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Microleakage Study of Three Adhesive Systems

Vanessa Gallego ARIAS[1]
Inger Teixeira CAMPOS[2]
Luiz André Freire PIMENTA[1]

[1]*Department of Restorative Dentistry, Dental School of Piracicaba, UNICAMP, Piracicaba, SP, Brazil*
[2]*Department of Restorative Dentistry, Dental School - Estácio de Sá University, Rio de Janeiro, RJ, Brazil*

The purpose of this *in vitro* study was to evaluate the efficacy of three hydrophilic dentin adhesive systems to reduce class II restoration microleakage. A total of 60 human molar teeth were used in which two box cavities were made on the distal and mesial surfaces, with a cervical margin in dentin. These cavities were randomly divided into 3 groups (n = 40 each), according to adhesive system tested: G1: OptiBond SOLO®; G2: Amalgambond Plus®; G3: Etch & Prime 3.0®. The cavities were restored with the composite resin Z-100®. The groups were thermocycled 2000 times (5 ± 1°C and 55 ± 1°C) with a dwell time of 1 min. The teeth were then immersed in 2% methylene blue, pH 7.0, for 4 h, sectioned and observed with a stereomicroscope MEIJI 2000 (35X). The evaluation was made using scores (0-4) and the results were expressed through the sum of the ranks. G1 = 1994.00; G2 = 2294.00; G3 = 2972.00. The three groups were significantly different. The self-etching adhesive system Etch & Prime 3.0 was less effective in preventing microleakage. The OptiBond SOLO adhesive was the most effective in reducing microleakage in dentin margins when compared with Amalgambond Plus and Etch & Prime 3.0.

Key Words: microleakage, adhesive systems, class II restorations.

INTRODUCTION

The efficacy of adhesive systems on the enamel surface has been proven. However, the same is not observed on dentin and cementum surfaces (1). The bonding agents etch the dentin with acids and use hydrophilic primers to promote micromechanical bonding. Currently, adhesive systems etch the dentin with phosphoric acid or similar acids to decalcify the dentin surface. The total-etching techniques, proposed by Fusayama et al. in 1979 (2), remove the smear layer, open the dentinal tubules, and increase dentinal permeability. Decalcification can be acheived by various factors, including pH, concentration, viscosity, and application time of the etchant (2).

Removal of hydroxyapatite crystals leaves a collagen network that can collapse and shrink because of loss of inorganic support and excess dry time. After the etchant is rinsed off, a primer containing one or more hydrophilic resin monomers is applied. The hydrophilic group has affinity for the dentinal surface and the hydrophobic group has affinity for resin, thereby forming the hybrid layer. This hybrid layer formation between dentin and resin was first described by Nakabayashi et al. in 1982 (3), and it is thought to be the primary bonding mechanism of most current adhesive systems.

In an attempt to simplify clinical procedures, 2-step bonding systems were developed consisting of an etchant gel and a combined primer and bonding agent consisting of hydrophobic and hydrophilic monomers in one bottle. These monomers are dissolved in high-vapor pressure organic solvents such as acetone or ethanol and result in high bond strengths especially when dentin is moist or has been rewetted (4).

Self-etching products, consisting of a mixture of acid monomers that etch cut enamel and dentin as well as primers that allow the penetration of resins into the demineralized dentin, also achieve good shear bond strengths (5).

These one-bottle and self-etching systems have been used in clinical practice and it has been demon-

Correspondence: Prof. Dr. Luiz André F. Pimenta, Faculdade de Odontologia de Piracicaba, UNICAMP, Av. Limeira 901, 13414-018 Piracicaba, SP, Brasil. e-mail: lpimenta@fop.unicamp.br

strated that the progress in formulation of dentin adhesives has improved the clinical performance of cervical resin-based composite restorations (6). However, no dentin bonding agent currently available completely eliminates the microleakage at the cementum/dentin interface. The causes of microleakage are usually associated with polymerization, shrinkage, the composite resin used, occlusal load, location of the prepared margins and the technique used.

The clinical symptoms associated with the occurrence of microleakage are breakdown and discoloration of margins, secondary caries, increase in post-operative sensitivity, and pulp pathology (6). Many different techniques have been used to demonstrate microleakage. These techniques include the use of bacteria, compressed air, chemical and radioactive tracers, electrochemical investigations, scanning electron microscopy, and perhaps most common of all, dye penetration (7).

