

## Microleakage assessment of one- and two-step self-etch adhesive systems with the low shrinkage composites

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### Original Article

#### Abstract

**BACKGROUND AND AIM:** Different studies evaluating one-step self-etch (SE) adhesive systems show contradictory findings, so the aim of this study was to compare the microleakage of one-step SE adhesive systems and CLEARFIL SE BOND (CSB) (that serves as the “gold-standard” SE adhesive) with low shrinkage composites.

**METHODS:** In this in vitro study, Class V cavities with the occlusal margin in enamel and cervical margin in cementum were prepared on the buccal and lingual surfaces of 36 human premolars and molars (72 cavities). The enamel surfaces of the cavities were etched with 37% phosphoric acid and then the specimens were divided into six groups of 6 (12 cavities) and the cavities were restored according bellow: Group 1 (Kalore-GC + G-Bond), Group 2 (Grandio + Futurabond NR), Group 3 (Aelite LS Posterior + All Bond SE), Group 4 (Kalore-GC + CSB), Group 5 (Grandio + CSB), and Group 6 (Aelite LS Posterior + CSB). All the specimens were thermocycled for 2000 cycles (5-55 °C) and then placed in 0.5% basic fuchsin dye for 24 hours at 37 °C and finally sectioned and observed under the stereomicroscope. Data were analyzed using Kruskal-Wallis, Mann-Whitney, and Wilcoxon tests at a  $P < 0.050$  level of significance.

**RESULTS:** In comparison between occlusal and gingival margins in each group, microleakage in occlusal margins was significantly less than the gingival margins (except Kalore + CSB) ( $P > 0.050$ ). There were no significant differences in microleakage among two-step and one-step SE adhesive systems on both the occlusal and gingival margins.

**CONCLUSION:** According to this study, two-step SE adhesive system (CSB) did not provide better marginal seal than the one-step SE adhesive systems.

**KEYWORDS:** Composite Resins, Adhesives, Polymerization

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The main reason for clinical failure of composite fillings is significantly related to the occurrence of marginal leakage, which eventually leads to marginal discoloration, recurrent caries, and post-operative sensitivity.<sup>1</sup> Marginal sealing ability in composite restorations is achieved by the mutual work of the bonding agent as well as the restorative material. A suitable bond can oppose and withstand the contraction stress during polymerization of composite resin and insure retention and marginal adaptation of the

restored teeth.<sup>2,3</sup>

The first bonding protocol that revealed a clinically acceptable result involved the complete removal of the smear layer by an “etch-and rinse” or “total-etch” approach.<sup>1</sup> Although long-term clinical success has been achieved with total-etch systems, but by using this approach, the quality of resin-dentin adhesion may be technique sensitive.<sup>4</sup>

The demand for simplified adhesive procedures led to the development of the alternative “self-etch (SE)” approach by Watanabe et al.<sup>5</sup> This approach eliminates the

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rinsing phase and there is no need to application of the primer in particular conditions of wetness due to the SE adhesives' water content; reduced technique-sensitivity of the material and post-operative sensitivity of patients, and the risk of making errors during application diminished. As a result, their popularity is increasing.<sup>6,7</sup>

Two-step SE adhesive systems were introduced into the market in the late 1990s. They are based on the separate application of SE primer and hydrophobic resin.<sup>6</sup>

Analysis of the clinical trials demonstrate that SE adhesives with good clinical performance and proper bonding to dentin, belong to the group of "mild" SE adhesives (pH  $\geq$  2).<sup>8</sup> Mild SE adhesives demineralize the dentin enough to provide micro-mechanical retention, while leaving hydroxyl apatite within the hybrid layer to enable additional chemical interaction.<sup>9</sup> Mild two-step SE adhesives are like three-step etch and rinse adhesives in terms of low annual failure rates.<sup>1</sup> In this context, CLEARFIL SE BOND (CSB) has been the most tested adhesive and is often used as a control group because of its satisfactory clinical performance.<sup>1</sup>

Further simplification has been achieved by introducing one-step SE adhesives that combine SE primer and hydrophobic resin into one application. These adhesives are more acidic and more hydrophilic than two-step SE adhesives.<sup>1,6</sup> Although studies show that one-step SE adhesives may not perform as well as two-step SE adhesive systems and their bond strength is relatively low,<sup>10,11</sup> but different clinical trials and laboratory studies evaluating SE systems show contradictory findings, and it seems that the bonding effectiveness of these adhesives is material dependent.<sup>4,8</sup>

The latest generation of one-step SE adhesives have performed better as they have superior characteristics in comparison with their earlier versions.<sup>1</sup> In addition, the marginal seal of composite restorations, also

affected by the polymerization shrinkage of composites, and one strategy to control polymerization contraction stress is the use of low-shrinkage composites,<sup>12</sup> so the aim of this research was to compare the microleakage of one-and two-step SE adhesive systems with the low shrinkage methacrylate-based composites of the same company in Class V cavities.

