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Original research

Comparing the effectiveness of desensitizing toothpastes on the dentin bond strength of a new self-cured universal adhesive

Purpose

This study aims to evaluate the shear bond strength (SBS) of dentin using a new selfcured universal adhesive after the application of various desensitizing toothpastes.

Materials and Methods

Fifty permanent third molar teeth were prepared by removing roots and enamel surfaces to expose the buccal and lingual dentin surfaces mesiodistally. Specimens were then randomly divided into five groups: Sensodyne Repair & Protect (Group 1), Ipana Pro-Expert (Group 2), Colgate Sensitive Pro-Relief (Group 3), Prevdent (Group 4), and Group 5 served as the control group where no toothpaste was applied. An electric toothbrush was used to brush the teeth twice daily for 14 days, each session lasting 15 seconds. Each group was further divided into two subgroups for bonding procedures using Clearfil SE-Bond (CSB) and Tokuyama Universal Bond (TUB). Composite resin was applied to all sample surfaces following the adhesive procedures. After undergoing 5,000 thermal cycles, the SBS test was conducted. Data were analyzed using two-way ANOVA and post hoc Tukey test (p<0.05). Scanning electron microscopy (SEM) was employed to assess the toothpaste's ability to occlude dentinal tubules, while stereomicroscopy (x40) was used for failure analysis.

Results

The data indicated that the highest mean SBS value among all groups was observed in CSB/Group-5 (13.83 MPa), while the lowest mean SBS value was recorded in TUB/ Group-4 (5.21 MPa). SEM analysis showed significant tubule occlusion in the group treated with nanohydroxyapatite-containing toothpaste.

Conclusion

The study found that toothpaste containing nanohydroxyapatite effectively occludes dentin tubules. Therefore, two-step self-etch adhesive systems might be preferred over self-curing universal adhesives. The selection of adhesive procedures should consider the desensitizing toothpaste's composition.

Keywords: Dentin hypersensitivity, desensitizing toothpaste, dentin tubule occlusion, shear bond strength, self-cure universal adhesive

Introduction

Dentin hypersensitivity (DH) is a common condition in clinical dentistry that significantly impacts patients' daily lives (1,2). For DH to manifest, two conditions must be met: the exposure of the dentin surface (lesion localization) and the opening of dentinal tubules (lesion initiation) (2). The hydrodynamic theory's acceptance has led to two primary approaches in DH treatment: reducing fluid flow within the dentinal tubules and blocking nerve transmission (2,3).

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This work is licensed under Creative Commons Attribution-NonCommercial 4.0 International License A variety of desensitizing agents are currently employed to manage DH. These include home-use products such as toothpastes, mouthwashes, and gels (4). Recent advancements have introduced active ingredients like fluoride, casein phosphopeptide-amorphous calcium phosphate (CPP-ACP) complex, tricalcium phosphate, bioactive glass (novamine), arginine bicarbonate, and nanohydroxyapatite into these products. These agents aim to occlude dentinal tubules through remineralization, addressing the root cause of DH (4,5).

When home treatments fail to alleviate pain, professional dental interventions become necessary. In some instances, teeth with sensitivity and hard tissue loss may require restorative treatments combined with sensitivity management (6). However, desensitizing agents can influence the bond strength of adhesive restorations to dentin by altering the dentin surface, which is crucial for the clinical success of these restorations (4).

Recent technological advancements have led to the development of "universal" or "multi-mode" adhesive systems. These systems, while similar to one-step self-etching adhesives, include unique components such as carboxylate and phosphate monomers, methacryloyloxydecyl dihydrogen phosphate (10-MDP), silane, and polyacrylic acid (7). The TUB represents a new generation of two-component, onestep universal adhesive systems that incorporate a three-dimensional self-reinforcing (3D-SR) monomer. This monomer forms strong, three-dimensional crosslinking polymers after polymerization, offering enhanced bonding to tooth structures (8). Universal adhesives stand out because of their ability to ionically bond to calcium in hydroxyapatite, thanks to their specific carboxylate and/or phosphate monomers. However, given their recent introduction to the market, research on their performance, especially the newer versions containing the 3D-SR monomer, is still limited (9,10).

This study aims to explore the impact of different desensitizing toothpastes on the dentin bonding strength of the new self-cured universal adhesive TUB and to assess the tubule occlusion capabilities of these pastes. The null hypothesis posits that desensitizing toothpastes will not influence the dentin shear bond strength values of the TUB adhesive system.

Materials and Methods

Ethical approval

This study has been approved by the Clinical Research Ethics Committee of the University with the decision dated 03.03.2020 and numbered 2020/40.

Preparation of specimens for bond strength testing

Fifty extracted permanent third molar teeth were stored in distilled water (4 °C) at room temperature for one week. The roots were separated from the crowns approximately 1.5 mm below the cemento-enamel junction using a diamond bur under copious water spray. The buccal and lingual enamel were removed using a low-speed cutting device (Isomet 1000, Buehler) and underwater cooling revealed the dentin surfaces. From each tooth, two dentin discs (1 mm thickness) were

obtained from the mid-coronal region by performing mesiodistal cuts perpendicular to the long axis of the tooth using a low-speed diamond disc under water cooling. To obtain a standardized smear layer, the sample surfaces were sanded with 600, 800, and 1000 grit silicon carbide papers for a total of 60 seconds under running water. The sanded specimens were washed under water and dried slightly with air spray. 17% EDTA was applied to each sample surface for 20 seconds to simulate dentin. After the specimens were rewashed with distilled water, they were placed in silicone molds (13x10) filled with acrylic and randomly divided into five groups (n=20).

