting and reheating was repeated several times to complete a single-phase high-purity TCS.

Table 1 shows the main components and contents of the experimental materials. The particle size distribution of the powders was measured using laser diffraction (Mastersizer S; Malvern Panalytical, Malvern, UK).

Application of experimental materials

After the specimens and experimental materials were prepared, 0.5 g of experimental materials was mixed with 5 mL of distilled water and applied to the exposed dentin surface via a toothbrushing motion according to ISO 11609 (n = 65). A total of 10,000 repeated strokes (1 stroke/s) were applied under a 150-g load with continuous contact with the test material and dentin surface. Following this, any excess experimental material remaining on the dentin surface was removed by washed it with distilled water.

The groups were randomly divided into five subgroups based on the PBS (D8662, Sigma—Aldrich) immersion period of 1, 15, 30, 60, and 90 days at 37 °C (n = 13). The PBS solution was replaced every 7 days. In this study, the PBS solution consisted of the following (in g/L): $CaCl_2 \cdot 2H_2O$, 0.133; $MgCl_2 \cdot 6H_2O$, 0.1; KCl, 0.2; KH_2PO_4, 0.2; NaCl, 8.0; and Na_2HPO_4 (anhydrous), 1.15. The pH of the solution was 7.2.

Table 1 Composition of the experimental materials.

Group	Component	Content (wt%)
DCS/TCS	Dicalcium silicate [$2CaO.SiO_2$]	10—15
	Tricalcium silicate [$3CaO.SiO_2$]	70—80
	Others	5
TCS	Tricalcium silicate [$3CaO.SiO_2$]	100

Abbreviations: Lp, hydraulic conductance; DCS, dicalcium silicate; TCS, tricalcium silicate.

Dentin permeability measurement

All surfaces, except for the upper surfaces of the 50 specimens in each group, were varnished twice with nail varnish to achieve a fluid-tight seal. The flow of fluid through the dentinal tubules was measured using a fluid flow measurement device (Fig. 2). Each specimen was connected to a water bath at a pressure of 20 cm H_2O using a microcapillary tube. The linear displacement of the air bubbles in capillary tube filled with water was detected by an infrared-emitting diode. A computer recorded the movement of the air bubble four times per second by and converted it into volume displacement (hydraulic

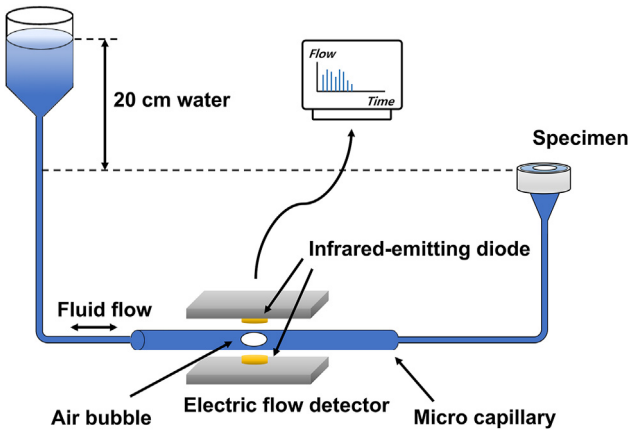

Figure 2 Schematic illustrating the measurement of hydraulic conductance. Evaporation through the exposed dentinal tubules caused movement of an air bubble, which was traced by an infrared-emitting diode. The flow by time was recorded on a computer.

conductance, Lp), in accordance with the dentinal permeability.

After initial dentin permeability was measured, experimental materials were applied to the dentinal surface of each specimen as described above. Specimens without experimental material application served as controls. Each group was divided into five subgroups based on the storage period (1, 15, 30, 60, and 90 days) in PBS. Dentin permeability was measured again after each specified period.

After plotting Lp over time, the slope (Lp/s) was calculated by selecting an interval with a straight line of more than 10 min and $R > 0.998$. The results were expressed as percentages of the initial Lp/s, Lp (%).

