Bonding Efficacy of 1-Step Self-Etch Adhesives: Effect of Additional Enamel Etching and Hydrophobic Layer Application

Naveen Manuja, MDS Rajni Nagpal, MDS
Seema Chaudhary, MDS

ABSTRACT

Purpose: The purpose of this study was to analyze the effect of additional enamel etching and hydrophobic layer application on microleakage of 1-step self-etch adhesives.
Methods: Class V cavities were made on the buccal surfaces of 80 extracted human premolars. The teeth were divided into 2 test groups according to the 1-step self-etch adhesive used: Clearfil S^3 Bond and Xeno III. Each test group was subdivided into 4 groups (N=10) according to application mode of the adhesive: (1) Group 1=adhesive applied following manufacturer's directions; (2) Group 2=enamel etching prior to adhesive application; (3) Group 3=same as Group 1, plus application of an additional coat of hydrophobic resin layer; (4) Group 4=same as Group 2, plus application of an additional coat of hydrophobic resin layer. The adhesives were light cured, and the cavities were filled with resin composite. Specimens were thermocycled and analyzed for leakage using Kruskal-Wallis and Mann-Whitney U test at a significance level of P=.05.
Results: Enamel etching significantly reduced leakage in both the adhesives. Hydrophobic resin layer application significantly reduced leakage in Xeno III.
Conclusions: An additional acid etching step and hydrophobic resin layer application can improve the bonding of 1-step self-etched adhesives to enamel and dentin, respectively. (**J Dent Child 2012;79:3-8**)
Received April 15, 2010; Last Revision May 7, 2010; Revision Accepted October 11, 2010.

KEYWORDS: 1-STEP SELF-ETCH ADHESIVES, ADDITIONAL ENAMEL ETCHING, HYDROPHOBIC LAYER, MICROLEAKAGE

Bonding to tooth hard tissues can be accomplished by using 1 of the 2 adhesion strategies: (1) the etch-and-rinse approach or the self-etch approach.[1] Initially, the self-etch adhesives were introduced for use in a 2-step procedure. Recently, however, 1-step self-etch (all-in-one) systems have been introduced that incorporate all the components of an adhesive system (etchant, primer, and bonding resin) into a single solution and combine all 3 bonding steps into a single-application. These 1-step self-etch systems hold a special importance in pediatric dentistry because of a reduction in the number of clinical procedural steps and less time consumption.

Simplification of the clinical procedure that is provided with these adhesives will be especially beneficial while treating uncooperative children. Compared to the etch-and-rinse systems, they do not require a separate rinsing step; therefore, technique sensitivity associated with over-drying or over-wetting of dentin is not a problem with these systems. Moreover, there is less chance of discrepancy between etching depth and resin infiltration, as both procedures take place simultaneously. As smear plugs are not completely removed from the dentinal tubules, postoperative sensitivity is less vs etch-and-rinse systems.

Different researchers have shown that some 1-step self-etch adhesives exhibit relatively low bond strength values to both enamel and dentin vs 2-step self-etch or etch-and-rinse systems.[2-6] These 1-step self-etch adhesives have been subdivided into 3 categories based upon their pH value as strong, moderate (intermediary strong), and mild.[1] The weak acidity of these 1-step self-etch adhesives raises the question of whether the adhesives are able to penetrate the enamel surface and yield durable bonding with the restored tooth. Studies have shown that the resultant

Dr. Manuja is a reader and Dr. Chaudhary is a professor, Department of Pediatric Dentistry, and Dr. Nagpal is reader, Department of Conservative Dentistry, all at Kothiwal Dental College and Research Centre, Moradabad, Uttar Pradesh, India.
Correspond with Dr. Manuja at naveenmanuja@yahoo.com

enamel surface is not etched to the same degree, and there is a significant difference in enamel roughness with the self-etch adhesive systems when compared with traditional phosphoric acid conditioning.[7,8] It has been suggested that the shallower etching pattern on enamel and subsequent reduced micromechanical retention might jeopardize bonding.[9]

