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Resin Tags Do Not Contribute to Dentin Adhesion in Self-etching Adhesives

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Purpose: Self-etching adhesives partly remove or dissolve the dentin smear layer, causing incomplete resin tag formation or low resin tag density. The quantitative contribution of properly formed resin tags to dentin adhesion was evaluated.

Materials and Methods: We assessed how the presence or absence of resin tags affects tubules of human deep-coronal dentin. G-Bond was used to bond Gradia resin composite. To ensure deep tubule penetration, we used a vacuum exsiccator. For eliminating tag formation, dentin tubules were presealed with adhesive and reverse bonded after finishing. Microtensile bond strength (μTBS) was measured on flat specimens and on Class I cavity floors. Thermocyclic loading was used to estimate the influence of resin tags on long-term behavior. Confocal laser scanning microscopy was used to evaluate adhesive interface dimensions.

Results: Hybrid layer thickness, tag length, and tag diameter increased under vacuum treatment. Presealing dentin tubules led to a residual tag area of 3.1% with a tag length of 10.8 μm. Under vacuum, 24.7% of the total dentin surface was covered with tags of 87.8 μm. Low C-factor preparations produced superior μTBS (71.8 to 92.7 MPa) compared with high C-factor Class I cavities (47.0 to 67.6 MPa). Thermocyclic fatigue differed from low to high C-factor situations. In Class I cavities, μTBS significantly decreased after thermocycling. On flat specimens, vacuum infiltration led to reduced μTBS after thermocyclic loading.

Conclusion: Initially, resin tag formation did not influence the μTBS in either type of C-factor preparation. After thermocyclic loading, μTBS decreased with or without resin tags. Adhesive fracture patterns occurred at the hybrid layer/dentin interface.

Keywords: dentin bonding, adhesion, tag formation, resin composites, hybrid layer, vacuum, C-factor.

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Adhesion mechanisms are a focus of research into developing optimal adhesive models. Using adhesive resins, the acid-etching technique provides well-established and reliable bonding to enamel.[20] Dentin adhesion remains less reliable because of greater substrate complexity and the technical sensitivity of the bonding procedure.[5,23] Conventional adhesive treatment follows a clinical protocol in three consecutive steps, including (1) demineralization of the cavity surface layer, exposing a thin collagen scaffold; (2) penetration of the collagen scaffold with hydrophilic monomers; and (3) application of a hydrophobic bonding agent to completely fill the intercollagen pores with resin.[23] The result of this three-step bonding process is commonly referred to as "hybridization" or the formation of a hybrid layer.[12] In addition to hybridization, resin tags are formed in the opened dentinal tubules, which are intended to contribute to the final dentin bond strength.[18]

Commercial dental adhesive systems are practically categorized into etch-and-rinse and self-etching systems. Modern self-etching products may have the priming and bonding step either combined (one-step) or separate (two-step). As a

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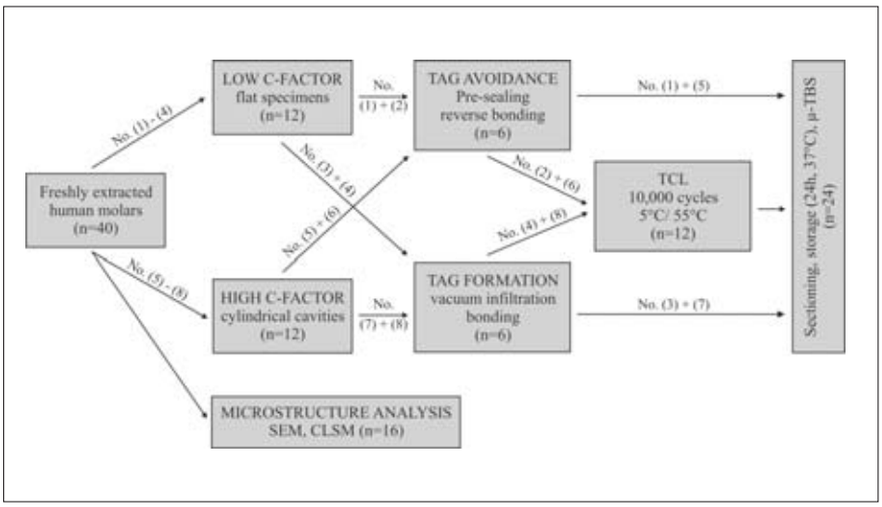