Scanning electron microscope analysis

All specimens were mounted on aluminum stubs, and the examined surfaces were coated with a 30-nm layer of gold. The occlusal surface and longitudinally sectioned surface of each specimen were examined via field emission scanning electron microscope (FE-SEM; Apreo S; Thermo Fisher Scientific, Waltham, MA, USA; n = 3 at five time points).

Field emission-electron probe microanalyzer examination

The longitudinal split surfaces of the specimens were analyzed using an FE–electron probe microanalyzer (FE-EPMA; JXA-8530F, JEOL, Tokyo, Japan) with area mapping for the composition of the chemical elements Ca, P, and O. The X-ray profiles and element quantification were performed at 15 kV and 10 nA (probe current).

To quantify the components of intratubular crystals, the crystals in the dentinal tubules were focused with a 2-mm probe, and the scattered X-ray wavelength was analyzed using wavelength dispersive spectrometry. Eight different analyzing crystals (LIF, LIFH, PETJ, PETH, TAP, TAPH, LDE1, LDE2H) were used.

Acid challenge

To simulate the acid challenge induced by the daily diet, 1 M acetic acid was applied on the treated dentinal surface for 3 min to the specimens stored for 3 months. After rinsing twice with distilled water, dentin permeability was measured again. The occlusal surfaces of specimens of the three groups were examined using SEM.

Statistical analysis

The means and standard deviations of dentin permeability were calculated, and the results were expressed as percentages of the pretreatment measurements. Data were analyzed using two-way analysis of variance and Fisher's least significant difference test using IBM SPSS 23.0 (SPSS Inc., Chicago, IL, USA). The level of significance was set at $\alpha = 0.05$.

Results

Particle size distribution of the experimental materials

Fig. 3 shows the particle size distribution of the experimental materials. The expressions D (0.1), D (0.5), and D (0.9) represent specific percentile points in the particle size distribution. The particle size of the DCS/TCS was distributed between 0.052 μm (D0.1) – 1.267 μm (D0.9), and the median value (D0.5) is 0.184 μm. The particle size of the TCS was distributed between 0.737 μm (D0.1) – 3.415 μm (D0.9), and the median value (D0.5) is 2.012 μm.

Change in hydraulic conductance

Table 2 presents the means and standard deviations of permeability for the groups. There were significant differences among the groups ($P < 0.001$), immersion periods ($P < 0.001$) and in the interactions between factors ($P < 0.008$). The Lp (%) of the two experimental groups was lower than that of the control group throughout the experimental period ($P < 0.05$). After 60 days, the Lp (%) of the DCS/TCS group was lower than that of the TCS group ($P < 0.05$).

After the acid challenge, there was a slight recovery in Lp (%) compared to the Lp (%) at 90 days in all three groups, but this difference was not significant ($P > 0.05$). However,

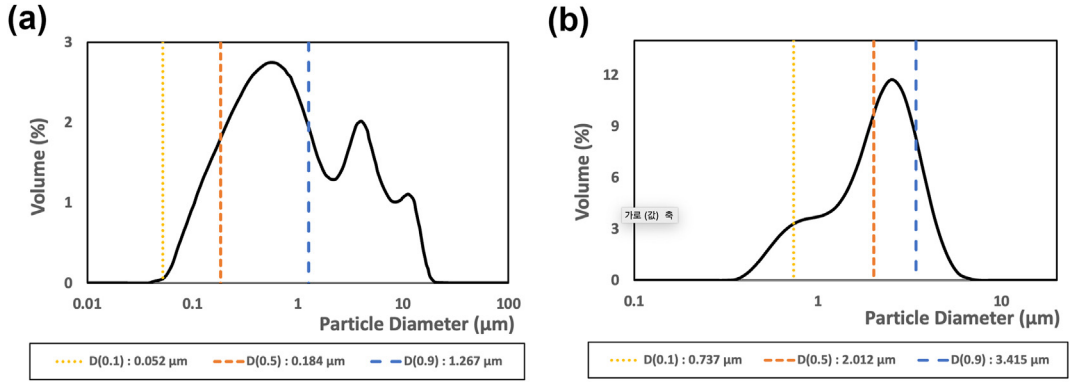

Figure 3 Particle size distribution of the experimental materials. (a) DCS/TCS, (b) TCS. D (0.1), D (0.5), and D (0.9) represent specific percentile points in the particle size distribution.

