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Bond strength and marginal adaptation of resin composites and correlations with clinical results

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ABSTRACT

Objectives: Due to innumerable confounding factors and a high number of types and brands of dental restorative materials, the clinical performance of restorative materials are sought predicted by various in vitro tests. However, only few such tests have been found to correlate well with clinical findings. Thus, the present study determined the in vitro dentin bond strength and marginal adaptation of Class II restorations and correlated the results to their clinical outcomes.

Methods: Dentin bond strength (μTBS and μSBS) and marginal gap formation of Class II restorations (replica technique and SEM) were measured after 24 h and 6 m water storage using eight combinations of adhesive and resin composite. Clinical outcomes (mean survival time, Hazard Ratio, annual failure rate; n = 10.695) were gained from a data set of a retrospective multicenter study of direct restorations.

Results: Significant differences were found for dentin bond strength and marginal gap formation between the restorative material groups, and negative effects of long-term storage were observed. μTBS correlated significantly with certain clinical outcomes of Class I restorations, while μSBS correlated with certain clinical outcomes of Class II, III, IV and V restorations. Marginal gap formation in enamel and number of paramarginal fractures correlated with certain clinical outcomes of Class II restorations.

Significance: Using the same restorative materials in vitro as in vivo, gave significant, but weak correlations between in vitro bond strength or marginal adaptation and clinical outcomes, lending support to the use of in vitro tests in early stages of material selection.

1. Introduction

Today, resin composite is the most used restorative material in dentistry and is the material of choice due to its good aesthetic properties and the minimally invasive preparation method rendered possible by the adhesive bonding technique. Although resin composite has many advantages and has developed and improved continuously since its introduction some sixty years ago, it still presents a number of problems, hampering even better clinical performance.

One factor that plays a main role in this scenario is the inherent polymerization shrinkage of most resin composites [1,2]. Polymerization shrinkage causes stress within the composite restoration, within the surrounding tooth substance and at the restoration-tooth interface, which may lead to marginal gap formation between the tooth and the restoration, nanoleakage, cusp deformation, postoperative sensitivity, paramarginal enamel fractures, marginal discoloration, and ultimately to fractures or secondary caries [3–5].

The negative side effects of polymerization shrinkage and stress are sought counteracted by elaborate restorative techniques and appropriate restorative materials. This includes, among others, an adhesive system able to reliably bond the hydrophobic resin composite to the hydrophilic dentin substance. Like resin composites, adhesive systems have undergone formidable development over the years and today an abundance of products of variable application complexity are available to the clinician.

The plethora of material types and brands on the market makes it challenging not only for the clinician to stay updated and to make wise choices, but also for researchers seeking to test the safety and efficiency of materials and techniques.

Ideally, all dental restorative materials should be tested in

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randomized, controlled clinical trials. However, due to innumerable confounding factors and the high number of types and brands of materials and application techniques, it is impossible to test and evaluate the performance of all materials and material combinations in vivo. Hence, in vitro studies are applied to evaluate materials and techniques and their potential clinical performance, although the fact that these studies can only attempt to simulate biological conditions raises the question of translatability to the clinical reality.

Numerous in vitro tests are in use for assessing the bonding effectiveness of resin composite to tooth structure. Static tests are the most used and can be either macro or micro tests according to the size of the bonding area. Macro tests (bonding area larger than 3 mm^2) have been criticized for resulting in a higher incidence of cohesive failure in the substrate (tooth or composite) and thus in inaccurate estimations of the bond strength [6,7], and micro tests (bonding area of 1 mm^2 or less) [6,8,9] are favored nowadays. Among the macro as well as the micro tests, the most frequently used are the tensile and the shear tests according to the direction of the applied load. While microtensile tests are thought to result in a more uniformly distribution of stress than the microshear test [7,10,11], they are also much more time consuming and technique sensitive to perform.

Not only the bond strength of the adhesive - resin composite restorations is important for the survival of the restoration, but also their marginal adaptation. Class II restorations may seem especially challenged by polymerization shrinkage and stress as there is typically a reduced amount of tooth structure available to distribute stresses. A potentially aggravating factor is the fact that the cervical margin of Class II cavities often finishes in dentin, to which it is generally more difficult to generate adequate adhesion than to enamel [5]. One of the most commonly used in vitro tests for evaluating the marginal adaption of Class II restorations is the replica technique according to which the marginal adaptation is studied and quantified on epoxy models of the restorations under the scanning electron microscope (SEM).

To evaluate the clinical relevance of in vitro bond strength tests and marginal adaptation assessments, several studies have been carried out to search for correlations between in vitro results and those of clinical studies, but only few in vitro tests have been found to correlate with clinical results [8,12–14].

A recent retrospective multicenter study of risk factors for failure of direct restorations reported the median survival time, Hazard Ratio and annual failure rate of 27.407 restorations in 7.858 patients from five private practices [15]. Since information on the adhesive - resin composite combinations used were available, the aim of the present study was, first, to determine the in vitro dentin bond strength and marginal adaptation of Class II restorations using the same adhesive – resin composite combinations that had been used also in that study [15] and, secondly, to test for correlations between, on the one hand, dentin bond strength or marginal adaptation and, on the other hand, the clinical survival outcomes. The null hypothesis was that neither the dentin bond strength results nor the marginal adaptation results would correlate with the clinical survival results.

2. Materials and methods

For the three in vitro tests (3 tests, 8 groups, n = 15/group), a total of 360 dentin specimens were produced from extracted permanent human molars (without restorations or caries) obtained from a pooled biobank. The local ethical committee considers pooled biobanks as irreversibly anonymized and waives the necessity of previous ethical approval. The molars were cleaned with a scaler under tap water to remove debris and soft tissue and then embedded in self-curing resin (Paladur pink; Heraeus Kulzer GmbH, Hanau, Germany). For the μTBS test the molars were poured into circular molds and with only the root surfaces in contact with the self-curing resin. Subsequently, the molars were ground from the occlusal surfaces on a grinding machine with grit #220 silicon carbide (SiC) abrasive paper under water-cooling (Struers 15/Tegra Pol 1, Struers, Ballerup, Denmark) until at mid-coronal dentin [16,17]. The dentin surfaces were air-dried and carefully checked for absence of enamel, caries and pulp perforations. For the μSBS [18] and the marginal gap formation tests [5,19], the roots of the molars were removed using a water-cooled diamond saw (IsoMet Low Speed Saw; Buehler, Lake Bluff, IL, USA; μSBS) or shortened with grit #220 SiC paper under water-cooling (Struers 15/Tegra Pol 1; marginal gap formation). Subsequently, the molars were embedded in self-curing resin (Paladur pink) poured into circular molds and for the marginal gap formation specimens with only the root surfaces in contact with the self-curing resin. All dentin specimens were stored in tap water in the refrigerator (4 °C) until the respective test specimens were produced.

