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Influence of filler existence on microleakage of a self-etch adhesive system

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Abstract

Aim: This study evaluated the effect of filler existence in self-etch adhesive resin on the marginal leakage of a class V restoration.

Materials and Methods: Class V cavities were prepared and restored with a resin composite on the buccal surfaces of 48 premolars lined with unfilled or filled adhesives (n = 24). After thermo cycling, teeth in each group were divided to two subgroups (n = 12), specimens of the first subgroup were incubated for 24 h in distilled water at 37°C, and for the second group three months in the same condition. Specimens were placed in 50% silver nitrate for 24 h at 37°C, and then were cut buccolingually 1 mm thick. Dye penetration was measured using a stereomicroscope and scaled from 0 to 5 in a blind method. SEM images were made to evaluate the dentin-adhesive interfaces. Collected data were analyzed using the nonparametric Kruskal-Wallis and Mann-Whitney U-tests at a significant level of P<0.05.

Results: There was no significant difference between microleakage of filled and unfilled adhesive at 24 h and 3 months (P<0.05). There was a significant difference in cervical microleakage between 24 h and 3 months, which was independent on filler load of the adhesive (P<0.001). In contrast, there was no significant difference in occlusal microleakage between 24 h and 3 months and the cervical microleakage was significantly higher than occlusal microleakage after 3 months. SEM images revealed that unfilled adhesive infiltrate slightly

better than filled adhesive.

Conclusion: The application of filler particles in a self etch adhesive system had no influence on marginal leakage at both the enamel and dentin margins. While the unfilled adhesive infiltrate better than the filled adhesive, its long term performance is not promising.

Keywords: Filled; microleakage; self-etch adhesive; unfilled

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Introduction

To ensure a successful restorative procedure, the bonded interface area must be capable of withstanding the stresses caused by polymerization shrinkage, occlusal loading and thermal changes. Marginal discoloration, recurrent caries and post-operative sensitivity may be associated with the penetration of bacteria and oral fluid due to an incomplete marginal adaptation at the enamel-dentin resin composite interface. ^[1]

There has been increasing interest in the incorporation of fillers into dentin adhesives. These fillers originate from conventional glass fillers, silica fillers, to nanometer-sized aerosol silica. ^{[2],[3]} For self-priming adhesives and single-step systems that do not require an additional bonding resin layer for coupling resin composite to the primed dentin, inclusion of fillers increases their viscosity that tends to prevent over-thinning of unfilled adhesive layers, thereby preventing incomplete polymerization caused by oxygen inhibition. ^[4] They may also provide stress relief capacities against shrinkage stresses generated during the polymerization of dental composites, in a way that is similar to the use of resin composite liners and flowable composites. ^{[5],[6]} Manufactures have added filler to adhesives in an attempt to increase the strength of the adhesive, to modify the viscosity and to provide fluoride-releasing and radio-opacity particles. ^[7] The perceived advantages of filler inclusion in dentin adhesives as stress buffer remains unpredictable and has not been substantiated in a recent clinical trial. ^[8] A recent study of Giannini *et al.* concluded that filler addition to bonding agents can increase the flexural strength and modulus; however, results are product dependent. ^[9] They also suggested that filler addition does not necessarily have to increase the flexural strength and flexural modulus. ^[10]

A previous study showed no significant deference in tensile bond strength of filled and unfilled adhesive using low-viscosity composites, while the failure mode of fractures were different. ^[11] However, it is difficult to prove the effect of filler inclusion in dentin adhesives if they are used in combination with flowable composites or different filled adhesives are used or when they are compared with unfilled adhesives that have dissimilar resin compositions. ^[12] Therefore, the objective of this study was to examine the microleakage

pattern of resin-dentin/enamel interfaces created by a filled and an unfilled version of the same self-etch bonding (Clearfil SE Bond) after thermo-cycling.

Materials and Methods

A total of 48 non-carious human premolars stored in 0.5% chloramine T at 4°C solution for maximum 1 month following extraction, were used for the experiments. After being cleaned and pumiced, the teeth, including the apical third of their roots, were embedded in cold cure polystyrene resin cylinders (Acropars, Tehran, Iran). The occlusal portion of the tooth was transversally sectioned under water irrigation, allowing the configuration of a flat standard "occlusal" surface 4 mm above the cemento-enamel junction (CEJ). Class V cavity preparations were performed on the buccal surface, with a high speed carbide bur (D and Z, Germany), under water spray. Each bur was used for the preparation of four cavities and was then replaced by a new bur. Preparations were centered on the CEJ and had the following dimensions: 1.5 mm axial depth, 3.0 mm occluso-gingival height (1.0 mm under CEJ and 2.0 mm above CEJ) and 2.0 mm mesio-distal width. Margins received 45° bevel width of 0.5-1.0 mm.