The null hypothesis tested was that there are no differences among microleakage of the adhesive systems tested.

## Methods

36 extracted intact human premolars and molars, free of cracks, decalcifications, or caries were immersed in 0.5% chloramine-T for 1-week and then stored in normal saline solution until use. Class V cavities (mesiodistal width of 3 mm, occlusogingival length of 3 mm and 1 mm in to dentin deep) were prepared, with the gingival margin 1 mm below the cemento-enamel junction, using a tapered fissure diamond bur (Tiz Kavan, Tehran, Iran) with a water cooled high speed hand piece on the buccal and lingual surfaces of the teeth. New burs were used after every five preparations. Materials used in this study with their chemical compositions are listed in table 1.

The prepared teeth were randomly divided into six groups of 6 teeth each (12 cavities). In all groups, the enamel part of the cavity was etched with 37% phosphoric acid (Total Etch, Ivoclar Vivadent) for 20 seconds, rinsed for 15 seconds and slightly air dried to achieve moist surface and then were restored as follows.

Group 1: G-Bond (one-step SE) (GC Corporation, Tokyo, Japan) was applied using a microbrush and leaved undisturbed for 10 seconds. Then, thoroughly air dried for 5 seconds. Another layer of G-Bond was applied and the process repeated again, and then cured for 20 seconds using the LED curing unit (Guilin Woodpecker Medical Instrument Co., China) with an intensity of 900 mw/cm<sup>2</sup>. The light intensity of the curing

unit was periodically checked with a light meter (LED Radiometer Demetron, Kerr, USA). Then each cavity was filled with three layers of Kalore-GC (GC Corporation, Tokyo, Japan) A3.5 shade composite, the first layer on the axial wall, the second layer extended from gingival wall to occlusal, and the third layer extended from occlusal wall to gingival, with each layer not being more than 2 mm thickness and each increment was separately light cured for 40 seconds.

Group 2: Futurabond NR (one-step SE) (Voco Cuxhaven, Germany) was applied for 20 seconds and air dried for 5 seconds. Another layer of bonding was applied and the process was repeated again, and then cured for 20 seconds. Then each cavity was filled with three layers of Grandio (Voco Cuxhaven, Germany) A3.5 shade composite.

Group 3: All Bond SE (one-step SE) (Bisco Inc., Schaumburg, IL, USA) was applied and agitated for 5-10 seconds. Then gently air dried from the distance of 5 cm for 5 seconds (without visible movement of the material). Then with greater pressure, thoroughly air dried for 5 seconds. Another layer of bonding was applied and the process repeated again, then cured for 20 seconds. Then each cavity was filled with three layers of Aelite LS Posterior (Bisco Inc., Schaumburg, IL, USA) A3.5 shade composite.

Group 4: The CSB primer (two-step SE) (Kuraray Medical Inc., Okayama, Japan) was applied for 20 seconds and gently air-dried. The adhesive was then applied and dispersed with a mild air stream, and light cured for 20 seconds. Then each cavity was filled with three layers of Kalore-GC A3.5 shade composite.

Group 5: Following the same bonding procedure applied in group 4, each cavity was filled with three layers of Grandio A3.5 shade composite.

Group 6: Following the same bonding procedure applied in group 4, each cavity was filled with three layers of Aelite LS Posterior A3.5 shade composite. After finishing with

fine-grit finishing diamond burs (Diatech Dental AG, Heerbrugg, Switzerland) and polishing with sequential disks (OptiDisc, Kerr, USA), all specimens were stored in distilled water at 37 °C for 24 hours.

The teeth were thermocycled (Malek Teb, Iran) at 2000 cycles at a temperature 5 °C and 55 °C, a dwell time of 30 seconds in each bath and a transfer time of 10 seconds. The apices of the teeth were then sealed with sticky wax, and whole surfaces of the teeth except a 1 mm wide zone around the margins of each restoration were coated with two layers of nail polish. The teeth were then immersed in a solution of 0.5% basic fuchsin for 24 hours at 37° C. Following immersion, the teeth were washed with distilled water, dried and embedded in self-curing acrylic resin (Acropars, Iran), and sectioned longitudinally in a buccolingual direction through the center of the restoration using a low speed diamond disc (Presi, MECATOME, T201A, France) under constant water irrigation.

