

Effect of desensitizing toothpaste on microtensile bond strength between resin composite and dentin

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Abstract

Objective To evaluate and compare the effects of two desensitizing toothpastes and a regular fluoride toothpaste on microtensile bond strength of two adhesive agents to dentin.

Materials and methods The labial surfaces of forty bovine incisor crowns were ground flat, exposing dentin. The teeth were then randomly divided into four groups corresponding to the toothpaste used: 1) Sensodyne[®] Rapid Relief (GlaxoSmithKline, UK), 2) Colgate Sensitive Pro-ReliefTM (Colgate– Palmolive, Thailand), 3) Colgate[®] Regular Flavor (Colgate–Palmolive, Thailand), and 4) immersed in artificial saliva (control). Each tooth in groups 1–3 was brushed with its respective dentifrice under constant loading (200 g) at 250 strokes/min for 2 minutes, twice daily for three days. Each group was then randomly divided for composite build–up using the following adhesive agents: 1) Optibond[®] XTR (Kerr, USA), or 2) Optibond[®] FL (Kerr, USA). After curing the adhesives, a light–cured resin composite (PremiseTM, Kerr, USA) was used for a core build–up. The samples were sectioned into four specimens with 1 ± 0.1 mm thick and wide. The microtensile bond strength test was performed using a universal testing machine at a cross–head speed of 0.5 mm/min. The data were analyzed using two–way ANOVA and Tukey's multiple comparison tests with significance set at *p* < 0.05. Fracture analysis of the debonded dentin surface was performed using a stereomicroscope.

Results Bond strength was statistically significantly reduced by the application of desensitizing toothpastes (p < 0.0001), and the type of adhesive agents had a significant effect on bond strength (p < 0.0001).

Conclusion The uses of desensitizing toothpaste reduce bond strength of adhesives to dentin.

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Key words: adhesive system; arginine; desensitizing toothpaste; microtensile bond strength; strontium acetate

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Introduction

The causes of presenting symptoms of sensitive teeth are multi-factorial. Diagnosis of tooth sensitivity can range from an abscessed or cracked tooth, diet sensitivity, medication sensitivity, restorative sensitivity, bleaching sensitivity, to dental decay or some form of hypersensitivity. Dentin hypersensitivity is defined by brief, sharp, well-localized pain in response to thermal, evaporative, tactile, osmotic, or chemical stimuli that cannot be ascribed to any other form of dental defect or pathology. There are many varieties of potential causes for dentin sensitivity. The loss of enamel and removal of cementum from the root with exposure of dentin is a major contributing factor. The loss of enamel may be a consequence of attrition, erosion, abrasion, and abfraction.¹ Dentin hypersensitivity can reduce the quality of life of those who suffer from it, because it can affect eating, drinking, and breathing habits. The most widely accepted explanation for dentin hypersensitivity is the hydrodynamic theory,² which states that the movement of fluids or semi-fluid materials in the dentinal tubules transmits peripheral stimuli that activate the sensory nerves in the pulp, causing sharp short pain. Dentin hypersensitivity can be treated by either 1) in-office treatment, which uses professional products (e.g. topical fluoride, oxalate salts, glutaraldehyde, desensitizing prophylaxis paste) or surgical intervention (e.g. restoration, periodontal soft tissue grafting) or 2) self-applied treatment with professionally dispensed products (e.g. casein phosphopeptide- amorphous calcium phosphate paste) or over-the-counter products (e.g. desensitizing toothpaste).³ Dentin hypersensitivity can be alleviated either by interfering with neural transmission or by occluding the dentin tubules.² One of the most common ingredients used to treat dentin hypersensitivity is potassium nitrate. The potassium ions are thought to increase the nerve depolarization threshold, thereby reducing the sensation of pain.²

Treatments which physically plug opened dentinal tubules have the potential to be more effective than potassium-based treatments.⁴ One treatment to occlude the tubules used high concentration fluoride gels or pastes. The high level of fluoride interacted with calcium in the saliva or on the tooth surface, and calcium fluoride precipitated within the tubules and occluded them.¹ Clinical studies have been performed to evaluate the effectiveness of treating dentin hyper-sensitivity with various fluoride products.⁵ Although these agents reduced dentin hypersensitivity, they were found to decrease the bond strength between composite resin and dentin.^{6–7} This was due to the precipitation of microcrystals and mineral in the dentinal tubules, preventing proper resin infiltration.

Oxalate materials also have been used successfully for desensitization.⁸ These materials react with calcium ions on dentin and in dentinal fluid to form insoluble calcium oxalate crystals. Calcium oxalate crystals occluded open tubules in dentin.⁸ "Oxa-Gel" (Art-Dent, Brazil) is a product that contains monohydrogenmonopotassium oxalate. However, a previous study indicated that adhesive resins did not bond well to oxalate-treated dentin, because the dentin surface, including tubule orifices, was covered with calcium oxalate crystals.⁹ Thus, using a topical desensitizing agent, prior to tooth restoration using composite resin and a bonding agent, may result in a reduction in bond strength between dentin and the restorative material.

