International Journal of Adhesion & Adhesives 34 (2012) 6–10

Contents lists available at SciVerse ScienceDirect

International Journal of Adhesion & Adhesives

journal homepage: www.elsevier.com/locate/ijadhadh

Effect of different adhesive systems on microleakage in class II composite resin restorations

S.J. Mauro [a,*], V.C.A. Durão [a], A.L.F. Briso [a], M.L.M.M. Sundefeld [b], V. Rahal [a]

[a] Department of Restorative Dentistry, Araçatuba Dental School, São Paulo State University – UNESP, Araçatuba, SP, Brazil
[b] Department of Pediatric and Community Dentistry, Araçatuba Dental School, São Paulo State University – UNESP, Araçatuba, São Paulo, SP, Brazil

ARTICLE INFO

Article history:
Accepted 15 December 2011
Available online 17 January 2012

Keywords:
Composites
Biological adhesion
Microscopy

ABSTRACT

The success of a restoration depends on the interaction between the adhesive system and the dental substrate. The aim of this study was to evaluate the occurrence of microleakage in class II resin restorations using different adhesive systems light cured in enamel and dentin: conventional and self-etching acrylic. For that purpose 40 human molars were sectioned in buccal–lingual direction, making up 80 samples (40 mesial and 40 distal halves). Each sample had cavities class II prepared and the occlusal margins were kept in enamel while the cervical margins were kept in dentin. The specimens were divided into four groups ($n=20$) according to the adhesive system used. After restorative and thermocycling procedures the degree of dye leakage was qualified using a stereomicroscopic loupe (45X). Data were analyzed by ANOVA and Kruskal–Wallis tests ($\alpha=0.05$). Results obtained for occlusal (enamel) and cervical (dentin) margins (μm) were, respectively: GI-11.45, 20.7; GII-15.4, 19.4; GIII-23.3, 24.05; GIV-31.85, 17.85. In enamel, the self-etching adhesive systems presented the less control of marginal leakage in the occlusal and cervical margins. Although for the dentin there was no difference among the adhesive systems used. It was concluded that no adhesive system hermetically sealed the restorations with cavity margins in enamel or dentin and self-etching adhesives did not improve microleakage controlling when compared to conventional systems.

1. Introduction

Dental professionals aim to perform procedures for esthetic restorations with longevity and no postoperative sensitivity [1–3]. Marginal sealing in composite resin restorations is important for the longevity and can be affected by dimensional alterations of the restorative material, polymerization shrinkage, thermal and hygroscopic expansion, C-factor, substrates available for adhesion, and sealing ability of the adhesive system [4–8].

Microleakage test with previous thermocycling is considered a low-cost and effective method to evaluate the marginal sealing provided by adhesive systems, considering the different linear thermal expansion coefficients of restorative materials and dental structures [9]. Class II cavities are excellent for this evaluation because they present cavity margins in enamel and dentin substrate.

Adhesion in enamel has been considered safe and is rarely questioned [10–13]. However, the same does not apply for dentin, because the humidity, collagen, cavity depth, amplitude and direction of dentin tubules may influence the marginal sealing [5,8,11,14–18].

The most used bonding technique establishes simultaneous enamel and dentin etching with phosphoric acid. The selective demineralization of hydroxyapatite in enamel favors the formation of microporosities for penetration of the hydrophobic and/or hydrophilic resin components of the adhesive system, promoting tags formation for retention of composites and control of marginal leakage [11–13,16]. In dentin, etching with phosphoric acid makes this tissue very dynamic and unstable. Removal of the smear layer, smear plug and underlying hydroxyapatite exposes an extremely organic structure with a mesh of fibrils that are separated and sustained by the humidity of this tissue and the water for acid rinsing. In ideal conditions, the space between collagen fibers is filled by the hydrophilic component of the adhesive system, generating tags into the dentin tubules and the hybrid layer in the peri and inter-tubular dentin. This structure has been indicated as responsible for retention of the restorative material and control of microleakage in this substrate [19]. However, perfect interaction is not achieved with the adhesive system by the total etching technique. Some reports state that the entire thickness of demineralized dentin with exposed collagen mesh is not completely filled by the resin components. This allows degradation of the unprotected collagen, jeopardizing the long-term marginal sealing [4,20–22].

