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ORIGINAL ARTICLE

Microleakage of silorane- and methacrylate-based class V composite restorations

Stephanie Krifka · Marianne Federlin · Karl-Anton Hiller · Gottfried Schmalz

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Abstract The marginal integrity of class V restorations in a silorane- and a group of methacrylate-based composite resins with varying viscosities was tested in the present study. Different adhesives (OptiBond FL, KerrHawe; AdheSE One, Vivadent; or Silorane System Adhesive, 3M ESPE) were applied to168 standardized class V cavities. The cavities (n=12) were filled with a wide range of different viscous composite resins: Filtek Silorane, 3M ESPE; els and els flow, Saremco; Tetric EvoCeram and Tetric EvoFlow, Vivadent; Grandio, Voco; and Ultraseal XT Plus, Ultradent. Microleakage of the restoration was assessed by dye penetration (silver staining) on multiple sections with and without thermocycling and mechanical loading (TCML: 5,000×5–55°C; 30 s/cycle; 500,000×72.5 N, 1.6 Hz). Data were statistically analyzed with the Mann–Whitney U test and the Error Rates Method (ERM). The silorane-based composite resin yielded the lowest dye penetration after TCML. Microleakage of methacrylate-based composite restorations, in general (ERM), was statistically significantly influenced by the adhesive system, Moreover, dye penetration at enamel margins was significantly lower than dye penetration at dentin margins. The chemical basis of composite resins and adjacent tooth substance seems to strongly influence marginal sealing of class V restorations for methacrylate-based materials. Moreover, the steps of dental adhesives used affected marginal integrity. The silorane-based composite resin evaluated in the present study exhibits the best marginal seal. The three-step adhesive yielded better marginal sealing than the one-step adhesive for methacrylate-based class V composite restorations.

Keywords Microleakage · Class V restoration · Adhesive · Composite resin · Enamel · Dentin

Introduction

Recurrent caries, marginal discoloration, or postoperative sensitivity may negatively affect the clinical performance of methacrylate-based adhesively bonded composite resin restorations. The three cardinal compounds of methacrylate-based composite resins are inorganic filler particles (glass, quartz), organic matrix (base monomers, pigments, photoinitioators, stabilizers), and the coupling agent (silane) to bond the inorganic filler particles to the organic matrix [1]. During the process of polymerization, volumetric shrinkage of up to 5% [2] occurs, as radicals react with methacrylate groups of the monomers to form a cross-linked network [3] resulting in a reduction of the intermolecular distance of the free monomer molecules (0.3–0.4 nm) to the polymerized molecules (0.154 nm); i.e., exchanging van der Waals spaces for shorter covalent bond spaces [4], contraction stresses consequently develop in the resin [5]. These internal stresses are transferred to the tooth/restoration interface as tensile forces, because the composite resin shrinks towards the bonded surface, but is constrained by the rest of its mass, which is bonded to the opposing surface [6]. Clinical problems are at least partially related with polymerization shrinkage [7] and thus, low-shrinking matrix resins were developed to prevent or to reduce microleakage by introducing a completely new chemical basis (combination of

S. Krifka (✉) · M. Federlin · K.-A. Hiller · G. Schmalz
Department of Operative Dentistry and Periodontology,
University of Regensburg Medical Centre,
Franz Josef Strauß Allee 11,
93042 Regensburg, Germany
e-mail: Stephanie.Krifka@klinik.uni-regensburg.de

siloxanes and oxiranes [8]). The volumetric shrinkage of the silorane-based composite resin is <1% [9] because these monomers connect by opening, flattening, and extending toward each other during the "ring-opening" cationic polymerization. Volumetric shrinkage and shrinkage stress [10] of silorane-based composite resins are comparatively low. However, when compared with methacrylate-based composite resins, similar mechanical properties [11, 12] and a reduction in bacterial adhesion [13] were demonstrated. Some authors reported on higher flexural strength, fracture toughness [1], chromatic stability [14], the lack of cytotoxic effects [15], or no water solubility [16], but less compressive strength, microhardness [1], and translucency [14] were observed. Although a considerable amount of performance data are available, information for silorane-based materials on microleakage is limited.