Dentifrices are the most common vehicles for desensitizing agents. They widely indicated because of their low cost, ease of use and home application. Active ingredients in desensitizing toothpaste include 2% potassium salt, 8% arginine and calcium carbonate, and 8% strontium acetate dentifrice. Kleinberg and colleagues developed a dentin hypersensitivity treatment consisting of 8% arginine (an amino acid found in saliva), bicarbonate, and calcium carbonate. This desensitizing formulation mimicked saliva's natural ability to plug and seal open dentin tubules.¹⁹ This formulation has been further developed as a daily-use dentin hypersensitivity dentifrice (Colgate Sensitive Pro-ReliefTM Colgate–Palmolive, Thailand). In addition to 8% arginine and calcium carbonate, the dentifrice also contains 1450 ppm fluoride and claims to protect against the development of caries. Both *in vitro* and clinical studies have reported the efficacy of this dentifrice in reducing dentin hypersensitivity.^{11–14} High resolution scanning electron microscopy images revealed that the arginine–calcium carbonate desensitizing paste provided complete occlusion of open dentinal tubules, and freeze–fracture SEM images demonstrated that the plug reached a depth of two microns into the tubule.¹²

Strontium-based dentifrices (10% strontium chloride) have been widely used in treating dentin hypersensitivity, and are believed to work by occluding dentinal tubules.¹⁵ Researchers have found that strontium acetate is more versatile than strontium chloride, can be formulated as a dentifrice base with almost no organoleptic downside, and was shown to successfully combine with sodium fluoride.¹⁵ A dentifrice containing 8% strontium acetate and 1040 ppm sodium fluoride was developed and has been extensively tested in vitro, in situ, and clinically.^{16–20} This technology is available as a daily-use dentin hypersensitivity dentifrice (Sensodyne[®] Rapid Relief, GlaxoSmithKline, UK). A study demonstrated that a single application of an 8% strontium acetate/1040 ppm sodium fluoride formulation occluded open dentinal tubules with a strontium-silica plug deep within the dentinal tubules, and this occlusion was resistant to dietary acids.¹⁹

Dental adhesives are used for several clinical applications and they can be classified based on the clinical regimen in "etch-and-rinse adhesives" and "self-etch adhesives". In the present study, we chose Optibond[®] FL, which has been extensively studied and widely used clinically, to represent etch and rinse adhesives and Optibond[®] XTR to represent self-etch

adhesives. Optibond[®] FL has had long-term clinical track,²¹⁻²² and has been considered to be the gold standard for adhesives.²³ Optibond[®] XTR is a simplified version from the same manufacturer utilizing a functional monomer similar to that of Optibond[®] FL, glycerol phosphate dimethacrylate, which is a phosphate monomer that has been used for bonding to dentin for over 50 years.²⁴

Therefore, we hypothesized that because the dentifrice containing 8% arginine and the dentifrice containing 8% strontium acetate function by occluding dentinal tubules, their use might affect the bond strength between dentin and bonding agents, as has been seen with other desensitizing agents. The aim of this study was to evaluate and compare the effect of these two desensitizing toothpastes and regular fluoride toothpaste on the microtensile bond strength of two different adhesives to dentin.

Materials and methods

Forty extracted bovine incisors were collected, cleaned, and stored in 0.01% thymol solution for 1 week, and then stored in distilled water at 4°C for a maximum of 1 month before use. The roots were removed at 1 mm below the cemento-enamel junction (Fig. 1A), and pulpal tissue was carefully removed. The teeth were then embedded in self-curing resin with their labial surfaces exposed with their surface parallel to the horizontal plane (Fig. 1B). The labial surfaces were ground flat using a polishing machine (NANO 2000 Grinder-polisher, Pace Technologies, USA) with 320, 600, and 1200 grit silicon carbide paper under running water until the enamel was completely removed (Fig. 1C) with an exposed dentin surface area of at least 6 x 3 mm². Labial surface of each tooth was carefully inspected using a stereomicroscope (ML 9300; Meiji Techno Co. Ltd., Japan) at 40X magnification to ensure that it was free of enamel. The teeth were equally randomly assigned into four groups (n=10) according to the type of toothpaste: Group 1-Sensodyne[®] Rapid Relief, Group 2-Colgate[®] Sensitive Pro-ReliefTM, Group 3-Colgate[®] Regular Flavor (Colgate-Palmolive, Thailand), and Group 4-no treatment (Fig. 1). The compositions of the desensitizing toothpastes used in the present study are summarized in Table 1. Respective dentifrice slurry, which was prepared by diluting 2 g of the dentifrice in 6 ml of distilled water, was placed on the exposed dentin surface of each tooth from groups 1-3. A toothbrush with bristles of medium hardness was applied perpendicular to the dentin surface under a constant loading (200 g) for 250 strokes/min in 2 minutes using a V-8 cross brushing machine (Sabri Dental Enterprise, Inc., USA). The teeth were brushed with the toothpastes twice a day (9.00 AM and 5.00 PM) for three days. To remove excess slurry or aqueous solution, the teeth were rinsed in distilled water

for 10 sec. During the three day brushing procedure at the time when the teeth were not being brushed by the brushing machine, the teeth were immersed in artificial saliva. After the brushing process, each group was further randomly assigned into two groups (n=5) (Fig. 1) for composite build up, one being bonded using Optibond[®] XTR and the other using Optibond[®] FL (Kerr, USA) (Table 2).

After applying the adhesive on the dentin surface according to the manufacturers' instructions, a silicone mold with a $14 \times 8 \times 4 \text{ mm}^3$ opening in the middle was placed on the dentifice treated dentin. Light-cured composite (PremiseTM, Kerr, USA) was used to filled up the mold 4 mm in height on the treated dentin surface by incremental placement. Each 2 mm increment was polymerized for 40 s using a visible light-polymerization unit (EliparTriLight Curing Light, 3M ESPE,



Fig. 1 Specimen preparation and the experimental design (XTR = treated with Optibond[®] XTR, FL = treated with Optibond[®] FL).