Fig 1 Experimental test design.

result, the number of consecutive steps and thus the sensitivity of the technique has been reduced.[13,24] Depending on the acidity of the incorporated functional monomers, self-etching adhesives exhibit either a mild or strong conditioning effect. With less acidic, mild adhesives, the smear layer on the dentin substrate will not be totally dissolved or removed, leading to a reduced resin tag density in dentin and hybrid layer thickness.[13]

The bond strength of conventional etch-and-rinse systems has been theoretically modeled by Pashley et al[18] as the sum of strength contributed by resin tags, the hybrid layer, and surface adhesion. For etch-and-rinse systems, resin tag formation contributes quantitatively up to one-third of the total shear bond strength.[22] No differences in shear bond strength were found in comparison of the hybrid layer after denaturation of the collagenous surface layer.[10] Self-etching adhesives are reported to benefit from additional chemical interaction between the functional mono-mers and the exposed hydroxyapatite phase in dentin.[24]

Together with the formation of a hybrid layer and chemical bonding to the anorganic phase of the dentin substrate, resin tags may become a key factor in the bonding of self-etching adhesives. Therefore, the aim of this study was to evaluate the contribution of resin tags to dentin adhesion, with respect to different C-factor configurations and to thermocyclic fatigue loading. The null hypothesis tested was that using a self-etching adhesive, resin tag formation does not quantitatively affect dentin adhesion.

MATERIALS AND METHODS

Experimental Procedure

A total of 40 freshly extracted caries-free human molars were used in this study. Bonding to dentin was performed on the occlusal surfaces of deep coronal dentin. Teeth were consecutively debrided, examined to ensure an absence of defects, and stored in antimicrobial 0.5% chloramine-T solution (Merck; Darmstadt, Germany) at 4°C for less than four weeks.

The experimental setup was based on formation or avoidance of resin tags in dentin tubules of the substrate. Figure 1 illustrates the experimental design indicating all test groups. A vacuum procedure was used to ensure deep penetration of the tubules. To consecutively eliminate any influence of tag contribution to dentin adhesion, the tag area was presealed with a nonstained adhesive (G-Bond, GC; Leuven, Belgium). Final finishing of the dentin substrates resulted in sealed tubules with a fully polymerized adhesive and freshly exposed intertubular dentin with an intact smear layer, ready for the reverse bonding procedure. Further experimental variables were C-factor variations and thermocyclic fatigue loading. Table 1 summarizes the test matrix with variables (1) – (8), which also were used to define groups.

For a low C-factor configuration, the occlusal enamel and half of the coronal dentin were removed by low-speed diamond-saw sectioning (Isomet, Buehler; Lake Bluff, IL, USA) under profuse water cooling. A standardized smear layer was created on the surface by wet grinding with 600-grit SiC paper for 60 s. For high C-factor groups, cylindrical Class I cavities (depth = 4 mm, height = 4 mm) were cut using coarse diamond burs (80-μm diamond bur, Two-Striper Prep-Set, Premier; St Paul, MN, USA) under profuse water cooling and finished with a 25-μm finishing diamond. Rounded bottom angles of the cavities were prepared.

Twelve teeth were randomly assigned to either the flat or the Class I preparation types and further subdivided according to the experimental variables (see Table 1).

