Original Article

Evaluation of biaxial flexural strength and modulus of filled and unfilled adhesive systems

Avaliação da resistência flexural biaxial e módulo de flexão de sistemas adesivos

Abstract

Purpose: The purpose of this study was to evaluate the flexural strength and modulus of two adhesive systems using biaxial flexural strength test.

Methods: The bonding agents (Pentron Clinical Technologies) tested were: Bond 1 (B1) and NanoBond (NB). Thirteen disks (6.1 mm diameter and 0.6 mm thick) were prepared with adhesive solutions of each bonding agents using Teflon molds. The adhesive solutions within the molds were light-activated with XL 3000 curing unit (3M ESPE) for 10 s on both sides. The disks were stored for 10 days and were tested in a universal testing machine (1.27 mm/min – Instron 5844). Data were statistically analyzed by one-way ANOVA (α=0.05).

Results: The mean values of fracture strength ($\pm$SD) for adhesive systems were (in MPa): B1- 89.7$\pm$7.6 e NB- 131.1$\pm$9.5. Modulus means ($\pm$SD) were (in MPa): B1- 1999.9$\pm$258.4 e NB- 2314.5$\pm$271.0.

Conclusion: The filled adhesive system (NB) exhibited higher strength and flexural modulus means than the unfilled adhesive B1.

Key words: Dentin-bonding agents; flexural strength; flexural modulus

Resumo

Objetivo: O objetivo deste estudo foi avaliar a resistência flexural e o módulo de flexão de dois sistemas adesivos, através de ensaio de resistência flexural biaxial.

Metodologia: Os adesivos (Pentron Clinical Technologies) estudados foram: Bond 1 (B1) e NanoBond (NB). Treze discos de cada adesivo foram preparados com dimensões aproximadas de 6,1 mm de diâmetro e 0,6 mm de espessura. Os discos de adesivos foram confeccionados utilizando-se moldes de teflon e fotopolimerizados com aparelho XL 3000 (3M ESPE). Após armazenamento por 10 dias, os discos foram testados em máquina universal de ensaio (Instron 5844), com velocidade de 1,27 mm/min. Os dados foram submetidos à análise de variância (1 fator) ao nível de significância de 0,05.

Resultados: Os valores médios ($\pm$DP) de resistência flexural para os adesivos foram (em MPa): B1- 89,7$\pm$7,6 e NB- 131,1$\pm$9,5. Os valores médios de módulo flexural ($\pm$DP) foram (em MPa): B1- 1999,9$\pm$258,4 e NB- 2314,5$\pm$271,0.

Conclusão: O adesivo contendo partículas de carga (NB) mostrou maiores valores de resistência flexural e módulo de flexão que o adesivo B1.

Palavras-chave: Adesivos dentinários; resistência flexural; módulo flexural

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Introduction

The composition of adhesive systems has constantly changed in the last few years. Some of the most important changes in composition include the addition of inorganic fillers and anti-bacterial agents (1-4). Some studies demonstrated the advantages of filler addition on the adhesive composition regarding hybrid layer formation and long-term bond strength to tooth tissues (5,6).

It has been demonstrated the importance of filler content on the improvement of resin composite physical properties (7,8). The bonding agents are basically composed of monomers and organic solvents, such as water, ethanol, or acetone, without filler particles, and other components responsible for the polymerization start (9). Therefore, it would be reasonable to speculate that filler addition improves the mechanical properties of bonding agents as observed in resin composite when filer is added to or had its content increased. However, filler addition may affect the chemical balance of the adhesive components, resulting in changes in hybrid layer properties and in the clinical performance of the bonding agent, as a consequence.

In vitro studies are required to provide further information regarding the differences in mechanical properties of bonding agents with and without filler particles. Therefore, the aim of the current study was to evaluate the flexural strength and modulus of two bonding agents, which have similar compositions except the presence of filler particles in one. The research hypothesis evaluated in the study was that the filler particle addition alters the flexural strength and modulus.

Methods

Two bonding agents from the same manufacturer were used: Bond 1 and NanoBond (Pentron Clinical Technologies, Wallingford, CT, USA). The bonding agent compositions and lot numbers are described in Table 1. The bonding agents were applied to Teflon molds using microbrushs provided by the manufacturer, to originate 0.6 mm thick disks having 6.1 mm in diameter. The bonding agents were air dried to evaporate solvent as recommended when they are applied to dentin surfaces. The Teflon molds containing the bonding agents were protect from light exposure for 5 min at 37°C to avoid polymerization, and were then exposed to light-activation for 10 s (power density: 670 mW/cm²; XL 3000; 3M ESPE, St. Paul, MN, USA).

As the specimen thickness was higher than that produced by the bonding agent when applied to the dentin surface, additional 10 s light exposure was performed on the other side of the specimen immediately after it was removed from the mold. The specimens were wet ground with 800-grit SiC paper to create flat surfaces and to adjust thickness. Thirteen specimens were created for each experimental group (n=13) and had their dimensions constantly measured (diameter and thickness) using a digital caliper (Starret 727-6/150, Itu, SP, Brazil).

