



In vitro microleakage of three adhesive systems with three different solvents

Microfiltración in vitro de tres sistemas adhesivos con diferentes solventes

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ABSTRACT

Micro-leakage can be described as the movement of bacteria, fluids, molecules, ions or air between the wall of the tooth cavity and the restorative material. This elicits clinical consequences such as sensitivity, discoloration in the internal phase between tooth and restorative material, secondary caries and pulp disease. The aim of this study was to quantify micro-leakage in dentin and enamel margins in class V cavities of extracted teeth restored with composite resin, using a bonding system provided by different solvents. An experimental study was conducted in 30 molars, extracted no longer than 3 months before. These molars were free of caries and with intact crown surface. Standard class V cavities were shaped, on lingual and vestibular surfaces, with margins in the enamel. Specimens were randomly assigned to three groups, according to their corresponding bonding system. The following bonding systems were applied: Prime & Bond NT, single Bond, and classic Syntec. Manufacturers instructions were closely adhered to. Cavities were restored with ceromer. After being finished and polished, specimens were subjected to thermo-cycling process, and later nail polish and wax were applied. After this procedure, samples were immersed in methylene blue for 24 hours. Using a diamond disk, specimens were then longitudinally sectioned. Samples were evaluated with a microscope, using a 0-4 scale. Data were analyzed using a relative frequency distribution test. The study reached the conclusion that none of the used systems was able to prevent micro-leakage.

Key words: Micro-leakage, adhesive systems, marginal seal.

Palabras clave: Microfiltración, sistemas adhesivos, sellado marginal.

RESUMEN

La microfiltración es el movimiento de bacterias, fluidos, moléculas, iones o aire entre la pared de la cavidad del diente y el material restaurativo, que trae consecuencias clínicas como sensibilidad, cambio de color en la interfase diente-material restaurador, caries secundaria y patología pulpar. El objetivo de este trabajo fue cuantificar la microfiltración en los márgenes de esmalte y dentina en cavidades clase V, en dientes extraídos, restaurados con resina compuesta usando sistemas de adhesión con diferentes solventes. Se realizó un estudio experimental en 30 molares extraídos de tiempo no mayor a 3 meses, no cariados, con superficie coronaria intacta. Se realizaron cavidades estandarizadas clase V en la superficie lingual y vestibular, con márgenes en el esmalte. Se asignaron aleatoriamente en 3 grupos, de acuerdo al sistema de adhesión. Se aplicaron los sistemas de adhesión Prime & Bond NT, Single Bond y Syntac clásico, siguiendo estrictamente las instrucciones del fabricante. Las cavidades fueron restauradas con cerómero. Después del terminado y pulido fueron sometidas a termociclado y las muestras fueron cubiertas con esmalte de uñas y cera. Posteriormente fueron sumergidas en azul de metileno durante 24 horas, después seccionadas longitudinalmente a través de la preparación con un disco de diamante. Las muestras fueron evaluadas con un microscopio usando una escala de 0-4. El análisis de datos fue realizado con un diagrama de frecuencia acumulada. Se concluye que ninguno de los sistemas usados evitó la microfiltración.

INTRODUCTION

In dentistry, along the years, bonding of different restoration materials and remaining dental structure has always been attempted. This was first tried through the establishment of anchoring elements, and a later stage, bonding restorations to the tooth with the help of micro-mechanical retention. First, bonding was only achieved to the enamel, with the help of selective enamel etching with ortho-phosphoric acid, protecting dentin with bases. After this, bonding to enamel and dentin was attempted. Adhesion must be pursued ac-

