Clin Oral Invest (2013) 17:1893–1900
DOI 10.1007/s00784-012-0878-y

ORIGINAL ARTICLE

Self-etching aspects of a three-step etch-and-rinse adhesive

Jose Bahillo · Miguel Roig · Tissiana Bortolotto · Ivo Krejci

Received: 20 June 2012 / Accepted: 31 October 2012 / Published online: 10 November 2012

Abstract
Purpose The purpose of this study is to assess the marginal adaptation of cavities restored with a three-step etch-and-rinse adhesive, OptiBond FL (OFL) under different application protocols.
Materials and methods Twenty-four class V cavities were prepared with half of the margins located in enamel and half in dentin. Cavities were restored with OFL and a micro-hybrid resin composite (Clearfil AP-X). Three groups ($n=8$) that differed in the etching technique were tested with thermomechanical loading, and specimens were subjected to quantitative marginal analysis before and after loading. Micromorphology of etching patters on enamel and dentin were observed with SEM. Data was evaluated with Kruskal–Wallis and Bonferroni post hoc test.
Results Significantly lower percent CM (46.9±19.5) were found after loading on enamel in group 3 compared to group 1 (96.5±5.1) and group 2 (93.1±8.1). However, no significant differences ($p=0.30$) were observed on dentin margins.
Conclusions Etching enamel with phosphoric acid but avoiding etching dentin before the application of OFL, optimal marginal adaptation could be obtained, evidencing a self-etching primer effect.
Clinical relevance A reliable adhesive interface was attained with the application of the three-step etch-and-rinse OFL adhesive with a selective enamel etching, representing an advantage on restoring deep cavities.

Key words Etch and rinse · OptiBond FL · Marginal adaptation · Class V · Etch pattern

J. Bahillo (✉)
Department of Operative Dentistry and Endodontics,
International University of Catalonia,
Sant Cugat del Valles,
08195 Barcelona, Spain
e-mail: jbahillovarela@gmail.com

M. Roig
Department of Operative Dentistry and Endodontics,
School of Dentistry, International University of Catalonia,
Sant Cugat del Valles,
08195 Barcelona, Spain

T. Bortolotto
Division of Cariology and Endodontology, School of Dentistry,
University of Geneva,
Rue Barthelemy-Menn, 19,
1205 Geneva, Switzerland

I. Krejci
Division of Cariology and Endodontology, School of Dentistry,
University of Geneva,
Rue Barthelemy-Menn, 19,
CH-1205 Geneva, Switzerland

Introduction

Adhesive systems can be classified according to their etching technique into etch-and-rinse (E&R) and self-etch (SE) products. E&R adhesive systems were the first to be introduced in the market and are often considered as being the adhesive system of reference [1–3].

OptiBond FL (OFL, Kerr, Orange, CA, USA) is a particle-filled, ethanol-based three-step E&R adhesive that has played an important role in adhesion, reporting favorable and reliable bonding effectiveness [4, 5]. The superior bonding effectiveness shown in vitro [6, 7] and the resultant clinical performance [5, 8] has been attributed to optimal enamel inter-locking and dentin hybridization, as demonstrated in various ultramorphologic interface analyses [6, 7, 9]. Based on all these data, OFL is considered by some authors as the gold standard.

One of the first chemical compounds that have been proposed to improve bonding to human dentin is the glycerol phosphate dimethacrylate (GPDM) [10], which is an acidic monomer containing methacrylated phosphoric acid esters, and it is present within the composition of OFL Primer.

Due to the poor etching pattern observed on enamel when SE adhesives are used, several studies proposed to transform SE adhesives to E&R by adding a phosphoric acid conditioning step [11–16]. However, due to the presence of GPDM in the primer, OFL may be used as a self-etching system when the etching step with phosphoric acid is avoided on dentin.

Therefore, the purpose of the present study was to evaluate the marginal adaptation of class V cavities restored with composite and an E&R adhesive system applied under three protocols that differ in the use of the phosphoric acid etching step. The null hypothesis tested was that there would be no effect on the marginal adaptation with different application protocols on enamel and dentin.

