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Influence of loading types on the shear strength of the dentin–resin interface bonding

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Small differences in the shear bond test can make critical differences in the bonding strength values. The purpose of this study was to compare the influence of the orthodontic-looped wire, stainless steel tape and chisel systems used in shear bonding tests to verify the resistance in the dentin-resin interface. Forty-eight human teeth were used and divided in three groups. The teeth were ground until a flat smooth surface was achieved, that was delimited with an adhesive tape containing a hole of 4 mm diameter. After, the dentine surface was treated with Scotchbond Multi Purpose and the composite Z-100 was applied in layers, through a stainless steel mold. The samples were stored at 37 °C and 100% of relative humidity for 24 h and, then, submitted to 500 thermal cycles. After, they were taken to an universal test machine (Otto Wolpert) with crosshead speed of 6 mm/min. The results were statistically analyzed using a Tukey's test ($p < 0.05$). The orthodontic-looped wire determined the highest values of shear bond (13.33 MPa), following by chisel (7.81 MPa) and stainless steel tape (4.87 MPa). The debonding values depend on a complex stress combination produced during the loading of the samples. Small variations in test methodologies give statistically different values for shear bond strength. Different shear strength methods *in vitro* make the comparative clinical performance of the resin filling materials difficult.

Introduction

The quality promoter of bonding of the dentine adhesives is frequently verified by several laboratory tests, using shear and traction efforts under certain limitations. These *in vitro* tests show that the obtained values hinder the standardization and they disable the direct comparison among different researchers [1, 2].

In agreement with Barkmeier and Cooley [3], clinical tests are considered the most reliable for the study of dentine adhesive behavior, although laboratory tests are also considered valuable in the evaluation of these materials. While, many laboratory studies cannot be extrapolated directly to clinical situations, they are useful in the establishment of clinical study protocols [4]. So, they reinforce the effectiveness of dentine adhesives and demonstrate the behavior of the adhesive systems employs in enamel and dentin.

In vitro tests also have some drawbacks. Previous studies have showed it is impossible to standardize the dental substratum, due to great variation of the dentine structure in the bonding area [5–7]. Other important factors such as tooth face, dentine depth [8], surface roughness [9], surface humidity [10, 11], tooth type [12], surface treatment type [13, 14], thermocycling [15], concentration and type of acid [16], conditioning time [17], load application and differences in the elastic properties of the materials [1], influence also the calculation of bonding values. These factors have equal influence in both traction and shear methods.

Another important consideration is the modifications in the test procedures commonly applied in different investigations seeking the same objective, to determine bonding values. For this reason, analyses of the same material unavoidably produces different data on the bonding resistance [18, 19].

Recently, the ISO (International Organization for Standardization) created a norm [20] in order to standardize the adhesion tests, including the shear test, to the dental structure. In spite of this, the literature still exhibits a discrepency between adhesion tests and the dental structure. Considering this, we thought it reasonable to conduct a study to compare the ISO specified test (chisel) with the non-specific test (stainless steel tape and orthodontic-looped wire), in addition to compare as well, the morphological characteristics of the fractured interface composite-dentin.

Material and methods

This study used the commercial adhesive system recommended for use in enamel and/or dentin, Scotchbond Multi-Purpose Plus (3M Dental Division, St. Paul, MN), and the composite Z-100 (3M Dental Division, St. Paul, MN).

Forty eight human teeth without caries, extracted for orthodontic reasons were used. The roots were sectioned and the dental crowns included in plastic tubes with chemically activated acrylic resin. The vestibular face of the tooth was worn in a vertical polisher (P.F. Dujardin & Co., Dusseldorf, Germany) respectively with 180 and 400 grit sandpapers, until a 5 mm diameter plane area in the dentine surface was acquired. After the preparation of the dentine surface, a circular adhesive tape with a 4 mm diameter central hole was bonded to the dentin. The defined area was treated with Scotchbond Multi-Purpose Plus adhesive system, according to the manufacturer's instructions. Afterwards, Z-100 restorative composite was inserted in the hole of a stainless steel matrix (4 mm in diameter and 5 mm in height), in three layers. Each layer was light cured for 40 s at a light intensity of 530 mW/cm[2] with a unit Visilux 2 (3M Dental Division, St. Paul, MN).

