Effect of mesoporous silica nanoparticles on dentinal tubule occlusion: An in vitro study using SEM and image analysis

Lili TIAN1, Ce PENG2, Ying SHI2, Xuan GUO1, Bo ZHONG1, Juanjuan QI1, Guanhong WANG1, Qiang CAI2 and Fuzhai CUI2

1 Center of Stomatology, China–Japan Friendship Hospital, 2 Yinghuayuan Dongjie, Chaoyang District, Beijing 100029, P. R. China
2 Department of Materials Science and Engineering, Tsinghua University, Haidian District, Beijing 100084, P. R. China
Corresponding author, 1st: Lili TIAN; E-mail: dentistlily@aliyun.com
2nd: Qiang CAI; E-mail: caiqiang@mail.tsinghua.edu.cn

INTRODUCTION

Dentin hypersensitivity is a common dental problem caused by the exposure and patency of dentinal tubules. The hydrodynamic theory of dentin hypersensitivity is widely accepted. It states that external irritation induces the movement of fluid into or out of dentinal tubules, which stimulates sensory nerve endings in the dentinal tubules and leads to dentin hypersensitivity. Based on this theory, there are two general approaches for the treatment of dentin hypersensitivity: nerve desensitization and/or tubular occlusion. Occlusion of patent dentinal tubules would be a more effective means of treating dentin hypersensitivity.

Besides physical agents (mainly laser treatment), various chemical agents which can form insoluble precipitates in dentinal tubules have been used for tubular occlusion. They include light-cured bonding materials, strontium chloride, sodium fluoride, potassium nitrate and potassium oxalate, silica, calcium-based remineralizing agents, and nanosized carbonate apatite. Some dentin hypersensitivity treatment products contain a variety of chemical agents. For example, Green-Or™ densitizer contains potassium phosphate, potassium carbonate, calcium chloride, strontium chloride, etc. Nonetheless, most chemical agents are used to close dentinal tubules, except for potassium phosphate which has the effect of nerve desensitization. Although these agents and products were reported to be effective, their therapeutic effects were generally short-lived or diminished with time. This is because these occluding materials are easily removed by daily brushing, food chewing, and the consumption of acidic drinks. To ensure sustained treatment effect, a novel material that produces a deeper sealing into the dentinal tubules needs to be developed.

Three types of materials have attracted researchers’ attention: calcium phosphate precipitation (CPP), silica, and nanomaterials. The CPP method has exhibited potential in the occlusion of dentinal tubules, as dentin is primarily composed of calcium and phosphate composites. CPP occluding agents may provide twofold functions in the initial mechanical plugging and in long-term remineralization. In previous studies, occlusion of dentinal tubules was obtained by wiping a mixture of calcium and phosphate on the dentinal surface. CPP which forms in this way on the dentinal surface cannot deeply penetrate into the dentinal tubules. Further, as dentin remineralization is a long-term process, the ideal way of releasing calcium and phosphate should be a slow release.

Artificial silica-based products also seemed to be effective at tubular occlusion. The stability of silica to adhere to and occlude tubules may result from hydroxyl groups binding silica to the calcium receptors on the dentin surface. Nanomaterials with superior dispersion can easily enter dentinal tubules of 2–3 μm diameters; thus, they can be prime candidates for dentinal tubule occlusion. The properties of nanoparticles clearly differ from those of their corresponding bulk materials. The solubility and reactivity of nanoparticles are significantly increased because of high surface energy and a large surface area. The large surface area of nanoparticles also provides a high affinity and allows them to easily deposit...
on irregular spaces\(^{15}\).

Inspired by the properties of CPP, silica, and nanomaterials, we introduced novel mesoporous silica nanoparticles (MSNs) for dentinal tube occlusion. As an important fundamental nanomaterial, MSNs have attracted significant amount of research interest due to their ordered porous structure, facile synthesis methods, and broad range of applications. As opposed to non-porous silica nanoparticles, both the surface and pore interior of mesostructured nanoparticles can be modified with functional groups, such that they become compatible in various solutions and are able to store different types of molecules. The biocompatibility and potential as delivery vehicles for proteins and anticancer drugs of these nanomaterials have been well demonstrated\(^{10}\).