Materials and methods

A universal restorative composite Clearfil AP-X (Kuraray, Okayama, Japan) and a three-step etch-and-rinse adhesive system Optibond FL were used for this study (Table 1). Twenty-four recently extracted sound molars were randomly assigned to three equal groups on the basis of the etching method used. After scaling and pumicing, the teeth were mounted on custom-made specimen holders with their roots at the center using a cold-polymerizing resin (Technovit 4071, Heraeus Kulzer GmbH, Wehrheim, Germany). Prior to the mounting procedure, the apices were sealed with two coats of nail varnish. To simulate dentinal fluid flow, a cylindrical hole was drilled into the pulpal chamber approximately in the middle third of the root, and a metal tube with a diameter of 1.4 mm was then adhesively luted using a dentinal adhesive (Syntac Classic, IvoclarVivadent AG, Schaan, Liechtenstein). The pulpal tissue was not removed. This tube was connected by a flexible silicone hose to an infusion bottle placed 34 cm vertically above the test tooth. The infusion bottle was filled with horse serum (PAA Laboratories GmbH, Linz, Austria) and phosphate-buffered saline solution (PBS; Oxoid Ltd, Basingstoke, Hampshire, UK) diluted in a 1:3 ratio under a hydrostatic pressure of about 25 mm Hg. Twenty-four hours before starting the cavity preparations, using a three-way valve, the pulp chambers were evacuated with a vacuum pump and subsequently bubble-free filled with the above solution. As of this moment, the intrapulpal pressure was maintained at 25 mm Hg throughout the testing, i.e., during cavity preparation, restoration placement, finishing, and stressing.

In each tooth, a mixed class V, V-shaped cavity was prepared using fine diamond burs (Intensiv SA, Grancia, Switzerland), including both enamel and dentin margins. The dimensions of the V-shaped cavities were 3.0–3.5 mm in diameter, 2.5–3.0 mm in height, and 1.5 mm in depth. A slight enamel cavosuperficial margin was beveled to a crescent shape with a maximum width of 1.2 mm. using an extra-fine (15 μm) diamond bur (Intensiv SA) under ×12 magnification.

The teeth were divided into three groups (n=8) that differed in the application of phosphoric acid: group 1, enamel and dentin was etched with 37.5% phosphoric acid gel (Kerr, Scafati, Italy), applied for 30 s to enamel and 15 s to dentin; group 2, enamel was etched for 30 s and no phosphoric acid was applied on dentin; and group 3, no phosphoric acid was applied on either enamel or dentin. Then, OFL primer was applied on enamel and dentin using a microbrush with a continuous scrubbing motion for 15 s. Removal of excess solvent was done by drying the cavity with compressed air for 5 s, then OFL adhesive was applied with a microbrush to the primed surface for 15 s and spread with air for 5 s before a 20-s light curing. The cavity preparations were restored with a microhybrid resin composite Clearfil AP-X under ×12 magnification and light-cured for 40 s (L.E.D. Demetron II, serial number 792026758, Kerr, Orange, CA, USA) with a relative intensity of 800 mW/cm^2 (Curing Radiometer, Demetron Research, Danbury, CT, USA). The same operator performed the restoration of all groups.

Immediately after light polymerization, finishing and polishing of the restorations were carried out using flexible

Table 1 List of materials with composition, batch number and application mode

Material	Component (batch no.)	Application mode
OptiBond FL (Kerr, Orange, CA, USA), according to Mine et al. [9]	Primer (3271580): HEMA, GPDM, MMEP, water, ethanol, CQ, BHT (pH1.9)	Scrub for 15 s. Gently air dry 5 s
	Bond (3437447): Bis-GMA, HEMA, GDMA, CQ, ODMAB, Filler (fumed SiO_2, barium aluminoborosilicate, Na_2SiF_6), coupling factor A174 (approximately 48 wt% filled)	Apply thin coat for 15 s and gently air dry 5 s. Light-cure for 20 s
Clearfil AP-X (Kuraray, Okayama, Japan), according to manufacturer recommendations for use	Principle ingredients (1067BA) Silanated barium glass, silanated colloidal silica, silanated silica, Bis-GMA, TEGDMA, dl-Camphorquinone	Apply composite and light cure for 40 s

Bis-GMA bisphenol A diglycidyl ether dimethacrylate, *HEMA* 2-hydroxyethyl methacrylate, *GPDM* glycerol phosphate dimethacrylate, *MMEP* mono-2-methacryloyloxyethyl phthalate, *CQ* camphorquinone (photo-initiator), *BHT* butylhydroxytoluene or butylated hydroxytoluene or 2,6-di-(tert-butyl)-4-methylphenol (inhibitor), *GDMA* glycerol dimethacrylate, *ODMAB* 2-(ethylhexyl)-4-(dimethylamino)benzoate (coinitiator) discs (SofLex PopOn, 3 M ESPE AG, Seefeld, Germany). Then, impressions with a polyvinylsiloxane material (President light body, Coltène-Whaledent, Altstätten, Switzerland) were made of each restoration and poured with epoxy resin (Epofix Resin, Struers, Germany) and 24 h after gold sputtered to obtain replicas. They were subjected to the computer-assisted quantitative margin analysis in a scanning electron microscope (XL20, Philips, Eindhoven, The Netherlands) at ×200 magnification using a custom-made module programmed with an image processing software (Scion Image, Scion Corp, Frederik, MA, USA [17]. For the quantitative evaluation, a blinded and trained lab technician examined the specimens. The following criteria were considered for enamel and dentin margin analysis: percentages of perfect/continuous margins and percentages of noncontinuous margins due to the presence of: pure gaps, marginal enamel fractures, marginal dentin fractures, marginal restoration fractures, and overhang and underfilled margins, at each interval before and after loading.

