

Influence of various dentin desensitizers on the effect of tooth whitening

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We evaluated the influence of the combined use of dentin desensitizer and bleaching agent on tooth whitening. Bovine teeth were immersed in a pigmentation liquid to discolor the specimens. The shade was measured before and after bleaching using TiON in Office after each treatment (control group). MS coat F, Nanoseal, and Teethmate Desensitizer were applied (after each treatment for the bleaching group). After bleaching treatments for the controls, the specimens were immersed in a staining solution. that was applied at the time of shade measurement every 7 days (after bleaching group). The measurements obtained were statistically tested using one-way layout analysis of variance and Tukey's test ($p < 0.05$). Regarding ΔE between before and after bleaching treatment (4 t measurements), the combined use of each hypersensitivity inhibitor showed no significant difference compared, with the controls. Regarding ΔE between completion of bleaching treatment and 4 weeks later, although there was no significant difference between the combined use of Nanoseal or Teeth Mate Desensitizer and the controls, the use of MS Coat F presented a significant difference with the controls. Because the action of dentin desensitizer on the hydroxyapatite did not influence the bleaching, we recommend its combined use during bleaching. (J Osaka Dent Univ 2015 ; 49(1) : 49–60)

Key words : Bleaching ; Desensitizer ; Hypersensitivity

INTRODUCTION

Since the FDI proposed minimal intervention in 2000,¹ whitening methods without drilling teeth have been actively and widely investigated, and many new bleaching agents have been developed. There are two methods of vital tooth-bleaching : home bleaching performed by patients using a custom tray, and office bleaching performed by dentists at clinics. Office bleaching has the advantage of obtaining the desired effect in a short time compared with home bleaching, and being applied under the control of a dentist. However, agents used for office bleaching contain a high concentration of hydrogen peroxide as the main ingredient, for which there are many points to be considered, such as the influence on tooth quality^{2–8} and protection of the soft tissue.^{9–11} In addition, hypersensitivity frequently develops.^{12–14} The incidence of dentin hypersensitivity developing during and immediately after

bleaching has been reported to be 55–75% when mild cases are included,¹⁵ and with involvement of enamel lamellae, there is a possibility of micro cracks developing in the enamel.

Desensitizers acting on hydroxyapatite are commercially available and expected to be effective for enamel, which contains a higher percentage of hydroxyapatite than dentin. In this study, we investigated the effect on tooth whitening of the concomitant use of dentin desensitizers and bleaching agents.

MATERIALS AND METHODS

Sample preparation

Stored frozen bovine teeth were thawed and the roots removed. After extirpation of the dental pulp, the pulp cavity was treated with 5% hypochlorous acid solution for one minute, washed with water, dried, and treated with 37% phosphate gel (K-echant, Kurary Noritake, Tokyo, Japan) for one minute. They were then wash-

ed with water and dried for use as test teeth. The samples were immersed in a tea-infused solution as a stainer for 7 days to prepare discolored samples. The color of the labial central region was measured using a dental colorimeter, Shade Eye (Shofu, Kyoto, Japan), and the CIE L* a* b* was determined before treatment.

Comparison of whitening effect

The bleaching and desensitizer application procedures are shown in Fig. 1. After pretreatment measurement, tooth bleaching was performed using TiON in Office (GC, Tokyo, Japan) following the method specified in the package insert of the product. TiON reactor was applied and the sample was left for 10 seconds. After drying, a mixed gel of syringes A and B was applied, followed by exposure for 10 minutes using an LED irradiator for bleaching (Cosmo Blue ; GC).

This bleaching procedure was applied to the same region three times, and the color was measured (first bleaching). In the control group, teeth were stored in saline thereafter and subjected to bleaching three more times followed by color measurement every 7 days (2nd–4th bleedings). The tooth was then im-

mersed in the staining solution again, and the color was measured every 7 days (1st–4th re-discoloration).

As with the treatment application groups, the teeth were subjected to bleaching using TiON four times, the same as with the controls, and the respective dentin desensitizers were applied immediately after each treatment (during bleaching group). As with the after treatment application groups, the teeth were subjected to bleaching four times, similar to the control group. After the 4th bleaching, dentin desensitizers were applied after the 1st–3rd re-discoloration every 7 days (after bleaching group).

Dentin desensitizers

The dentin desensitizers used in the experiment are shown in Table 1. Three dentin desensitizers were used : MS Coat F (MS) (Sun Medical, Moriyama, Japan), Nanoseal (NS) (Nippon Shika Yakuhin, Shimonoeki, Japan), and Teethmate Desensitizer (TD) (Kurary Noritake, Tokyo, Japan).

Color difference ΔE^*ab

The tooth color was measured before treatment, after each of the four bleedings, and after each of the four

