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ORIGINAL ARTICLE

Marginal adaptation of an etch-and-rinse adhesive with a new type of solvent in class II cavities after artificial aging

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Abstract This in vitro study evaluated the marginal adaptation of etch-and-rinse adhesives. Standardized class II cavities were cut in 40 human molars with one proximal box limited within enamel and one proximal box extending into dentin. Teeth were assigned randomly to five groups ($n=8$) and restored with incrementally placed composite restorations. Five combinations were tested: G1, XP Bond+Ceram-X Mono; G2, P&B NT+Ceram-X Mono; G3, Optibond Solo Plus+Ceram-X Mono; R1, Syntac Classic+Tetric EvoCeram; R2, Scotchbond 1 XT+Z250. After finishing and polishing, teeth were stored for 48 h in water at 37°C before subjected to artificial aging by thermal stress (5/55°C; ×2,000; 30 s) and mechanical loading (50 N; ×50,000). Marginal adaptation of the restorations was evaluated in a SEM (×200) using a replica technique. Statistical analysis was performed with nonparametric test methods ($p<0.05$). The percentages of "perfect margin" after aging ranged from 95.9% to 99.6% in enamel and 85.9% to 96.0% in dentin. "Marginal opening" was observed between 0.1% to 2.6% in enamel and 2.6% to 11.8% in dentin. In enamel and dentin, both, G3 showed significantly more gap formation than G1 and G2. Comparing marginal adaptation to enamel and dentin within each group yielded only for G1 no significant differences. Tert-butanol-based XP Bond showed excellent marginal adaptation in both enamel and dentin.

Keywords Class II restorations · Dentin adhesives · Composite · Marginal adaptation · Solvent

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Introduction

Despite improvements in the formulation of modern dentin adhesive systems, the bond strength and marginal adaptation of composite resins to dentin seems still inferior and less predictable than adhesion to enamel [1, 2]. However, in clinical dentistry, most of the cavities, especially when restorations in posterior teeth are replaced, are not confined exclusively within enamel but show a mixed type configuration with finishing lines in enamel as well as in dentin [1, 3]. In particular, the adhesive interface between tooth and restorative material at the gingival finish line has been recognized as one of the most problematic regions [4, 5]. While a great number of self-etching primers and adhesives have emerged on the market, which are considered less technique sensitive and less time consuming than etch-and-rinse adhesives [6], the latter are still considered gold standard with respect to long-term bond strength and marginal seal, especially for the restoration of high-load class II cavities or when restorations are bonded to uninstrumented tooth tissues such as sclerotic dentin in class V lesions or virgin enamel in anterior diastema closures.

The monomers of adhesive systems are carried by a solvent which is usually either water, ethanol, acetone, or a combination of those [7]. The type of solvent strongly influences the clinical application protocol of etch-and-rinse adhesive systems, which are—despite the growing influence of self-etch adhesives—still an important group of bonding agents that are clinically widely used [8]. Etch-and-rinse adhesives require dentin demineralization using phosphoric acid to expose the collagen network. Concerns regarding drying of the dentin protein mesh prior to application of the bonding system are discussed extensively in the literature [9–11]. Especially acetone-based systems require a moist dentin surface after acid etching in order to enable the monomers of the bonding system to completely penetrate the decalcified area. A collapse of the exposed collagen network due to overdrying would seriously lower bond strengths and increase the risk of postoperative symptoms [12–14]. On the other hand, it is a real and well-documented risk that acid-etched dentin with excess water shows detrimental effects even with bonding systems that advocate bonding to moist dentin, being referred to as the “overwet phenomenon” [15–18]. It has been proposed that an adhesive system capable of penetrating dry and collapsed demineralized dentin would improve the bonding procedure and result in a less technique-sensitive application procedure and better clinical performance [8, 11].

XP Bond (Dentsply, Konstanz, Germany) is a two-step one-bottle etch-and-rinse adhesive which first-time uses the alcohol tert-butanol to dissolve the monomers. It is claimed to be less technique sensitive due to an improved ability to diffuse through partially collapsed demineralized dentin [8].

