EFFECT OF INCREASED DWELL TIMES FOR SOLVENT EVAPORATION ON THE BOND STRENGTH AND DEGREE OF CONVERSION OF AN ETHANOL-BASED ADHESIVE SYSTEM

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ABSTRACT

This study evaluated the influence of the prolonged setting time of an ethanol-based adhesive system on the dentin bond strength and degree of conversion. Labial and lingual surfaces of fifteen human third molars were flattened until the dentin was exposed and randomly allocated to 3 groups (n=10), according to the dwell time between the application of two consecutive layers of the adhesive system (Adper Single Bond Plus, 3M ESPE) and light activation: G1 – control (no extra dwell time); G2 and G3 – dwell time of 30 seconds and 60 seconds, respectively. After light curing, two cylinders (1.4 x 1 mm) of composite resin (Filtek Flow, 3M ESPE) were bonded to each surface and submitted to micro-shear testing, 24 hours after light curing. A similar adhesive procedure was used for the degree of conversion evaluation using Fourier transform infrared spectroscopy (FTIR). Significant differences between bond strength values (p=0.0003) and degrees of conversion (p=0.0004) were detected. The bond strength of G3 (60-second dwell time) was statistically higher than that of other groups. G1 (control) and G2 (30-second dwell time) presented similar results. Values of degree of conversion indicated that both the 30-second and 60-second dwell times resulted in similar and greater percentages of conversion. The use of a longer dwell time (60 seconds) might provide better solvent volatilization and monomer infiltration; bringing benefits to dentin bonding using simplified etch & rinse adhesive systems.

Key-words: Adhesives, shear strength, composite resins, solvents.

EFEITO DO TEMPO PROLONGADO DE ESPERA PARA A EVAPORAÇÃO DO SOLVENTE NA RESISTÊNCIA DE UNIÃO E NO GRAU DE CONVERSÃO DE SISTEMA ADESIVO A BASE DE ETANOL

RESUMO

Este estudo avaliou a influência do tempo de espera entre a aplicação do adesivo e a polimerização na resistência de união à dentina e no grau de conversão (GC) de um sistema à base de etanol. Superfícies vestibular e lingual de 15 terceiros molares foram desgastadas até a exposição da dentina e aleatoriamente distribuídas em 3 grupos (n=10), segundo a forma de aplicação do sistema adesivo (Single Bond 2, 3M ESPE): G1 (controle) – uso seguindo recomendações do fabricante; G2 e G3 (experimental) – aguardou-se tempo de espera de 30 e 60 segundos, respectivamente, entre a aplicação das camadas do adesivo e a fotoativação. Dois cilindros (1,4 x 1 mm) de resina composta (Filtek Flow, 3M ESPE) foram aderidos à cada superfície; e submetidos ao ensaio de micro-cisalhamento 24h após a polimerização. Semelhante procedimento adesivo foi utilizado para a avaliação do grau de conversão por meio de um espectrômetro de infra-vermelho transformado de Fourrier (FTIR). Diferenças significativas nos valores de resistência de união (p=0,0003) e GC foram observados (p=0,0004). A resistência de união do grupo 3 (espera de 60 segundos) foi estatisticamente maior que a dos demais grupos. Os grupos 1 (protocolo do fabricante) e 2 (espera de 30 segundos) apresentaram resultados estatisticamente semelhantes entre si. O resultado do GC indicou que tanto o tempo de espera de 30 segundos quanto o de 60 segundos apresentam semelhança estatística. De acordo com os achados obtidos, verifica-se que a utilização do tempo de espera prolongado (60 segundos) traz benefícios uma vez que permite melhor infiltração do agente de união e volatilização do solvente.

Palavras-Chave: Adesivos, resistência ao cisalhamento, resina composta, solventes.

INTRODUCTION

One of the main goals of adhesion between restorative materials and dental tissues is to guarantee long-term sealed cavity margins. The morphophysiological variation of dentin is cited as one of the major complicating factors in this process[1-3]. Depending on the region of the tooth, bonding to dentin might be adversely affected by the volume of pulp fluid flow, by variations in the degree of calcification of the substrate, and by the number and arrangement of tubules[4]. Over time, research has investigated the effect of clinical and substrate-related factors, providing major breakthroughs in the use of adhesive systems[2,5-7].