The specimens were stored in dry and dark conditions for 10 days and were tested in a Universal Testing Machine (5844, Instron Corp., Canton, MA, USA). Each specimen was individually positioned on a specific device attached to the universal testing machine to evaluate the biaxial flexural strength at 1.27 mm/min until failure occurred. The results of flexural strength and modulus were submitted to one-way ANOVA at a pre-set alpha of 5%.

Table 1. Composition and lot number of the tested bonding agents.

Bonding agent	Composition	Lot number
Bond 1	Trimethylolpropane trimethacrylate pyromellitic dianhydride and glycerol dimethacrylate; Ethyl Alcohol; Acetone Unknown; Pyromellitic Dianhydride; H_2O; Photocuring system.	129121
Nano-Bond	Trimethylolpropane trimethacrylate pyromellitic dianhydride and glycerol dimethacrylate; Ethyl Alcohol; Acetone Unknown; Pyromellitic Dianhydride; H_2O; Photocuring system; POSS (Polyhedral oligomeric silsesquioxane).	126356

Results

The flexural strength and modulus values are displayed on Table 2. The ANOVA detected significant difference between bonding agents regarding flexural strength ($P<0.001$) and modulus ($P=0.0084$). The filled bonding agent Nanobond showed higher flexural strength and modulus than the unfilled bonding agent Bond 1.

Table 2. Mean flexural strength and modulus (MPa±standard deviation) of the bonding agents (n=13).

Bonding agent	Flexural strength (MPa)	Flexural modulus (MPa)
Bond 1	89.7±7.6 [A]	1999.9±258.4 [a]
Nano-Bond	131.1±9.5 [B]	2314.5±271.0 [b]

Significant differences between bonding agents are indicated by different letters (no comparison between columns was performed).

Discussion

In vitro and *in vivo* studies have shown the advantages of using filled bonding agents. Some studies demonstrate a decrease in microleakage (10) and an increase in the bond strength to dentin when filled bonding agents are used (11,12). Besides, it has been shown a good clinical performance of such bonding agents (13) and low absorption to fluids from the oral cavity (14). Conversely, other studies did not find clinical advantages when filled bonging agents were used (15) and indicated that the use of filled bonding agents is not a determinant factor to provide high bond strength values on dentin (16).

The filler particles added to NanoBond consist of nano-sized silica particles to allow filler diffusion within the

demineralized dentin. The filler addition in bonding agent composition results in a decrease in shrinkage stress and improved mechanical properties of the hybrid layer once the fillers are able to properly infiltrate the demineralized intertubular dentin along with monomers (5,6), although fillers may agglomerate and impair filler diffusion through the dentin (6). However, the fillers are apparently capable of penetrating into the dentin tubules and participating on the resin tag formation (1,5,17).

The current results demonstrated that the bonding agent having filler particles in its composition showed higher flexural strength (NanoBond: 131.1±9.5 MPa) than the unfilled version (Bond 1: 89.7±7.6 MPa). As the hybrid layer is formed after *in situ* polymerization of resin monomers infiltrated into the demineralized dentin (4,9), it is expected higher bond strength of such layer when filled bonding agent is used. Besides, other factors may be related to the hybrid layer strength, such as filler particle distribution within the hybrid layer and shrinkage stress produced during polymerization (16).

The use of adhesive systems on cavity preparations to create hybrid layer aims to bond the restorative material to tooth and to create an elastic layer capable of relieving all composite shrinkage stress and the chewing forces (18,19). The concept of elastic walls infers that the resilience/elasticity of the resin-dentin interface must compensate for the dimensional changes of the restorative materials without jeopardizing the bonding or tooth structures and therefore assure the durability of the restorative procedure.

Besides the increase in flexural strength, an increase in flexural modulus was observed as a consequence of filler addition in the bonding agent composition. Such finding deserves concern when an elastic layer formation is expected to absorb the stress created by polymerization shrinkage of resin composite during the restorative procedure. In other words, the polymerized filled bonding agent is more rigid than the unfilled bonding agent, so changes in the mechanical properties of the hybrid layer are expected. Therefore, filler addition created a polymer chain with lower flexibility than the polymer chain without fillers, leading to limited chain mobility during polymer deformation (7).

Two bonding agents from the same manufacturer were evaluated in the current study to assure similar compositions between bonding agents besides the presence of filler particles in one product. Therefore, it would be possible to attribute the differences in flexural strength and modulus mainly to the presence of filler particles. Other adhesive systems commercially available present their filled versions having filler particles in their compositions. Among all commercially available adhesive systems, the most known products on the market are Optibond Solo Plus (Kerr, Orange, CA, USA), Prime&Bond NT (Dentsply Caulk, Milford, DE, USA), Single Bond 2 (3M ESPE, St. Paul, MN, USA), and One Step Plus (Bisco Inc., Schuamburg, IL, USA). Some products had their unfilled versions replaced by the filled versions, while others have both versions. Despite the improvements in the mechanical properties of the filled bonding agent observed in the current study, little-to-no information regarding the short- and long-term mechanical properties of such products is available. For this reason, further studies evaluating the effects of the range in filler content on other features, such as degree of conversion, shrinkage and contraction stress are required.

Conclusions

The results of the current study demonstrated that the filled bonding agent NanoBond showed higher flexural strength than the unfilled bonding agent Bond 1. Besides, filler addition increased flexural modulus, so the filled bonding agent presented higher stiffness than the unfilled bonding agent.

Acknowledgements

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