The samples were stored at 37 °C and 100% relative humidity for 24 h. After, they were divided into 3 groups of 16 samples each. Half of the samples from each group were submitted to 500 thermal cycles with 30 s in bathings of 5 °C, 37 °C and 60 °C.

Each sample was submitted to the shear bond test in a universal machine (Otto Wolpert–Werke, Ludwigshafen, Germany), with a cross-head speed of 6 mm/min. Each sample was horizontally housed in a metallic glove (20.5 mm in internal diameter and 20 mm in height) fastened to the superior mordant of the machine. The extremities of either a stainless steel tape (5 mm in width and 10 cm in length) or an orthodontic-looped wire (1 mm in diameter and 10 cm in length) were fastened in the inferior mordant. Both formed a loop that enclosed the composite cylinder bonded to the dentine surface, during the traction effort. The third used system was a chisel form loading device with a 1.0 mm border.

The surfaces of fractured samples of each material group were covered with gold-paladium in a high vacuum (Balzers–SCD 050, sputter coater, Germany) for scanning electron microscopy observation (Zeiss DSM 960, Germany).

Results

Shear bond strength

The results obtained from the shear bond test of the interface between dentin/adhesive system, in agreement with the type of loading and thermocycling, were submitted to variance analysis and the medium values were submitted to the Tukey's test at 5% probability level (Tables I and II).

Morphologic analysis of the dentine surface in the fractured area

When the stainless steel tape was used, the photomicrographs taken of the dentine surface showed that the failure was interfacial fracture between the adhesive and the dentine (Fig. 1). Fig. 2 illustrates the fractured adhesive, filling dentinal tubules and the adhesive residues on the surface. When the orthodontic-looped wire was used, the failure more commonly observed was a cohesive type in the composite (Fig. 5). Fig. 6 shows residues of the adhesive bonded to the dentine surface (cohesive fracture of adhesive). When the chisel was used the observed failure was cohesive fracture of the adhesive (Fig. 3). Fig. 4 shows adhesive and composite residues bonded to the dentine surface.

Discussion

In scientific investigations, different methods and modifications of the same test are routinely used even though the objectives are similar. It is difficult to compare the data and results of investigations because of the lack of technical standardization.

In this laboratory study, the discrepancies were considered when the different shear bond strength

TABLE I Shear bond averages using different loading methods (MPa)

Type of loading	Not cycled average	S.D.*	Cycled average	S.D.*
Orthodontic-looped wire	15,23 a	1,43	13,33 a	0,77
Chisel	9,10 b	1,14	7,81 b	0,64
Stainless steel tape	5,63 c	0,45	4,87	c 0,41

* Standard deviation.
D.M.S. 5% = $1,08244$.
Averages followed by different letters in the column differ from each other at a 5% probability level, when the Tukey's test is used.

TABLE II Shear bond averages of the submitted samples, both thermocycled and non-thermocycled

Procedure	Orthodontic-looped wire average	S.D.*	Chisel average	D.P. *	Stainless steel tape average	S.D. *
Not cycled	15,23 a	1,43	9,10 a	1,14	5,63 a	0,45
Cycled	13,33 b	0,77	7,81 b	0,64	4,87 a	0,41

* Standard deviation.
D.M.S. 5% = $0,90004$.
Averages followed by different letters in the column differ from each other at the 5% probability level, using the Tukey's test.

