

DOES BONDING TO DENTIN REDUCE MICROLEAKAGE OF COMPOSITE RESTORATIONS?

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ABSTRACT

This study evaluated the effect of adhesive application only to enamel on the marginal microleakage of composite resin restorations performed with different adhesive systems. Standardized cylinder-shaped cavities were prepared on the buccal surface of eighty bovine incisors. Two etch-and-rinse (Adper Scotchbond Multi-purpose [3M ESPE, St. Paul, MN, USA] and Adper Single Bond 2 [3M ESPE]) and two self-etching (Clearfil SE Bond [Kuraray, Osaka, Japan] and Adper Prompt [3M ESPE]) adhesive systems were evaluated. The adhesives were applied only to enamel or to both dentin and enamel. After adhesive light-activation, the cavities were restored with composite resin. The samples were coated with two layers of nail polish, except an area of 1-mm wide around of the restoration, and immersed in a methylene blue solution. Afterwards, the speci-

mens were ground in order to obtain powder, which was immersed in absolute alcohol. The solutions were centrifuged and the supernatant was analyzed using an absorbance spectrophotometer. Linear regression was used to estimate the dye concentration. Data were analyzed using ANOVA and Tukey's tests ($\alpha=0.05$). The etch-and-rinse adhesives showed lower microleakage means compared to those of the self-etching adhesives. Adper Prompt presented higher microleakage means. There was no difference between the modes of application of the adhesive on the cavity for all adhesive systems, except for Clearfil SE Bond. This showed lower microleakage when applied to the whole cavity. Bonding to dentin may not reduce microleakage of composite restorations.

Key-words: adhesives, dental bonding, dental leakage.

A UNIÃO À DENTINA REDUZ A MICROINFILTRAÇÃO DE RESTAURAÇÕES DE RESINA COMPOSTA?

RESUMO

Este estudo avaliou o efeito da aplicação do adesivo apenas ao esmalte na microinfiltração marginal de restaurações de resina composta realizadas com diferentes sistemas adesivos. Cavidades cilíndricas padronizadas foram preparadas na superfície vestibular de oitenta incisivos bovinos. Dois sistemas adesivos convencionais (Adper Scotchbond Multi-purpose [3M ESPE, St. Paul, MN, USA] e Adper Single Bond 2 [3M ESPE]) e dois auto-condicionantes (Clearfil SE Bond [Kuraray, Osaka, Japan] e Adper Prompt [3M ESPE]) foram avaliados. Os adesivos foram aplicados apenas no esmalte ou tanto na dentina quanto esmalte. Após a fotoativação do adesivo, as cavidades foram restauradas com resina composta. As amostras foram cobertas com duas camadas de verniz, exceto um área de 1 mm de largura da restauração, e imersas em solução de

azul de metileno. As soluções foram centrifugadas e o sobrenadante foi analisado usando um espectrômetro de absorvância. Uma regressão linear foi usada para estimar a concentração de corante. Os dados foram analisados usando ANOVA e teste de Tukey ($\alpha=0,05$). Os adesivos convencionais mostraram menores médias de microinfiltração comparadas às dos adesivos auto-condicionantes. Adper Prompt apresentou a maior média de microinfiltração. Não houve diferença entre os modos de aplicação do adesivo na cavidade para todos os adesivos, exceto para o Clearfil SE Bond. Este mostrou menor microinfiltração quando foi aplicado em toda a cavidade. A união à dentina pode não reduzir a microinfiltração de restaurações de resina composta.

Palavras-chave: Adesivos, Adesão dental, Infiltração dental.

INTRODUCTION

Despite the improvements in restorative materials in recent decades, the marginal integrity of restoration remains a challenge for dentistry. Poor margin-

al adaptation may produce marginal discoloration, postoperative sensitivity and secondary caries.¹ These are the most frequent reasons for replacing or repairing an adhesive restoration^{2,3}. The margin-

al failure of composite resin restorations is related mainly to the polymerization shrinkage of composites⁴. However, it is also affected by other factors such as the restorative technique and adhesive system used⁵.