When MSNs are used for dentinal tubule occlusion, we speculated that nano-sized MSNs with superior dispersion, high solubility and reactivity would easily enter the dentinal tubules of 2–3 μm diameters. Due to their high affinity and the aid of hydroxyl groups on their surfaces, they could easily adhere to the dentin surface. If calcium and phosphates can be encapsulated in MSNs as calcium and phosphate sources which slowly release Ca\(^{2+}\) and PO\(_4^{3-}\) in the dentinal tubules, it could improve the long-term efficacy of dentinal tubule occlusion and remineralization. In a previous study\(^{12}\), mesoporous silica powder loaded with a large amount of nano-sized CaO particles was mixed with 30% phosphoric acid to form supersaturated Ca\(^{2+}\) and HPO\(_4^{2-}\), which then entered the dentinal tubules and formed a calcium phosphate precipitate. In this study, MSNs were directly used for dentinal tubule occlusion. MSNs were expected to have great potential as a clinical treatment option for dentin hypersensitivity.

The aims of this study were: (1) prepare MSNs and MSNs with independently encapsulated calcium and phosphates; (2) prove the hypothesis that MSNs or Ca\(^{2+}/P0_{4}^{3-}\)@MSNs desensitizing slurry could efficiently occlude dentinal tubules. The structural features of the materials were characterized by scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray powder diffraction (XRD), and X-ray fluorescence (XRF). The occlusion efficacy of the desensitizing slurry at both the open exterior end of dentinal tubules and in the depth of dentinal tubules were evaluated using SEM images, and the degree of occlusion of dentinal tubules was also evaluated using SEM and image analysis methodology for statistical analysis. Comparisons were made with the commonly used Green-Or\textsuperscript{TM} densitizer.

**MATERIALS AND METHODS**

**Preparation of MSNs and Ca\(^{2+}/P0_{4}^{3-}\)@MSNs**

The MSNs were synthesized as described in our previous studies\(^{18,19}\). Typical syntheses of Ca\(^{2+}\)@MSNs and PO\(_4^{3-}\)@MSNs were as follows: 0.900 g of sodium dihydrogen phosphate (NaH\(_2\)PO\(_4\cdot2\text{H}_2\text{O}\)) and 0.952 g of anhydrous calcium chloride (CaCl\(_2\)) were separately dissolved in a sealed vial with 100 mL of distilled water and stirred for 30 min at room temperature. Then, 0.300 g of MSNs was added and stirring continued for 2 h. The powder of Ca\(^{2+}/P0_{4}^{3-}\)@MSNs was collected by filtration and washed with deionized water. Rinsing was required because the powder particles of Ca\(^{2+}/P0_{4}^{3-}\)@MSNs had some residual chemicals on their surfaces. Then, the samples were obtained by centrifugation and drying in an oven at 60°C. After grinding, the dried powder was collected for the next procedure. All the chemicals mentioned herein were purchased from Beijing Xizhong Chemical Plant.

The structural features of MSNs and Ca\(^{2+}/P0_{4}^{3-}\)@MSNs were examined by SEM (JSM-6301F, Jeol, Japan; S-4800, Hitachi, Japan) and TEM (Tecnai G2 20, FEI, Japan) operated at 200 kV. The mesoporous structure of the three kinds of products was analyzed with XRD (D/max-ra diffractometer, Rigaku, Japan) using Cu K\(_\alpha\) radiation at \(\lambda=1.5418\ \text{Å}\). Each sample was scanned from 0.8° to 10° (2\(\theta\)) with a step size of 0.02° and a count time of 1 s at each point. It was performed to characterize the chemical compositions of the samples of Ca\(^{2+}\)@MSNs and PO\(_4^{3-}\)@MSNs by using XRF (XRF-1800, Shimadzu, Japan).

**Preparation of samples**

Freshly extracted, caries-free, human third molars were collected and stored in physiological saline at 4°C. Coronal dentin disks of 1.0 mm thickness were cut perpendicular to the long axis of the tooth at a level of 1.0 mm above the cementoenamel junction with a diamond saw (Accutom-50, Struers, Denmark) (Schematic 1A).

To observe the surface morphological changes of dentin samples, a central area of 5 mm diameter on the dentin disks was defined as the experimental area, as shown in Schematic 1B. Five dentin disks were randomly selected. Each disk was divided into four equal parts for control and testing (Schematic 1C). All the four parts were pretreated with 6% citric acid for 2 min and then subjected to ultrasonic cleaning with distilled water for 30 s, before being randomly divided into four groups as follows.