Fig. 2 Cut specimens from the top view. Only four sticks in the middle were used.

USA) and then storing in distilled water at 37°C for 24 h. Each specimen was mounted on a low speed cutting machine (ISOMET 1000[®], Buehler, USA) and sectioned perpendicular to the surface both mesialdistally and inciso-cervically direction in order to obtain stick-shaped microtensile specimens. Only four sticks in the middle were used for the test (Fig 2). Dimensions of each specimen was measured using a digital vernier caliper (Mitutoyo Co., Japan) approximately 1.0 mm² (1 ± 0.1 mm x 1 ± 0.1 mm). Each test specimen surface was carefully examined under a stereomicroscope at 40X to ensure that it was homogeneous without bubbles or cracks. The specimens were subsequently attached to the tensile testing apparatus mounted in a universal testing machine (EZ-S, Shimadzu, Japan) with a cyanoacrylate adhesive (Model Repair II Blue, Dentsply-Sankin, Japan), and stressed to failure at a cross-head speed of 0.5 mm/min. The microtensile bond strength of each specimen was calculated as the ratio of the maximum load force at fracture divided by the cross-sectional bonding area of each individual fractured specimen.

Analysis the mode of failure of the composite bonded dentin surfaces was performed using a

stereomicroscope at a 40X magnification. Failures were classified as adhesive (> 75% of failure was between the tooth and the restorative material), cohesive (> 75% of the failure was within the restorative material or dentin) or a mixture of the two.^{25–26} Specimens with pre-test failure were excluded from the study.

Randomly selected samples with adhesive fractures were processed for scanning electron microscopy (JSM-5410LV, JEOL, Japan) using standard SEM specimen processing techniques; i.e. fixed in a 2.5% glutaraldehyde cacodylate buffer solution, dehydrated in graded ethanols, chemically dried using hexamethyl-disilazane, and gold-sputter coated.

The microtensile bond strength data were statistically analyzed using two-way ANOVA to examine the effect of the factors (type of toothpaste and type of adhesive agent). As there were significant interactions between these factors, the data were further analyzed with one-way ANOVA followed by Tukey's post-hoc comparison test, with significance set at p < 0.05. All statistical analyses were performed using SPSS statistics for windows version 17.0 (Chicago: SPSS Inc.).

Toothpaste	Manufacturer	Composition		
Sensodyne®	GlaxoSmithKline Ltd.,	Aqua, Sorbitol, Hydrated Silica, Glycerin, Strontium Acetate		
Rapid Relief	UK	hemihydrate (8%wt), Sodium Methyl Cocoyl Taurate, Xanthan		
		Gum, Titanium Dioxide, Aroma, Sodium Saccharin, Sodium		
		Fluoride, Sodium Propylparaben, Sodium Methylparaben,		
		Limonene, Sodium Fluoride 0.23% w/w (1040 ppm F)		
Colgate®	Colgate-Palmolive,	Calcium Carbonate, Water, Sorbitol, Arginine Bicarbonate		
Sensitive	Thailand	(8%wt), Hydrated Silica, Sodium Lauryl Sulfate, Flavor,		
Pro-Relief TM		Cellulose Gum, Sodium Monofluorophosphate (1450 ppm		
		F), Sodium Bicarbonate, Tetrasodium Pyrophosphate, Benzyl		
_		Alcohol, Sodium Saccharin, Xanthan Gum		
Colgate®	Colgate-Palmolive,	Dicalcium phosphate dihydrate, water, glycerin, sorbitol, sodium		
Regular Flavor	Thailand	lauryl sulfate, cellulose gum, flavor, tetrapotassium pyrophosphate,		
		sodium saccharin, Sodium monofluorophosphate (1000 ppm F)		

 Table 1 Composition of tested toothpastes⁴⁷

Results

Table 3 summarizes the mean microtensile bond strength values and standard deviations of the test and control groups. Two-way ANOVA indicated that the type of toothpaste (p < 0.0001), the type of adhesive agent (p < 0.0001) and their interaction (p < 0.05) had a significant effect on microtensile bond strength. The microtensile bond strengths in Colgate[®] Regular Flavor (group 3) and the control groups (group 4) were significantly higher than in Sensodyne[®] Rapid Relief (group 1) and Colgate[®] Sensitive Pro-ReliefTM group (group 2) (p < 0.05). However, there was no significant difference in bond strength between groups 1 and 2 (p = 0.760) and groups 3 and 4 (p = 0.104). There were significant differences in bond strength between adhesive agents in group 1 (p < 0.05) and 2 (p < 0.001), but no significant differences were found in group 3 (p = 0.859) and 4 (p = 0.879). Premature failures occurred in group $1 + \text{Optibond}^{\text{(B)}} \text{ XTR } (n = 2)$ and

group 2 + Optibond[®] XTR (n = 1). The distribution of failure modes is presented in Table 4. The bond failure type in each group was predominantly adhesive (83% or higher of total number of specimens), with the remainder exhibiting cohesive failures, and no mixed failures.

Figures 3 and 4 show representative SEM images of the debonded dentin specimens. It was possible to observe partial obstruction of dentinal tubules in specimens of group 1 + Optibond[®] XTR (Fig. 3a) and group 2 + Optibond[®] XTR (Fig. 3b).

