

Influence of desensitizing procedures on adhesion of resin cements to dentin

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ABSTRACT

This study evaluated the effect of two desensitizer agents with different contents and Nd:YAG laser irradiation on the shear bond strength (SBS) of adhesive resin cements to dentin. New treatment options of Nd:YAG laser irradiation and tetracalcium phosphatecontaining agent applications were compared with routinely used glutaraldehyde-containing agents. One hundred and twenty human, caries-free premolars were embedded in acrylic resin blocks 2 mm below the cementoenamel junction. Buccal surfaces of the teeth were ground to expose dentin. The specimens were randomly assigned into three different surface treatments (desensitizing agents, Nd:YAG laser) and the control, then into three different adhesive resin cement applications (n = 10). Resin cements (Panavia SA cement (PA), Panavia SA cement with Clearfil Universal Bond (PACU), and Multilink N (MN)) were applied to the conditioned teeth surfaces using Teflon tubes. The specimens were thermocycled (5000 cycles, 5–55 \pm 1 °C, dwell time 30 s). The SBS test was performed in all groups. The results were submitted to two-way ANOVA and Tukey HSD tests (p < .05). Further, SEM analysis was performed on the dentin surfaces. SBS values were significantly difference among the surface treatment groups and also among adhesive resin cement groups (p < .05). The specimen cemented with PA showed lower SBS values than PACU- and MNapplied specimens. The highest SBS value was obtained in the Nd:YAG laser group which was cemented with PACU cement. The lowest SBS value was obtained in the control group which was cemented with PA cement. In addition, SEM evaluation revealed that desensitizing agents and Nd:YAG laser occluded dentin tubules.

Introduction

The short, sharp pain that patients experience when exposed dentin is brought into contact with thermal, tactile, osmotic, and mechanical stimuli (such as tooth brushing, sweet and sour foods, and hot or cold water) is defined as dentin hypersensitivity (DH).[1] Most DH cases are the result of abrasion, attrition, erosion, abfraction, gingival recession, and improper brushing habits.[2] DH may be seen in patients of any age, and women are affected

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Desensitizing agents; calcium–phosphate desensitizer; Nd:YAG laser; adhesive resin cement; universal bond; shear bond strength more often than men.[1] The prevalence of this condition has been reported to vary from 4 to 73%.[1,3] However, an increase in this prevalence is possible, as more adults retain their teeth into later life.[4]

Among the many theories proposed for the mechanism of DH, Brannstrom's hydrodynamic mechanism theory notes that in the presence of an external stimulus, fluid inside dentinal tubules move inwardly or outwardly, causing mechanical deformation of nerve endings at the pulp/dentine interface, which is perceived as a painful sensation.[5] When the dentinal tubules are blocked, fluid shifts and dentin sensitivity can be prevented. Desensitizing agents (DA) may function by desensitizing the nerves (potassium nitrate), precipitating proteins (glutaraldehyde, silver nitrate, zinc chloride), or by plugging dentinal tubules (sodium fluoride, potassium oxalate). Dentin adhesive sealers, lasers, and homeopathic medication may also be used for this purpose.[6]

Hydroxyethyl methacrylate (HEMA)/glutaraldehyde, the most commonly used desensitizer, is a dentin adhesive sealer, which also contains benzalkonium chloride and fluoride. Glutaraldehyde proteins inside the dentinal fluid coagulate and precipitate. Deep resinous tags are formed by HEMA and they occlude the dentinal tubules.[7]

Recently, desensitizers with calcium phosphate have gathered much attention due to their biocompability, and their superior ability to occlude dentinal tubules and reduce dentine permeability.[8,9] Teethmate Desensitizer (TMD; Kuraray Noritake Dental Inc., Tokyo, Japan) is a newly developed calcium–phosphate-containing material; tetracalcium phosphate (TTCP; $Ca_4(PO_4)_2O$) and dicalcium phosphate anhydrous (DCPA; $CaHPO_4$), which can spontaneously transform to hydroxyapatite (HA; $Ca_{10}(PO_4)_6(OH)_2$).[10] When TMD powder is mixed with water, the chemical reaction between particles takes place. Within seconds after mixing, calcium and phosphate ions are released, and this layer makes up HA crystals which physically occlude dentin tubules.[11]

