

## Effect of previous desensitizer and rewetting agent application on shear bond strength of bonding systems to dentin

### *Efeito da aplicação prévia de dessensibilizante e reidratante na resistência adesiva por cisalhamento de sistemas adesivos a dentina*

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#### **ABSTRACT**

This study evaluated the effect of desensitizer and rewetting agent on dentin shear bond strength. One hundred thirty five bovine incisive teeth had their buccal surfaces ground down to produce a flat superficial dentin surfaces and received the following treatments: G1- One Step Plus (OSP) and resin cylinder adhesive fixation (RI); G2- Gluma One Bond (GOB) and RI; G3- Single Bond (SB) and RI; G4- Aqua-Prep (AP) + OSP and RI; G5- Gluma Desensitizer (GD) + GOB and RI; G6- GD + SB and RI; G7- GD + OSP and RI; G8- AP + GOB and RI; G9- AP + SB and RI. The specimens were stored at 37°C and 100% humidity for 24 hours and a shear bond test was performed with a mechanical testing machine, at a crosshead speed of 0.5 mm/min. The data were submitted to one-way ANOVA followed by the Tukey test ( $p < 0.05$ ). The results (MPa) were: G1: 10.75(2.64)a; G2: 10.28(2.58)a; G3: 11.63(4.59)a; G4: 10.93(4.88)a; G5: 10.15(3.95)a; G6: 11.82(4.14)a; G7: 9.85(2.15)a; G8: 5.48(1.94)b; G9: 10.62(2.83)a. The resistance of the GOB adhesive system was negatively affected by AP application. The use of desensitizer and rewetting agent does not compromise the bond strength when they are compatible with the adhesive system used.

#### **UNITERMS**

Dentine hypersensitivity, dentin-bonding agents, desensitizers

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#### **INTRODUCTION**

The hydrodynamic theory of dentin sensitivity is now widely accepted. Dentin hypersensitivity is thought to be caused by displacement of dentin fluid within the dentin tubules<sup>2</sup>. The dentin tubules of hypersensitive teeth are open, more numerous and larger than in normal teeth<sup>19</sup>.

Dentin sensitivity presents a challenge to the dentist. Modern treatments for hypersensitive teeth are intended either to reduce tubular fluid movements by reducing dentin permeability or to reduce the excitability of intradental nerves with neurally active agents<sup>13</sup>. In addition, tubule occlusion is thought to reduce subjacent pulpal inflammation.

A variety of materials, such as calcium hydroxide, cavity varnishes, topical fluorides, fluoride iontophoresis, laser irradiation, strontium chloride and potassium nitrate dentifrices have been used in an attempt to alleviate dentin sensitivity<sup>12</sup>. Oxalates, glutaraldehyde, benzalkonium chloride and dentin bonding agents with and without resin-based composite are some of the materials currently being used for the treatment of this condition<sup>8</sup>. Oxalates and dentin bonding agents have been evaluated for their influence on dentin permeability. Ferric oxalate reduces dentin permeability to nearly 35% of the original smear layer values<sup>14</sup>. Glutaraldehyde reacts with serum albumin in the dentin fluid by coagulation, thus counteracting the hydrodynamic mechanism of dentin hypersensitivity<sup>4</sup>.

Combining a resin adhesive with a previous application of a desensitizing agent seems to be contradictory at first sight, since effective adhesives are expected to seal the etched dentin surface by intertubular and peritubular hybridization and by resin tag formation in the opened dentin tubules<sup>5</sup>. This seal prevents fluid shifts across the tubules occurring in response to mechanical, thermal or osmotic stimuli. However, if the tags formed within the dentin tubules were too long, it could cause post-operative pain. With the continued development of bonding systems with improved physical properties<sup>3</sup> that seal dentinal tubules more effectively, desensitizing agents may be a useful treatment option in the management of persistent dentin hypersensitivity<sup>17</sup>. Although the influence of chlorhexidine<sup>6</sup>, formalin cresol and hydrogen peroxide/sodium hypochlorite<sup>11</sup> on dentin bonding has been reported, little information is available concerning the influence of desensitizers used immediately before bonding procedures. It is hypothesized that the use of desensitizers does not influence the bond strength

with different adhesive systems. This hypothesis was tested by determining the shear bond strengths of three bonding systems applied to bovine dentin in combination with desensitizer and rewetting agent.