This study assesses the marginal adaptation of XP Bond in large class II cavities after artificial aging in comparison with well-established competitive adhesive and composite systems. The null hypothesis tested was that the type of restorative system used does not significantly affect the marginal seal in enamel or dentin.

Materials and methods

Specimen preparation

Forty freshly extracted caries-free human molars of the permanent dentition, stored in a 0.25% mixture of sodium azide in Ringer's solution until the date of use, were used in this in vitro study. After cleaning the teeth with a scaler and polishing with pumice, standardized class II inlay cavities were prepared, with one proximal box limited within enamel (1–1.5 mm above the cemento-enamel junction) and one proximal box extending into dentin (1–1.5 mm below the cemento-enamel junction; Fig. 1). The dimensions of the cavities were 4.0 mm in width and 3–3.5 mm in depth at the occlusal isthmus and 5.0 mm in width at the proximal boxes. The depth of the proximal boxes in direction to the axial pulpal walls was 1.5 mm. To achieve divergence angles between opposing walls of 10° to 12°, cavities were prepared using coarse diamond burs with a slight taper (855.314,

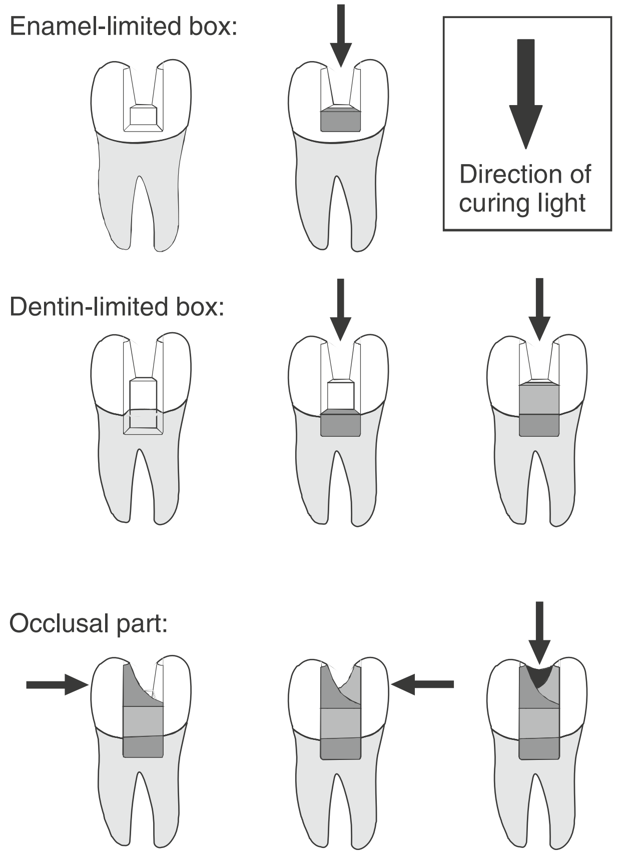

Fig. 1 Incremental restoration technique and light-curing direction [2]

Komet, Lemgo, Germany) in a high-speed dental handpiece with copious water spray. Fine-grained diamond burs of the same shape (8855.314, Komet) were used for finishing the preparations. The internal point and line angles were rounded and enamel margins were not beveled but prepared in butt-joint configuration [2]. After visual inspection of the cavities for imperfect finish lines, the 40 prepared teeth were randomly assigned to five experimental groups with eight teeth each (Table 1).

Two different types of comparisons were made:

(a) Comparison of different adhesives with different solvents: XP Bond (G1) was compared with Prime & Bond NT and Optibond Solo Plus, each of these groups used the same composite (Ceram-X Mono).
(b) Comparison of different restorative systems (adhesive+ respective composite of same manufacturer): XP Bond+ Ceram-X Mono (G1) was compared with Syntac Classic+Tetric EvoCeram (R1) and Scotchbond 1 XT+ Z250 (R2)

Instructions for use for each material were strictly followed.