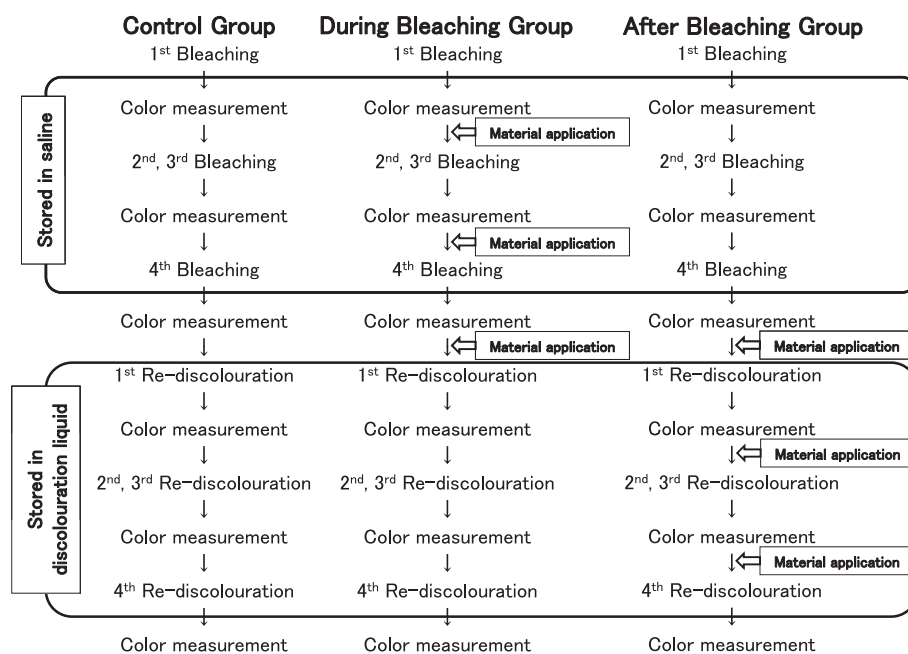


Fig. 1 Experimental procedures.

Table 1 Desensitizers

Material	Manufacturer	Lot. No.	Composition	Code
MS Coat F	Sun Medical, Moriyama, Japan	GX1/10-2016	Polymethyl-methacrylate, Polystyrene sulphonic acid copolymer, Oxalic acid, fluoride, water	MS
NanoSeal	Nippon Shika Yakuhin, Shimonoseki, Japan	C57/C59-2016	(A) F-Ca-Al-Si glass in aqueous dispersion (B) H ₃ PO ₄ aqueous solution	NS
Teethmate Desensitizer	Kuraray Noritake Dental, Tokyo, Japan	11123/10-2015	Powder : Tetra-calcium phosphate, Dicalcium phosphate anhydrous Liquid : Water, preservatives	TD

re-discolourations. The color differences, ΔE^{*ab} , between the pretreatment value and value after the 4th bleaching and between the value after the 4th bleaching and 4th re-discolouration, were determined as the tooth-bleaching effect. The color difference was calculated using the following formula : $\Delta E^{*ab} = [(\Delta L^{*})^2 + (\Delta a^{*})^2 + (\Delta b^{*})^2]^{1/2}$.

Statistical analysis

The measurement results were analyzed using one-way layout analysis of variance and Tukey's test ($n = 5$, $p < 0.01$).

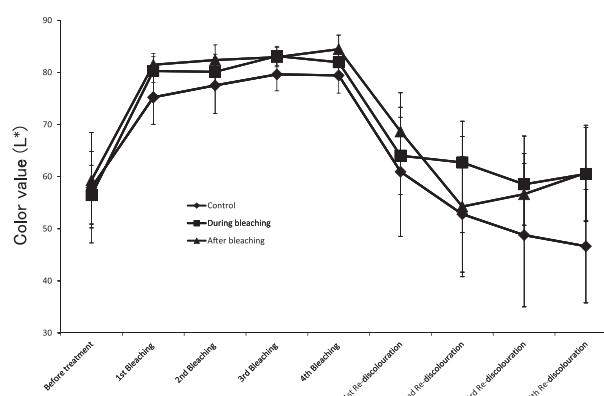
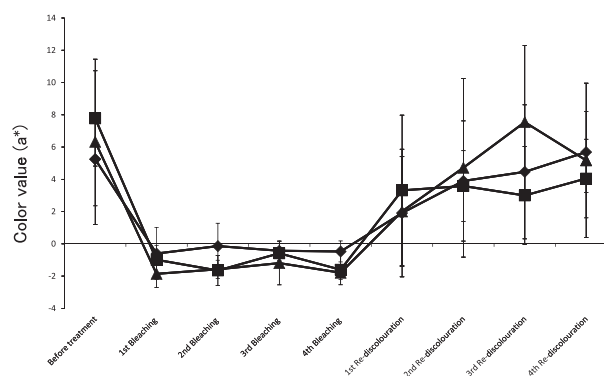
SEM observation of the dentin surface

Gold evaporation was performed on the teeth by conventional methods, and the specimens were observed using a scanning electron microscope (JSM-5610 LV ; JEOL, Tokyo, Japan).

RESULTS

Influence of various dentin desensitizers on color change ($L^*a^*b^*$)

The results of $L^*a^*b^*$ are shown in Figs. 2–10, and the color difference values are shown in Figs. 11 and 12. Regarding the influence of MS on L^* , the value in the during treatment application group was similar to that in the controls throughout the four bleaching periods. After completion of bleaching treatment, L^* tended to be higher in the during- and after bleaching group than in the controls throughout the four re-discolouration periods. Regarding the influence of MS on a^* , the value in the during treatment application group was similar to that in the controls group throughout the

**Fig. 2** Changes in the color value (L^*) (MS).**Fig. 3** Changes in the color value (a^*) (MS).

four bleaching periods. After completion of the bleaching treatment, the values in the during and after bleaching groups were similar those in the controls throughout the four re-discolouration periods. Regarding the influence of MS on b^* , the value in the during treatment application group was similar to that in the controls throughout the four bleaching periods. After completion of the bleaching treatment, the values

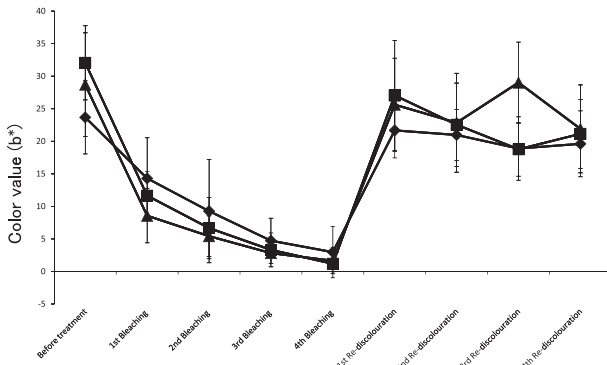


Fig. 4 Changes in the color value (b*) (MS).