After storage for 7 days at 37 °C in the dark, the teeth were loaded with simultaneous repeated thermal (×600 from 5 to 55 °C with a dwell time of 2 min) and mechanical stresses (240,000 chewing cycles at 1.7 Hz) by an antagonistic natural molar cusp with a maximum load of 49 N under the constant simulation of dentinal fluid flow according to a protocol described before by Krejci et al. [18]. After thermomechanical loading, the teeth were cleaned with toothpaste, rinsed with tap water, and impressions were taken again in order to perform the marginal replicas for SEM analysis after loading.

To qualitatively assess the self-etch pattern obtained with the different techniques, intact caries-free extracted human molars were selected. The crowns were sectioned perpendicular to their longitudinal axis above the roof of the pulp chamber using a precision slow-speed diamond saw (Isomet, Buehler Ltd., Evanston, IL, USA) under water cooling, to obtain a flat surface of dentin surrounded by enamel. Then, two grooves perpendicular to the flat surface were performed so that the surface could be divided into three sections. A metal matrix was fixed into each groove in order to achieve three separate flat surfaces that would not be contaminated by the different etching procedures. The first third was etched with 37.5 % phosphoric acid gel, applied 30 s to enamel and 15 s to dentin. After water rinsing and slight drying, OFL primer was applied to enamel and dentin for 15 s. On the second third, only OFL primer was applied, and the third control part was included without phosphoric acid or primer treatment. Fixation was performed by immersing the specimens in 2.5 % glutaraldehyde in 0.1 M sodium cacodylate buffer (pH7.4) for 12 h at 4 °C. After rinsing with sodium cacodylate for 1 h in three different baths and then in deonized water for 1 min, dehydration was performed by immersing the specimens in ethanol with increasing concentrations (50, 70, 90, and 100 %) and transferred to HMDS and allowed air-dry for 10 min [19].

Specimens were gold sputtered and observed in a scanning electron microscope at ×1,000 magnification (XL20, Philips, Eindhoven, The Netherlands).

Statistical analysis

In some groups, data of marginal adaptation was not normally distributed, as proved by Shapiro–Wilk test and therefore evaluated with Kruskal–Wallis and Bonferroni post hoc test. The level of confidence was set to 95 %. We used a one-way Bonferroni to assess whether there were significant differences between experimental groups both before and after loading on enamel and dentin margins.

Results

Percentages of continuous margins (%CM) before loading are shown in Table 2. No significant differences between groups were observed at dentin margins (p=0.33). However, on enamel margins, significantly lower %CM (80.2±10.1) were observed before loading in the group without phosphoric acid etching.

After loading (Table 3), significantly lower %CM on enamel margins (46.9±19.5) were observed in group 3, without phosphoric acid etching, in comparison to group 1 (96.5±5.1) and group 2 (93.1±8.1). Interestingly, the results on dentin were not statistically different (p=0.30) for the three groups.

Figure 1a and b represents the percentage of noncontinuous margins due to enamel fractures or pure gaps after loading. It can be observed that while the percentage of enamel fractures was similar in the three groups, a significantly higher percentage of pure open gaps was observed in the group in which phosphoric acid etching was avoided, indicating the absence of adhesion at this level.

Representative SEM images of enamel and dentin margins are presented in Figs. 2, 3 and 4 for the three groups.

The micromorphology of enamel and dentin surfaces after the different treatments is presented in Figs. 5 and 6. On enamel (Fig. 5), the best morphology was achieved when phosphoric acid was applied on the surface. On dentin (Fig. 6), while the morphology of the surface was quite similar for both OFL primer and H_3PO_4 treatment, we observed that in the surface treated with OFL primer, tubule openings were less evident, and some of them were still covered by smear layer, suggesting that the primer was less aggressive compared to phosphoric acid etching.

Table 2 Percentage of continuous margins (%CM, mean ± standard deviation) of each group before loading on enamel and dentin. Levels connected by the same letter are statistically similar and apply to each column

	%CM enamel before loading $p=0.012$	%CM dentin before loading $p=0.984$
Group 1 (H_3PO_4 E&D+OFL)	97.5±4.1 A	89.4±8.6 A
Group 2 (H_3PO_4 E+OFL)	89.9±14.5 AB	89±10.9 A
Group 3 (OFL no H_3PO_4)	80.2±10.1 B	90.1±16.6 A

Discussion

Based on recommendations of the American Dental Association for dentin and enamel adhesives, we performed class V restorations on noncarious teeth because of the following reasons [20]. The lesions do not have any macromechanical retention, and they have a small C-factor, which plays an important role in the performance of the adhesive system. Class V restorations may include margins on enamel and dentin with no major difficulties in cavity preparation, thus minimizing the operator factor variable and providing an appropriate location for the restorative and evaluation procedure.