The material characteristics of composite resin restorations depend on various factors, such as amount and type of matrix resin [16], curing chemistry [17], initiator level [18], and the addition of non-bonded microfiller particles [19]. Due to 20–25% less filler content, inferior material properties (e.g., low fracture strength [20], less rigidity [21], and high polymerization shrinkage [22]) are observed for flowable composite resins than for conventional composite resins. Therefore, its application as sealant, liner, and filling material in small cavities or non-occlusal surfaces is advocated [23, 24]. Information comparing a wide range of methacrylate-based flowable and conventional composite resins is rare for class V restorations. Most reports are based on basic mechanical testing, gap formation, and microleakage, without taking the simulation of the clinical situation into account. Here, flowables are mostly used as liners for larger composite resin restorations.

Nonmaterial-related factors, like cavity configuration [25, 26], application technique [27], and curing method [28, 29], also influence the marginal sealing ability or material's properties. Bonding systems are generally used to reduce or to prevent microleakage [5, 8]. Depending on how the three steps (etching, priming, and bonding) are combined, numerous dental adhesives are available today for adhesively bonding composite resins to the tooth. The suitability of adhesive systems is related to the resulting bond strength between the composite resin and the different dental hard tissues (dentin and enamel). Although bond strength data reported for special products depend on the experimental conditions or the method used, general trends for the different adhesive systems can be noted: Lower bond strength for enamel and dentin has been reported for one-step self-etch adhesives when compared to two-step and three-step adhesives [30]. The literature presents inconsistent results regarding the bond strength of dental adhesives necessary to provide retention and to prevent microleakage. Some authors found no correlation between bond strength and microleakage [31]. Other authors suggested that an in vitro bond strength of approximately 20 MPa as sufficient to avoid marginal leakage of class V cavities [32]. However, no systematic studies about microleakage are available in the literature for a group of methacrylate-based flowable and conventional composite resins using different adhesives and a silorane-based composite resin.

This study hypothesized that artificial aging as well as viscosity (flowables vs. conventional complements) and not the chemical basis impair marginal sealing of class V restorations. Furthermore, it was hypothesized that one-step and three-step adhesives equally prevent microleakage of methacrylate-based class V restorations. Therefore, microleakage of adhesively bonded composite resin restorations in standardized class V cavities under the same application conditions was tested. A silorane-based composite system and diverse methacrylate-based composite resins and two adhesive systems (etch&rinse adhesive and self-etch adhesive) were used for this purpose. Clinical use was simulated by simultaneous thermal and mechanical loading.

Methods and materials

Specimen preparation

One hundred sixty-eight extracted molars, which had been stored in 0.5% chloramine solution, were cleaned, the apices sealed with gutta-percha, and mounted in Pattern Resin (GC Corporation, Tokyo, Japan) up to 3 mm below the cementoenamel junction (CEJ). The teeth were then stored in distilled water (4°C) for a maximum of 1 week until use. Bullet-shaped diamond burs with a diameter of 2.3 mm and the matching diamond finishing burs (46 µm particle size) (Brasseler, Lemgo, Germany) in a high-speed handpiece with sufficient water cooling were used to cut a buccal class V cavity preparations on each tooth with the cavity margins being located, in both dentin and enamel. Preparations were about 5.0 mm in the mesiodistal and 3.0 mm in the occluso-cervical directions. The depth of the cavity was about 1.5 mm and the gingival cavosurface margins were placed 1.5 mm below the CEJ. The enamel surface was chamfered 1.0 mm using flame diamond finishing burs (Brasseler, Lemgo, Germany). Finally, the cavity size was checked using a periodontal probe (PCP UNC 127, Hu Friedy Mfg. Co.Inc., Chicago, IL, USA). The teeth were randomly assigned to 14 groups of 12 teeth each.