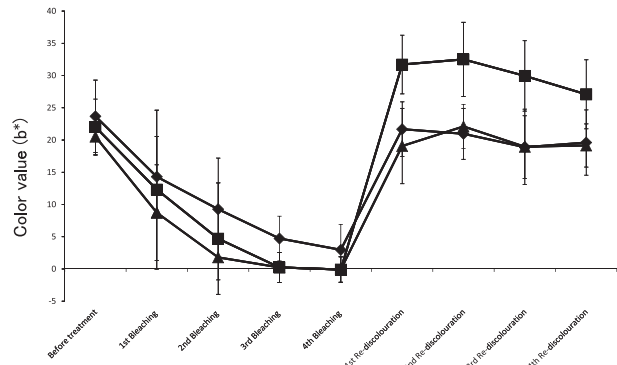


Fig. 7 Changes in the color value (b*) (NS).

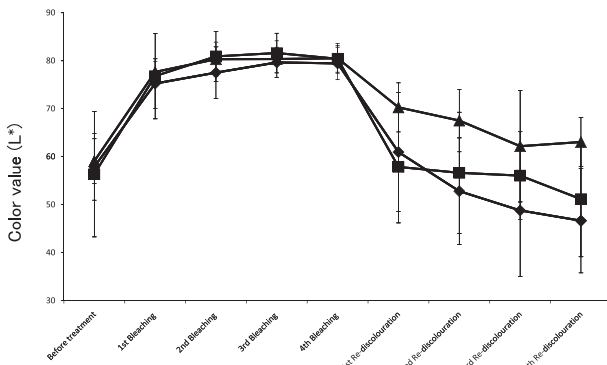


Fig. 5 Changes in the color value (L*) (NS).

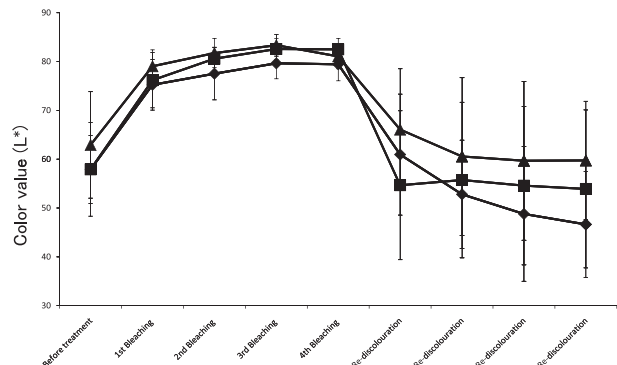


Fig. 8 Changes in the color value (L*) (TD).

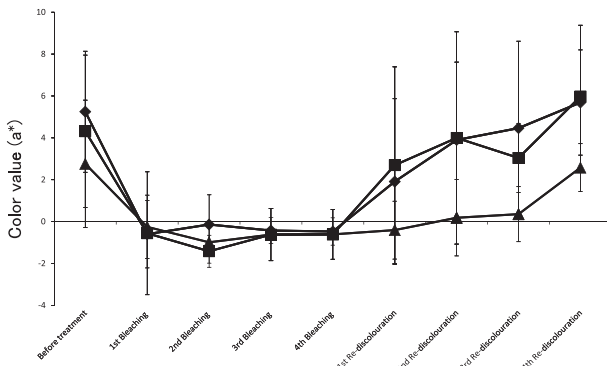


Fig. 6 Changes in the color value (a*) (NS).

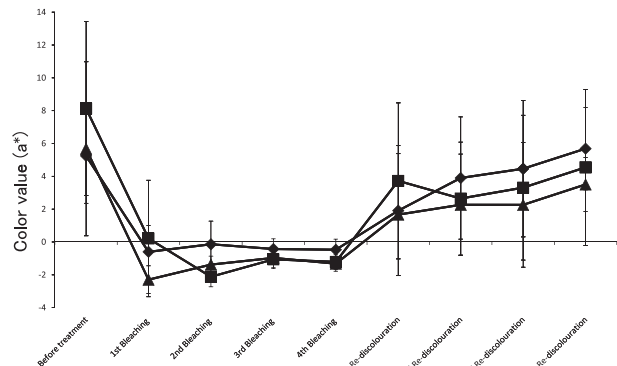


Fig. 9 Changes in the color value (a*) (TD).

in the during and after bleaching groups were similar to those in the controls throughout the four re-discolouration periods.

Regarding the influence of NS on L*, the value in the during treatment application group was similar to that in the controls throughout the four bleaching periods. After completion of the bleaching treatment, the

value tended to be higher in the after treatment application group than in the controls throughout the four re-discolouration periods. Regarding the influence of NS on a*, the value in the during treatment application group was similar to that in the controls throughout the four bleaching periods. After completion of the bleaching treatment, the value tended to be lower in the after

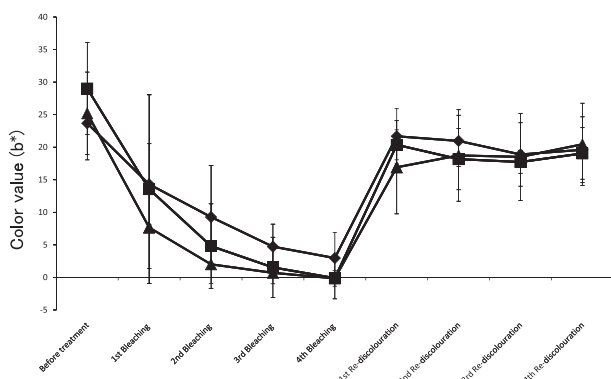


Fig. 10 Changes in the color value (b^*) (TD).

