Original Article

Effect of Desensitizing Toothpaste Containing Calcium Sodium Phosphosilicate on Microtensile Bond Strength of Adhesive Systems

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Abstract

The purpose of this study was to evaluate the effect of a desensitizing toothpaste, containing calcium sodium phosphosilicate, on the microtensile bond strength of adhesive systems treated with dentine. Fifty-two human third molars were embedded into acrylic resin, and cut to expose a flat dentin surface. The specimens were randomly divided into two groups, 1) no brushing, and 2) brushing with Sensodyne Repair & Protect (GSK, London, UK) for 10,000 cycles with a V-8 cross brushing machine (Sabri Dental Enterprise, Inc., USA). Subsequently, both groups were divided into three groups for resin composite build-up using different adhesive agents: OptiBond FL® (Kerr, Orange, CA, USA), Clearfil SE Bond® (Kuraray Medical Inc, Japan), and Single Bond Universal® (3M ESPE, USA). All samples were subsequently sectioned to obtain microtensile test specimen, after which the sectioned sticks in the same tooth were divided into two subgroups: 1) microtensile bond strength test, and 2) thermocycling for 10,000 cycles, followed by microtensile bond strength test. Two-way ANOVA revealed that the μTBS values of each adhesive system was not significantly affected by brushing with desensitizing toothpaste containing calcium sodium phosphosilicate. After brushing with desensitizing toothpaste containing calcium sodium phosphosilicate, OptiBond FL® had the highest μTBS value. Clearfil SE bond® showed no significantly different immediate μTBS value compared to Single Bond Universal®, but showed a significantly higher μTBS value than Single Bond Universal® after 10,000-cycle thermocycling. In addition, 10,000-cycle thermocycling significantly decreased the μTBS value of Single Bond Universal® after brushing. In conclusion, desensitizing toothpaste containing calcium sodium phosphosilicate had no effect on OptiBond FL® or Clearfil SE bond®, either for immediate μTBS or after 10,000-cycle thermocycling. Meanwhile, Single Bond universal® adhesive showed decreased μTBS after 10,000-cycle thermocycling.

Keywords: Bonding agents, Calcium sodium phosphosilicate, Desensitizing, toothpaste, Microtensile bond strength, Thermocycling

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Introduction

Tooth hypersensitivity is characterized by short sharp pain arising from exposed dentin in response to stimuli that can be thermal, evaporative, tactile, osmotic or chemical[1,2]. It is usually found in a tooth where underlying dentin has been exposed.[3,4] Using desensitizing toothpaste is one of the treatments for tooth hypersensitivity. The advantage of using desensitizing toothpastes is that they are immediately available for treatment when compared to agents applied by a professional.[5] Their function is either to block pulp nerve response or occlude opened dentine tubules.[6,7] To block the nerve, some products contained potassium salts, which are thought to diffuse inside the dentinal tubules and lower the excitability of pulpal nerve fibers.[8] Occlusive therapies for the treatment of dentinal hypersensitivity are frequently used. It was believed that sealing dentinal surface subsided movement of fluid inside the tubules and reduced the sensitivity.[5] Strontium salt provided layers of deposited small particles to block the opened dentinal tubules.[9] However, several clinical trials failed to demonstrate the superior efficacy of strontium-based formulations containing silica over that of conventional fluoridated toothpaste.[10,11] A study reported that arginine-calcium carbonate desensitizing paste provided complete occlusion of open dentinal tubules.[12] There were also in vitro and clinical studies showing that arginine-calcium carbonate toothpastes reduced sensitivity.[6,12,13] Recently, a component of calcium sodium phosphosilicate has been introduced. It has been used as a component in dentifrice to provide relief from dentine hypersensitivity. Several studies have shown that dentifrice containing calcium sodium phosphosilicate formed a deposit over dentine and in the tubules. When calcium sodium phosphosilicate was exposed to saliva, calcium and phosphate ions were released from particles, the pH was increased to facilitate the precipitation of calcium and phosphate from the particles and from saliva to form a calcium phosphate layer on tooth surfaces, or into tubules. This layers crystalized into hydroxycarbonate apatite-like deposits, which were chemically and structurally similar to minerals found in a tooth.[14,15] The study demonstrated that a deposition of calcium sodium phosphosilicate on dentine was more acid-resistant and showed better dentinal tubule occlusion and retention than the application of arginine-containing toothpaste.[16] However, topical desensitizing agent had a temporary effect on occluding the dentinal tubule. If sensitivity persisted or the lesion became more extensive, stronger and more adhesive materials were preferred for longer-lasting desensitization.[17] When extended to consider restorative strategies, resin-based composite restoration has been preferred based on its excellent esthetic properties and good clinical performance in studies one year or more in duration.[3,18] A study showed that using dentifrice was significantly less effective in reducing sensitivity than sealants and the restorative treatment, either in clinical or reported patients.[3] When long-term desensitization using toothpaste fails as the tooth surface loss becomes extensive, definitive restoration of the hypersensitive area using resin composite may be needed. Previous studies showed that the use of desensitizing toothpaste resulted in occlusion of the dentinal tubules, which might affect the bonding performance of subsequent restoration.[18,19] A study found that the microtensile bond strength of adhesive to dentin specimens treated with arginine or strontium acetate desensitizing toothpaste was significantly lower than that of regular toothpaste when using a three-step etch-and-rinse and a self-etch bonding agent.[19] On the other hand, another study showed that prolonged use of desensitizing toothpaste containing 8% arginine/calcium carbonate, 8% strontium acetate and 5% calcium sodium phosphosilicate did not influence the bond strength of a self-etching adhesive system to dentin.[20] Even though there have been studies concerning the effect of desensitizing toothpaste on dental adhesives, very few studies have focused on the effect of calcium sodium phosphosilicate. Therefore, the objective of this study was to evaluate the effect of desensitizing toothpaste containing calcium sodium phosphosilicate on microtensile bond strengths of adhesive systems treated to dentin.