Material application

One silorane-based composite resin [Filtek Silorane (SI)] and six different viscous methacrylate-based composite resins [els (EL) and els flow (EF), Tetric EvoCeram (TC) and Tetric EvoFlow (TF), Grandio (GR), and Ultraseal XT Plus (US)] were inserted according to the manufacturers' instructions using two different adhesives (OptiBond FL and AdheSE One) with the methacrylate-based composite resins and Silorane System Adhesive with the silorane-based composite resin as shown in Table 1. The composite resins were placed in one increment and light cured for 40 s (3M ESPE Elipar Trilight, Seefeld, Germany) with an intensity of 750 mW/cm^2.

After light curing, the restorations were finished with Komet finishing diamonds (Brasseler, Lemgo, Germany) and polished with Sof-Lex flexible disks (3M, St. Paul, MN, USA). The samples were then stored in physiological saline solution at 37°C for 24 h.

Thermocycling and mechanical loading (TCML)

The teeth of half of the groups were exposed to thermocycling (5,000×5°C and 55°C, 30 s/cycle) and central mechanical loading (500,000×72.5 N at 1.6 Hz) simultaneously. To perform the mechanical loading, a stainless steel stop was used to represent the opposing cusp. The stainless steel stop was placed in the occlusal central fissure of the tooth.

Dye penetration

Before and after TCML, microleakage was determined by means of dye penetration. Except for the areas within 1.0 mm from restoration margins, specimens were covered with nail varnish and placed in 50 wt.% silver nitrate ($AgNO_3$) aqueous solution (S-6506: Sigma-Aldrich Chemie GmbH, Steinheim, Germany) at room temperature for 2 h in the dark. Then, the teeth were placed in a photo-developing solution/fluorescent light (Tetenal Ultrafin Plus, Tetenal AG & Co. KG, Norderstedt, Germany) for 6 h. After dye penetration, the specimens were cleaned, mounted onto stubs with acrylic resin, and consequently sectioned longitudinally in the vestibulo-oral direction (Fig. 1) into as many as possible, approximately 300 μm thick sections (up to 12 sections), using a rotating diamond saw (blade thickness, 300 μm) (Innenlochsäge Leitz 1600, Leitz, Wetzlar, Germany) with sufficient water cooling. Each section provided two sites for the evaluation of dye penetration. Digital images of the sections were used to measure microleakage, which was recorded using an image analyzing system (Optimas 6.1, Stemmer, Munich, Germany).

Three dye penetration measurements per section (rendering up to 72 measurements per tooth) were recorded along the restoration: i.e., the length of dye penetration at enamel (l_e) and dentin (l_d) margins as well as the entire length of the restoration (l_r). Total dye penetration (DP_t) was expressed as percentage of the entire length of the restoration (100% reference):

$$DP_t = \frac{l_e + l_d}{l_r}$$

The extent of dye penetration was expressed separately for enamel (DP_e) and dentin (DP_d) as a percentage of the entire length of restoration:

$$DP_e = \frac{l_e + l_d}{l_r} \quad \text{and} \quad DP_d = \frac{l_e + l_d}{l_r}$$

The maximum value of these measurements was selected for each tooth and used for further statistical analysis of the tooth as a statistical unit. Only the results of the total dye penetration (DP_t) were put into graphs, whereas the results of individual dye penetration of enamel (DP_e) and dentin (DP_d) without/with TCML are shown in Table 2.

