

Effect of adhesive system on microleakage in class II direct composite restorations

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This study evaluated microleakage at the dentin/composite and enamel/composite interfaces in Class II restorations using 4 different newly-developed experimental hydroxyapatite-filled adhesive systems. Ninety-six box-type Class II standardized cavities were prepared on both proximal surfaces of previously extracted sound human molars with the cervical margin located 1mm above or below the cemento-enamel junction (CEJ). Teeth were randomly assigned to 4 groups (n=12) and conditioned with one of the 4 adhesive systems. Premise nanocomposite was incrementally inserted as the final restoration. The teeth were thermocycled and immersed in 0.5% basic fuchsin solution for 24 hours. The specimens were cut into 3 slices and the resulted sections were examined using a stereomicroscope under 40x magnification. The microleakage at the enamel and cementum margins was evaluated using an ordinal scale (0-3). Data were analyzed using the Kruskal-Wallis and Wilcoxon test at a $p < 0.05$ level of significance. Significantly less microleakage was associated with the use of etch & rinse adhesive systems at the enamel surface. Between the enamel and the dentin margins significant statistical differences were found just for etch & rinse adhesives. Etch & rinse three-step adhesive containing water and ethanol as solvent performed significantly better at the dentin margins.

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1. Introduction

Resin composites with adhesive systems have been available on the dental market for about four decades and are widely used for both anterior and posterior restorations [1]. The ultimate goal of a dental adhesive system is to achieve a good and durable bond to the dental substrates. In order to be able to achieve this goal in the clinics, it is necessary for the clinicians to be aware of the limitations of these materials [2]. One is polymerization shrinkage which produces contraction stresses generally concentrated at the adhesive interface leading to marginal debonding [3].

Many efforts have been undertaken to minimize the extent of the interfacial gaps, including thicker adhesive layers or liners which may act as an elastic intermediate layer between the cavity walls and the adjacent composite resin [3,4]. Based on this idea, filled adhesives have been introduced [5-7]. These adhesives contain various types of inorganic fillers such as conventional glass, ion leachable glass, silica and aerosol silica nanofillers [8-10]. They have been reported to improve marginal seal of composite restorations, [1,6,11-13] to have higher mechanical properties and sufficient radiopacity to be discernible on dental X-ray films [7].

Compared to total-etch adhesives, the more recent self-etch adhesive systems are becoming increasingly popular, because of the reduced postoperative [14] and technique sensitivity [15]. In addition to this, fillers incorporated in adhesive resins may increase the adhesive viscosity, resulting in a reduction of adhesive penetration into the demineralized dentin and bonding to dentin [16,17].

The purpose of this study was to evaluate the effect of solvent formulation and application procedures of four experimental adhesive systems filled with hydroxyapatite on the microleakage in proximal cavities with the gingival margin located in either enamel or dentin. The adhesives are: (1) a three-step etch & rinse system with ethanol as solvent; (2) a three-step etch & rinse system with ethanol and water as solvent; (3) a two-step self-etching primer with ethanol as solvent and (4) a one-step all-in-one adhesive with ethanol as solvent. The null-hypothesis was that the more recent less technique sensitive all-in-one adhesive system will perform better in terms of microleakage, on both enamel and dentin margins than the others.

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2. Experimental

Forty-eight freshly extracted human molars were used for microleakage analysis. The teeth were cleaned in tap water, with calculus and soft tissue deposits being removed with a hand scaler and then stored in 0.9% NaCl containing 0.02% sodium azide at 4°C until used. Ninety-six box-type Class II standardized cavities were prepared on both proximal surfaces using a medium-grained diamond bur (SG881KS.014, Edenta AG, Switzerland) in a water-cooled high-speed turbine. The dimensions of the cavity preparation were: 4.0 mm buccolingual width, 1.0 mm gingival wall depth. The cervical margin was located 1.0 mm above the cemento-enamel junction (CEJ) for the mesial cavities and 1.0 mm below CEJ for the distal cavities [18]. The cavosurface margins were prepared at a 90° angle, and all internal angles were rounded. The teeth were randomly divided into four groups of 12 teeth.

All the adhesive systems used in this study were manufactured by the Dental Materials Section, Chemistry Research Institute "Raluca Ripan", Cluj-Napoca (Table 1).

Table 1. The adhesives used in this study.