The inferior performance of 1-step self-etch adhesives in dentin has been attributed to certain factors. First, these products create very thin coatings, which may be oxygen inhibited, resulting in a poorly polymerized adhesive layer.[10-13] Second, they are prone to phase separation as the solvent evaporates from the solution. Finally, they behave as permeable membranes after polymerization.[14,15] It has also been demonstrated that employing simplified self-etch adhesives in enamel can result in osmotic blistering and, consequently, bond failure when they are not covered by a hydrophobic resin layer.[16] Some authors have indicated that treating 1-step self-etch systems as a primer and covering them with hydrophobic resin coating can be an option for resolving their drawbacks.[12,17,18] The performance of this alternative technique, seems to be dependent on the brand of adhesive tested.[18,19]

Various authors have either evaluated the effect of prior acid-etching on bonding of 1-step self-etch adhesives to enamel or the effect of application of an additional hydrophobic resin layer on bonding of 1-step self-etch adhesives to dentin. No study, however, has yet evaluated the combined effect of such surface treatments on bonding to enamel and dentin simultaneously in prepared cavities. Furthermore, most of the studies have been performed on flat surfaces, which underestimates the effect of C-factor on bonding. Therefore, the objective of this study was to analyze the effect of additional enamel etching and hydrophobic layer application on microleakage of 1-step self-etch adhesives in Class V cavities. The null hypothesis tested was that additional acid etching of enamel and an additional hydrophobic layer application on the prepared tooth surface will not affect the microleakage of 1-step self-etch adhesives at enamel and dentin margins in Class V cavities.

METHODS

Eighty intact caries-free human premolars extracted for orthodontic purpose were selected for the study. Teeth were debrided and disinfected in 1% thymol solution, stored in distilled water, and used within 6 months of extraction. Class V cavity preparations were made on the buccal surfaces of the premolars using an ISO 012 straight fissure diamond bur (Dentsply, Konstanz, Germany) in an air/water-cooled high-speed handpiece. After every 5 preparations, the bur was discarded and replaced with a new bur. The preparations were centered on the cementoenamel junction and standardized with a width of 3 mm mesiodistally and 3 mm occlusogingivally and a depth of 1.5 mm. Enamel margins received a 45° bevel (1 mm), while gingival cavosurface margins were prepared butt joint in dentin. No additional mechanical retention was placed. The teeth were divided into 2 test groups according to the 2 different 1-step self-etch adhesives used: (1) Clearfil S³ Bond (Kuraray Medical, Tokyo, Japan); and (2) Xeno III (Dentsply). Each test group was further subdivided into 4 groups (N=10) according to 4 application modes (Table 1):

1. Group 1: The adhesive was applied according to the manufacturer's directions.
2. Group 2: The enamel margin was etched with 37% phosphoric acid (Etchant, 3M ESPE, St. Paul, Minn) for 15 seconds prior to adhesive application as in Group 1.
3. Group 3: The adhesive was applied, as in Group 1, followed by application of an additional coat of hydrophobic resin layer (Adhesive from Clearfil SE Bond, Kuraray Medical).
4. Group 4: The enamel margin was etched with 37% phosphoric acid for 15 seconds prior to

Table 1. Adhesive Systems: Composition and Application Modes of Different Groups*

Adhesive systems	Composition	Group 1: Manufacturer's directions (MD)	Group 2: Enamel etching (EE)	Group 3: Hydrophobic layer (HL)	Group 4: EE+HL
Xeno III (Dentsply)	Liquid A: HEMA, ethanol, water, aerosol, stabilizers Liquid B: Pyro-EMA, PEM-F, UDMA, camphorquinone, stabilizers, ethyl-4dimethylaminobenzoate (coinitiator)	1. Mix liquids A and B for 5 secs. 2. Apply 1 thick coat of the adhesive under pressure (30 secs). 3. Apply gentle air stream (10 secs at 20 cm). 4. Light-activation (10 secs – 600 mW/cm²).	1. Enamel etching with 37% phosphoric acid for 15 secs. 2. Steps 1-4 from Group 1.	1. Steps 1-4 from Group 1. 2. Application of 1 coat of adhesive from Clearfil SE. 3. Air stream to make the bond film uniform (3 secs at 20 cm). 4. Light-activation (10 secs – 600 mW/cm²).	1. Steps 1-2 from Group 2. 2. Steps 2-4 from Group 3.
Clearfil S³ Bond (Kuraray Medical)	10 MDP, HEMA, bis-GMA, water, ethanol, silanated colloidal silica, camphorquinone, hydrophobic dimethacrylate	1. Wet brush tip with adhesive. 2. Apply; leave on for 20 secs. 3. Dry via high-pressure air for >5 secs. 4. Light-activation (10 secs – 600 mW/cm²).	1. Enamel etching with 37% phosphoric acid for 15 secs. 2. Steps 1-4 from Group 1.	1. Steps 1-4 from Group 1. 2. Application of 1 coat of adhesive from Clearfil SE. 3. Air stream to make the bond film uniform (3 secs at 20 cm). 4. Light-activation (10 secs – 600 mW/cm²).	1. Steps 1-2 from Group 2. 2. Steps 2-4 from Group 3.