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ording to the sub-stratus over which the bonding is to be achieved. Enamel is mainly inorganic, and has a content of 92% hydroxyapatite and small amounts of water. When compared to enamel, dentin is composed of 45% hydroxyapatite crystals, about 30% organic matrix, and 25% water. Dentin presents as well a very complex physical structure, which varies according to the depth where it is found. Any material or substance can be called adhesive, when, between two surfaces, has the ability of keeping them joined through a mechanical locking interaction between them, through chemical bonds with them, or through the interaction of both.¹ In the course of the last decade, dentin adhesives materials have experimented changes pertaining to composition and clinical handling. They presently try to adapt to the ever-increasing knowledge on dentin and dentinal fluid behavior.² Fusayama³ in 1980, proposed to etch dentin with the aim of creating micro-retentions, as is the case in enamel. He did not bear in mind the fact that dentin is a basically organic substrate and inside the tubules there is fluid pressure which hinders to the extreme the penetration of hydrophobic substances. Such was the case of resin materials used in that period of time. At a later stage, bi-functional molecules were designed. They possessed the ability to chemically react with organic and inorganic components found in dentin, and simultaneously, copolymerize with restorative materials,^{4,5} although the presence of a layer of dentinal debris during the preparation of the cavity would preclude the intimate contact between resin and dentin which is essential for chemical adhesion.⁵ Due to all the aforementioned reasons, a dentin conditioner has been used in treatment. In our days, it is accepted that bonding to dentin has a micro-mechanical component, through the formation of resin extensions within the dentinal tubules. This bonding would improve with the formation of a dentin-resin inter-diffusion area, which Nakabayashi calls hybrid layer.⁶ It was commonly thought that humidity in dentin reduced adhesion success. This was sustained with research conducted by Glasspoole et al. in 1991, Mitchem et al. in 1998 and Terika et al. in 1987.⁷⁻⁹ In later research, it was pointed out that strong bonding to dentin can be elicited in the presence of humidity, and that this bond can be stronger than when dentin is dry. As shown in research conducted by Kanca in 1992 and Gwinnett in 1992.^{10,11} Kanka¹² has attributed this adhesion ability to bond with humid dentin to the use of a first hydrophilic agent which contains acetone. When acetone combines with water, the water vapor pressure would increase and enhance its partial volatilization. Acetone addition decreases water superficial tension, therefore, when mixing *primer* with acetone,

the water is «collected» until reaching a state of balance. It is supposed that this affects the light within the tubule as well as dentin surface, and improves adaptation of resin tubular walls thus enhancing adhesion.¹¹

In present days there is talk about adhesive dentistry, and, within it, usage and knowledge of dentin adhesive materials is paramount.

The aim of the present paper is to quantify micro-leakage in enamel and dentin margins in class V cavities, using bonding systems with different solvents.

METHODS

Thirty human impacted third molars extracted from male and female patients of different ages, caries free and intact crown surface were used. These teeth were thoroughly cleansed with cures and pumice powder, using a rubber cup and low speed hand-piece. After this procedure, for a period no longer than three months, teeth were immersed in distilled water at room temperature to avoid dehydration. Class V cavities were prepared on buccal and lingual surface. Cavities were placed over the cement-enamel juncture. Cavity measures were as follows: 4 mm width, 2 mm depth and 3 mm occluso-cervical length. Cavities were built with a pear-shaped diamond burr (SS White). All burrs were replaced after five preparations, with refrigerated Synea TA-98 (W&H) high speed hand-piece at 370,000 rpm speed. Enamel margins were beveled at 0.5 mm with a fine grain, flame-shaped burr (Brasseler, USA, Dental Rotary Instruments).

Cavity dimensions were ascertained with a digital Vernier (Max-Cal, USA) device. Once the cavities were performed, samples were randomly assigned to three groups. Before building restorations, cavity margins were cleansed with pumice powder and rubber cup.

Adhesive systems were applied according to manufacturer's instructions. Prime & Bond NT (Dentsply Caulk, Milford DE, USA) was used for the first group. This system provides an acetone-based solvent. Dentin and enamel surfaces were etched during 15 seconds with phosphoric acid at 37% solution Total Etch gel (Ivoclar Vivadent AG Liechtenstein), after this, the area was thoroughly cleansed with water produced by the triple syringe and later dried with filtered air from the compressor. A layer of NT adhesive was placed and distributed for 20 seconds with an applicator. After this, the solvent was eliminated with air. The sample was later photo-polymerized during 10 seconds with an Astralis 5 lamp (Ivoclar, Vivadent AG Liechtenstein) (A5). Intensity of light was measured with a radiometer (Demetron Research Corp.) model 100, at a range 400-500 mW/cm². The amount of generated heat was

measured with a radiometer (Demetron Research Corp), model 200, at a range 5-10mW/cm². Polymerization was conducted at a 2 mm distance.

Single Bond (3M Dental Products ST Paulo, MN, USA) (SB) with alcohol solvent was used for the second group. Enamel and dentin surfaces were etched with TE for 15 seconds. Samples were thoroughly cleansed and dried with compressor filtered air, leaving surfaces visibly damp. Two SB layers were consecutively applied and gently distributed with air during 5 seconds. After this, they were photo-polymerized with A5 during 20 seconds.

Syntac (Ivoclar, Vivadent, AG, Liechtenstein) (S) was used for the third group. This system provided as solvent acetone in aqueous solution. With TE, enamel surfaces were etched for 30 seconds and dentin was etched for 15 seconds. Samples were thoroughly washed and dried with compressor air. A layer of S Primer was applied to enamel and dentin. After 15 seconds, the sample was air dried. After this, a layer of S adhesive was applied to enamel and dentin. 10 seconds were allowed to elapse to then proceed to air-drying the samples. After this, Heliobond was applied to enamel and dentin. It was then spread with air and light-cured for 10 seconds with A5.