(HEMA = hydroxyethylmethacrylate;
DMAEMA = dimethylaminoethylmethacrylate;
CQ = camforquinone; BisGMA = bis-phenol glycidyl
methacrylate;
TEGDMA = triethyleneglycoldimethacrylate)

Product/ Group	Component	Adhesion strategy
AD5.I Group I	Acid: 37% phosphoric acid Primer: acidic monomer, HEMA, ethanol, CQ, DMAEMA Adhesive: BisGMA, HEMA, acid monomer, CQ, DMAEMA, hydroxyapatite	Total etch 3 step
AD11.I Group II	Acid: 37% phosphoric acid Primer: acidic monomer, HEMA, ethanol, H ₂ O, CQ, DMAEMA Adhesive: BisGMA, HEMA, acid monomer, CQ, DMAEMA, hydroxyapatite	Total etch 3 step
AD11.II Group III	Primer: acidic monomer, polyacrylic acid, HEMA, Ethanol, CQ, amine Adhesive: BisGMA, TEGDMA, HEMA, acid monomer, CQ, DMAEMA, hydroxyapatite	Self-etch 2 step
AD5 Group IV	Primer&Adhesive: acidic monomer, poliacyrylic acid, HEMA, Bis-GMA, ethanol, CQ, amine, hydroxyapatite	Self-etch One step

The teeth were restored as follows:

Group I and II: The teeth were etched with 37% phosphoric acid gel for 15 s, rinsed with water spray for 15 s and dried with a gentle air stream for 2 s. Primers of AD5.I respectively AD11.I were applied with a microbrush for 15 s with a light brushing motion and air-thinned for 5 s with canned compressed air to achieve a visibly uniform layer. Adhesives of AD5.I respectively AD11.I were applied using a microbrush for 15 s, air-thinned for 5 s and light-cured for 20 s using an Optilux 501 curing unit (Kerr Corp., Orange, CA, USA).

Group III: The primer of AD11.II was applied with a microbrush for 15 s using a light brushing motion and air-thinned for 3 s with canned compressed air to achieve a visibly uniform layer. The adhesive of AD11.II was applied using a microbrush for 15 s, air-thinned for 3 s and light-cured for 20 s (Optilux 501, Kerr Corp.).

Group IV: Solution of AD5 was well mixed for 3 s and then applied for 20 s with a light brushing motion. A second layer of AD5 was placed using the same protocol and the excess solvent was evaporated by air drying for 5 s. The adhesive was light-cured for 20 s (Optilux 501, Kerr Corp.).

A steel matrix (Adapt Super Cap, KerrHawe, Bioggio, Switzerland) was applied with Super Mat (KerrHawe, Bioggio, Switzerland), and each tooth was restored using Premise Packable nanocomposite (Kerr Corp.). Composite resin was incrementally inserted using oblique layering technique [19] (Fig. 1). Each increment of resin-based composite (RBC) was light-cured for 40s from the occlusal, and after removal of the steel matrix, also from the buccal and lingual surfaces, using an Optilux 501 curing unit (Kerr Corp.) operating in standard mode at a light intensity of 740 ± 36 mW/cm². The restorations were finished with BluWhite diamond burs (Kerr Corp.) and polished using a series of abrasive disks (OptiDisc, Kerr Corp.) and rubber points (HiLuster Dia Polishers, Kerr Corp.).

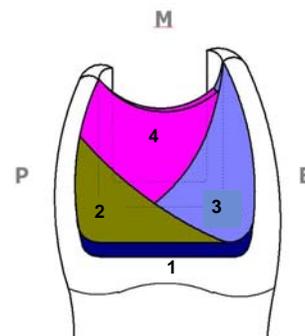


Fig. 1. Schematic representation of the oblique incremental placement of the composite resin.

The restored teeth were stored 7 days in water at 37°C and then subjected to thermocycling between 5°C/55°C for

1000 cycles, with an immersion time of 25 s in each bath and a dwell time of 10 s.

The apices of the teeth were sealed with resin composite and the tooth surfaces were covered with two layers of nail varnish with the exception of 1 mm around the tooth-restoration. The teeth were then immersed in 0.5% basic fuchsin for 24 hours, then removed from the dye, washed in tap water and air-dried. Finally, their roots were mounted in self-curing acrylic resin.

Each specimen was sectioned in a mesiodistal direction into three sections of approximately 1.5 mm width using a water-cooled microtome (Isomet Low Speed Saw, Buehler Ltd). Each section was evaluated for dye penetration at the gingival enamel and dentin margins at 40x magnification with a stereomicroscope (Leitz, Wetzlar, Germany). The microleakage was evaluated using scores, according to an ordinal scale:

0 = No dye penetration

1 = Penetration along the half of the cervical wall

2 = Penetration along the entire depth of the cervical wall

3 = Penetration extended along the axial wall

The highest score (of the 4 scores per cavity) was recorded and used in the statistical analysis. Kruskal-Wallis test and Wilcoxon test were used for statistical analysis and comparison of dye penetration scores between groups at a $p < 0.05$ level of significance.