Investigation of leakage has been carried out both *in vivo* and *in vitro*, but the latter is more common. *In vitro* experiments fall broadly into two categories – one that uses a clinical simulation and the other that is purely a test of the behavior of materials (8).

The purpose of this *in vitro* study was to evaluate the effects of three hydrophilic adhesive systems on microleakage on dentin in class II restorations with the total-etching technique.

MATERIAL AND METHODS

Sixty human molars were selected, cleaned, and stored in a 2% formol solution, pH 7.0, for 7 days. In each tooth, two vertical box cavities on the distal and mesial surfaces were made. The dimensions of the cavities were: 1 mm beyond the cementum-enamel junction; 3 mm in width; 1.5 mm in depth.

The cavities were made with #245 carbide burs (JET Brand, Wheeling, IL, USA), at high speed with water cooling, and were replaced after every 10 preparations. The 120 cavity preparations were randomly assigned into 3 groups (n=40), according to the adhesive system used (Table 1).

The bonding agent systems were applied following manufacturer instructions. The cavities were restored with Z100 (3M/ESPE, St. Paul, MN, USA) composite resin in three increments. Each increment of composite resin was light-cured for 40 s. After 24 h, the restorations were finished with Sof-lex disc systems (3M/ESPE) in decreasing granulation. The specimens were thermocycled 2000 times between water baths at 5 ± 1°C and 55 ± 1°C for 1 min dwell time in a MCT2-AMM Instrumental machine (ERIOS, São Paulo, SP, Brazil).

After thermocycling, the root apex was sealed with epoxy resin and the teeth were covered with two coats of nail varnish. All areas of the teeth were covered with the varnish except for the restorations and a 1-mm rim of the tooth structure around each restoration. The teeth were immersed in 2% methylene blue for 4 h, rinsed with distilled water, dried for 10 min, and sectioned with a diamond disc (KG Sorensen, São Paulo, SP, Brazil). The samples were analyzed with a stereomicroscope MEJI 2000 (Beijing, China) (35X).

The following criteria were used to score penetration: 0 = no microleakage, 1 = dye penetration within 1/3 of cavity wall, 2 = dye penetration within 2/3 of cavity wall, 3 = dye penetration within the last 1/3 of

Table 1. Adhesive systems evaluated.

Material	Manufacturer	Characteristics of adhesives	Composition
Amalgambond Plus®	Parkell Farmingdale, NY	Multi-step adhesive system (a,b,c)	10% citric acid, 3% chloride acid, HEMA, META, MMA, TBB
OptiBond SOLO®	Kerr Orange, CA	One-bottle adhesive system (a,d)	37.5% H_3PO_4, Bis-GMA, GPDM, HEMA, PAMM, barium glass, silica, sodium hexafluorosilicate, ethanol
Etch & Prime 3.0®	Degussa Dusseldorf, Germany	Self-etching adhesive system (e)	Pyrophosphate, 2-hydroxy-ethlymethacrylate, ethanol, distilled water, initiators and stabilizers

a: etching; b: priming; c: adhesive application; d: simultaneous primer and adhesive application; e: simultaneous etch, primer and adhesive application.

cavity wall up to the axial wall, 4 = dye penetration spreading along the axial wall.

Statistical analyses were done using the Kruskall-Wallis test and Mann-Whitney non-parametric analysis, and the results were expressed through the sum of the ranks.

RESULTS

Marginal microleakage was observed in varying levels in all groups (Figure 1). The results of this study were expressed through the sum of the ranks and significant statistical differences were observed between all groups: G1 = 1994.00; G2 = 2294.00; G3 = 2972.00 ($p \leq 0.05$). The Kruskal-Wallis and Mann-Whitney tests ($p \leq 0.05$) showed that the Optibond SOLO (OB) adhesive controlled microleakage more efficiently than Amalgambond Plus (AMP). The Etch & Prime 3.0 (EP) adhesive was the least effective system in controlling microleakage.

DISCUSSION

The hybrid layer and resin tags are essential for a strong bond between the composite resin and the dentin surface (3). Complete dissolution of the smear layer, dentin peritubular and intertubular decalcification, resin infiltration in this decalcified dentin and the polymerization of polymer molecules are responsible for creating this bonding mechanism.

Self-etching primers do not completely remove the smear layer from dentin, rather, they impregnate the smear plug, fixing it at the entrance of the tubules. Dentin is demineralized up to 7.5 µm depending on the type of acid, the etching time, and the concentration of the etchant.