Table 1 Composite resins and adhesives

Composite resin	Adhesives	
Methacrylate based Els (EL), Saremco (#07.2011-38) Tetric EvoCeram (TC), Ivoclar Vivadent (#J23963) Grandio (GR), Voco (#711044) Els flow (EF), Saremco (#07.2011-06) Tetric EvoFlow (TF), Ivoclar Vivadent (#J18892) Ultraseal XT Plus (US), Ultradent (#B2MSV)	OptiBond FL, KerrHawe (#2722728), etch&rinse three-step adhesive; shear bond strength[a]: enamel, 26.7 MPa; dentin, 45.6 MPa	AdheSE One, Ivoclar Vivadent (#K14344), self-etch one-step adhesive; shear bond strength[b]: enamel, 20 MPa; dentin, 20 MPa
Silorane based Filtek Silorane (SI), 3M ESPE (#4771A2)	Silorane System Adhesive, 3M ESPE (self-etch primer #20071222, bond #20071226) self-etch two-step adhesive; shear bond strength: enamel, 20.2 MPa[b], dentin, 21.5 MPa[c]	

[a] Data from reference [40]

[b] Manufacturer information

[c] Data from reference [42]

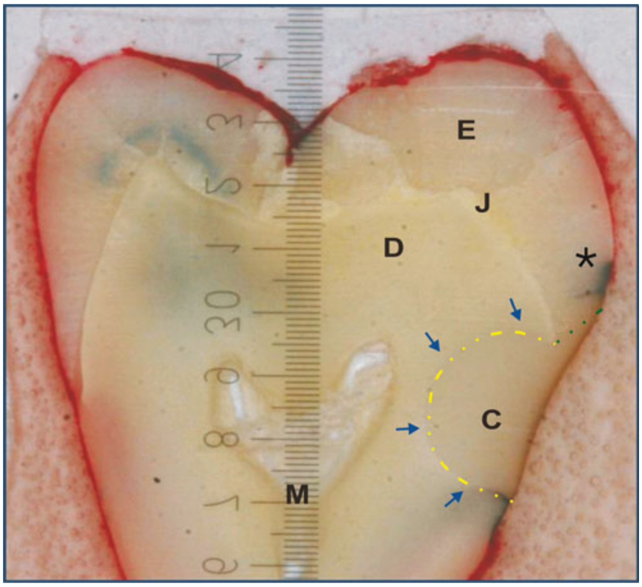

Fig. 1 Example of tooth section: *E* enamel, *D* dentin, *J* dentin–enamel junction, *M* measure, *C* composite resin; *Arrows* indicate class V restoration; *green dotted line* indicates the entire length of enamel (l_e); *yellow dotted line* indicates the entire length of dentin (l_d); *asterisk* artifact

Statistical analysis

Medians and 25% and 75% quartiles were determined from 12 replications of each experimental group and pairwise comparisons between groups were performed using the Mann–Whitney *U* test for independent and the Wilcoxon rank sum test for paired samples (SPSS version 19, SPSS Inc, Chicago, IL, USA) at the α=0.05 level of significance. For evaluating the influence of the adhesives in general, the level of significance was adjusted to $\alpha^*(k)=1-(1-\alpha)1/k$ by applying the error rates method (ERM), where *k* denotes the number of pairwise tests to be performed.

Results

The results of dye penetration for one silorane-based composite resin and six methacrylate-based composite resins (flowable/conventional) in combination with two different adhesives for both situations, without and with TCML, are shown in Fig. 2 (DP_t). The present study focused on the whole restoration with the “worst case scenario” for marginal sealing. However, results of individual tooth substances, enamel, and dentin are included (Table 2; DP_e and DP_d). Dye penetration of silorane- and methacrylate-based composite resins without TCML (ranging from 9% to 70%) was generally lower than with TCML (ranging from 23% to 100%). This can be substantiated by statistical analysis showing a significant difference in dye penetration between the groups with and without TCML (ERM, p=0.000). However, in three materials (GR, TC, and US) TCML had no significant influence upon total dye penetration (pairwise comparison).

The three-step adhesive generally led to less dye penetration than the one-step adhesive when used together with the methacrylate-based composite resin. This could be substantiated by statistical analysis (ERM, p=0.001).