** HEMA=2-hydroxyethyl methacrylate; Pyro-EMA=tetromethacrylo-yloxyethyl pyrophosphate; PEM-F=pentamethacryloyloxyethylcyclohexaphosphazene monofluoride; UDMA=urethane dimethacrylate; MDP=10-methacryloxydecyl dihydrogen phosphate; bis-GMA=bisphenol A diglycidyl methacrylate.*

adhesive application (as in Group 2), which was followed by application of an additional coat of hydrophobic resin layer (as in Group 3).

The adhesives were light cured for 10 seconds at a light intensity of 600 mW/cm^2. The cavity preparations were bulk filled with resin composite (Z 250, 3M ESPE) and light cured for 40 seconds at 600 mW/cm^2. The restorations were polished using Enhance system disks (Dentsply). The restored teeth in each group were thermocycled for 500 cycles at 5°C and 55°C. Immersion time was 30 seconds in each bath.

In preparation for the dye penetration test, the specimens were air dried and coated with 2 layers of sticky wax, leaving a 1 mm window around the cavity margins. The samples were immersed in freshly prepared 2% methylene blue dye for 48 hours, rinsed with water, the sticky wax was removed, and the teeth were left to air dry at room temperature for 24 hours. The teeth were sectioned longitudinally in a buccolingual direction by a cut through the center of the restoration. Dye penetration at the tooth restoration interface was assessed by stereomicroscope at a 10X magnification by 2 independent precalibrated examiners who were unaware of the treatment groups. In case of any disagreement, new readings were performed until a consensus was reached. The following scoring system was used[20]:

0 = no evidence of microleakage;
1 = dye penetration up to half the cavity depth;
2 = dye penetration of more than half the cavity depth; and
3 = dye penetration along the axial wall.

STATISTICAL ANALYSIS

The dye penetration results were analyzed via Kruskal-Wallis nonparametric analysis followed by the Mann-Whitney U test to evaluate differences among the experimental groups at a significance level of P=.05.

RESULTS

Microleakage scores for both the adhesives at the enamel and dentin margins are presented in Tables 2 and 3, respectively. Mean scores for dye penetration at the enamel and dentin margins are presented in Tables 4 and 5 and Figures 1 and 2, respectively. At the enamel margins, Clearfil S^3 showed significantly less leakage than Xeno III when applied according to the manufacturer's directions. Additional acid etching of enamel significantly reduced the microleakage for both 1-step self-etch adhesives at the enamel margin. At dentin margins, Xeno III, when applied according to manufacturer's directions, depicted significantly less leakage than Clearfil S^3. With the application of an additional hydrophobic, solvent-free resin layer, a decrease in leakage scores at dentin margins was observed for both the adhesives tested, but the effect was significant only for Xeno III.