Discussion

The hydrodynamic theory has been widely accepted as the principal mechanism for dentin hypersensitivity. Based on this theory, a substance occluding the dentinal tubules can cause a decrease in dentinal fluid flow, thereby reducing the clinical symp-

Adhesive agent	Composition	Manufacturer's instructions Apply Primer to the dentin surface using a disposable applicator brush, scrub the surface using a brushing motion for 20 s, apply adhesive to the dentin surface using light brushing motion for 1 s, air thin for 5 s, light cure with visible light- polymerization unit for 10 s. Place etchant 37.5% phosphoric acid on dentifi- surface for 15 s, rinse with water until etchan has been completely removed, gently air dry fo 5 s, apply primer over dentin surface with a light agitating motion for 15 s, gently air dry fo approximately 5 s, apply adhesive over dentifi- surface with uniformly creating a thin coating air thin for 5 s, light cure with visible light- polymerization unit for 20 s.		
Optibond [®] XTR (Kerr, USA) (self-etch)	Primer: GPDM, hydrophilic co-monomers, water, ethanol, acetone Adhesive: resin monomers, HEMA, inorganic fillers, ethanol			
Optibond [®] FL (Kerr, USA) (etch-and-rinse)	Etchant: 37% phosphoric acid Primer: HEMA, GPDM, ethanol, water, PAMA, camphorquinone Adhesive: BisGMA, HEMA, GDMA, camphorquinone, fumed SiO_2 , barium aluminoborosilicate, Na_2SiF_6 , coupling factor A174			

Table 2 Adhesive agents, composition and the manufacture's instructions⁴⁸⁻⁴⁹

GDMP	=	glycerol phosphate dimethacrylate
BHT	=	2,6-di-(tert-butyl)-4-methylphenol
PAMA	=	phtailic acid monomethacrylate
BisGMA	=	bis-phenol-A-bis-(2-hydroxy-3-methacryloxypropyl) ether

GDMA = glycerol dimethacrylate

toms of dentin hypersensitivity.²⁷ Previous studies have demonstrated that using 8% arginine 8% strontium acetate toothpaste resulted in significant tubule occlusion compared with the negative control.^{12,28-31} However, the use of these dentifrices may alter the microtensile bond strengths of adhesives to dentin.

The results of the present study revealed that both 8% arginine and 8% strontium toothpaste significantly reduced the microtensile bond strengths of adhesives to dentin. This may be because these two desensitizing toothpastes occluded dentinal tubules, and made dentin more resistant to acid challenge.^{12,28–30} *In vitro* and

in situ studies have demonstrated that following acid challenge (grapefruit juice^{29–30}, Coca–Cola^{®12}, and citric acid³²), dentin samples treated with 8% arginine or 8% strontium toothpaste had significantly more occluded dentin tubules than the negative control.^{12,28–30,32} These desensitizing toothpastes formed occluding layers that were resistant to acid challenge.^{12,28–30} Therefore, these layers may be resistant to acid etching used in bonding procedures, and may chemically and physically prevent complete penetration of the bonding agents. We found that the desensitizing paste groups bonded with Optibond[®] XTR (pH = 1.6–2.4), which

	Sensodyne®	Colgate®	Colgate [®] Regular		
Adhesive agent	Rapid Relief Sensitive		Flavor	None	
		$\mathbf{Pro-Relief}^{\mathrm{TM}}$			
Optibond [®] XTR	27.95 ^{Aa} ± 4.91	$28.37^{AC} \pm 5.43$	$41.88^{Be} \pm 3.45$	$43.86^{Bf} \pm 2.75$	
Optibolia ATK	(2)	(1)	(0)	(0)	
	$32.73^{\text{Cb}} \pm 3.20$	34.12 ^{Cd} ± 3.76	$43.56^{\text{De}} \pm 3.73$	45.49 ^{Df} ± 2.58	
Optibond [®] FL	(0)	(0)	(0)	(0)	

 Table 3
 Means ± standard deviations of microtensile bond strength values (MPa) between dentin surface and resin composite and numbers of pre-testing failures in brackets

Means followed by the same superscript capital letters in the same row or lowercase letters in the same column indicates no statistical difference (p > 0.05)

Table 4 The fracture modes of the experimental groups

Desensitizing		Adhesive	Cohesive	Mixed	
toothpaste	Adhesive agents	failure	failure	failure	
Sensodyne®	Optibond [®] XTR $(n = 18)$	15 (83.33%)	3 (16.67%)	0	
Rapid Relief	Optibond [®] FL $(n = 20)$	20 (100%)	0	0	
Colgate [®]	Optibond [®] XTR (n=19)	16 (84.21%)	3 (15.79%)	0	
Sensitive					
Pro-Relief TM	Optibond [®] FL $(n = 20)$	17 (85%)	3 (15%)	0	
Colgate [®] Regular	Optibond [®] XTR (n = 20)	17 (85%)	3 (15%)	0	
Flavor	Optibond [®] FL $(n = 20)$	20 (100%)	0	0	
None	Optibond [®] XTR (n = 20)	17 (85%)	3 (15%)	0	
none	Optibond [®] FL (n = 20)	18 (90%)	2 (10%)	0	



Fig. 3 Scanning electron micrographs (x 3,500) of debonded specimens treated with Optibond[®] XTR : a) group 1–Sensodyne[®] Rapid Relief, b) group 2–Colgate[®] Sensitive Pro–ReliefTM, c) group 3–Colgate[®] Regular Flavor, and d) group 4–Negative control (no toothpaste). Arrows indicate tubular partial obstruction and arrowheads show partially demineralized intertubular dentin. (id: intertubular dentin)

was less acidic than Optibond[®] FL (pH = 1.8), demonstrated a significantly lower mean microtensile bond strength than the Optibond[®] FL groups. An etchant with lower acidity may result in less tubular penetration of the bonding agent, resulting in lower microtensile bond strength.

