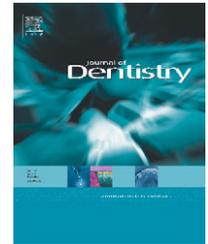


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Marginal quality of flowable 4-mm base vs. conventionally layered resin composite

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ABSTRACT

Objective: This study evaluated marginal integrity of bonded posterior resin composite fillings to enamel and dentine with and without 4 mm flowable base, before and after thermo-mechanical loading (TML).

Methods: 80 MOD cavities with one proximal box beneath the CEJ were prepared in extracted human third molars. Direct resin composite restorations (SDR with CeramX mono, Tetric EvoCeram, Filtek Supreme XT, and Venus Diamond or the respective resin composites alone) were bonded with system immanent adhesives XP Bond, Xeno V, Syntac, Adper Prompt L-Pop, and iBond self-etch. Before and after thermomechanical loading (100,000 × 50 N, 2500 thermocycles between 5 and 55 °C), marginal gaps were analysed using SEM of epoxy resin replicas. Results were analysed with Kruskal–Wallis and Mann–Whitney U-tests ($p < 0.05$). After thermomechanical loading, specimens were cut longitudinally in order to investigate internal dentine adaptation by epoxy replicas under a SEM (200× magnification).

Results: In enamel, high percentages of gap-free margins were initially identified for all adhesives. After TML, etch-and-rinse adhesives performed better than self-etch adhesives ($p < 0.05$). Also in dentine, initially high percentages of gap-free margins were found for all adhesives. After TML, etch-and-rinse adhesives again performed better than self-etch adhesives for both marginal and internal adaptation ($p < 0.05$). The presence of a 4 mm layer of SDR had no negative influence on results in any group ($p > 0.05$).

Conclusions: SDR as 4 mm bulk fill dentine replacement showed a good performance with the material combinations under investigation.

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1. Introduction

Long-term adhesion of bonded dental biomaterials to tooth hard tissues is an important factor for clinical success at least with materials shrinking on polymerization.^{1–3} When bonding performance is inferior, it is possible that both

initial and residual polymerization stresses lead to gap formation, leakage, recurrent caries, pulpal irritation and maybe retention loss.^{2,4–8} Therefore a tight marginal seal still has to be the primary goal for the clinician, because once happened, gap formation cannot be counteracted with restorative materials that prevent demineralization along with cavity margins.^{9,10}

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Despite many new and innovative developments in the field of adhesives, a 100% perfect margin is not realistically achievable.^{6,11-15} Nevertheless, conventional multi-step adhesives such as OptiBond FL on the etch-and-rinse sector and Clearfil SE Bond on the self-etch sector were repeatedly called gold standards, providing clinically successful adhesion to enamel and dentine.^{1,3,7,14,16,17} On the other hand, many of the more modern self-etch adhesives performed worse in vitro and in vivo.^{15,18-20} Although this is proven in a variety of preclinical and clinical investigations, simplification still acts as strong merchandising argument and all-in-one adhesives could get considerable portion of the adhesive market all over the world. Finally, a certain amount of technique sensitivity is always involved in the use of adhesive systems of all kinds.^{5,16}

In contrast to adhesives, simplifications in resin composite materials have been less frequently reported during the last decade in adhesive dentistry. There was definitely improvement in the field of polymerization shrinkage and wear resistance,^{4,21,22} however, a meticulous incremental layering technique is still mandatory to meet the above mentioned prerequisites for effective sealing of margins of resin composite restorations.^{1,7,12,23,24} SDR or Surefil SDR (Dentsply, Konstanz, Germany) was introduced to the market as flowable resin composite claiming that it would allow a 4 mm bulk placement in one layer due to reduced polymerization stress,²⁵ being mandatorily covered by a 2 mm layer of conventional resin composite.²¹ Although flowable resin composite materials have been repeatedly discussed to act as stress breakers or adaptation promoters,²⁶ clinical investigations could not confirm this issue so far.^{4,24,27} Up to now, there is only one study describing relevant parameters for SDR, however, polymerization stress was reported to be considerably lower than for conventional flowable materials.²⁵

Therefore, the objective of the present study was to compare the novel flowable low-shrinkage-stress resin composite SDR in a Class II fatigue loading design when used as 4 mm dentine replacement compared to conventionally layered resin composite restorations being bonded with different approaches. Evaluation of marginal adaptation to

enamel and dentine and internal adaptation to dentine was performed with examination of epoxy resin replicas of the restorative margins by scanning electron microscopy (SEM).