- **Group 1 (Control)**: Citric-etched samples without any treatment.
- **Group 2 (Green-Or\textsuperscript{TM})**: Liquid 1 of Green-Or\textsuperscript{TM} (Ital Med, Italy) contained potassium phosphate, potassium carbonate, sodium methylparaben, and deionized water; while Liquid 2 contained calcium chloride, strontium chloride, sodium benzoate, and deionized water.
- **Group 3 (MSNs)**: MSNs powder was mixed with distilled water at a ratio of MSNs:H\(_2\text{O}=0.015\text{ g}:75\text{ μL}\) to make the desensitizing slurry.
- **Group 4 (Ca\(^{2+}/P0_{4}^{3-}\)@MSNs)**: Ca\(^{2+}\)@MSNs and PO\(_4^{3-}\)@MSN powders were mixed with distilled water in a ratio of Ca\(^{2+}\)@MSN:PO\(_4^{3-}\)@MSN:H\(_2\text{O}=0.015\text{ g}:0.015\text{ g}:150\text{ μL}\) to make the desensitizing slurry.

In Groups 3 and 4, the desensitizing slurry was immediately applied on the dentin samples with a dental brush. After 30 s, the slurry was applied again. In Group
2, the application procedure followed the manufacturer’s instruction. Treated samples were gently rinsed with distilled water for 10 s.

To observe morphological changes of dentin samples in the longitudinal section, four dentin disks were randomly selected. Each disk was sectioned in half (Schematic 1D) and pretreated with 6% citric acid. Eight samples were randomly divided into four groups, with two samples per group. After applying the desensitizing materials, each sample was further sectioned into halves (Schematic 1E) to evaluate the efficacy of dentinal tubule occlusion in the longitudinal section.

**Evaluation of dentinal tubule occlusion efficacy**

The surface morphological changes of dentin samples and the depths of desensitizing materials penetrating the dentinal tubules were evaluated by SEM. Length of the precipitate formed along the dentinal tubules from the dentinal surface to the bottom of precipitate was measured in at least 20 dentinal tubules for each sample. In addition, the degree of occlusion of the dentinal tubules on the dentinal surface was quantified using SEM and image analyses (Image-Pro PLUS 6.0, Media Cybernetics, Silver Spring, MD, USA). Three images were taken at a constant magnification of ×1000, with a 50-μm scale bar from the central portion of each sample (Schematic 1C). SEM images were saved as TIF files for image analysis. The polygon tool in the major tool bar was used to measure the area of open dentinal tubules. The relative area (RA) of open dentinal tubules was calculated in this study. Levene’s test showed that group variances were not homogeneous (p<0.05), and Kruskal-Wallis test was applied to RA which was designated as the surface treatment factor. Tukey’s post hoc test was used for multiple comparisons. Statistical significance was preset at α=0.05. All statistical analyses were performed using SPSS 13.0 software (SPSS, Chicago, IL, USA).

**RESULTS**

**Characterization of MSNs, Ca²⁺@MSN and PO₄³⁻@MSN**

Characterization results by SEM (Figs. 1A–C) and TEM (Figs. 1D–F) demonstrated that the morphologies and structures of the particles of MSNs, Ca²⁺@MSNs and PO₄³⁻@MSNs were similar. Particles of MSNs, Ca²⁺@MSNs and PO₄³⁻@MSNs were uniform spheres of about 50–80 nm in size with no agglomeration. They presented a highly ordered porous structure with a hexagonal lattice (indicated by arrows in Figs. 1D–F) and streak structural feature (indicated by dovetail arrows in Figs. 1D–F). Pores were 3 nm in diameter.

However, there were some differences between MSNs and Ca²⁺/PO₄³⁻@MSNs in the fine structure. TEM images revealed that the clarity of steak feature of Ca²⁺@MSNs and PO₄³⁻@MSNs was not better than that of MSNs, probably indicating Ca²⁺ or PO₄³⁻ embedded in MSNs. In other words, Ca²⁺ or PO₄³⁻ nano-composites encapsulated in the pores of MSNs led to reduced interference of periodic mesostructure of MSNs. Notably in Fig. 1F, the particle with a dark contrast feature implied that phosphate was well embedded in the pores of MSNs. The XRD patterns of these three kinds of products were all the same, as shown in Fig. 1G. Four sharp Bragg peaks were shown, which could be indexed as (100), (110), (200), and (210) of MSNs respectively, suggesting a perfect long-range order in these materials. The XRF results of Ca²⁺@MSNs and PO₄³⁻@MSNs are presented in Table 1. Data given in Table 1 showed that CaO, P₂O₅, and SiO₂ were mainly present in quantity while other chemical components were present in trace amounts. This confirmed the chemical compositions of the samples.