In contrast to the present study, a previous study indicated that Colgate Sensitive Pro-ReliefTM desensitizing paste did not have a significant effect on the shear bond strength of the composites tested to enamel.33 Similarly, a previous study by Canares, *et al.* reported that 8% arginine desensitizing toothpaste had no effect on the bond strength of composites bonded to dentin.³⁴ The differences in findings between these *in vitro* studies and the present study may be due to differences in study design. The formers only applied the desensitizing paste once and did not immerse the samples in artificial saliva to simulate the oral environment, therefore, the environment and application methods of these studies may not be sufficient for the precipitation process to occur, generating results dissimilar to those of the present study.

SEM analysis of our samples demonstrated partial obstruction of the dentinal tubules in group 1 using Optibond[®] XTR and group 2 using Optibond[®] XTR. The groups with blocked tubules also had significantly lower bond strength compared to the other groups suggesting that tubule occlusion was responsible, at least in part, for decreasing bond strength. However, further compositional analysis may be needed to determine exactly what is obstructing the dentinal tubules of these specimens. Petrou, et al. treated dentin specimens with an 8% arginine desensitizing paste, which occluded the dentinal tubules, and analyzed them by electron spectroscopy for chemical analysis,¹² finding that calcium, oxygen, and phosphorus levels were significantly increased. Carbonate compound was also detected on the treated dentin surface. They concluded



Fig. 4 Scanning electron micrographs (x 3,500) of debonded specimens treated with Optibond[®] FL : a) group 1– Sensodyne[®] Rapid Relief, b) group 2–Colgate[®] Sensitive Pro–ReliefTM, c) group 3–Colgate[®] Regular Flavor, and d) group 4–Negative control (no toothpaste). Arrowheads indicate partially demineralized intertubular dentin. (id: intertubular dentin)

that the treated dentin surfaces had been remineralized, and that calcium carbonate was simultaneously deposited on the dentin surface.¹² Earl, *et al.* analyzed dentin specimens treated with an 8% strontium acetate denti– frice using energy dispersive x-ray spectroscopy (EDX). EDX analysis indicated the presence of strontium within the dentin tubules.²⁰

Both Sensodyne[®] Rapid Relief and Colgate[®] Sensitive Pro-ReliefTM were able to plug dentinal tubules through an interaction between their respective active ingredients, abrasive agents, and the dentin itself.^{11,29} However, there were some differences between these two desensitizing pastes. Arginine was absorbed onto the surface of calcium carbonate forming a positively charged alkaline agglomerate.¹² This alkaline agglomerate had a high affinity to dentin, and relied on the deposition of calcium and phosphate from saliva to occlude the dentin tubules. The presence of saliva was therefore essential for the mechanism of action of arginine.¹² However, strontium-based dentifrices function based on a different mechanism of action. Strontium is an alkaline earth metal, which has a strong inherent absorptive capacity to calcified tissues, especially those with a high organic content such as dentin.¹⁸ This may be because strontium permeated into dentin and adsorbed into or onto organic connective tissues, including odontoblast processes, as was shown in an earlier study using the metallic compound strontium chloride.¹⁸ Strontium has been shown to penetrate dentinal tubules, and is thought to occlude the tubules by substituting for calcium in hydroxyapatite.³⁰

In the present study, we chose Optibond[®] FL to represent etch and rinse adhesives and Optibond[®] XTR to represent self-etch adhesives. Optibond[®] FL has had long-term clinical track,35-36 and has been considered to be the gold standard for adhesives.³⁷ Optibond[®] XTR is a simplified version utilizing a functional monomer similar to that of Optibond[®] FL, glycerol phosphate dimethacrylate, which is a phosphate monomer that has been used for bonding to dentin for over 50 years.³⁸ Optibond[®] XTR is a two-step mild self-etch adhesive. However, it has only recently been intro-duced; therefore, there have been few studies on its dentin bonding strength. In the studies that have been published, Optibond[®] XTR did not demonstrate a lower bond strength compared with Optibond[®] FL.³⁷⁻³⁸ These findings are consistent with the results of the present study, where no significant difference was found between specimens bonded with Optibond[®] XTR and Optibond[®] FL in the regular fluoride toothpaste group or the negative control group.

The present study employed microtensile bond strength test to minimize the occurrence of dentin cohesive failure, which has been reported to occur in up to 80% of the specimens in conventional shear and tensile tests.³⁵ A characteristic feature common to all variations of the microtensile bond strength test method is the use of a relatively small cross-sectional surface area of 1 mm^2 or less. A smaller bonding area reduces the probability of sample internal defects and provides a more homogeneous distribution of stress during loading, thus minimizing the chance of dentin cohesive failure. The small size of the dentin/resin composite slabs allows for testing multiple specimens derived from the same tooth, which makes it necessary to treat the respective bond strength values as repeated measurements in the statistical analysis.³⁵

Tooth region and remaining dentin thickness may have an effect for bond strength. Superficial dentin at the dentin-enamel junction occupied by tubule lamina approximately 1% of total surface area, while that of dentin near the pulp is about 22%. Since tubule lamina area is occupied by dentinal fluid, these areas are also approximately equal to the tubular water content. This difference in intrinsic moisture has been deemed responsible for the differences in bond strengths between superficial and deep dentin.³⁶ Superficial dentin normally results in higher composite-dentin bond strengths than deep dentin. The orientation of the dentinal tubules may also influence dentin bond strengths. Adebayo et al. reported that shear bond strengths were affected by dentin depth, orientation of the tubule and the adhesive material, but not by location of dentin (occlusal or cervical).³⁷ On the contrary, Sattabanasuk et al. and Phrukkanon et al. reported that bond strengths were not affected by tubule orientation.³⁸⁻³⁹ There was also a study reported that there was no significant differences between the morphology of dentinal tubules between human and bovine tooth.⁴⁰ However, in order to reduce these problems, we only used four specimens in the middle of each tooth (Fig. 2), which have resemble dentin thickness and dentin tubule orientation.