Laser applications are another option for DH treatment. Laser desensitization has been suggested as an effective and rapid treatment option for terminating or reducing DH. Recently, neodymium-doped:yttrium, aluminum and garnet (Nd:YAG), CO_2 , galliumealuminumearsenide (diode), and erbium-doped:yttrium, aluminum and garnet (Er: YAG) have been commonly used lasers for the treatment of DH. The success of the treatment is mainly determined by the laser type and the parameters used.[12,13]

The effect of the Nd:YAG laser is accomplished by the occlusion or narrowing of dentinal tubules causing a form of nerve analgesia. After a short exposure to Nd:YAG laser, dentin fuses, and its surface becomes non-porous and glazed.[14] Followed by the application of the Nd:YAG laser, some physical changes in dentin such as a melted and re-solidified surface as well as craters, cracks, and globules are observed.[15–18]

Long-lasting dentin and luting material bond is a key to the success of the restorations' retention and avoiding microleakage, secondary caries, and tooth fracture.[19–21] Zinc phosphate, zinc polycarboxylate, conventional glass ionomer cements, and resin-based cements are some of the frequently used luting materials.[22–24] Adhesive resin cements can be used for the cementation of many types of fixed partial dentures.[21] Self-adhesive resin cements have the advantages of having simpler and less technique-sensitive pretreatment steps, eliminating application errors of the cementation process in fixed prosthodontics.[25] The impact of the desensitizer use before cementation with conventional or resin cements is still under debate. Some authors suggest that there is a significant loss in retention in cases using a desensitizer,[21,26–29] while some researchers have concluded that desensitizers

have no negative effect on crown retention.[30,31] However, there is a lack of literature comparing the bond strengths of various desensitizers used before the application of the newly developed self-adhesive resin cements.[32]

The aim of this study was to assess the impact of three different surface treatments and control on the shear bond strength (SBS) of adhesive resin cements to tooth surface. This study also examined dentin surfaces under a field emission scanning electron microscope (SEM) after the application of DAs. The null hypothesis was that DAs and laser irradiation procedures do not affect the SBS of adhesive resin cements to dentin surface.

Materials and methods

Dentin DAs and methods used in the study are shown in Table 1. Dentin surface with no treatment acted as the control group.

One hundred and twenty unrestored, caries-free, extracted human premolars were used in the study. The teeth were cleaned mechanically and stored in distilled water at room temperature until used. They were then embedded in custom made molds using autopolymerizing acrylic resin (Vertex Self Curing; Vertex-Dental, Netherlands) with the areas to be treated placed 2 mm above the resin surface. The specimens were then ground with a coarse grit diamond rotary cutting instrument (Mecatome T180, Presi, Grenoble, France) until the dentin surfaces were exposed. To standardize the dentin surface texture, the occlusal surface of each tooth was further ground off in a mechanical grinder (Minitech 233, Presi, Grenoble, France) with 600-grit silicon carbide abrasive paper under running water for 30 s.[33] The specimens were randomly assigned into the surface treatments (Gluma, TMD, Nd:YAG laser irradiation, and the control) and then into two different adhesive resin cements and a universal bond which constituted the 12 test groups (n = 10).

Applied resin cement groups were as follows:

PA: Self-adhesive resin cement (Panavia SA cement,) was applied without bonding agent application.

Brand Name (Batch No.)	Chemical composition	Application steps as recommended by the manufacturer	Manufacturer
Gluma 10,096	Glutaraldehyde (5%), distilled water, HEMA (35%)	 Apply on dried dentin and leave for 30 to 60 s. Apply air until the fluid film has disappeared. Rinse with water. 	Heraeus Kulzer, Hanau, Ger- many
Nd:YAG laser	N/A	 The dentin surface was irradiated with a pulse 10 Hz-1 W, with a total irradiation time of 60 s to simulate clinical manipulation. 	Lightwalker, Foto- na, Ljubljana, Slovenia
Teethmate Desensitiz- er (TMD) 041,117	Powder: Tetracalcium phos- phate, Dicalcium phosphate anhydrous Liquid: Water, Preservative	 Mix powders and water within 30 s, apply the slurry to the affected area using the applicator brush, and then let it penetrate sufficiently by rubbing the area for more than 30 s. Rinse the excess slurry with water spray or by having the patient rinse 	Kuraray Noritake Dental Inc, Tokyo, Japan

Table 1. The brand names, batch numbers, chemical compositions, application steps, and manufacturers of the test groups utilized in the study.