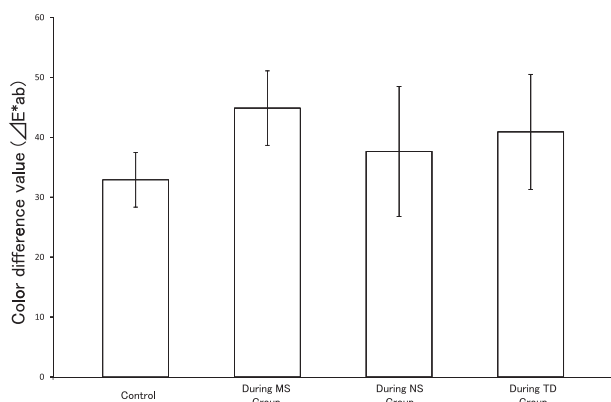


Fig. 11 Changes in the color difference value (ΔE^*ab) before and after bleaching.

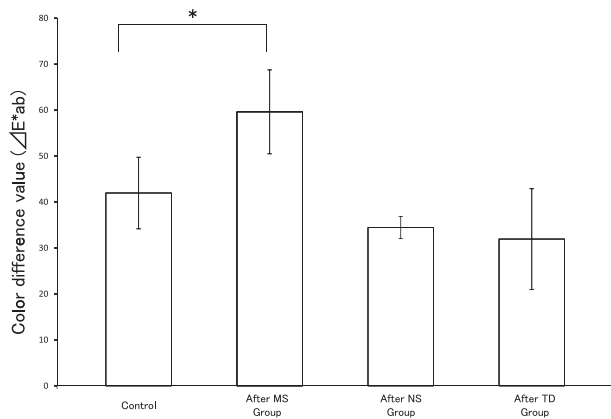


Fig. 12 Changes in the color difference value (ΔE^*ab) after treatment and 4th re-discolouration (* $p < 0.05$).

treatment application group than in the controls throughout the four re-discolouration periods. Regarding the influence of NS on b^* , the value in the during treatment application group was similar to that in the con-

trols throughout the four bleaching periods. After the completion of bleaching treatment, the value tended to be higher in the during treatment application group than in the controls, and the value in the after treatment application group was similar to that in the controls throughout the four re-discolouration periods.

Regarding the influence of TD on L^* , the value in the during treatment application group was similar to that in the controls throughout the four bleaching periods. After completion of the bleaching treatment, the value tended to be higher in the after treatment application group than in the controls throughout the four re-discolouration periods. Regarding the influence of TD on a^* , the value in the during treatment application group was similar to that in the controls throughout the four bleaching periods. After completion of the bleaching treatment, the value tended to be lower in the after treatment application group than in the controls throughout the four re-discolouration periods. Regarding the influence of TD on b^* , the value in the during treatment application group was similar to that in the controls throughout the four bleaching periods. After the completion of bleaching treatment, the values in the during and after bleaching groups were similar to those in the controls throughout the four re-discolouration periods.

Regarding the color difference, ΔE^*ab , ΔE after the 4th bleaching compared to the pretreatment value was 32.9 ± 4.5 in the controls and 44.9 ± 6.2 , 7.7 ± 10.9 , and 40.9 ± 9.6 in the during bleaching group with MS, NS and TD, respectively. This shows there was no significant difference between the controls and the teeth that received the concomitant use of the dentin desensitizers.

ΔE after the 4th re-discolouration compared to the value after the 4th bleaching was 42.0 ± 7.8 in the control group and 59.6 ± 9.1 , 34.4 ± 2.4 , and 31.9 ± 10.9 in the after bleaching group with MS, NS and TD, respectively, showing a significant difference with the concomitant use of MS applied after bleaching treatment compared to the controls.

Influences of various dentin desensitizers on SEM

The results of SEM observation are shown in Figs. 13 –16. Figure 13 shows an SEM image of a discolored

sample surface (before treatment). Adherent materials of staining and enamel structures were observed on the surface. Figure 14 shows SEM images after the 4th bleaching in the control and during treatment application groups. Structures thought to be enamel were observed on the surface of the controls. Struc-

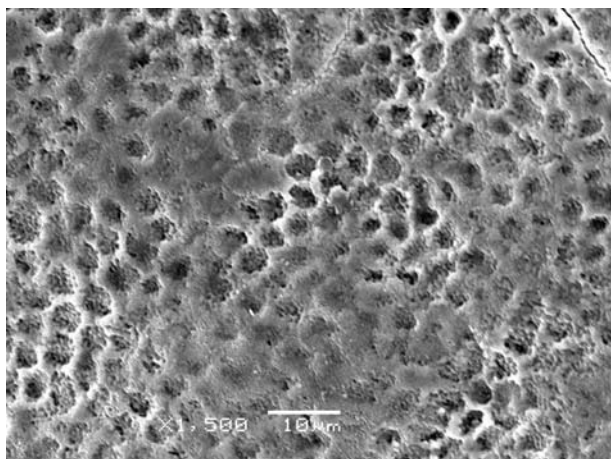


Fig. 13 SEM image of the surface of a discolouration specimen in the before treatment group ($\times 1500$).