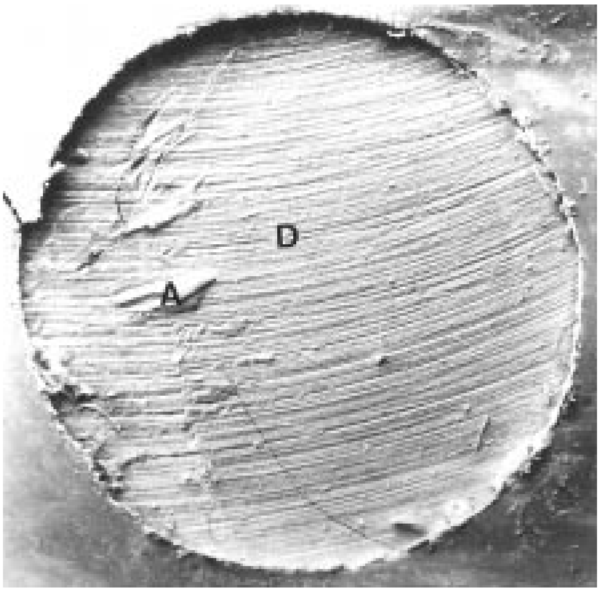

Figure 1 Morphological aspect of the dentine surface after shear testing using the stainless steel tape (20 ×). Adhesive residues (A) stuck in the dentinal surface (D).

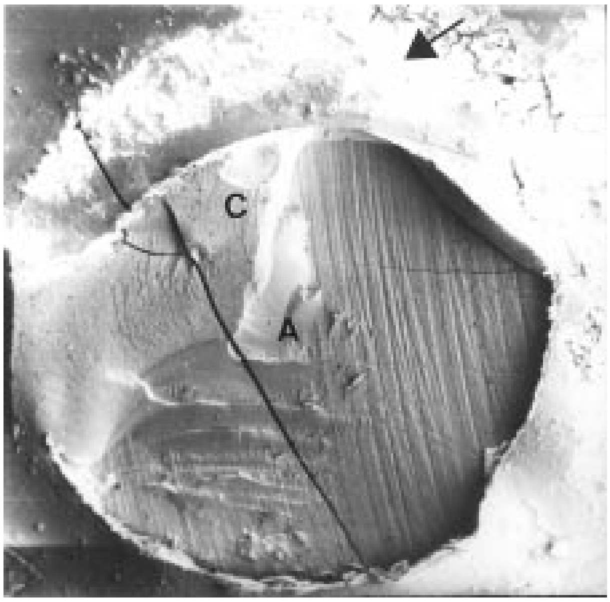

Figure 3 Morphological aspect of the dentine surface after the shear testing using the chisel (20 ×). Adhesive residues (A) and composite (C) on the dentine surface. The arrow indicates loading area.

values was calculated. The data obtained from shear bond strength tests which had different method modifications showed significant differences ($p < 0.05$). The orthodontic-looped wire determined the highest values of shear bond, reaching median values of 15.23 MPa. There are studies (Ishioka and Caputo [21] and Berry and Powers [22]) where this type of loading was frequently used.

The microscopic inspection of the fractured surface showed that the adhesion failures which occurred in certain specimens, tested with the orthodontic-looped wire, were cohesive in the composite (Fig. 5). The resin remained closely bonded to the desmineralized layer of the dentin, forming the hybrid layer [23]. In other samples, the failure was cohesive fracture of the adhesive. This fracture mode left material residue on the dentinal surface and at the entrance of the dentinal tubules (Fig. 6). In these conditions, it is hypothesized that the pattern determined by orthodontic-looped wire was flexion stress. Under these conditions in the initial loading, the tensile and compressive stresses produced in the axial loading would be concentrated in diametrically opposed directions and perpendicular to the interface (Fig. 7). Later, the bonding force in the dentin-resin interface would be completely overcome, promoting cohesive fracture of the composite or cohesive fracture of adhesive. On the one hand, the intensity of the tensile and compressive forces which occur at the interface, can