It is important to highlight that this phenomenon encouraged researchers and manufacturers to improve the self-etching

* Corresponding author at: Faculdade de Odontologia de Araçatuba – Unesp, Departamento de Odontologia Restauradora, Rua José Bonifácio 1193, Vila Mendonça, 16015-050, Araçatuba, São Paulo, Brazil. Tel.: +55 18 3636 3349; fax: +55 18 36363343.
E-mail address: sjmauro@foa.unesp.br (S.J. Mauro).

0143-7496/$ - see front matter © 2012 Elsevier Ltd. All rights reserved.
doi:10.1016/j.ijadhadh.2012.01.004

systems, aiming at easy application and the involvement of exposed collagen with the acidic monomers.

Adhesion in dental substrates is traditionally obtained by acid etching with phosphoric acid and posterior application of a conventional adhesive, which contains hydrophobic and hydrophilic monomers found separately or all-in-one bottle [10–15]. Thus, the resin monomer penetrates into the gaps created by acid etching, generating excellent interaction between the adhesive system and the substrate [16]. However, in dentin, demineralization by the phosphoric acid may create areas of exposed collagen that is not impregnated with the hydrophobic adhesive monomer, which compromises bonding stability [19–21,23–25].

To solve this problem and facilitate the bonding technique, self-etching adhesive systems have been developed which are capable of etching enamel and dentin, without the need of phosphoric acid application. In addition, this adhesive system does not allow formation of demineralized areas not impregnated by the resin components [20,21,25,26]. When these systems are applied on enamel, there is significant damage in tags formation and bonding [9,12,16,27–30,]. However, in dentin, the behavior is similar to the conventional adhesives [9,12,27,31,32].

Otherwise, self-etching adhesives consist of self-etching adhesive monomers, cross-linking monomers and additional monofunctional co-monomers. Thus, strongly acidic adhesives monomers containing dihydogenphosphate phosphonic acid or carboxylic acid groups are the most efficient adhesives available. These monomers promote the formation of a strong and durable adhesive layer by free-radical copolymerization with the other monomeric components of the adhesive. At the moment, serious problems of water-based, highly acidic self-etching enamel-dentin adhesives, in particular single-bottle conventional adhesives arise from the hydrolytic instability of the methacrylate monomers used [33].

adhesive systems and immediate sealing of the tooth/restoration interface, the stress generated by polymerization of the restorative resin materials and the dimensional alterations frequently occurring in the mouth may compromise the marginal sealing and longevity of composite resin restorations [17]. Therefore, laboratory analyses are required to test the effectiveness of adhesives. Thus, thermal and/or mechanical challenges are carried out to test the interface at its most vulnerable point, namely its longitudinal stability [5,9,11,12,27,31]. So, thermal cycling is for the main purpose of failure acceleration.

Considering that enamel and dentin are involved in bonding, studies should compare the behavior of different adhesive materials in these substrates to obtain enough information, so that the clinicians may perform restorative procedures safely. Thus, the null hypotheses tested were that: 1—self-etching adhesive systems do not avoid the occurrence of microleakage in enamel and dentin margins, and 2—self-etching adhesive systems are not better than conventional systems in controlling marginal microleakage in enamel and dentin.

This paper is intended to qualitatively evaluate the control of microleakage in class II cavities, with occlusal margin in enamel and cervical margin in dentin, restored with composite resin and different acrylic and light curing adhesive systems (two self-etching adhesive systems, one one-bottle conventional adhesive system and one two-bottle conventional adhesive system). Dye penetration after thermal cycling will be used to evaluate leakage.

2. Materials and methods

Forty intact recently extracted human molars were stored in 0.2% formalin at neutral pH. The project was approved by the Institutional Review Board, FOA, Proc. 600/2004.

2.1. Specimen preparation

Forty human molars were sectioned in buccal–lingual direction, making up 80 samples (40 mesial and 40 distal halves). Afterwards, standard class II cavities were prepared using a # 245 bur (KG Sorensen Ltd.—Barueri, SP, Brazil) mounted in high-speed handpiece under water/air spray. The cavities presented 3 mm in buccal–lingual direction, 1.5 mm in mesial–distal direction and 6 mm in occlusal–gingival direction. The cervical margin was 1 mm above the dentin–enamel junction.

The eighty samples were randomly divided into 4 experimental groups ($n=20$) that received the adhesives presented in Table 1, followed by the composite resin Filtek Z-250 (3 M ESPE, St. Paul, Mn, USA).