One of the main objectives in adhesive restorative dentistry is to obtain a reliable and durable bonding interface creating restorations with clinical longevity, trying to avoid future leakage, recurrent caries, or pulpal irritation. Therefore, the simulation of oral conditions by thermomechanical loading, together with dentinal fluid simulation, assessing the marginal adaptation may serve as an appropriate model for the in vitro evaluation of adhesive systems [17, 18, 21]. Nevertheless, in vitro evaluations of marginal adaptation have been severely criticized in the last years, mainly due to the common belief that retention loss (and not the presence of marginal defects) is the most obvious sign of failure of an adhesive system [22]. However, it is known from the previous literature that clinical failure of restorations occurs most often due to inadequate sealing, with subsequent discoloration of the cavity margins, than due to restoration loss [23]. Moreover, the criterion marginal adaptation (together with cavosurface marginal discoloration, color match, anatomic form, and caries) is part of the US Public Health Service or Ryge guidelines to judge on the clinical performance of a restoration. These guidelines are by far the ones that had the greatest scientific impact in dentistry since their creation several decades ago [24]. Thus, it is difficult to explain the reason why marginal integrity is a widely accepted test when it is used in vivo and so criticized when it is used in vitro. Furthermore, Frankenberger et al. [25] reported that even if marginal integrity is only one among several factors, responsible for clinical success or clinical failure over time, thermomechanical loading and marginal analysis may be the in vitro test that is closest to the clinical situation. These authors reported that when high percentages of gap-free margins are observed in vitro, it could be assumed that the restoration's clinical behavior regarding marginal quality will not be problematic. This assumption was confirmed in a recent study [26], in which a correlation was observed between in vitro marginal adaptation and clinical outcome of class V restorations, when the same restorative composite was used in both in vitro and in vivo tests, justifying why in our study the primary criterion of evaluation was the percentages of continuous or gap-free margins, in class V cavities restored with the same composite resin.

Bond formation to enamel has proved reliable since Buonocore [27] demostrated that phosphoric acid etching increased resin-enamel bond strengths. Since then, several publications confirmed this assertion [11, 28–30]. Creation of a bond to dentin is more complicated due to the composition of the dentin substrate, presence of collagen, water, and smear layer deposition.

While several studies have transformed an SE adhesive to an E&R [11–16] by adding a phorphoric acid-etching step, there is not much literature evaluating a

Table 3 Percentage of continuous margins (%CM, mean±standard deviation) after loading on enamel and dentin

	%CM enamel after loading $p<0.001$	%CM dentin after loading $p=0.305$
Group 1 (H_3PO_4 E&D+OFL)	96.5±5.1 A	83.3±11.6 A
Group 2 (H_3PO_4 E+OFL)	93.1±8.1 A	68.7±25.1 A
Group 3 (OFL no H_3PO_4)	46.9±19.5 B	69.7±22.5 A

Levels connected by the same letter are statistically similar and apply to each column

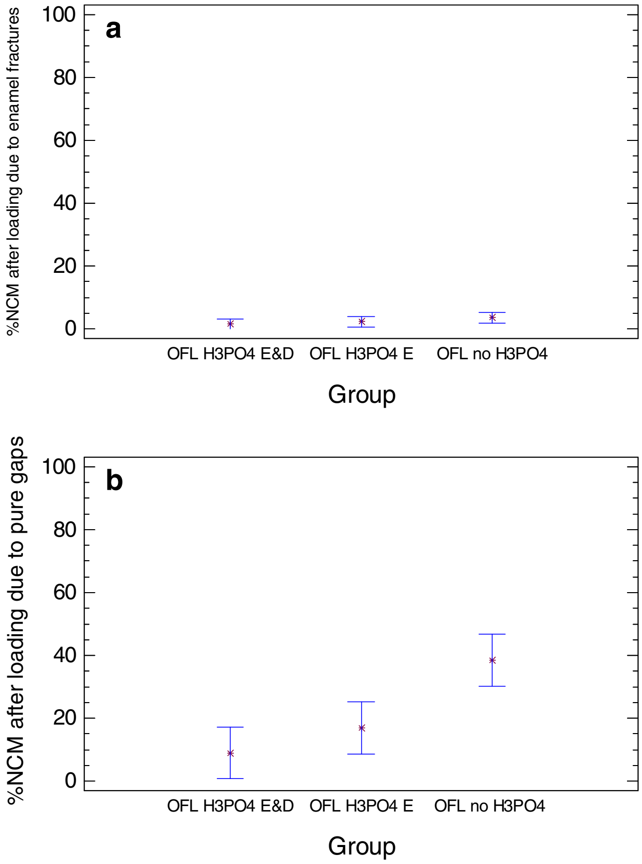

Fig. 1 Percentage of noncontinuous margins due to the presence of enamel fractures (**a**) and pure gaps (**b**). It can be observed that in the group in which H_3PO_4 was avoided, increased percentages of pure gaps were observed after loading, evidencing a lack of adhesion at the marginal level