**SEM evaluation of the occlusion of dentinal tubules**

According to the SEM images, morphologies of the dentinal surfaces showed different changes when treated with different desensitizing materials (Fig. 2).
Fig. 1  SEM images (Figs. 1A–C) and TEM images (Figs. 1D–F) of MSNs and Ca^{2+}/PO_{4}^{3−}@MSNs. In TEM images, a highly ordered hexagonal lattice (indicated by arrow) and streak structural feature (indicated by dovetail arrow) could be seen. Fig. 1G shows XRD patterns of MSN, Ca^{2+}@MSNs, and PO_{4}^{3−}@MSNs samples.

Fig. 2  SEM images of surface morphological changes of dentinal tubules pretreated with 6% citric acid (Figs. 2A, 2a) and treated with MSNs (Figs. 2B, 2b), Ca^{2+}/PO_{4}^{3−}@MSNs (Figs. 2C, 2c), and Green-Or™ desensitizing material (Figs. 2D, 2d). Figs. 2a–d are high-magnification images of Figs. 2A–D. In Fig. 2A, a large number of distinctly open dentinal tubules could be seen. In Figs. 2B and 2C, dentinal tubules were almost completely occluded. In Fig. 2D, some open dentinal tubules were exposed. Figs. 2b and 2e were high-magnification SEM images, where nano-sized particles could be seen entering the dentinal tubules (indicated by dovetail arrow). In Figs. 2d and 2f, occlusion of open dentinal tubules was insufficient (indicated by arrow).

### Table 1  The XRF results of Ca^{2+}@MSNs and PO_{4}^{3−}@MSNs products

<table>
<thead>
<tr>
<th></th>
<th>Analyte Amount (wt. %)</th>
<th>Analyte Amount (wt. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ca^{2+}@MSNs</td>
<td>SiO_{2} 94.7876%</td>
<td>SiO_{2} 96.3363%</td>
</tr>
<tr>
<td></td>
<td>CaO 3.0893%</td>
<td>P_{2}O_{5} 2.6623%</td>
</tr>
<tr>
<td></td>
<td>Others 2.1231%</td>
<td>Others 1.0014%</td>
</tr>
<tr>
<td>PO_{4}^{3−}@MSNs</td>
<td>SiO_{2}</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The baseline sample that was etched with 6% citric acid showed a large number of distinctly open dentinal tubules whereby the tubules were more or less round (Figs. 2A, 2a). Dentin samples treated with MSNs and Ca\(^{2+}\)/PO\(_4\)^{3-}\@MSNs resulted in almost complete occlusion of dentinal tubules (Figs. 2B, 2b; 2C, 2c). High-magnification SEM images showed that the surfaces of the samples were covered by very small particles which were similar to the morphology of MSNs (Figs. 2b, 2e). However, in the dentin sample...
treated with Green-Or™, open dentinal tubules and big crystalloids were observed to cover the dentin surface (Figs. 2D, 2d, 2f).

SEM images in Fig. 3 showed the dentinal tubules pretreated with 6% citric acid and the depths of different desensitizing materials penetrating the dentinal tubules. The magnified SEM images showed that a large quantity of nano-sized particles filled the dentinal tubules (Figs. 3b, 3c) and the morphologies of these small particles were similar to that of MSNs. In Green-Or™ group, the dentinal tubules were almost empty and there was only a small amount of material entering the dentinal tubules.

Piecewise measurement method was used to measure the depths of materials penetrating the dentinal tubules, as shown in Fig. 4. The precipitate depths of MSNs and Ca²⁺/PO₄³⁻@MSNs Groups were measured in at least 20 dentinal tubules for each sample. To accurately measure the depth of nanoparticles entering the dentinal tubules, the nanoparticles must be clearly seen in the dentinal tubule in the SEM image. However, MSNs of 50–80 nm were too small compared with the dentinal tubules with diameters of 2–3 μm. To observe the nanoparticles in a dentinal tubule, it must be done at high magnification. As a result, the length of a dentinal tubule was limited under a certain field of vision in SEM. If nanoparticles deeply penetrated the dentinal tubules, piecewise measurement method must be adopted. In Fig. 4, summation of the four figures was approximately 105 μm, which was the complete precipitate length in a dentinal tubule. Based on this method, precipitate lengths of MSNs Group and Ca²⁺/PO₄³⁻@MSNs Group were measured to be about 150 μm for deepest depth and 105 μm for average depth. Figure 4a was the high-magnification image of a local part (indicated by circle) of a precipitate in one of the measured dentinal tubules, in which a large number of nano-sized particles was clearly seen, suggesting that the precipitate was probably MSNs. Sealing depth of Green-Or™ Group was about 5 μm only.