2.1. Microtensile bond strength (μTBS)

2.1.1. Preparation of μTBS specimens

The dentin specimens were retrieved from the refrigerator at least 1 h before use, randomly assigned to one of the 8 groups and stored in tap water at room temperature. Immediately before the adhesive treatment, the dentin of each specimen was ground on abrasive paper, SiC grit size #500 (Struers) for 5 s to create a standardized smear layer [16,17]. The SiC paper was replaced after grinding of ten dentin specimens. Subsequently, the dentin was gently air-dried and immediately treated with one of the three adhesives according to the manufacturers` instructions (Supplementary material). Immediately after adhesive treatment, a transparent circular matrix (Lucifix Molar, KerrHawe, Bioggio, Switzerland), on which the increment thickness had been marked with a waterproof felt-tip pen, was placed around each dentin specimen, which was then restored with two resin composite layers of 2 mm and one layer of 1 mm with different resin composites (Supplementary material) depending on the adhesive.

Each composite layer was light-cured according to the manufacturers` instructions, i.e., 20 s per layer. All light-curing was done using an LED light-curing unit (bluephase 16, Ivoclar Vivadent AG, Schaan, Lichtenstein; irradiance 1500 mW/cm^2) in “high power” mode, and the light power density was verified with a radiometer (bluephase meter Ser. No. 001556, Ivoclar Vivadent AG) at the beginning and end of each day of specimen preparation. This same light-curing unit was used for all three tests. The restored dentin specimens were kept in tap water at 37 °C for 24 h (Memmert UM 500, Memmert & Co., Schwabach, Germany).

2.1.2. μTBS testing

After the 24 h storage, the restored dentin specimens were sectioned with an electronically programmable water-cooled diamond saw (Struer Accutom; Struers) in x and y directions perpendicular to the adhesive interface to obtain as many sticks as possible from the most central part of each restored dentin specimen [16,17] (Fig. 1). Half of the sticks were stored in 0.5% Chloramine T for 6 m, the other half were immediately subjected to μTBS testing. Before testing the μTBS, the width and breadth of each stick were measured with a digital caliper with an accuracy of 0.001 mm (Mitutoyo IP 65; Kawasaki, Japan) for calculation of bonding surface (BSU $\approx 1.0 \pm 0.1$ mm^2). Each stick was then fixed by its ends to a notched Ciucchi`s jig mounted in a universal testing machine (Instron 5942, High Wycombe, UK) with a low viscosity resin (Heliobond, Ivoclar Vivadent AG, Schaan, Lichtenstein). The stick was stressed in tension at a crosshead speed of 1 mm/min until fracture, and the maximum force (F_{max} (N)) was recorded. The μTBS values (MPa) were calculated according to the formula μTBS = F_{max} / BSU. Pre-testing failure was given the value “0” while any failure due to manipulation error were excluded [16]. The mean μTBS per tooth was used as the statistical unit.

2.2. Microshear bond strength (μSBS)

2.2.1. Preparation of μSBS specimens

The dentin specimens were retrieved from the refrigerator at least

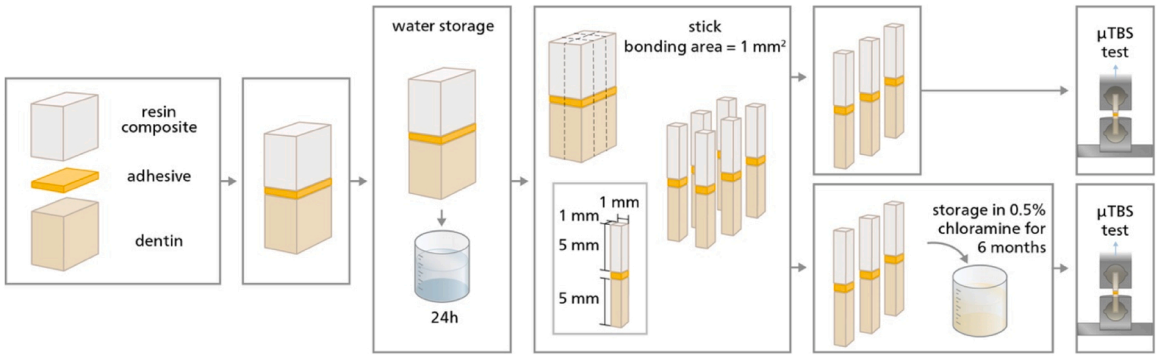

Fig. 1. Preparation of the µTBS specimens and test procedure.

1 h before use, randomly assigned to one of the 8 groups, and stored in tap water at room temperature. A standardized smear layer was created as described above, and the dentin was gently air-dried and immediately treated with one of the three adhesives according to the manufacturers` instructions. Immediately after the adhesive treatment, a cylinder of resin composite was bonded to the adhesive area using a split Teflon mold (inner diameter: 1.5 mm ≈ adhesive area: 1.8 mm^2, height: 2 mm) mounted in a holding device [18] (Fig. 2). The resin composite cylinders were light-cured according to the manufacturers` instruction, i.e., 20 s. After 5 min, the Teflon mold was removed, and all specimens were stored in tap water at 37 °C for 24 h or in 0.5% Chloramine T for 6 m.

2.2.2. µSBS testing

After storage, the specimens were subjected to bond strength testing performed in a universal testing machine (Zwick Z1.0 TN, Zwick, Ulm, Germany). The µSBS specimens were loaded until fracture. The load was applied at a right angle to the resin composite cylinder at a cross-head speed of 1 mm/min [18]. The strength required for fracture (F_{max}(N)) was determined and the µSBS (MPa) was calculated as (F_{max} (N) / bonding area (mm^2)).

2.3. Marginal gap formation

2.3.1. Cavity preparation and restoration

In each molar, a standardized Class II cavity was prepared on the mesial and on the distal surfaces by use of a coarse-grained preparation diamond bur (Intensiv 8113NR; Intensiv AG, Montagnola, Switzerland) [19]. The dimensions of the standardized cavity were 4 mm in oro-vestibular width, 6 mm in occluso-cervical height, including a margin below the cemento-enamel junction, and 2 mm in mesio-distal depth. The margins of the cavity were not beveled. The two cavities in each molar were restored with the same adhesive - resin composite combination. A circular curved matrix (Automatrix, Dentsply Sirona, Konstanz, Germany) was placed and the cavities were treated with one of the three adhesives, according to the manufacturers' instructions (Fig. 3). Each cavity was then built up in three resin composite layers of 2 mm each, with different resin composites depending on the adhesive. Each resin composite layer was light-cured according to the manufacturers` instructions, i.e., 20 s per layer. After removal of the matrix, each restoration was polished with Sof-Lex XT Discs (Sof-Lex XT Discs coarse, medium, fine, and superfine; 3 M ESPE) and then stored in tap water for 24 h at 37 °C. The discs were changed after polishing of the two restorations in each molar.