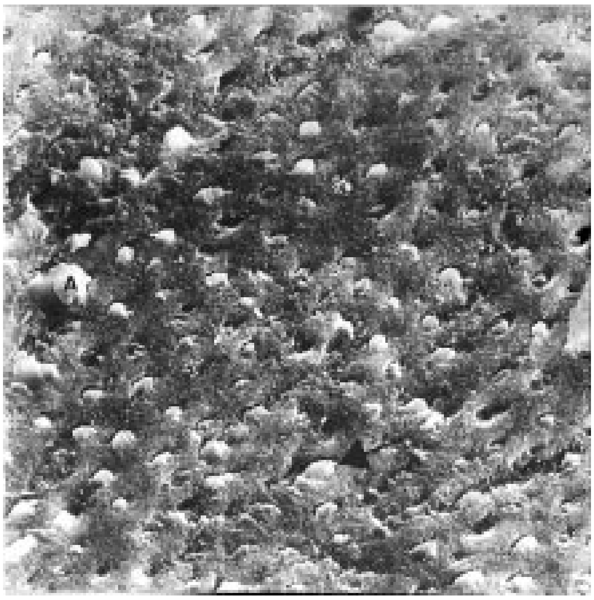

Figure 2 Morphological aspect of the dentine surface after the shear testing using the stainless steel tape (1000 ×). Letter A shows adhesive residue on the dentinal surface. The arrow shows the entrance of dentinal tubules filled with adhesive.

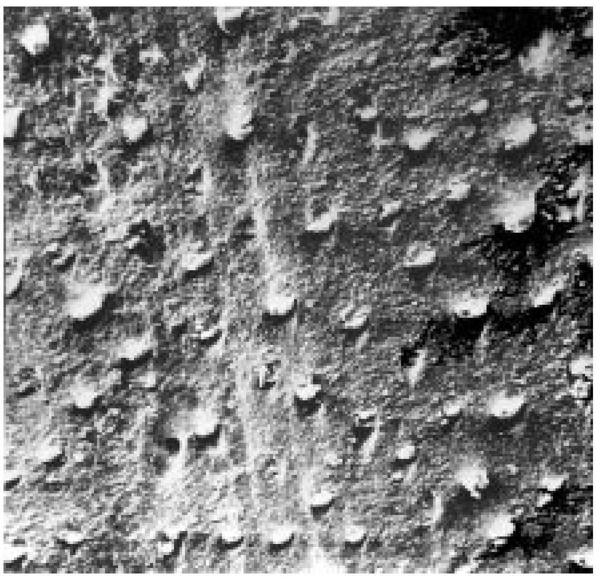

Figure 4 Morphological aspect of the dentine surface after the shear testing using the chisel (1000 ×). In a magnification of the seemingly resin free area, dentinal tubules filled with adhesive can be seen.

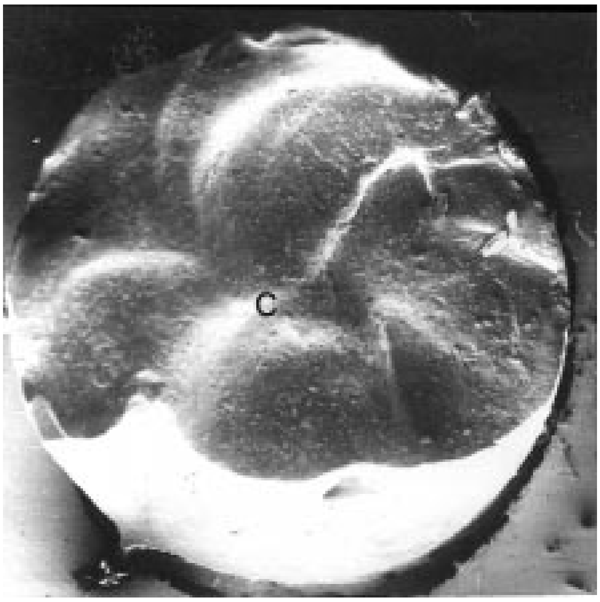

Figure 5 Morphological aspect of the fractured composite after the shear testing using the orthodontic-looped wire (20×). Composite residue (C) on the dentine surface.

grow with increase in the distance between the load application point and the bonding surface. This situation occurs with the increase of the flexion moment value by the distancing between the load application point in relation to the interface [2, 24, 25].

