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Original Research Article

Effect of dentin desensitizers on resin cement bond strengths

Rubens Nazareno Garcia[1,2]
Marcelo Giannini[3]
Tomohiro Takagaki[4]
Takaaki Sato[4]
Naoko Matsui[4]
Toru Nikaido[4]
Junji Tagami[4]

Corresponding author:
Rubens Nazareno Garcia
Rua Uruguai, n. 458 – Centro
CEP 88302-202 – Itajaí – SC – Brasil
E-mail: rubensgarcia@univali.br

[1] Department of Dentistry, University of Joinville Region – Joinville – SC – Brazil.
[2] School of Dentistry, University of Itajai Valley – Itajai – SC – Brazil.
[3] Department of Restorative Dentistry, Piracicaba Dental School, State University of Campinas – Piracicaba – SP – Brazil.
[4] Department of Cariology and Operative Dentistry, Division of Oral Health Sciences, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University – Tokyo – Japan.

Abstract

Introduction: The crown preparation promotes the exposure of dentin tubules. Thus, to avoid post-operative sensitivity, the first approach involves the use of dentin adhesives, and the second one the use of dentin desensitizers. **Objective:** This study evaluated the effect of dentin desensitizers on microtensile bond strengths (μTBSs) of a resin cement to dentin. **Material and methods:** Twenty bovine teeth were prepared until obtaining flat dentin surfaces. A standardized smear layer was created (#600-grit SiC paper). The samples were randomly divided into the following four groups (n = 5): no treatment (Control), treatment with Gluma Desensitizer (Heraeus Kulzer), Super Seal (Phoenix Dental) and Teethmate Desensitizer (Kuraray Noritake Dental). The dentin surfaces were then treated with ED Primer II (Kuraray Noritake Dental). Twenty composite blocks, 4 mm thick (Estenia C&B, Kuraray Noritake Dental) were used. The composite surfaces were abraded with aluminum oxide (50 μm), and then silanized. The composite block was bonded to the dentin surface with a resin cement (Panavia F 2.0, Kuraray Noritake Dental) according to

the manufacturer's instructions. After 24-hour storage (37°C, 100% RH), the bonded samples were cut into beam–shaped microtensile specimens and loaded in tension until failure. Data were analyzed with one-way ANOVA and the Dunnett's test ($\alpha = 0.05$). An SEM was used to examine the failure modes. **Results:** The μTBSs (MPa ± SD) were: 24.4 ± 3.2 (Control), 14.0 ± 5.6 (Gluma Desensitizer), 8.6 ± 4.7 (Super Seal), and 34.7 ± 4.6 (Teethmate Desensitizer), in which there were significant differences among the four groups ($p < 0.05$). The Teethmate Desensitizer group showed the highest μTBS, while the Super Seal group showed the lowest mean of μTBS to dentin. **Conclusion:** The efficacy of the desensitizers is material-dependent; Gluma Desensitizer and Super Seal decreased the μTBSs, however, Teethmate Desensitizer improved it.

Introduction

The exposure of the dentin tubules is inevitable during cavity or crown preparations [20]. Once the tubules are opened, they act as channels that transmit mechanical, chemical and bacterial stimuli to the pulp [18]. Provisional sealing materials do not cohesively bind to dentin and may permit leakage to bacteria and their products before the luting of final restoration. During the provisional stage, the dentin may also encounter external stimuli that include impression taking, rinsing, drying, and removal of temporary sealing, which may all encourage tooth sensitivity and potential pulp damage [3]. Thus, coating should be performed immediately after cutting the dentin and provide a *biological seal* that acts as a dentin-pulp protector. To serve this aim, it has been suggested that freshly cut dentin surfaces for indirect restorations could be sealed with resin-based adhesives prior to the taking impressions, so-called *resin coating technique* [16]. In addition to its favorable effects on reduction of post-preparation and post-cementation sensitivities, this also called *immediate dentin sealing* technique [15] can result in significantly increased retention, reduced marginal leakage, and improved bond strengths [7, 8], when used for traditional crown preparations of vital teeth [13].

The most widely accepted mechanism of dentin sensitivity is the so-called hydrodynamic theory of sensitivity. It postulates that rapid shifts, in either direction, of the fluids within the dentinal tubules, following stimulus application, result in activation of sensory nerves in the pulp/inner dentin region of the tooth [1, 9].