Materials and Methods

This study was approved by the ethical committee of the Faculty of Dentistry, Chulalongkorn University, Thailand (approval number: HREC-DCU 2019-041).

Dentin samples were prepared from 52 permanent third molar teeth extracted with informed consent stored in a 0.1% thymol solution at 4°c and used within three months of extraction. Teeth were carefully inspected using a stereomicroscope (ML 9300; Meiji Techno Co. Ltd., Japan) at 40X magnification to ensure that they were free of caries, cracks or restoration. Teeth were embedded in a self-curing resin with their occlusal surfaces exposed parallel to a horizontal plane at 2 mm below the cemento-enamel junction. Occlusal one-third of the crown was removed perpendicular to the long axis of the tooth using a low-speed diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA) under running water until the enamel was completely removed. Each tooth was carefully inspected using a stereomicroscope to ensure that it was free of enamel. One percent citric acid solution was used to immerse specimens for 20s and rinsed with distilled water for 20s to open up dentinal tubule to mimic a dentine hypersensitivity scenario. Then, two teeth were confirmed for the tubular opening using a scanning electron microscope (Quanta 250, FEI, Hillsboro, OR, USA). Teeth were randomly divided into two groups; Group A (n=24): non brush (control) and Group B (n=26): brushed with Sensodyne Repair & Protect®. In Group B, teeth were brushed with the dentifrice slurries, which were prepared by diluting 2 g of the dentifrice in 15 ml of distilled water. A toothbrush with bristles of medium hardness was applied to the dentin surface at an inclination of about 90° under a constant loading (200 g) using a speed of 250 cycles/min for 2 minutes with a V-8 cross brushing machine (Sabri Dental Enterprise, Inc., USA). Teeth were brushed with the tested toothpaste twice a day for ten days. To remove excess slurry or aqueous solution, teeth were rinsed using distilled water for 10s. During the brushing procedure, teeth were immersed in artificial saliva except for when being brushed with the brushing machine. After the brushing procedure, two teeth from group B were confirmed for the tubular occlusion using a scanning electron microscope. Both groups A and B were then divided into three groups (n=8 per group) for resin composite build-up using different adhesive agents as follows:

OptiBond FL® (Kerr, Orange, CA, USA): 37.5% phosphoric acid etching gel was applied onto the prepared dentin and allowed to react for 15s, then the specimens were rinsed thoroughly with water and dried with foam pellets. OptiBond FL® primer was applied with a light scrubbing motion for 15s and gently air-dried for 5s until there was no visible movement of liquid. OptiBond FL® adhesive was then applied uniformly creating a thin coating for 15s, then light cured for 20s using a LED light curing unit (Demi Plus, Kerr Corporation, Orange, CA, USA) with 1,100 mW/cm^2 intensity.