2.3.2. Production of replicas

After 24 h storage, each molar was cut in half in oro-vestibular direction between the two restorations, with a water-cooled diamond saw, resulting in two specimens (restorations) per molar. One specimen was immediately placed in 0.5% Chloramine T and stored for 6 m at 37 °C. The other specimen was cleaned for 2 min in an ultrasonic bath with deionized water, before being embedded in cylindrical stainless-steel molds with self-curing acrylic resin (Paladur pink), letting the restoration protrude from the acrylic resin [19]. After curing of the acrylic resin, the specimen was removed from the mold and cleaned again for 2 min in an ultrasonic bath. Subsequently, polyvinylsiloxane impressions were taken (Aquasil Ultra LV Regular Set and Aquasil Ultra

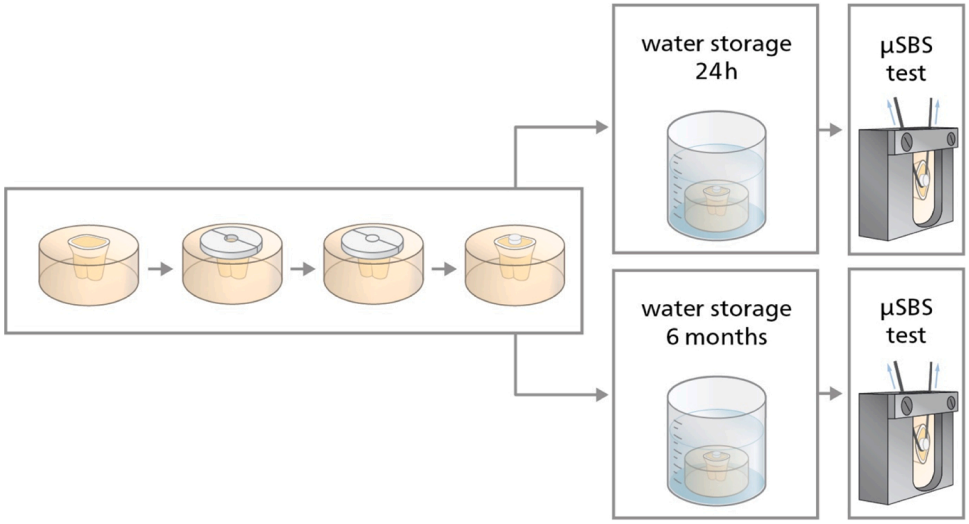

Fig. 2. Preparation of the µSBS specimens and test procedure.

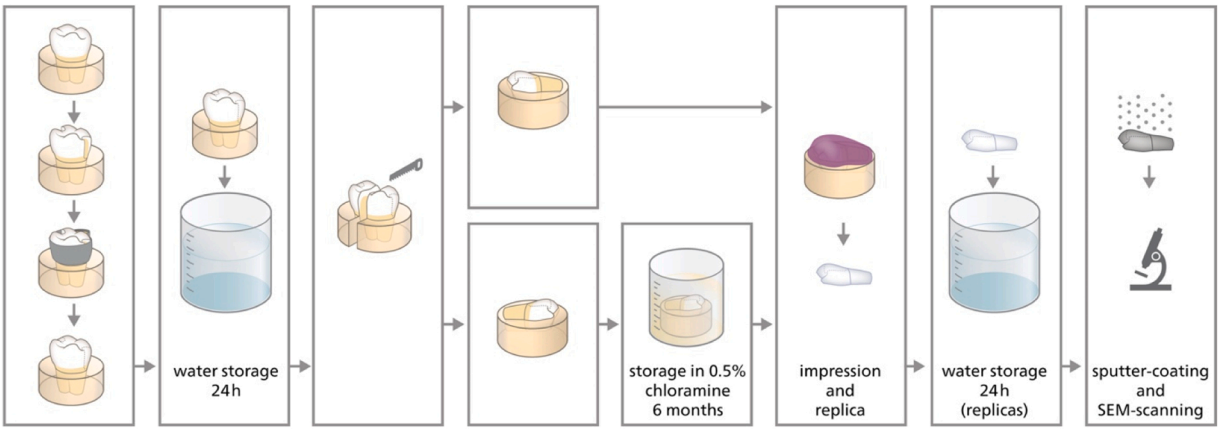

Fig. 3. Preparation of the gap formation specimens and test procedure.

Medium Fast Set; Dentsply Sirona, Konstanz, Germany) of the restoration and poured with epoxy resin (EpoFix; Struers) to produce 24 h replicas. Immediately following impression taking, the specimen was transferred to tap water for 24 h. The same procedure was used to produce replicas after 6 m of storage.

2.3.3. Measurement of marginal gap formation

All replicas, the 24 h and the 6 m, were fixed onto an object carrier and then on aluminum stubs and sputter-coated with gold/palladium (100 s 50 mA) by use of a sputter-coating device (Balzers SCD 050; Balzers, Liechtenstein). The replicas were examined under a scanning electron microscope (SEM; JEOL JSM6010PLUS/LV; JEOL, Tokyo, Japan) and SEM micrographs were produced from each replica. Since the restorations were located partly in enamel and partly in dentin (i.e., below the cemento-enamel junction), marginal gap formation of the restorations was assessed separately for the "margin located in enamel" and for the "margin located in dentin". First, the length of the entire enamel margin of each restoration was measured (in µm). In case of gaps along the margin, the length of each gap was measured (in µm) and the individual gap lengths were added. The percentage of the total gap length was then calculated relative to the entire enamel margin. This procedure was repeated for the restorative margin located in dentin. The measurements of marginal gap formation were performed with the ImageJ software version 1.53 t (http://imagej.nih.gov) [5] by one operator and blinded to the adhesive - resin composite group. Any paramarginal gap formation was registered as being either present or absent, and the number of teeth with paramarginal gaps was calculated for each group.

2.4. Calculation of in vivo outcomes (median survival time (MST), Hazard Ratio (HR) and annual failure rates (AFR))

To calculate the median survival time (MST), Hazard Ratio (HR) and annual failure rates (AFR) of the chosen adhesive - resin composite combinations, the data set of a recent retrospective multicenter study was used [15]. The data were analyzed and correlated for Classes I (n = 2.536), II (n = 4.285), III (n = 2.005), IV (n = 952) and V (n = 917) as well as for the total number of all direct restorations (n = 10.695).

2.5. Statistical analysis

The in vitro results were analysed by Kruskal-Wallis tests and in case of significant effects further analyses were conducted by Mann-Whitney U tests to test for differences between material combinations, adhesives, resin composites as well as storage times (24 h, 6 m). Spearman correlation tests were carried out between the results of the various in vitro tests (µTBS, µSBS, marginal gap formation, paramarginal fractures) as well as between the in vitro results and the clinical outcomes (MST, HR, AFR). Correlations between µTBS or µSBS results were calculated for each class of restoration separately as well as for the total number of restorations (10.695). In contrast, the results of the marginal gap formation test in Class II cavities were only correlated with the clinical parameters of the 4.285 Class II restorations. All statistical analyses were performed using the IBM SPSS Statistical tool (v23) and the level of significance was set at $\alpha = 0.05$.