DISCUSSION

In tooth-colored restorative materials, early loss of the restoration is no longer a clinical problem. Marginal leakage and consequential marginal discoloration, however, remain the most frequent reasons to replace/repair an adhesive

Table 2. Microleakage Scores at Enamel Margins (N=10)

Restorative groups*	Dye leakage scores			
	0	1	2	3
Clearfil S^3 (MD) Group 1[a]	5	3	2	0
Clearfil S^3 (EE) Group 2[b]	10	0	0	0
Clearfil S^3 (HL) Group 3[a]	3	6	1	0
Clearfil S^3 (EE+HL) Group 4[b]	9	1	0	0
Xeno III (MD) Group 1[c]	0	4	6	0
Xeno III (EE) Group 2[a,d]	6	4	0	0
Xeno (HL) Group 3[c]	0	6	4	0
Xeno III (EE+HL) Group 4[a,b,d]	6	3	1	0

* Same superscript letters indicate no statistically significant difference between the 2 groups at a significant difference level of P=.05.

Table 3. Microleakage Scores at Dentin Margins (N=10)

Restorative groups*	Dye leakage scores			
	0	1	2	3
Clearfil S^3 (MD) Group 1[a]	0	6	4	0
Clearfil S^3 (EE) Group 2[b]	0	7	3	0
Clearfil S^3 (HL) Group 3[a,d]	3	5	2	0
Clearfil S^3 (EE+HL) Group 4[a,d]	2	6	2	0
Xeno III (MD) Group 1[b,d]	5	3	2	0
Xeno III (EE) Group 2[b,a]	4	6	0	0
Xeno (HL) Group 3[c]	10	0	0	0
Xeno III (EE+HL) Group 4[c]	9	1	0	0

* Same superscript letters indicate no statistically significant difference between the 2 groups at a significance level of P=.05.

Table 4. Mean Microleakage Scores of Both the Adhesives at Enamel Margins in all 4 Groups

Group	Clearfil S^3	Xeno III
1 (MD)	0.7	1.6
2 (EE)	0.0	0.4
3 (HL)	0.8	1.4
4 (EE+ HL)	1.0	0.5

Table 5. Mean Microleakage Scores of Both the Adhesives at Dentin Margins in all 4 Groups

Group	Clearfil S^3	Xeno III
1 (MD)	1.4	0.7
2 (EE)	1.3	0.6
3 (HL)	0.9	0.0
4 (EE+ HL)	1.0	0.1

restoration.[1] Additionally, a "retained" restoration is always assessed as "bonded" to the cavity walls, yet, retention and bonding are 2 different concepts. A composite restoration might be retained in a Class V cavity without being totally bonded at the resin dentin interface.[21] Therefore, besides bond strength, testing of marginal sealing effectiveness of adhesives is needed. The design of this study, in which the restorations have a relatively high C-factor and are subjected to thermal stress, is likely to challenge the marginal integrity of restorations.

Self-etch adhesives are a promising development in adhesive dentistry, especially regarding reduction of the necessary application steps. Despite their user-friendliness, however, these 1-step self-etch adhesives are composed of high concentrations of hydrophilic and ionic resin monomers; this fact, associated with the lack of nonsolvented resin coating, turns them into permeable membranes that permit rapid dentinal fluid transudation across the polymerized adhesives.[15,22-26] Besides that, a high amount of water is added to these 1-step adhesive solutions. Water is required to dissociate the weak acidic methacrylate monomers into ionized form for permeation into the smear layer and underlying mineralized dentin.[1] The excess water, however, may prevent the optimal polymerization of the adhesive monomers and lead to phase separation, thereby reducing the mechanical properties of the adhesive layer and the resulting resin-dentin bond strength values.[14,27-31]

When applied according to manufacturer's directions, Xeno III showed significantly less leakage at the dentin margins vs Clearfil S³. At the enamel margins, however, Clearfil S³ performed significantly better than Xeno III when applied according to the manufacturer's directions. This could be due to the fact that Clearfil S³ contains 10-methacryloxydecyl dihydrogen phosphate (**MDP**), which has been reported to have a high chemical bonding potential to hydroxyapatite.[32] Furthermore, the calcium salt of MDP is highly insoluble. According to the adhesion-decalcification concept, the less soluble the calcium salt of an acidic molecule, the more intense and stable the molecular adhesion to a hydroxyapatite-based substrate.[33] A chemical interaction between hydroxyapatite and functional monomers in an adhesive leads to higher bond strengths compared with those that rely on micromechanical retention to the enamel substrate alone.