All samples were restored with a one-step, self-etching, one-component adhesive (G-Bond, GC; Leuven, Belgium, Lot: 0609061; 09-2007) and a hybrid resin composite (Gradia direct, GC, Lot: 0502232; 02-2007, A3). G-Bond represents a

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Table 1 Dimensional data of the adhesive layer measured on flat specimens, given as means (SD)

Hybrid layer [µm]		Tag length [µm]		Tag diameter [µm]		Tag surface area [%]	
No vac.	Vacuum	No vac.	Vacuum	No vac.	Vacuum	No vac.	Vacuum
12.0	1.81	10.82	87.81	2.09	2.37	3.14	24.68
(0.16)	(0.61)	(9.6)	(27.9)	(0.41)	(0.39)	(1.48)	(1.97)

Superscript letters indicate no statistical differences (independent samples t-test, $p < 0.05$).

Table 2 Results of the microtensile bond strength to dentin

	Low C-factor				High C-factor, Class I cavity			
	No vacuum		Vacuum		No vacuum		Vacuum	
	no TC	TC	no TC	TC	no TC	TC	no TC	TC
Test group	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
µTBS [MPa]	92.7[a]	90.9[a]	86.7[a]	71.8[b]	67.6[b,c]	50.2[d]	57.2[c,d]	47.0[d]
SD	31.8	26.6	27.5	27.0	26.5	20.3	19.4	14.2
PFS [%]	0	0	0	0	5	0	4	0

Superscript letters indicate no statistical difference (one-way ANOVA/ LSD, $p < 0.05$). TC: thermocycling; SD: standard deviation; PFS: prematurely failed specimens.

mild self-etching one-step adhesive. Based on water and acetone as solvent, this filled adhesive contains a phosphoric acid monomer, the acidic monomer component 4-methacryloxyethyl trimellitate anhydride (4-META) and the hydrophobic resin compound urethane dimethacrylate (UDMA).[10] The adhesive was applied according to the manufacturer's protocol (10 s brushing, 5 s strong air drying, 10 s light curing). For the no-vacuum specimens, the dentin substrate was presealed under vacuum according to the described adhesive procedure and finished with 600-grit SiC paper or with the 20-µm finishing diamond. For confocal laser scanning microscopy (CLSM) evaluation of the adhesive layer, the adhesive was stained in advance (see microscopic analysis). The resin composite was applied for both preparation types in four 1-mm-thick layers. Each layer was polymerized for 20 s using a halogen light curing unit (Elipar Trilight, 3M ESPE; Seefeld, Germany, 750 mW/cm^2). Half of the flattened teeth and of the Class I cavities were adhesively bonded under vacuum. A vacuum exsiccator was used to penetrate the dentin surface at an atmospheric pressure of 10 KPa for 10 s prior to the air-drying step. After restorative treatment, half of the differently treated specimens were subjected to alternating 10,000 thermal cycles in water of 5°C and 55°C using a thermocycling apparatus (Willytec; Munich, Germany). The dwell time for each temperature was set to 30 s, and water temperature was checked continuously.

For producing rectangular µTBS specimens, the peripheral parts of the preparations were removed, resulting in a central area 9 mm^2. The remaining restored complex was vertically sectioned to produce 4 beams per tooth with a cross-sectional area of 0.5 mm^2 and a minimum dentin thickness to the pulp of 2.0 ± 0.5 mm. In case of beam fracture prior to testing, the percentage of prematurely failed specimens was recorded (PFS [%]) and set to 0 MPa for the final results and statistics. The µTBS beams were stored in distilled water for 24 h at 37°C. The beams were mounted in a tensile testing machine (Z 2.5, Zwick, Ulm, Germany), fixed with a special cyanoacrylate glue (Zapit, Dental Ventures of America; Corona, CA, USA), and tested with a 100-N load cell operating at a crosshead speed of 0.75 mm/min. µTBS was determined by dividing the maximum load with the individually measured adhesion area.