Group 1: Sensodyne Repair and Protection Group 2: Ipana Pro-Expert (Sensitive Protection) Group 3: Colgate Sensitive Pro-Relief Group 4: Prevdent Toothpaste Group 5: Control

Desensitizing toothpastes used in the study were applied to the sample surfaces with an electric toothbrush (Oral B Professional Care Triumph) under standard pressure for 14 days, twice a day for 15 seconds. If a force above 2.4 N (Newton) was applied, the toothbrush gave an alert and stopped spontaneously. The head of the toothbrush was positioned parallel to the sample surfaces and fixed. Toothpastes were mixed with artificial saliva in a ratio of 1: 2. A single operator brushed the samples, and four different heads were used to prevent contamination. The samples were stored in artificial saliva during the brushing process. An artificial saliva solution was formulated by dissolving 1.5 mmol/L of CaCl₂, 0.9 mmol/L of $\rm KH_2PO_{4\prime}$ 130 mmol/L of KCl, and 20 mmol/L of 4-(2-hydroxyethyl-)-1-piperazineethanesulfonic acid (HEPES). The pH was then adjusted to 7.0 using potassium hydroxide (1 mmol/L) (11). Desensitizing toothpaste was not applied to the control group. All samples on which desensitizing toothpaste was used were washed with distilled water for 30 seconds and then stored in artificial saliva at 37° C for 24 hours.

After the desensitizing toothpaste application, each group was divided into two subgroups: Clearfil SE Bond (CSB) and Tokuyama Universal Bond (TUB). After the adhesive systems were applied according to the manufacturer's instructions, Filtek Z 250 (B3, 3M ESPE, St Paul, MN, USA), a microhybrid universal composite resin, was applied to the dentin surface of all samples using cylindrical plastic molds of 4 mm in height and 3 mm in diameter (Figure 1). The composite resins placed with the incremental technique were polymerized for 20 seconds with a LED light device (Woodpecker, 1200 mW / cm²). The materials used in the study are listed in Table 1 and 2 with their composition.

Bond strength testing

The samples were aged for a total of 5,000 cycles (5-55 ° C, retention time: 25 sec, transfer time: 10 sec) with a thermal cycle device (SD Mechatronik Thermocycler, Germany) before the shear bond strength test. After storage in artificial saliva for 24 h, the shear bond strength test was performed using a universal tester (Universal Testing Machine LRX, Lloyd, England). The samples were fixed to the test device, and the crosshead speed was adjusted to 1 mm/sec. The values obtained were converted into MPa.

Failure mode analysis

After the bond strength test, the specimens' failure mode was determined using a stereomicroscope (Olympus, SZX10, Japan) with X40 magnification. Adhesive failure has been classified as dentin cohesive failure (failure on the dentin surface), resin cohesive failure (failure on the composite resin surface), and mixed failure (both adhesive and cohesive failure on the same surface).

Scanning electron microscopy (SEM)

To evaluate the tubule occlusion efficiency of toothpastes, a total of 10 samples, two from each group, were prepared. After removing the occlusal enamel of the specimens, the dentin surfaces were exposed. The samples were then sanded un-



Figure 1. Demonstration of the experimental design used in the present study.

der running water with 600, 800, and 1000 grit silicon carbide papers for 60 seconds in total. After the sanded specimens were washed with distilled water and dried with air spray, 37% orthophosphoric acid was applied on each sample surface for 30 seconds to expose the dentinal tubules. The samples were rewashed with distilled water and dried. The desensitizing toothpastes were applied to each sample surface for 15 seconds, twice a day for 14 days, under standard pressure with an electric toothbrush (Oral B Professional Care Triumph). After the desensitizing toothpaste procedure, the samples were washed with distilled water for 30 seconds and stored in artificial saliva at 37° C for 24 hours. After the samples were dried, they were examined with a SEM device (Zeiss Sigma 300 VP, 15.00 kV) at X2000 magnification (figure 2).

Statistical analysis

The obtained values were analyzed with a statistical package program (SPSS, 25.0, IBM Corp. Released 2017. IBM SPSS Statistics for Windows, Version 25.0. Armonk, NY, USA). The normality of the distribution and homogeneity of the variances were checked with Shapiro-Wilk and Levene's tests, respectively. Two-Way analysis of variance (ANOVA) test was used to evalaute the interaction between adhesive and toothpastes. The post hoc Tukey test was used in cases where there was a statistically significant difference in the variances. The confidence level was set to 95% and p values less than 0.05 were considered significant.



Figure 2. Preparation process of the electron microscopy samples for the examination of dentin tubule occlusion.