## MATERIALS AND METHODS

One hundred thirty five bovine incisors were extracted and the roots were removed from the crown at the cementum-enamel junction. After embedding the crowns in resin, the labial surface of each tooth was ground on a water cooled mechanical grinder with 600-grit silicon carbide abrasive papers (Norton, Campinas, SP, Brazil) in order to get a flat superficial dentin sample, to standardize smear layer formation. The teeth were stored at 4°C in a solution of 0.2 % thymol for a week. Adhesive masking tape with a 3.0mm diameter hole was placed on the dentine surface to control the bonding area. Specimens were randomly divided into nine groups (n=15). Table 1 and 2 give descriptions of the materials used.

**Table 1 – Chemical formulations of the bonding agents used**

Bonding Agent	Etching gel	Chemical Composition	Solvent
Gluma One Bond	35% PhA*	UDMA*, HEMA*, 4-META*	Acetone
Single Bond	35% PhA*	BisGMA*, HEMA*, dimetacrylates, polyalkenoic acid copolymer, water.	Ethanol
One Step	35% PhA*	BisGMA*, BPDMA*, HEMA*	Acetone

\* PhA: Phosphoric acid; UDMA: urethane di-methacrylate; HEMA: 2-hidroxyethylmetacrylate; 4-META:4-methacryloxyethyl trimellitate anhydride; BisGMA: bis-phenol- A- diglycidylmethacrylate; BPDMA: Biphenyl dimethacrylate

**Table 2 – Chemical formulations of the desensitizing and rewetting agents used**

Desensitizing Agent	Chemical composition
Gluma Desensitizer	HEMA* 36%, Glutaraldehyde 5%, Purified water, Mequinol.
Aqua Prep	HEMA* 35%, Purified water 65%.

\*HEMA: 2- hidroxyethylmetacrylate.

The dentin surface of samples was conditioned with 35% phosphoric acid gel for 15s and was rinsed for the same time with air-water spray. The etched dentin surface was gently dried with absorbent paper, to produce a visibly moist and not desiccated surface.

- *Group 1:* One-bottle adhesive system (Single Bond, 3M-ESPE, St. Paul, MN, USA) (SB), was applied with a disposable brush, waiting for 20s before the second coat application to evaporate the solvent and was then light-cured for 20s (SB control group).
- *Group 2:* On conditioned dentin, rewetting agent (Aqua-Prep, BISCO, Schamburg, IL, USA) was applied according to the manufacturer's instructions, and SB, was applied in the same way as in Group 1.
- *Group 3:* On conditioned dentin, desensitizer agent (Gluma Desensitizer, Heraeus Kulzer, Dormagen, Germany) was applied according to the manufacturer's instructions before SB application.
- *Group 4:* One-bottle adhesive system (One Step plus, BISCO, Schamburg, IL, USA) (OSP), was applied with a disposable brush, in two consecutive coats, waiting for 20s before the second coat application to evaporate the solvent and then light-cured for 20s (OSP control group).
- *Group 5:* Aqua-Prep was applied according to the manufacturer's instructions before OSP adhesive system application.
- *Group 6:* Gluma Desensitizer was applied according to the manufacturer's instructions before OSP adhesive system application.
- *Group 7:* One-bottle adhesive system (Gluma One Bond, Heraeus Kulzer, Dormagen, Germany) (GOB), was applied with a disposable brush, in two consecutive coats, waiting for 20s before the second coat application to evaporate the solvent and then light-cured for 20s (GOB control group).

- *Group 8:* Aqua-Prep was applied according to the manufacturer's instructions before GOB adhesive system application.
- *Group 9:* Gluma Desensitizer was applied according to the manufacturer's instructions before GOB adhesive system application.