3. Results

At the enamel margin, Group I (median=0) recorded significantly less microleakage than Group III (median=3) ($p=0,00004$), and Group IV (median=3) ($p=0,00005$), with no significantly difference when compared with Group II (median=1) ($p=0,39$). Significantly less microleakage was recorded also for Group II compared with Group III ($p=0,0001$) and Group IV ($p=0,0001$) (Table 2).

Table 2. Frequency of dye penetration scores as an indicator of marginal microleakage in enamel margin ($n = 12$ per group).

Group (n=12)	Enamel Scores			
	0	1	2	3
I	8	3	1	0
II	6	4	2	0
III	0	1	3	8
IV	0	1	4	7

At the dentin margin, microleakage for Group II (median=2) was significantly reduced than for Group III (median=3) ($p=0,0006$) and Group IV (median=3) ($p=0,008$), but similar in comparison with Group I (median=2) ($p=0,12$) (Table 3).

Table 3. Frequency of dye penetration scores as an indicator of marginal microleakage in cementum margin ($n = 12$ per group).

Group (n=12)	Dentin Score			
	0	1	2	3
I	0	3	4	5
II	0	5	6	1
III	0	0	3	9
IV	0	1	4	7

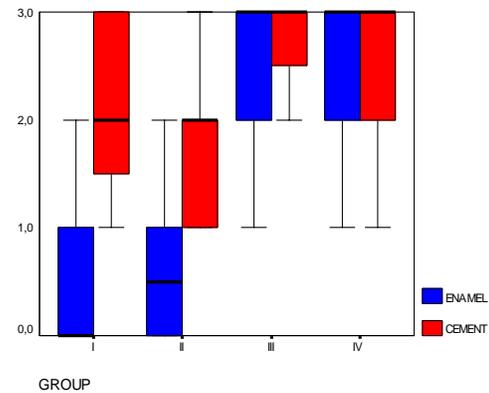


Fig. 2. The microleakage scores of the restored Class II cavities.

4. Discussion

For many years the dental profession has strived to achieve good adhesion of resin composites to tooth substrates, since reliable bonding should produce less microleakage and a higher rate of clinical success [20].

The filler of the adhesives used in this study consists of 30% hydroxyapatite. Several studies have shown that filled adhesives are expected to act as an intermediate shock-absorbing elastic layer between the composite resin and dentin, thus reducing the stress at the interface and diminishing microleakage [3,4]. However other studies have shown that filled adhesives were not capable to penetrate into the spaces between the collagen fibers because the width of interfibrillar spaces is about 20 nm [1,9]. In this study, the microleakage scores at the dentin margins showed that three of four adhesives (Group I, III and IV) performed similarly, regardless of the application procedure.

Self-etch adhesives have been classified based on their ability to penetrate smear layers and their depth of demineralization into the subsurface dentin as mild, intermediary strong and strong [15,21]. The self-etch adhesives used in this study (AD11.II and AD5) can be considered as mild because of their 2.0 pH value. „Mild” self-etch adhesives demineralize dentin only to a depth of 1 μm , keeping residual hydroxyapatite still attached to collagen [3]. The preservation of hydroxyapatite within the submicron hybrid layer may serve as a receptor for additional chemical bonding with the functional monomers

and may help prevent marginal leakage [15]. Furthermore, self-etch adhesives used in this study contain high concentration of ethanol which should remove all water and solvent at the end of the etching time. On the other hand acidic monomers, dissolved calcium and phosphate ions may lower their vapor pressure [1]. Residual water in the dentin subsurface may interfere with polymerization of the mixture of the self-etching primer and the adhesive resin, thereby lowering the quality of the hybrid layer. This may be an explanation for the higher level of microleakage obtained with these self-etch adhesive systems compared to the total-etch ones.

Over- or under drying of acid-etched dentin is a very technique-sensitive step. Gentle post-conditioning air-drying of acid-etched dentin and enamel following a „dry-bonding” technique still guarantees effective bonding when a water/ethanol-based adhesive is used [15]. The presence of 5% water along with ethanol as a solvent in AD11.I total-etch adhesive system seems to offer better marginal seal than the total-etch adhesive system AD5.I when applied at the dentin margin. As revealed by other studies, three-step ethanol-water-based etch-and-rinse adhesives are still regarded as the „gold standard” in terms of sealing durability, especially in demanding cavity preparations that have exposed dentin margins [22].

5. Conclusions

Within the limits defined in the experimental design, the following conclusions may be drawn:

- Significantly less microleakage was associated with the use of three-step etch & rinse adhesives at the enamel margin compared with the dentin margin.
- Etch & rinse adhesives sealed better the enamel margins compared to the self-etch adhesives.
- At the dentin margin only etch & rinse three-step adhesive system with water and ethanol as solvent performed significantly better.
- The all-in-one adhesive system did not perform better than three-step etch-and-rinse adhesive systems.

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