Table 1. Toothpaste ingredients and manufacturer brands used in the study.				
Desensitizing toothpaste	Ingredients	Manufacturer		
Sensodyne Repair and Protection	Glycerin, PEG-8, Hydrated Silica, Calcium Sodium Phosphosilicate (%5 Novamin), Cocamidopropyl Betaine, Sodium Methyl Cocoyl Taurate, Titanium Dioxide, Aroma, Carbomer, Sodium Saccharin, Limonene. Sodium Fluoride (1450 ppm)	GlaxoSmithKline, Ireland		
Ipana Pro-Expert (Sensitive Protection)	Aqua, Sorbitol, Hydrated Silica, Sodium Lauryl Sulfate, Sodium Gluconate, Carrageenan, Aroma, Xanthan Gum, Zinc citrate, Cl 77891, Stannous Fluoride (1100ppm), Sodium Hidroxide, Stannous Chloride, Sodium Saccharin, Glycerin, Sodium Hexametaphosphate, Sodium Fluoride (350ppm), Sucralose, Citric acid, Sodium Citrate, Sodium Benzoate, Potassium Sorbate	Procter&Gamble Manufacturing GmbH, Germany		
Colgate Sensitive Pro Relief	%8 Arginine, Calcium Carbonate, Aqua, Sorbitol, Aroma,Poloxamer 407, Cocamidopropyl betaine, Zinc oxide, Zinc citrate, Sodium Monofluorophospate, Cellulose Gum, Sodium Bicarbonate, Tetrasordium Pyrophosphate, Sodium Saccharin, Benzyl Alcohol, Xanthan Gum, Sucralose, Limonene,Cl 77891.	Colgate-Palmolive Co, Poland		
Prevdent	Water, Hydrated Silica, Sorbitol, Glycerin, Xylitol, Potassium Nitrate, Nano-Hydroxyapatite, Magnesium Aluminum Silicate, Mentha Piperita Oil, Sodium Lauroyl Sarcosinate, Xanthan Gum, Phenoxyethanol, Potassium Chloride, Sodium Sulfate, Sodium Saccharin, Linalool, Limonene, Cl 77891.	Prevdent, Netherland		

Table 2. Description of	f composite and adhesive materials used in the pres	sent studv.

Materials	Composition	Manufacturer	Application	Lot No.
Clearfil SE- Bond	Primer: MDP, HEMA, dimethacrylate, Di- camphoroquinone, N, N: Dietanol-p-toluidin, water. Bond: MDP, BisGMA, HEMA, dimethacrylate, Di- camphoroquinone, N-N Dietanol- p-toluidin, silanized colloidal silica.	Kuraray, Kurashiki, Japan	The primer is applied to the tooth surface for 20 seconds. Dry for 5 seconds with light air. Then bond is applied for 10 seconds. It is dried with air for 5 seconds and polymerized with light for 10 seconds.	000065
Tokuyama Universal Bond	A: Metakrilat monomers, phosphoric acid monomer (3D SR), Bis-GMA, TEGDMA, HEMA, MTU-6, acetone. B: Silane coating agent, peroxide, borate catalyst, water, isopropyl alcohol.	Tokuyama, Japan	One drop of each bottle A and B is mixed and applied to the tooth surface by rubbing for 20 seconds. Air dry for 5 seconds.	066EZ9
Filtek Z250 Micro hybrid Universal Composite	BisEMA, BisGMA, TEGDMA, UDMA, Zirconia, Silica, Camphoroquinone Inorganic filler weight: 82% volume: 60%	3M ESPE, St Paul, MN, USA	It was applied by incremental technique and light cured for 20 seconds.	NA47392

MDP: Methacryloyloxydecyl dihydrogen phosphate, HEMA: Hydroxyethyl Methacrylate, BisGMA: bisphenol A-glycidyl methacrylate, TEGDMA: triethylene glycol dimethacrylate, UDMA: Urethandimethacrylate, MTU-6: 6-methacryloxyhexyl 2-thiouracil-5-carboxylate, BisEMA: bisphenol A diglycidyl methacrylate ethoxylated.

Results

Bond strength test results

The dentin SBS values corresponding to each toothpaste per each adhesive system are shown in Table 3. The Two Way Anova analysis revealed no statistically significant difference when toothpaste and adhesive system were evaluated together. Among all groups, the highest shear bond strength value (MPa) was observed in the Clearfil SE Bond (CSB) subgroup of Group 5 (13.83), while the lowest average bond strength value was observed in the Tokuyama Universal Bond (TUB) subgroup of Group 4 (5.21). All CSB subgroups showed statistically significantly higher bond strength than the TUB subgroups (p<0.05).

As a result of comparisons made using post hoc Tukey Test in subgroups where CSB was used, only Group 4 showed significantly lower bond strength than Group 5 (p<0.05), but no statistically significant difference was observed between other groups. When the post hoc Tukey Test was applied in subgroups with TUB application, Groups 1, 3, and 4 showed significantly lower bond strength than Group 5 (p<0.05), while Group 2 was found to be similar to all groups (Table 4).

Evaluation of failure modes

In CSB subgroups, mostly mixed and cohesive type failure was observed, except for Group 4, and primarily adhesive type failure was observed in Group 4. The distribution of the samples' fracture surfaces by failure types is shown in figure 3 and 4.





Scanning electron microscopy observations

The highest tubule occlusion was observed in Group 4, and the least occlusion was observed in Group 2. Since the smear layer was removed entirely in the control group, no occlusion was observed. The images we obtained correspond to the bond strength values of our study. SEM images of the samples are shown in Figure 5.