3. Results

The results of the µTBS and µSBS tests are presented in Table 1 and those of the marginal gap formation test in Table 2 and Fig. 4.

3.1. µTBS

After a storage time of 24 h statistically significant differences were found between the µTBS of the eight adhesive - resin composite groups ($p < 0.001$), with One Up Bond F Plus-Enamel Plus HFO showing the significantly lowest µTBS and Prime&Bond NT-Tetric EvoFlow followed by Optibond FL-Tetric EvoFlow and Optibond FL-Herculite XRV showing the significantly highest µTBS. After storage for 6 m, the µTBS had been significantly reduced for all eight adhesive - resin composite groups ($p < 0.001$) (Table 1). The µTBS results were found to vary with statistical significance ($p < 0.001$), and with Optibond FL-Tetric EvoFlow showing higher µTBS than Optibond FL-Herculite XRV ($p < 0.001$), Optibond FL-Grandio ($p = 0.007$), One Up Bond F Plus-Enamel HFO ($p = 0.006$) and Prime&Bond NT-Tetric EvoFlow ($p = 0.005$). As regards any effect of adhesive and of resin composite, respectively, the adhesive Prime&Bond NT resulted in higher µTBS after 24 h than did Optibond FL ($p = 0.028$) and One Up Bond F Plus ($p = 0.002$), while the resin composites Tetric EvoFlow and Herculite XRV resulted in higher µTBS than did Grandio ($p < 0.05$) and Enamel Plus HFO ($p = 0.036$). After 6 m storage, there were no longer any differences between the three adhesives, while Ceram.x Spectra ST and Tetric EvoFlow showed higher µTBS than did Herculite XRV ($p = 0.002$).

3.2. µSBS

After a storage time of 24 h no significant differences in µSBS were found between the eight adhesive - resin composite groups ($p = 0.066$) (Table 1). Whereas storage for 6 m did not reduce the µSBS for any of the eight adhesive - resin composite groups, significant differences were found among the eight 6 m µSBS results. Thus, Optibond FL-Ceram.x Spectra ST yielded higher µSBS than did One Up Bond F Plus-Tetric

Table 1
Mean (standard deviation) μTBS and μSBS values according to adhesive – resin composite combination, type of composite and type of adhesive.

		Microtensile bond strength (μTBS) [MPa]				Microshear bond strength (μSBS) [MPa]							
		24 h		6 m		24 h			6 m				
Adhesive -resin composite	Optibond FL-Herculite XRV	28 (16,3)	ABC	3,8 (6,6)		A	*	7,2 (4,5)	A	6,3 (3,3)	AB		
	Optibond FL-Grandio	18,7 (11,6)	AB	3,9 (4,1)		AB	*	7,5 (3,2)	A	5,5 (2,2)	AB		
	Optibond FL-Ceram.x Spectra ST	22,8 (13,2)	ABC	9,6 (6,7)		BC	*	6,6 (4,3)	A	8,7 (3,9)	B		
	Optibond FL-Tetric EvoFlow	27,8 (15,3)	BC	12,3 (8,7)		C	*	6,6 (3)	A	7,4 (4,5)	AB		
	Optibond FL-Enamel Plus HFO	21,5 (13,9)	ABC	9 (10,9)		BC	*	6,8 (4)	A	5,8 (3,6)	AB		
	One Up Bond F Plus-Tetric EvoFlow	21,7 (8,6)	ABC	9,6 (8,1)		BC	*	4,5 (3,1)	A	3,5 (3,7)	A		
	One Up Bond F Plus-Enamel Plus HFO	17,3 (7,8)	A	4,3 (3,2)		AB	*	4,6 (2,4)	A	3 (1,8)	A		
	Prime&Bond NT-Tetric EvoFlow	29,1 (9,6)	C	4,9 (4,3)		AB	*	5,1 (3,7)	A	3,2 (2,9)	A		
Resin composite	Herculite XRV	28 (16,3)	**AC**	3,8 (6,6)		**A**	*	7,2 (4,5)	**A**	6,3 (3,3)	**AB**		
	Grandio	18,7 (11,6)	**A**	3,9 (4,1)		**AB**	*	7,5 (3,2)	**A**	5,5 (2,2)	**AB**		
	Ceram.x Spectra ST	22,8 (13,2)	**ABC**	9,6 (6,7)		**B**	*	6,6 (4,3)	**A**	8,7 (3,9)	**B**		
	Tetric EvoFlow	26,1 (11,8)	**C**	8,7 (7,6)		**B**	*	5,4 (3,4)	**A**	4,8 (4,2)	**A**		
	Enamel Plus HFO	19,4 (11,3)	**AB**	6,7 (8,4)		**AB**	*	5,7 (3,5)	**A**	4,3 (3,1)	**A**		
Adhesive	Optibond FL	23,5 (14,1)	a	7,6 (8,3)		a	*	7 (3,8)	b	6,8 (3,7)	b		
	One Up Bond F Plus	19,5 (8,4)	a	7 (6,6)		a	*	4,6 (2,7)	a	3,2 (2,8)	a	*	
	Prime&Bond NT	29,1 (9,6)	b	4,9 (4,3)		a	*	5,1 (3,7)	ab	3,2 (2,9)	a		

Different letters indicate significant differences between groups ($p < 0.001$; Mann-Whitney-U tests adjusted by the Bonferroni correction for multiple tests).
Asterisks indicate significant differences between the two time points ($p < 0.001$; Wilcoxon signed rank tests).

Table 2
Mean percentage (standard deviation) of gap formation in enamel and dentin as well as paramarginal fractures according to the different adhesive-composite combinations, the type of adhesive, and the type of composite.

