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ORIGINAL RESEARCH

An *in vitro* Evaluation of Microleakage of Three Different Self-Etch Adhesives with Ethanol, Acetone and Water as Solvents

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ABSTRACT

Purpose: The aim of this study is to evaluate and compare the microleakage of three different self-etch adhesives with ethanol, acetone and water as solvents.

Materials and methods: Standardized class V cavities were prepared on both facial and lingual surfaces of 60 caries-free human premolar teeth. The teeth were divided in three groups and different self-etch adhesives (OptiBond All in One, Tetric N Self-Etch, Adper Easy One) were applied according to manufacturer's instructions. The samples were then restored with composite and subjected to thermocycling. Later, the teeth were stained with methylene blue, sectioned and measured for microleakage under stereomicroscope.

Results: This study showed that at the occlusal margin and gingival margins, the preparations treated with OptiBond all in one showed significantly less leakage than the other groups. Enamel margins provided better marginal sealing than dentin/ cementum margins.

Conclusion: The study demonstrated that OptiBond All in One bond had a better sealing ability at both occlusal and gingival margins compared with the other adhesives used.

Keywords: Microleakage, Self-etch adhesives, Solvents.

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INTRODUCTION

The primary aim of dental adhesives is to provide retention to composite fillings or composite cements. In addition to withstanding mechanical forces, and in particular shrinkage stress from the lining composite, a good adhesive also should be able to prevent leakage along the restoration's margins. Clinically, failure of restorations occurs more often due to inadequate sealing, with subsequent discoloration of the cavity margins than due to loss of retention.[1]

Microleakage is defined as the clinically detectable passage of bacteria, fluids, molecules or ions between cavity wall and the restorative materials applied to it. Achieving a micromechanical and biomechanical bond between the restoration and tooth is considered effective and a standard procedure in clinical practice. Therefore, good insights in the components of the adhesives are paramount to understand or even predict their behavior.[2]

The addition of solvents to resins is indispensable to the composition of adhesives that need to bond to dentin. The wet nature of dentin only allows good wetting when a hydrophilic bonding is applied.[3] The wetting behavior of adhesive is drastically improved by addition of hydrophilic monomers and solvents.[4] The low viscosity of primers and/ or adhesive resins is partly due to the dissolution of the monomers in a solvent and will improve its diffusion ability in the microretentive tooth surface. In adhesives, water, ethanol and acetone are the most commonly used solvents.

The objective of the present *in vitro* study is to compare the sealing ability of three different self-etch adhesives with ethanol, acetone and water as solvents.

MATERIALS AND METHODS

Sixty freshly extracted, caries-free human premolars were kept in distilled water at 4°C for 24 hours. Class V cavities were prepared on facial and lingual surfaces, with the gingival margin 1 mm below the CEJ, using a #4 round bur with a high speed handpiece and copious amounts of water. The preparations were standardized as 4 mm long, 3 mm wide and 2 mm in depth, and no bevels were placed. The teeth were randomly divided into three groups corresponding to each adhesive system (Table 1). Twenty teeth were assigned to each group and adhesives were applied according to manufacturer's instructions.

The cavities were later restored with resin composite (Charisma, Heraeus Kulzer) and finished using carbide burs. The restored teeth were thermocycled 1000× at a temperature of 5°C and 55°C. The dwell time was 30 seconds in each water bath, with a transfer time of 30 seconds between each bath. The samples were then blotted dry with a paper towel and the roots were sealed with sticky wax. An acid-resistant varnish was applied to all surfaces of the teeth except for 1 mm adjacent to the restoration margins.

The specimens were then subjected to dye leakage tests. They were immersed in 0.5 methylene blue dye solution for 24 hours. After staining, the teeth were rinsed in running water and radicular parts of the teeth were cut 4 to 5 mm below the CEJ. The coronal parts were then sectioned mesiodistally in the approximate center of the restoration with a low-speed diamond saw (Fig. 1).

Two specialists in restorative dentistry (Cohen's Kappa 0.74) assessed the restorations independently for dye

Table 1: Different self-etch adhesives evaluated in the study

Groups	*Adhesive system*	*Composition*	*Manufacturer*
I	Adper Easy One	2-hydroxymethyl methacrylate (HEMA), Bis-GMA, methacrylated phosphoric esters,1,6 hexanediol methacrylate, Vitrebond copolymer, filler particles, ethanol, water, initiators and stabilizers	Batch# 426422 Manufacturer: 3M ESPE, St Paul, MN, USA
II	Opti-Bond All in One	Glycerol phosphate dimethacrylate (GPDM), mono-, and di-functional methacrylate monomers, ethanol, acetone and water as solvents, photoinitiators, nanofillers and fluoride-releasing fillers	Batch# 3332328 Manufacturer: Kerr Co, Orange, CA, USA
III	Tetric N-Bond Self-Etch	Bis-acrylamide derivative, bismethacrylamide dihydrogen phosphate, amino acid acrylamide, hydroxyalkyl methacrylamide, water, fillers, initiators and stabilizers	Batch# N16261 Manufacturer: Ivoclar Vivadent, Amherst, NY, USA

Fig. 1: Sectioning of a tooth with diamond disk

penetration at occlusal and gingival margin using a stereomicroscope (20×).