Discussion

The tubular and hydrophilic structure of dentin tissue, pulpal pressure, the smear layer formed after preparation, and changes in dentin structure are among the numerous biological and clinical factors that affect dentin adhesion. In recent years, universal or multi-mode adhesive systems have been developed to minimize the drawbacks in adhesion (9-12). Therefore, in our study, we chose to use a new adhesive system, Tokuyama Universal Bond (TUB), which does not require light for polymerization.

In this study, it was determined that within different desensitizing toothpaste groups, CSB demonstrated higher bond strength values compared to the TUB groups (table 3). MDP (10-Methacryloyloxydecyl dihydrogen phosphate), present in CSB, is a hydrophobic monomer with the ability to chemically interact intensely and stably with the calcium in hydroxyapatite. The stable accumulation of MDP-Ca salt and the resulting nanolayer, as observed in several studies, can explain the high stability of MDP-based adhesives (13-15). Furthermore, studies comparing MDP monomer with other monomers (4-META, phenyl-P) have reported that MDP-based adhesion exhibited high bond strength and long-term stability (16). The newly developed 3D-SR monomer contained in TUB is a three-dimensional self-reinforcing multifunctional acidic monomer. There are limited studies in the literature on the bond strength of the TUB. Katsumata *et al.* compared the dentin microtensile bond strengths of different universal adhesives (single bond universal, TUB) using different restorative materials and found no statistically significant difference (17). According to the literature, it has been reported that the 3D-SR monomer forms strong three-dimensional crosslinked polymers after polymerization and contributes to bond strength by creating a hydrolysis-resistant calcium salt on dentin (18, 19). In our study, the reason for choosing this adhesive system is its different monomer composition and the absence of light requirement during polymerization. When the SBS values were evaluated, it was observed

Table 3.	Means and stan	dard deviations c	of shear bond str	ength (SBS) in N	MPa for toothpaste:	s and adhesive sys	items.

Groups	Clearfil SE-Bond	Tokuyama Universal Bond
	Mean (SD)	Mean (SD)
Group 1: Sensodyne Repair and Protection	10,83 (± 3,49) ^{A,ab}	5,51 (±1,82) ^{в,а}
Group 2: Ipana Pro- Expert (Sensitive Protection)	11,55 (± 3,08) ^{A,ab}	7,10 (±1,86) ^{B,ab}
Group 3: Colgate Sensitive Pro-Relief	10,42 (± 3,54) ^{A,ab}	5,92 (±1,82) ^{B,a}
Group 4: Prevdent	8,72 (± 2,55) ^{A,a}	5,21 (±1,37) ^{B,a}
Group 5: Control	13,83 (± 2,88) ^{A,b}	8,31 (±1,62) ^{B,b}

In the line A, B letters show a statistical difference (p<0.05). In the column a, b letters show statistical difference (p< 0.05). (SD: Standard Deviation)

Clearfil SE-Bond				Tokuyama Universal Bond		
C	Groups	P value		Groups	P value	
Group 1	Group 2	0,986	Group 1	Group 2	0,252	
	Group 3	0,998		Group 3	0,984	
	Group 4	0,565		Group 4	0,994	
	Group 5	0,223		Group 5	0,006 *	
Group 2	Group 1	0,986	Group 2	Group 1	0,252	
	Group 3	0,927		Group 3	0,544	
	Group 4	0,274		Group 4	0,116	
	Group 5	0,491		Group 5	0,516	
Group 3	Group 1	0,998	Group 3	Group 1	0,984	
	Group 2	0,927		Group 2	0,544	
	Group 4	0,744		Group 4	0,882	
	Group 5	0,126		Group 5	0,025 *	
Group 4	Group 1	0,565	Group 4	Group 1	0,994	
	Group 2	0,274		Group 2	0,116	
	Group 3	0,744		Group 3	0,882	
	Group 5	0,006 *		Group 5	0,002 *	
Group 5	Group 1	0,223	Group 5	Group 1	0,006 *	
	Group 2	0,491		Group 2	0,516	
	Group 3	0,126		Group 3	0,025 *	
	Group 4	0,006 *		Group 4	0,002 *	



Figure 4. Stereomicroscope images of failure types a) adhesive b) dentin cohesive c) composite cohesive d) mixed type.



Figure 5. SEM images of the dentine surface morphology after treatment desensitizing toothpastes (X2000). a) Sensodyne Repair and Protection (Group 1), b) Ipana Pro-Expert (Sensitive Protection) (Group 2), c) Colgate Sensitive Pro-Relief (Group 3), d) Prevdent (Group 4), e) Control group- no toothpaste applied (Group 5).

that the TUB values were significantly lower compared to the CSB in each toothpaste group (table 3). It has been reported that the quality and degree of polymerization, which are important factors affecting adhesion, are higher in light-cured adhesives compared to chemically cured adhesives (20, 21). Due to the chemical polymerization process of TUB, as opposed to the light-cured CSB, the bond strength values may vary. We hypothesize that the adhesive of CSB, characterized by its higher acidity (pH=2) and inclusion of MDP monomer, contributes to higher bond strength values compared to TUB (12,22).

Previous studies examining fracture types following bond strength testing have reported a predominance of cohesive fractures in high bond strength groups, while adhesive fractures were more prevalent in groups with low bond strength (23, 24). In line with these findings, the present study observed predominantly mixed and cohesive type failures in CSB subgroups, and adhesive failures in TUB subgroups, supporting the bond strength results (figure 3).