The adhesive systems of this study present different etchants. OptiBond SOLO uses a separate phosphoric acid-based conditioner in a concentration of 37.5% to etch enamel and dentin simultaneously (total-etch). Amalgambond Plus uses a solution of 10% citric acid and 3% ferric chloride to remove the smear layer and demineralize the dentin. The self-etching Etch & Prime 3.0 simultaneously conditions and primes the enamel and dentin substrate without rinsing the self-etching primer. This adhesive contains a weaker acid, pyrophosphate acid, in the primer composition. The self-etching primers produced less etching because of their relatively high pH (1.5-3.0), when compared with the pH of 32-37% phosphoric acid (-0.43 to 0.02) (9). Ogata et al. (10) reported that bond strengths of self-etching primer bonding systems to dentin could be affected by differences in the quantity of residual smear layer left on the surface due to the weak acidity of self-etching primers (10).

The results of the present study demonstrated that this bonding agent is less effective in controlling microleakage when compared with Amalgambond Plus and OptiBond SOLO. It could be that the mixing of the pyrophosphate acid with water did not result in an adequate quantity of phosphoric acid to decalcify the dentin to a sufficient depth which would cause a regular and efficient hybrid layer to control microleakage. The 35-40% phosphoric acid concentration was more effective than other dentin conditioners, however, Chan and Swift reported that 10% maleic acid, 10% phosphoric acid and 1.6% oxalic acid reduced microleakage at restoration margins in dentin and enamel (11).

When dentin is etched, the smear layer and the mineral phase of dentin are dissolved and the collagen is exposed. The desiccation of the dentin can cause a collapse of the unsupported collagen network, inhibiting adequate wetting and the penetration of the resin to create the hybrid layer. The clinician must be aware that pooled moisture should not be allowed to remain on the tooth because excess water can dilute the primer and reduce its effectiveness. This phenomenon is known as overwet. Some hypotheses have been made concerning the Etch & Prime 3.0 adhesive. This system presents a 55% water volume (manufacturer's information). It has been suggested that the presence of water might influ-

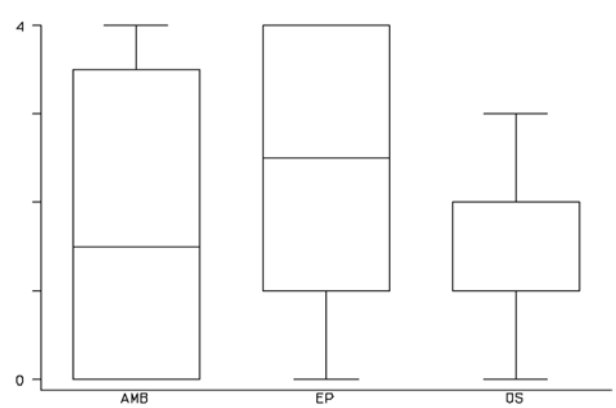

Figure 1. Box plot illustrating the distribution of scores.

ence the polymerization (12).

The results of this study are in agreement with those of Cardoso et al. (13) and Bedran de Castro et al (14), which demonstrated that the use of Etch & Prime 3.0 resulted in high leakage values.

Primer has been used to improve the bonding between the composite resin and the cavity walls. Current adhesive systems contain hydrophilic primers that utilize acetone, alcohol and/or water as solvent. These solvents carry the resin primers into the demineralized dentin by displacing water from the collagen network. Resin penetration into the collagen network and its occupation of the demineralized dentin is responsible for forming the interdiffusion zone or hybrid layer. HEMA is a hydrophilic monomer that penetrates into the collagen network. HEMA molecules are usually dissolved in different solutions with acetone, alcohol and/or water which work as chasers. These chasers compete with water present at the dentin surface by promoting a union of the water molecules and displacing water when compressed air is applied, permitting the penetration by the monomer (12). The removal of water from the collagen fibrils may stabilize the structure by increasing the amount of interaction of weak forces between adjacent collagen molecules. Water removal may also permit additional hydrogen bonds to form between collagen molecules that were previously bonded to water molecules (15).