Self-etch adhesives use acidic monomers to condition tooth structure rather than traditional phosphoric acid; however, they do not produce the same degree of porosity in enamel surfaces as that attained with phosphoric acid etching.[34] Bonding of 1-step self-etch systems to enamel still remains critical and is controversially discussed by various authors.[4,35] In the current investigation, when the adherent enamel surfaces were treated with phosphoric acid, a significant decrease in leakage was observed for both Xeno III and Clearfil S³ (Table 2). Various studies have also indicated the potential benefit of additional enamel etching with phosphoric acid prior to the use of self-etch adhesives.[19,36-41] The most plausible explanation for decreased leakage at the enamel margin after additional phosphoric acid etching of enamel is the increase in enamel porosity, resulting in an increased resin-interlocking and micromechanical retention. In spite of the weak correlation between enamel-etching depth/pattern and bond strength found in the literature, the aggressiveness of the enamel treatment may play an important role.[42-44] Therefore, the null hypothesis that prior acid-etching did not affect the microleakage was rejected. Although the effect was not statistically significant in this study, strengthening the enamel bond through prior etching may direct polymerization stresses to the gingival margin in Class V restorations placed with self-etch adhesives.

By executing the rationale behind the use of 2-step self-etch systems, the performance of 1-step self-etch adhesives may also be improved by treating them as a primer and covering it with a less hydrophilic resin coating such as those employed in conventional 3-step etch-and-rinse adhesives.

In our study, the additional application of a hydrophobic, solvent-free resin layer improved the performance of both 1-step self-etch adhesives in dentin. The effect was significant, however, for Xeno III (Table 3). Our results are supported by the studies of Brackett et al., Reis et al., and Albuquerque et al., who reported increase in resin-dentin bond strength of 1-step self-etch adhesives after hydrophobic resin layer application.[18,45,46] Several mechanisms could account for the better performance of adhesives after hydrophobic layer application.

First, the additional application of a hydrophobic resin layer increased the concentration of hydrophobic monomers, reducing the relative concentration of solvents and hydrophilic monomers within the adhesive interface that explains the decreased leakage at the dentin margin.[46] Second, the application of a hydrophobic coat also seems to limit the diffusion of water through the hybrid layer to the interface between the adhesive and resin composite.[15] Third, the additional layer of hydrophobic adhesive increased the thickness of the adhesive layer that is known to reduce polymerization stresses.[47] On the other hand, a slight increase in leakage was observed at the enamel margin after hydrophobic layer application, although the effect was not statistically significant.

CONCLUSIONS

1. Additional enamel etching significantly reduced the microleakage of both 1-step self-etch adhesives at the enamel margins in Class V cavities.
2. The effect of hydrophobic resin layer application on microleakage at dentin margins varied with the brand of 1-step self-etch adhesive tested, but had no detrimental effect on dentin bonding regardless of the adhesive system used.

3. An additional acid etching step could be considered with the use of 1-step self-etch adhesives for restorations whose retention primarily depends on a strong bond to the enamel surface, such as large Class IV restorations or restorations with a high C factor. Application of an additional hydrophobic resin layer may also be beneficial for 1-step self-etch adhesives where maximum dentin adhesion is desirable.