Microscopic Analysis

The remaining teeth (n = 16) were subjected to microstructural analysis using scanning electron microscopy (SEM) and CLSM. Vertical and horizontal cross sections were prepared using the sectioning procedure already described. The SEM

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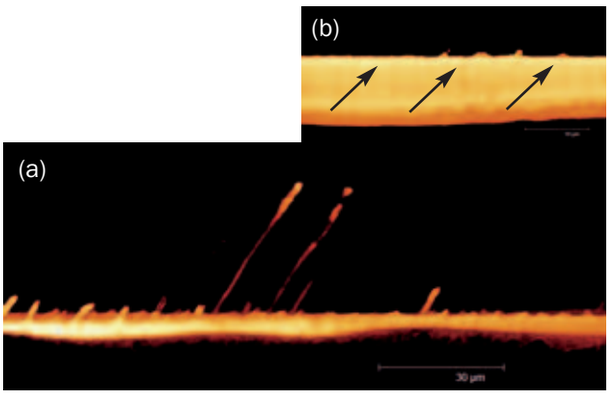

Fig 2 CLSM fluorescence image of the adhesive zone between composite and dentin (a) and magnification (b) of a thin hybrid layer (arrows). Only the stained adhesive can be seen.

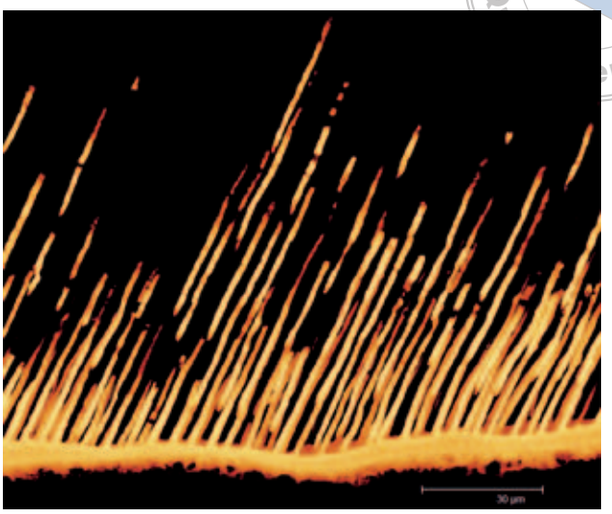

Fig 3 CLSM fluorescence image of the adhesive zone between composite and dentin under vacuum. Only the stained adhesive can be seen.

(ISI SR 50, Leitz; Wetzlar, Germany) was operated under 20 kV acceleration voltage. The backscatter electron mode was applied for improved surface contrast. The samples were gold sputtered under high vacuum. A CLSM (TCS SL, Leica; Bensheim, Germany) was used in fluorescence mode to highlight dentin tags and the hybrid layer. The fluorochrome rhodamine B isothiocyanate (Merck, maximum absorption = 540 nm, maximum emission = 625 nm) was compounded into the adhesive at a concentration of approximately 0.01%. The 514-nm excitation line of an argon ion laser was selected, and the emissions were detected using a DD 458/514 bandpass filter. The confocal Z-sections were taken at 1.6-µm increments under 1000X magnification (HC PL Fluotar 100, Leica, NA = 0.9).

Dimensional data were collected from the cross-sectional images and summarized in Table 2. Tag length, tag diameter, tag surface area, and hybrid layer thickness were calculated from 30 measurements on three different CLSM images of the low C-factor specimens. Tag surface area was calculated from a 3D projection of CLSM fluorescence images of the fractured interfacial plane. The respective measurements were taken from cross sections at 5 µm subsurface depth to minimize the influence of surface irregularities.

Statistical Analysis

Statistical analysis was performed using SPSS 14.0 for Windows (SPSS; Chicago, IL, USA). The values were found to have a normal Gaussian distribution (Kolmogorov-Smirnov test); thus, one-way ANOVA and LSD multiple comparison test were employed to determine statistical differences between the different groups ($p < 0.05$). Statistical differences between dimensional data were computed using the t-test for independent samples at a significance level of $\alpha = 0.05$.

RESULTS

Table 1 shows the µTBS data of the measured preparations. A total of 151 available specimens from 40 teeth were used for µTBS testing. All 80 specimens (4 specimens from each tooth) were available for low C-factor measurements, and 9 pre-test failures were recorded for Class I cavity preparations. Regarding the low C-factor bond strength, only group 4 (see Table 1) showed a significantly decreased µTBS of 71.8 MPa compared to groups 1 to 3, which performed between 92.7 and 86.7 MPa. For Class I cavity preparations, groups 6 to 8 showed no statistical differences, with values between 57.2 and 47.0 MPa, and group 5 proved to be statistically homogeneous with group 7.