Table 2 Hydraulic conductance, Lp (%), after desensitizer treatment and immersion in phosphate-buffered saline.

Time	Lp (%)		
	Control	DCS/TCS	TCS
Pretreatment	100 ^{Aa}	100 ^{Aa}	100 ^{Aa}
1 day	99.6 ± 1.8 ^{Aab}	64.8 ± 2.0 ^{Bb}	64.1 ± 3.3 ^{Bb}
14 days	94.1 ± 2.4 ^{Aabc}	64.4 ± 2.9 ^{Bb}	64.5 ± 2.4 ^{Bb}
30 days	93.0 ± 2.5 ^{Aac}	56.6 ± 2.8 ^{Bbc}	59.7 ± 2.9 ^{Bbc}
60 days	90.9 ± 2.0 ^{Abc}	45.5 ± 3.3 ^{Bcd}	54.3 ± 3.1 ^{Cbc}
90 days	87.0 ± 2.7 ^{Ac}	35.9 ± 2.9 ^{Bd}	49.1 ± 2.9 ^{Cc}
After acid challenge	92.9 ± 2.5 ^{Aabc}	47.1 ± 4.1 ^{Bcd}	56.5 ± 3.4 ^{Bbc}

Values are presented as mean ± standard deviation. Significant differences are represented by different uppercase letters within the same time and different lowercase letters within the same group (two-way analysis of variance and Fisher's least significant difference test; $P < 0.05$).
Abbreviations: Lp, hydraulic conductance; DCS, dicalcium silicate; TCS, tricalcium silicate.

Lp (%) in both experimental groups was significantly lower ($P < 0.05$) compared to the pretreatment value, whereas Lp (%) in the control group was significantly increased after the acid challenge, showing no significant difference from the pretreatment value ($P > 0.05$).

Crystal formation on the occlusal surface

Fig. 4 shows the SEM images of the specimens immersed for 90 days in PBS after the application of the experimental materials. There was no smear layer or crystal formation over the exposed dentin surface, and all dentinal tubules remained open in the control group. In the experimental groups, petal-shaped crystals were formed on the exposed dentin surface, and plug-shaped precipitates below the dentinal tubule orifice and intratubular crystals were formed.

After 1 M acetic acid was applied for 3 min, the dentin surface was clean, and the dentinal tubules were empty in control group. In the two experimental groups, the crystals on the occlusal surface were removed after acid treatment; however, the occluding materials in the dentinal tubules were retained.

Intratubular crystal formation

Fig. 5 presents the SEM images of the dentinal tubules within a range of 20–100 μm from the dentinal surface of longitudinally sectioned specimens of the experimental groups. Both experimental groups showed that crystals inside the dentinal tubules became larger over time, filling the dentinal tubules more densely.

Analysis of crystals by electron probe microanalysis

Fig. 6a and b shows the representative EPMA mapping for Ca and P in the experimental group specimens. The wavelength dispersive spectra of the intratubular crystals are also exhibited. Ca, P, and O were confirmed to exist at a similar level to the dentin in the crystals formed in the dentinal tubules. Based on the results of quantifying the content of each element (Fig. 6c and d) by specifying an area of 2 μm (white circles in Fig. 6a and b), the Ca/P ratio of the intratubular crystals of the DCS/TCS and TCS groups was 1.70, which was similar to the Ca/P ratio of hydroxyapatite.

Discussion

Based on hydrodynamic theory, blocking the movement of fluid through the exposed dentinal tubules can reduce discomfort caused by DH. In this study, dentin permeability change by biomineralization reaction from experimental materials in dentinal tubules was evaluated.