**Statistical image analysis results**

Table 2 presents the statistical results of the relative area (RA), expressed in mean and standard deviation, of open dentinal tubules using image analysis methodology for the four groups. In the etched baseline sample, RA was 5.615%. RA decreased after treatment with the three desensitizing materials. The RA values of Groups 3 and 4 were smaller than that of Group 2. Statistical analysis revealed significant differences among the groups, and post hoc comparisons revealed statistically significant differences (p<0.001) between Group 2 and Group 3, and between Group 2 and Group 4. There was no significant difference (p>0.05) between Group 3 and Group 4.

**DISCUSSION**

In this study, MSNs and MSNs loaded with calcium and phosphates were independently prepared. Then, MSNs or Ca²⁺/PO₄³⁻@MSNs were mixed with distilled water to form desensitizing slurries, which were used to treat sample dentin surfaces. Compared with Green-Or™ densitizer, results demonstrated that MSNs or Ca²⁺/PO₄³⁻@MSNs desensitizing slurry could efficiently occlude dentinal tubules at both the exterior open end of dentinal tubules and in the depth of dentinal tubules. It was suggested that Ca²⁺/PO₄³⁻@MSNs as calcium and phosphate sources could supply Ca²⁺ and PO₄³⁻ for the long-term remineralization process and further improve tubular occlusion efficacy. For a more objective and accurate evaluation of the efficacy of MSNs or Ca²⁺/PO₄³⁻@MSNs in occluding dentinal tubules, sample preparation methods and evaluation methodology used in this study were modified from previous studies²⁰-²².

MSNs were successfully prepared, and preliminary experiment of MSNs loaded with calcium or phosphates (Ca²⁺/PO₄³⁻@MSNs) was further performed. The method of MSNs hydrothermally synthesized at low surfactant concentration by our group has been accepted in the academic community²³. Synthesized MSNs had uniform nano-sized spheres, a high-order porous structure, and superior dispersion, as shown in the SEM and TEM images in Figs. 1A and 1D. MSNs loaded with calcium or phosphates were obtained by soaking MSNs in anhydrous calcium chloride (CaCl₂) or sodium dihydrogen phosphate (NaH₂PO₄·2H₂O) solution. CaCl₂ and NaH₂PO₄·2H₂O were incorporated into the MSNs through adsorption. This synthesis method was simple, practicable, and effective²³, but was not previously reported to be used for MSNs loaded with calcium or phosphates. After Ca²⁺ and PO₄³⁻ were encapsulated in MSNs, the basic structure of Ca²⁺/PO₄³⁻@MSNs did not change (Figs. 1B, 1C, 1E, 1F). The XRD patterns of these three kinds of products also indicated that they all shared a typical mesoporous structure (Fig. 1G). The efficacy of Ca²⁺ or PO₄³⁻ encapsulated in MSNs could be proved by TEM observation and XRF analysis.

<table>
<thead>
<tr>
<th>Group</th>
<th>RA</th>
<th>Group2 (GreenOr™)</th>
<th>Group3 (MSNs)</th>
<th>Group4 (Ca²⁺/PO₄³⁻@MSNs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RA</td>
<td>5.615 (0.427)</td>
<td>0.457 (0.116)</td>
<td>0.006 (0.003)</td>
<td>0.006 (0.002)</td>
</tr>
</tbody>
</table>

Sample images (n=15 for each group) were used. The data were expressed as mean and standard deviation and statistically analyzed using one-way ANOVA with Tukey's test. Different superscript letters indicate statistically differences (a, VS control, p<0.001; b, VS GreenOr™ p<0.001).
In TEM images (Figs. 1E, 1F), the absence of clear streak feature of Ca²⁺@MSNs and PO₄³⁻@MSNs which comprised MSNs or even the particle with a dark contrast feature of PO₄³⁻@MSNs implied that Ca²⁺ and PO₄³⁻ were embedded in MSNs. XRF analysis further showed the existence of a certain amount of calcium and phosphorus in Ca²⁺/PO₄³⁻@MSNs product.