Dimensional data for the adhesive interface layer were measured under CLSM. In the comparison of vacuum application, Table 2 gives the dimensions for hybrid layer thicknesses, tag lengths, tag diameters, and tag surface areas for low C-factor geometries. Under vacuum, all variables exhibited statistically significantly increased values. Tag length dramatically increased from 10.82 to 87.81 µm after vacuum infiltration. It should be noted that even though the dentin tubules in the no-vacuum group were presealed, a tag length of 10.82 µm was derived from residual unsealed tags, as shown in Fig 2, and expressed in a tag surface area of 3.14%. As expected, the resin tag surface area at a depth of 5 µm was increased significantly from presealed substrates (3.14%) to vacuum infiltrated dentin (24.68%). Figures 2 to 4 show dimensional data on the adhesive interfaces under investigation. The low vs high tag density can be clearly distinguished in Figs 2 and 3. Figure 2 shows the stained adhesive after pre-sealing of the tubules. Some small tag rudiments and residual stained resin tags are shown. The hybrid layer was easily detected by a light zone

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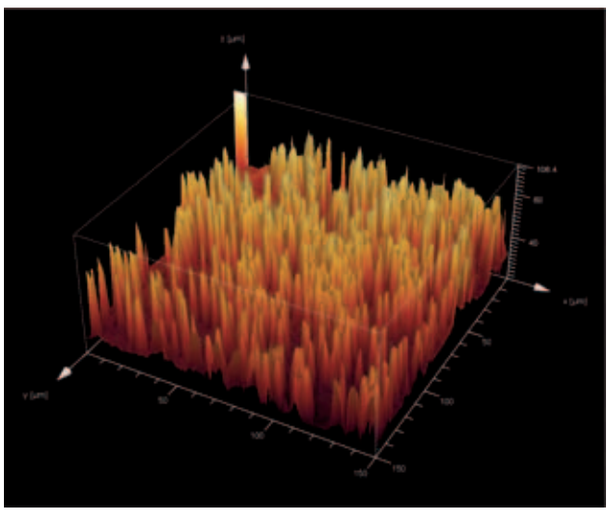

Fig 4 3D projection of a CLSM fluorescence image of a representative fractured specimen used for calculation of dimensional data, such as tag length, tag diameter, and tag surface area.

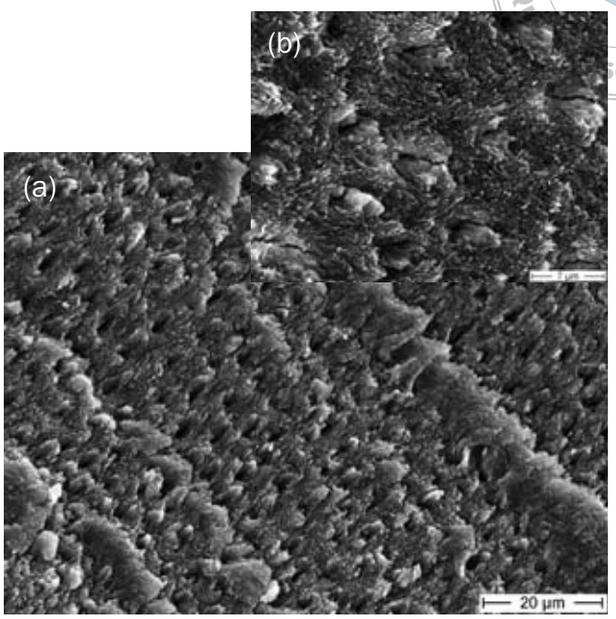

Fig 5 SEM image of the cavity floor of a fractured Class I specimen under vacuum (a) and magnification of the fracture surface morphology (b) exhibiting exposed collagen structures.