Factors related to the chemical structure of the bonding agent also have significant importance to the success and durability of bonding to dentin. Among them are the chemical composition of the material, the effectiveness of the curing sources, solvent volatilization and the temperature and humidity of the oral environment[8-11]. Current adhesive systems have dissimilar mechanisms of action, chemical composition and application techniques; which all have substantial effects on the stability of the interface between dentin and bonding agent[12].

The presence of solvents in adhesive systems is critical to the effectiveness of the wet-bonding technique[12-14]. Solvents can be organic, such as acetone or ethanol, or inorganic, such as water, compounding the adhesive systems alone or in various combinations[15]. The water and organic solvents contained in adhesives are also known as "water-chasers"[12]. These components are responsible for the displacement of water within the network of collagen fibrils and the infiltration of resin monomers into the interior of the network[11,16]. Effective action of the solvent is directly related to its proper evaporation[17]. When high levels of solvents are trapped into adhesive layers, polymerization can be inhibited, mechanical properties of the adhesive layers can be jeopardized and bond strengths to dentin can be reduced[12,15,17].

The vapor pressure ratio of each type of solvent should determine the technique for applying the adhesive. Therefore, it is important to indicate the particular dwell time required for the solvent to perform its function and then evaporate, thus ensuring that the adhesive layer will have suitable properties[8,16]. Thus, the aim of this *in vitro* study was to evaluate the influence of different times elapsed between the application of an ethanol-based bonding agent and the light-curing, on the dentin bond strength and degree of conversion of the adhesive layer. The experimental hypothesis tested was that longer dwell times would increase the bond strength to dentin and the degree of conversion of the ethanol-based bonding agent.

MATERIALS AND METHODS

Micro-shear testing

In this study, 15 recently extracted human third molars, free of caries, cracks or fractures, were used after the approval from the Committee for Ethics in Research EBMSP (protocol 106/2008). Teeth were cleaned to remove organic debris and were stored in a 0.1% thymol solution at 37ºC until the start of the work.

Teeth were sectioned in the mesio-distal direction and embedded in acrylic resin for rapid polymerization (Jet Acrylic autopolymerized, Articles Dental Classic, Sao Paulo, SP, Brazil), with the buccal/lingual side up. After inclusion, the enamel surface was worn until superficial dentin was exposed, using silicon carbide sandpaper with decreasing abrasiveness (320, 400, and 600-grit) under constant refrigeration (Arotec APL- 4; Arotec S.A., Sao Paulo, SP, Brazil). The dentin surfaces were randomly divided into 3 groups (n=10), according to the technique for applying the bonding agent:

G1 (control)

Surfaces were conditioned with 35% phosphoric acid for 15 seconds (Magic Acid Gel, Vigodent, Rio de Janeiro, RJ, Brazil), washed with water for 15 seconds and dried with paper towels, keeping the surface moist. Immediately after drying, two consecutive and similar drops of the adhesive system (Adper Single Bond Plus, 3M ESPE, St Paul, MN, USA) were actively applied for 15 seconds each. Then a gentle air flow was applied for 10 seconds from a distance of 10 cm. The technique was completed with 10 seconds of light curing (Optlight 600, Medical and Dental Equipment Gnatus, São Paulo, SP, Brazil).

G2 (30-second time lapse)

During bonding procedures, the bonding technique was modified by introducing a 30-second dwell time after the active application and gentle air flow of each layer. The subsequent light activation was done as described above.

G3 (60-second dwell time): The application of the adhesive was carried out as in G2, but the time elapsed after applying each layer was 60 seconds, followed by light curing for 10 seconds.

After the bonding procedures, two cylindrical molds with an internal diameter of 1.4 mm and height of 1 mm (Scalp-23G, EMBRAMED, Sao Paulo, SP, Brazil) were fixed to each dentin surface. The low viscosity composite resin (Filtek Flow, 3M ESPE, St Paul, MN, USA) was inserted into the molds in a single increment and light-cured for 40 seconds. The molds were removed with scalpel blades (15C, LAMEDID Commercial and Services Ltd., Barueri, Sao Paulo, SP, Brazil) and light-cured for other 40 seconds. Thus, two resin cylinders, approximately 1.4 mm in diameter and 1 mm in height, were bonded to the tooth surface. Specimens were stored in distilled water at 37°C for 24 hours.