REFERENCES

1. Van Meerbeek B, De Munck J, Yoshida Y, et al. Buonocore Memorial Lecture. Adhesion to enamel and dentin: Current status and future challenges. Oper Dent 2003;28:215-35.
2. Inoue S, Vargas MA, Abe Y, et al. Microtensile bond strength of 11 contemporary adhesives to enamel. Am J Dent 2003;16:329-34.
3. Armstrong SR, Vargas MA, Fang Q, Laffoon JE. Microtensile bond strength of a total-etch 3-step, total-etch 2-step, self-etch 2-step, and a self-etch 1-step dentin bonding systems through 15-month water storage. J Adhes Dent 2003;5:47-56.
4. Brackett WW, Ito S, Nishitani Y, Haisch LD, Pashley DH. The microtensile bond strength of self-etching adhesives to ground enamel. Oper Dent 2006;31: 332-7.
5. De Munck J, Van Meerbeek B, Satoshi I, et al. Microtensile bond strengths of 1- and 2-step self-etch adhesives to bur-cut enamel and dentin. Am J Dent 2003;16:414-20.
6. Perdigao J, Gomes G, Duarte S Jr, Lopes MM. Enamel bond strengths of pairs of adhesives from the same manufacturer. Oper Dent 2005;30:492-9.
7. Breschi l, Gobbi P, Falconi M, Mazzotti G, Prati C, Perdigao J. Ultra-morphology of self-etching adhesives on ground enamel: A high resolution SEM study. Am J Dent 2003;16:57A-62A.
8. Barkmeier WW, Erickson RL, Kimmes NS, Latta MA, Wilwerding TM. Effect of enamel etching time on roughness and bond strength. Oper Dent 2009; 34:217-22.
9. Miyazaki M, Sato M, Onose H. Durability of enamel bond strength of simplified bonding systems. Oper Dent 2000;25:75-80.
10. Frankenberger R, Perdigao J, Rosa BT, Lopes M. No-bottle vs multi-bottle dentin adhesives: A microtensile bond strength and morphological study. Dent Mater 2001;17:373-80.
11. Pashley EL, Agee KA, Pashley DH, Tay FR. Effects of one versus two applications of an unfilled, all-in-one adhesives on dentin bonding. J Dent 2002;30: 83-90.
12. Ito S, Tay FR, Hashimoto M, et al. Effects of multiple coating of two all-in-one adhesives on dentin bonding. J Adhes Dent 2005;7:133-41.
13. Rueggeberg FA, Margeson DH. The effect of oxygen inhibition on an unfilled/filled composite system. J Dent Res 1990;69:1652-8.
14. Van Landuyt KL, De Munck J, Snauwaert J, et al. Monomer-solvent phase separation in 1-step self-etch adhesives. J Dent Res 2005;84:183-8.
15. Tay FR, Pashley DH, Suh BI, Carvalho RM, Itthagarum. Single-step adhesives are permeable membranes. J Dent 2002;30:371-82.
16. Tay FR, Lai CN, Chersoni S, et al. Osmotic blistering in enamel bonded with 1-step self-etch adhesives. J Dent Res 2004;83:290-5.
17. King NM, Tay FR, Pashley DH, et al. Conversion of 1-step to two-step self-etch adhesives for improved efficacy and extended application. Am J Dent 2005; 18:126-34.
18. Brackett WW, Ito S, Tay FR, Haisch LD, Pashley DH. Microtensile dentin bond strength of self-etching resins: Effect of a hydrophobic layer. Oper Dent 2005;30:733-8.
19. Van Landuyt KL, Peumans M, De Munck J, Lambrechts P, Van Meerbeek B. Extention of 1-step self-etch adhesive into a multi–step adhesive. Dent Mater 2006;22:533-4.
20. Munro GA, Hilton TJ, Hermesch CB. In vitro microleakage of etched and rebonded Class V composite resin restorations. Oper Dent 1996;21:203-8.
21. Perdigao J. Dentin bonding as a function of dentin structure. Dent Clin North Am 2002;46:277-301.
22. Van Landuyt KL, Snauwaert J, De Munck J, et al. Systematic review of the chemical composition of contemporary dental adhesives. Biomater 2007;28: 3757-85.
23. Cheong C, King NM, Pashley DH, Ferrari M, Toledano M, Tay FR. Incompatibility of self-etch adhesives with chemical/ dual cure composites: Two-step vs one-step systems. Oper Dent 2003;28: 747-55.
24. Tay FR, Pashley DH, Garcia-Godoy F, Yiu CK. Single-step, self-etch adhesives behave as permeable membranes after polymerization. Part II. Silver tracer penetration evidence. Am J Dent 2004;17: 315-22.
25. Tay FR, Pashley DH, Suh B, Carvalho R, Miller M. Single-step, self-etch adhesives behave as permeable membranes after polymerization. Part I. Bond strength and morphologic evidence. Am J Dent 2004;17:271-8.
26. Itthagarun A, Tay FR, Pashley DH, Wefel JS, Garcia-Godoy F, Wei Sh. Single-step, self-etch adhesives behave as permeable membranes after polymerization. Part III. Evidence from fluid conductance and artificial caries inhibition. Am J Dent 2004;17: 394-400.
27. Jacobson T, Soderholm KJ. Some effects of water on dentin bonding. Dent Mater 1995;11:132-6.