After immersion in PBS for 1 day, the Lp (%) was lower than that before the application of the experimental materials ($P < 0.05$). The rapid decrease in Lp (%) can be described as an occluding plug observed directly below the dentinal tubule orifice in the SEM images (Fig. 4b). In the process of applying the experimental materials to the brushing motion, the occluded plugs penetrated the dentinal tubules, and this action effectively reduced dentin permeability after the initial setting.

After DCS and TCS come in contact with H_2O, the solution is quickly supersaturated with calcium hydrosilicate as described below.[17,18]

$$2(2CaO\cdot SiO_2)+4H_2O \rightarrow 3CaO\cdot 2SiO_2\cdot 3H_2O+Ca(OH)_2 \quad (1)$$

$$2(3CaO\cdot SiO_2)+6H_2O \rightarrow 3CaO\cdot 2SiO_2\cdot 3H_2O+3Ca(OH)_2 \quad (2)$$

As a result of these reactions, calcium and hydroxyl ions are released, resulting in a highly alkaline environment.

Therefore, the occluding plug that formed below the dentinal tubule orifice can act as a reservoir of calcium ions, allowing for the presence of excess calcium ions inside the dentinal tubules through continuous release of calcium.

The supersaturation condition was expected to cause local aggregation of calcium and phosphate ions, which caused the growth of intratubular crystals.[19] The growing crystals could block the dentinal tubules and continuously decrease dentin permeability (Table 2). This suggests that experimental materials have potential as relieving agents for DH, and the effect increases with time. These results are consistent with those of previous studies.[20–25]

The analysis of intratubular crystals using EPMA showed the Ca, P, and O existed at levels similar to those of dentin