In the treatment of dentin hypersensitivity, dentinal tubule occlusion can be achieved through the use of nanohydroxyapatite, novamin, and pro-arginine technologies to stimulate tubular mineralization (25-27). When examining the SEM images obtained in this study, it was observed that tubular occlusion occurred at specific rates in all groups where the desensitizing paste was applied (figure 5).

SEM analysis in many in vitro studies has demonstrated the ability of nanohydroxyapatite to effectively remineralize dentin, form an acid-resistant layer on the dentin surface, and occlude dentinal tubules (28-30). In an in vitro study comparing toothpastes containing nanohydroxyapatite, novamin, and pro-arginine in terms of dentinal tubule occlusion, it was reported that the nanohydroxyapatite group achieved 98.1% occlusion, the novamin group achieved 83.1% occlusion, and the pro-arginine group achieved 69.1% occlusion (31). Consistent with our study, the SEM images of dentin surfaces treated with a nanohydroxyapatite-containing toothpaste revealed nearly complete occlusion of all dentinal tubules (figure 5). The findings of Earl *et al.* (32) and Shah *et al.* (33) also support our study.

Various studies have been published on the clinical efficacy of stannous fluoride with conflicting results (34-36). Arnold *et al.* (37) and West *et al.* (38) reported no superior tubule occlusion on dentin surfaces where toothpaste containing stannous fluoride was applied in their studies investigating the tubule occlusion efficiency of different toothpastes. The images that provide insights into the tubule occlusion efficiency of the toothpastes were consistent with the results of the present study (figure 5). In contrast, Takamizawa *et al.* (39) evaluated the tubule occlusion efficiency of toothpastes containing different concentrations of stannous fluoride and reported that stannous fluoride was more effective in tubule occlusion compared to the control group (distilled water).

A review of the literature reveals various findings regarding the effect of desensitizing agents on bond strength (23, 40-42). The acid-resistant layer and tubule occlusion feature created by desensitizing agents in dentinal tubules can hinder the penetration of adhesive systems into dentin. In this study, lower bond strength values were observed in the groups using nanohydroxyapatite-containing toothpaste (group 4). We attribute this result to the high tubule-occluding characteristics of nanohydroxyapatite. Pei et al. (43) have claimed that toothpastes containing nanohydroxyapatite can decrease the bond strength of self-etching adhesives, which supports our findings. Aguiar et al. (40) reported that the long-term use of desensitizing toothpastes does not affect the bond strength of self-etch adhesive (CSB). These results are consistent with our study because in our study, no statistically significant difference (p>0.05) was observed when comparing the control group with subgroups of CSB in Groups 1, 2, and 3 (table 4). When examining the studies, it has been observed that variables such as the composition of desensitizing toothpastes, their short or long-term usage, and conducting the bond strength test immediately

after 24 hours can lead to variations in bond strength values (23,44,45). Wang *et al.* (46) and Canares *et al.* (41) claimed that desensitizing toothpaste containing arginine and calcium carbonate effectively occludes dentinal tubules and has no adverse effect on dentin bonding performance when used with adhesives. In another study, it was observed that after the use of calcium-based desensitizing toothpaste, there was a decrease in bonding strength in the self-etch mode of universal adhesive, while the bonding strength was not affected in the acid etching and rinsing mode (47). In our study, the use of Colgate Sensitive Pro-Relief did not cause a significant change in CSB bond strength values, which is consistent with the findings of the mentioned studies and supports our results (47,48).

In studies investigating the effect of desensitizing agents on dentin bond strength, it has been observed that commonly used fluoride-containing toothpastes do not significantly affect dentin bond strength (23, 24, 40). The Ipana Pro-Expert (Sensitive Protection) toothpaste used in the current study did not show a statistically significant difference in bond strength for both adhesive systems, which is consistent with previous studies.

When evaluating the results obtained in this study, it can be observed that different desensitizing toothpastes caused changes in bond strength values, leading to a partial rejection of the null hypothesis. Among the limitations of the study is the absence of reflection of forces, thermal changes, oral microflora, and saliva factors to which restorative materials are exposed in the oral environment in our samples. The samples were stored in artificial saliva between tooth brushing sessions, but neither the buffering effect of saliva nor enzyme activity could be simulated. Additionally, the evaluation did not include dentin's hydraulic conductivity and pulp pressure. Clinical adhesion surfaces may vary depending on differences in dentin structure and can be considered a limitation of this study. The dentin samples used in the study were obtained from the middle part of the tooth. The orientation and diameter of dentinal tubules in this area may differ when compared to cervical, coronal, or root dentin. There are a limited number of studies on the bond strength of the newly developed and chemically polymerized TUB. Since the results of this study are limited to the adhesives and desensitizing toothpaste used, further laboratory and clinical studies are needed to test materials with different mechanisms of action.

Conclusion

Considering the potential for tubule occlusion, toothpastes containing nanohydroxyapatite can be recommended for the treatment of dentin hypersensitivity. However, restoration planning should take into account the possibility of reduced bond strength of the adhesives. Despite showing low SBS values in all groups, further in vivo and in vitro studies are needed to evaluate the effectiveness and potential clinical application of the newly developed self-curing universal adhesive.