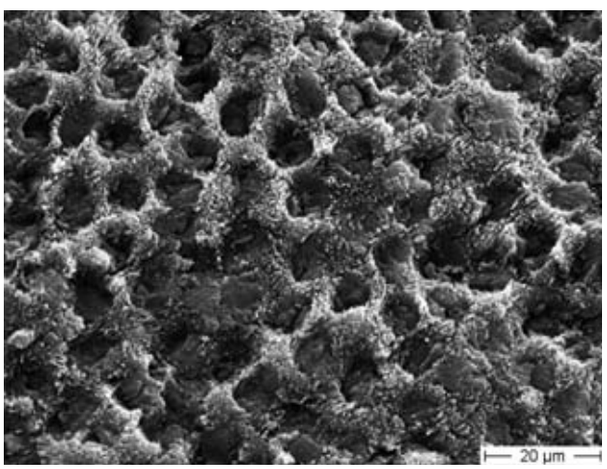

Fig 6 SEM image of the cavity floor of a fractured Class I specimen exhibiting presealed dentin tubules and exposed collagen structures.

at the interface to dentin. Figure 3 shows the completely filled dentin tubules with stained adhesive. Figure 4 represents a CLSM-3D projection of an exemplary infiltrated adhesive layer for calculation of dimensional data such as tag length, thickness, and surface area.

Figures 5 and 6 represent fracture surfaces of Class I cavity floors with and without vacuum application. Both images clearly exhibit an adhesive fracture mode within the hybrid layer, regardless of the presence of resin tags. In Fig 5, the empty tubules can be seen because of tag pullout during fracture. The presealed dentin tubules of the no-vacuum group are shown in Fig 6.

DISCUSSION

Microtensile bond strength decreased with an increasing C-factor. Low C-factor preparations produced superior bond strength compared with the high C-factor cavity preparations. The effect of thermocyclic fatigue differed from low to high C-factors. In the Class I cavity scenario, the µTBS decreased significantly after thermocycling regardless of the presence of resin tags. On flat surfaces, only the vacuum-infiltrated group showed a significant decrease after thermocyclic loading. Within the different preparation types, the presence of resin tags did not lead to significantly different

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results compared to the no-vacuum substrates. On the basis of these findings, the null hypothesis can be partly accepted, since using a self-etching adhesive, resin tag formation does not quantitatively affect dentin adhesion. A reduced μTBS was observed only in low C-factor situations after thermocyclic loading.

Depending on the applied adhesive concepts, the literature provides a wide span of μTBS data. Applying a similar preparation procedure on flat specimens, Sidhu et al[21] reported values for all-in-one adhesives up to 65 MPa. They measured a μTBS for G-Bond of 39.7 MPa. Other mild self-etching adhesives have been estimated between 30 and 50 MPa.[23] De Munck et al[4] measured up to 51 MPa for one-step self-etching adhesives in occlusal Class I cavities. A reduced bond strength is expected in high C-factor Class I cavities because of an increased stress state through polymerization shrinkage.[17,26] The data under investigation here correlate with literature findings in Class I cavities but not on flat surfaces. One reason might be found in the highly technique-sensitive nature of self-etching adhesives. A susceptibility to phase separation has been reported for hydroxyethyl methacrylate (HEMA)-free, one-step, self-etching adhesives.[24] Thus, void formation within the hybrid layer might result in a reduced μTBS.[25] In addition, the sensitive smear-layer–modifying and hybrid-layer–forming potential of mild self-etching adhesives account for an increased technique sensitivity.[13] One key influence on the final bond strength is the intensity and duration of the air-drying step during adhesive application.[3,16]

Hybrid layer thickness increased after vacuum infiltration. However, the measured values for the hybrid layers for groups 1 and 3 correlate with the literature. A layer thickness of 0.7 to 1.5 μm is reported for self-etching adhesives, with mild adhesives producing a rather thin layer.[13] Tag length does not correlate with the literature due to the study goals of complete formation or avoidance of resin tags. A resin tag length of 10.82 μm was measured on presealed substrates. This value was derived from residual tag rudiments, as shown in Fig 2. The measurement of tag surface area at a depth of 5 μm, however, led to an area coverage of only 3.14% of the total dentin surface. After vacuum infiltration, the surface area increased up to 24.68% and tag length to 87.81 μm. Assuming a properly polymerized presealed adhesive, no additional monomer would contribute to chemical bonding at the resin/resin interface. Theoretically, the effective bonding area in the no-vacuum groups would be reduced about 3.14%. In consequence, the estimated μTBS for group 1 might increase from 92.7 to 95.7 MPa.