For the micro-shear testing, specimens were fitted in a universal testing machine (EMIC DL 500, Sao Jose dos Pinhais, Parana, Brazil) and the test was conducted at a constant speed of 0.5 mm/min until fracture. [18] Bond strength in MPa was calculated according to the area of adhesion. The mean bond strength of the two cylinders bonded to each surface was calculated, and considered as the specimens' micro-shear bond strength.

Degree of Conversion

To evaluate the degree of conversion of the adhesive system in the different activation protocols, Fourier transform infrared spectroscopy (FTIR) (Spectrum 100 Optica, PerkinElmer, MA, USA) was used coupled with an attenuated total reflectance element (ATR). In each experimental group, 3 µl of the adhesive system was dispensed on a horizontal crystal of zinc selenide (Pike Technologies, Madison, WI, USA), which acts as a substrate for active infra-red rays. The protocol established for each group was performed (no time elapsed, and 30- or 60-second dwell time) (n = 5).

The degree of conversion was calculated from the ratio between the aliphatic and aromatic carbon double bonds, used as an internal standard, in the uncured and cured states. During the light-activation reaction, the absorbance of aromatic carbon double bonds remains constant, while there is a decrease in the amount of aliphatic double bonds (- c = c). The aliphatic carbon double bond absorbs energy at a wavelength of 1638 cm^{-1}, while the length of the aromatic is 1608 cm^{-1}. For the calculation, the baseline technique [19] was used, traced by the Spectrum software (Spectrum 100 Optica, PerkinElmer, MA, USA). The corrected intensity of the peaks observed at 1638 and 1608 cm^{-1} wavelengths was used in the following formula: R = intensity at 1638cm^{-1}/intensity at 1608cm^{-1} and the degree of conversion (%) as follows:

$$x(\%) = \left(100 - \frac{\dfrac{1638\ \text{cm}}{1608\ \text{cm}^{-1}}\ \text{polymerized}}{\dfrac{1638\ \text{cm}}{1608\ \text{cm}^{-1}}\ \text{not polymerized}} \right) \text{x } 100$$

Statistical Analysis

Initially, an exploratory data analysis was performed to verify the adequacy to the parameters of the analysis of variance (ANOVA). Both inferential statistical analyses from micro-shear bond strength and from the degree of conversion were performed by one-way ANOVA. The post-hoc Tukey test was used for multiple comparisons, when significant differences were observed among micro-shear bond strengths and among degrees of conversion. Both analyses were performed in the statistical package SAS, version 9.1, with a significance level of 5%.

RESULTS

Significant differences were observed between the values of micro-shear bond strength (p=0.0003) and degree of conversion (p=0.0004) (Table 1). The micro-shear strength of G3 (60-second dwell time) was statistically higher than that of other groups. G1 (control) and G2 (30-second dwell time) were statistically similar. The result for degree of conversion showed that both the 30-second and the 60-second dwell times have statistical similarity, both having an average conversion significantly higher than that of G1.

Table 1: Mean (standard deviation) of micro-shear bond strength and degree of conversion in the three experimental groups.

	Bond Strength (MPa)		Degree of Conversion (%)	
Control (G1)	7.3 (9.1)	B	65.2 (9.1)	B
Dwell time of 30 seconds (G2)	9.5 (9.2)	B	69.3 (0.9)	A
Dwell time of 60 seconds (G3)	12.0 (5.6)	A	69.5 (0.5)	A

Different letters represent statistically significant differences (one way ANOVA/Tukey, alfa=5%).