Clearfil SE Bond® (Kuraray Medical Inc, Japan) : primer was applied with rubbing motion for 20s, then dried with mild airflow for 10s. After that, adhesive was applied and light cured for 20s.

Single Bond Universal® (3M ESPE, USA) adhesive was applied to the prepared tooth with a rubbing motion for 20s, then gently air dried for approximately 5s to evaporate the solvent and light cured for 20s.

After bonding procedures, a silicone mold with a 14 x 8 x 4 mm^3 opening at the center was placed on the treated dentin. Resin composite (Premise, Kerr, USA) was built up incrementally to 4 mm in height, 2 mm for each layer, onto the treated dentin. Each increment was light-cured with an LED light curing unit (Demi Plus, Kerr Corporation, Orange, CA, USA) with 1,100 mW/cm^2 intensity for 40s from the top, with a light tip held perpendicularly and within 1 mm superior to resin composite. Light output from the light-polymerizing unit was checked using a radiometer (Model 100 Optilux, Kerr, Orange, CA, USA) throughout the experiment.

All samples were stored in water at 37°c for 24 hours, and then mounted onto a low-speed sectioning machine (ISOMET 1000TM, Buehler, USA), which were subsequently sectioned in order to obtain stick-shaped microtensile specimens. Eight sticks from the middle of the dentin portion were selected from each tooth. Every stick was examined using a stereo microscope at 40X to ensure its homogeneity, without bubbles or cracks, and also to verify the exact dimension. All samples were stored in water at 37°c for 24h. Subsequently, the sectioned sticks in the same tooth were divided into two subgroups: 1) microtensile bond strength test and 2) thermocycling for 10,000 cycles between 5°c and 55°c for 30s at each temperature. All stick specimens were attached to the test apparatus using a cyanoacrylate adhesive (Model Repair II Blue, Dentsply-Sankin, Japan) and subjected to microtensile bond strength testing using a universal testing machine (EZ-S; Shimadzu Corporation, Kyoto, Japan) at a cross-head speed of 0.5 mm/min until the bond ruptured. The microtensile bond strength of each specimen was calculated as the ratio of maximum load force at the fracture and cross- sectional bonding area, which was measured in each individual fractured specimen. Specimens with pretest failure were calculated as mean between 0 MPa and the lowest measured value in the specific experimental group.[21] Fracture mode analysis of the bonded dentin surface was performed using a stereo microscope at 40X magnification. Failure mode were classified as follows:

• Adhesive failure: fracture occurred in the adhesive layer, or where adhesive remained on the dentin surface or the resin composite. (>75 % of failure between resin/dentin interface)

• Cohesive failure:

Cohesive failure in dentin: >75 % of fracture or failure occurred within dentin

Cohesive failure in restoration: >75 % of fracture or failure occurred within the resin composite.

• Mixed failure: failure at resin/dentin interface that included cohesive failure of the neighboring substrates.

All data of microtensile bond strength was analyzed statistically using a two-way ANOVA, a Tukey's (HSD) test and a paired sample *t*-test, with significance set at $p<0.05$. All statistical analyses were performed using SPSS 20.0 software (SPSS Inc., Chicago, IL, USA).

Abbreviation : FL= OptiBond FL® non brush group, FLT= OptiBond FL® non brush group with 10,000-cycle thermocycling, FLB= OptiBond FL® brush group, FLBT= OptiBond FL® brush group with 10,000-cycle thermocycling, SE= Clearfil SE Bond® non brush group, SET= Clearfil SE Bond® non brush group with 10,000-cycle thermocycling, SEB= Clearfil SE Bond® brush group, SEBT: Clearfil SE Bond® brush group with 10,000-cycle thermocycling, SU= Single Bond Universal® non brush group, SUT= Single Bond Universal® non brush group with 10,000-cycle thermocycling, SUB= Single Bond Universal® brush group, SUBT= Single Bond Universal® brush group with 10,000-cycle thermocycling.