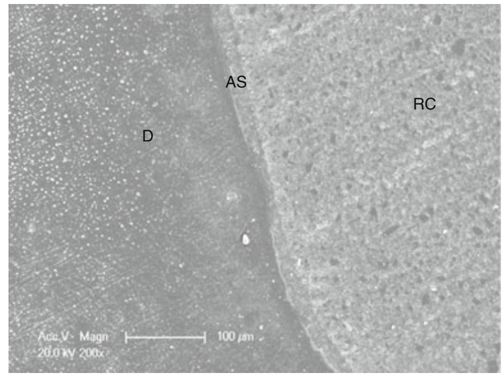

Fig. 3 SEM image of group 2 (H_3PO_4 E+OFL), showing a continuous margin between dentin (*D*), adhesive system (*AS*), and composite (*RC*) (magnification, ×200)

selective enamel etching for a three-step E&R adhesive system. The %CM after loading (Table 3) were significantly higher when enamel was conditioned with H_3PO_4 previous to the application of the primer (96.5 ±5.1 and 93.1±8.1) with respect to the group in which H_3PO_4 was avoided (46.9±19.5). This is in agreement with similar findings that have been reported in the literature [11, 31]. These better results on enamel were due to an enhanced mechanical interlocking resulting from the use of H_3PO_4 as shown on Fig. 5, which has a low pH. GPDM was probably not acidic enough to properly etch enamel, explaining why the %CM after loading on enamel was below 50 % (46.9±19.5). More, pure marginal gaps were observed when H_3PO_4 was avoided (Fig. 1b), showing a clear lack of adhesion at this level. Therefore, the null hypothesis was rejected for enamel margins.

Etching dentin with phosphoric acid did not improve significantly marginal adaptation either before or after loading; indicating a self-etching effect most probably due to the presence of the acidic monomer (GPDM) within the composition of OFL primer. These findings led to accept the null hypothesis on dentin. Nevertheless,

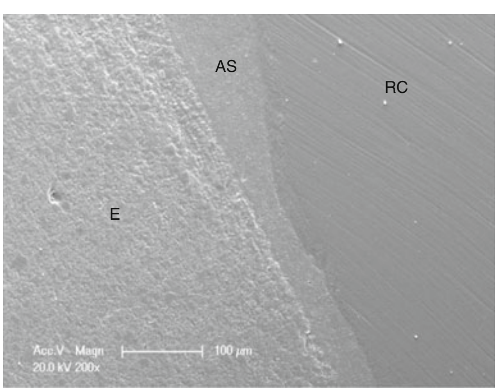

Fig. 2 SEM image of group 1 (H_3PO_4 E&D+OFL), showing continuous adhesive interface between enamel (*E*), adhesive system (*AS*), and composite (*RC*) (magnification, ×200)

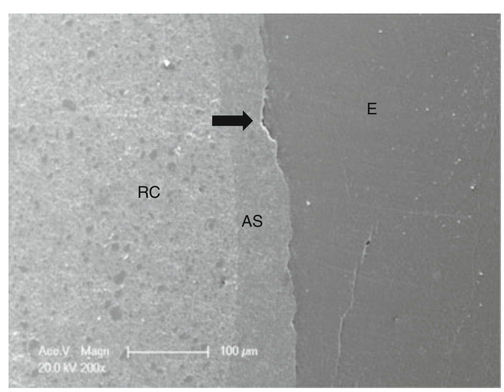

Fig. 4 Group 3 (OFL no H_3PO_4), SEM image with a noncontinuous margin observing a open gap (*arrow*) between enamel (*E*), adhesive system (*AS*), and composite (*RC*) (magnification, ×200)