The null hypothesis tested was twofold, (1) that the different adhesives under investigation would not exhibit significant differences regarding adhesive performance being displayed as percentage of gap-free margins to enamel and dentine and (2) that there would be no differences in marginal and internal integrity of either enamel or dentine margins in Class II cavities that were restored either with or without SDR as first 4 mm increment within the different restorative approaches.

2. Materials and methods

Eighty intact, non-carious, unrestored human third molars, extracted for therapeutic reasons under informed consent of the patients and with a votum of an ethics committee (University of Giessen, Germany), were stored in an aqueous solution of 0.5% chloramine T at 4 °C for up to 30 days. The teeth were debrided of residual plaque and calculus, and examined to ensure that they were free of defects under a light microscope at $\times 20$ magnification.

Eighty standardised Class II cavity preparations (MOD, 4 mm in width bucco-lingually, 4 mm in depth occlusally, and 2 mm in medio-distal direction at the bottom of the proximal box) with the distal proximal margin located 1-2 mm below the cemento-enamel junction were performed. The cavities were cut using coarse diamond burs under profuse water cooling (80 μ m diamond, Komet, Lemgo, Germany), and finished with a 25 μ m finishing diamond (one pair of diamonds per four cavities). Inner angles of the cavities were rounded and the margins were not bevelled to deliver comparable results to previous experiments. Prepared cavities were treated with different classes of dentine adhesives according to the manufacturers' instructions (XP Bond, Xeno V, Syntac, Adper Prompt L-Pop, and iBond self-etch). Eight teeth were randomly selected for each adhesive (Table 1). The dentine

Table 1 – Adhesives under investigation.

Adhesive	Components	Manufacturer
XP Bond	Etchant: 36% phosphoric acid (0506000765) Primer/Bond: TCB resin, PENTA, UDMA, TEGDMA, BHT, CQ, amorphous silica (0503004020), mixed with SCA (self-cure activator 041203)	Dentsply DeTrey, Konstanz, Germany
Xeno V	Bifunctional acrylic amides, acryloamido alcylysulfonic acid, functionalized phosphoric acid ester, acrylic acid, camphorquinone, butylated benzenediol, water, tert-butanol, photoinitiators, stabilizers (0809001936)	
Syntac	Etchant: 35% phosphoric acid (G07234) Primer: Maleic acid 4%, TEGDMA, water, acetone (G19483) Adhesive: Water, PEGDMA, glutaraldehyde (J0076) Heliobond: bisGMA, UDMA, TEGDMA (H14837)	Ivoclar Vivadent, Schaan, Principality of Liechtenstein
Prompt L-Pop	Blister A: Methacrylated phosphates, photoinitiator, stabilizer (367978) Blister B: Water, complexed fluorides, stabilizer (367978)	3M Espe, Seefeld, Germany
iBond SE	UDMA, 4-Meta, glutaraldehyde, acetone, water, photoinitiators, stabilizers (010076)	Heraeus Kulzer, Hanau, Germany

adhesives and resin composite were polymerised with a Translux CL light-curing unit (Heraeus Kulzer, Hanau, Germany). The intensity of the light was checked periodically with a radiometer (Demetron Research Corp, Danbury, CT, USA) to ensure that 650 mW/cm² was always delivered during the experiments. The adhesive was polymerized for 40 s prior to application of the resin composite in all cases.¹⁴ The resin composite SDR (Dentsply DeTrey, Konstanz, Germany) was used for all experimental restorations, being combined with resin composites of the same product lines of manufacturers, i.e., XP Bond and Xeno V with CeramX mono+ (Lot 0903000338), Syntac + Tetric EvoCeram (L56579), Adper Prompt L-Pop + Filtek Supreme XT (20090402), iBond self-etch + Venus Diamond (010025).