2.2. Bonding procedures

The prepared cavities were submitted to prophylaxis with pumice and water, rinsed and dried. Then, the adhesive system of each group was applied according to the manufacturer's instructions (Table 1). The specimens were positioned in an acrylic resin model to allow positioning of wooden wedges and circumferential flexible pre-contoured Hawe Neos matrices (KerrHawe SA, Bioggio, Switzerland) to simulate the clinical conditions for restoration with composite resin.

The cavities were restored with composite resin hade, using three horizontal layers. Each layer was polymerized for 40 s with quartz–tungsten–halogen light source with an intensity of 500 mW/cm^2 (Ultralux-Dabi Atlante, Ribeirão Preto, São Paulo, Brazil).

After the restorative procedures, specimens were stored in a humidified chamber at 37 °C during 7 days followed by finishing and polishing using diamond burs (# 1190F KG Sorensen Ltd., Barueri, SP, Brazil), silicone tips (Enhance-Dentsply, Calk, United States of America) and aluminum oxide disks (Pop-On—3M ESPE, St. Paul, MN, USA).

After polishing, the specimens were stored and thermocycled for 1,000 cycles using a thermocycling machine (Ética Equipamentos Científicos S/A—São Paulo, São Paulo, Brazil). Each cycle consisted of one minute at a temperature of 5 ± 2 °C, and one further minute at a temperature of 55 ± 2 °C. After thermocycling, the specimens received 2 layers of nail polish, except for the restoration and the 1 mm surface around it [11,27].

Thereafter the specimens were immersed in 2% basic fuchsine solution at 37 °C for 24 h [9,11,27,34]. After this period, the specimens were rinsed in running water during 2 min and dried for 24 h at room temperature.

2.3. Microleakage analysis

The specimens were sectioned in mesio–distal direction at the center of the restorations to obtain two similar dental fragments. Twenty analysis were performed in each group, since the fragment that exhibited greater dye leakage was evaluated and the other was discarded ($n=20$).

The degree of dye leakage was analyzed with the aid of a stereomicroscope Stemi SV 11 (Carl Zeiss Company—DSM-940A, Oberkochen, Baden-Württemberg, Germany) at 45 × magnification according to scores previously established for enamel (Table 2) and dentin (Table 3) margins.

Data were statistically analyzed by the non-parametric Kruskal–Wallis test and multiple comparisons test at a significance level of 5%.

Table 1
Experimental groups according to the adhesive system, composition and application method recommended by the manufacturer.

Group	Product manufacturer	Composition	Application method
GI	Adper Scotchbond Multi-Purpose Plus (**3 M** ESPE, St. Paul, Mn, USA)	*ScotchEtch Conditioner*—35% H_3PO_4 silica thickened.	Etch for 20 seg. Rinse with water spray for 45 s.
		Primer—2-hydroxyethil methacrylate, polyalkenoic acid copolymer, water.	Apply Primer for 20 s, Mild air stream
		Adhesive—Bis-phenol A diglycidylmethacrylate, 2-hydroxyethil methacrylate	Apply Adhesive. Gentle air stream. Light cure for 20 s
GII	Adper Single Bond (**3 M** ESPE, St. Paul, Mn, USA)	*ScotchEtch Conditioner*—35% H_3PO_4 silica thickened	Etch for 20 seg. Rinse with water spray for 45 s.
		Adhesive—Bis-phenol A diglycidylmethacrylate, 2-hydroxyethil methacrylate, diurethane dimethacrylate, polyalkenoic acid copolymer, camphorquinone, water, ethanol and glycerol dimethacrylate 1.3, 10% by weight of silica nanoparticles	Apply Adhesive. Gentle air stream. Light cure for 20 s
GIII	Clearfil SE Bond (**Kuraray** Medical Inc., Sakazu, Kurashiki, Okayama, Japan)	*Primer*—10-methacryloxydecyl dihydrogen phosphate; 2-hydroxyethyl methacrylate; hydrophilic dimethacrylate; decamphorquinone; N, N-diethanol-p-toluidine; water	Apply Primer for 20 s Mild air stream
		Bond—10-methacryloxydecyl dihydrogen phosphate; Bis-phenol A diglycidylmethacrylate; 2-hydroxyethil methacrylate; hydrophobic dimethacrylate; dicamphorquinone; N, N-diethanol-p-toluidine; silanated colloidal silica	Apply Bond. Gentle air stream. Light cure for 10 s
GIV	Tyrian-SPE+One Step Plus (**BISCO**, INC., 1100 W. Irving Park Rd.—Schaumburg, IL USA)	*Tyrian SPE* Primer A: Thymol blue, ethanol and water. Primer B: 2-Acrylamino-2-methy propanesulfonic acid, Bis{2-[metacryloyloxy]ethyl}phosphate, 2,4,6-trymethylbenzoyldiphenylphosphine oxide and ethanol.	Activate Tyrian SPE (Mixture of Primer A and Primer B) and apply two coats, with slight agitation for10 s: air-dry for 10 s
		One Step—Adhesive Bisphenol-A diglycidyl ether dimethacrylate and Bisphenyl dimethacrylate (15%–40%), 2-hydroxyethil methacrylate (15%–40%), dental glass (1%–10%) and acetone (40%–70%)	Apply two consecutive coats of adhesive for 10 s each; air-dry for 10 s; light-activate for 10 s