Table 2 Results of the dye penetration of flowable and conventional composite resins without and with TCML at the enamel/restoration interface (DP_e) and the dentin/restoration interface (DP_d) (median and 25–75% quartiles)

		Without TCML									With TCML								
A		EF			TC			US			EF			TC			US		
		50	25	75	50	25	75	50	25	75	50	25	75	50	25	75	50	25	75
OptiBond FL	Enamel	4.7	3.5	8.0	0.0	0.0	0.0	7.0	5.6	8.9	8.4	6.0	11.9	0.0	0.0	7.0	8.4	6.0	11.9
	Dentin	22.6	14.7	41.6	27.9	24.5	33.8	25.4	15.4	34.3	23.7	14.8	36.6	59.8	51.4	70.8	23.7	14.8	36.6
AdheSE One	Enamel	16.0	12.0	20.1	5.4	0.7	10.8	9.1	4.7	14.6	22.4	20.3	26.2	17.5	14.6	22.1	20.4	17.3	24.0
	Dentin	53.1	22.2	60.7	7.5	5.8	9.3	29.1	12.3	55.3	79.7	65.5	85.0	52.3	27.6	80.0	31.0	15.8	61.1
B		EL			TC			GR			EL			TC			GR		
		50	25	75	50	25	75	50	25	75	50	25	75	50	25	75	50	25	75
OptiBond FL	Enamel	3.7	0.5	5.4	1.3	0.0	4.8	1.7	0.0	5.4	0.0	0.0	4.2	3.3	2.2	5.6	0.0	0.0	4.2
	Dentin	26.5	17.1	30.6	24.9	18.8	69.0	18.1	15.1	22.8	22.4	13.5	37.6	57.3	25.6	70.9	22.4	13.5	37.6
AdheSE One	Enamel	7.3	4.4	10.0	16.6	14.4	23.6	4.8	1.0	9.4	22.7	18.0	25.6	22.7	18.2	26.0	22.7	18.0	25.6
	Dentin	68.1	38.6	73.5	53.1	38.0	62.7	15.1	11.4	17.8	70.5	61.2	76.7	66.2	59.0	71.3	25.2	7.0	44.6
C		SI									SI								
		50			50			25			75			25			75		
Silorane System Adhesive	Enamel	4.7			4.2			7.4			8.2			7.5			14.0		
	Dentin	5.3			4.6			7.6			5.8			4.8			8.2		

A flowable methacrylate-based composite resins, *B* conventional methacrylate-based composite resins, *C* silorane-based composite resin

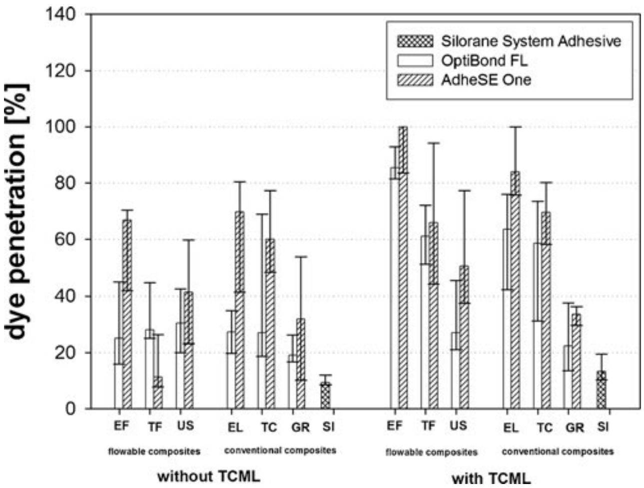

Fig. 2 Results of the total dye penetration test (DP_t) of flowable and conventional composite resins without and with TCML at the tooth/composite resin interface (median and 25–75% quartiles). Test materials are arranged by flowable and conventional composite resins

