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# Effects of polymerization contraction on interface's µTBS of luting material and dentin

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Abstract Polymerization contraction of composite resin luting materials is known to produce high stresses in the interfaces being cemented that are described as perpendicular to them. This study describes the effect of shearing strains of curing luting materials on microtensile bond strength (µTBS) of interfaces. A flat surface of labial dentin of bovine incisors was exposed and teeth randomly assigned to A (n=12) or B (n=6) groups. Adoro rectangular  $(2 \times 3 \times 11 \text{ mm})$  restorative composite resin blocks were cemented (Excite DSC+ Variolink II) completely (group A) or partially (group B, only on extremes and center) occupying luting space. After visible light curing, stick compound bars were sectioned perpendicular to interface and submitted to tension until detachment. µTBS decreased from the center to the extremes in group A (Spearman tests p < 0.0008) and not in group B, where µTBS was higher in extremes than in correspondent locations in group A and equivalent to that in group A in the central location. Weibull's analysis showed that m modulus and characteristic stresses also decreased from the center to periphery of restorations in group A. Mechanical resistance of bonded interface of a luting material and dentin decreases peripherally, and this reduction is caused by polymerization contraction.

**Keywords** Polymerization contraction · Luting · Contraction stress · Weibull's analysis · Microtensile test

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#### Introduction

In adhesive dentistry, resin-based luting cements are commonly used for ceramic- or resin-based esthetic restorations [1]. Despite of their clear advantages regarding their superior mechanical resistance, the possibility of accomplishing adhesion both to tooth and restoration, the compatibility with other materials, or the many colors in which they are available, polymerization contraction stresses have been an issue nearly since their introduction [2] because they have been shown to potentially have a detrimental effect on the adhesive strength of the composite cement [3, 4].

These stresses were described in 1991 as affecting the bond to dental substrate instead of its cohesive strength because attachment to dentin was not yet formed when the hardening stress developed [3]. Polymerization stresses in light-cured composite resins develop fast with conventional light polymerization and extremely faster when high output lights are used [5–9]. As an example, stress–building rates of 500-µm layers (C factor=5) of dual–cured resin–based luting materials have been measured to range 9 to 31 MPa/min [4] and to be of 0.32 MPa/s for a 840-µm layer of a restorative composite resin in a cementing situation (C factor=3) [10]. These stresses in resin cement films are supposed to depend on the ability to yield elastically when a force is applied (i.e., on the compliance) of the substrate materials [11].

It is likely that they may have an effect on the interface because of its incomplete development. Although the time required to reach the conversion plateau for polymerization of an actual adhesive is impossible to specify to all resins and lights, it is obvious that different comonomer systems contain different weight fractions of the cross-linking monomers [12] and that the efficacy of the initiator systems varies between materials [13]. Total reaction times (time after which no reaction may occur) of various adhesives have been measured, in anaerobic conditions in order to avoid presence of an oxygen inhibited layer, to range between 46 and 126 s when a 600-mW/cm<sup>2</sup> output was used [14]. In these circumstances, polymerization contraction stresses are formed before completion of maturation of the adhesive.

Presence of high stress can be expected at the interface of a contracting gel mass bonded to a rigid structure [15, 16]. Accordingly, a tangential, shearing component of polymerization contraction stresses has been described in studies using finite–element analyzed (FEA) models of direct restorations [17–20]. These stresses, calculated at the end of the theoretical polymerization process, ranged from *circa* 4 to 6 MPa [21, 22].

Such lateral contributions to volumetric shrinkage [23], that would cause shearing stresses, would not be measured in experimental setups assessing stress or strain perpendicularly to interface [24-26], as in the bonded-disk method [27], in which a disk of composite resin is light-cured while adhesively sandwiched between a rigid glass-base and a flexible glass cover-slip. Shrinkage of the composite resin disk would, in this situation, be restricted to the vertical dimension being the lateral components of stress negligible. Nevertheless, in a photoelastic analysis of cylindrical resin cavities restored in bulk with a resin composite showed, at internal angles, a trend for higher stress values when greater diameters were used [28]. In an FEA analysis in which a perfect adhesion of the cement was assumed, authors state that the substrate would restrict the contraction of the resin cement [29], leading to large compressive stresses of the flaws of the substrate, at the interface with the cement. Such stresses would not be expected to build up if perfect adhesion did not exist but a displacement of the cementing material.