Prepared specimens were randomly allocated into two groups ($n = 24$) in accordance with the filler existence of the adhesive system. For the first group Clearfil SE Bond (Kuraray Medical Inc., Kurashiki, Okayama, Japan), which is a filled two-step self-etch and for the second group experimental SE Bond provided by the same manufacture, as an unfilled bond with the same composition, were used following the manufacturer's instructions. Restorations were finalized with Filtek Z250 composite in 3 increments (first inciso-axially, second gingivo-axially and finally gingivo-incisally) and light cured for 40 s between each increment using a light-emitting diode curing unit (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein).

Teeth were stored in deionized water at 37°C in a humidity chamber (maintained at 95% relative humidity) for 24 h prior to thermo cycling. Thermo cycling was achieved with a programmed robot (Vafae Manufacture, Tehran, Iran) that alternated the specimens between two temperature controlled water baths at 5°C and 55°C, respectively with a dwell time of 30 s in each bath for 2 days (1,000 cycles). After thermal cycling, teeth in each group were divided into two subgroups ($n = 12$), specimens of the first subgroup were incubated for 24 h in distilled water at 37°C and for the second group 3 months in the same condition.

After the corresponding period for each subgroup the entire surface of each tooth (with the exception of the restoration and 1 mm of tooth structure adjacent to the restoration) was covered with two layers of nail varnish (Max Factor, France) to prevent dye penetration into the tooth except at the resin-tooth interface. The specimens were placed in 50% silver nitrate for 24 h at 37°C. After washing the teeth, they were sectioned using an Isomet slow-speed saw (Buehler Ltd, Evanston, IL, USA) with a diamond blade in water, longitudinally in a bucco-lingual direction yielding a 1.0-1.5 mm section through the center of the restoration.

Dye penetration was measured using a stereomicroscope at $\times 32$ magnification according to the following 5 point interval scale: 0 = no leakage, 1 = leakage restricted to the enamel, 2 = leakage into dentin but not reaching the axial cavity wall, 3 = leakage reaching the axial cavity wall and 4 = leakage beyond the axial cavity wall reaching the pulp. All evaluations were carried out in a blind study with no information to the examiner about the identity of the

adhesive system used. One examiner measured each section separately resulting in 24 data points for each subgroup (12 teeth × 2 sections).

Scanning electron microscopy (SEM) analysis

The sections were demineralized in hydrochloric acid solution (6 mol/l) for 10 s and deproteinated in 1% sodium hypochlorite solution for 10 min., sputter-coated with gold and examined in a SEM (Seron, model S-2500, Gocheon-dong, Uiwang-si, Gyeonggi-Do, 437-801, Republic of Korea) using an accelerating voltage of 30.0 kV.

Statistical analysis

The results of the microleakage investigation were analyzed using the nonparametric Kruskal-Wallis on ranks and *post-hoc* Mann-Whitney U-test at a significant level of $P < 0.05$ (SPSS 14.0 for Windows, SPSS Inc., Chicago, IL, USA).

Results

The microleakage index at resin-dentin/enamel interfaces at two different locations after 24 h and 3 months for an unfilled and filled bonding system are summarized in [Table 1]. The Mann-Whitney tests showed that there was no significant difference between the filled and unfilled adhesive systems ($T = 2447.5$, $P = 0.383$). The non-parametric Kruskal-Wallis on ranks showed that specimens of 3 months at the cervical location had a significant higher microleakage index (3.1) compared to occlusal specimens at 24 h (1.7) and 3 months (0.6) and the cervical specimens at 24 h (1.2) ($H = 38.9$, $P \leq 0.001$). This implies that the microleakage index after 24 h for the cervical and occlusal location were not significantly different. Furthermore, there was no significant different between leakage at 24 h and 3 months at the occlusal surface.

Leakage Index	Cervical				Occlusal							
	0	1	2	3	4	Average	0	1	2	3	4	Average
24 h filler	2	3	4	3	0	1.7	2	3	3	4	0	1.8
24 h no filler	8	1	1	2	0	0.8	3	1	6	2	0	1.6
24 h total	10	4	5	5	0	1.2	5	4	9	6	0	1.7
3 months filler	1	0	3	2	6	3.0	8	2	1	0	1	0.7
3 months no filler	0	1	2	2	7	3.2	6	6	0	0	0	0.5
3 months total	1	1	5	4	13	3.1	14	8	1	0	1	0.6

Table 1: Frequency distribution together with the averages of filled and unfilled self-etch bond after thermo cycling at 24 h and 3 months

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SEM analysis revealed that unfilled SE bond can better infiltrate the dentinal tubules than filled SE bond by having longer resin tags [Figure 1].