Because of the low pressure from vacuum infiltration, not only the tag length but also the tag diameter increased significantly from 2.09 to 2.37 μm. As a result of the reduced acidity involved (mild self-etching adhesive), no cone-shaped tags or lateral tag branches could be observed, as has been described by Ferrari and Davidson.[6] Whether or not the orientation of the resin tags substantially influences bond strength remains a question. Figure 3 shows a tubule orientation of approximately 20 degrees to the adhesive layer. Lang et al[14] found no statistical difference in tensile bond strength comparing tubule orientations of 0, 45, and 90 degrees. Gwinnett measured a dependency of resin tags on shear bond strength.[10] Of course, the stress relations under shear loading are different from the tensile stresses. Under tensile conditions, dimensional changes attributable to polymerization shrinkage have to be considered. For monomers such as UDMA, a volumetric shrinkage of 5.3% has been reported.[1] Assuming a polymerization shrinkage of 5.3% in volume for the adhesive under investigation, a remaining gap size of 0.125 μm can be calculated between the resin tag and the tubule walls. These gaps reduce microretention within the dentin tubules and thus might explain the lack of influence of resin tags on the total bond strength. Figure 5 shows the empty tubules resulting from tag pullout during fracture. On the other hand, tag diameters may contribute more to shear bond strength because they must actually be broken during the shear process. This is not the case when perpendicularly arranged tags are pulled out during tensile testing.

After thermocyclic loading, Class I cavities showed a significant decrease in μTBS, but the low C-factor preparations exhibited a less deleterious effect. In addition to the stress state arising from polymerization shrinkage, thermal changes of the complex also stress the preparations. According to reports, the differences in the coefficient of thermal expansion between tooth structure and resin restorative materials might induce degradation of the resin/dentin interface.[8] On the other hand, some authors have hypothesized that especially filled adhesives infiltrate the surface collagen network, forming a low elastic modulus hybrid layer that could work as a stress absorber and allow for more homogeneous distribution and relief of thermal stresses.[4,15] An in vitro investigation involving Class II slot cavities showed no degradation after 300,000 cycles of combined mechanical and thermal fatigue.[15] Another study discussed the influence of water storage on long-term degradation of Class I cavities. The authors observed a worse long-term strength performance for one-step self-etching adhesives compared with conventional three-step etch-and-rinse systems.[4]

From the results shown here, a possible conclusion is that the formation of resin tags does not influence the bonding strength of the one-step self-etching adhesive under investigation. However, durability and longevity of the adhesive interface are major concerns, and degradation of the hybrid layer is repeatedly described in the literature.[7,11,23] A plasticizing effect of water on adhesive polymers arises from water sorption through intrinsically wet dentin, which results in nanoleakage and hydrolysis of especially hydrophilic resin components.[2] Degradation within the hybrid layer is further driven by breakdown of unprotected collagen fibrils via the activation of host-derived matrix metalloproteinases.[19] However, because of incorporation of functional monomers in self-etching adhesives, chemical bonding to the anorganic phase of dentin might compensate for or prevent hybrid layer degradation over time. With that premise arises the question of whether or not resin tags do contribute to dentin adhesion in terms of long-term stability. Additional in vitro fatigue studies or long-term clinical trials should be conducted to further evaluate the role of resin tag formation on bond strength.

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Clinical relevance: Using mild one-step self-etching adhesives, short-term dentin adhesion is not influenced by formation of resin tags. The long-term outcome might be different.