Conventional therapy for dentin sensitivity is based on using topical application of desensitizing agents which can be applied either professionally or can be described to the patient for home use. The ideal desensitizer agent should not irritate or endanger the integrity of the pulp, should be relatively painless on application or shortly afterward, should be easily applied, rapid in action, permanently effective and finally should not discolour tooth structure [4].

The aim of the present study was to investigate the effect of dentin desensitizers on resin cement bond strengths to dentin, by evaluating microtensile bond strength and failure analysis. Null hypothesis of this study was that dentin bond strengths of a resin cement were not affected by the different dentin desensitizing agents.

Material and methods

Materials used in this study

Three desensitizers (Gluma, Heraeus Kulzer, Dormagen, Germany; Super Seal, Phoenix Dental, Fenton, MI, USA; and Teethmate Desensitizer, Kuraray Noritake Dental, Tokyo, Japan), a resin cement (Panavia F 2.0, Kuraray Noritake Dental) and an indirect composite resin (Estenia C&B, Kuraray Noritake Dental) were used in this study (Table I).

Table I – Materials used in this study

Material	Batch	Composition	
Estenia C&B Kuraray Noritake Dental	0098AA/ 0101AA	Bis-GMA, TEGDMA, photoinitiator, silanized ceramic microfillers, silica	Estenia blocks (preparation): Apply the material into matrix (3 mm thick), light cure (40 sec each side), lab light cure oven (3 min), lab hot oven (15 min), sandblasting to aluminum oxide (50 μm / 0.1MPa / 10mm distance / 10sec) Estenia side (silanation): Air drying (5 sec), K-etchant GEL (5 sec), wash (5 sec), Air drying (5 sec), mix 1 drop of Clearfil Mega Bond primer and 1 drop of Clearfil Porcelain Bond Activator, apply and wait (30 sec), air drying (5 sec)
K-etchant GEL Kuraray Noritake Dental	00522A	37% phosphoric acid	
Clearfil SE Bond Primer Kuraray Noritake Dental	01170A	MDP, HEMA, hydrophilic dimethacrylate, dl-camphorquinone, N,N-diethanol-p-toluidine, water	
Clearfil Porcelain Bond Activator Kuraray Noritake Dental	00262B	3-trimethoxysilylpropyl methacrylate, hydrophobic aromatic dimethacrylate, others	
Panavia F2.0 Kuraray Noritake Dental	Paste A: 00550A Paste B: 00276A	Paste A: Methacrylate, MDP, quartz-glass, microfillers, photoinitiator Paste B: Methacrylate, barium glass, sodium fluoride, chemical initiator	Dentin side: Air drying (5 sec), mix ED primer II A and B (1 drop each one), apply ED primer to dentin surface by small brush or sponge (just waiting 30 sec), air drying (5 sec), put paste A and paste B same amount on paper and mix (20 sec), apply the paste on Estenia, light cure (40 sec each side)
ED Primer II Kuraray Noritake Dental (pH 2.4)	Liquid A: 00313A Liquid B: 00187B	HEMA, MDP, MASA, water, accelerators MASA, water, catalysts, accelerators	
Teethmate Desensitizer Kuraray Noritake Dental	000001	Powder: Tetrocalcium phosphate, dibasic calcium phosphate Liquid: water	Mix powder and liquid (more than 15 sec), apply (rubbing more than 30 sec), wash (5 sec), clean the dentin surface by wet swab/cotton (more than 10 sec/use distilled water)
Gluma Desensitizer Heraeus Kulzer	010209	35% HEMA, 5% glutaraldehyde, water	Air drying (5 sec), apply (rubbing 30 sec), air drying (5 sec), wash (5 sec), air drying (5 sec)
Super Seal Phoenix Dental	991631	Oxalic acid, potassium salt, water	Air drying (5 sec), apply at least 5 sec (rubbing 10 sec), air drying (3 sec – mild 10 cm distance)

Abbreviations: Bis-GMA: bisphenol A glycidyl methacrylate; TEGDMA: triethyleneglycol dimethacrylate; MDP: 10-methacrylate oxydecyl dihydrogen phosphate; NaF: sodium fluoride; BPO: benzoyl peroxide; MASA: N-methacrylolyl-5-aminosalicylic acid; HEMA: 2-hydroxyethylmethacrylate

Specimen preparation for microtensile bond test

Twenty bovine lower central incisors were used as bonding substrates. The teeth were stored in water at 4°C and used within one month after extraction. Each tooth was sectioned in the root and in the labial surface, approximately 1 mm below the enamel-dentin junction using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under water stream. The teeth were prepared until obtaining middle portion of flat dentin surfaces.