		Marginal gap in enamel [%]					Marginal gap in dentin [%]				Paramarginal fractures [%]				
		24 h		6 m			24 h		6 m			24 h		6 m		
Adhesive -resin composite	Optibond FL-Herculite XRV	24 (20)	ABC	32 (25)	BC		19 (35)	A	76 (33)	A	*	33	A	87	AB	*
	Optibond FL-Grandio	15 (19)	C	18 (24)	C		20 (24)	A	86 (18)	A	*	47	A	93	AB	*
	Optibond FL-Ceram.x Spectra ST	19 (17)	BC	48 (27)	ABC	*	14 (28)	A	78 (25)	A	*	27	A	73	AB	*
	Optibond FL-Tetric EvoFlow	42 (28)	AB	56 (32)	AB		9 (14)	A	83 (29)	A	*	29	A	71	AB	*
	Optibond FL-Enamel Plus HFO	29 (25)	ABC	52 (31)	ABC		28 (30)	A	98 (7)	A	*	27	A	73	A	*
	One Up Bond F Plus-Tetric EvoFlow	53 (28)	A	79 (26)	A	*	44 (36)	A	86 (22)	A	*	33	A	20	B	
	One Up Bond F Plus-Enamel Plus HFO	35 (21)	ABC	71 (15)	AB	*	29 (36)	A	85 (23)	A	*	13	A	67	AB	*
	Prime&Bond NT-Tetric EvoFlow	46 (25)	AB	63 (23)	AB		29 (34)	A	76 (30)	A	*	20	A	47	AB	
Resin composite	Herculite XRV	24 (20)	**B**	32 (25)	**C**		19 (35)	**A**	76 (33)	**A**	*	33	**A**	87	**A**	*
	Grandio	15 (19)	**B**	18 (24)	**C**		20 (24)	**A**	86 (18)	**A**	*	47	**A**	93	**A**	*
	Ceram.x Spectra ST	19 (17)	**B**	48 (27)	**BC**	*	14 (28)	**A**	78 (25)	**A**	*	27	**A**	73	**AB**	*
	Tetric EvoFlow	47 (27)	**A**	66 (28)	**A**	*	27 (32)	**A**	83 (23)	**A**	*	27	**A**	45	**B**	
	Enamel Plus HFO	32 (23)	**AB**	61 (26)	**AB**	*	28 (33)	**A**	91 (18)	**A**	*	20	**A**	70	**AB**	*
Adhesive	Optibond FL	26 (23)	b	41 (31)	bc	*	18 (27)	b	84 (24)	a	*	33	a	79	a	*
	One Up Bond F Plus	44 (26)	a	75 (22)	a	*	37 (36)	a	85 (22)	a	*	23	a	43	b	
	Prime&Bond NT	46 (25)	a	63 (23)	ab		29 (34)	ab	76 (30)	a	*	20	a	47	b	

Different letters indicate significant differences between the different groups within each of the three categories for marginal gaps ($p < 0.001$; Mann-Whitney-U tests adjusted by the Bonferroni correction for multiple tests) and paramarginal fracture ($p < 0.001$; Chi Square).
Asterisks indicate significant differences between the two time points ($p < 0.001$; Wilcoxon signed rank tests and Chi Square, respectively).

EvoFlow ($p = 0.009$), One Up Bond F Plus-Enamel Plus HFO ($p < 0.002$), and Prime&Bond NT-Tetric EvoFlow ($p = 0.002$). As regards any effect of adhesive and resin composite, respectively, when measured after 24 h, Optibond FL had higher μSBS than did One Up Bond F Plus ($p = 0.006$), whereas there were no significant differences in μSBS between the five resin composites ($p = 0.257$). After 6 m μSBS had only decreased for the adhesive One Up Bond F Plus, and Optibond FL had higher μSBS than did One Up Bond F Plus ($p < 0.001$) and Prime&Bond NT ($p = 0.001$), whereas Ceram.x Spectra ST resulted in higher μSBS than did Tetric EvoFlow ($p = 0.005$) and Enamel Plus HFO ($p = 0.007$).

3.3. Marginal gap formation in enamel

After 24 h storage, significant differences were found for marginal gap formation in enamel between the eight adhesive - resin composite groups ($p < 0.001$) (Table 2). One Up Bond F Plus-Tetric EvoFlow, Prime&Bond NT-Tetric EvoFlow, and Optibond FL-Tetric EvoFlow showed most gaps, while Optibond FL-Grandio showed least gaps. After the 6 m storage, gap formation had increased for Optibond FL-Ceram.x Spectra ST ($p = 0.006$), One Up Bond F Plus-Tetric EvoFlow ($p = 0.015$), and One Up Bond F Plus-Enamel Plus HFO ($p < 0.001$). Significant differences were found between the eight adhesive - resin composite groups ($p < 0.001$). Optibond FL-Grandio showed less marginal gap formation than did Optibond FL-Tetric EvoFlow ($p = 0.047$), One Up Bond F Plus-Tetric EvoFlow ($p < 0.001$), Prime&Bond NT-Tetric EvoFlow ($p = 0.005$), and One Up Bond F Plus-Enamel Plus HFO ($p < 0.001$). As regards any effect of adhesive and resin composite, when measured after 24 h, Optibond FL showed less gap formation in enamel than did One Up Bond F Plus ($p = 0.004$) and Prime&Bond NT ($p = 0.019$), and Tetric EvoFlow showed more gap formation than did Grandio ($p < 0.001$), Ceram.x Spectra ST ($p = 0.005$), and Herculite XRV ($p = 0.038$). After 6 m, marginal gap formation had significantly increased for the adhesives Optibond FL ($p = 0.002$) and One Up Bond F Plus ($p < 0.001$), and for three of five resin composites, Ceram.x Spectra ST ($p = 0.006$), Tetric EvoFlow ($p = 0.002$) and Enamel Plus HFO ($p < 0.001$). Thus after 6 m, Optibond FL showed less marginal gap formation in enamel than did One Up Bond F Plus ($p = 0.062$), and