Results

The µTBS values of all experimental groups were normally distributed ($p>0.05$). Mean µTBS values and standard deviations of both the brush and the non brush groups in each adhesive system at 24 hours and after 10,000 cycles of thermocycling are summarized in Table 1. Two-way ANOVA revealed that µTBS values of each of the adhesive system were not significantly affected by brushing with desensitizing toothpaste containing calcium sodium phosphosilicate at both 24-hour water storage ($p=0.857$) and 10,000-cycle thermocycling ($p=0.787$). On the other hand, after brushing with desensitizing toothpaste containing calcium sodium phosphosilicate, some types of adhesive had a statistically significant effect on µTBS values ($p<0.001$) as shown in Table 2 and 3. According to Tukey's (HSD) test, OptiBond FL® brush groups (FLB,FLBT) gave significant higher µTBS values than Clearfil SE bond® brush groups (SEB,SEBT) ($p=0.036$, $p<0.001$) and Single Bond Universal®

brush groups (SUB,SUBT) (p=0.011, p<0.001). Although SEB showed no significant difference in μTBS values to SUB group (p=0.853) (Table 4), SEBT had statistically significant higher μTBS values than SUBT group (p=0.038) (Table 5). In addition, when focused on the thermocycling effect, 10,000-cycle thermocycling did not significantly affect the μTBS values in OptiBond FL® groups (p=0.06, p=0.284) and Clearfil SE bond® groups (p=0.168 , p=0.414) in both the brush group and the non brush group. In contrast, it significantly affected the μTBS values in Single Bond Universal® groups (p=0.043 , p=0.006). Failure modes are given by group in Figure 1. Adhesive failure was noticed to be a major finding in all testing groups. No pre-test failure was recorded for any other adhesives tested.

SEM images of dentin at 10000x magnification were shown in Figure 2. Picture (a) showed opened dentinal tubule after immersion in a 1% citric acid. Picture (b) showed dentinal tubules occluded with deposits after brushing with desensitizing toothpaste containing calcium sodium phosphosilicate for 10,000 cycles with V-8 cross brushing machine. All arrows indicated dentinal tubules.

Table 1 *μTBS values of brush and non brush groups in 24-hour water storage and 10,000-cycle thermocycling (means ± standard deviations (MPa) of the different experimental groups*

GROUP	24-HOUR	10,000-CYCLE THERMOCYCLING (T)
FL	$33.05 \pm 5.37^{A,1}$	$32.08 \pm 4.97^{a,1}$
FLB	$34.58 \pm 4.36^{A,1}$	$32.04 \pm 2.52^{a,1}$
SE	$28.36 \pm 3.20^{B,1}$	$27.69 \pm 4.75^{b,1}$
SEB	$30.23 \pm 3.43^{B,C,1}$	$28.94 \pm 3.96^{b,1}$
SU	$26.57 \pm 3.99^{D,1}$	$21.66 \pm 5.59^{c,2}$
SUB	$27.39 \pm 4.63^{C,D,1}$	$23.60 \pm 1.88^{c,2}$

** Similar superscripts capital letters indicate no significant differences between groups at 24-hr (left columns), similar superscript lowercase letters indicate no significant differences between groups after 10,000-cycle thermocycling (right columns), and similar superscript numbers indicate no significant differences between adhesive systems within each group (rows) according to Tukey's (HSD) test (p>0.05)*