Twenty indirect composite restorative blocks were fabricated using Estenia C&B (Table I). A cylindrical Teflon mold (4 mm deep and 10 mm in diameter) was made. The Estenia C&B was placed into this mold by two increments. Each increment was light cured for 40 s (Optilux 501; Kerr Corp, Orange, CA, USA / 600mW/cm^2). Afterwards, the blocks were light cured 3 min and heat cured for 15 min with an Estenia polymerization device (CS-110 light and heat curing unit; Kuraray Noritake Dental). Then, the surfaces of the composite blocks were abraded with 50-μm aluminum oxide (0.1 MPa, 10 mm distance, 10 s). Also, the Estenia C&B blocks were silanized.

The dentin surface was ground with 600-grit SiC paper under a water spray to create a standard smear layer, just before each adhesive procedure to simulate to the clinical treatment method, in which the smear layer was created by rotary instruments [17]. The teeth were randomly distributed into four groups (n = 5) according to the experimental groups: [1] control group, only ED Primer II (Kuraray Noritake Dental); [2] Gluma + ED; [3] Super Seal + ED; [4] TMD + ED. Afterwards, blocks of Estenia C&B were cemented to the samples under a load (500 g weight) using Panavia F 2.0 resin cement, which was light-activated for 40 s (Optilux 501; 600 mW/cm^2). After 24-hour storage (37ºC, 100% RH), the bonded samples were then perpendicularly sectioned with a diamond saw (Isomet 1000) under water lubrication. The samples were cut into beam–shaped microtensile specimens with an adhesive area of approximately 1 mm^2. These specimens were fixed to an universal testing machine (EZ-Test / Shimadzu, Kyoto, Japan) with a cyanoacrylate adhesive (Zapit, DVA, Anaheim Hills, CA, USA) and subjected to microtensile bond strength (μTBS) testing at a crosshead speed of 1 mm/min.

Scanning Electron Microscopy (SEM) observations

The representative specimens for each failure mode were examined by using SEM (JSM-5310LV; Jeol, Tokyo, Japan). Prior to the SEM observations, the specimens were air-dried and sputter-coated with gold. Failure modes were categorized as: adhesive failure at the resin cement-dentin interface (AD), cohesive failure within dentin (CD), cohesive failure within resin cement (CRC), mixed failure (AD + CRC), and mixed failure (CRC + TMD layer).

The dentin surfaces treated with four different treatment groups were also examined (SEM / JSM-5310LV; Jeol, Tokyo, Japan). After the specimens were treated in the same manner of the adhesive procedures described above, the treated specimens were air-dried and sputter-coated with gold for SEM examination.

Statistics

The μTBS data were statistically analyzed using a One-way ANOVA and the Dunnett's test. The statistical significance level was always set at $\alpha = 0.05$. The survival rate was analysed by Chi-square test with Bonferroni correction (p < 0.05).

Results

Microtensile bond strength testing and failure modes

Means μTBS are presented in Table II. One-way ANOVA showed significant differences among the groups (p < 0.001). The TMD group showed the highest μTBS, while the Super Seal desensitizer showed the lowest one. As presented in Table III, the control group showed mainly CRC failure. The Gluma group showed 50% mixed (AD + CRC) failure, 30% AD failure and 20% CRC failure. The Super Seal group showed 90% AD failure and the TMD showed 50% CRC failure and 50% mixed (CRC + TMD) failure.

The SEM micrographs of the dentin surfaces treated with four different treatment groups were shown in Figures 1 to 4. For the control group (Group 1), the dentin surface was covered by the smear layer. The dentinal tubules were closed (Figure 1). For Gluma Desensitizer (Group 2), the dentinal tubules were partially closed. A coating layer with the desensitizing material was not visually confirmed under the low magnification of the observation (Figure 2). For Super Seal (Group 3), small precipitates were deposited on the intertubular dentin. The dentinal tubules were occluded by the depositions (Figure 3). For Teethmate Desensitizer (Group 4), the scratches on the dentin surface with #600-grit SiC paper disappeared. Depositions covered the intertubular dentin and occluded the dentinal tubules (Figure 4).