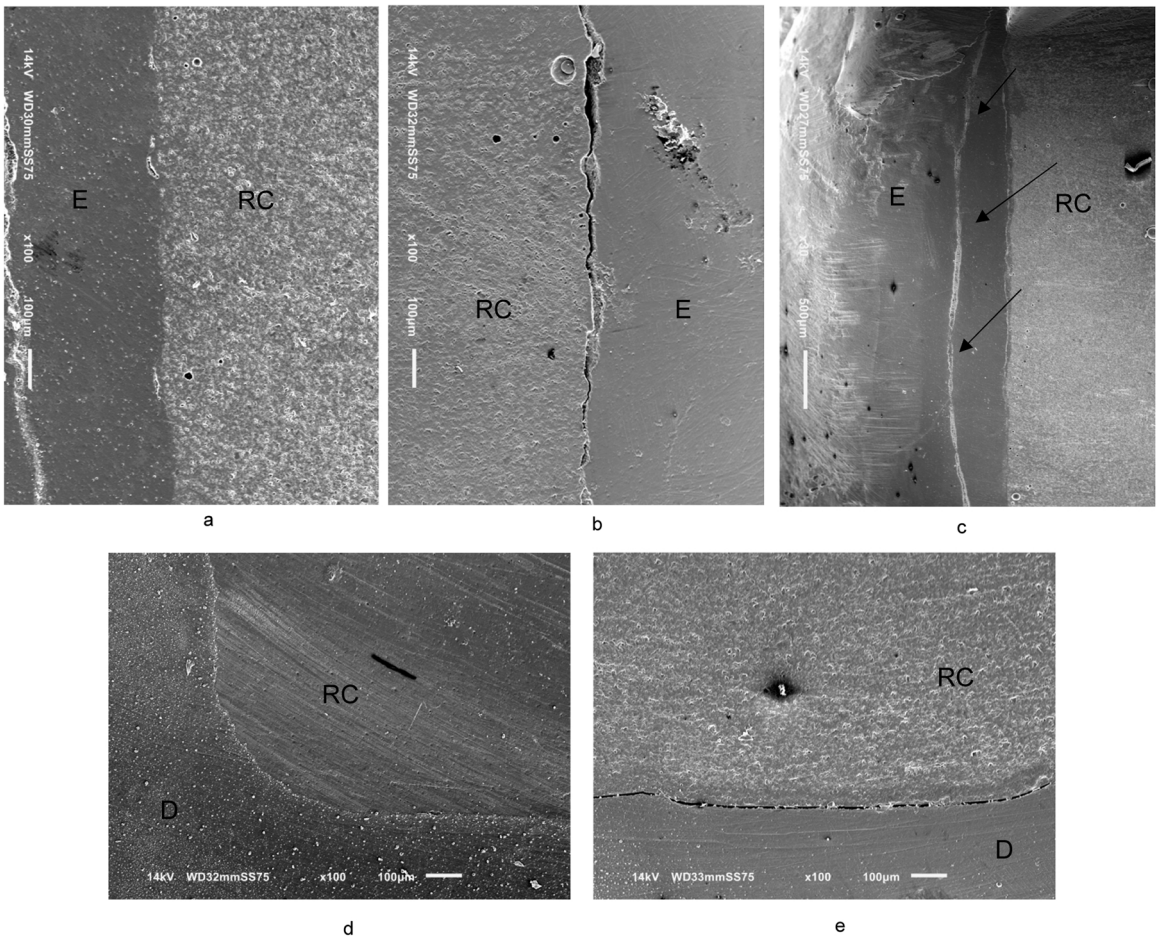

Fig. 4. a-e. Representative SEM micrographs of restorative margins in enamel (E) and dentin (D) with (b, e) and without (a, d) gap formation and paramarginal enamel fracture (c, arrows). RC, resin composite.

Grandio showed less marginal gap formation than did Tetric EvoFlow ($p < 0.001$) and Enamel Plus HFO ($p < 0.001$).

3.4. Marginal gap formation in dentin

After 24 h storage ($p = 0.091$) as well as after 6 m storage ($p = 0.286$), there were no significant differences in dentin gap formation between the eight adhesive - resin composite groups (Table 2). The extent of dentin gap formation increased significantly from 24 h to 6 m for all eight groups ($p = 0.021$). While there were no differences between the five resin composites at either time point, the adhesive One Up Bond F Plus resulted in more dentin gap formation than did Optibond FL ($p = 0.037$) after 24 h storage.

3.5. Paramarginal fractures

After a storage time of 24 h no significant difference in the number of paramarginal fractures was found between the eight adhesive - resin composite groups ($p = 0.665$) (Table 2). After storage for 6 m the number of paramarginal fractures had increased significantly for all adhesive - resin composite groups except Prime&Bond NT-Tetric EvoFlow ($p = 0.123$) and One Up Bond F Plus-Tetric EvoFlow ($p = 0.341$), as well as for the adhesive Optibond FL ($p < 0.001$) and all composites except Tetric EvoFlow ($p = 0.06$). After 6 m storage, significant differences were found between the eight adhesive - resin composite groups ($p = 0.001$), the adhesives ($p < 0.001$), and the composites ($p = 0.002$). Thus, One Up Bond F Plus-Tetric EvoFlow resulted in fewer paramarginal fractures ($p = 0.001$) than did Optibond FL-Herculite XRV and Optibond FL-Grandio, the adhesive Optibond FL showed more paramarginal fractures than did One Up Bond F Plus and Prime&Bond NT ($p < 0.001$), and finally Tetric EvoFlow resulted in fewer paramarginal fractures ($p = 0.002$) than did Herculite XRV and Grandio.

3.6. Correlations between in vitro data

Significant, positive correlations were found between the 24 h μTBS results and the 6 m μTBS results (rho=0458, $p < 0.001$) and between the 24 h marginal gap formation in enamel and 6 m gap formation in enamel results (rho=0.306, $p = 0.001$). There was no significant correlation between μTBS and μSBS, but a negative correlation between gap formation in enamel and the number of teeth displaying paramarginal fractures after 6 m (rho=−0.538, $p < 0.001$).

3.7. Correlations between in vitro data and in vivo data

Correlation analyses between the in vitro results and the clinical results showed the following: the 24 h μTBS correlated positively with mean survival time (MST) (rho= 0.248, $p = 0.008$) and negatively with HR (rho=−0.204, $p = 0.031$) and AFR (rho=−0.218, $p = 0.02$) of the in vivo Class I restorations ($n = 2.536$) as well as positively with MST of all classes of restorations combined ($n = 10.695$) (rho=0.187, $p = 0.048$). The 6 m μTBS correlated negatively with HR (rho=−0.208, $p = 0.036$) and AFR (rho=−0.230, $p = 0.02$) of Class I restorations. The 6 m μSBS correlated positively with MST (rho=0.275, $p = 0.007$) and negatively with HR (rho=−0.252, $p = 0.013$) and AFR (rho=−0.202, $p = 0.048$) of Class II ($n = 4.285$) restorations, as well as positively with MST of Class

III (n = 2005) restorations (rho=0.387, p < 0.000), negatively with HR of Class IV (n = 952) (rho=−0.338, p = 0.002), and with AFP of Class V (n = 917) (rho=−0.256, p = 0.012) restorations. The marginal gap formation in enamel of the in vitro Class II restorations at 24 h as well as at 6 m correlated positively with HR (24 h: rho=0.288, p = 0.003; 6 m: rho=0.225, p = 0.022) and AFR (24 h: rho=0.295, p = 0.002; 6 m: rho=0.209, p = 0.034) of the in vivo Class II restorations. No significant correlations were found for in vitro marginal gap formation in dentin and the in vivo clinical data. The number of teeth with paramarginal fractures at 24 h correlated negatively with MST rho= −0.591, p < 0.001) and positively with HR (rho=0.334, p < 0.001) and AFR (rho=0.417, p < 0.001) of the in vivo Class II restorations.

4. Discussion

The present study determined the dentin bond strength and the marginal gap formation of Class II restorations of eight adhesive - resin composite combinations and searched for correlations to in vivo results of those same material combinations. For this, a data set of a recent retrospective multicenter study was used [15].

When bond strength to dentin was measured using the μTBS method, significant differences were found between the eight adhesive - resin composite groups at both time points tested. While Prime and Bond NT-Tetric EvoFlow and Optibond FL-Tetric EvoFlow exhibited high bond strength values after 24 h, Optibond FL-Grandio showed poor bond strength values both after 24 h and after 6 m. Tetric EvoFlow showed unexpectedly high values at both time points, while Grandio performed rather poorly. Previous studies have found bond strength to increase with flexural strength [20,21] and flexural modulus [7,21] of the resin composite. Considering that Tetric EvoFlow has much lower flexural strength and flexural modulus than the other four resin composites [22–25], the higher values for this composite were unexpected. It could be speculated that the lower viscosity of Tetric EvoFlow ensures superior contact with the adhesively treated dentin surface, fewer voids, and thus higher bond strength.