tures thought to be enamel were also observed in all during treatment application groups, and the NS- and TD-applied surfaces were partially covered with aggregates. Figure 15 shows SEM images after the 4th relapse in the control and during treatment application groups. In the control group, more aggregates were deposited on the surface, making it smooth, compared to those immediately after the completion of bleaching treatment. More aggregates were also deposited on the surface, making the surface smooth, in all the during treatment application groups. Figure 16 shows SEM images after the 4th relapse in the control and after treatment application groups. In the control group, more aggregates were deposited on the surface, making it smooth, compared to those immediately after the completion of bleaching treatment. In the after treatment MS application group, more aggregates were deposited on the surface. Deposition was marked, compared to that in the control group, and the surface was uneven and rough. In the after treatment NS and TD application groups, enamel structures

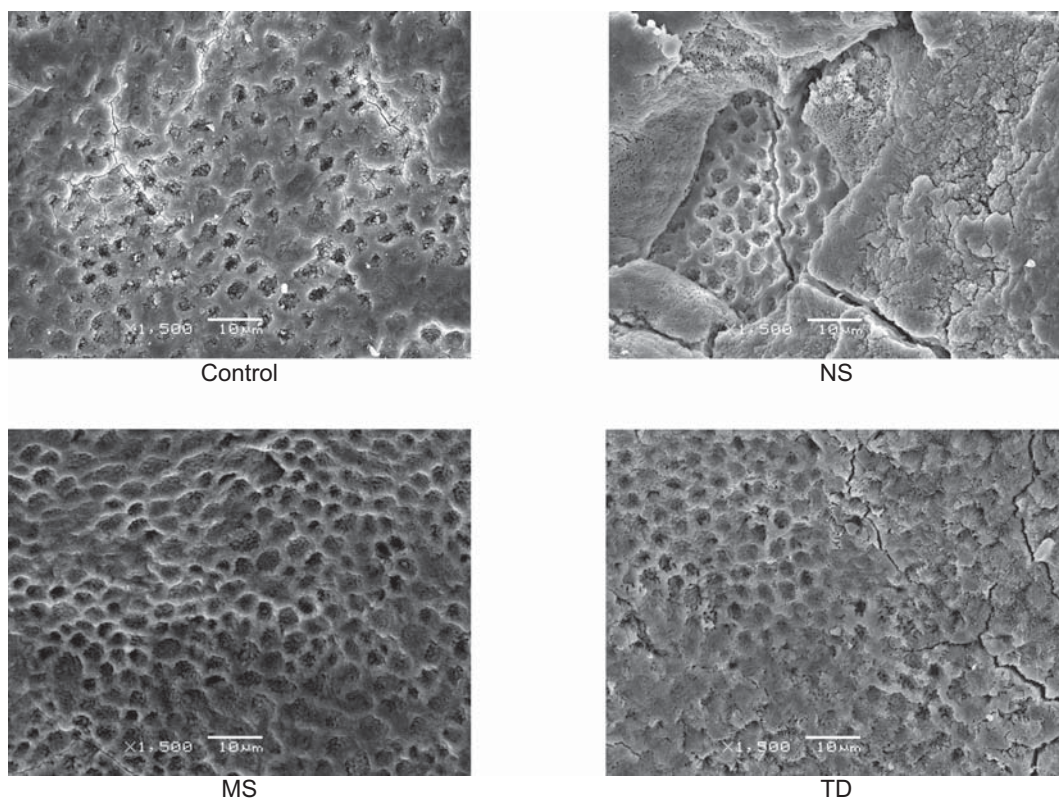


Fig. 14 SEM image of the surface after the 4th bleaching in the during bleaching group ($\times 1500$).