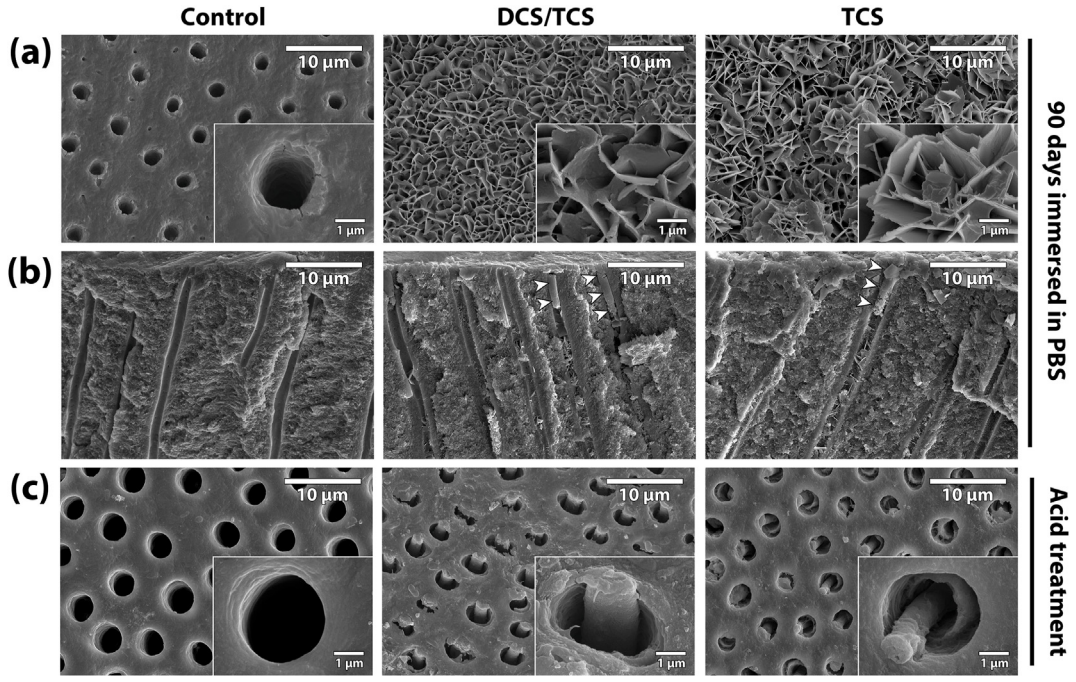

Figure 4 SEM images of the specimens immersed for 90 days in PBS. All images were taken at a magnification of 10,000×, and the enlarged images in the white square were taken at 50,000 × magnification. (a) No crystal formation was observed in the control group. In the experimental groups, petal-shaped crystals were formed on the occlusal surface. (b) In the longitudinal sectioned specimen, the plug-shape precipitate was inserted inside the dentinal tubules in the experimental groups (white arrowheads). (c) SEM images of the occlusal surface of specimens after application of 1 M acetic acid for 3 min. The occlusal surface was clear, and the dentinal tubules were empty in the control group. However, precipitate in the dentinal tubules was observed in the DCS/TCA and TCS groups.

and the calcium–phosphorus ratio (1.67) was similar to that of hydroxyapatite. This is consistent with studies that mentioned the formation of hydroxyapatite-like crystals in contact with PBS after the hydration reaction of calcium silicate.[20,25,26]

The Lp (%) of the TCS group on day 1 was slightly lower than the Lp (%) of the DCS/TCS group. This could be explained by the initial reaction of calcium silicate. TCS provides a rapid hydration reaction and setting than DCS does.[27] The total initial reaction rate was faster in the TCS group than in the DCS/TCS group, and the formation of the initial precipitate progressed rapidly. The results can be confirmed using SEM images. After 1 day, clear plate-shaped crystals were observed in the dentinal tubules of the TCS group, whereas small plate-shaped crystals were observed in the DCS/TCS group.

After 90 days, the Lp (%) was smaller in the DCS/TCS group than in the TCS group. This difference resulted from the particle size applied on the exposed dentin surface. DCS/TCS had smaller particle sizes compared to TCS (Table 1), allowing relatively more particles to penetrate deep dentinal tubules. The particles released more calcium ions, and more intratubular crystals were formed in the DCS/TCS group.

It was confirmed that the materials used in this experiment formed crystals through continuous chemical reactions without additional application, and the effect persisted. However, the effect of most desensitizers is rapidly reduced by saliva, food, and mechanical stimulation.[9] After acid challenge with 1 M acetic acid, Lp (%) was significantly increased in the control group compared to the value after 90 days, whereas only insignificant difference was observed in the Lp (%) of the two experimental groups after 90 days. This result can be explained through SEM analysis. Although the crystals on the exposed dentin surface were removed because they were loosely bound to the smear layer formed by the toothbrushing motion, the intratubular precipitates remained intact after acid treatment. The intratubular crystals were not easily dissolved or washed out for acid, and they had the potential to reduce DH even under acid conditions.

In the present experimental design, the experimental materials were applied to the extracted teeth as a brushing method. Because vital teeth experience outward hydrostatic pressure, it is crucial to reproduce this condition to accurately assess the desensitizing effect of the experimental material. Hence, to address these limitations and provide additional insights, further research is required.

Within the limitations of this *in vitro* study, the DCS/TCS mixture and TCS effectively reduced the permeability of dentin and occluded opened dentinal tubules under PBS. The crystals observed on the exposed dentin surface and in the dentinal tubules had a Ca/P ratio similar to that of