Table II – µTBSs means to bovine dentin

Group	Mean ± SD*	Survival rate (%)**
[1] Control	24.3 ± 3.2 B	100 a
[2] Gluma	14.0 ± 5.6 C	100 a
[3] Super Seal	08.6 ± 4.7 D	40 b
[4] Teethmate Desensitizer	34.7 ± 4.6 A	100 a

Means (SD) in MPa, (n = 5). Same letters in the column are not statistically significant ($p < 0.05$)
* One-way ANOVA and Dunnett's test. ** Chi-square test with Bonferroni correction

Table III – Failure modes (%)

Group	AD	CD	CRC	AD+CRC	CRC+TMD layer
[1] Control	0	0	90	10	0
[2] Gluma	30	0	20	50	0
[3] Super Seal	90	0	0	10	0
[4] Teethmate Desensitizer	0	0	50	0	50

AD = Adhesive / CD = Cohesive in dentin / CRC = Cohesive in resin cement / AD+CRC = Adhesive and cohesive in resin cement / CRC+TMD layer = Cohesive in resin cement and TMD layer
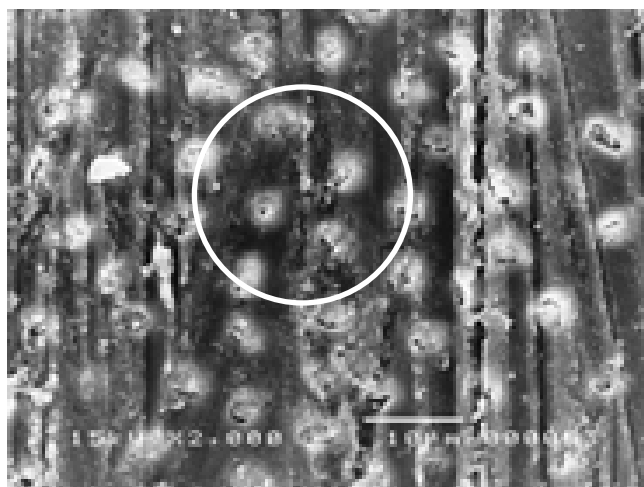

Figure 1 – SEM micrographs of the dentin surface ground with #600-grit SiC paper (Group 1 - Control) (x2,000). The dentin surface was covered by the smear layer. The dentinal tubules were closed
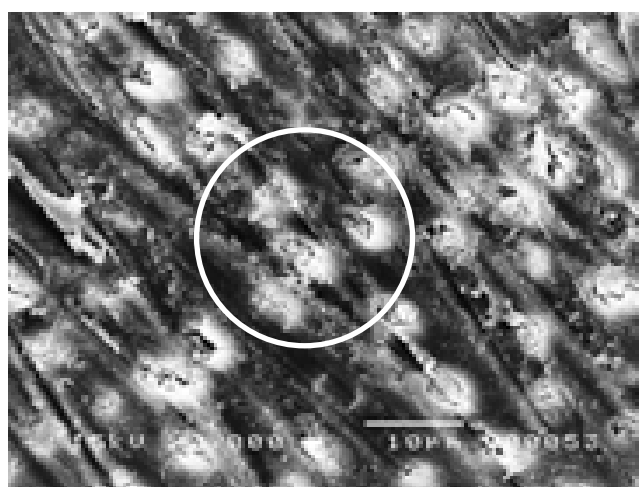