Forty cavities were surrounded with a metal matrix band, bonded with the respective adhesives, and restored with the SDR resin composite in a first 4 mm layer and the respective resin composite for the residual height of the cavity in increments of 2 mm thickness. Forty cavities were restored with the high viscosity resin composites in a conventional horizontal layering technique of 2 mm thickness. The increments were separately light-cured for 40 s each with the light source in contact with the coronal edge of the matrix band. After removal of the matrix band, the restorations were light-cured from their buccal and lingual aspects for an additional 20 s on each side. Prior to the finishing process, visible overhangs were removed using a posterior scaler (A8 S204S, Hu-Friedy, Leimen, Germany). Proximal margins were finished with flexible disks (SofLex Pop-on, 3 M ESPE, St. Paul, USA).¹⁴ All restorations were performed by one operator.

After storage in distilled water at 37 °C for 21 days, impressions (Provil Novo, Heraeus Kulzer, Hanau, Germany) of the teeth were taken and a first set of epoxy resin replicas (Alpha Die, Schuetz Dental, Rosbach, Germany) was made for SEM evaluation.

Thermo-mechanical loading of specimens was then performed in an artificial oral environment (“Quasimodo” chewing simulator, University of Erlangen, Germany). Two specimens were arranged in one simulator chamber and occluded against a steatite (a multi-component semi-porous crystalline ceramic material) antagonist (6 mm in diameter) hitting two lateral ridges of restorations for 100,000 cycles at 50 N at a frequency of 0.5 Hz. The specimens were simultaneously subjected to 2500 thermal cycles between +5 °C and +55 °C by filling the chambers with water in each temperature for 30 s. The mechanical action and the water temperature within the chewing chambers were checked periodically to ensure a reliable thermo-mechanical loading (TML) effect.

After completion of the 100,000 mechanical loading and the 2500 thermal cycles, impressions of the teeth were made again and another set of replica was made for each restoration. The replicas were mounted on aluminium stubs, sputter-coated with gold and examined under a SEM (Phenom, FEI, Amsterdam, The Netherlands) as before at ×200 magnification.

SEM examination was performed by one operator having experience with quantitative margin analysis who was blinded to the restorative procedures. The marginal integrity between resin composite and dentine was expressed as a percentage of the entire margin length in enamel and dentine. Marginal qualities were classified according to the criteria

“gap-free margin”, “gap/irregularity” and “not judgeable/artefact”. Afterwards the percentage “gap-free margin” in relation to the individual judgeable margin was calculated as marginal integrity. After having been investigated marginally, previously loaded teeth were embedded in a slow self-curing epoxy resin material (Epoxy-Die, Ivoclar, Schaan, Liechtenstein) and sectioned mesio-distally with one central cut using a slow-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). Both sections were polished (600# SiC paper) and etched for 2 min using a 36% phosphoric acid etching gel (Total Etch, Vivadent), rinsed and dried. Impressions were taken from the two surfaces using a high (Permagum, Espe, Seefeld) and a low viscosity (Permagum Garant) impression material and both internal surfaces were investigated. Drying and impression processes were carried out carefully to limit dentine dehydration.

Statistical analysis was performed using SPSS/PC+, Version 12 (SPSS Inc., Chicago, IL, USA) for Windows. As the majority of groups in each of the two investigations (i.e., enamel or dentine marginal integrity) did not exhibit normal data distribution (Kolmogorov-Smirnov test), non-parametric tests were used (Kruskal-Wallis test, Wilcoxon matched-pairs signed-ranks test, Mann-Whitney-U test) for pairwise comparisons at the 95% significance level.

3. Results

The results of the present investigation are displayed in Table 2. In enamel, high percentages of gap-free margins were initially identified for all adhesives, providing amounts of gap-free margins of 89.9–100%.