Table 2
Scores for microleakage evaluation in enamel.

Score	Degree	Marginal leakage
0	*Absent*	*No dye leakage at the enamel/restoration interface*
1	*Moderate*	*Dye leakage at the enamel/restoration interface until the dentinoenamel junction*
2	*Severe*	*Dye leakage at the enamel/restoration interface beyond the dentinoenamel junction*

Table 3
Scores for microleakage evaluation in dentin/cement.

Score	Degree	Marginal leakage
0	*Absent*	*No dye leakage at the dentin/restoration interface*
1	*Moderate*	*Dye leakage at the dentin/restoration interface including the cervical wall*
2	*Severe*	*Dye leakage at the dentin/restoration interface including the cervical and axial walls*

Table 4
Kruskal–Wallis test applied to the means of scores obtained in the occlusal margins (enamel—$p < 0.05$).

Group	Median	Sum of ranks	Mean of ranks*
G I (conventional two-bottle)	0.000	114.50	5.73 **A**
G II (conventional one-bottle)	0.000	154.00	7,70 **A**
G III (self-etching)	0.000	233.00	11.65 **B**
G IV (self-etching)	0.000	318.50	15.93C

* Same letters indicate statistical similarity.

Table 5
Result of the Kruskal–Wallis test applied to the means of scores obtained in the cervical margins (dentin—$p < 0.05$).

Group	Median	Sum of ranks	Mean of ranks*
G I (conventional two-bottle)	1.000	207.00	10.35**A**
G II (conventional one-bottle)	1.000	194.00	9.70**A**
G III (self-etching)	1.000	240.50	12.025**A**
G IV (self-etching)	0.5000	178.50	8.925**A**

* Same letters indicate statistical similarity.

3. Results

None of the adhesive systems tested completely eliminated microleakage in both substrates.

In occlusal margins (enamel), the Kruskal–Wallis and Dunn's multiple comparison tests demonstrated better behavior of the two-bottle (GI) and single-bottle (GII) adhesive systems in comparison to the self-etching adhesive systems (GIII and GIV). Thus, different letters indicate statistical significant values and same letters indicate statistical similar values (Tables 4 and 5). Tests also demonstrated that GIII and GIV exhibited the worst performance in microleakage control (Table 4).

In cervical margins (dentin), the Kruskal–Wallis test demonstrated statistical similarity among the experimental groups (Table 5).

4. Discussion

The results of the present study accepted both null hypotheses since it was demonstrated that the self-etching adhesive systems did not avoid microleakage in the occlusal (enamel) and cervical (dentin) margins. Additionally, these systems were not better than single- and two-bottle systems with complete etching for microleakage in enamel and dentin. These results are in accordance with those of Opdan et al. [2], Arisu et al. [12], Brandt et al. [27] and Osório et al. [11].

Probably, the poor performance of self-etching adhesives in enamel is related to the little micromechanical interaction of their resin components with enamel. Microscopic observations exhibit scarce and short tags that are probably insufficient to control microleakage at the adhesive interface [11,16,35]. The alterations in enamel generated by the acidic primers of self-etching systems are worse than those obtained with phosphoric acid etching (GI and GII). Similar observations were reported by Peumans et al. [28] and Van Meerbeek et al. [29], who assessed the clinical performance of a self-etching adhesive system and stated that it can be improved if the enamel is previously etched with phosphoric acid.