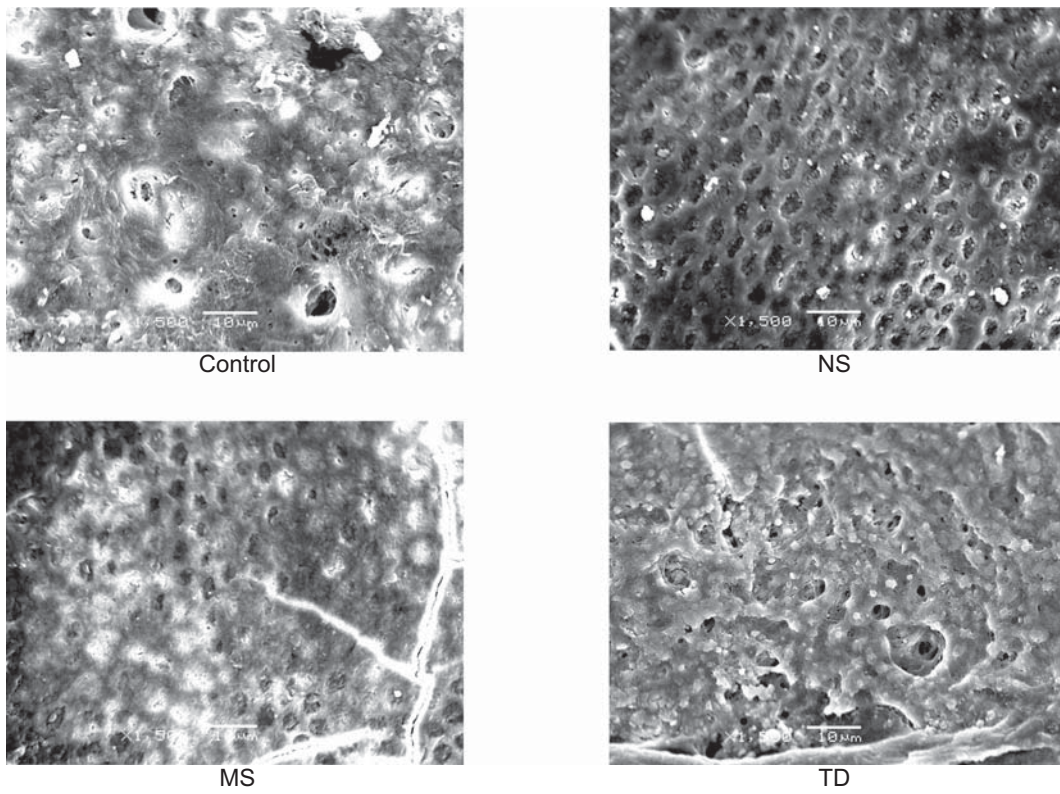


Fig. 15 SEM image of the surface after the 4th re-discolouration in the during bleaching group ($\times 1500$).

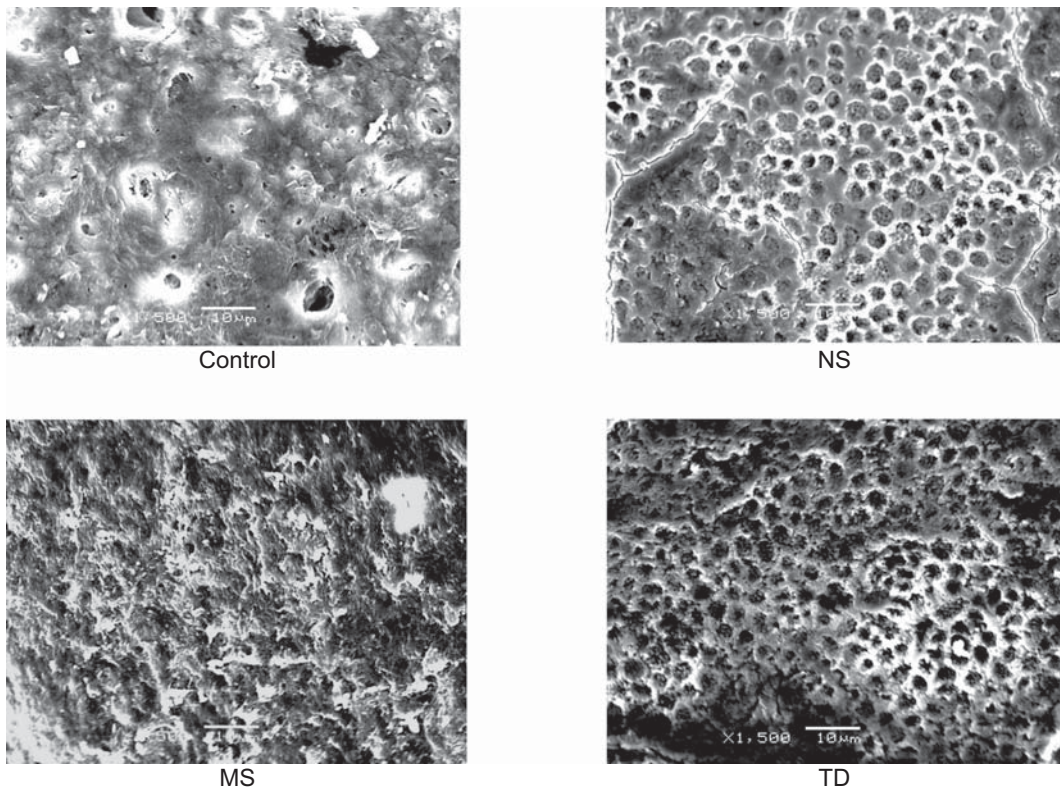


Fig. 16 SEM image of the surface at 4th re-discolouration in the after bleaching group ($\times 1500$).

were observed similar to those after the 4th bleaching. No aggregate deposition on the surface was noted.

DISCUSSION

Bleaching methods improving the tooth color without drilling are selected based on the causes of staining and discoloration. For exogenous stains due to food, drink, and other factors, improvement with mechanical tooth surface cleaning is possible when the stain is mild, however, for exogenous stains not improved by mechanical cleaning, mild tetracycline-stained teeth, and age-associated discoloration, bleaching of vital teeth is preferred. There are two methods for vital tooth bleaching: home bleaching performed by patients using a custom tray, and office bleaching performed by dentists at clinics. Office bleaching has the advantage of obtaining a bleaching effect within a short time compared to home bleaching and being applied under the control of a dentist. However, the main ingredient of agents used for office bleaching is a high concentration of hydrogen peroxide which markedly influences tooth quality. Moreover, hypersensitivity frequently develops.

In home bleaching, which uses bleaching agents containing a lower concentration of hydrogen peroxide, the main ingredient is 10–22% carbamide peroxide, which has less of an influence on tooth quality.^{16–19} In addition, the teeth can be homogeneously whitened. It is in common use. However, a reliable tooth bleaching effect cannot be readily obtained because it is performed by patients themselves at home without the direct control of a dentist. Moreover, it takes time to achieve a whitening effect, which reduces patient motivation causing them to discontinue treatment.¹⁹ Although products comprised of bleaching agents for vital tooth bleaching containing a hypersensitivity-inhibiting ingredient, such as potassium nitrate and CPP-ACP, are commercially available in the USA,^{20,21} they have not been approved in Japan, where hypersensitivity is dealt with as a separate treatment.^{22,23}