The adhesive system Amalgambond Plus uses acetone as the priming solvent. The adhesive systems with acetone-based primers required more diligence to achieve a primed dentin surface because of their material sensitivity. The Etch & Prime 3.0 and OptiBond SOLO systems are ethanol-based primers. Jacobsen et al. (16) showed that adhesive systems with alcohol are less sensitive to the technique utilized. Requirements for an effective dentin adhesive system include the ability of the system to thoroughly infiltrate the collagen network and partially demineralized zone, to comingle and encapsulate the collagen and hydroxyapatite crystallites at the surface of the demineralized dentin, and to produce a well-polymerized durable hybrid layer. It was been suggested that poor infiltration of adhesive resin into the rich collagen area of the demineralized dentin leaves gaps in the hybrid layer where water and microleakage can infiltrate, producing hydrolysis of the exposed collagen peptides not protected by hydroxyapatite or resin (17).

Marginal leakage of the composite resin restorations might be influenced by external stress produced during thermocycling, which causes thermal variation that permits the formation of gaps, and internal stress produced by polymerization shrinkage and differences in the thermal expansion characteristics of the materials and the teeth. These stresses may hinder the properties of the materials by creating openings and deforming the tooth substrate.

However, the internal reorganization of the polymer molecules formed during polymerization contributes to a significant reduction in the generated stress that is dependent upon the configuration factor as well as the properties of the material. It has been suggested that the stress reduction produced by a resin-dentin bonding agent is attributable to the low elastic modulus of both the adhesive resin layer itself and the resin-dentin interdiffusion zone (18). The adhesive system should present median flexural strength between the dentin and the composite resin to increase the resistance of the adhesive thereby reducing the effects of polymerization shrinkage.

Another factor to be considered is the variation of layer thickness. The differences of thickness could be due to a number of factors, such as differences in viscosity of the adhesive systems, filled vs unfilled primer/adhesives, and variations in the number of coats and application techniques as indicated by the manufacturers (19). Vargas et al. (19) observed that OptiBond SOLO, a filled adhesive system, had a significantly thicker primer/adhesive layer with a thickness of up to 50 μm.

The gradient of elastic modulus is more pronounced in systems that form a thicker layer or include a filled low-viscosity resin. Thus, dentin adhesives that contain a filled resin may have sufficient elasticity to relieve the stress that develops from the contraction of the composite resin used as the final restorative material (15).

Filled adhesives are designed to provide stress relief between the tooth and restorative materials. OptiBond SOLO is filled with nanoparticles. The filler is a nanoscale silica which functions by a special silinization process. This process makes the nanofiller more compatible with the resin matrix and allows it to serve as a cross-linker. Choi et al. (20) demonstrated that the contraction stress generated during the placement of composite resin restorations was significantly relieved by an application of an increasing thickness of low-stiffness adhesive. Increasing the adhesive thickness can lead to improvement in marginal integrity and, as a consequence, may prolong the life of a restoration (20).

The present study showed that the one-bottle adhesive system Optibond SOLO was more effective in reducing microleakage in dentin margins when compared with the self-etching primer Etch & Prime 3.0 and the multi-step adhesive system Amalgambond Plus. However, although there has been progress in the development of adhesive systems, current bonding agents have not entirely eliminated microleakage even when the direct polymerization technique is used.

RESUMO

O objetivo deste estudo foi avaliar a eficácia de 3 sistemas adesivos hidrófilos na redução da microinfiltração de restaurações classe II. Foram selecionados 60 dentes molares humanos. Foi realizado em cada dente dois preparos classe II do tipo "slot vertical" nas superfícies mesial e distal, com margem cervical em dentina. As 120 cavidades foram sorteadas aleatoriamente em 3 grupos (n = 40), de acordo com o sistema adesivo testado G1: OptiBondo SOLO® (Kerr); G2: Amalgambond Plus® (Parkell); G3: Etch & Prime 3.0® (Degussa). As cavidades foram restauradas com resina composta Z 100 (3M/ESPE). As amostras foram termocicladas 2000 vezes (5 ± 1ºC e 55 ± 1ºC), durante 1 min em cada banho. Após a termociclagem, os dentes foram imersos em uma solução de azul de metileno a 2%, pH 7,0, durante 4 h, seccionados e observados em microscópio ótico MEIJI 2000 (35X). A avaliação foi realizada utilizando-se escores (0-4) e os resultados expressos através da soma das ordens. G1: 1994.00; G2: 2294.00; G3: 2972.00. Os 3 grupos foram significativamente diferentes. Os resultados indicaram que o sistema adesivo autocondiconante Etch & Prime 3.0 foi menos efetivo no controle da microinfiltração marginal. Já o sistema adesivo OptiBond SOLO foi o mais efetivo no controle da microinfiltração quando comparado com o Amalgambond Plus e o Etch & Prime 3.0.

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