The indirect restorations were made with hybrid resin (TPH Spectrum – Dentsply De Trey, Konstanz, Germany) in 2 increments using a teflon matrix with a diameter of 3mm, and was light-cured for 40s using a halogen light-curing unit XL 3000 (3M-ESPE, St Paul, USA). Indirect restorations were sandblasting with 50 $\mu$ m aluminum oxide at 4 bars pressure and silane coupling agent (Ceramic Primer, 3M-ESPE, St Paul, USA) was applied. Next, they were fixed with dual cure resin cement (Rely X, 3M-ESPE, St Paul, USA) under a standard weight of 500g and light-cured on two faces for 40s. Specimens were stored in distilled water at 37°C immediately after bonding, for 24hs. Specimens were shear loaded using a Mechanical Testing Machine (EMIC DL 2000, São José dos Pinhais, Brazil) at a crosshead speed of 0.5mm/minute. The knife device was applied parallel to and approximately 0.2 from the dentin surface, perpendicular to the composite restoration. Shear bond strengths were calculated by relation of load (Kg) by bonded surface area (mm<sup>2</sup>). Mean shear bond strength values were expressed in MPa and data were analyzed by one-way ANOVA, followed by Tukey test ( $p < 0.05$ ).

## RESULTS

Data presented a normal and homogeneous distribution, which enabled parametric analyses to be made. Two-way ANOVA revealed that there was significant difference in the bond strength of the different groups. Thus, the interaction was analyzed by the multiple range Tukey test, and the shear bond strength values are presented in Table 3.

**Table 3 – Means standard deviation for shear bond strength and statistical ranking by Tukey test (p<0.05)**

Adhesive System	Previous Treatment	Mean ± S.D (MPa)
Single bond	without desensitizer	10.75 (2.64)a
	Aqua-Prep	10.28 (2.58)a
	Gluma Desensitizer	11.63 (4.59)a
One Step Plus	without desensitizer	10.93 (4.88)a
	Aqua-Prep	10.15 (3.95)a
	Gluma Desensitizer	11.82 (4.14)a
Gluma One Bond	without desensitizer	9.85 (2.15)a
	Aqua-Prep	5.48 (1.94)b
	Gluma Desensitizer	10.62 (2.83)a

(Different letters indicate significantly different means for each treatment.)

No statistically significant difference was found among the shear bond strength values of bonding systems applied alone and when they were associated with Gluma Desensitizer. The only association that suffered a statistically significant decrease in adhesion values was Gluma One Bond + Aqua-Prep.

## DISCUSSION

While enamel is predominantly composed of an inorganic, homogenous phase, the organic, tubular and heterogeneous composition of dentin renders it more complex as a bonding substrate<sup>15</sup>. Since dentin is intrinsically wet, the development of hydrophilic resin primers dissolved in organic solvents was an important achievement in dentin bonding technology. These solvents, commonly acetone and ethanol, solubilize the resins and facilitate the embedding of the exposed collagen fibrils with resin. The solvents act as carriers, delivering the resin components where they are necessary<sup>15</sup>.

HEMA, a methacrylate derivative, is a component of many current hydrophilic adhesive systems due to its ability to promote dentin adhesion<sup>18</sup>. HEMA is also used as a monomer in collagen-HEMA hydrogels in a variety of biomedical applications. It has been reported

that HEMA infiltrates into the intertubular dentin during adsorption, thus facilitating the diffusion of resin monomers and the formation of the hybrid layer<sup>10</sup>. The potential chemical reaction between the ester function group of HEMA and dentin collagen has been described<sup>18</sup>. The hydroxyl group in HEMA associates with the exposed collagen due to its polar, hydrophilic nature. On the other hand, the homopolar methacrylate group of the HEMA molecule has a high affinity for hydrophobic monomers<sup>1</sup>. This ambiphilic nature makes HEMA a very convenient component of adhesive resins, since these materials act as a link between the hydrophilic dentin surface and the hydrophobic restorative resins.