The direction of shrinkage is towards the bonded interface [30–33] or the mechanical interlocking [34], if any is present, because it is the consequence of a complex interplay of the direction of the curing light, the material's attachment, and its thickness [30, 34, 35]. This is especially interesting in cementing resins, where available space for the luting material is almost completely surrounded by two confronted bonded interfaces, near one to each other. Apart from the extremely unfavorable configuration factor of the cementing space, extension of these interfaces has hypothetically to play a role because the luting material will, while curing, act as a unique mass and tend to maintain its cohesion. This is conflicting with the fact that because volumetric contraction the outer extremes of the material will be in theory obliged to approach towards the center.

Whether such shearing strains deteriorate the immature interface is an interesting research question. This report assesses the mechanical resistance (through its microtensile bond strength,  $\mu TBS$ ) of a bonded interface created by a composite resin cementing a rectangular resin composite inlay to dentin. Hypothesis is that, if such strains have an effect on interface, this effect will be proportionally related to distance to the center of the mass.

## Material and methods

Eighteen bovine incisors, stored in distilled water at room temperature for a maximum of 1 month after extraction, were used. Roots were removed (Fig. 1, 1; 3031 CP/N Exakt diamond band saw, Norderstedt, Germany) and each incisor's labial surface was grounded in a grinder polisher (Struers A/S Dap–7, Rodøvre, Denmark) with 500–grit SiC paper until dentin was exposed (Figs. 1 and 2). Pulpal tissue was carefully removed with pliers. Specimens were kept humid during all procedures, with their pulp chamber moist.

Dentin surfaces were etched (total etch, 15 s), washed (15 s), and gently air-dried (5 s). Adhesive (Excite DSC) was applied and solvent was evaporated (gentle air blow, 5 s). Luting material (Variolink II base + catalyst) was applied, as described below, to Adoro parallelepiped



Fig. 1 Preparation of specimens

Fig. 2 Relationship between  $\mu$ TBS and BA, *per* groups. In group A, relationship is not evident because it is masked by the effect of distance. This does not happen in group B, where relationship is obvious, because the effect of distance is not present



(approximately  $2 \times 3 \times 11$  mm) restorative resin blocks previously cleaned with phosphoric acid (total etch, 60 s), thoroughly washed (30 s), and thoroughly air-dried (5 s). See Table 1 for description of the materials used.

To guarantee a consistent cement thickness in all samples, a peripheral rigid spacer plastic sheet (20- $\mu$ m thickness) was used in all specimens. Restorative resin blocks were cemented to exposed dentin following two different protocols, according to which two groups were defined A (*n*=12) and B (*n*=6). In group A, resin blocks were cemented by placing the luting material on the complete extension of their larger aspect, facing dentin (Figs. 1 and 3, a). In Group B, luting material was applied limited to small portions exclusively in incisal, central, and gingival parts of the larger aspect of the restorative resin block (Figs. 1 and 3, b). Extreme care was taken to ensure that such portions of cementing material were restricted to extremes and center of the respective cementation space by using only limited amounts of cementing material.

Excess material was removed, and cements were evenly visible–light-cured (800 mW/cm<sup>2</sup>, Optilux 501 KerrHawe SA, Bioggio, Switzerland) with three separate light applications (20 s each) at incisal, central, and gingival locations, in both groups.

After 24 h of storage in a 100% humidity chamber, restored teeth were sectioned perpendicularly to bonded interfaces (3031 CP/N Exakt, Norderstedt, Germany) to obtain rectangular compound (Adoro–cement–dentin) samples (Figs. 1 and 4, a, b) which were glued (SuperGlue 3 gel, Madrid, Spain) to a rigid tensile device and submitted to tension (Hounsfield HTI 500 N, Croydon, UK, 1 mm/min) until adhesive separation from dentine. Debonded areas were measured with a digital caliper (Mitutoyo 500 series, Mitutoyo UK Ltd., England) with a 0.01-mm definition and examined in a stereoscopic microscope (×40, Leica MZ12, Leica Imaging Systems Ltd., Cambridge, UK) to determine the type of fracture (adhesive, cohesive, or mixed). Only adhesive fractures were submitted to statistical analysis.