Dye penetration using stereomicroscope was done according to following criteria (Fig. 2):

0 – No leakage.
1 – Dye penetration up to one-third of the cavity.
2 – Dye penetration up to two-third of the cavity.
3 – Dye penetration up to full cavity depth.
4 – Dye penetration onto the axial wall of the cavity preparation.

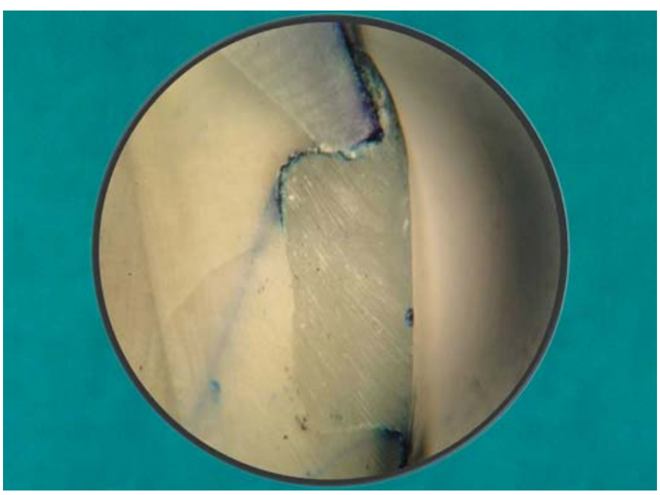

Fig. 2: A score of 4 at occlusal margins and score 1 at gingival margins

RESULTS

Data were analyzed using nonparametric analysis of variance (ANOVA) (Kruskall-Wallis test). Between group comparisons were made using Mann-Whitney U test and within group comparisons between two locations (occlusal *vs* gingival) were made using Wilcoxon signed rank test. The confidence of the study was kept at 95%; hence, a p-value less than 0.05 indicated a significant difference.

Dye penetration scores for the occlusal and gingival walls are presented in Table 2.

Kruskal-Wallis one-way ANOVA indicated significant differences between groups for occlusal and gingival scores ($\chi^2_{df=2}$ = 14.958, p < 0.001, $\chi^2_{df=2}$ = 37.989, p < 0.001, respectively).

The Mann-Whitney test (Table 3) was performed to evaluate significant differences of occlusal and gingival scores between groups. The results demonstrated no significant leakage differences among Adper Easy One and OptiBond All in One. Conversely, Tetric N Self-Etch had significantly more dye penetration when compared to Adper Easy One and OptiBond All in One.

Gingival scores between groups showed some significant differences: Tetric N Self-Etch showed significantly more dye penetration when compared to Adper Easy One and OptiBond All in One.

When comparing the occlusal and gingival scores for each group, the Wilcoxon rank test (Table 4) showed the mean microleakage at occlusal location was significantly lower as compared to that at gingival location in all the three groups.

DISCUSSION

Solvents are substances that are capable of dissolving or dispersing one or more other substances. When a solvent dissolves a solid or a liquid, the molecules (or ions) become separated from each other and the spaces in between become occupied by solvent molecules. The energy required to break the bonds between solute molecules is supplied by the formation of bonds between the solute particles and the solvent molecules: the old intermolecular forces are replaced

Table 2: Dye penetration scores at coronal and gingival margins (Kruskal-Wallis test)

	0	1	2	3	4	Mean	Standard deviation
Coronal							
Adper Easy One	24	13	3	0	0	0.475	0.640
OptiBond All in One	26	14	0	0	0	0.350	0.483
Tetric N Self-Etch	12	19	9	0	0	0.925	0.730
Gingival							
Adper Easy One	9	14	10	3	4	1.475	1.219
OptiBond All in One	12	16	8	2	2	1.150	1.075
Tetric N Self-Etch	0	1	19	11	9	2.700	0.853

Table 3: Mann-Whitney test to evaluate differences between groups

S.no.	Comparison	Coronal			Gingival		
		Mean difference	z-value	p-value	Mean diff.	z-value	p-value
1.	Adper Easy *vs* OptiBond All in One	0.125	0.693	0.488	0.325	1.216	0.224
2.	Adper Easy *vs* Tetric N	–0.450	2.832	0.005	–1.225	4.673	<0.001
3.	OptiBond All in One *vs* Tetric N	–0.575	3.642	<0.001	–1.550	5.810	<0.001

Table 4: Microleakage at coronal *vs* gingival locations (Wilcoxon signed rank test)

S. no.	Group	z-value	p-value
1.	Adper Easy	5.122	<0.001
2.	OptiBond All in One	4.815	<0.001
3.	Tetric N	5.857	<0.001

by new ones. The solubility characteristics of molecules are determined chiefly by their polarity.[5]

Chemists have classified solvents into three categories according to their polarity: polar protic, dipolar aprotic and apolar solvents. Polar protic solvents consist of a hydroxyl-group that can form strong hydrogen bonds. Examples are water and ethanol. Polar aprotic solvents do not have the required hydroxyl-group to form hydrogen bonds but do have a large dipole moment. They usually also contain a ketone group. Typical example is acetone. Apolar solvents have both a low dielectric constant and dipole moment.