All enamel and dentin surfaces of the cavities were conditioned with 36% phosphoric acid gel (DeTrey Conditioner 36, Dentsply DeTrey, Konstanz, Germany),

Table 1 Experimental groups and materials used

Group	Adhesive	Type	Solvent of adhesive	Composite
G1	XP Bond[a]	2-step etch-and-rinse	t-butanol	Ceram-X Mono (M2)[a]
G2	Prime & Bond NT[a]	2-step etch-and-rinse	Acetone	Ceram-X Mono (M2)[a]
G3	Optibond Solo Plus[d]	2-step etch-and-rinse	Ethanol	Ceram-X Mono (M2)[a]
R1	Syntac Classic[b]	3-step etch-and-rinse	Primer: acetone–water Adhesive: water Heliobond: –	Tetric EvoCeram (A2)[b]
R2	Scotchbond 1 XT[c]	2-step etch-and-rinse	Ethanol–water	Z250 (A2)[c]

[a] Dentsply DeTrey (Konstanz, Germany)

[b] Ivoclar-Vivadent (Schaan, Liechtenstein)

[c] 3M Espe (Seefeld, Germany)

[d] KerrHawe (Bioggio, Switzerland)

starting acid application on enamel, leaving undisturbed for 15 s, then covering the dentin preparation surfaces for an additional 15 s (total-etch technique). After thoroughly rinsing with water, the cavities were then gently dried with oil-free compressed air, taking care to avoid desiccation of the tooth substrate (moist bonding technique). Following the application and light-curing of the adhesive systems, the cavities were restored with composite using a horizontal and oblique layering technique with five increments in the dentin-limited proximal box and four increments in the enamel-limited box (Fig. 1). Each increment with a maximum thickness of 2 mm was light-cured individually with a LED curing unit (SmartLite PS, Dentsply DeTrey, Konstanz, Germany) according to manufacturers' recommendations. The light output of the curing unit was monitored at 1065 mW/cm^2 with a calibrated light meter (CureRite, Dentsply DeTrey, Konstanz, Germany). All restorations were finished and polished immediately after placement using finishing diamond burs and flexible aluminum oxide polishing disks (Sof-Lex, 3 M ESPE, Seefeld, Germany).

Thermocycling and mechanical loading

After 48 h storage in distilled water at 37°C, the restored teeth were subjected to artificial aging by thermocycling and mechanical loading. All specimens were immersed alternately in water baths at 5°C and 55°C for 2,000 cycles, with a dwell time of 30 s in each bath and a transfer time of 15 s. Mechanical loading of the teeth, which were mounted on metallic specimen holders with a light-curing composite, was conducted in the Munich Oral Environment [19]. The carefully aligned teeth were loaded in the central fossa of the restorations in axial direction with a force of 50 N for 50,000 times at a frequency of 1 Hz. The antagonist material was a Degusit sphere (6 mm in diameter), which exhibits a hardness and wear resistance similar to natural enamel [19–22]. The metal specimen holders were mounted on a hard rubber element, which allowed a sliding movement of the tooth between the first contact on an inclined plane to the central fossa [23]. During mechanical loading, the teeth were continuously immersed in Ringer's solution. This oral simulation device exhibits similar functions to the machine developed by Krejci et al. [24].

Specimen evaluation

After thermal and mechanical loading, impressions of the restored teeth were taken using a polyether material (Impregum F, Espe, Seefeld, Germany). Replicas were made by casting the impressions with an epoxy resin and gold sputtered (SEM Autocoating Unit E5200, Polaron Equipment Ltd., Watford, England). The interface between composite and tooth tissues at the proximal extensions was analyzed with a well-established quantitative and qualitative marginal analysis in a scanning electron microscope (Leitz AMR1200, Leitz, Wetzlar, Germany) at ×200 magnification [1, 23, 25–28]. Differentiations between enamel-limited and dentin-limited interfaces were made. The SEM evaluation was performed by a single qualified operator using the criteria listed in Table 2. Results of the marginal quality, based on the four rating categories, are expressed as the percentage of the total length of the particular margin in enamel and dentin.