28. Cadenaro M, Antoniolli F, Sauro S, et al. Degree of conversion and permeability of dental adhesives. Eur J Oral Sci 2005;113:525-30.
29. Spencer P, Wang Y. Adhesive phase separation at the dentin interface under wet bonding conditions. J Biomed Mater Res 2002;62:447-56.
30. Takahashi A, Sato Y, Uno S, Pereira PNR, Sano H. Effects of mechanical properties of adhesives on bond strength to dentin. Dent Mater 2002;18:263-8.
31. Reis A, Grandi V, Carlotto L, et al. Effect of smear layer thickness and acidity of self-etching solution on early and long-term bond strength to dentin. J Dent 2005;33:549-59.
32. Yoshioka M, Yoshida Y, Inoue S, et al. Adhesion/decalcification mechanisms of acid interactions with human hard tissues. J Biomed Mater Res 2002; 59:56-62.
33. Yoshida Y, Nagakane K, Fukuda R, et al. Comparative study on adhesive performance of functional monomers. J Dent Res 2004;83:454-8.
34. Hannig M, Bock H, Bott B, Hoth-Hannig W. Intercrystallite nanoretention of self-etching adhesives at enamel imaged by transmission electron microscopy. Eur J Oral Sci 2002;110:464-70.
35. Watanabe T, Tsubota K, Takamizawa T, et al. Effect of prior acid etching on bonding durability of single-step adhesives. Oper Dent 2008;33:426-33.
36. Brackett MG, Brackett WW, Haisch LD. Microleakage of Class V resin composites placed using self-etching resins: Effect of prior enamel etching. Quintessence Int 2006;37:109-13.
37. Miguez PA, Castro PS, Nunes MF, Walter R, Pereira PN. Effect of acid-etching on the enamel bond of two self-etching systems. J Adhes Dent 2003;5: 107-12.
38. Peumans M, De Munck J, Van Landuyt KL, Lambrechts P, Van Meerbeek B. Three-year clinical effectiveness of a two-step self-etch adhesive in cervical lesions. Eur J Oral Sci 2005;113:512-8.
39. Van Landuyt KL, Kanumilli P, De Munck J, Peumans M, Lambrechts P, Van Meerbeek B. Bond strength of a mild self-etch adhesive with and without prior acid-etching. J Dent 2006;34:77-85.
40. Van Meerbeek B, Kanumilli P, De Munck J, Van Landuyt KL, Lambrechts P, Peumans M. A randomized controlled study evaluating the effectiveness of a two-step self-etch adhesive with and without selective phosphoric-acid etching of enamel. Dent Mater 2005;21:375-83.
41. Luhrs AK, Guhr S, Schilke R, Borchers L, Geurtsen W, Gunay H. Shear bond strength of self-etch adhesives to enamel with additional phosphoric acid etching. Oper Dent 2008;33:155-62.
42. Pashley DH, Tay FR. Aggressiveness of contemporary self-etching adhesives. Part II: Etching effects on unground enamel. Dent Mater 2001;17:430-44.
43. Perdigao J, Lopes L, Lambrechts P, et al. Effects of a self-etching primer on enamel shear bond strengths and SEM morphology. Am J Dent 1997;10:141-6.
44. Hashimoto M, Ohno H, Yoshida E. Resin-enamel bonds made with self-etching primers on ground enamel. Eur J Oral Sci 2003;111:447-53.
45. Reis A, Albuquerque M, Pegoraro M, et al. Can the durability of 1-step self-etch adhesives be improved by double application or by an extra layer of hydrophobic resin? J Dent 2008;33:309-15.
46. Albuquerque M, Pegoraro M, Mattei G, Reis A, Loguercio AD. Effect of double-application or the application of a hydrophobic layer for improved efficacy of 1-step self-etch systems in enamel and dentin. Oper Dent 2008;33:564-70.
47. Chic KK, Condon JR, Ferracane JL. The effects of adhesive thickness on polymerization contraction stress of composite. J Dent Res 2000;79:812-7.