Bovine teeth have been the most widely used substitute for human teeth in dental studies because they are easy to obtain in large quantities, in good condition and width a more uniform composition than that of human teeth. Moreover, bovine teeth have a relatively large flat surface, and do not have caries lesions and other defects.⁴¹ Schilke et al. reported no statistically significant differences in the number of dentin tubules per mm² or in the tubule diameter among coronal dentin layers of human deciduous or permanent molars, and coronal bovine incisors.⁴⁰ In addition, there are studies reported the use of bovine teeth as a substitute for human teeth in adhesion tests.⁴²⁻⁴³ They used microtensile bond strength tests to measure and compare bond strength of adhesive resins on human and bovine dentin. They all found no statistically significant differences between these hard tooth tissues. Therefore, it can be suggested that bovine teeth could be used to evaluate the adhesive materials or techniques before clinical implementation.

Our *in vitro* study was performed using extracted teeth without simulating dentinal fluid pressure, so it is difficult to compare the results with the clinical situation. When dentin is clinically exposed to the oral cavity, dentinal fluid flows from pulp to exposed dentin surface because of the interstitial fluid pressure in the pulp. Studies have reported that dentinal fluid flow affected the ingress of adhesive resins into the dentinal tubules.⁴⁴⁻⁴⁵ Therefore, the results of the present study should be confirmed by a clinical study.

To provide a scientific supported clinical recommendation, further study should be in human teeth, and in the future clinical investigation. Moreover, additional studies should include in-office treatments, for example, in-office desensitizing paste, which have also proven effective.⁴⁶

Conclusion

This study demonstrated the microtensile bond strength of bovine dentin specimens treated with 8% arginine and 8% strontium acetate desensitizing tooth– paste with resin composite were significantly lower than the specimens treated with a regular fluoride tooth– paste and specimens in the negative control groups.

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References

- Haywood VB. Dentine hypersensitivity: bleaching and restorative considerations for successful management. Int Dent J. 2002;52:7–15.
- Porto IC, Andrade AK, Montes MA. Diagnosis and treatment of dentinal hypersensitivity. J Oral Sci. 2009;51:323-32.
- Trushkowsky RD, Oquendo A. Treatment of dentin hypersensitivity. Dent Clin North Am. 2011;55:599–608.
- Markowitz K, Pashley DH. Discovering new treatments for sensitive teeth: the long path from biology to therapy. J Oral Rehabil. 2008;35:300–15.
- Petersson LG. The role of fluoride in the preventive management of dentin hypersensitivity and root caries. Clin Oral Investig. 2013;17:S63-71.
- Itota T, Torii Y, Nakabo S, Yoshiyama M. Effect of fluoride application on tensile bond strength of self-etching adhesive systems to demineralized dentin. J Prosthet Dent. 2002;88:503–10.
- Lehmann N, Degrange M. Effect of four dentin desensitizer on the shear bond strength of three bonding systems. Eur Cell Mater. 2002;9:52–3.
- Santiago SL, Pereira JC, Martineli AC. Effect of commercially available and experimental potassium oxalate-based dentin desensitizing agents in dentin permeability: influence of time and filtration system. Braz Dent J. 2006;17:300-5.
- Aranha AC, Siqueira Junior Ade S, Cavalcante LM, Pimenta LA, Marchi GM. Microtensile bond strengths of composite to dentin treated with desensitizer products. J Adhes Dent. 2006;8:85–90.
- Kleinberg I. SensiStat. A new saliva-based composition for simple and effective treatment of dentinal sensitivity pain. Dent Today. 2002;21:42–7.
- 11. Cummins D. Dentin hypersensitivity: from diagnosis to a breakthrough therapy for everyday sensitivity relief. J Clin Dent. 2009;20(Spec Iss):1-9.

- 12. Petrou I, Heu R, Stranick M, Lavender S, Zaidel L, Cummins D, et al. A breakthrough therapy for dentin hypersensitivity: How dental products containing 8% arginine and calcium carbonate work to deliver effective relief of sensitive teeth. J Clin Dent. 2009;20(Spec Iss):23-31.
- 13. Ayad F, Ayad N, Zhang YP, DeVizio W, Cummins D, Mateo LR. Comparing the efficacy in reducing dentin hypersensitivity of a new toothpaste containing 8.0% arginine, calcium carbonate, and 1450 ppm fluoride to a commercial sensitive toothpaste containing 2% potassium ion: an eight-week clinical study on Canadian adults. J Clin Dent. 2009;20(Spec Iss):10-6.
- 14. Docimo R, Montesani L, Maturo P, Costacurta M, Bartolino M, DeVizio W, et al. Comparing the efficacy in reducing dentin hypersensitivity of a new toothpaste containing 8.0% arginine, calcium carbonate, and 1450 ppm fluoride to a commercial sensitive toothpaste containing 2% potassium ion: an eight-week clinical study in Rome, Italy. J Clin Dent. 2009;20:17–22.
- 15. Markowitz K. The original desensitizers: strontium and potassium salts. J Clin Dent. 2009;20:145–51.
- Gillam DG, Bulman JS, Jackson RJ, Newman HN. Comparison of 2 desensitizing dentifrices with a commercially available fluoride dentifrice in alleviating cervical dentine sensitivity. J Periodontol. 1996;67:737-42.
- Claydon NC, Addy M, MacDonald EL, West NX, Maggio B, Barlow A, *et al.* Development of an in situ methodology for the clinical evaluation of dentine hypersensitivity occlusion ingredients. J Clin Dent. 2009;20:158–66.
- Pearce NX, Addy M, Newcombe RG. Dentine hypersensitivity: a clinical trial to compare 2 strontium densensitizing toothpastes with a

conventional fluoride toothpaste. J Periodontol. 1994;65:113-9.