Statistical analysis

Mean values and standard deviations of eight replications were calculated for each assessed marginal interface. Preliminary analysis of the data (SPSS for Windows, SPSS Inc., Chicago, USA) showed not for all experimental groups normal distribution (Shapiro-Wilks test) and homogeneity of variances (Levene's test). Further statistical

Table 2 Criteria for assessment of marginal quality of adhesive restorations

Criterion	Description
Perfect margin	The interface between the restorative material and tooth structure exhibits a smooth surface without any interruption in continuity
Marginal opening	The interface between the restorative material and tooth structure is separated by a gap caused by an adhesive failure
Marginal swelling	The interface between the restorative material and tooth structure exhibits a swelling phenomenon at the cavity margin
Artefact	The interface between the restorative material and tooth structure cannot be exactly assessed, e.g., due to overhanging excess material or errors in the replication procedure (voids)

analysis among the experimental groups for each assessed marginal interface was performed with nonparametric test methods, using the Kruskal–Wallis H and Mann–Whitney U tests with Bonferroni correction for pairwise multiple comparisons at a significance level of $p<0.05$. Performance differences for enamel margins and dentin margins within each experimental group were statistically analyzed using the Mann–Whitney U test at a significance level of $p<0.05$.

Results

Marginal areas assessed with the criterion "artefact" are a result of overhanging excess material or errors during the replication procedure. As margin segments scored "artefact" or "marginal swelling" did not exceed 2.5% in all groups, these results are therefore not reported in detail. Mean percentages, standard deviations and statistical significances of "perfect margins" and "marginal opening", differentiated by enamel-limited and dentin-limited interfaces are reported in Tables 3 and 4.

Proximal adaptation to enamel

The proximal adaptation of the adhesive restorations to enamel exhibited high percentages of perfect margin after artificial aging, ranging from 95.9% to 99.6%, and minor areas with marginal openings (0.1% to 2.6%). Comparing different adhesives placed with Ceram-X Mono composite showed significantly more perfect margins and significantly less marginal gap formation for tert-butanol-based XP Bond and acetone-based Prime & Bond NT than Optibond Solo Plus (Table 3). Data analysis of the performance of three complete restorative systems (G1, R1, and R2) revealed no significant differences among the groups (Table 3).

Table 3 Marginal quality of the interface between composite and enamel after thermal and mechanical loading as mean percentage (standard deviation) of the entire length of the particular margin

Experimental group	Marginal quality in enamel	
	Perfect margin	Marginal opening
Adhesives		
H-test	P=0.03	P=0.04
G1	98.9 (1.5) b	0.5 (1.2) a
G2	98.5 (2.3) b	0.7 (1.3) a
G3	95.9 (2.6) a	2.6(2.7) b
Restorative systems		
H-test	P=0.22	P=0.26
G1	98.9 (1.5) a	0.5 (1.2) a
R1	99.6 (0.8) a	0.1 (0.3) a
R2	98.3 (1.9) a	1.0 (1.4) a

Lowercase letters indicate statistically homogeneous subsets within each criterion among the different experimental groups

Proximal adaptation to dentin

The proximal adaptation of the adhesive restorations to dentin exhibited lower percentages of perfect margin after artificial aging compared with enamel, ranging from 85.9% to 96.0%. Also, a trend towards more marginal gaps could be observed, presenting a range from 2.6% to 11.8% marginal openings. Comparing different adhesives placed with Ceram-X Mono composite showed significantly more perfect margins and significantly less marginal gap formation for tert-butanol-based XP Bond and acetone-based Prime & Bond NT than Optibond Solo Plus (Table 4). Data

Table 4 Marginal quality of the interface between composite and dentin after thermal and mechanical loading as mean percentage (standard deviation) of the entire length of the particular margin