MSNs or Ca²⁺/PO₄³⁻@MSNs slurry efficiently occluded the dentinal tubules. Nano-sized MSNs were easily and rapidly dispersed in distilled water due to their high specific surface area. Therefore, when mixed with distilled water, a large quantity of instantly formed MSNs easily entered the dentinal tubules and deposited on the walls of dentinal tubules. SEM observation results and statistical image analysis proved that MSNs not only almost completely occluded the dentinal tubules at the dentin surface, but also penetrated the dentinal tubules to a depth of about 105 μm, presenting results which were superior to the previous work11-13). The tubular occlusion efficacy of Ca²⁺/PO₄³⁻@MSNs group was similar to that of MSNs group at both the dentin surface and in the depth of dentinal tubules. According to previous studies8,10, it was suggested that MSNs or Ca²⁺/PO₄³⁻@MSNs adhered to the walls of dentinal tubules with the aid of hydroxyl groups on their surface to achieve better sealing of dentinal tubules. In Ca²⁺/PO₄³⁻@MSNs group, Ca²⁺ or HPO₄²⁻ independently embedded in MSNs prevented Ca²⁺ and HPO₄²⁻ from contacting and reacting with each other, thus leading to rapid precipitation of calcium phosphate at the dentin surface alone. It was speculated that Ca²⁺/PO₄³⁻@MSNs enabled more ions to be released, such that reactions in the dentinal tubules improved the function of initial mechanical plugging. The precipitate of MSNs or Ca²⁺/PO₄³⁻@MSNs in the dentinal tubules was deeper than that of Green-Or™ densitizer. In the latter, many large crystalloids piled up on the dentin surface while only a small amount of the material was found in the dentinal tubules, as shown in Fig. 2D. Based on these results, it was speculated that MSNs or Ca²⁺/PO₄³⁻@MSNs could render superior dentinal tubule occlusion. These results also explained why the efficacy of Green-Or™ densitizer was relatively short-lived in clinical treatment.

MSNs embedded with Ca²⁺ or PO₄³⁻ respectively could act as calcium and phosphate sources to supply Ca²⁺ and PO₄³⁻ for the long-term remineralization process. It was speculated that the slow release of Ca²⁺ and PO₄³⁻ by Ca²⁺/PO₄³⁻@MSNs in the dentinal tubules was beneficial to the long-term dentin remineralization process. Moreover, according to a previous work on simulated body fluids and synthetic saliva, some silica compounds could induce apatite formation20. The use of bioactive hydroxyapatite and silica nanoparticles promoted infiltration and subsequent remineralization of dentin25. The efficacy of dentin mineralization induced by MSNs and the efficacy of CPP derived from Ca²⁺/PO₄³⁻@MSNs were combined to achieve long-term tubular occlusion.

In this study, sample preparation methods and the evaluation methodology of the efficacy of MSNs or Ca²⁺/PO₄³⁻@MSNs in occluding dental tubules were modified from previous studies20-22), and they were considered to be more objective and accurate. Dentin disks were standardized as much as possible with regard to the age of tooth, site of tooth, and direction of dentinal tubules. Experimental areas were carefully inspected to ensure that they were free of coronal enamel, pulpal exposures, secondary dentin, or microcracks. Each disk was divided into four equal parts for both control and test groups to ensure reliability of the experiment. For a systematic image analysis, the relative area (RA) of open dentinal tubules was calculated based on three continuous SEM images at a constant magnification on each sample to assess the efficacy of tubular occlusion quantitatively.

Low content of calcium/phosphates in Ca²⁺/PO₄³⁻@MSNs was one of the limitations of this study. Therefore, further work to increase the amount of calcium and phosphates embedded in MSNs will be carried out. Besides, the controlled release systems for Ca²⁺ and PO₄³⁻ will also be studied, such as using light stimuli for controlled release systems23) to release Ca²⁺ and PO₄³⁻ completely into the dentinal tubules, so as to achieve better initial mechanical plugging and long-term remineralization.

CONCLUSION

In conclusion, MSNs and Ca²⁺/PO₄³⁻@MSNs mixed with distilled water demonstrated superior performance in occluding dental tubules. Thus, this novel nanomaterial could improve or open up new possible applications in clinical dentistry.

ACKNOWLEDGMENTS

This study was funded by China-Japan Friendship Hospital (No. 2010-M-25), Capital Medical Development Scientific Research Fund (No. 2009-3025), and NSFC (No. 50830102). The authors would like to thank Crest Oral Nursing Research Institute for providing the dental cutting machine to prepare the dentin disks.

REFERENCES

6) Kishore A, Mehrotra KK, Saimhi CS. Effectiveness of...