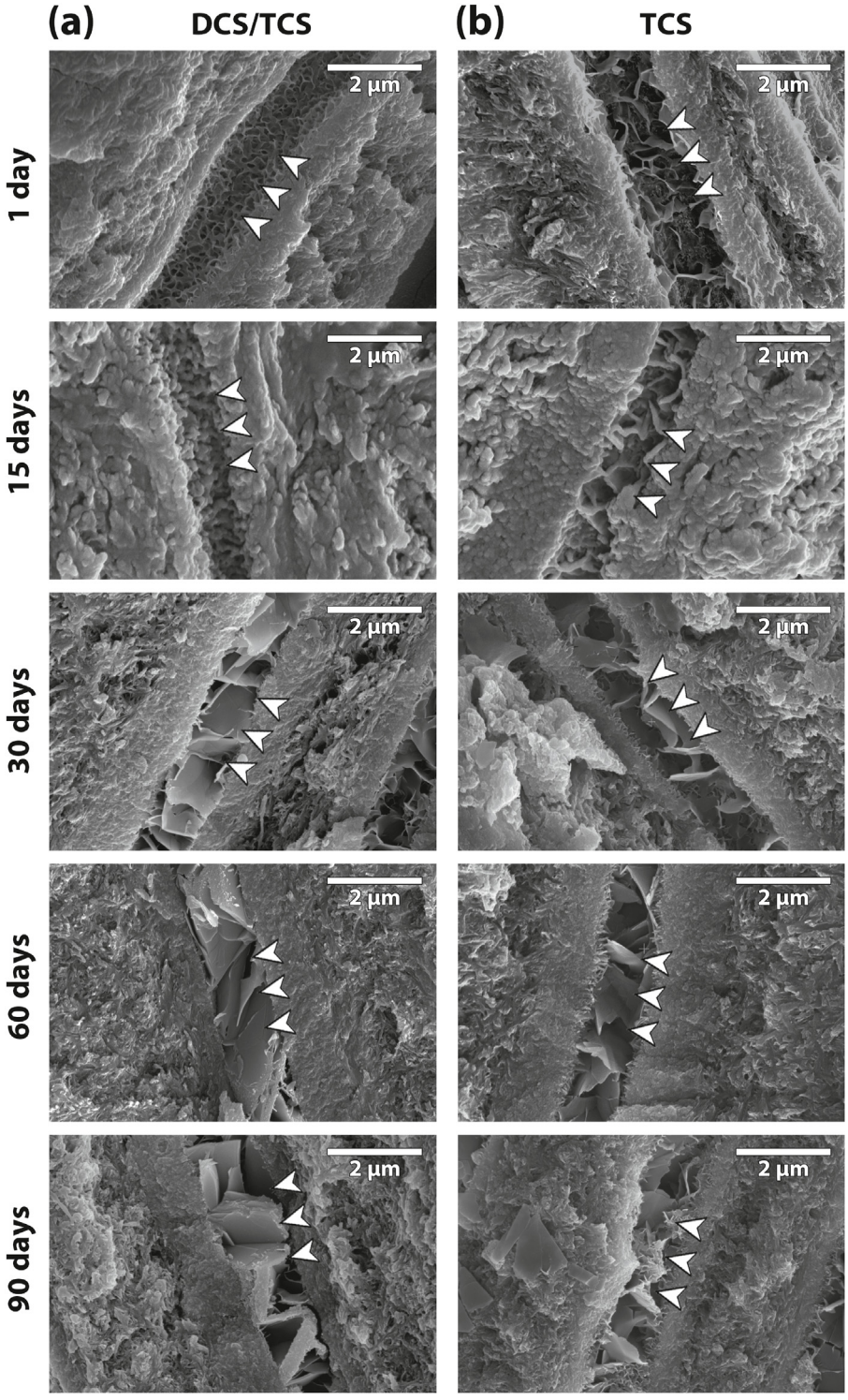

Figure 5 SEM images of the dentinal tubules of the two experimental groups over time. (a) DCS/TCS group and (b) TCS group at 1, 15, 30, 60, and 90 days after immersion in PBS, respectively. Crystal formation was observed in the dentinal tubules, forming a denser crystal complex as the period of storage in PBS increased (white arrowheads).