- Banfield N, Addy M. Dentine hypersensitivity: development and evaluation of a model *in situ* to study tubulepatency. J Clin Periodontol. 2004;31: 325–35.
- Earl JS, Ward MB, Langford RM. Investigation of dentinal tubule occlusion using FIB-SEM milling and EDX. J Clin Dent. 2010;21:37-41.
- 21. Peumans M, De Munck J, Van Landuyt KL, Poitevin A, Lambrechts P, Van Meerbeek B. A 13-year clinical evaluation of two three-step etch-and-rinse adhesives in non-carious class-V lesions. Clin Oral Investig. 2012;16:129-37.
- 22. Wilder AD, Swift EJ, Heymann HO, Ritter AV, Sturdevant JR, Bayne SC. A 12-year clinical evaluation of a three-step dentin adhesive in noncarious cervical lesions. J Am Dent Assoc. 2009;140:526-35.
- Walter R, Swift EJ, Jr., Boushell LW, Braswell K. Enamel and dentin bond strengths of a new self-etch adhesive system. J Esthet Restor Dent. 2011;23:390-6.
- Sezinando A, Perdigao J, Regalheiro R. Dentin bond strengths of four adhesion strategies after thermal fatigue and 6-month water storage. J Esthet Restor Dent. 2012;24:345-55.
- 25. Turkun M, Celik EU, Kaya AD, Arici M. Can the hydrogel form of sodium ascorbate be used to reverse compromised bond strength after bleaching? J Adhes Dent. 2009;11:35–40.
- Cunningham MP, Meiers JC. The effect of dentin disinfectants on shear bond strength of resin-modified glass-ionomer materials. Quintessence Int. 1997;28: 545-51.
- 27. Markowitz K, Pashley DH. Discovering new treatments for sensitive teeth: The long path from biology to therapy. J Oral Rehabil. 2008;35:300–15.
- 28. Olley RC, Pilecki P, Hughes N, Jeffery P, Austin

RS, Moazzez R, *et al.* An *in situ* investigating dentine tubule occlusion of dentifrices following acid challenge. J Dent. 2012;40:585–93.

- Parkinson CR, Butler A, Willson RJ. Development of an acid challenge-based *in vitro* dentin disc occlusion model. J Clin Dent. 2010;21:31-6.
- 30. Seong J, Macdonald E, Newcombe RG, Davies M, Jones SB, Johnson S, *et al.* In situ randomised trial to investigate the occluding properties of two desensitising toothpastes on dentine after subsequent acid challenge. Clin Oral Investig. 2013;17:195–203.
- 31. Wang Z, Sa Y, Sauro S, Chen H, Xing W, Ma X, et al. Effect of desensitising toothpastes on dentinal tubule occlusion: A dentine permeability measurement and SEM *in vitro* study. J Dent. 2010; 38:400–10.
- 32. Sauro S, Watson TF, Thompson I. Dentine desensitization induced by prophylactic and air-polishing procedures: an *in vitro* dentine permeability and confocal microscopy study. J Dent. 2010;38: 411-22.
- 33. Garcia-Godoy A, Garcia-Godoy F. Effect of an 8.0% arginine and calcium carbonate in-office desensitizing paste on the shear bond strength of composites to human dental enamel. Am J Dent. 2010;23:324-6.
- 34. Canares G, Salgado T, Pines MS, Wolff MS. Effect of an 8.0% arginine and calcium carbonate desensitizing toothpaste on shear dentin bond strength. J Clin Dent. 2012;23:68-70.
- 35. Pashley DH, Sano H, Ciucchi B, Yoshiyama M, Carvalho RM. Adhesion testing of dentin bonding agents: a review. Dent Mater. 1995;11:117-25.
- Perdigao J. Dentin bonding-variables related to the clinical situation and the substrate treatment. Dent Mater. 2010;2:e24-37.