On the other hand, the chisel promoted a cleavage stress, which was initially concentrated on the sub-superficial layer of composite (Fig. 3), localized in the loading area (arrow). At this loading point, the resin fractured and the cleavage propagation reached the interface, causing the tooth-composite debonding (Fig. 8). Moments before the bond rupture, this loading type produced complex stresses, involving cleavage, tension and compression, with median values of 9.10 MPa. Fig. 4, shows that the dentine close to the chisel loading area has resinous protrusions inside the dentinal tubules and fractured resin close to the dentine surface. The fractographic view also suggests that the resinous fragments have been pulled from the inside of the tubule.

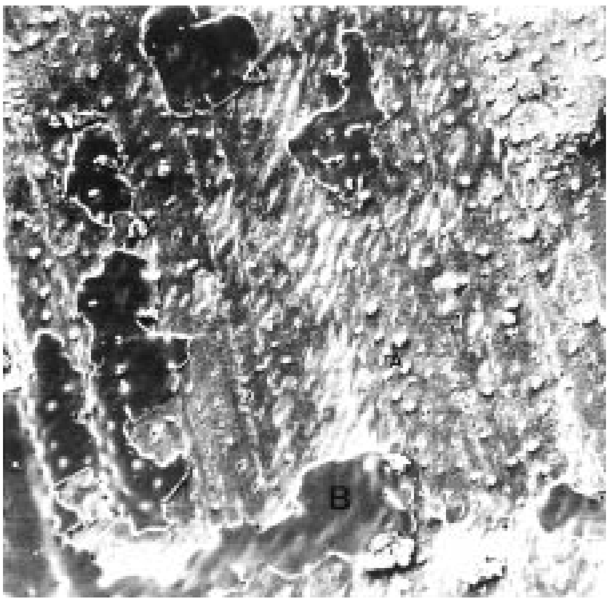

Figure 6 Morphological aspect of the dentine surface after the shear testing using the orthodontic-looped wire (300×). Dentinal tubules filled with adhesive on the surface (A) and adhesive residues (B) in a sample with mixed failure.

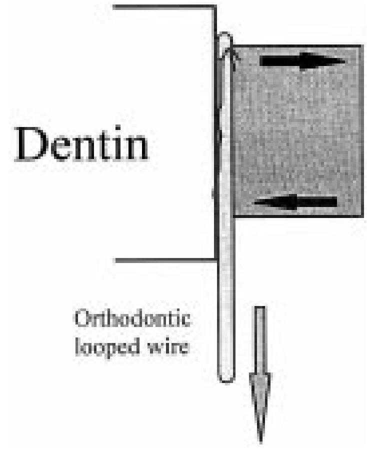

Figure 7 Schematic illustration of the fracture direction caused by the stress in the orthodontic-looped wire system.

The lowest median values of shear resistance (5.63 MPa) were obtained when the stainless steel tape method was used, which indicated that the mechanism of loading in this test were less complex. From this evidence, we can assume that the stainless steel tape created the best conditions for the establishment of the true shear loading test (Fig. 9). Our results confirm the statements of Retief [26], when a rectangular point chisel (contact area similar to the metallic tape) produces smaller shear resistance values than an angulated point chisel, which causes puncture loading (smaller contact area) instead of compression loading.

Therefore, as seen in Fig. 1, the loading with stainless steel tape promoted interfacial fracture between adhesive layer and dentin. This debonding occurred under the influence of sliding along the interface, as a result of the high concentration of tangencial force (parallel lateral forces), similar to that found in the inclined plan. If present, this stress type was not significant in other tests

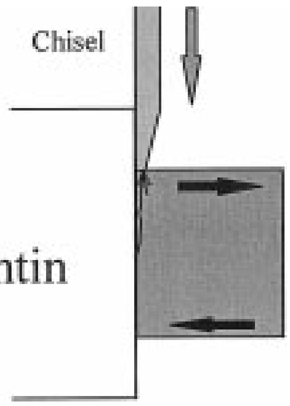

Figure 8 Schematic illustration of the fracture direction caused by the stress with the chisel system.