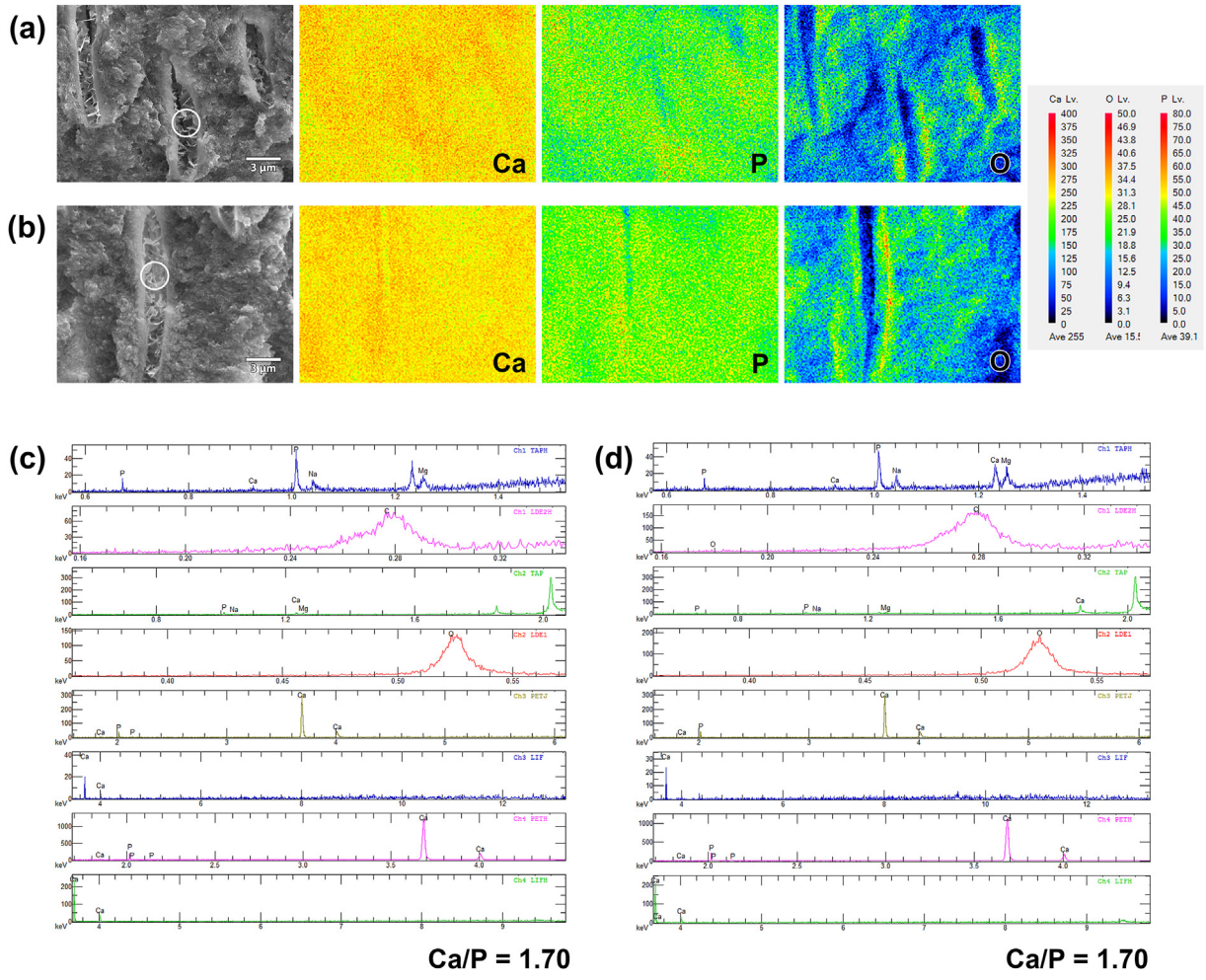

Figure 6 Three EPMA chemical element mappings for Ca, P, and O of the specimens. (a, b) Due to the structure of the dentinal tubules, fewer components were detected than that of dentin; however, it can be seen that substances with Ca, P, and O were formed in the dentinal tubules in both experimental groups. (c, d) Scattered X-ray wavelength analysis by wavelength dispersive spectrometers of intratubular crystals (white circles in a and b) showing a Ca/P ratio of 1.70 for the DCS/TCS group and TCS group. Abbreviations: LIF, lithium fluoride; PET, pentaerythritol; TAP, thallium acid phthalate; LDE, artificial layered dispersive element, LDE, layered dispersion element, H designated for H-type spectrometer, J designated for high reflectivity crystal.

hydroxyapatite. The effect of the DCS/TCS mixture and TCS on reducing discomfort due to DH has resistance potential for acid challenge.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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