Some studies report that removal of the superficial enamel layer with abrasive disks or diamond burs improves the performance of self-etching adhesive systems [36,37]. Such findings were not confirmed in the present study, since the self-etching systems presented the worst performance for microleakage control. The presence of the byproducts calcium, phosphate and water at the adhesive interface, generated by acidic dissolution of hydroxyapatite by the self-etching primer, may have impaired bonding between the composite and the enamel and its maintenance during thermocycling. Although Shinohara et al. [38] demonstrated that the enamel etching with a self-etching adhesive system is similar to that obtained with phosphoric acid, the results of this study do not confirm these observations and are in accordance with the results of Hanning et al. [39]. The present results also reinforce the statements of Brackett et al. [30] that suggested caution for use of this material.

Considering the proximity between enamel and dentin, two technical difficulties are presented during the adhesive procedure. The first one is the humidity in enamel near dentin, since humid dentin is necessary for the application of adhesive systems after phosphoric acid etching. The second difficulty is the contamination of enamel etched with phosphoric acid when the primer is applied on dentin. Even considering such difficulties, the two and single-bottle adhesive systems exhibited better performance for microleakage control when compared to self-etching systems. The presence of a functional copolymer of methacrylate of polyacrylic and polyalkenoic acids in the conventional adhesive systems (GI and GII) increases their resistance to the harmful effect of humidity in an environment with high relative humidity [40] and satisfactory amount of resin component in these adhesives. Thus, this copolymer that completely fills the micropores created by acid etching may eliminate the two technical difficulties previously presented, which ensures good performance for these materials even in humid enamel.

No adhesive system avoided microleakage in the cavity margins in dentin and statistically similar outcomes were observed in all groups, which is in agreement with other authors [9,11,12,27,31,41].

The self-etching adhesive systems were developed to improve the performance of conventional systems, presenting a technique with easier application and complete involvement of the demineralized area. However, the results of the present study demonstrated that no adhesive system eliminated the occurrence of microleakage.

The low pH of self-etching adhesive systems (1.4 for GIII and 0.5 for GIV) allows dentin demineralization and its involvement by the resin components in a single step, allowing formation of the hybrid layer [32].

However, the hybrid layer formed in these conditions may be insufficient to create a stable and strong bonding to support thermal and mechanical stresses [42]. This statement was observed in the present study considering that demineralization by self-etching adhesives may destabilize the collagen and decrease the adhesive resistance. In addition, the hybrid layer may not present enough resin material to establish a strong bonding interface between the dentin and composite resin, increasing the probability of dye leakage [19,24,25].

Furthermore, the poor performance of self-etching adhesive systems in dentin can result from the acids in these adhesives, which promote dissolution of the smear layer, smear plug and dentin, avoiding complete opening of the dentin tubules [11,24] and allowing little monomer penetration into dentin. Additionally, this interaction without rinsing of the etching agent generates a hybrid layer impregnated with calcium and phosphate ions associated to the unstructured components of the smear layer and smear plug, which results in a hybrid layer more susceptible to degradation during storage and thermocycling [42].

The microleakage observed in the cervical walls using adhesive systems with total etching may have resulted from inability of the primers to involve the collagen fibers exposed after acid etching that did not penetrate into the demineralized area, which makes this interface more susceptible to degradation. The residual water may also have influenced polymerization of resin components of the adhesive systems [43], damaging the involvement of collagen fibers by the resin components of the adhesive system and generating gaps at the dentin/composite resin interface [20–22,26,43].

The literature shows that the 2-hydroxyethil methacrylate present in the conventional systems collaborates with expansion of the collagen fibers, decreasing their collapse [44]. However, this monomer decreases the partial pressure of steam, impairing the removal of residual water. In this condition, the hydrophobic monomers do not penetrate into the areas with residual water [45], leading to the occurrence of leakage. Even with their development, porosities are still observed in the hybrid layer with no resin component in the hybrid layer base [45]. Additionally, damage may have occurred in polymerization of the adhesive or low molecular weight oligomers may have retained water in the hybrid layer at the adhesive/hybrid layer interface [4,5,11,19,23].

Based on these results, both null hypotheses should be accepted, since they do not confirm the statements of Osório et al. [11], Hashimoto et al. [46] and Haning et al. [41], who suggested the use of self-etching adhesive systems. The similar behavior in dentin between the total etching and self-etching adhesive systems associated to the supremacy of those systems with total etching in enamel indicates that the adhesive systems with total etching should be considered as the first alternative, mainly for extensive occlusal restorations [30].