Türkçe öz: Yeni Bir Self-Cured Universal Adezivin Dentine Bağlanma Dayanımında Hassasiyet Giderici Diş Macunlarının Etkinliğinin Karşılaştırılması. Amaç: Bu çalışma, hassasiyet giderici farklı diş macunlarının kullanımı sonrası yeni bir self-cured universal adezivin dentindeki makaslama bağlanma dayanımını (SBS) değerlendirmeyi amaçlamaktadır. Gereç ve Yöntem: Elli adet daimi üçüncü molar dişin kökleri ve mine yüzeyleri uzaklaştırıldı, meziodistal olarak bukal ve lingual dentin yüzeyleri elde edildi. Dişler rastgele beş gruba ayrıldı: Grup-1: Sensodyne Repair-Protect; Grup-2: Ipana Pro-Expert; Grup-3: Colgate Sensitive Pro-Relief; Grup-4: Prevdent; Grup-5: Kontrol (diş macunu uygulanmadı). Fırçalama işlemi, elektrikli diş fırçası kullanılarak günde iki kez 14 gün boyunca 15 saniye süreyle uygulandı. Her grup, Clearfil SE-Bond (CSB), Tokuyama Universal Bond (TUB) olmak üzere iki alt gruba ayrıldı. Adheziv prosedürü takiben tüm örnek yüzeylerine kompozit rezin uygulandı. Termal döngü (5,000) cihazından sonra SBS testi gerçekleştirildi. Veriler iki yönlü ANOVA ve post hoc Tukey testi ile analiz edildi ($p \le 0,05$). Diş macununun dentinal tübülü tıkama miktarının değerlendirilmesi için taramalı elektron mikroskobu (SEM) ve başarısızlık analizi için bir stereomikroskop (x40) kullanıldı. Bulgular: Tüm gruplar arasında en yüksek ortalama SBS değerini CSB/Grup-5 (13.83 MPa) gösterirken; en düşük ortalama SBS değeri TUB/Grup-4 (5.21 MPa)'de gözlendi. SEM analizi sonucu, nanohidroksiapatit içeren diş macunu grubunda anlamlı bir tübül tıkanması olduğu belirlendi. Sonuç: Nanohidroksiapatit içeren diş macununun dentin tübül tıkama etkinliğinin yüksek olduğu belirlendi. Bu durumda, self-cured universal adezivler yerine iki aşamalı self-etch adeziv sistemler tercih edilebilir. Adheziv prosedür, hassasiyet giderici diş macununun içeriği dikkate alınarak planlanmalıdır. Anahtar Kelimeler: dentin hassasiyeti, dentin tübül oklüzyonu, hassasiyet giderici diş macunu, makaslama bağlanma dayanımı, self-cured universal adeziv

Ethics Committee Approval: This study has been approved by the Clinical Research Ethics Committee of the University with the decision dated 03.03.2020 and numbered 2020/40.

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References

- Bekes K. Clinical presentation and physiological mechanisms of dentine hypersensitivity. Dentine Hypersensitivity: Developing a Person-centred Approach to Oral Health. 2014; 21-32. [CrossRef]
- West N, Seong J, Davies M. Dentine hypersensitivity. Monogr Oral Sci. 2014;25:108-22. doi: 10.1159/000360749. [CrossRef]
- Consensus-based recommendations for the diagnosis and management of dentin hypersensitivity. In: Journal (Canadian Dental Association). 2003;69(4):221-6.
- Talioti E, Hill R, Gillam DG. The Efficacy of Selected Desensitizing OTC Products: A Systematic Review. ISRN Dentistry. 2014;2014:865761. doi: 10.1155/2014/8657615. [CrossRef]
- Amaechi BT, Van Loveren C. Fluorides and non-fluoride remineralization systems. Monogr Oral Sci. 2013;23: 15-26. doi: 10.1159/000350458. [CrossRef]