- 37. Adebayo OA, Burrow MF, Tyas MJ. Bonding of one-step and two-step self-etching primer adhesives to dentin with different tubule orientations. Acta Odontol Scand. 2008;66:159-68.37.
- Sattabanasuk V, Shimada Y, Tagami J. The bond of resin to different dentin surface characteristics. Oper Dent. 2004;29:333-41.
- Phrukkanon S, Burrow MF, Tyas MJ. The effect of dentine location and tubule orientation on the bond strengths between resin and dentine. J Dent. 1999;27:265-74.
- Schilke R, Lisson JA, Bauss O, Geurtsen W. Comparison of the number and diameter of dentinal tubules in human and bovine dentine by scanning electron microscopic investigation. Arch Oral Biol. 2000;45:355-61.
- 41. Yassen GH, Platt JA, Hara AT. Bovine teeth as substitute for human teeth in dental research: A review of literature. J Oral Sci. 2011;53:273-82.
- 42. Reis AF, Giannini M, Kavaguchi A, Soares CJ, Line SR. Comparison of microtensile bond strength to enamel and dentin of human, bovine, and porcine teeth. J Adhes Dent. 2004;6:117–21.
- Muench A, da Silva EM, Ballester RY. Influence of different dentinal substrates on the tensile bond strength of three adhesive systems. J Adhes Dent. 2000;2:209-12.
- Hiraishi N, Yiu CK, King NM, Tay FR. Effect of pulpal pressure on the microtensile bond strength of luting resin cements to human dentin. Dent Mater. 2009;25:58-66.
- 45. Mazzitelli C, Monticelli F, Osorio R, Casucci A, Toledano M, Ferrari M. Effect of simulated pulpal pressure on self-adhesive cements bonding to dentin. Dent Mater. 2008;24:1156-63.
- 46. Hamlin D, Mateo LR, Dibart S, Delgado E, Zhang

YP, DeVizio W. Comparative efficacy of two treatment regimens combining in-office and at-home programs for dentin hypersensitivity relief: a 24-week clinical study. Am J Dent. 2012;25:146-52.

- Bavbek AB, Goktas B, Cekic-Nagas I, Egilmez F, Ergun G, Eskitascioglu G. Micro-shear bond strength of resin cement to dentin after application of desensitizing toothpastes. Acta Odontol Scand. 2013;71:952-6.
- 48. Marchesi G, Frassetto A, Visintini E, Diolosa M, Turco G, Salgarello S, *et al.* Influence of ageing on self-etch adhesives: one-step vs. two-step systems. Eur J Oral Sci. 2013;121:43-9.
- A9. Nicola C, Sava S, Dudea D, Bondor CI, Soanca A, Colceriu L, *et al.* Influence of adhesive systems composition on dual-cured resin cements. J Optoelectron Adv M. 2009;4:359-65.

ผลของยาสีฟันลดเสียวฟันต่อกำลังแรงยึด แบบดึงระดับจุลภาคระหว่างเรซินคอมโพสิต และเนื้อฟัน

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บทคัดย่อ

วัตถุประสงค์ เพื่อประเมินและเปรียบเทียบผลของยาสีฟันลดเสียวฟันสองชนิดกับยาสีฟันฟลูออไรด์ปกติ ต่อกำลังแรงยึดแบบดึงระดับจุลภาคของสารยึดติด 2 ชนิดต่อเนื้อฟัน

วัสดุและวิธีการ ฟันวัวจำนวน 40 ซี่ นำมาขัดให้เรียบจนเผยเนื้อฟันด้านริมฝีปาก สุ่มแบ่งเป็น 4 กลุ่มตาม ยาสีฟันที่ใช้ในการทดลองดังนี้ 1) เซนโซดายน์ แรปิดรีลิฟ 2) คอลเกตเซนซิทิฟโปรรีลิฟ 3) คอลเกตรสยอดนิยม และ 4) แซ่ในน้ำลายเทียม (กลุ่มควบคุม) แปรงฟันกลุ่มที่ 1–3 ด้วยยาสีฟันตามกลุ่มที่กำหนดภายใต้น้ำหนักกด คงที่ (200 กรัม) 250 ช่วงชักต่อนาที เป็นเวลา 2 นาที 2 ครั้งต่อวัน เป็นเวลาสามวัน แบ่งฟันในแต่ละกลุ่มอีกครั้ง โดยการสุ่มเพื่อก่อเรซินคอมโพสิตโดยใช้สารยึดติดดังนี้ 1) ออพติบอนด์ เอกซ์ทีอาร์ หรือ 2) ออพติบอนด์ เอฟแอล หลังจากบ่มสารยึดติด ก่อแกนคอมโพสิตชนิดบ่มตัวด้วยแสง (พรีมิส) นำฟันตัวอย่างมาตัดเป็นชิ้นตัวอย่าง จำนวน 4 ชิ้น แต่ละซิ้นหนาและกว้าง 1 ± 0.1 มิลลิเมตร นำไปทดสอบกำลังแรงยึดแบบดึงระดับจุลภาคโดยใช้ เครื่องทดสอบสากล ความเร็วของหัวกด 0.5 มิลลิเมตรต่อนาที นำข้อมูลมาวิเคราะห์ความแปรปรวนสองทางและ เปรียบเทียบเชิงซ้อนชนิดทูกีย์ ที่ระดับนัยสำคัญ *p*<0.05 และวิเคราะห์ลักษณะการแตกที่เกิดขึ้นของพื้นผิวเนื้อฟัน ที่เกิดพันธะด้วยกล้องจุลทรรศน์ชนิดสเตอริโอ

ผลการศึกษา กำลังแรงยึดลดลงอย่างมีนัยสำคัญทางสถิติจากการใช้ยาสีฟันลดเสียวฟัน (*p* < 0.0001) และชนิด ของสารยึดติดส่งผลต่อกำลังแรงยึดอย่างมีนัยสำคัญ (*p* < 0.0001)

สรุป การใช้ยาสีฟันลดเสียวพันลดกำลังแรงยึดแบบดึงระดับจุลภาคของสารยึดติดต่อเนื้อพัน

(ว ทันต จุฬาฯ 2557;37:225-40)

คำสำคัญ: กำลังแรงยึดแบบดึงระดับจุลภาค; ยาสีฟันลดเสียวฟัน; ระบบการยึดติด; สตรอนเทียม อะซิเตท; อาร์จินิน

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