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PACU: Self-adhesive resin cement (Panavia SA cement,) was applied with self-etch bonding agent (Clearfil Universal Bond) application.

MN: Self-etching resin cement (Multilink N) was applied.

The DAs and Nd:YAG laser were applied to the dentin surfaces according to the manufacturer's instructions (Table 1). A steel mold with a hole in the center (3 mm diameter and 2 mm deep) was used for the application of the resin cements (Table 2) to the dentin surfaces according to the manufacturer's instructions.

The resin cements were then cured by use of an LED-unit (BAOPTIMA 10, B. A. International, Northampton, U.K., 1200 mW/cm²) with a curing time of 20s. After removal of the mold, the specimens were subjected to thermocycling for 5000 cycles between 5 °C and 55 °C in deionized water. The dwelling time at each temperature was 30 s, and the transfer time from one bath to the other was 2 s according to the International Organization for Standardization (ISO NO:11405).[34] The SBS testing was performed with a universal testing machine (Shimadzu AG-50 kNG, Kyoto, Japan) at a crosshead speed of 0.5 mm/ minute. Results were expressed in megapascal (MPa) values.

Means and standard deviations of bond strengths were calculated, and mean values were compared by two-way analysis (surface treatments and adhesive resin cements) of variance (ANOVA; SPSS 12.0, SPSS Inc., Chicago, IL, U.S.A.) followed by a multiple comparison test performed with a Tukey's honestly significant difference (HSD) test (p < .05).

To investigate the changes in the dentinal tubules after applying different surface treatments, one specimen from each group was air-dried overnight and gold sputtered with a sputter coater (S150B, Edwards, Crawley, England), and examined by means of a field emission SEM (JSM-6335F, JEOL Ltd., Tokyo, Japan) at 15.0 or 20.0 kV. There was no resin cement applied. For visual assessment, the SEM photomicrographs were developed at 1000× magnification.

Results

The SBS test results of two-way ANOVA revealed significant differences among the surface treatment groups and also among adhesive resin cement groups (p < .05; Table 3). The mean SBS values and the differences among the groups are shown in Table 4.

There were significant differences among the surface treatment groups. The specimens cemented with PA showed lower SBS values than PACU- and MN-applied specimens (p < .05). However, there were no significant differences between PACU- and MN-applied specimens (p > .05).

The control group's specimens showed significantly lower SBS values than Gluma and Nd:YAG laser groups (p < .05). However, there was no significant difference between Nd:YAG laser and Gluma group specimen (p > .05). The specimens cemented with TMD did not show any significant differences from other groups (p > .05).

The highest SBS value was obtained in the Nd:YAG laser group which was cemented with PACU cement (20.8 \pm 4.52 MPa). The lowest SBS value was obtained in the control group which was cemented with PA cement (5.96 \pm 2.9 MPa) (p < .05).

SEM evaluation

The microstructures of the dentin surfaces treated with two different types of DAs and Nd:YAG laser are shown in Figure 1. The specimens treated with Gluma (a) showed

Brand Name (Batch No.)	Chemical composition	Application steps as recommended by the manufacturer	Type	Manufacturer
Multilink N (MN) T12873	Automix: Dimethacrylate, HEMA, barium glass, ytterbium trifluoride, spheroid mixed oxide Primer A: Aqueous solution of initiators Primer B: HEMA, phosphonic acid, acrylic acid monomers	 The tooth is rinsed with water and dried with air. Primer A and B are mixed with same amount. The mixture is applied on the tooth surface for thirty seconds. It is dried with mild air. Later on it is dispenced into the crown and taken by ligth- 	Self-etch adhesive, dual-cure resin cement	lvoclar, Vivadent, Liechtenstein
Panavia SA cement (PA) 0015BE	PASTE A: MDP, Bis-GMA, TEGDMA, Hydro- phobic aromatic dimethacrylate, Silanated barium glass filler, Silanated colloidal silica, dl-Camphorquinone, Benzoyl peroxide, Ini- tiators. PASTE B: Bis-GMA, Hydrophobic aro- matic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Silanated barium glass filler,	 cure for twenty seconds. Clean the crown and cavity. Dispence Panavia SA Cement into the crown. Seat the crown onto the abuttment allowing excess cement to seep out. Tack light-ture for 2–5 s. Simply start removing the excess cement. Wait a total of five minutes (from seat to finish) for final cure. 	Self adhesive, dual-cure resin cement	Kuraray, NY, America
Clearfil Universal Bond 100,004	Silanated colloidal silica, Surface treated sodium fluoride, Accelerators, Pigments MDP, Bis-GMA, HEMA, Hydrophilic aliphatic dimethacrylate, Colloidal silica dl-Cam- phorquinone, Silane coupling agent, ethanol, Accelerators, Initiators, Water	 The tooth is bonded and rubbed by universal bond for 10 s. It is dried by blowing mild air. Panavia SA cement is dispenced into the crown and then cementaion is applied according to the manufecturer's instructions. 	Total etch, Self etch, Selective etch	Kuraray, NY, America