In adhesives, water, ethanol and acetone are the most commonly used solvents (Table 5). Other polyvalent alcohol solvents have been evaluated but are not used commercially.[6] The use of these organic solvents in adhesives must be explained by their inexpensiveness, their wide availability and their good biocompatibility. Most important characteristics of a solvent are its dipole moment, dielectric constant, boiling point, vapor pressure and H-bonding capacity. The vapor pressure of a solvent is important to ensure good evaporation of the solvent after application of the adhesive onto tooth tissue.[7,8] Remaining solvent in the adhesive may jeopardize polymerization due to dilution of the monomers and may result in voids and hence permeability of the adhesive layer.[9,10]

The results showed that at both occlusal and gingival margins group II (OptiBond All in One) exhibited lowest microleakage than the other bonding systems, followed by Adper Easy One and Tetric N Self-Etch.

OptiBond All in One contains a unique combination of ternary solvents that is ethanol, acetone and water. Ethanol is a polar solvent that forms hydrogen bonds with its solutes. Its high vapor pressure as compared to water allows better evaporation by air drying. Ethanol when used in conjunction with water forms an ‘azeotropic mixture’. This implies the formation of hydrogen bonds between water and ethanol molecules, resulting in a better evaporation of these water-ethanol aggregates than pure water. Self-evidently, this results in more water removal from the adhesive and lesser chances of void formation.[5,11]

The high vapor pressure of acetone, which is four times that of ethanol, is a main advantage. It facilitates better evaporation by air-drying. Similar to ethanol, acetone and water make an azeotrope. It has a very good water removing capacity, because of its high dipole moment and excellent evaporation capacities.[12] This is often referred to as the ‘water-chasing’ capacity of acetone.[13]

Table 5: Main characteristics of solvents used in adhesives

Solvent	Dipole moment in gaseous state in Debye at 25°C	Dielectric constant at 293°K (20°C)	Boiling temperature (°C)	Vapor pressure in mm Hg at 25°C
Water	1.85	80	100	23.8
Acetone	1.69	24.3	78.5	54.1
Ethanol	2.88	20.7	56.2	200

Also, the filled adhesive technology of OptiBond All in One, i.e., the presence of a nanofiller and fluoride-releasing filler ensures the higher level of protection against microleakage.

Adper Easy One also contains ethanol and water as solvents which explain for its comparatively lesser microleakage than Tetric N self-etch**,** which contains only water as the solvent. The high boiling temperature and low vapor pressure of water imply that this solvent is difficult to remove from adhesive solutions after application on the tooth. In addition, the equilibrium of water between fluid and gaseous state is also in favor of the fluid state, which will decrease the rate of evaporation even more.[14] Tay et al,[15] showed that excess water in the adhesive resin compromises with the marginal adaptation due to entrapment of water blisters resulting in overwet phenomenon.

The presence of a specific polyalkenoic copolymer–Vitrebond also plays a significant role in the inferior performance of Adper Easy One when compared to OptiBond All in One. The rationale for the use of this polymer was to provide better moisture stability. However, several authors demonstrated that this monomer does not dissolve in the adhesive's solution, leading to a separate phase producing many globules within the polymer of the adhesive layer, thus compromising with its marginal adaptation.[16,17]

The results also showed that the microleakage at the enamel margins is less as compared to that of the microleakage at the dentin/cementum margins. This difference can be explained by the composition of these two tissues which is very different. Dentin is a wet tissue and contains more water than enamel and this water interferes with the adhesive particles. These ones can penetrate the dentin only if they are hydrophilic. The formation of smear layer at the surface of the dentin, after the use of burs, blocks dentinal tubules and prevents all direct contact of any material with the dentinal substrate. The bond obtained in this situation is insufficient and fragile, with much higher possibilities for microleakage.

CONCLUSION

Dental adhesives are intricate mixtures of ingredients. Profound knowledge of these ingredients is one key to better understanding the behavior of adhesives in studies and in clinic. Good understanding also provides better insights in the correct clinical use of adhesives. Each ingredient has to some extent a specific effect on the microleakage, bonding efficiency, bonding durability, shelf life and biocompatibility of the adhesive system. In addition, ingredients may affect each other in a complicated interplay of factors. Unbalanced mixtures of ingredients may lead to reduced bonding effectiveness, sealing ability, durability and to phase-separation reactions, while a well thoughtout formulation will be the key to long-term clinical success.

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