The fundamental principle of bonding to the tooth substrate is based upon micromechanical interlocking by which the inorganic phase of dentine or enamel is exchanged for adhesive resin⁶. Enamel etching removes calcium phosphate and creates porosities for the infiltration and subsequent *in situ* polymerization of resin. On dentin, the resin adhesive diffuses through the collagen fibrils exposed by etching and forms the hybrid layer with them. The etching procedure can follow two different approaches. With etch-and-rinse adhesive systems, the tooth substrate is etched by an acidic solution (usually with 30-40% phosphoric acid gel) followed by rinsing with water. Simpler adhesives were introduced with the development of self-etching primers/adhesives, eliminating the previous conditioning, rinsing, and drying steps that were critical for the adhesion protocol. However, it has been demonstrated that this simplification did not improve bonding performance^{7,8}.

Most studies of adhesive systems have demonstrated that the bonding procedure of adhesive to enamel is predictable^{6,7,9,10}. However, the bonding procedure to dentin is more complex, resulting in more failures.⁸ Clinically, keeping the margins sealed is the main factor in the clinical success of adhesive restorations¹¹⁻¹⁴. Considering that most restorations have all margins at the enamel, bonding the composite only to enamel may, theoretically, be sufficient to maintain the proper marginal seal. Thus, the

aim of this study was to evaluate the effect of adhesive application only to enamel on microleakage in cavities of class V with a margin at the enamel. The null hypothesis was that bonding to dentin does not reduce microleakage of composite restorations presenting all margins at enamel.

MATERIAL AND METHODS

One week after extraction, sound bovine incisors were cleaned, polished, and examined under a light microscope (Eclipse E 600; Nikon, Shinagawa-ku, Tokyo, Japan) in order to exclude any with cracks. Eighty teeth were selected and stored in distilled water at 5°C for less than one month before the restorative procedure. Cubic 5.0mm blocks were obtained from the buccal surfaces using a diamond disc. The surface was slightly wet-ground with 1200-grit SiC abrasive paper to obtain a flat area of enamel. Then a circular-shaped class V cavity (2.0 ± 0.05 mm diameter by 2.0 mm depth) was prepared on the central part of the block using a #4054 diamond bur (KG Sorensen Ind. Com. Ltda. – Barueri, SP, Brazil). The cavities were made 4 mm from the cemento-enamel junction using a water-cooled high-speed turbine attached to a standard cavity preparation device. A new bur was used for each of the five preparations.

Cavities were randomly assigned to eight groups according to a combination of the adhesive system and application mode. The adhesive systems used in this study and respective application descriptions are summarized in Table 1. The application was performed only to enamel or to all cavity walls (both enamel and dentin). When the adhesive was applied only to enam-

Table 1: Classification and adhesive procedure of adhesive systems used in this study.

Adhesive systems	Category	Adhesive procedure*
Adper Scotchbond Multi-purpose (3M ESPE)	3-steps etch-and-rinse	1. Acid etching (15s), rinsing (15s) and air-drying (10s) leaving dentin moist. 2. Primer and air-stream (10s). 3. Adhesive and light-activation (10s).
Adper Single Bond 2 (3M ESPE)	2-steps etch-and-rinse	1. Acid etching (15s), rinsing (15s) and air-drying (10s) leaving dentin moist. 2. Two consecutive coats of adhesive, air-stream (10s) and light-activation (10s).
Clearfil SE Bond (Kuraray)	2-steps self-etching	1. Primer with slight agitation (20s) and air-stream (10s). 2. Adhesive and light-activation (10s).
Adper Prompt (3M ESPE)	Single-step self-etching	1. Liquids A and B were mixed. 2. Mixed adhesive with slight agitation (15s), air-stream (10s) and light-activation (10s).

* Manufacturers' instructions.

el, all adhesive procedures were performed under an optical microscope (30x, EMZ-TR, Meiji Techno Co., Saitama, Japan) and using an extra-fine microbrush (Cavibrush, FGM, Joinville, SC, Brazil).

The cavities were restored with a microhybrid resin composite (Filtek Z-250, 3M ESPE, St. Paul, MN, USA), filled in one 2mm (bulk) increment and light-activated for 20 seconds. An Optilux 501 light-curing unit (Demetron Kerr, Danbury, USA) with an output intensity of 650 mW/cm² was used in this study. The output of the light-curing unit was periodically checked using a handheld radiometer (Model 100, Demetron Kerr). After restoration, all specimens were stored in distilled water at 37°C for 24h and polished with flexible aluminum oxide disks (Sof-Lex Pop-on®, 3M ESPE, St. Paul, MN, USA) under a water spray. All specimens were kept in water at 37°C for 24h.