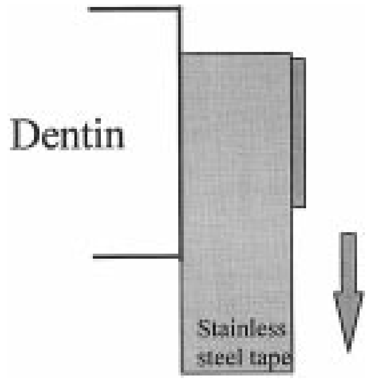

Figure 9 Schematic illustration of the fracture direction caused by the stress in with the stainless steel tape system.

where the loading produced flexion of the composite cylinder (orthodontic-looped wire) or surface cleavage (chisel). Fig. 2 shows the dentinal tubules filled with adhesive, which failured at the dentinal surface level, indicating interfacial fracture between adhesive and dentin. However, this situation does not show the displacement aspect of the resin protrusions outside of the tubules promoted by flexion stress, as verified in the Figs 4 and 6.

The orthodontic-looped wire and the chisel act axially on the composite cylinder, but it was not possible to establish an identical point of load application. This creates a situation with different stresses which consequently produces different debonding values. For this reason, Van Noort *et al.* [2] demonstrated that uniformity of the traction or shear resistance in the interface between dentine and composite resin was not reached because of these different stresses.

For some loading conditions, the interfacial stress is not uniform due to alterations in the specimem geometry and composite modulus of elasticity. Therefore, the bond failure can be initiated in the composite body close to the interface, but not necessarily in the interface, as occurred in the case of the chisel.

The stainless steel tape loading system did not produce a support point (fulcrum or moment) in the composite cylinder nor superficial cleavage. However, it promoted sliding between two surfaces. In this case, the traction and compression efforts produced in the interface are smaller than those obtained from the chisel and orthodontic-looped wire systems. The stainless steel tape loading method perhaps explains the presence of interfacial failure between adhesive layer and dentine, because this structure directly receives the sliding force.

In this study, thermocycling was also implemented in order to submit samples to the critical conditions during the *in vitro* test. The obtained values showed that the thermocycling did not interfere in the results. The orthodontic looped wire loading produced the largest values of shear resistance (13.33 MPa), proceeded by the chisel (7.81 MPa) and stainless steel tape (4.87 MPa) systems. The chisel and stainless steel tape systems were statistically different from one another. When each loading system was individually compared in relation to the thermocycling effect, the loadings with orthodontic-looped wire and chisel were statistically significantly decreased ($p < 0.05$). However, the thermocycling did not have a statistically significant effect with the stainless steel tape loading despite smaller numeric values. The thermocycling produced fatigue in the interfacial adhesion [27]. This differing result probably occurred due to the fact that stainless steel tape is not subject to the complexity of stress produced by loading test. The complex stresses produced in the orthodontic looped wire and chisel systems were affected more significantly by the detrimental thermocycling action.

The results from this investigation can be used by other researchers (as an indication of the phenomenon) who have the objective to evaluate the shear resistance values obtained from different laboratory tests.

Conclusion

The orthodontic-looped wire loading method produced the statistically superior ($p < 0.05$) and largest shear bond resistance, compared to the chisel and stainless steel tape methods, which were statistically different ($p < 0.05$) from one another. This sequence was derived as much in the samples with thermocycling as those without thermocycling.

The thermocycling produced a statistically significant reduction ($p < 0.05$) in the shear bond resistance, except in the stainless steel tape method.

The photomicrographs showed that orthodontic-looped wire loading caused mixed and cohesive failure in the composite.

The more commonly observed failure in the chisel was mixed type with cohesive in the composite resin and in the adhesive layer.

The most common failure caused by the stainless steel tape was the cohesive type in the adhesive layer.

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