Figure 2 – SEM micrographs of the dentin surface treated with Gluma Desensitizer (Group 2) (x2,000). The dentinal tubules were partially closed. A coating layer with the desensitizing material was not visually confirmed under the low magnification of the observation
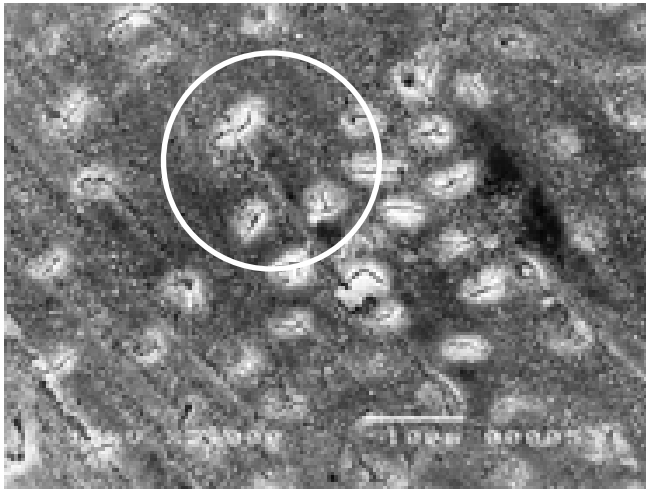
Figure 3 – SEM micrographs of the dentin surface treated with Super Seal (Group 3) (x2,000). Small precipitates were deposited on the intertubular dentin. The dentinal tubules were occluded by the depositions
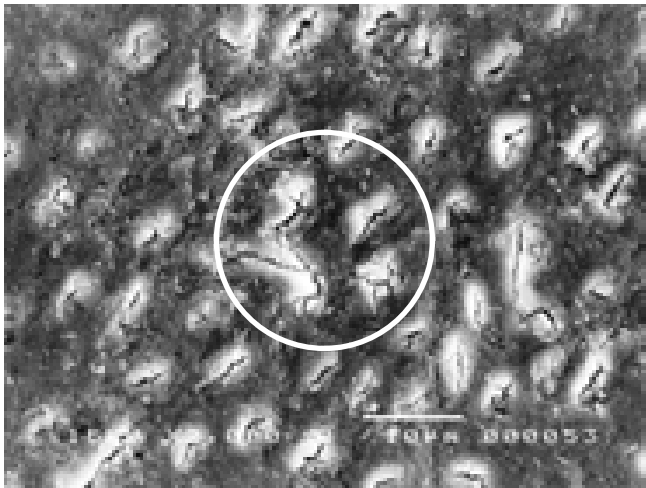

Figure 4 – SEM micrographs of the dentin surface treated with Teethmate Desensitizer (Group 4) (x2,000). The scratches on the dentin surface with #600-grit SiC paper disappeared. Depositions covered the intertubular dentin and occluded the dentinal tubules

Discussion

There is an understanding that the treatment of the dentin sensitivity mainly has being focused on the tubular occlusion [12, 19]. For the sealing of the dentinal tubules, there is the method of using a smear layer that can decrease the level of dentin permeability by 98%, when compared to dentin treated with acid [5]. Regarding the SEM analysis of the control group, the micrographs showed closed

dentinal tubules by the smear layer and 90% of cohesive failures in the resin cement (Figure 1).

The ED Primer II and Panavia F 2.0 contain MDP (10-methacrylate oxydecyl dihydrogen phosphate). The preservation of hydroxyapatite (HAp) within the submicron hybrid layer may serve as receptor for additional chemical bonding. MDP has this chemical bonding potential to calcium of residual HAp [30]. One may hypothesize that a self-etching effect of MDP is mandatory in order to deal with the smear layer resulting from tooth preparation and achieving shallow micromechanical interlocking through hybridization at dentin. In addition, the exposed HAp crystals that remain around collagen are expected to be particularly advantageous. They enable more intimate chemical interaction with the functional monomers on a molecular level and may help prevent or retard marginal leakage [27]. Keeping HAp around collagen may also better protect the collagen against hydrolysis and thus, prevent from early degradation of the bond [10, 21]. It is reported that ionic bond of MDP with calcium appeared to be hydrolytically stable, as compared with 4-META (4-methacryloxyethyl trimellitic acid) and Phenyl-P (2-metha-cryloyloxyethyl phenyl phosphoric acid). It was capable of forming strong ionic bonds with calcium due to the superficial dissolution of HAp induced by the MDP adsorption and subsequent deposition of MDP-calcium salt with a lower solubility [30].

In this study, a well-established μTBS protocol was used to investigate the adhesive luting of one indirect composite resin to dentin, using three different treatments of dentin desensitizer agents. In the present study, the control group [1] showed higher bond strength when compared to group [2] and [3].