Symptoms of hypersensitivity include pain to abrasion with a toothbrush, transient pain induced by hot and cold liquids, and sweet-induced pain. However,

spontaneous pain is not a characteristic of hypersensitivity. Micro cracks in enamel made by abfraction caused by stress-induced bruxism and clenching, excess ingestion of low-pH health drinks, such as sports drinks and black vinegar, gastric acid reflux due to eating disorders, and mouth dryness are recently thought to aggravate the symptoms. When hypersensitivity develops during the bleaching treatment period, it can be dealt with by reducing the frequency or duration of treatment in most patients. However, effective hypersensitivity treatment becomes necessary when the hypersensitivity is severe and persists the following day, or when suspension of bleaching treatment is required. This hypersensitivity is treated by firstly reducing the duration and frequency of bleaching agent application, widening the interval, and then applying dentin desensitizers.²⁴

Dentin hypersensitivity is treated by drug application, iontophoresis, laser treatment, and coating with an adhesive material.^{25–35} Drug applications are the first-line treatment for many cases of dentin hypersensitivity because it is simple and fast-acting.³⁶ They have diverse mechanisms of action. Various products are applied clinically, such as those containing potassium nitrate and aluminum lactate as the main ingredients that act to desensitize the teeth,^{25,26} products that seal the dentinal tubules with inorganic salt crystals produced by reaction with calcium in the teeth,^{27–30} products containing glutaraldehyde as the main ingredient that seal the dentinal tubules through tissue fluid coagulation,³¹ and products sealing dentinal tubules with resin and glass ionomer cements.³²

However, dentin is not exposed when hypersensitivity develops during bleaching treatment of vital teeth, and treatment may be applied through the enamel surface. In this case it is important to block dental pulp stimulation through microcracks in the enamel to avoid excitation of dental pulp cells, calm down the hypersensitized dental pulp nerve, and promote deposition of calcified substances in microcrack openings in the enamel surface to seal the tooth. Accordingly, we selected three dentin desensitizers that act on the main constituent of enamel, hydroxyapatite, and investigated their influence on whitening effect when used concomitantly with bleaching treat-

ments.

TiON in Office

The application of photocatalytic titanium dioxide has been attracting attention as a catalyst. Nonami discovered that titanium dioxide, previously used mainly in the field of dentistry for washing and sterilization, primarily for its antifungal effect on dentures,^{37,38} can be used during whitening by mixing with a low-concentration hydrogen peroxide. They reported that it was effective for enamel surface bleaching. It has been confirmed that this low-concentration hydrogen peroxide bleaching agent containing titanium dioxide exhibits a marked whitening effect through the action of titanium dioxide as a photocatalyst. In addition, although the hydrogen peroxide concentration is low, the effect is equivalent to that of conventional bleaching agents that have a high concentration of hydrogen peroxide as the main ingredient.

Several products containing titanium dioxide as a catalyst and hydrogen peroxide at a reduced concentration are now sold for office bleaching. Generally, a titanium dioxide photocatalyst strongly reacts with ultraviolet light which is harmful to the human body, and does not react with visible light. TiON, which contains a visible light-responsive photocatalyst, became commercially available in 2010. Originally, active oxygen, such as the OH radical, is produced when titanium dioxide is irradiated with ultraviolet light, and the resulting active oxygen degrades various organic compounds that are not readily degradable.³⁹ Although titanium dioxide catalysts previously reacted only with ultraviolet light, Tion which was used in this study, contains the visible light-responsive photocatalyst (V-CAT), which reacts not only with the ultraviolet range, but also with 400–420-nm visible light.⁴⁰ Therefore, the photocatalytic action of titanium dioxide is induced by violet-blue visible light irradiation, and bleaching is promoted through the oxidizing action of the resulting OH radicals.

TiON is biocompatible because its pH is 5.26 ± 0.01 , which is almost equivalent to the physiological pH. SEM observation of the enamel surface treated with TiON has shown it to be noninvasive for enamel.⁴¹ Al-Salehi reported that the amounts of Ca and P

eluted from the tooth increase as the hydrogen peroxide concentration in the bleaching agent increases.⁴² Regarding the hydrogen peroxide concentration in bleaching agents for office bleaching, Titley³ reported that no structural changes were noted in the enamel after the application of 35% hydrogen peroxide solution for 60 minutes. In contrast, Kobayashi⁴ and Pugh⁵ described the influences of prolonged bleaching on the enamel surface. Nakazawa⁶ noted that the prolonged application of high-concentration hydrogen peroxide bleaching agents increases surface roughness, which tended to decrease after the application of a low-concentration of hydrogen peroxide bleaching agents. However, they noted that the change was not significant. Soma⁴³ reported that bleaching with low-concentration hydrogen peroxide is desirable because the reduction in surface roughness protects against re-discolouration after bleaching. The hydrogen peroxide concentration in TiON is about 23%. Although this is not low, we observed no adverse effects in our study. This may have been due to the pH being close to neutral during bleaching because of the effect of the acidity regulator. It has been found that TiON has no negative effect, such as decalcification, on the enamel.⁴¹

MS coat F (MS)

MS acts to seal dentinal tubules with inorganic salt crystals and polymer. When methyl methacrylate-styrenesulfonic acid copolymer emulsion and oxalic acid aqueous solution are applied, the copolymer reacts with hydroxyapatite in intertubular dentin and forms a macromolecular laminar capsule on the dentin surface, which precipitates inorganic polymer plugs containing calcium oxalate crystals and seals the openings of the dentinal tubules, inhibiting dentin permeation. Clinically, repeated applications followed by air drying are necessary.⁴⁴ The product has been modified as MS Coat F formulated with a high concentration (3,000 ppm) of fluoride with the goal of promoting tooth re-calcification.⁴⁵