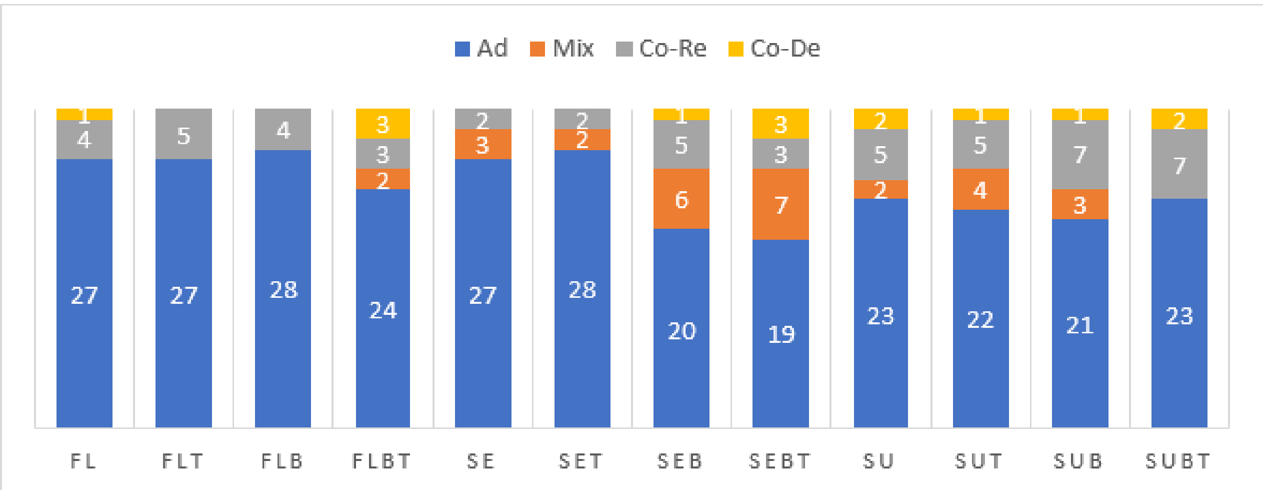

Figure 1 *Failure modes of three types of adhesive systems bonded to dentin at 24-hour water storage and after 10,000-cycle of thermocycling (T)*

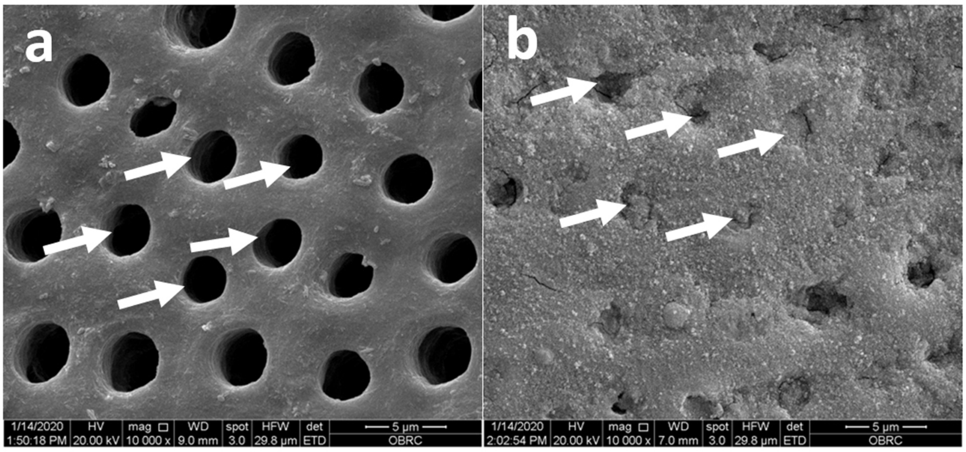

Figure 2 *Representative SEM micrograph of dentinal tubule (a) after immerse in 1% citric acid and (b) after brushing with Sensodyne Repair & Protect® 10,000 cycles with V-8 cross brushing machine (Sabri Dental Enterprise, Inc., USA) (b) (10000x magnification)*

Discussion

This study evaluated the effect of a desensitizing toothpaste containing calcium sodium phosphosilicate on the microtensile bond strength of adhesive systems treated to dentine. This desensitizing toothpaste released sodium, calcium, and phosphate ions, which consequently interacted with oral fluids and formed a crystalline hydroxycarbonate apatite (HCA) layer, chemically and structurally similar to natural tooth mineral.[15] A previous study revealed that the mineral deposits formed by calcium sodium phosphosilicate desensitizing toothpastes were unstable[22] and not strong enough to affect the formation of the hybrid layer resulting in no interference on bond strength.[23] This was in agreement with the present study that showed there was no significant difference in the microtensile bond strength of adhesive systems treated to dentin between the groups using desensitizing toothpaste containing calcium sodium phosphosilicate compared to non brush group in each adhesive system. It could be explained that phosphoric acid from the OptiBond FL® bonding system probably dissolved the calcium phosphate deposits covering the dentin leading to a reopening of tubules allowing infiltration of resin monomers, favoring the bonding.[22] However, the other two adhesive systems in this study are Clearfil SE bond® and Single Bond Universal®, which were less acidic when compared to phosphoric acid, the mineral deposits formed on the dentin surface were not able to act as a physical barrier and compromised dentin hybridization. In agreement with the study by Aguiar *et al.,* which showed that prolonged use of a desensitizing toothpaste with 5% calcium sodium phosphosilicate had no influence on bond strength of a self-etching adhesive system to dentin.[20]