Gluma Desensitizer is a glutaraldehyde-base substance that contains HEMA (2-hydroxyethylmethacrylate), causing the coagulation of the dentin fluid proteins in the dentinal tubules and plugs the tubules [6, 24]. In fact, glutaraldehyde causes coagulation of proteins inside the dentinal tubules, reacting with the albumin in the dentinal fluid, thus causing the precipitation of albumin and blockage of tubules. Therefore, Gluma reduces dentin permeability and disinfects the dentin at the same time. Especially when glutaraldehyde was combined with HEMA, bond strengths were improved [22].

Despite dentin precipitation after tooth preparation, the diffusion of monomers to dentin is likely to be accelerated by the presence of HEMA, because this product has the ability to

promote dentin adhesion and helps in facilitating diffusion of resin monomer and the formation of hybrid layer.

However, some in vitro studies have showed an inhibiting effect of Gluma on the bond strength of conventional resin cements, as a consequence of this coagulation of dentin fluid proteins and plugging the tubules [11, 28].

In the present study, a self-etching primer (ED Primer II) was used. Gluma did not contribute to improve the bond strength, because Gluma has a better mechanism of action when used to etch-and-rinse approach, when occurs the rewet or the restablishment of the collagen fibrils [24]. Regarding the SEM analysis, the micrographs showed semi-closed dentinal tubules, but it did not form a thick coating, and 50% mixed failures (between adhesive and cohesive in resin cement), 30% adhesive and 20% cohesive failures in resin cement failures (Figure 2).

Super Seal desensitizer agent is an oxalate-based substance. When acidic oxalates are applied to the dentin surface, they liberate calcium from the dentin to produce an insoluble calcium oxalate crystals that block dentinal tubules [11]. Hydrophilic calcium oxalate forms an insoluble crystalline layer on intratubular dentin matrix. It is acidic enough to remove the smear layer, and replace it with a layer of calcium oxalate crystals. To the present study, Super Seal showed the lowest bond strength mean among the groups. This might be because of the pH = 2.4 of ED Primer II, once there is an incompatibility of oxalate desensitizers agents with the acidic materials [29]. The solubility of calcium oxalate is affected by pH, since the anion is the conjugate base of a weak acid [14].

The SEM analysis showed precipitates that were deposited on dentin surface with limited penetration into the tubules (Figure 3), resulting in 90% of adhesive failures and a survival rate of only 40% of the specimens.

Teethmate Desensitizer is a calcium-phosphate desensitizer agent that contains TTCP (tetracalcium phosphate) and DCPA (dicalcium phosphate anhydrous) with water, whose combination could spontaneously transform to HAp. Previous reports [2, 25, 26] showed that the precipitates or crystallites were found in tubular orifices and on dentin surface, suggesting that the presence of HAp in the dentin substance could enhance the setting reaction of TMD and serve as a substrate for heterogeneous nucleation (deposition of crystals on foreign bodies, considered as potentiators of crystallization). The chemical bond might be formed between the material layer and the smear layer deprived dentin surface in a clinically reasonable time. According to the same authors, the effectiveness of TMD in forming a layer on dentin regardless of pretreatment and maintaining tubule occlusion should be attributed to its chemical composition. The mixing of the two components (TTCP and DCPA) provided a thick paste which could penetrate into the dentinal tubules by mean of scrubbing on dry dentin surface. This occluding effect resulted in the immediate dentinal permeability reduction and, hence, clinical sensitivity reduction could be expected [23]. To this study, TMD (group 4) showed the highest microtensile bond strength mean among all groups. This is because of this supposed chemical interaction created between the TMD and the smear layer and dentin, was possible to confirm in the SEM images.

The images showed a deposited layer of TMD covering the dentin surface and the occlusion of the dentinal tubules (figure 4). The TMD showed 50% cohesive failures in resin cement and 50% mixed failures (cohesive in resin cement and in TMD layer).

Based on the findings of the present study, the formulated null hypothesis was rejected, because the results differ significantly in μTBS among all groups tested. The current results indicate a relevant clinical significance because the TMD desensitizer is expected to be a new generation of material forming a stable calcium-phosphate rich layer and enhancing the calcification under oral conditions. It has biocompatible property, outstanding characteristic in dentinal tubule occlusion and favorable reduction in dentin permeability in the oral environment.

Conclusion

The efficacy of the desensitizers is material-dependent; Gluma Desensitizer and Super Seal decreased the μTBSs, however, Teethmate Desensitizer improved it.

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