Abbreviation: Bis-GMA: bisphenol A diglycidylether methacrylate; MDP: 10-Methacryloyloxydecyl dihydrogen phosphate; TEGDMA: triethylene glycol dimethacrylate.

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Table 3. Two-way ANOVA results.

	Sum of squares	df	Mean square	F	Sig.
Surface treatment groups	229.28	3	76.43	4.0	.010
Adhesive resin cements groups	2503.61	2	1251.80	65.57	.000
Between groups	361.40	6	60.23	3.16	.007

Table 4. Mean and SD values for shear bond strength (MPa) Same supercripts in same column and row show not statistically significant differences.

Adhesive resin cements and surface treatments	Gluma (Mean ± SD)	Nd: YAG Laser (Mean ± SD)	Teethmate desensi- tizer (Mean ± SD)	Control (Mean ± SD)
PA	14.5 ± 4.71 ^{bc}	10.84 ± 3.37^{ab}	7.3 ± 4.08^{a}	5.96 ± 2.9 ^a
PACU	19.06 ± 6.51°	$20.8 \pm 4.52^{\circ}$	20.17 ± 3.63 ^c	16.6 ± 5.4^{bc}
MN	17.88 ± 3.33 ^c	$20.74 \pm 3.56^{\circ}$	$20.16 \pm 4.07^{\circ}$	19.28 ± 5.01°

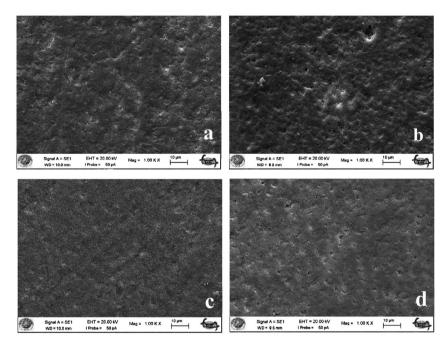


Figure 1. Scanning electron microscopic views of the dentin surface after treatment with the desensitizing agents: a, Gluma; b, Nd: YAG laser; c, TMD; d, control.

completely occluded dentinal tubules. In the specimens treated with Nd:YAG laser (b) and TMD (c)-treated group, the dentine tubules were occluded as well. However, TMD completely covered and obliterated the tubules. In the control group specimens (d), exposed dentin tubules were observed.

Discussion

The hypothesis of the present study was that DAs and laser irradiation procedures do not affect the SBS of self-etch, self-adhesive resin cements to dentin surface. However, the study

concluded that the SBS was increased after the application of the DAs. Therefore, the null hypothesis was rejected.

Adhesive cementation is a more technique-sensitive procedure compared to conventional cementation, and clinical success may be overridden by the technical challenges involved. Self-adhesive resin cements can be a solution to such difficulties.[32] Clearfil Universal Bond, used in the presented study, is a one-bottle, newly produced universal bond which can be used with self-adhesive resin cement. When used with Panavia SA Cement, no additional primer or activator is needed. In the current study, the combined application of the Panavia SA Cement with Clearfil Universal Bond significantly increased the SBS values.

Because of its chemical composition, TMD is very successful at forming a layer on dentin and at occluding tubules regardless of pretreatment. TMD consists majorly of TTCP and DCPA. When scrubbed on dry dentin surface, thick paste, which is the mixture of the two components, can penetrate into the tubules. This occluding mechanism reduces the dentinal permeability and clinical hypersensitivity.[35] However, in the results of current study, the SBS values of the TMD group were not significantly different from the other groups. In the literature, there is a lack of study on this newly introduced desensitizer on the market.