- Davari A, Ataei E, Assarzadeh H. Dentin hypersensitivity: etiology, diagnosis and treatment; a literature review. J Dent (Shiraz, Iran). 2013; 14(3):136-45.
- 7. Perdigão J, Swift EJ. Universal Adhesives. J Esthet Restor Dent. 2015; 27(6):331-4. doi: 10.1111/jerd.12185. [CrossRef]
- Kawano S, Fu J, Saikaew P, Chowdhury AA, Fukuzawa N, Kadowaki Y, et al. Microtensile bond strength of a newly developed resin cement to dentin. Dent Mater J. 2015; 34(1):61-9. doi: 10.4012/ dmj.2014-122. [CrossRef]
- Ahmeda AA, Mustafa M. Hassanb AIA. Microshear bond strength of universal adhesives to dentin used in totaletch and selfetch modes. Tanta Dental Journal. 2018; 15(2):91-98. doi: 10.4103/ tdj._52_17. [CrossRef]
- Wang R, Shi Y, Li T, Pan Y, Cui Y, Xia W. Adhesive interfacial characteristics and the related bonding performance of four self-etching adhesives with different functional monomers applied to dentin. J Dent. 2017; 62:72-80.doi: 10.1016/j. jdent.2017.05.010. [CrossRef]
- Tao S, He L, Xu HHK, Weir MD, Fan M, Yu Z, et. al. Dentin remineralization via adhesive containing amorphous calcium phosphate nanoparticles in a biofilm-challenged environment. J Dent. 2019;89:103193. doi: 10.1016/j.jdent.2019.103193. [CrossRef]
- Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, Van Landuyt KL. State of the art of self-etch adhesives. Dent Mater. 2011; 27(1):17-28. doi: 10.1016/j.dental.2010.10.023. [CrossRef]
- Carrilho E, Cardoso M, Ferreira MM, Marto CM, Paula A, Coelho AS. 10-MDP based dental adhesives: Adhesive interface characterization and adhesive stability-A systematic review. Materials. 2019; 12(5):790. doi: 10.3390/ma12050790. [CrossRef]
- 14. Yoshida Y, Yoshihara K, Nagaoka N, Hayakawa S, Torii Y, Ogawa T, et al. Self-assembled nano-layering at the adhesive interface. J Dent Res. 2012; 91(4):376-81. doi: 10.1177/0022034512437375. [CrossRef]
- Yoshihara K, Yoshida Y, Hayakawa S, Nagaoka N, Irie M, Ogawa T, et al. Nanolayering of phosphoric acid ester monomer on enamel and dentin. Acta Biomater. 2011; 7(8):3187-95.doi: 10.1016/j.actbio.2011.04.026. [CrossRef]
- Hiraishi N, Tochio N, Kigawa T, Otsuki M, Tagami J. Monomer-collagen interactions studied by saturation transfer difference NMR. J Dent Res. 2013; 92(3):284-8.doi: 10.1177/0022034512474310. [CrossRef]
- Aiichiro Katsumata, Pipop Saikaew, Shihchun Ting, Tamaki Katsumata, Tomohiro Hoshika, Hidehiko Sano Yn. Microtensile Bond Strength Bonded to Dentin of a Newly Universal Adhesive. J Oral Tissue Engin. 2017;15(1):18–24. https://doi.org/10.11223/ jarde.15.18.
- Yoshida Y, Yoshihara K, Nagaoka N, Hanabusa M, Matsumoto T, Momoi Y. X-ray diffraction analysis of three-dimensional selfreinforcing monomer and its chemical interaction with tooth and hydroxyapatite. Dent Mater J. 2012;31(4):697-702. doi: 10.4012/dmj.2012-074. [CrossRef]
- Fu J, Kakuda S, Pan F, Hoshika S, Ting S, Fukuoka A, et al. Bonding performance of a newly developed step-less all-in-one system on dentin. Dent Mater J. 2013; 32(2):203-11. doi: 10.4012/ dmj.2012-204. [CrossRef]
- Agaccioglu M, Aytac F. Analysis Methods Related to Polymerization Properties of Composite Resins. Turkiye Klinikleri J Dental Sci. 2019; 25(2):201–12. [CrossRef]
- 21. Lu H, Mehmood A, Chow A, Powers JM. Influence of polymerization mode on flexural properties of esthetic resin luting agents. J Prosthet Dent. 2005; 94(6):549-54. doi: 10.1016/j. prosdent.2005.09.016. [CrossRef]
- Nagarkar S, Theis-Mahon N, Perdigão J. Universal dental adhesives: Current status, laboratory testing, and clinical performance. J Biomed Mater Res B Appl Biomater. 2019;107(6):2121-2131. doi: 10.1002/jbm.b.34305. [CrossRef]
- 23. Kochanipa Saisopa SS. Effect of desensitizing toothpaste on microtensile bond strength between resin composite and dentin. Cumhuriyet Dent J. 2014; 37:225–40. [CrossRef]