Prime&Bond NT showed higher initial bond strength than did the other two adhesives, but was significantly less stable after 6 m. The superior result of Prime&Bond NT over Optibond FL confirms the results of Almahady et al. who also used the μTBS test [26]. However, determining fatigue bond strength, Tsujimoto et al., found higher bond strength of Optibond FL than of Prime&Bond NT, explaining the superiority of Optibond FL by a much thicker, stronger, and more hydrophobic adhesive layer as well as more and longer resin tags [27]. Furthermore, Nikolaenko et al. found Optibond FL to promote a higher μTBS to dentin than did One Up Bond F Plus [28] which was not corroborated in the present study, as statistically similar bond strengths were found for the two adhesives at both time points.

Storage for 6 m decreased the μTBS values of all groups, adhesives, and composites. This is in corroboration with the findings of the meta-analysis by De Munck et. al. who reported significant reductions in μTBS for all adhesive categories after 1 year storage [29]. Among these categories, they found the 3-step etch & rinse adhesive Optibond FL to be more hydrolytically stable than the 2-step etch & rinse adhesive Prime&Bond NT and the 1-step self-etch adhesive One Up Bond F because of the presence of the separate, more hydrophobic resin layer as mentioned above. In contrast, the present study found One Up Bond F Plus to be as hydrolytically (un)stable as Optibond FL. This difference between the two studies may lie in the fact that we used the newer, optimized version, One Up Bond F Plus, while the version in the meta-analysis was One Up Bond F.

When bond strength to dentin was measured using the μSBS method, fewer differences were found between the adhesive - resin composite groups, the resin composites, and the adhesives. However, at both time points, Optibond FL showed higher μSBS than One Up Bond F Plus and Prime&Bond NT. The higher bond strength of the etch-and rinse adhesive is in line with previous results [30–32]. The pretreatment with phosphoric acid creates better adhesion than many acidic monomeric primers, not only because of a more efficient smear layer removal and creation of a well-defined etch pattern, but also because of more complete infiltration of adhesive and resin composite monomers in the demineralized collagen network producing longer resin tags, a thicker hybrid layer, and improved micromechanical retention [33,34].

In contrast to the μTBS results, no effect of prolonged storage was found when the bond strength was measured with the μSBS test, except for the One Up Bond F Plus adhesive. Possible reasons for this lack of effect of storage time on μSBS results include a bigger cross-sectional bonded area for the μSBS specimens (1.8 mm^2) than for the μTBS (1.0 mm^2), possibly delaying hydrolytic degradation due to longer diffusional distances, and a lower discriminatory power of the μSBS test.

In general, the μTBS test showed higher values than the μSBS test. This finding is in agreement with the literature [6,21,35] and thought to be the result of uniform stress distribution, stress concentration at the substrate area, and predominantly tensile stresses rather than shear stresses [8,25,27,36]. In particular, the much higher μTBS reported by Heintze et al. [37] for Optibond FL and Prime&Bond NT Dual Cure may, at least in part, be explained by the dumbbell trimming technique to arrive at a round cross-sectional area of 0.5 mm^2, i.e., half the area of the square area of 1 mm^2 used in the present study.

There was no significant correlation between μTBS and μSBS. This corroborates previous findings [12,21,38]. We had expected that using the exact same adhesive -resin composite groups in the two tests and the same operator would have improved the chance of the groups being ranked in the same order. The lack of a correlation may be explained by the high variability in bond strength in the present study, which has also been reported previously for all bond strength tests [12,38]. Possible reasons for this high variability are many, including flaws (e.g., air bubbles) in the adhesive layer, variations in the thickness of the adhesive layer, as well as flaws created during cutting of specimens into sticks, interference of the resin/product used to glue the sticks to the notched Ciucchi's jig and lack of alignment of the stick's bond line with its gripping surfaces particular for the μTBS test [35,36], factors which will result in variation in stress concentration between specimens and which may also explain the relatively many sticks lost due to pre-testing failure (43 in total) and manipulation error (37 in total) that occurred with the μTBS test, confirming the technique sensitivity of the method.

The significant differences in gap formation along the enamel margins observed among the eight adhesive - resin composite groups at both storage times seem the result of two main findings: first, Tetric EvoFlow showing more and Grandio showing less gap formation at enamel margins than the other three composites and secondly, Optibond FL showing significantly less gap formation than One Up Bond F Plus and Prime&Bond NT. In accordance with Hook`s Law, polymerization stress is determined by the multiplication of the elastic modulus and strain, as described by Ferracane [39]. This suggests that resin composites exhibiting both high polymerization shrinkage and a high elastic modulus are anticipated to yield the greatest polymerization stresses. Specifically, a higher filler content corresponds to reduced polymerization shrinkage and an increased elastic modulus [19]. While Tetric EvoFlow has a lower filler volume and lower resulting strength and stiffness, Grandio has a higher filler volume and high elastic modulus [5]. However, Grandio restorations not only exhibited less gap formation in enamel than did Tetric EvoFlow restorations, but they also showed more paramarginal fractures. These results are in line with previous in vitro studies which found resin composites with higher elastic modulus to have fewer marginal gaps in enamel, but more paramarginal fractures [5,19] and with finite element analyses that indicated a reduction in marginal stress under occlusal loading with an increase in the elastic modulus of the bonded resin composite [5,40]. While there seems to be a trade-off between gaps at the interface and fracture within the enamel, paramarginal fractures may be argued to have less serious consequences than marginal gaps at the restoration-enamel interface [19]. Regarding the adhesives, despite not

resulting in a consistently higher bond strength, Optibond FL showed less gap formation than did the other two adhesives. These results are in line with those of Park et al. [41], who explained their findings by etch-and-rinse adhesives generally exhibiting better adhesive performance, particularly when compared to a 1-step self-etch adhesive, because of more complete penetration into acid-demineralized dentin and superior bonding for systems with separate primers and bonding agents [42].

As for the marginal gap formation in dentin, no significant differences were found between the eight adhesive – resin composite groups, except that they all had more marginal gaps after 6 m storage. These findings are in agreement with those of previous studies [5,19] and may reflect the decrease in μTBS bond strength likewise observed for all eight groups. Furthermore, the absence of differences in dentin gap formation between the groups despite using resin composites of highly varying elastic modulus confirms the results of previous studies and their conclusion that the elastic modulus is not as important for gap formation in dentin as it is in enamel [5,19].