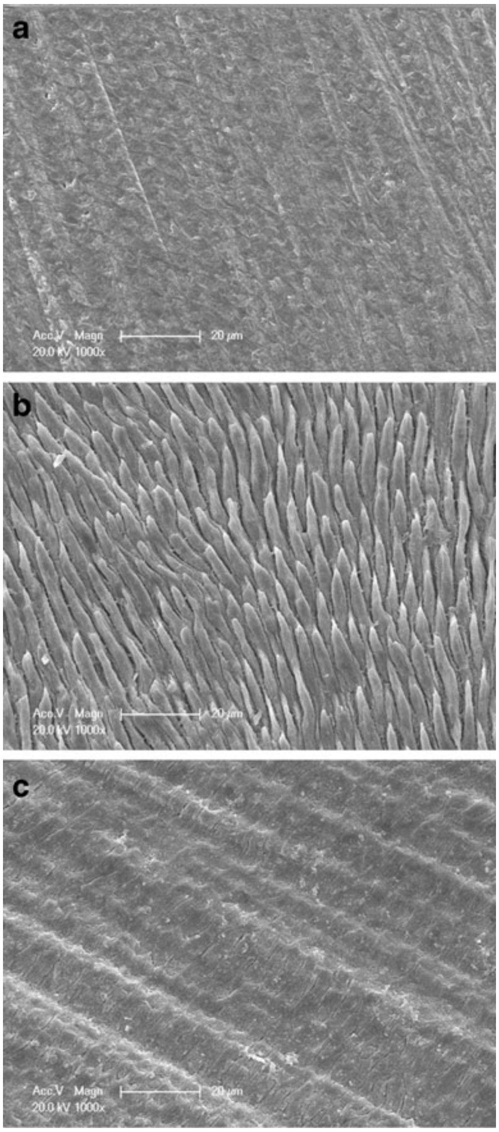

Fig. 5 SEM micrographs (×1,000) of enamel original surface without treatment (**a**), after H_3PO_4 etching (**b**), and after the application of OFL primer (**c**). See that micromorphology of enamel is clearly visible in **b**, that is, after acid conditioning with phosphoric acid

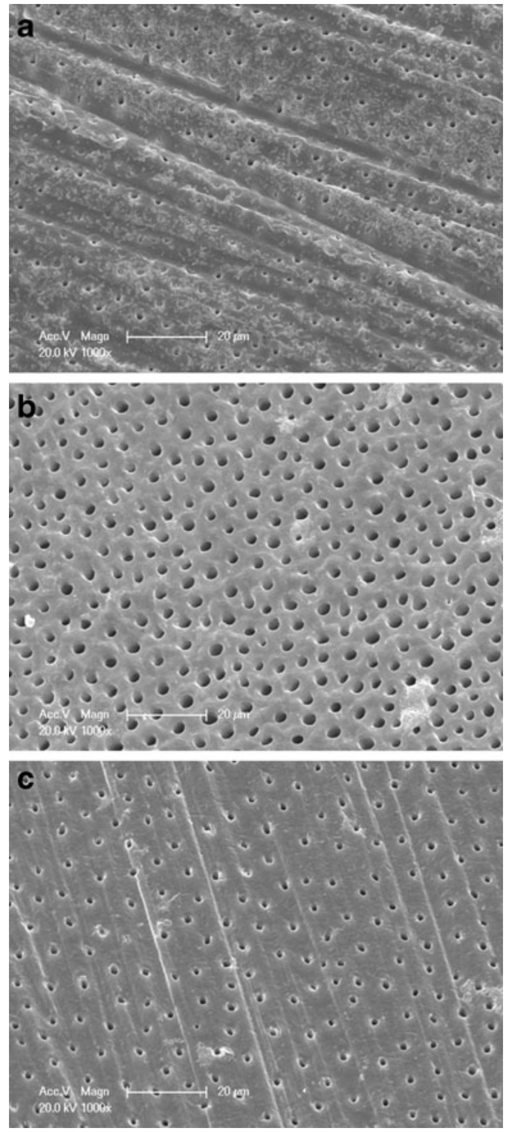

Fig. 6 SEM micrographs (×1,000) of dentin original surface without treatment (**a**), after H_3PO_4 etching (**b**), and after the application of OFL primer (**c**). Note that in **a** dentin is partially covered by smear layer; in **b**, dentinal tubules are completely opened, and in **c**, dentinal tubules are open due to the effect of the acidic monomer. However, the etching pattern looks less aggressive than the one obtained with H_3PO_4 etching

greater amount of variability in the bonding results were obtained in groups 2 and 3 compared to group 1. This may indicate that although there may be a self-etch effect from GPDM, it may not be as reliable as phosphoric acid. However, acid-etching deep dentin could have deleterious effects, justifying why some authors even recommend to avoid this procedure [32]. Phosphoric acid etching of cavities approaching the pulp may induce a moderate inflammatory response or pulpal irritation [33, 34]. Avoiding phosphoric acid etching of dentin thereby reduces the technique sensitivity of etch-and-rinse adhesives. Therefore, eliminating as many steps as possible in the bonding protocol could increase the efficiency of the procedure and would reduce technique sensitivity, as shown in the literature [35].