The results of this study indicated that previous application of Aqua-Prep associated with Gluma One Bond resulted in a statistically significant reduction in shear bond strength values. Probably, this occurred due the composition of the two products. Aqua-Prep is composed of 65% water and 35% HEMA, and HEMA is the main absorption path of Gluma One bond. The high concentration of hydrophilic components, due the combination of Aqua-Prep and Gluma One Bond, decreased the bond strength values, because this made it difficult for the hydrophobic component of the bond agent to penetrate, which is responsible of the hybrid layer resistance.

The use of glutaraldehyde as a pre-treatment agent before bonding is advantageous because of the antibacterial and desensitizing effects<sup>7</sup>, due the coagulation of dentin fluid proteins within dentin tubules.

The application of Gluma Desensitizer on acid-etched dentin has been shown to improve the efficacy of dentin bonding systems in vitro. The enhanced bond strengths observed could be related to the covalent cross-linking between collagen and glutaraldehyde. HEMA-collagen interaction has never been studied at a biochemical level<sup>16</sup>.

The interaction between HEMA, glutaraldehyde and collagen components was studied by Munksgaard<sup>9</sup>(1990). This author proposed that amino group-containing substances in dentin react with glutaraldehyde and start the formation of a HEMA-polymer. It is conceivable that the ε-amino groups in these amino acids of a collagen molecule react with glutaraldehyde-derived aldehyde, forming reducible Schiff based cross-links<sup>16</sup>.

In spite of there being no statistically significant difference, this explains the increase in bond

strength values of the groups where the Gluma Desensitizer was used in association with bond systems.

## CONCLUSION

The hypothesis of this study was partially accepted. According to the methodology used and to the results presented by this study, it is possible to conclude that the shear bond strength of the Gluma One Bond adhesive system was negatively affected by previous application of Aqua-Prep; there was no statistically significant difference in shear bond strength values of the bond systems applied alone and the use of a desensitizing agent and rewetting agent did not compromise the bond strength when the two products are compatible.

Therefore, desensitizer application and rewetting agent in association with a compatible adhesive system can be used as a dentist's ally when endeavoring to control hypersensitivity, without interference in the bond treatment system.

## RESUMO

*Este trabalho avaliou o efeito de dessensibilizante e agente reidratante na resistência adesiva ao cisalhamento à dentina. Cento e trinta incisivos bovinos tiveram suas superfícies vestibulares lixadas produzindo uma superfície plana de dentina que receberam os seguintes tratamentos: G1- One Step Plus (OSP) e cimentação adesiva do cilindro de resina (CR); G2- Gluma One Bond (GOB) e CR; G3- Single Bond (SB) e CR; G4- Aqua-Prep (AP) + OSP e CR; G5- Gluma Desensitizer (GD) + GOB e CR; G6- GD + SB e CR; G7- GD + OSP e CR; G8- AP + GOB e CR; G9- AP + SB e CR. Os espécimes foram armazenados em 37°C e umidade de 100% por 24 horas e o teste de resistência adesiva foi executado com máquina de ensaio mecânico, em uma velocidade de 0.5 mm/min. Os dados foram submetidos a análise de variância seguido pelo teste de Tukey ( $p < 0.05$ ). Os resultados (MPa) foram: G1: 10,75(2,64)a; G2: 10,28(2,58)a; G3: 11,63(4,59)a; G4: 10,93(4,88)a; G5: 10,15(3,95)a; G6: 11,82(4,14)a; G7: 9,85(2,15)a; G8: 5,48(1,94)b; G9: 10,62(2,83)a. A resistência adesiva do sistema adesivo GOB foi negativamente afetada pela aplicação do AP. O uso de dessensibilizante e agente reidratante não compromete a resistência adesiva quando são compatíveis com o cimento resinoso adesivo utilizado.*

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*Hipersensibilidade dentinária, agentes dentinários, dessensibilizantes*

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