Table 1	Materials	used in	the	study

Material	Manufacturer	Composition	Batch
Adoro	Ivoclar Vivadent AG. Bendererstrasse	Paste of dimethacrylates, copolymer, SiO <sub>2</sub> , catalysts, stabilizers, and pigments	J11663
Total etch	2. 9494 Schaan (Liechtenstein)	37% phosphoric acid (gel)	A11254
Excite DSC		Total etch 37% H3PO4 adhesive: mixture of dimethacrylates, alcohol, phosphonic acid acrylate, HEMA, SiO <sub>2</sub> , initiators, and stabilizers	B00502
Variolink II translucent		Base and catalyst: dimethacrylates, inorganic fillers (silica, barium glass, ytterbium trifluoride), catalysts and stabilizers, pigments	K00889

Fig. 3 Regression analysis of  $\mu$ TBS to BA, for all pooled specimens. Relationship between  $\mu$ TBS and BA is weak ( $r^2=0.12$ ), but statistically significant (ANOVA's p=1.3E-7)



# Preparation of data

## Microtensile test results

The force (N) required to adhesively detach each sample was annotated and transformed to  $\mu$ TBS (in megapascal) by dividing it into the magnitude of the adhesive surface being tested (BA, in square millimeter). Results confirm the known relationship between BA and  $\mu$ TBS [36–43], which has been described as not proportional (see Fig. 2). Mean (SD) BAs of valid samples of both A and B groups together were 0.72 (1.18)mm<sup>2</sup>, ranging from 0.34 to 1.27 mm<sup>2</sup>. To preclude the anticipated bias that such differences in BAs among samples would cause on  $\mu$ TBS results, an inverse regression of  $\mu$ TBS to BA of all valid samples (groups A and B together) was calculated (SPSS 15.0, SPSS Inc., Chicago, IL, USA; Fig. 3). Underlying principle is that, if microtensile result variations would have been caused only by BA differences among samples, all data should be exactly situated on the regression line. Actual vertical distances (residues) of each point to regression line must be therefore caused by other factor(s). Principal factor is, in our protocol, the independent variable, i.e., the distance to the gingival end of restoration.

In our study, 18 bovine teeth were used. It is likely that diversities between them existed (age, alimentation, health condition of animal, or other individual dissimilarity) that could cause differences in microtensile results. This study assesses intratooth variations in microtensile results, caused

Fig. 4 PResid values, distributed *per* PDistances and groups. It can be seen how values increase from 0% PDistance to center (Spearman's rho=0.65), and decrease from centre to 100% PDistance (Spearman's rho=-0.61), in group A



by the site of the measure related to an origin (gingival end of restoration). To make results comparable among specimens, normalization was done, consisting in transforming each residue to its percentage value in its specimen (PResid). Maximum value in each specimen was assigned a value of 100, and all other were considered as a fraction of this value. In our hypothesis, 100% values would tend be around and close to centers of specimens.

#### Distances of samples to gingival end of luting material

In our study, the independent variable is the distance of each sample to a given location (distance), specifically the gingival extreme of the luting material. The theorized effect on microtensile results is supposed to be distance dependant.

Again, diversities exist between teeth that did cause differences in total length of the layer of luting in group A: two specimens had a length of 10 mm, three of nine and seven of eight. For this reason, to make specimens comparable, distances of samples were transformed to their percentage value within its specimen. Larger distances of samples placed at furthest (incisal) extreme are assigned a value of 100, and all other values of Dist in the same specimen are rated as a percentage of this value into the normalized variable PDistance.

## Statistical analysis

### Intragroup analysis

Correlation between PResid and PDistance was tested in the first (0% to 50%) and second halves of PDistances (50% to 100%) through Spearman's nonparametric tests.

### Intergroup analysis

Mann–Whitney's U test was used to compare PResid values between groups A and B for incisal (PDistance=100%), central (PDistance=50%), and gingival (PDistance=0%) locations.