Because the effect of additional water storage was not assessed in the present study, it is not possible to know how these adhesive interfaces will behave when confronted by prolonged hydrolytic degradation. A previous study [36] tested the same adhesive system and found a significant decrease in bond strength from 1 to 6 months of water storage. These authors explained their findings by a combination of collagen and resin degradation within the hybrid layer. It is possible that due to the use of H_3PO_4 on dentin and then the application of the mentioned acidic primer, dentin was in fact etched "twice." The type of acid used to demineralize deep coronal dentin may significantly affect the quality of such bonding interface [37]. Phosphoric acid etching considerably increases dentin permeability as shown in the micromorphology in Fig. 6 and thus monomer diffusion into the pulp producing cytotoxicity. It is well known that inadequate etching procedures with a collapsed collagenous fibrillar network can decrease up to 90 % of the maximal level of bond strength values [38]. In this sense, additional studies are being performed by our research group in order to evaluate the hydrolytic degradation of these interfaces after long-term water storage.

Conclusions

Within the limitations of the present study, it can be concluded that when bur-prepared class V cavities were restored with the use of Optibond FL adhesive with selective enamel etching, high percentages of continuous margins were observed on dentin being not significantly different with the etch-and-rinse application procedure. These results might be due to a self-etching effect of the primer, owing to the presence of an acidic monomer within its composition. However, marginal adaptation on enamel was still enhanced by etching with phosphoric acid.

Acknowledgments The authors wish to express their gratitude to Marie-Claude Raymond for the help with the SEM evaluation.

Conflict of interest statement The authors declare that they have no conflict of interest.

References

1. Ermis R, De Munck J, Cardoso M, Coutinho E, Van Landuyt K, Poitevin A, Lambrechts P, Van Meerbeek B (2008) Bond strength of self-etch adhesives to dentin prepared with three different diamond burs. Dent Mater 24:978–985
2. Hamouda I, Samra N, Badawi M (2011) Microtensile bond strength of etch and rinse versus self-etch adhesive systems. J Mech Behav Biomed Mater 4:461–466
3. Pashley DH, Tay F, Breschi L, Tjäderhane L, Carvalho R, Carrilho M, Tezvergil-Mutluay A (2011) State of the art etch-and-rinse adhesives. Dent Mater 27:1–16
4. Peumans M, De Munck J, Van Landuyt KL, Kanumilli P, Yoshida Y, Inoue S, Lambrechts P, Van Meerbeek B (2007) Restoring cervical lesions with flexible composites. Dent Mater 23:749–754
5. Peumans M, De Munck J, Van Landuyt K, Poitevin A, Lambrechts P, Van Meerbeek B (2012) A 13-year clinical evaluation of two three-step etch-and-rinse adhesives in non-carious class-V lesions. Clin Oral Investig 16:129–137
6. De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, Van Meerbeek B (2005) A critical review of the durability of adhesion to tooth tissue: methods and results. J Dent Res 84:118–132
7. Van Landuyt KL, Mine A, De Munck J, Jaecques S, Peumans M, Lambrechts P, Van Meerbeek B (2009) Are one-step adhesives easier to use and better performing? Multifactorial assessment of contemporary one-step self-etching adhesives. J Adhes Dent 11:175–190
8. Van Meerbeek B, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, Peumans M (2004) A randomized, controlled trial evaluating the three-year clinical effectiveness of two etch & rinse adhesives in cervical lesions. Oper Dent 29:376–385
9. Mine A, De Munck J, Van Landuyt KL, Poitevin A, Kuboki T, Yoshida Y, Suzuki K, Lambrechts P, Van Meerbeek B (2008) Bonding effectiveness and interfacial characterization of a HEMA/TEGDMA-free three-step etch&rinse adhesive. J Dent 36:767–773
10. Söderholm KJ (2007) Dental adhesives … How it all started and later evolved. J Adhes Dent 9:227–230
11. Frankenberger R, Lohbauer U, Roggendorf MJ, Naumann M, Taschner M (2008) Selective enamel etching reconsidered: better than etch-and-rinse and self-etch? J Adhes Dent 10:339–344
12. Ikeda M, Tsubota K, Takamizawa T, Yoshida T, Miyazaki M, Platt JA (2008) Bonding durability of single-step adhesives to previously acid-etched dentin. Oper Dent 33:702–709
13. Lührs A-K, Guhr S, Schilke R, Borchers L, Geurtsen W, Günay H (2008) Shear bond strength of self-etch adhesives to enamel with additional phosphoric acid etching. Oper Dent 33:155–162
14. Taschner M, Nato F, Mazzoni A, Frankenberger R, Krämer N, Di Lenarda R, Petschelt A, Breschi L (2010) Role of preliminary etching for one-step self-etch adhesives. Eur J Oral Sci 118:517–524
15. Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, Van Landuyt KL (2011) State of the art of self-etch adhesives. Dent Mater 27:17–28
16. Watanabe T, Tsubota K, Takamizawa T, Kurokawa H, Rikuta A, Ando S, Miyazaki M (2008) Effect of prior acid etching on bonding durability of single-step adhesives. Oper Dent 33–4:426–433
17. Bortolotto T, Ferrari M, Onisor I, Tay F, Krejci I (2007) Degradation of thermo-mechanically loaded adhesive class V restorations after 18 months of water storage. Am J Dent 20:83–89
18. Krejci I, Kuster M, Lutz F (1993) Influence of dentinal fluid and stress on marginal adaptation of resin composites. J Dent Res 72:490–495
19. Perdigao J, Lambrechts P, Van Meerbeek B, Vanherle G, Lopes AL (1995) Field emission SEM comparison of four postfixation drying techniques for human dentin. J Biomed Mater Res 29:1111–1120
20. Van Meerbeek B, Perdigão J, Lambrechts P, Vanherle G (1998) The clinical performance of adhesives. J Dent 26:1–20
21. Frankenberger R, Strobel WO, Kramer N, Lohbauer U, Winterscheidt J, Winterscheidt B, Petschelt A (2003) Evaluation of the fatigue behavior of the resin–dentin bond with the use of different methods. J Biomed Mater Res B Appl Biomater 67:712–721
22. Heintze SD (2007) Systematic reviews: I. The correlation between laboratory tests on marginal quality and bond strength. II. The correlation between marginal quality and clinical outcome. J Adhes Dent 9:77–106
23. Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, Coutinho E, Suzuki K, Lambrechts P, Van Meerbeek B (2007) Systematic review of the chemical composition of contemporary dental adhesives. Biomaterials 28:3757–3785
24. Bayne SC, Schmalz G (2005) Reprinting the classic article on USPHS evaluation methods for measuring the clinical research performance of restorative materials. Clin Oral Invest 9:209–214
25. Frankenberger R, Krämer N, Lohbauer U, Nikolaenko SA, Reich SM (2007) Marginal integrity: is the clinical performance of bonded restorations predictable in vitro? J Adhes Dent 9:107–116