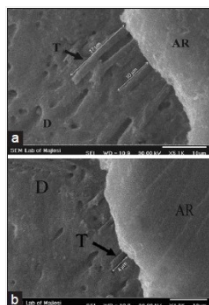


Figure 1: (a) Scanning electron microscopy (SEM) image, analysis for unfilled SE bond showing a view of a bonded area in unfilled adhesive version, (dentin [D] and adhesive resin [AR]), arrow showing the resin tag (T). The length of resin tags is indicated in μm . (b) SEM image, analysis for filled SE bond showing view of a bonded area in filled adhesive version

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Discussion

In the current study significantly greater leakage at the gingival wall was indicated compare to occlusal wall for all groups. This finding is in agreement with some authors, who used different combinations of bonding agents and resin composites in both Class II and Class V restorations. ^{[13],[14]} The higher leakage scores detected in gingival wall compare with occlusal wall can be related to the structure of these two walls. Enamel thickness is greater in occlusal wall compare to gingival wall and bonding to enamel is a relatively simple process without major technical requirements or difficulties. On the other hand, bonding to dentin presents a much greater challenge, as it contains a substantial proportion of water and organic materials, which presents a moist surface that impairs the bonding mechanism. ^[15] Moreover, it has been indicated that marginal dentin reveals more sensitivity to microleakage after thermo-cycling.

Microleakage is frequently used for assessing the quality of the tooth/restoration interface. ^[16] Dye penetration measured on sections of restored tooth is the most common technique for evaluating microleakage at the tooth/restoration interface. ^[17] Although this method is simple, economic and fast the subjectivity of reading the specimens has been noted as being a shortcoming relating to this methodology. To overcome this problem all evaluations were carried out in a blind study.

Clearfill SE Bond was used in this study as it was used in different previous studies and the results could be compared in this way. Self-etching systems are generally considered less technique-sensitive, when compared with systems that utilize separate acid conditioning and rinsing steps. Clearfil SE Bond contains 10% silanated colloidal silica particles in micro size. ^[18] To compare the influence of presence of fillers in adhesive system, an experimental unfilled SE bond was provided by the factory. Results indicated no significant difference between microleakage in SE Bond and experimental SE Bond. Cardoso *et al.* in their study also found no statistical difference between microleakage in Prime and Bond NT and Exp. NT (NT contains nanofillers, Exp. NT without filler). They showed that these two adhesives were quite successful in reducing leakage at dentin margins and resulted in virtually no leakage at the enamel margins. ^[19] Nunes *et al.* evaluated the effects of adhesive composition on microtensile bond strength (μ TBS) to human dentin and they reported no significant difference between μ TBS of filled and unfilled NT. ^[20] In contrast they found a statistically significant difference between μ TBS of Single Bond and Exp. Single Bond (SB unfilled, Exp. SB 10% ZrO/SiO₂). This can be explained by the different in size of fillers as NT contains nanofillers, which theoretically can penetrate through the demineralized dentine substrate specially the spaces between collagen fibrils, ^[21] while in Exp. SB Z100 fillers were added and they are in micro size (0.6 μ m). The width of the interfibrillar spaces is about 20 μ m, ^[3] therefore, the etched dentin may act as a sieve, with filler accumulating on the top and obstructing the resin penetration into the dentin below. It needs to be mentioned that Tay *et al.* found that also nanofillers from the adhesive layer were congested around the tubular orifices, but were not found within the interfibrillar spaces of the hybrid layer. ^[3]

SEM analysis in this study revealed, the unfilled adhesive performed slightly better than the filled SE bond, as the length of the resin tags were almost 3 times than the filled adhesive

[Figure 1]. Both filled and unfilled SE bond revealed the presence of incompletely infiltrated collagen fibrils. Moreover the presence of resin tags in filled adhesive was more irregular, which can be a prove for congestion of filler particles within the interfibrillar space. There is an ongoing debate on the importance of the resin tags influence on the bond strength to dentine. Recently Lohbauer *et al.* reported that resin tag formation did not influence the μ TBS, which is also unrelated to the C-factor. [22]

Filled adhesives were expected to act as an intermediate shock-absorbing elastic layer between resin composite and dentin, thus increasing the bond strength to dentin. [20]. [23] Many studies evaluated comparisons between filled and unfilled adhesives; however, the advantages of these adhesives as stress buffers remain unpredictable, [8] and have not been substantiated with *in vitro* bond tests [24] and a clinical trial. [25] According to the results of the current study no statistically significant difference was observed between microleakage of SE Bond (filled) and Exp. SE Bond (unfilled) before and after 3 months water storage. These findings shows that presence of filler has no influence on microleakage of SE Bond immediately or after 3 months hydrolytic degradation.

The simplified procedure for dental bonding systems are of great interest, but the presence of fillers appears questionable if the bonding systems are employed in only one layer. However, future studies are needed to confirm this.

Conclusion ▲

The application of filler in a self-etch adhesive system had no influence on marginal leakage at both the enamel and dentin margins. While the unfilled adhesive infiltrate better than the filled adhesive, its long-term performance is not promising.

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Figures

[\[Figure 1\]](#)

Tables

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