Besides, desensitizing toothpaste containing calcium sodium phosphosilicate had no influence on the 24-hour microtensile bond strength test results of OptiBond FL®, Clearfil SE bond® and Single Bond Universal®. Meanwhile, there was a significant decrease in microtensile bond strength of Single Bond Universal® after 10,000-cycle thermocycling in both brush and non brush groups in this study. Due to different compositions, universal bonding contained mixtures of hydrophilic and hydrophobic components within the same solution which exhibited residual solvents entrapped in the adhesive layer and might increase the permeability of the adhesive layer after polymerization leading to compromised long-term performance.[24] In addition, it was found that MDP chemically bonds to hydroxyapatite resulting in the formation of MDP-Ca salt which contribute to better bond stabiliy.[25] Although Clearfil SE Bond® and Single Bond Universal® contain MDP as a functional monomer, they have different concentrations.

It has been reported that the purity of MDP and its concentration in the adhesive had an influence on the bonding potential.[26,27] A previous study reported that a higher purity MDP is used in Clearfil SE Bond®.[27] According to a study by Yoshida *et al.,* Clearfil SE Bond®, containing MDP in both primer and adhesive, showed more MDP-Ca salt formation than Single Bond Universal® because of the higher concentration of MDP. As a previous study revealed that the higher the concentration of MDP, the more nano-layering intensity was found.[25] Moreover, Single Bond Universal® composed of polyalkenoic-acid copolymer which have been reported to interfere with nano-layering as it competed to react with the same calcium ion depleted from hydroxyapatite as 10-MDP.[25,28] In addition, Nano-layering was discovered not only within the hybrid layer but also extending into the adhesive layer in Clearfil SE Bond®. In Single Bond Universal®, it was found particularly near the dentinal tubule.[25]

Moreover, a SEM image of dentin showed dentinal tubules occluded with deposits after brushing with desensitizing toothpaste containing calcium sodium phosphosilicate for 10,000 cycles. However, compositional analysis was not done to identify the nature of minerals occluded in tubules. A previous study revealed that EDX analysis of teeth brushed with calcium sodium phosphosilicate toothpaste showed high amounts of calcium, phosphate and a small amount of silica and titanium at dentine surfaces and tubules.[8,14] A study by Li *et al.* also reported that the formation of calcium phosphate as well as calcium fluoride could occur in fluoridated toothpaste. In addition, the abrasive component in toothpastes may help to form smear layers, which varied widely in composition, and was composed mainly of toothpaste abrasives, on dentin after brushing.[29]

In this present investigation, the microtensile bond strength test was used because the small-sized bonding area could reduce the probability of sample internal defects and provide a more homogenous distribution of stress during loading, therefore, fewer cohesive failures in substrates occured.[30,31] As seen in this study, most failure modes were observed at the adhesive interface which indicated that the value measured when cracked specimen represented a more reliable microtensile strength in nature.[32,33] Moreover, thermocycling was performed at 10,000 cycles in this study to simulate approximately one year of clinically oral function.[34] A study by Ozcan *et al.* found that this method was appropriate in inducing degradation of the composite resin restorations compared to other aging methods. Therefore, it represented a more challenging condition for the material tested.[35] Furthermore, no pretest failure was found. There are some limitations of this *in vitro* study that could not be inferred totally to clinical situations. Moreover, this study investigated particular brands of toothpaste and adhesive systems, so the results of this study might not be inferred to other products.

Conclusion

Desensitizing toothpaste containing calcium sodium phosphosilicate had no effect on OptiBond FL® and Clearfil SE bond® in both immediate microtensile bond strength and after aging by 10,000-cycle thermocycling. Meanwhile, the microtensile bond strength of Single Bond universal® adhesive decreased after 10,000-cycle thermocycling. From the perspective of the clinician, this present study shows that OptiBond FL® and Clearfil SE bond® may be suitable to use as an adhesive system to bond resin composite to dentin in teeth brushed with calcium sodium phosphosilicate toothpaste, however, a study concerning the long-term performance of Single Bond universal® is warranted.

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