A significant difference was noted in ΔE after the 4th re-discolouration compared with the value after the 4th bleaching in the group treated with MS after bleaching treatment when this value was compared with

the controls. In addition, aggregates were deposited on the enamel surface treated with MS, making the surface rough, compared with the controls. Nishimura *et al.*²⁴ reported that MS reacts with calcium of the tooth and becomes gelatinous, resulting in capsule formation by the polymer component. This suggests that the staining component of the stainer was incorporated during the capsule formation process. Nakabayashi *et al.*⁴⁶ reported that dispersed fine particles react with hydroxyapatite and form aggregates which are larger than the dentinal tubules, sealing the tubules. Soma⁴³ reported that the reduced surface roughness is advantageous against re-discolouration after bleaching, suggesting that MS enhanced re-discolouration due to the formation of a rough, thick capsule compared to the situation with other test dentin desensitizers.

NanoSeal (NS)

Nanoseal may have the characteristics of silicate cement, which is the antecedent of glass ionomer cement, because its main ingredient is a glass powder of fluoro-alumino-calcium silicate, the same as silicate cement. Nanoseal may induce a phenomena based on two chemical reactions: reaction of the material on the tooth surface receiving the application, and reaction between Nanoseal and the tooth surface. Acid erosion of the surface of fluoro-alumino-calcium silicate particles may occur as the materials react. This is similar to silicate cement, which elutes Al, Ca, F, and Si, leading to the formation of phosphate salts of Al and Ca and fine particles (nanoparticles) of fluoride and silicate compounds. These reactions elevate the pH of Nanoseal on the tooth surface receiving the application and induce nanoparticle deposition. Through this process, a low-soluble nanoparticle deposition layer may be formed on the tooth surface.^{32, 47, 48} It is possible that this nanoparticle deposition is effective in repairing the decalcified regions of early caries. Nanoparticle deposition and its binding with tooth defects in the tooth microstructure caused by bleaching-induced mineral loss may repair the decalcified tooth.

In the group with NS application during bleaching treatment, no significant difference was noted in ΔE

after the 4th bleaching compared to the pretreatment when this value was compared with the controls. In addition, structures assumed to be enamel were confirmed on SEM, similar to those in the controls. These findings suggest that the mechanism of action of NS did not inhibit the whitening effect during treatment.

In the group with NS application after bleaching treatment, structures assumed to be enamel and similar to those seen after the 4th bleaching were observed. However, no significant difference was noted in ΔE after the 4th re-discolouration compared to the value after the 4th bleaching when this value was compared with the controls, showing that re-discolouration was inhibited compared to the controls. It is believed that NS formed a capsule with thin, nano-level microcrystals on the hydroxyapatite of the tooth surface receiving treatment, compared to that formed by MS, and the reaction of NS with the tooth surface did not interfere with bleaching by TiON.

Teethmate Desensitizer (TD)

Brown and Chow⁴⁷ reported that Hap ($\text{Ca}_5(\text{PO}_4)_3\text{OH}$) is readily produced at room temperature in the presence of water when TTCP ($\text{Ca}_4(\text{PO}_4)_2\text{O}$) and DCPD ($\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$) or DCPA (dicalcium phosphate anhydrous: CaHPO_4) are present at a molar ratio of 1 : 1 through the reaction whereby calcium phosphate-re-calcifying solution comprised of TTCT and DCPD produces hydroxyapatite under physiological conditions. They confirmed that there was sealing of openings of the exposed dentinal tubules with apatite crystals.⁴⁸⁻⁵⁰ Since TD has been confirmed to seal microcracks of enamel with similar crystalline compounds, it may be expected to inhibit microcrack-induced hypersensitivity. The main ingredients of TD, TTCT and DCPD strongly bind to inorganic compounds of the dentinal tubular wall using hydroxyapatite produced by curing as a core. Although it has been observed that vital tooth bleaching causes calcium elution from the enamel, a slight reduction in hardness, and surface roughness, changes in the enamel surface properties are within the range where re-calcification is possible. Events similar to those noted in dentinal tubules were observed in microcracks of the enamel, suggesting that the effect leads to the protection of tooth quality and

promotion of re-calcification after bleaching.¹⁵

In the group with TD application during bleaching treatment, there was no significant difference in ΔE after the 4th bleaching compared to pretreatment when this value was compared with the controls. Structures assumed to be enamel were confirmed on SEM, similar to those noted in the controls, suggesting that the action mechanism of TD did not inhibit the whitening effect during treatment.

In the group with TD application after bleaching, structures assumed to be enamel were observed on SEM, similar to those noted after the 4th bleaching, and no significant difference was noted in ΔE after the 4th re-discolouration compared to that after the 4th bleaching, when this value was compared with the controls. This shows that re-discolouration was inhibited compared to the controls. This seems to suggest that TD forms a thin capsule with microcrystals on the tooth surface, receiving treatment similar to NS, and that the capsule is formed without de- and re-calcification of the tooth surface because the pH of the mixture is weakly alkaline.¹⁵

CONCLUSIONS

We investigated how various dentin desensitizers applied during and after bleaching treatment affected whitening. We found that when dentin desensitizers were applied during treatment, none of them influenced the whitening effect. Also, when the dentin desensitizers were applied after bleaching treatment, Nanoseal and Teethmate Desensitizer did not influence the whitening. We concluded that Nanoseal and Teethmate Desensitizer, which act on hydroxyapatite and form a thin capsule, can be concomitantly applied during and after bleaching treatment.

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