After TML, etch-and-rinse adhesives performed better (87.4–91.5%) than self-etch adhesives (42.1–64.1%) ($p < 0.05$). Also in dentine, initially high percentages of gap-free margins were found for all adhesives (92.2–100%). After TML, etch-and-rinse adhesives (63.2–66.6%) again performed better than self-etch adhesives (38.5–56.2%) for marginal adaptation ($p < 0.05$) (Table 2). During the marginal quality evaluation, paramarginal enamel fractures occurred only in groups bonded with etch-and-rinse adhesives with slightly more of these fractures having been observed with SDR ($p > 0.05$). Not judgeable margins were <2% in all cases.

Also for internal adaptation to dentine, etch-and-rinse adhesives (66.3–70.1%) exhibited significantly more areas of gap-free transition compared to self-etch adhesive systems (31.0–57.0%) ($p < 0.05$). For all adhesives under investigation, the presence of a 4 mm layer of SDR had no negative influence on results ($p > 0.05$) (Table 2).

4. Discussion

The results of this study confirmed previous investigations showing that conventional phosphoric acid-etching remains the most reliable mode of pre-treatment to achieve fatigue-resistant enamel bonds.^{3,8,14,15,17} Although the self-etch adhesives under investigation performed well with cut enamel prior to functional and thermal stresses, this particular adhesive approach was significantly less effective after

Table 2 – Overview of results. Same superscript letters within columns mean $p > 0.05$. CXm, CeramX mono+; TEC, Tetric EvoCeram; FSXT, Filtek Supreme XT; VD, Venus Diamond.

Adhesive	Resin composite	Enamel initial	Enamel (TML)	Dentine initial	Dentine (TML)	Internal adaptation (TML)
XP Bond	SDR + CXm	100 ^a	87.4 (7.0) ^a	100 ^a	64.9 (8.7) ^a	70.1 (12.0) ^a
	CXm	100 ^a	90.3 (6.7) ^a	100 ^a	63.2 (7.6) ^a	66.3 (9.9) ^a
Xeno V	SDR + CXm	96.8 (3.9) ^b	62.4 (10.1) ^b	100 ^a	50.3 (7.4) ^b	56.2 (10.4) ^b
	CXm	100 ^a	64.1 (9.6) ^b	100 ^a	52.5 (7.0) ^b	58.9 (9.4) ^b
Syntac	SDR + TEC	100 ^a	91.5 (3.2) ^a	100 ^a	66.6 (7.9) ^a	73.8 (9.4) ^a
	TEC	100 ^a	90.2 (2.1) ^a	100 ^a	64.3 (7.0) ^a	69.4 (11.9) ^a
Prompt L-Pop	SDR + FSXT	100 ^a	59.9 (10.4) ^b	94.3 (4.5) ^b	38.5 (11.1) ^c	36.2 (14.3) ^c
	FSXT	100 ^a	63.2 (11.0) ^b	92.2 (3.4) ^b	41.6 (10.0) ^c	31.0 (12.0) ^c
iBond SE	SDR + VD	90.3 (4.5) ^c	46.2 (10.4) ^c	100 ^a	56.2 (6.9) ^b	54.6 (12.3) ^b
	VD	89.9 (3.9) ^c	42.1 (9.7) ^c	100 ^a	55.1 (5.8) ^b	57.0 (9.8) ^b

fatigue testing (Table 2). It is known that self-etching systems provide a network of intercrystallite retention leading to a large surface for bonding. It was shown that this type of enamel bond was able to initially compensate polymerization shrinkage stresses, however, after thorough fatigue testing marginal quality of these interfaces exhibited more gaps than margins obtained under the etch-and-rinse approach. As a consequence, null hypothesis (1) had to be rejected.