5. Conclusions

Considering the conditions and limitations of the present study, it was concluded that:

1. No adhesive system hermetically sealed the restorations with cavity margins in enamel or dentin.
2. Self-etching adhesives did not improve microleakage controlling when compared to conventional systems.
3. Systems by self-etching systems should be carefully indicated.

Acknowledgments

Supported by São Paulo State Research Foundation – FAPESP – Process: 04/02656-1.

References

[1] Briso ALF, Mestrener SR, Delício G, et al. Clinical assessment of postoperative sensitivity in posterior composite restorations. Oper Dent 2007;32:421–6.
[2] Opdan NJM, Roeters FJM, Feilzer AJ, et al. Marginal integrity and postoperative sensitivy in Class 2 resin composite restorations in vivo. J Dent 1998;26:555–62.
[3] Wilson NH, Dunne SM, Gainsford ID. Current materials and techniques for direct restorations in posterior teeth. Part 2: Resin composite systems. Int Dent J 1997;47:185–93.

[4] Toledano M, Osorio R, Albadejo A, et al. Differential effect of in vitro degradation on resin–dentin bonds produced by self-etch versus total-etch adhesives. J Biomed Mater Res 2006;77A:128–35.
[5] Vaidyanathan TK, Vaidyanathan J. Recent advances in the theory and mechanism of adhesive resin bonding to dentin: a critical review. J Biomed Mater Res B Appl Biomater 2009;88:558–78.
[6] Hashimoto M, Fujita S, Endo K, et al. In vitro degradation of resin–dentin bonds with one-bottle self-etching adhesives. Eur J Oral Sci 2009;117:611–7.
[7] Toledano M, Osorio R, Osorio E, et al. Durability of resin–dentin bonds: effects of direct/indirect exposure and storage media. Dent Mater 2007;23: 885–92.
[8] Besnault C, Attal JP. Influence of a simulated oral environment on microleakage of two adhesive systems in Class II composite restorations. J Dent 2002;30:1–6.
[9] Abo T, Uno S, Sano H. Comparison of bonding efficacy of an all-in-one adhesive with a self-etching primer system. Eur J Oral Sci 2004;112:286–92.
[10] Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surface. J Dent Res 1955;34:849–53.
[11] Osorio R, Toledano M, Leonardi G, et al. Microleakage and interfacial morphology of self-etching in class V resin composite restorations. J Biomed Mater Res Part B Appl Biomater 2003;66B:399–409.
[12] Arisu HD, Uçtasli MB, Eligüzeloglu E, et al. The effect of occlusal loading on the microleakage of class V restorations. Oper Dent 2008;33:135–41.
[13] Swift EJ, Perdigão J, Heyman HO. Bonding to enamel and dentin: a brief history and state of the art. Quint Int 1995;26:95–110.
[14] Hashimoto M, Ohno H, Kaga M, et al. In vivo degradation of resin-dentin bonds in humans over 1 to 3 years. J Dent Res 2000;79:1385–91.
[15] Carvalho RM, Fernandes CA, Villanueva R, et al. Tensile strength of human dentin as a function of tubule orientation and density. J Adhes Dent 2001;3:309–14.
[16] de Alexandre RS, Sundfeld RH, Giannini M, et al. The influence of temperature of three adhesive systems on bonding to ground enamel. Oper Dent 2008;33:272–81.
[17] Costa Pfeifer CS, Braga RR, Cardoso PE. Polymerization contraction stress of low-shrinkage composites and its correlation with microleakage in class V restorations. J Dent 2004;32:407–12.
[18] Fusayama T. Factors and prevention of pulp irritation by adhesive composite resin restorations. Quint Int 1987;18:633–40.
[19] Nakabayashi N, Pashley DM. Hybridization of dental hard tissue. first ed. Tokio: Quintessense; 1998.
[20] Sano H, Shono T, Takatsu T, et al. Microporous dentin zone beneath resin-impregnated layer. Oper Dent 1994;19:59–64.
[21] Sano H, Yoshiyama M, Ebisu S, et al. Comparative SEM and TEM observations of nanoleakage within the hybrid layer. Oper Dent 1995;20:160–7.