DISCUSSION

This experimental study evaluated the hypothesis that longer dwell times for solvent evaporation increase the bond strength to dentin and the degree of conversion of an ethanol-based bonding agent. The bonding agent used in this study contains the components Bis-GMA, HEMA, polyalkenoid acid copolymer, camphorquinone, water, ethanol and glycerol dimethacrylate, 10% by weight of silica nanoparticles (5 nanometers). Because of the size of its particles, this bonding agent provides adequate diffusion into dentinal tubules, and consequently better infiltration of resin monomers within the interfibrillar spaces formed after the demineralization of dentin, which are approximately 20 nm in size[20].

Microshear testing[18] was selected because it is versatile and very useful for evaluating the bond strength between the mineralized tissues and restorative materials, as it allows better standardization in the production of evidence[21]. The degree of conversion was used to determine whether prolonged waiting times directly influence the conversion of the polymeric bonding agent, since recent studies indicate that the remaining solvent in the monomeric mixture, prior to its polymerization, has been identified as crucial for the formation of areas of incomplete conversion monomer and polymer formation with low mechanical properties[12,15,17].

According to the results of the present investigation, groups that had 30- and 60-second dwell times between the application of layers of adhesive and light curing showed higher values of degree of conversion compared to the adhesive applied without extra dwell time. These results can be related to a greater evaporation of the solvent, contributing to the reduction of distances between monomers, and therefore increasing the degree of conversion of adhesives[4,6,22-24]. Similar results were obtained in a previous study[15], which evaluated the effect of four methods of solvent evaporation on the degree of conversion of seven one-step conventional adhesives systems. The results indicated that the degree of conversion does not depend exclusively on the type of organic solvent (ethanol or acetone) used, but also on the way that each adhesive system is applied. For etch & rinse adhesive systems such as One-Step and Adper Single Bond, the use of an air spray for 10 seconds to accelerate the evaporation of the solvent was important in increasing monomer conversion. In another study [17], the authors concluded that the use of a warm air flow of might be an interesting clinical tool to improve the quality of dentin bonding and to achieve lower nanoleakage, since the number of pores in the adhesive layer can be reduced. Considering the findings of the works mentioned above and the results from the present investigation, it may be noted that adequate solvent evaporation through strategies such as air sprays or prolonged times elapsed appear to result in a greater degree of conversion and consequently provide adhesive layers with improved properties.

Although a high degree of conversion represents improved mechanical performance for adhesive layers, the micromechanical interlocking of the cured adhesive system in the mesh of exposed collagen fibrils is another important parameter for long-term dentin bonding[1,4,11]. Therefore, both the greater monomer conversion and the well-defined and entrapped in dentin polymer matrix correspond to mechanical properties suitable for increasing the longevity of restorations[10]. The findings of the present study have shown that 30- or 60-second dwell times might result in similar degrees of conversion for the adhesive layers; nonetheless, this similarity was not observed in bond strength values. The 30-second dwell time presented bond strength values similar to those obtained in the control groups (no extra time elapsed); and both groups have lower values than the 60-second dwell time. Therefore, it is not possible to conclude that bond strength is directly related to the degree of conversion, although the latter has significant influence on the quality of the polymer[8].

A previous study[16] examined the extent of penetration of organic solvents through five versions of monomers with different degrees of hydrophilicity. The authors concluded that, ideally, all solvents should be completely eliminated from the adhesive prior to light activation. Ideally, the vapor pressure ratio of each type of solvent should determine the technique for applying the adhesive. Thus, the particular dwell time required for the solvent to perform its function and then evaporate, ensuring suitable properties of the adhesive layer, should be known. Nevertheless, in a clinical situation, temperature, humidity and other conditions could interfere in solvent's dwell time. Therefore, in the present investigation, the 60-second period was suggested as a possibility for clinically standardizing the dwell times for ethanol-based simplified adhesive systems. Although the findings of this investigation indicate that the 60-second delay might have provided more time for solvent evaporation, favoring monomer conversion and enabling a better infiltration of monomers through the network of collagen fibrils, further studies are needed to confirm these effects on different classes of adhesive systems, with other mechanism of action and solvents.

According to the limitations of the present *in vitro* investigation, it can be concluded that a prolonged time lapse (60 seconds) enables both better solvent volatilization and infiltration of the monomer, bringing the benefits to dentin bonding with a simplified etch & rinse system, and this should be a common recommended practice.

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