Although the difference of total dye penetration between both adhesives without and with TCML (Fig. 2) is equal to the individual dye penetration of enamel and dentin (Table 2), the three-step adhesive produced statistically significant less dye penetration at the enamel and dentin margin than the one-step adhesive (ERM, $p=0.000$). Furthermore, statistical analysis (ERM) revealed a significant influence of the composite materials on dye penetration. The silorane-based composite resin (SI) showed significantly less dye penetration (without TCML, 9%; with TCML, 13%) than all methacrylate-based composite resins tested; e.g., after TCML, the methacrylate-based composite resins US (27–50%) and GR (22–30%) showed less dye penetration than TF (61–65%), TC (59–70%), and EF (85–100%). Flowable methacrylate-based composite resins did not show more dye penetration than conventional methacrylate-based composite resins. When used together with the three-step adhesive, dye penetration of the flowables before TCML (ranging from 25% to 31%) was similar to dye penetration of the conventional composite resins (ranging from 19% to 27%). After TCML, the methacrylate-based flowables performed statistically equal to the conventional methacrylate-based composite resins; e.g., US (27%) versus GR (22%) and TF (61%) versus TC (59%).

When combining dye penetration of flowable composite resins and their conventional composite resin counterparts from the same manufacturer (TF vs. TC and EF vs. EL), without TCML, TF (11.4%) when used together with the one-step adhesive, produced significantly less dye penetration than TC (60.2%; $p=0.000$). With TCML, no statistically significant difference of dye penetration between the two materials (TF vs. TC) was observed. For EF vs. EL without TCML, no difference in dye penetration was found; however, a statistical significant difference was found with TCML, since less dye penetration was observed with EL (63.6%) when compared with EF (85.4%) for the three-step adhesive ($p=0.004$).

Discussion

Method

The clinical relevance of the method used in the present study is discussed in the literature. Heintze et al. [31] stated the clinical relevance of various in vitro tests, such as the evaluation of microleakage by dye penetration, as problematic. Different results of dye penetration in vitro seem to be affected by many factors [31] and various test methods [33]. Furthermore, the results of sparse comparative studies are varying [31] and a direct correlation between the results of dye penetration studies and the clinical outcome appears to be difficult. Therefore, clinical studies cannot be replaced by in vitro microleakage studies or used to solely predict clinical performance. However, dye penetration may provide an easy, fast, and commonly applied preclinical screening method [34–36] to compare diverse parameters. Many different in vitro techniques have been used, such as dyes, radioactive isotopes, bacteria, or scanning electron microscopy. In the current study, marginal sealing was evaluated using the silver staining technique because silver nitrate, enhanced by the addition of a photo developer and made visible by photo development in fluorescence light, presents as black staining [37] which is stable, resists disruption by coolant when sectioning the tooth, and allows for a unequivocal evaluation. Although clinical performance is the ultimate test method for the assessment of restorations, in vitro evaluations remain an important method for an initial screening [33].

To simulate in vivo conditions, TCML was used. On the one hand, thermocycling induces repetitive contraction or expansion stresses at the tooth–material interface resulting from the high thermal contraction or expansion coefficient of composite resins [38]. On the other hand, mechanical loading leads to a decrease in bonding performance because of fatigue at the adhesive interface [39].

Choice of test materials

The rationale for the selection of the current test materials was to have a large range of diverse methacrylate-based materials with different viscosities, which are frequently used for class V restorations. The silorane-based composite resin was chosen because of its new chemical formulation and it presents the lowest polymerization shrinkage values of the materials available on the market today.

Additionally, two currently used adhesive systems for methacrylate-based composite resins, representing different adhesion concepts and different values of bond strength, were evaluated. OptiBond FL represents the group of three-step, multi-bottle, or “etch&rinse” adhesives with bond strength values of 26.7 MPa for enamel and 45.6 MPa for dentin [40]. AdheSE One represents a single-step, strong self-etch adhesive (pH 1.5) with bond strength values of 20 MPa for enamel and 20 MPa for dentin (according to the manufacturer's information). Those adhesive systems were used with six flowable and conventional methacrylate-based composite resins. Combinations of adhesives with methacrylate-based composite resins of different manufacturer are a common practice [41]. Because of the extremely hydrophobic properties of the silorane-based composite resin, it may only be used with its corresponding adhesive system, which represents a two-step (self-etch primer (pH 2.7) and bond) methacrylate-based adhesive and bond strength values of 20.2 MPa (according to the manufacturer) for enamel and 21.5 MPa for dentin [42] (Table 1).