The sections were examined under stereomicroscope (Nikon, 30-DS, SMZ800, Tokyo, Japan) at ×10 and ×40 magnification and degree of dye penetration at occlusal and gingival margins was then scored according to the following criteria:<sup>3</sup>

0 = No evidence of dye penetration

1 = Dye penetration into half extension of the occlusal or gingival wall

2 = Dye penetration into more than half extension of the occlusal or gingival wall, without reaching the axial wall

3 = Dye penetration into the axial wall

Data were analyzed by SPSS software (version 18, SPSS Inc., Chicago, IL, USA) using the Kruskal-Wallis test and Mann-Whitney test. The difference between the occlusal and gingival dye penetration scores in each group was analyzed by the Wilcoxon test at a  $P < 0.050$  level of significance.

## Results

Data showing the dye penetration scores for

the occlusal and gingival margins of the restorations are shown in table 2.

When comparing the microleakage between gingival and occlusal margins in each group, there were significantly more dye penetration at the gingival wall than occlusal wall ( $P < 0.050$ ) except Kalore + CSB (Table 3).

There were no significant differences in microleakage among three groups of 1-3 (restored with one-step SE) on both occlusal

( $P = 0.860$ ) and gingival margins ( $P = 0.070$ ). Also, there were no significant differences in microleakage among three groups of 4-6 (restored with two-step SE) on both occlusal ( $P = 0.860$ ) and gingival margins ( $P = 0.090$ ) (Table 4).

There were no significant differences in microleakage among two-step and one-step SE adhesive systems on both the occlusal and gingival margins ( $P > 0.050$ ) (Table 5).

**Table 1.** Materials used in this study and their composition

Material	Composition	Manufacturer	Batch #
CSB (two-step SE)	Primer: MDP, HEMA, hydrophilic dimethacrylate, photoinitiator, water bond: 10-MDP, Bis-GMA, HEMA, hydrophilic dimethacrylate, microfiller, photoinitiator	(Kuraray Medical Inc., Okayama, Japan)	01531A
Futurabond NR (one-step SE)	Liquid A: methacryl phosphorus acid ester and carbonic acid modified methacrylic ester Liquid B: water, ethanol, silicon Part I-Ethanol, sodium benzenesulfinate dehydrate	(Voco Cuxhaven, Germany)	610458
All Bond SE (one-step SE)	Part II-Bis (Glyceryl 1,3 dimethacrylate) phosphate; hydroxyethylmethacrylate, biphenyl dimethacrylate	(Bisco Inc., Schaumburg, USA)	0600010905
G-Bond (one-step SE)	4-MET, pHA-m, DMA, ethanol, water, filler, photo-initiator, stabilizer	(GC Corporation Tokyo, Japan)	0507279
Grandio	Resin matrix: Bis-GMA, TEGDMA, Filler: Fluorosilicate glass, SiO <sub>2</sub>	(Voco Cuxhaven, Germany)	1106467
Aelite LS Posterior	Resin matrix: Ethoxylated Bis-GMA, Filler: Glass filler, amorphous silica	(Bisco Inc., Schaumburg, USA)	0900001308
Kalore-GC	Resin matrix: DX-511 monomer, UDMA, dimethacrylate co-monomers Filler: (30-35 wt% prepolymerized filler, 20-30 wt% fluoroaluminosilicate glass, 20-33 wt% strontium/barium glass, 1-5 wt% silicon dioxide nanofiller)	(GC Corporation Tokyo, Japan)	1004121

10-GDP: 10-methacryloxydecyl dihydrogen phosphate; HEMA: Hydroxyethyl methacrylate; UDMA: Urethane dimethacrylate; TEGDMA: Triethyleneglycol-dimethacrylate; Bis-GMA: Bisphenol A-glycidyl methacrylate; SE: Self-etch; CSB: CLEARFIL SE BOND

**Table 2.** Microleakage score of different adhesive systems and composite restorations

Groups		Occlusal margins				Gingival margins			
		Score 0 [n (%)]	Score 1 [n (%)]	Score 2 [n (%)]	Score 3 [n (%)]	Score 0 [n (%)]	Score 1 [n (%)]	Score 2 [n (%)]	Score 3 [n (%)]
Grandio	CSB	10 (83)	2 (17)	0 (0)	0 (0)	5 (42)	5 (42)	2 (17)	0 (0)
	Futurabond NR	10 (83)	2 (17)	0 (0)	0 (0)	2 (17)	7 (58)	3 (25)	0 (0)
Kalore-GC	CSB	9 (75)	3 (25)	0 (0)	0 (0)	7 (58)	4 (34)	1 (8)	0 (0)
	G-Bond	9 (75)	3 (25)	0 (0)	0 (0)	3 (25)	6 (50)	3 (25)	0 (0)
Aelite LS	CSB	9 (75)	3 (25)	0 (0)	0 (0)	2 (17)	6 (50)	4 (34)	0 (0)
Posterior	All Bond SE	9 (75)	3 (25)	0 (0)	0 (0)	2 (17)	5 (42)	4 (34)	1 (8)