The blocks were then coated with two layers of nail polish, except for an area 1mm wide around of the restoration, and immersed in a 2% methylene blue solution for 12 hours at 37°C. After this time, the specimens were rinsed in tap water and dried. The surface layer of the composite restorations was abraded with Sof-Lex to remove possible superficial dye penetration in the restorative material. Each dental block was weighed and ground into powder in a mill for hard tissues (Marconi Equip. Ltda., Piracicaba, SP, Brazil). Each powdered specimen was weighed again and the samples in which initial and final weights differed by more than 10% were discarded. Each powdered sample was individually immersed in a glass tube containing 4 ml of absolute alcohol (Merck, Darmstadt, Germany) for 24 hours in order to dilute the methylene blue. Then the solutions were centrifuged (Tomy, IC 15NA, Tomy Ind., Tokyo, Japan) at 3,000 rpm for 3 minutes. The supernatant was analyzed using an absorbance spectrophotometer (Beckman DU 65 – Instruments, Inc., Fullerton, CA, USA) adjusted to a wavelength of 668 nm.

In order to determine the absorbance, the spectrophotometer was adjusted to an appropriate wavelength for the methylene blue, corresponding to the maximum absorbency for the dye. To calibrate the spectrophotometer, the absorbance of standard solutions (0.1; 0.2; 0.3; 0.5; 1; 2; 4; 6 mg/ml) was determined at wavelengths ranging from 400 to 700 nm, and the maximum value was obtained at 668 nm. To estimate the dye concentration in the experimental samples, a linear regression was obtained. The regression equation is expressed as: $y = 0.2716 x - 0.0075$, where y is the absorbance and x the dye concentration. The microleakage of each specimen was expressed as μg of dye/ml. Two-way ANOVA and Tukey's tests were performed on the data at the 0.05 confidence level. The factors evaluated were "adhesive system" and "substrate where the adhesives were applied."

RESULTS

ANOVA showed that there were statistically significant differences for the factors "adhesive system" ($p < .001$), "substrate where the adhesives were applied" ($p < .001$) and for interaction between factors ($p < .001$). The comparisons according to Tukey's test are shown in Table 2. No statistical difference was found between the etch-and-rinse adhesive systems, independently of the application mode. These adhesive systems presented the lowest microleakage values. The samples bonded with Adper Prompt showed the highest microleakage means, with no differences between the adhesive application modes. In contrast, using Clearfil SE Bond only on enamel produced higher microleakage than its application to both dentin and enamel.

DISCUSSION

Microleakage tests are usually used in dentistry as an *in vitro* evaluation of the quality of restoration margins. The purpose is to predict the clinical performance with regard to the occurrence of postoperative sensitivity and/or secondary caries. This method involves the immersion of a restored tooth in a dye solution. Traditionally, the specimens are washed and cut into two or more slices after their removal from the solution, and the extension of microleakage is determined visually¹⁵. The

Table 2: Mean values (standard deviation) for microleakage in μg of dye/ml.

Adhesive system	Bonded substrates	
	Only enamel	Dentin and enamel
Adper Scotchbond Multi-purpose	0.034 (0.009) Ac	0.025 (0.007) Ac
Adper Single Bond 2	0.079 (0.01) Ac	0.067 (0.009) Ac
Clearfil SE Bond	0.533 (0.074) Ab	0.229 (0.047) Bb
Adper Prompt	0.840 (0.074) Aa	0.799 (0.094) Aa

Means followed by different letters (upper case – row, lower case – column) differ by Tukey test ($\alpha = .05$).

main problem of this method is the fact that it involves a qualitative evaluation. Generally, the results obtained in each study group differ only slightly, making the interpretation of results difficult and reducing the sensitivity of the test. The quantitative microleakage evaluation method was developed by Douglas and Zakariasen.¹⁶ This methodology eliminates the subjective operator evaluation that is used in qualitative evaluations and measures all of the infiltrated dye¹⁷.