Results

The hypothesis of the present study had to be rejected in part, since Filtek Silorane, the silorane-based composite resin, produced the lowest values of dye penetration, which were statistically different from the other methacrylate-based composite resins. Only limited information is currently available regarding microleakage of this recently marketed composite resin in a clinical simulation experiment. Bagis et al. [35] compared the influence of different layering techniques upon marginal integrity of methacrylate- and silorane-based composite resins in wide MOD cavities in vitro. No dye penetration for Filtek Silorane restorations was observed. Schmidt et al. [43] did not find significant differences in marginal adaptation of the low-shrink silorane-based class II restoration when compared to methacrylate-based restorations in vivo.

The results of the present study indicate that TCML generally increases dye penetration and are in agreement with a large number of similar studies (e.g., [39, 44]). However, both adhesives did not equally prevent microleakage as the three-step adhesive revealed less dye penetration than the single-step adhesive. Furthermore, enamel margins exhibited higher marginal integrity than dentin margins. These results are in agreement with data from Cardoso et al. [45] on microleakage at enamel and dentin margins of class V cavities after thermocycling using one two-step (Etch & Prime 3.0/Degussa) and four one-step adhesives (Single Bond/3M ESPE, PQ1/Ultradent, Prime & Bond NT/Dentsply DeTrey and Experimental BEH/Dentsply DeTrey). This was also supported for cementum–dentin margins of class V cavities by Pilo et al. [46], who compared microleakage of three one-bottle (Single Bond/3M ESPE, One-Step/Bisco, and Solobond/KerrHawe) and three multistep (OptiBond FL/KerrHawe, All-Bond 2/Bisco, and Scotchbond MP/3M ESPE) adhesives after thermocycling and mechanical loading. Interestingly, the adhesive of the silorane-based composite resin exhibited the lowest dye penetration in the current study. This supports the idea that factors other than bond strength determine the amount of microleakage. Some authors have suggested no correlation between bond strength tests and marginal integrity [31, 47].

Due to the inferior material properties of flowable methacrylate-based composite resins, it may be speculated that marginal sealing in class V restorations is impaired; however, this cannot be supported by the results of the present study. Conventional composite resins generally did not show less dye penetration than flowable composite resins, not even from the same manufacturer. The data of the present study are in line with Ikeda et al. [48], who stated no differences in marginal integrity of small class I cavities (1-mm deep) filled with three composite resins (low filler and high filler loaded flowable, hybrid composite) using the bulk technique. This was also supported by Yazici et al. [49] who showed equal marginal behavior of different filled composite resins (Filtek Flow, Tetric Flow, Solitaire, Admira, and Z100) of class V cavities. However, those studies have been based on testing without simulating clinical conditions. Jang et al. [50] observed no significant differences in microleakage of flowable and packable composite resins without and with TCML. Kubo et al. [51] showed no significant differences in microleakage among the flowable composites resins, but less microleakage of the hybrid composites could be observed without and with thermocycling while mechanical loading did not impair marginal integrity of flowable composites resin restorations.

A consolidated view indicates that various material properties (e.g., thermal expansion, elasticity) seem to play a role in modifying the marginal sealing ability; nonmaterial-related factors (e.g., cavity configuration, application technique, and curing method) have to be taken into account as well. The physical properties of the material selected for a special study may be rather similar or rather different and thus may play a major or a minor role because all properties are not defined in these studies. This may explain diverging results reported in the evaluation. However, this also means that generally predicting microleakage based on some material properties is not possible for class V cavities.

Conclusion

Within the limitations of the present study, it can be concluded that microleakage could not be prevented entirely. The silorane-based composite resin had the lowest dye penetration, but neither flowable nor conventional methacrylate-based composite resins provided an indication of better sealing abilities. However, dental adhesives significantly influence marginal adaptation of methacrylate-based composite resins in class V cavities.

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

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