Each cavity was restored with Tetric Ceram direct ceromer (Ivoclar, Vivadent AG Liechtenstein) color B2, in two diagonal increments, following Lutz's¹³ technique. Each increment was photo-polymerized for 40 minutes with A5.

Restorations were later polished using the Polishing Esthetic EP discs complete system (Brasseler USA Dental Rotary Instruments). Discs were of thick, medium, fine and extra-fine grit. Every 3 restorations, discs were replaced. All samples were kept in bi-distilled water, at 37 °C for 24 hours. Samples were thermo-cycled for 300 cycles, between 5 and 55 °C for 60 seconds. This procedure was performed in the device developed at the Dental Materials Laboratory of the Research and Graduate School, National School of Dentistry, National University of Mexico. At a later stage, apexes were sealed with all purpose pink wax, and teeth were covered with three layers of transparent nail polish (Renova), exception made of restorations and the 1 mm area surrounding these restorations. Samples were immersed in 2% methylene blue solution for 24 hours as recommended by Philip et al.¹⁵ Samples were thoroughly cleansed with tap water and brushed for three minutes with an Oral B number 60 brush. After this they were vertically fixated with acrylic resin in acrylic rulers measuring 20 cm in length and 4 cm width. Samples were then placed in a cutting device (Guillins Hamco machine Inc, Rochester N.Y.)

to be longitudinally sectioned through the restoration. Once the samples were sectioned, only mesial parts of molars were selected to be assessed.

Tincture penetration in the ceromer-tooth interphase was assessed through observation in a CARL ZEISS, Germany, 2 objective 10 X microscope. Examiners were 10 dentists who had previously been informed about which scale to use. This scale was proposed by Capel et al.¹⁴ and was graded according to the following values:

- 0 = no micro-leakage.
- 1 = tincture penetration into the upper third of the cavity depth.
- 2 = Tincture penetration more than one third, but less than two thirds of the cavity depth.
- 3 = Tincture penetration surpassing two thirds of the cavity depth, but lesser than the axial wall.
- 4 = Tincture penetration encompassing the axial wall.

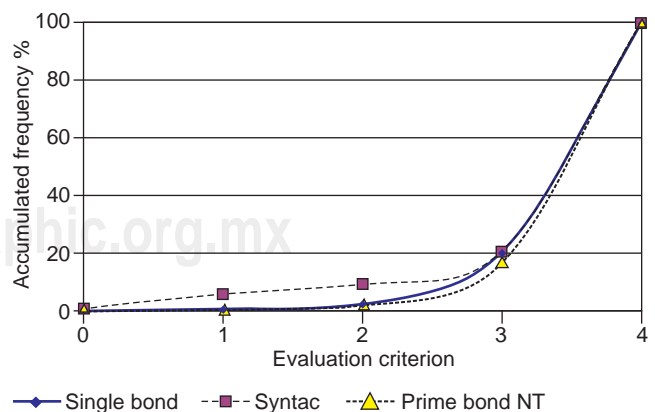
Results were analyzed with a cumulative frequency histogram.

RESULTS

The *figure 1* shows micro-leakage frequency found in this study. It shows that samples with value 4 (tincture penetration encompassing the axial wall) were more frequent; 485 were found in all three adhesive systems (*Table I*).

DISCUSSION

Results in the present study indicate there is no statistically significant difference among bonding systems



Source: Primary.

Figure 1. Micro-leakage accumulated frequency histogram according to bonding agent group.

Table I. Accumulated frequency.

Evaluation criterion	Frequency	Accumulated frequency	Accumulated frequency	Bonding system
0	0	0	0	Single Bond
1	1	1	0.5	
2	3	4	2.0	
3	37	41	20.5	
4	159	200	100.0	Classic syntac
0	1	1	0.5	
1	10	11	5.5	
2	7	18	9.0	
3	22	40	20.0	Prime and Bond NT
4	160	200	100.0	
0	0	0	0.0	
1	1	1	0.5	
2	3	4	2.0	
3	30	34	17.0	
4	166	200	100.0	