Experimental group	Marginal quality in dentin	
	Perfect margin	Marginal opening
Adhesives		
H-test	P=0.01	P=0.01
G1	95.1 (5.1) b	2.7 (4.9) a
G2	92.6 (3.2) b	5.4 (4.3) a
G3	85.9 (4.9) a	11.8 (4.4) b
Restorative systems		
H-test	P=0.01	P=0.01
G1	95.1 (5.1) b	2.7 (4.9) a
R1	96.0 (2.7) b	2.6 (1.9) a
R2	89.5 (4.7) a	8.0 (4.1) b

Lowercase letters indicate statistically homogeneous subsets within each criterion among the different experimental groups analysis of the performance of three complete restorative systems (G1, R1, and R2) revealed significantly more perfect margins and significantly less marginal gap formation for tert-butanol-based XP Bond+Ceram-X Mono and acetone–water-based Syntac Classic+Tetric EvoCeram than ethanol–water-based Scotchbond 1 XT+Z250 (Table 4).

Comparing the marginal adaptation of all adhesive restorative systems to enamel and dentin within each experimental group yielded significantly less perfect margins and more gap formation at the dentin interface compared with enamel margins for all groups except XP Bond.

Discussion

The effectiveness of adhesives and adhesive restorative systems can be generally judged by the marginal adaptation of these materials/material combinations at the interface with tooth substrate [29]. Within the limitations of laboratory studies, quantitative marginal analysis by scanning electron microscopy has proven to be an exact and reliable assessment method for the evaluation of the marginal adaptation of adhesive restorations [25, 30, 31]. The evaluation of gap formation between composite materials and tooth structures is a realistic and valid test for adhesive restorations [32]. A computer-assisted quantitative SEM analysis on the replicas of adhesive restorations was performed to evaluate marginal adaptation. This method is truly quantitative as the presence or absence of gaps is expressed as percentage of “perfect margin” or “marginal opening” along the tooth/restoration interface [33]. The method is nondestructive as well as highly discriminative, which allows the potential of different operative techniques or the performance of various adhesive materials to be quantified in terms of percentage of perfect margin or gap formation [26]. Marginal adaptation of the restorative systems to enamel and dentin was assessed in the present study only after artificial aging, because no significant information could be obtained from measurements prior to the thermal and mechanical fatigue test in preliminary studies [23].

Marginal adaptation is influenced by many different factors, among others quality of enamel and dentin, shrinkage and shrinkage stress of the restorative materials, chemistry of the adhesive system, cavity size and geometry, C-factor, mode of composite application and polymerization protocol [34, 35].

The type of solvent strongly influences the clinical application protocol of etch-and-rinse adhesive systems. While acetone-based systems only work well on a moist dentin surface as acetone is a water-chaser and can lead to rather poor results on overdried acid-etched dentin surfaces on the other hand water-based systems are not so sensitive with regard to dentin moisture content, as they have inherent rewetting properties, but require a longer evaporation time for the solvent as water has a considerable lower vapor pressure [7]. If the solvent is not completely evaporated before light-curing the adhesive, flaws can weaken the hybrid layer probably causing premature restoration failure [36]. A new type of solvent for adhesives, namely tert-butanol was introduced for XP Bond. Tert-Butanol (2-*methyl*-2-propanol) consists of a C4-body with an alcohol group surrounded by three methyl groups, making it totally miscible with water and polymerizable resins both. Although tert-butanol has a higher molecular weight than ethanol, evaporation rate is almost the same, with a latent heat of vaporization of 41 kJ/mol and 42 kJ/mol, respectively [37]. Vapor pressure of the different kinds of solvents at 20°C is given 2,330 Pa for water, 4,133 Pa for tert-butanol, 5,900 Pa for ethanol, and 23,300 Pa for acetone [38]. The properties of tert-butanol provide the ability of using a dappen dish and increasing the resin content of the adhesive which results in an increase of adhesive layer thickness and a higher degree of technique robustness as compared with acetone-based systems [36]. The solvent used for etch-and-rinse adhesives is a major factor affecting handling characteristics and performance [36, 39].