Neither of the two tests used to determine bond strength to dentin resulted in correlations with the in vitro marginal gap formation in dentin. This finding corroborates the conclusion of a systematic review which found no correlation between bond strength and neither microleakage nor gap formation for 80% of the included studies [43]. The reason for the lack of correlation also found in the present study may lie in the fact that bond strength was measured to flat dentin surfaces, while gap formation was assessed in Class II restorations [43]. The bond strength measured will reflect the adhesive's ability to create a homogenous hybrid layer, to penetrate effectively into the dentin tubules and to effectively bond to the hydrophobic composite. The resultant gap formation of Class II restorations will reflect not only these parameters but also the interplay between polymerization shrinkage and polymerization stress, elastic modulus and flowability of the composite,

Clinically, an adhesive may have different primary roles depending on the type of restoration. Thus, in Class V non-carious, non-retentive restorations where retention is low or absent, adhesion is required to first and foremost retain the restoration. For posterior restorations, on the other hand, excavation and/or preparation will often have created sufficient mechanical retention to retain the restoration per se, and in these situations the primary role of the adhesive may be to ensure that margins are sealed to avoid marginal staining, gap formation and caries and to reduce the risk of post-operative sensitivity by sealing the dental tubules [14]. Indeed, a meta-analysis on the clinical performance of cervical restorations, found the adhesive to have the most significant influence on the retention rate and marginal discoloration of these restorations [44]. Likewise, summing up on results from clinical trials, Heintze concluded that the retention and marginal staining of posterior restorations is influenced by the long-term bonding properties of the adhesive systems [14].

Due to innumerable restorative techniques and types of restorative materials on the market and the frequent introduction of new brands and versions, it is logistically impossible to test the performance all materials, material combinations and techniques in clinical trials. Hence, in vitro studies are applied to screen materials and techniques and attempt to predict their clinical performance. Consequently, numerous attempts have been made to correlate the results of in vitro tests, including bond strength results and marginal adaptation results, with the outcomes of clinical trials [6,12–14,37,38,45–47].

In the present study, we determined the dentin bond strength and marginal adaptation of Class II restorations of eight specific adhesive - resin composite groups and correlated these in vitro results with the clinical outcomes for the same eight material combinations found in a previously published retrospective multicenter study [15]. As regards the two bond strength methods applied, we found significant correlations between 24 h and 6 m μTBS and two or three clinical outcomes of the in vivo Class I restorations, while 6 m μSBS correlated with one or more clinical outcomes of Class II, III, IV and Class V restorations. Previous attempts to correlate bond strength values to clinical outcomes, primarily using retention rates of cervical restorations, have not been straightforward. However, it has been concluded that results from μTBS and macrotensile tests correlate better with clinical data than do results from macroshear and μSBS tests, and that the correlations improved if data from different test institutes are pooled [13,14]. The fact that any significant correlations were found for the μSBS in the present study is therefore unexpected. First, whereas all studies on which these conclusions were based included macrotensile, macroshear and μTBS, only few included μSBS [12,13,37,38] and it thus seems that the critique of the μSBS is based less on correlations analyses with clinical date and more on theoretical assumptions on the stress formation. Secondly, as mentioned, most previous attempts of correlating in vitro bond strength results to clinical outcomes have been made to retention rates of cervical restorations. In contrast, the present study sought correlations to all classes of restorations, and indeed the μTBS and μSBS correlated to each their class(es) of restorations. This might indicate that the requirements to the adhesive and the stress formation and stress distribution during bond strength testing do in fact differ, an interplay that should be researched further. A high incidence of cohesive failure in dentin has frequently been cited as a main disadvantage of the macroshear bond strength test [7,16,38]. However, the incidence of cohesive failure in dentin is lower for the μSBS that for macroshear [7]. Thus, a further possible reason for the significant correlations found between the μSBS results and clinical outcomes in this study is the exceptionally low number of cohesive fractures (1.3% cohesive failures, 90% adhesive failures between adhesive and dentin, and 8.7% mixed failures between adhesive and dentin or between adhesive and resin composite), which were maybe the result of the relatively low range of μSBS values. Finally, considering that the stress that forms in static bond strength tests such the μTBS and μSBS tests, in most situations do not reflect the stresses that develop in the clinical setting it would be interesting to also correlate the presently used clinical data with results from dynamic or fatigue bond strength tests as well as results from fracture mechanics tests.

As pointed out by Heintze [14], marginal gaps are presumed to facilitate penetration of bacteria and fluids which may lead to hypersensitivity, pulpitis, marginal staining, and debonding and loss of retention and consequently to influence the longevity of restorations. Thus, attempts have been made to correlate in vitro and in vivo results on marginal integrity. A weak correlation was found for Class V cavities provided that the same resin composite had been used [46] while a strong correlation was found for Class I restorations [47]. As for Class II restorations, a weak correlation was found between in vitro marginal gap formation in proximal enamel and the percentage of in vivo Class II restorations with marginal staining after 2 years and placed with the same eight adhesives [14]. As regards the in vitro marginal adaptation of Class II restorations measured in the present study, the in vitro marginal quality parameters gap formation in enamel and number of paramarginal fractures were found to correlate with the clinical outcomes HR and AFR of the in vivo Class II restorations. To the knowledge of the authors, no such correlations have been investigated previously and they lend support to the hypothesis that laboratory tests on marginal integrity are able to predict clinical performance and even longevity. In this context it should born in mind that the restorative technique, i.e. the preparation principle (e.g. ± beveling of enamel margins), the filling technique (increment size and direction), and the light-curing step (irradiance of the light-curing unit and curing times) used by the clinicians in the retrospective multicenter clinical study were not controlled and are bound to have varied among the clinicians and also to have differed from the restorative technique used in the present in vitro study. One factor that might be of particular importance for the marginal gap formation in enamel is that the enamel margins of the Class II cavities in the present study were not beveled. While numerous studies have reported enamel beveling to improve marginal adaptation and to reduce marginal leakage [48–50], other studies have not found any positive effect of beveling [51–53]. The effect of beveling has been reported to

depend on the adhesive system [49,54], and it seems that with the superior bonding potential of current adhesive systems, along with the lower polymerization shrinkage of modern resin composites, any positive effect of enamel beveling is reduced or even annulled [53].

Several limitations of this study should be considered when interpreting its results and impact. These limitations include the very high variability of the measured values obtained with the in vitro tests. Thus, the coefficient of variation superseded by far the recommended 20% mentioned by Heintze [14] implying that the results should be interpreted with caution and that the variability should be sought reduced by careful analysis and better control of the influencing factors and/or by an increase in the number of specimens per group. Secondly, the composition of the various adhesives and resin composites used may have been adjusted or changed, to a smaller or higher degree, in the years that had passed between the placement of the restorations included in the clinical study and the in vitro tests of the present study. Such adjustments are sometimes accompanied by a slight change in the brand name, but other times not. Thirdly, the restorative technique and procedure followed by the clinicians in the multicenter study was not monitored and not aligned with the restorative procedure followed in the present study. All three limitations may have influenced the probability of finding significant correlations between in vitro and in vivo data. A final factor to consider is that clinical results also show variability due to patient- and tooth-related factors (e.g., variations in the dentin substrate caused by erosion or caries) and differences among operators when applying the materials.

5. Conclusions

The null hypothesis was partially rejected as some significant correlations were found between in vitro results and clinical outcomes. Even though the bond strength and in vitro gap formation tests correlated with clinical results to some extent, justifying the continued use of in vitro tests, numerous other parameters co-determine the clinical performance and longevity of adhesively bonded resin composite restorations.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.dental.2024.05.004.

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