For a self-etching/self-adhesive system, the monomer infiltration into the dentin and the formation of a resin-infiltrated layer is essential.[19] Self-etching/self-adhesive resin conditions and primes the enamel and dentinal surface without any need of rinsing. Therefore, the remnants of DAs cannot be removed from the dentinal surface, and they may cause a decrease in the bond strength.[36] Only a few studies have evaluated the impact of DAs on the self-adhesive resin-dentin interface.[21,28,29,37] Huh et al. [21] evaluated the influence of four different DAs (SuperSeal; Copalite Varnish, Cooley and Cooley, Houston, TX, U.S.A.; MS Coat; and Gluma) on the bond strength of a self-etching adhesive resin cement to dentin. According to their study, the Gluma group showed a lower bond strength value compared to SuperSeal and the control groups. Previously, it had been found that the bond strength of resin cement was mostly influenced by the HEMA concentration, with a maximum of 35% and nearly independent of the glutaraldehyde in Gluma is not able to cross-link mineralized dentin. HEMA reduces the surface tension of water and enables monomer diffusion into dentin.[40]

However, Sailer et al. [41] concluded that glutaraldehyde-containing dentin desensitizers and bonding agents (Gluma and Syntac) were likely to increase the SBS of the self-adhesive resin cement (RelyX U200) to dentin. Additionally, they found that the bond strength was increased when glutaraldehyde/HEMA-containing desensitizers were used before self-adhesive cement luting.[42] The glutaraldehyde reacts with protein and produces a precipitate on the dentin surface, while HEMA accelerates the diffusion of monomers into dentin regardless of such precipitation.[19,30,43,44] Dündar et al. [46] showed that the application of two brands of adhesive cement in combination with the same DA caused increased SBS. In the researchers' opinion, HEMA induces rehydration, allowing for the penetration of primer into dentin and causing an increase in the bond strength.[46] In the results of the current study, the HEMA-containing DA (Gluma) group showed a higher bond strength than the control group.

Desensitizing treatment with Nd:YAG laser has also been investigated. This kind of laser irradiation causes a reduction or complete obliteration of the dentinal tubule lumen [47] and closure of exposed dentin tubules,[48] as shown in many of SEM inspections. When self-etch and etch-and-rinse systems are used after Nd:YAG laser irradiation, the bond strength usually decreases.[49,50] This might be caused by the tubule obliteration when self-etching

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primer is used on the interface of dentin and composite resin. Obliterated tubules may block the penetration of resin monomer and the resin tag formation.[49] However, in a study that evaluated the influence of five different DAs(Gluma, Aqua-Prep F, Bisblock, Cervitec Plus, Smart Protect) and Nd:YAG laser pre-treatment on microtensile bond strength of self-adhesive resin cement to dentin, the results showed higher bond strength values with the Gluma group.[32] In the results of the current study, there were no significant differences between the Nd:YAG laser-irradiated group and the Gluma group.

The present study has several limitations, making it difficult to compare results directly with clinical studies. Resin cement thickness tested in the current study was 2 mm, which is 25–150 µm in the clinical situations. Future investigations should be performed using different resin cement thicknesses for greater clinical relevance. Another limitation of the present study was that it was performed under *in vitro* conditions, but the results still provide guidance for clinicians. The differences among the materials and methods used make the results difficult to compare. In the present study, the effect of two different DAs (of all the types on the market today, with one newly introduced product) and Nd:YAG laser pretreatment on the bonding compatibility of adhesive resin cements were compared. Hence, this protocol is believed to make a more conclusive statement regarding which desensitizing material to use with adhesive resin cements. Also, to the authors' knowledge, there is no study conducted with all the different kinds of DAs combined with these cements. Future clinical studies are required to evaluate this combined usage of DAs and adhesive resin cements.

Conclusion

Within the limitations of this study, it is concluded that:

- (1) The specimen cemented with PA showed lower SBS values than the PACU- and MN-applied specimens.
- (2) The highest SBS value was obtained in the Nd:YAG laser group which was cemented with PACU cement. The lowest SBS value was obtained in the control group which was cemented with PA cement.
- (3) SEM evaluation revealed that DAs and Nd:YAG laser occluded dentin tubules.

Disclosure statement

No potential conflict of interest was reported by the authors.

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