In composite restorations, microleakage is often related to polymerization shrinkage that causes tensile stress between the cavity wall and the restoration⁴. This stress can disrupt the bond and lead to the formation of gaps. Thus, proper bonding of an adhesive to dental tissue contributes to avoiding marginal microleakage^{18,19}. The current study used bovine teeth as the bonding substrate to evaluate the microleakage of adhesive restorations. Reis et al.²⁰ analysed bond strength and enamel and dentine morphology as possible substitutes for human teeth in bonding tests. The values of bond strengths obtained with bovine and human teeth are similar for either enamel or dentine. In addition, the morphology of these two substrates was also similar. Thus, it is expected that the performance of adhesives would not be compromised by the use of bovine teeth and that the outcomes would be similar for human teeth.

In the current study, the etch-and-rinse adhesives presented the lowest microleakage values, while the single-step adhesive produced the highest microleakage. Based on the outcomes, there is a tendency to correlate the values of microleakage with the bond strength of the adhesive to enamel. A positive correlation could be observed when the adhesives presenting high bond strength to enamel²¹ produced low dye microleakage. However, most studies have not found a correlation between marginal microleakage and bond strength³. In contrast, a positive correlation has been demonstrated for the formation of gap and bond strength⁹. This demonstrated that gaps are not the only pathway for microleakage. As the dye molecules used in this study are so small, they can penetrate through other smaller and invisible paths through the dental tissue/restorative material interface²².

The application of adhesive only to enamel produced similar microleakage values as its application to the whole cavity, except for Clearfil SE Bond. Thus, the null hypothesis of the study was

partially rejected. The adhesive application only to enamel reduces the bonding area of the restoration, resulting in a lower C-factor. Thus, lower polymerization stress can be expected in this situation, favoring the maintenance of marginal sealing. However, Braga et al.²³ showed that microleakage is mainly related to the volume of the restoration, but not to its C-factor. When the volume of restoration was the same for all samples, microleakage was expected to be similar to both modes of adhesive application. Thus, the difference found for Clearfil SE Bond is probably explained by its ability to bond to enamel and dentin.

Clearfil SE Bond contains the acidic monomer 10-MDP, which includes two hydroxyl groups in its chemical structure. This monomer is able to chemically bond to dental tissue by chelation with calcium²⁴. This approach helps to reduce the permeability of the adhesive. Thus, the higher microleakage obtained with Clearfil SE Bond compared to that of etch-and-rinse adhesive is probably related to the gap formed by de-bonding under shrinkage stress. The weak bond of Clearfil SE Bond to the enamel along with a proper bond to the dentin may explain these results²¹. Clearfil SE Bond has a pH of around 2 and is classified as a mild self-etching system. Self-etching adhesives with relatively high pH are unable to produce an acidic environment that will efficiently etch the enamel. On the other hand, mild self-etching adhesives bond properly to dentin. Thus, de-bonding can occur only on enamel, with the adhesive bonded to dentin remaining. The possible gap formed at the margin by the de-bonding of the adhesive permitted the dye penetration. Probably, after penetrating through the enamel margins, the dye could not pass through the sealed interface between the adhesive and the dentine. This could explain the reduction in microleakage when Clearfil SE Bond was applied to the whole cavity.

Independently of the application mode, the single-step adhesive presented the highest microleakage values. Adper Prompt contains methacrylated phosphoric acid-HEMA esters, which are hydrolytically instable²⁵. In aqueous solutions, they will dissociate into HEMA and the strongly acidic phosphoric acid. Despite the proper etching of enamel and dentin, the high acidity of Adper Prompt may generate some disadvantages. First, the incomplete polymerization of the adhesive causes blisters throughout the adhesive layer²⁶. These blisters permit the for-

mation of water trees, weakening the bond strength and reducing the stability of the bonding.²⁷ Furthermore, the presence of water causes hydrolysis of the adhesive after curing and releases phosphoric acid. Thus, continuous dental demineralization is expected²⁸. These factors may contribute to increased microleakage^{28,29}.

The main aim of a dental restoration is to create an adequate seal, preventing the microleakage of contaminants contained in the oral environment. The outcomes of this study show that proper

bonding to enamel seems to be sufficient to obtain marginal integrity and to reduce microleakage. Based on this, etch-and-rinse adhesive systems had the best performance. The utilization of these adhesives on dentin did not alter the microleakage values. However, it is important to emphasize that the restorations used in this study were not subjected to artificial aging. Thus, further studies are needed before definite conclusions can be drawn regarding whether similar findings will be found elsewhere.

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