- Aguiar JD, Medeiros IS, e Souza Junior MHS, Loretto SC. Influence of the extended use of desensitizing toothpastes on dentin bonding, microhardness and roughness. Braz Dent J. 2017; 28(3):346-353. doi: 10.1590/0103-6440201601292. [CrossRef]
- Lavender SA, Petrou I, Heu R, Stranick MA, Cummins D, Kilpatrick-Liverman L, et al. Mode of action studies on a new desensitizing dentifrice containing 8.0% arginine, a high cleaning calcium carbonate system and 1450 ppm fluoride. Am J Dent. 2010; 23 Spec No A:14A-19A.
- Tschoppe P, Zandim DL, Martus P, Kielbassa AM. Enamel and dentine remineralization by nano-hydroxyapatite toothpastes. J Dent. 2011; 39(6):430-7. doi: 10.1016/j.jdent.2011.03.008. [CrossRef]
- Wang Z, Jiang T, Sauro S, Pashley DH, Toledano M, Osorio R, et al. The dentine remineralization activity of a desensitizing bioactive glass-containing toothpaste: An in vitro study. Aust Dent J. 2011; 56(4):372-81.doi: 10.1111/j.1834-7819.2011.01361.x. [CrossRef]
- Pepla E. Nano-hydroxyapatite and its applications in preventive, restorative and regenerative dentistry: a review of literature. Annali di Stomatologia. 2014; 20;5(3):108-14. [CrossRef]
- 29. Jena A, Kala S, Shashirekha G. Comparing the effectiveness of four desensitizing toothpastes on dentinal tubule occlusion: A scanning electron microscope analysis. J Conserv Dent. 2017; 20(4):269-272. doi: 10.4103/JCD. 34_17. [CrossRef]
- Kunam D, Manimaran S, Sampath V, Sekar M. Evaluation of dentinal tubule occlusion and depth of penetration of nanohydroxyapatite derived from chicken eggshell powder with and without addition of sodium fluoride: An in vitro study. J Conserv Dent. 2016; 19(3):239-44.doi: 10.4103/0972-0707.181940. [CrossRef]
- Kulal R, Jayanti I, Sambashivaiah S, Bilchodmath S. An invitro comparison of nano hydroxyapatite, novamin and proargin desensitizing toothpastes -A SEM study. JCDR. 2016; 10(10):ZC51-ZC54. doi: 10.7860/JCDR/2016/18991.8649. [CrossRef]
- 32. Earl JS, Topping N, Elle J, Langford RM, Greenspan DC. Physical and chemical characterization of the surface layers formed on dentin following treatment with a fluoridated toothpaste containing NovaMin. J Clin Dent. 2011; 22(3):68-73.
- Shah S, Shivakumar AT, Khot O, Patil C, Hosmani N. Efficacy of NovaMin- and Pro-Argin-containing desensitizing dentifrices on occlusion of dentinal tubules. Dental Hypotheses.2017;8(4):104. doi:10.4103/denthyp. _30_17. [CrossRef]
- 34. Sharma N, Roy S, Kakar A, Greenspan DC, Scott R. A clinical study comparing oral formulations containing 7.5% calcium sodium phosphosilicate (novamin), 5% potassium nitrate, and 0.4% stannous fluoride for the management of dentin hypersensitivity. J Clin Dent. 2010; 21(3):88-92.
- 35. He T, Barker ML, Biesbrock AR, Miner M, Qaqish J, Sharma N. A clinical study to assess the effect of a stabilized stannous fluoride dentifrice on hypersensitivity relative to a marketed sodium fluoride/triclosan control. J Clin Dent. 2014; 25(2):13-8.
- Ni LX, He T, Chang A, Sun L. The desensitizing efficacy of a novel stannous-containing sodium fluoride dentifrice: An 8-week randomized and controlled clinical trial. Am J Dent. 2010; 23 Spec No B:17B-21B.
- Arnold WH, Prange M, Naumova EA Effectiveness of various toothpastes on dentine tubule occlusion. J Dent. 2015; 43(4):440-9. doi: 10.1016/j.jdent.2015.01.014. [CrossRef]
- West NX, Seong J, Hellin N, Macdonald EL, Jones SB, Creeth JE. Assessment of tubule occlusion properties of an experimental stannous fluoride toothpaste: A randomised clinical in situ study. J Dent. 2018; 76:125-131.doi: 10.1016/j.jdent.2018.07.001. [CrossRef]
- Takamizawa T, Tsujimoto A, Ishii R, Ujiie M, Kawazu M, Hidari T, Suzuki T, Miyazaki M. Laboratory evaluation of dentin tubule occlusion after use of dentifrices containing stannous fluoride. J Oral Sci. 2019; 61(2):276-283. doi: 10.2334/josnusd.18-0176. [CrossRef]

- Aguiar JD, de Amorim ACS, Medeiros IS, Souza Júnior MHS e, Loretto SC. Influence of Prolonged use of Desensitizing Dentifrices on Dentin Bond Strength of Self-Etching Adhesive System. IJO. 2016; 10(1):135-142. doi:10.4067/s0718-381x2016000100021. [CrossRef]
- 41. 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(2):68-70.
- 42. Anithakumari R, Sureshbabu NM. The effect of desensitizing agents on the bond strength of dentin bonding agents: A systematic review. J Conserv Dent. 2022;25(6):580-587. doi: 10.4103/jcd.jcd_248_21. [CrossRef]
- Pei D, Meng Y, Li Y, Liu J, Lu Y. Influence of nano-hydroxyapatite containing desensitizing toothpastes on the sealing ability of dentinal tubules and bonding performance of self-etch adhesives. J Mech Behav Biomed Mater. 2019; 91:38-44. doi: 10.1016/j.jmbbm.2018.11.021. [CrossRef]
- Andreatti LS, Lopes MB, Guiraldo RD, Borges AH, Dorilêo MCO, Gonini A. Efffect of desensitizing agents on the bond strength of dental adhesive systems. Appl Adhes Sci. 2014;2(24). https:// doi.org/10.1186/s40563-014-0024-y. [CrossRef]

- 45. Pei D, Liu S, Huang C, Du X, Yang H, Wang Y, et al. Effect of pretreatment with calcium-containing desensitizer on the dentine bonding of mild self-etch adhesives. Eur J Oral Sci. 2013; 121(3 Pt 1):204-10.doi: 10.1111/eos.12047 [CrossRef]
- 46. Wang Y, Liu S, Pei D, Du X, Ouyang X, Huang C. Effect of an 8.0% arginine and calcium carbonate in-office desensitizing paste on the microtensile bond strength of self-etching dental adhesives to human dentin. Am J Dent. 2012; 25(5):281-6.
- 47. Wang S, Meng Y, Zhang Y, Huang F, Teng R, Lu Y, et. al. Influence of calcium-based desensitizing toothpastes on the bonding performance of universal adhesive. Microsc Res Tech. 2023;86(4):402-413. doi: 10.1002/jemt.24280. [CrossRef]
- Palhari FTL, Almeida LDM, Liporoni PCS, Hilgert LA, Zanatta RF. Influence of the combined effect of desensitizing dentifrices and universal adhesives on dentin bond strength under erosive conditions. J Appl Oral Sci. 2023;7;31:e20230224. doi: 10.1590/1678-7757-2023-0224. [CrossRef]