26. Heintze SD, Blunck U, Göhring TN, Rousson V (2009) Marginal adaptation in vitro and clinical outcome of class V restorations. Dent Mater 25:605–620
27. Buonocore G, Wileman W, Brudevold F (1956) A report on a resin composition capable of bonding human dentin surfaces. J Dent Res 35:846–851
28. Dietschi D, Herzfeld D (1998) In vitro evaluation of marginal and internal adaptation of class II resin composite restorations after thermal and occlusal stressing. Eur J Oral Sci 106:1033–1042
29. Erickson RL, Barkmeier WW, Latta MA (2009) The role of etching in bonding to enamel: a comparison of self-etching and etch-and-rinse adhesive systems. Dent Mater 25:1459–1467
30. Frankenberger R, Krämer N, Petschelt A (2000) Long-term effect of dentin primers on enamel bond strength and marginal adaptation. Oper Dent 25:11–19
31. De Munck J, Van Meerbeek B, Yudhira R, Lambrechts P, Vanherle G (2002) Micro-tensile bond strenght of two adhesives to Erbium: YAG-lased vs. bur-cut enamel and dentin. Eur J Oral Sci 110:322–329
32. Rathke A, Alt A, Gambin N, Haller B (2007) Dentin diffusion of HEMA released from etch-and-rinse and self-etch bonding systems. Eur J Oral Sci 115:510–516
33. Costa CA, Giro EM, Do Nascemento AB, Teixeira HM, Hebling J (2003) Short-term evaluation of the pulpo-dentin complex response to a resin-modified glass-ionomer cement and a bonding agent applied in deep cavities. Dent Mater 19:739–746
34. De Souza Costa CA, Do Nascimento ABL, Teixeira HM (2002) Response of human pulps following acid conditioning and application of a bonding agent in deep cavities. Dent Mater 18:543–551
35. Pegado RE, Do Amaral FL, Flório FM, Basting RT (2010) Effect of different bonding strategies on adhesion to deep and superficial permanent dentin. Eur J Dent 4:110–117
36. Armstrong SR, Vargas MA, Chung I, Pashley DH, Campbell JA, Laffoon JE, Qian F (2004) Resin-dentin interfacial ultrastructure and microtensile dentin bond strenght after five-year water storage. Oper Dent 29:705–712
37. Caiado AC, de Goes MF, de Souza-Filho FJ, Rueggeberg FA (2010) The effect of acid etchant type and dentin location on tubular density and dimension. J Prosthet Dent 103:352–361
38. Pashley DH, Ciucchi B, Sano H, Carvalho RM, Russell CM (1995) Bond strength versus dentine structure: a modelling approach. Arch Oral Biol 40:1109–1118