CSB: CLEARFIL SE BOND

**Table 3.** Wilcoxon test results for comparing microleakage scores of occlusal and gingival margins

Filling materials	Adhesive systems	Margin	P
Kalore-GC	CSB	Occlusal	0.350
	G-Bond	Gingival	
Grandio	CSB	Occlusal	0.010
	futurabond NR	Gingival	
Aelite LS Posterior	CSB	Occlusal	0.030
	All Bond SE	Gingival	
	CSB	Occlusal	0.001
	futurabond NR	Gingival	
	CSB	Occlusal	0.003
	All Bond SE	Gingival	
	CSB	Occlusal	0.002
	All Bond SE	Gingival	

CSB: CLEARFIL SE BOND

**Table 4.** Effect of filling material on microleakage scores of occlusal and gingival margins

Margin	Adhesive systems	Filling materials	P
Occlusal	One-step SE	Kalore-GC	0.860
		Grandio	
	Two-step SE	Aelite LS Posterior	
		Kalore-GC	
Gingival	One-step SE	Grandio	0.070
		Aelite LS Posterior	
	Two-step SE	Kalore-GC	
		Grandio	
		Aelite LS Posterior	0.090
		Kalore-GC	
		Grandio	
		Aelite LS Posterior	

SE: Self-etch

**Table 5.** Effect of adhesive systems on microleakage scores of occlusal and gingival margins

Margin	Filling materials	Adhesive systems	P
Occlusal	Kalore-GC	CSB	> 0.999
		G-Bond	
	Grandio	CSB	
		Futurabond NR	
Gingival	Aelite LS Posterior	CSB	> 0.999
		All Bond SE	
	Kalore-GC	CSB	
		G-Bond	
		CSB	0.090
		G-Bond	
		CSB	0.250
		Futurabond NR	
		CSB	0.660
		All Bond SE	

CSB: CLEARFIL SE BOND

## Discussion

Marginal sealing is a significant factor influencing the longevity of composite restorations.<sup>13</sup> In this investigation, in each group, there were significantly more dye penetration at the gingival wall than at the occlusal wall except Kalore + CSB; which is in agreement with the previous study results that demonstrated less microleakage at the occlusal margins than at the gingival margins.<sup>3,7,13</sup> Different clinical and laboratories studies demonstrated that selective etching of enamel margins with 37% phosphoric acid before using SE adhesives provide better marginal integrity and increase bond strength.<sup>8,14,15</sup>

Abdalla and Garcia-Godoy reported better marginal adaptation of Futurabond NR in Class V cavities when an adhesive resin was applied following enamel etching with 37% phosphoric acid.<sup>4</sup> Moreover, the GC Corp for G-Bond recommends the use of phosphoric acid to ensure a good enamel bond.<sup>10</sup> Also, in this study, before using SE adhesives, enamel was etched with 37% phosphoric acid for 15 seconds.

Furthermore, more dye penetration at the gingival margin was expected as the bond strength to enamel is usually higher than the bond strength to dentin and dentin is a less favorable bonding substrate, while enamel makes a uniform bonding substrate that consists of almost 90% inorganic material, dentin is a complex substrate with < 50% inorganic material, and high water content (21%) offering a moist surface that impairs the bonding mechanism. Moreover, the tubular structure of dentin makes it a complex substrate.<sup>16,17</sup>

But in Kalore + CSB group, no significant differences were detected between gingival margins and occlusal margins. Some studies reported that Aelite and Grandio composites have higher elastic modulus and polymerization contraction stress.<sup>18-20</sup> According to Hooke's law, polymerization contraction stress is determined by the elastic

modulus and the volumetric shrinkage of the material. Since the viscoelastic properties, is considered as the most influential factor on stress development, their high elastic modulus and stiffness result in high contraction stress levels.<sup>18,19,21</sup>

On the other hand, Kalore showed low polymerization shrinkage due to the presence of DX511 monomer (a monomer with low number of C = C double bonds and high molecular weight).<sup>22</sup> The combination of the strong bond produced by CSB and low polymerization shrinkage of Kalore decreased gingival microleakage.