[22] Sano H, Yoshikawa T, Pereira PN, et al. Long-term durability of dentin bonds made with a self-etching primer, in vivo. J Dent Res 1999;78:906–11.
[23] Koshiro K, Inoue S, Tanaka T, et al. In vivo degradation of resin-dentin bonds produced by a self-etch vs. a total-etch adhesive system. Eur J Oral Sci 2004;112:368–75.
[24] Tay FR, Carvalho R, Sano H, et al. Effect of smear layers on the bonding of a self-etching primer to dentin. J Adhes Dent 2000;2:99–116.
[25] Tay FR, Pashley DH. Aggressiveness of contemporary self-etching systems. I: Depth of penetration beyond dentin smear layers. Dent Mater 2001;17: 296–308.
[26] Tay FR, Sano H, Carvalho R, et al. An ultrastructural study of the influence of acidity of self-etching primers and smear layer thickness on bonding to intact dentin. J Adhes Dent 2000;2:83–98.
[27] Brandt PD, de Wet FA, du Preez IC. Self-etching bonding systems: in-vitro micro-leakage evaluation. SADJ 2006;61(248):250–1.
[28] Peumans M, De Munck J, Van Landuyt K, et al. Five-year clinical effectiveness of a two-step self-etching adhesive. J Adhes Dent 2007;9:7–10.
[29] Van Meerbeek B, Kanumilli P, De Munck J, et al. 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.
[30] Brackett MG, Brackett WW, Haisch LD. Microleakage of Class V resin composites placed using self-etching resins: effect of prior enamel etching. Quint Int 2006;37:109–13.
[31] Deliperi S, Bardwell DN, Wegley C. Restoration interface microleakage using one total-etch and three self-etch adhesives. Oper Dent 2007;32:179–84.
[32] Toledano M, Osório R, Leonardi de G, et al. Influence of self-etching primer on the resin adhesion to enamel and dentin. Am J. Dent 2001;14:205–10.
[33] Moszner N, Salz U, Zimmermann J. Chemical aspects of self-etching enamel–dentin adhesives: a systematic review. Dent Mater 2005;21:895–910.
[34] Toledano M, Perdigão J, Osorio R, et al. Effect of dentin deproteinization on microleakage of class V composite restoration. Oper Dent 2000;25:497–504.
[35] Pashley DH, Tay FR. Aggressiveness of contemporary self-etching adhesives Part II: etching effects on unground enamel. Dent Mater 2001;17:430–44.
[36] Di Francescantonio M, Oliveira MT, Shinohara MS, et al. Bond strenth total-etch adhesive systems on intact and ground human enamel. Braz J Oral Sci 2007;6:1462–6.
[37] Loguercio AD, Moura SK, Pellizzaro A, et al. Durability of enamel bonding using two-step self-etch systems on ground and unground enamel. Oper Dent 2008;33:79–88.
[38] Shinohara MS, Oliveira MT, Hipólito VD, et al. SEM analysis of the acid-etched enamel patterns promoted by acidic monomers and phosphoric acids. J Appl Oral Sci 2006;14:427–35.
[39] Hannig M, Friedrichs C. Comparative in vivo and in vitro investigation of interfacial bond variability. Oper Dent 2001;26:3–11.
[40] Douglas WH, Fundingsland JW. Microleakage of three generically different fluoride-releasing liner/bases. J Dent 1992;20:365–9.
[41] Hannig M, Reinhardt KJ, Bott B. Self-etching primer vs phosphoric acid: an alternative concept for composite-to-enamel bonding. Oper Dent 1999;24:172–80.
[42] Miyazaki M, Sato M, Onose H, et al. Influence of thermal cycling on dentin bond strength of two-step bonding systems. Am J Dent 1998;11:118–22.
[43] Amaral CM, Hara AT, Pimenta LA, et al. Microleakage of hydrophilic adhesive systems in Class V composite restorations. Am J Dent 2001;14:31–3.
[44] Tittley K, Chernecky R, Maric B, et al. The morphology of the demineralized layer in primed dentin. Am J Dent 1994;7:22–6.
[45] Phrukkanon S, Burrow MF, Tyas MJ. The effect of dentine location and tubule orientation on the bond strengths between resin and dentine. J Dent 1999;27:265–74.
[46] Hashimoto M, Ohno H, Yoshida E, et al. Resin–enamel bonds made with self-etching primers on ground enamel. Eur J Oral Sci 2003;111:447–53.