#### Weibull's analysis

Tensile bond strength tests to dentin of resin–based adhesives show high variability that cannot always be controlled throughout the experiment [44]. In this report, an important part of such variability, caused by fluctuations in specimens size that vary the probability of finding a larger defect that would act as a stress breaker [36], is controlled *via* the adjustment of microtensile test results to bonded area (see above).

However, the most important remaining source of variability is size of defects [44]. Weibull [45] described a function valid to calculate the survival probability (Ps) at any defect tensile stress ( $\sigma$ ), as Ps = 1-exp ( $\sigma/\sigma_0$ )*m*, where the dimensionless constant *m* (shape parameter, slope, or Weibull's modulus) is related to the variability of  $\mu$ TBS values (actually to the one of PResid values, in this report). The higher *m* values are, the more predictable is the behavior and a closer grouping of the fracture stress values can be expected.

Constant  $\sigma_0$  (characteristic tensile bond strength or characteristic stress or scale parameter [46, 47]) is related to the extension of the distribution along the stress axis. It is the fracture stress that we can expect that 63.2% of the samples reach (i.e., at this stress, the probability of failure is p=0.632). Higher values of this parameter match with higher dentin bond strength. In our study, stresses at fracture of interfaces are computed as the fractional ranks of distances to the regression line, i.e., PResid *per* specimen, and are given in percentages.

Weibull ++7 software (Reliasoft Corporation, Tucson, AR, USA) was used to find the two parameters m and  $\sigma_0$  (and their 95% confidence interval (CI)).

Critical failure ( $\sigma_{Pf=0.05}$ ) PResid was also calculated. This will be the PResid at which 5% of the specimens would fail and is a measure of the adhesive performance and of its reliability. In our study, these values are the fractional ranks of residues (PResid), *per* specimen, and are given in percentages.

#### Results

Numbers of valid and lost specimens are shown in Table 2.

Regression of  $\mu$ TBS to BA (all samples pooled, Fig. 3) results was:  $\mu$ TBS = 10.8 + 6.9/BA, with  $r^2$ =0.12 and analysis of variance (ANOVA's) regression p = 1.3E-7.

Figure 4 shows PResid values, distributed *per* PDistances and groups. Table 3 shows Tukey's hinges of PResid values, *per* groups, for the more relevant PDistances (0%, 50%, and 100%).

### Intragroup analysis

Spearman's rho nonparametric correlation test results (95% CI) for the first half (0% to 50%) of PDistances in group A was rho=0.65 (0.54 to 0.75), with a significance p=0.1E-15, and for the second (50% to 100%) half rho=-0.61 (-0.71 to -0.48), with a significance p=5.2E-13. Both correlations were significant.

## Intergroup analysis

Results of Mann–Whitney's U tests comparing PResid values between groups A and B, for 0%, 50%, and 100% PDistances, showed that differences were statistically

Table 2 Number of samples, per groups and PDistances

PDistance	Group A				Group B		
	Valid	Lost		Total	Valid	Lost	Total
		С	М			Т	_
0	13	11		24	12		12
11.1	4			4			
12.5	6			6			
14.3	14			14			
22.2	4			4			
25	6			6			
28.6	14			14			
33.3	4			4			
37.5	6			6			
42.9	14			14			
44.4	4			4			
50	6			6	11	1	12
55.6	4			4			
57.1	14			14			
62.5	6			6			
66.7	3		1	4			
71.4	14			14			
75	6			6			
77.8	3		1	4			
85.7	14			14			
87.5	6			6			
88.9	1	3		4			
100	14	8	2	24	12		12
Total	180	22	4	206	35	1	36

*PDistance* distance to gingival margin of restoration (in percent), C samples lost during cutting, M samples lost during manipulation, T samples lost during testing

significant for 0% and 100% PDistances (p=0.0008 and p=0.0001, respectively) but not for 50% (p=0.15).