The present study demonstrated that it is possible to successfully work with a simplified application procedure with a 4 mm base layer as open sandwich, which as possible in all specimens with boxes deeper than 6 mm. In none of the groups SDR caused less gap-free margins compared to the control groups using incrementally layered resin composites (Table 2). The basal part of the cavity is just filled with the flowable resin composite and consecutively covered with a more wear resistant conventional resin composite. The present study showed that the combination of SDR with the chosen five resin composites apparently shows no lack of adhesive performance in terms of marginal quality to enamel and dentine as well as internal dentine adaptation. Regarding internal dentine adaptation for SDR vs. conventionally layered resin composite, there was no difference even when both materials were part of the measured internal adaptation. Therefore, the null second hypothesis had to be confirmed.

In this study, marginal quality of a novel approach in resin-based composites was evaluated with the use of a thermo-mechanical loading scenario.¹¹⁻¹⁴ Although clinical trials remain the gold standard in evaluating the performance of dental materials,^{2,4-9,17,22,28} it has to be taken into account that individual products under investigation maybe not up to date anymore once useful clinical data are collected.^{8,24} This situation is deteriorated by the backlog between the point when clinical results are obtained and having them published in peer-reviewed journals. Thus, preclinical screening via laboratory tests is still an important tool for the evaluation of dentine adhesives and it allows to evaluate much more experimental groups compared to clinical studies where the number of variables has to be kept small.^{3,29}

There are several approaches to predict clinical behaviour of dental biomaterials in the laboratory. Bond strength tests are commonly carried out with quasistatic load until fracture.^{13,19,30} However, failure of clinical restorations due to high loads is exceptional incidents being primarily observed with nonvital teeth. Under normal clinical conditions with vital

teeth being in the focus, materials or interfaces fail after repeated sub-catastrophic loading with stresses being too small to provoke spontaneous failures.^{6,13,23,29} As a result after months and years of clinical service, the most frequent observation is gap formation between resin composite and tooth substrates.^{1,3,4,7,9,29}

Gap formation may occur at the margins in enamel and dentine as well as along the resin-dentine interface as loss of internal adaptation.²⁶ These gaps result from either insufficient compensation for initial polymerization shrinkage stresses occurring prior to the first occlusal loading, or from lower, repeated stresses which are below the maximum stress the adhesive restoration can resist.^{3,15,16} As a consequence, in vitro fatigue tests provide a better understanding of the in vivo behaviour of adhesives.^{6,22}

Limitations of the present results are related to the experimental setup. First of all, in the control groups with conventionally layered resin composite restorations the resin composites have been applied in a simplified horizontal incremental technique. Horizontal layering in the proximal box as carried out during the present study has the advantage to be easy to perform compared to more sophisticated layering regimens having been described in the literature of the field.¹² On the other hand, horizontal layering was described to result in unfavourable configuration factors of the individual increment, being added up to the top of the proximal box resin composite modulation. Due to the fact, that SDR is also applied in a 4 mm horizontal increment, it may be one argument for the results of the present study after thermomechanical loading. It is possible that using a more intelligent and c-factor-aware layering technique in the control groups may have produced different results, more in favour of the control groups where no SDR was used. Another limitation is that not all adhesives on the market could be covered, however, we chose characteristic versions of all kinds of adhesives.

Clear advantage of the present methodology was that the adhesives under investigation have been evaluated with both possible resin composites, one of the same manufacturer and additionally SDR from a different manufacturer.¹¹⁻¹⁴ So there is no remaining unanswered question whether the resin composite or the adhesive was evaluated. At the end, no incompatibilities between adhesives or resin composites and the SDR flowable resin composite were found. So even facing the potential shortcomings of the present investigation, the results confirm the conclusion.

Depending on cavity size and presence of bevelled margins, paramarginal enamel fractures are observed in this kind of preclinical investigations.^{12,23} Due to the large cavity size in the present preparation and restoration setup, this kind of enamel fractures did not play an important role during the investigation, however, they were found to be slightly more frequent compared to the conventional resin composite groups.

Altogether, the present study reports no detrimental effect of a 4 mm base layer of SDR compared to two separate layers of rein composite. The marketing term "bulk fill" was clearly not investigated here, because bulk fill was simply not done.

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