used with different solvents. In the same manner, in Capel et al.¹⁴ study, it was reported that, when using Prime & Bond NT, micro-leakage was totally eliminated in the enamel margins. Nonetheless, none of them totally removed micro-leakage in dentin. Philip et al.¹⁵ study differs from this exposition: in it, techniques of drying and dampening the tooth were followed. This study states that adhesive systems using acetone solvents, along with drying technique, reduce micro-leakage at gingival level, nevertheless, in enamel margins there is no statistical difference. Kanca¹⁶ in 1989 reported similar findings in his evaluation of five adhesive systems. He speculated that in some materials there could be areas where adhesion is interrupted, and that around these areas micro-leakage takes place. This phenomenon could be produced by contraction occurring during polymerization, with the resulting creation of spaces. According to scientific literature, the ability of a restorative material to seal the Internal phase with dental structure, is the most important factor to determine resistance to formation of future caries. Capel et al.¹⁴ found that dimensional changes of materials such as polymerization contraction, differences in thermal expansion coefficient and incomplete hygroscopic absorption can lead to micro-leakage production, originating thus recurrent caries, post operative sensitivity, margin pigmentation and pulp damage, leading then to the failure of the restoration. We need to point out as well that the material's resistance to dislodgement cannot be considered as a factor to predict sealing, since most bonding systems are estimated to be in the 17-20 MPa range.

When attempting to clinically prove dentin adhesion effectiveness one must be prudent. This prudence must also prevail when observing clinical indications of adhesive restorations, especially those located close to or below the cement-enamel junction. Restoration technique and dentist expertise are paramount to obtain proper sealing and adaptation while there are no contraction-free materials.

It is as well important to consider the fact that we must promote prevention. When dealing with restored teeth, proper control and follow-up are paramount in order to avoid complications in adhesive restorations which have encountered failure in a short period of time.

CONCLUSIONS

- There is no difference in micro-leakage degree among adhesion systems used with different solvents.
- Micro-leakage is present in greater amounts in the lower third of the class V cavity when compared to the upper third. This is due to the fact that permeability of dentinal tubules is greater in this area, added to the fact that enamel thickness is lesser.
- More research is needed to define proper polymerization form. In this process, light source can be an influencing factor, as well as deciding to polymerizing or not through tooth tissue. Filling technique followed is also of paramount importance.
- Adhesion systems must comply with different requirements depending on the substratum character-

istics. These can be: vitality and age of tooth, caries induced demineralization or amount of inorganic material.

REFERENCES

1. Asociación Americana para Ensayos de Materiales—ASTM. Date June 2002: Available to URL: <http://www.astm.org/cgi-bin/Soft-Cart.exe/index.shtml?E+mystore>
2. Going RE. Microleakage around dental restorations: A summarizing review. *J Am Dent Assoc* 1972; 84: 1349-1357.
3. Fusayama T, Nakamura M, Kurosaki N, Iwaku N. Non-pressure adhesion of a new adhesive restorative resin. *J Dent Res* 1979; 58: 1364-1370.
4. Asmussen E. Clinical relevance of physical, chemical and bonding properties of composite resins. *Op Dent* 1985; 10: 61-73.
5. Pashley D, Michelich U, Kiel T. Dentin permeability: Effects of smear layer removal. *J Prosthet Dent* 1981; 46: 531-537.
6. Nakabayashi N. Biocompatibility and promotion of adhesion to tooth substances. *Biocompatibility* 1984; 1: 25-52.
7. Glasspoole RA, Erickson RL, Pashley DH. The effect of surface moisture on bonding to dentin. *J Dent Res* 1991; 70: 457.
8. Mitchem ZC, Terkla LG, Gronas DG. Bonding of resin dentin adhesives under simulated physiological conditions. *Dent Mater* 1988; 4: 351-3.
9. Terkla LG, Brown AC, Hainisch AP, Mitchem JC. Testing sealing properties of restorative materials against moist dentin. *J Dent Res* 1987; 66: 1758-1764.
10. Kanca J. Effect of resin primer solvent and surface wetness on resin composite bond strength to dentin. *Am J Dent* 1992; 5: 213-215.
11. Gwinnett AJ. Moist versus dry dentinitis effect on shear bond strength. *Am J Dent* 1992; 5: 127-129.
12. Kanca J. Resin bonding to wet substratum. I: bonding to dentin. *Quintessence Int* 1992; 23: 39-41.
13. Lutz F, Krejci I, Oldenburg R. Elimination of polymerization stresses at the margins of posterior composite resin restorations: a new restorative technique. *Quintessence Int* 1986; 17: 777-784.
14. Capel PE, Placido E, Francci C, Perdigao J. Microleakage of class V resin-based composite restorations using five simplified adhesive systems. *Am J Dent* 1999; 12: 291-294.
15. Philip SW, Muir ES. Microleakage of bonding agents with wet dry bonding techniques. *Am J Dent* 1996; 9: 34-35.
16. Kanca J. Microleakage of five dentin bonding systems. *Dent Mater* 1989; 5: 415-416.

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