#### Weibull's analysis

Table 4 shows Weibull's analysis results (Weibull's modulus *m*, characteristic stress  $\sigma_0$ , and critical stress  $\sigma_{Pf=0.05}$ , for

each PDistance having five or more valid values; see Table 2). All parameters decreased from the specimens located in the centers of the restorations (where PDistance= 50%) towards gingival or incisal extremes (PDistance=0% or 100%; Fig. 5)

### Discussion

Bovine dentin has been accredited as a suitable substitute for human one [48–50]. It was used on purpose in this report because the magnification of the dimensions: to cement restorations up to 10 mm in length on flattened dentin surfaces required labial surfaces of specimens clearly larger than the ones normally found in human teeth. As the hypothesized effect is distance–related, larger specimens magnified the hypothesized effect correspondingly. Whether the level of decreasing in  $\mu$ TBS found in this report would be the same in clinical circumstances has still to be established.

Height of luting space was kept alike for all specimens by the use of a plastic sheet  $(20\,\mu\text{m})$  used as spacer. Not only did these sheets guarantee a regular height of luting space but also helped to maintain it throughout the curing process: it is likely that the polymerization contraction stress of the luting material would otherwise produce a central bending of the restorative resin block beams towards dentin and reversely of dentin towards resin blocks.

The volumetric contraction of a composite resin, which is normally distributed in three dimensions, is possibly converted into only one direction when the C factor increases (i.e., when the wall–to–wall distance of the luting space decreases). Feilzer et al. [51] explain this by a decrease of the flow capacity parallel to the walls of the resin materials at high C factors (as the ones found in luting spaces) and state that the remaining volumetric contraction left a stress along the bonded surfaces. This stress would be, in our study, responsible for the reduction of  $\mu$ TBS.

Contraction stress of luting materials possibly has no detrimental effect on the cohesive strength of the composite resin cement [3] because bonding to dentin is immature

Table 3 PResid results	PDistance	Distance Tukey's hinges						
		Group A			Group B			
		25 (Q1)	50 (M)	75 (Q3)	25 (Q1)	50 (M)	75 (Q3)	
<i>PDistance</i> distance to gingival margin of restoration (in	0	12.5	15.8	25.0	46.1	63.6	72.1	
percent), $Q1$ first quartile, M	50	81.3	90.6	94.4	62.9	76.2	89.0	
second quartile (median), Q3 third quartile	100	10.5	15.3	31.3	50.0	64.3	75.9	

#### Table 4 Weibull's analysis results

PDistance	Group A		Group B			
	m (95% CI)	σ <sub>0</sub> (95% CI)	$\sigma_{ m Pf=0.05}$	m (95% CI)	$\sigma_0$ (95% CI)	$\sigma_{ m Pf=0.05}$
0	1.6 (1.1 to 2.3)	23.9 (16.6 to 34.6)	3.6	2.3 (1.4 to 3.8)	63.9 (48.6 to 84.0)	17.2
12.5	3.1 (1.8 to 5.3)	39.2 (29.1 to 52.8)	14.9			
14.3	1.9 (1.2 to 3.0)	30.0 (22.2 to 40.5)	6.3			
25	1.3 (0.6 to 2.8)	53.9 (26.5 to 109.6)	5.6			
28.6	3.9 (2.6 to 5.8)	49.0 (42.5 to 56.5)	22.8			
37.5	6.7 (33 to 13.7)	91.3 (80.1 to 104.0)	58.7			
42.9	5.1 (3.3 to 7.7)	76.03 (68.2 to 84.8)	42.3			
50	11.2 (5.8 to 21.7)	92.8 (86.1 to 100.1)	71.2	3.7 (2.2 to 6.1)	82.4 (69.4 to 97.8)	36.8
57.1	11.8 (7.9 to 17.7)	91.0 (86.8 to 95.4)	70.8			
62.5	3.4 (1.8 to 6.5)	76.7 (59.5 to 99.0)	31.8			
71.4	4.3 (2.7 to 6.9)	90.8 (79.8 to 103.3)	45.4			
75	2.0 (1.0 to 3.9)	42.8 (28.0 to 65.5)	9.8			
85.7	1.9 (1.3 to 3.0)	36.5 (27.3 to 48.7)	7.8			
87.5	1.4 (0.7 to 2.7)	25.4 (13.9 to 46.3)	3.1			
100	1.4 (0.9 to 2.0)	24.7 (16.4 to 37.2)	2.8	4.0 (2.5 to 6.4)	68.8 (59.3 to 79.8)	32.9

Characteristic stress and critical failure stress are PResid values and are given in percentages

*PDistance* distance to gingival margin of restoration (in percent), *m* (95% CI) Weibull's modulus and 95% confidence interval,  $\sigma_0$  (95% CI) characteristic stress and 95% confidence interval,  $\sigma_{Pf=0.05}$  critical failure stress

before the stress development starts. This may lead to incomplete bonding and sealing of the cementing material, which will cause the effect identified in our study.