In vitro and in vivo studies have proved that multi-bottle multi-step adhesives such as Syntac Classic in combination with the etch-and-rinse approach provide reliable bond and marginal sealing qualities even after long-term observation [32, 36, 40–43]. A comparison of bond strength values of XP Bond, Scotchbond 1 XT and Optibond Solo Plus showed similar values (range, 24.2 to 28.3 MPa) for all three adhesives to enamel and dentin being not statistically different from each other [44]. Only Syntac Classic yielded significantly lower bond strengths to enamel and dentin respectively in the aforementioned study. However, bond strength seems not to be correlated to marginal adaptation [45]. XP Bond/Ceram-X Mono exhibits in the present study a marginal performance after accelerated artificial aging that is similar to the well-established one-bottle system Prime & Bond NT/Ceram-X Mono and being significantly better than Optibond Solo Plus/Ceram-X Mono.

Physico-mechanical properties of the composite material such as modulus of elasticity, shrinkage stress, hygroscopic expansion, etc.—determine to a large extent the marginal adaptation of the combination adhesive/composite. Thus, it is advisable to use the same composite for all tested adhesives within a study. On the other hand, it is recommended to stay within one manufacturer's product chain when placing adhesive restorations. To reflect this, XP Bond/Ceram-X Mono was further compared with two well-established restorative systems, multi-bottle multi-step Syntac Classic+Tetric EvoCeram and Scotchbond 1 XT+Z250. Keeping the aforementioned limitations in mind, XP Bond/Ceram-X

Mono performed in enamel equally well than Syntac Classic/Tetric EvoCeram and Scotchbond 1 XT/Z250. In dentin, XP Bond/Ceram-X Mono and Syntac Classic/Tetric EvoCeram were significantly better than Scotchbond 1 XT/Z250.

The results comparing one composite with different adhesives and different complete restorative systems show that the new formulation of XP Bond with a tertiary butanol used as solvent yields a competitive adhesion to both enamel and dentin combined with the advantages to be more user-friendly than multiple-bottle bonding agents. Latta suggests, based on the results of a micro-Raman spectroscopy investigation, a chemical interaction between XP Bond and dentin by observing the formation of calcium phosphate complexes derived from phosphate esters which are contained in the formulation of XP Bond and mineral apatite in the dentin [44]. In addition, a complete resin infiltration into the phosphoric acid demineralized dentin was shown, supporting the assumption that the new formulation of XP Bond will be less sensitive to residual dentin moisture and will allow full adhesive resin penetration under a wide range of dentin conditions [44].

In this in vitro study, marginal adaptation to dentin was found to be significantly worse compared with enamel-limited sections for all adhesive/composite restorations except XP Bond/Ceram-X Mono. Confirming the results of this study, several authors reported that adhesion to dentin for both direct and inlay techniques demonstrated less perfect margins and more marginal openings compared with the enamel-limited cavity segments [1, 30, 46]. Even with newest generation bonding systems which establish considerable high bond strengths to dentin, a perfect bond to dentin without gap formation cannot be established yet [47]. Dentin shows a wider biologic variability than enamel which makes it much more difficult to create a good adhesion which is able to resist the negative effects of polymerization shrinkage and subsequent thermal and mechanical stress factors. Bonding to dentin is a challenge because of its morphology, composition and high water content [27, 48]. Several studies proved that aging of the resin-dentin interface results in significantly reduced marginal quality and bond strength and more microleakage [46, 49]. XP Bond/Ceram-X Mono was the only combination in the present study that showed no significant decrease of marginal quality in dentin. The chemical composition of the adhesive is a major factor in achieving a strong and durable bond to enamel and dentin [50]. XP Bond uses tert-butanol as solvent and provides increased resin content which will result after light curing in a thicker bonding layer consisting of a dense polymer matrix that promotes better sealing [50]. This is confirmed by Blunck et al., who found 100% retention and absence of discoloration for XP Bond/Ceram-X duo after 6 month in a clinical study that restored noncarious cervical lesions [8].

Conclusions

Two-step one-bottle tert-butanol-based XP Bond showed excellent marginal adaptation in both enamel and dentin.

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