CSB has repeatedly been shown to be excellent performer in clinical and laboratory studies.<sup>9</sup> CSB as a two-step SE adhesive with an almost pH of 2 contains the functional monomer 10-methacryloyloxydecyl dihydrogen phosphate, which has the ability to adhere to hydroxyl apatite tightly. In addition, its calcium salt hardly dissolved in water.<sup>9,23,24</sup>

But, in the current study, there were no statistically significant differences in microleakage among CSB and three single-step SE adhesive systems (G-Bond, All Bond SE and Futurabond NR), which is in agreement with the results of some of the previous studies reporting that the microleakage of two-step SE adhesives is not significantly different from one-step SE adhesives.<sup>25-27</sup>

However, Frankenberger and Tay evaluated the microleakage of CSB, Adhe SE, I Bond, and Xeno III, in Class II composite restorations and reported that marginal seal of two-step SE adhesives at gingival margin was significantly better than one-step SE adhesives.<sup>11</sup> Osorio et al. evaluated the microleakage of CSB and Etch and Prime 3.0 in Class V composite restorations and reported that the degree of microleakage at the gingival margins for CSB was significantly lower than that of Etch and Prime 3.0.<sup>28</sup> This finding was not in agreement with the current study results.

Microleakage of composite restorations is affected by bonding type and polymerization shrinkage of composite.<sup>3</sup> G-Bond is a mild (pH = 2) SE adhesive, demineralizes the dentin surface only slightly and produces approximately no exposure of the collagen fibers yielding an extremely thin adhesive interface (300 nm or less), but, in the “nano interaction zone” the 4-methacryloyloxyethyl (MET) monomer may react with residual hydroxyl apatite to form an insoluble calcium compound, that result in a durable and strong interface that is less likely to be enzymatically deteriorated.<sup>7,29</sup>

Moreover, G-Bond contains 5% filler. Studies reported that filled adhesives demonstrated significantly less microleakage and higher bond strength than unfilled adhesives.<sup>30,31</sup>

The favorable clinical performance and strong bond of Futurabond NR is also reported. Futurabond NR contains poly functional adhesive monomers that could react with remaining hydroxyl apatite. Moreover; Futurabond is a nano filled adhesive which forms a thicker adhesive layer and a more flexible interface. Such a flexible intermediate resin layer may absorb stress resulting from polymerization shrinkage of resin composite.<sup>4</sup> Another contributing factor to such desirable performance of Futurabond NR is the presence of Ormocer in the bonding agent. Ormocer was reported to have a calcium complexing function which improved the bond strength to tooth structure.<sup>4</sup>

In the current study, two layers of one-step SE adhesives were used.

Different studies reported better marginal adaptation of one-step SE adhesives when double application of bonding were used due to several mechanisms.<sup>32,33</sup> As the first layer of bonding agent begins to etch dentin, it may be rapidly buffered by hydroxyl apatite, so that additional layers of unpolymerized comonomers may improve the etching ability of adhesives.

On the other hand, by applying more coats of adhesive, the thickness of the adhesive layer increases enough to prevent oxygen inhibition of its entire thickness, resulting in better polymerization.<sup>34</sup> Moreover, composite resins used in this study were methacrylate-based low shrinkage composites. Grandio is a highly filled nano hybrid composite (87.0% by weight and 71.4% by volume) and its low polymerization shrinkage (1.6%) is due to its nano structures.<sup>35</sup>

Kalore is a nano-hybrid resin composite that contains high molecular weight urethane dimethacrylate monomer (DX-511). It is based on a recently developed DuPont technology. The DuPont molecule, DX-511, is compatible with the current composite and bonding systems. This monomer has a long rigid molecular core and flexible arms in the structure. The long rigid core prevents monomer deformation and reduces polymerization shrinkage. The molecular weight of this monomer is 895 which is twice that of urethane dimethacrylate (UDMA) or bisphenol A-glycidyl methacrylate (Bis-GMA). The combination of low number of C = C double bonds and high molecular weight reduces its polymerization shrinkage (1.7).<sup>22,36</sup>

Aelite LS Posterior is a highly filled hybrid resin composite (88.5% by weight and 74.0% by volume) and its low polymerization shrinkage (1.39) is due to its high filler content.<sup>21</sup> In the current study, samples were only subjected to 2000 thermal cycles, thus the application of mechanical load cycling are suggested in future research.

## Conclusion

According to this study, two-step SE adhesive system (CSB) did not provide better marginal seal than the one-step SE adhesive systems.

## Conflict of Interests

Authors have no conflict of interest.

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