The confounding effect of the eventual bending of the restorative beam is apparently in contradiction with our results, first, because the plastic spacer was placed along all peripheries of restorations. Second, in the central position (PDistance=50%) of restorations of both groups, maximum  $\mu$ TBS is attained, and the greatest Weibull's modulus, characteristic stress, and critical stress are found. Results showing that, in group B, where cement was placed separately in gingival, central, and incisal locations, differences in PResid were not significant between locations (Kruskal–Wallis test p=0.14) and that differences (difference of medians is roughly of 50%) were significant between groups A and B for 0% PDistance (p=0.0008) and for 100% PDistance (p=0.0001) indicate it.

Polymerization could also cause part of our results. All restorations were cured as stated in the "Materials and methods" section: with three light periods (20 s.) at incisal, central and gingival locations. Although it would still be possible that central positions (PDistance=50%) would have received more radiation, the possible effect of this is in contradiction with our results, for the same reasons as indicated above.

Microtensile test results are known to be influenced by BA size of the sample [36–42].  $\mu$ TBS of any sample can be

separated in two components: one that can be explained by its BA and one that cannot. Both are identified, in our report, through a nonlinear regression of  $\mu$ TBS to BA for all samples. ANOVA test would not be an appropriate test because one of its assumptions is that this relationship would be proportional, as in some previous reports [37]. But it has also been described as inverse [40], logarithmic [43], or exponential [38]. Our results are that it is inverse (Fig. 3).

The fraction of the  $\mu$ TBS tests results that is not explained by BA is called residue in our protocol. If all residues were equal to 0, all results would have been exactly aligned along the regression line, all variations of  $\mu$ TBS would have been explained by BA differences, and no effect of distance to the center would have been found.

Regression coefficient of this line was very low  $r^2=0.12$  while ANOVA's regression probability was highly significant, p=1.3E-7. We explain this low regression value because there are two known sources of variation in calculations, apart from the intrinsic biological variability: BA and the effect of distance to the center.

A stronger  $\mu$ TBS to BA relationship can be noted in Fig. 2 for group B than for A. Samples in group B were obtained from separated portions (gingival, central, incisal) of luting, where no effect of PDistance was expected, while in the B group an effect of the distance to the center was present as the luting material was continuous from incisal to gingival limits.

Standard deviation of microtensile test results of stick samples, as the ones used in this report, is known to be higher than with other ways of sample preparation [52, 53]. In this research, an effect was demonstrated even with a very high standard deviation in PResid results. This important divergence was caused by the intrinsic biological nature of samples and because of the relative low values of PResid, the vertical distances to regression line, for each sample, excluding the  $\mu$ TBS value that could be explained by BA (the vertical distance of line to cero, at that point).

Residue values were transformed to their percent fraction, for each specimen, into PResid-independent variable. Distance of each sample to gingival outermost of restoration was also transformed to its percent fraction for each specimen, into the PDistance-independent variable. These transformations make interpretation of results more complex but were necessary because in our protocol it was not possible to produce specimens having exactly the same distance. Pertaining to test results, an estimated conventional µTBS value could have been calculated assuming all samples had a BA=1 mm<sup>2</sup>, thus circumventing the inconvenience of transforming µTBS results into PResid variable. Such values are not presented in this report because the objective was to assess the existence of the hypothesized effect. In further studies where performance of distinct materials was to be compared, testing of differences in percent decreases could be appropriate. It can be anticipated that material's distinctive characteristics should produce such differences.

Invalid or missing samples appeared principally in extreme (lower or higher, see Table 2) PDistances. Most of them happened during cutting procedure, probably caused by vibrations of the cutting band that distressed previously weakened interfaces. A small amount of invalid samples was caused by manipulation and occurred also in PDistances separated from the center. These distributions are in accordance with the initial hypothesis: interfaces are less resistant as PDistance increases.

All debonded samples (Table 2) were excluded from analysis. In group A, 22 were prematurely debonded during cutting process and four during manipulation, in total, rendering 180 for tensile testing. In group B, a total of 35 valid samples were analyzed, after one was lost during testing. Our explanation is that centripetal contraction stresses produced an important impairment of bonded interface in some specimens, particularly the ones located in extreme PDistances, in group A.

These samples were not analyzed because otherwise an arbitrary unreal  $\mu$ TBS value (normally, zero) would have then be assigned to them. They debonded because their mechanical resistance was low but not zero. Real value remained undetermined. In addition, in the event that debonded samples had been included in the analysis, such values would have been considered expected values. Mean—or median—PResid values are different from zero, and extremely low values, the ones that one would expect debonded specimens to have, are different. To exclude them from analysis is to acknowledge that they were exceptional and do not represent their group.

Nonparametric correlation tests showed that at both the first (gingival to centre) and the second (center to incisal) halves, there was a statistically significant correlation between PDistance and PResid. PResid values significantly decreased from the center to the outside edges of restorations.

Significant differences of PResid, between groups A and B, for 0% and 50% PDistances were found. In group B,

**Fig. 5** Weibull's analysis results, *per* PDistances. *m*: Weibull's modulus,  $\sigma_0$ : characteristic stress. Note *Y*-axis scales: 0 to 12 (*left*, *m* modulus) and 20 to 100 (*right*,  $\sigma_0$ ). Modulus and characteristic stresses decrease from the center to extremes in group A and not in group B



where luting was discontinuously placed in gingival (PDistance=0%), central (50%), and incisal (100%) positions,  $\mu$ TBS values at the extreme boundaries of restorations were higher than the correspondent ones in group A. This excludes local tooth characteristics that would reduce  $\mu$ TBS values. In central positions, differences were not statistically significant.

Weibull's analysis was performed including only PDistances counting at least five valid PResid values. These distances were evenly distributed and included extreme and central values. Results showed that Weibull'sm modulus consistently increased towards the center of specimens (Fig. 5). This modulus is related to predictability of behavior and grouping of PResid results. Same performance showed characteristic stress  $\sigma_0$ , directly related to tensile bond strength. Results were more inconsistent as measure moved towards outer boundaries. This is a new concept: not only do  $\mu$ TBS values decrease, but reliability declines when measured apart from the center of the curing mass.

It is difficult to project these results to clinical consequences or establish direct considerations. It has to be taken into account that the restorations used in this study were parallelepiped in shape and had their largest face cemented to dentin.

Although our results reveal that there was a shearing effect of polymerization contraction on  $\mu$ TBS values, it is difficult to disclose the mechanism. Flow of curing material during its gel phase was greatly restricted by its attaching to restorative and dentin walls, a quite relevant limitation in a 20- $\mu$ m luting space. As noted above, curing time for a typical adhesive can be 20 to 30 s [12] or even higher [14, 54]. This confirms the idea that micromechanical attachment of luting to form both restorative and tooth interfaces is undeveloped while stress buildup is probably faster [4–11].

While using the bonded disk method, Watts and Marouf [24] stated that if small specimen diameters are used or if composites are bonded to a compliant restorative material, then the configuration factor (C) is altered, and some lateral contribution to volumetric shrinkage may occur that is not uniaxially measured. Local shrinkage–strain will translate into an observable macroscopic shrinkage–strain unless significant free volume is induced within the material bulk [55]. In this study, no morphological measurements were effected, but such free volume can correspond with the stress relaxation effect of material's centripetal displacement. Although it has been stated that only uniaxial stresses are clinically relevant [26], this report highlights some potentially important effects of nonaxial stresses.

In conclusion, microtensile bond strength of a visible– light-cured composite resin material to bovine dentin and a prepolymerized resin restorative was reduced in proportion to the distance to the center of the luting space. Weibull's analysis showed that predictability of adhesive behavior of the luting resin and its characteristic bond strength decreased when measure moved from the center to periphery. These differences were not caused by local factors.

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