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Analysis of the specimen shape and aging protocols in the adhesive strength between dentin and resin composite

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ABSTRACT

The objective of this study was to evaluate the aging protocols and the specimen shape (sticks and dumbells) in the adhesive strength between dentin and resin composite during micro-tensile stress. The specimens were submitted to no aging protocol, mechanical cycling (using macro and micro-rotation methods), thermal cycling and micro-tensile test. The stress distribution for the two specimen shapes was performed by finite elements analysis. Data were submitted to 3-way ANOVA and Tukey Test ($\alpha = 0.05$). There is no statistical difference considering the interactions among the factors: specimen shape, thermal cycling, and mechanical cycling. Also, the mechanical cycling, for both methods, micro or macro-rotation, and the thermal cycling did not affect the adhesive strength of the samples. However, the specimen with the dumbbell shape showed higher adhesive strength (16 ± 3 MPa) than the stick shape specimen (11 ± 2 MPa). The stress distribution in dumbbell shape was more homogeneous than in the sticks. It can be concluded that the aging protocols tested were not enough to degrade the adhesive interface and the dumbbell shape specimen is better to predict the real adhesive strength developed in the interface.

1. Introduction

Even with the increasing development of resin composites, the durability of the bonding interface is still a challenge in dentistry. The achievement of high bond strength is intended not only to support the stress generated by the volumetric shrinkage but also to withstand the challenges inherent to the oral environment, related to chewing [1], pH changes [2], temperature variation [3] and hydrolytic and enzymatic degradations [4].

New materials and techniques are constantly launched on the market, evaluating those materials clinically would be ideal for determining their actual performance; however, clinical studies are complex, time-consuming, run into ethical norms, are difficult to perform and standardize variables. Laboratory studies, on the other hand, have advantages, such as their lower cost and time spent, and the possibility of greater standardization and isolation of variables.

One of the most used laboratory tests in literature for the analysis of adhesive strength is the micro-tensile test, as this type of test provides better stress distribution on the specimen, decreases the defects in the interface and has a lower incidence of cohesive type fractures compared to the macro-tensile test [5]. However, it is still necessary to improve this technique, as it still presents some limitations. Several parameters related to the specimens and the test itself directly influenced the results obtained. The influence of the format of the specimen (cylindrical, hourglass-shape, stick-shape and dumbbell-shape), its area, as well as the way it is made can be highlighted [6–8].

Laboratory studies using accelerated aging of adhesive restorations before the bond strength test have been widely used and considered as an adequate way to predict the performance of restorative materials in the long term. The most used methods for this have been: the storage of specimens in water [9] or different aqueous solutions such as artificial saliva [10], and solution with enzymes from the host [11]; cariogenic challenges and pH cycling [12,13]; and mechanical and thermal cycling [14,15]. These techniques challenge the adhesive interfaces, allowing their laboratory analysis. However, there is no consensus in literature related neither to its standardization nor to its simulation capability of

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what occurs within the oral cavity.

Thus, the need is evident to continue efforts in research that evaluate and standardize protocols for the preparation of specimens for micro-tensile, as well as aging techniques to find effective solutions to predict the clinical performance of materials. The objectives of this study were: to evaluate, *in vitro,* the effect of different accelerated aging protocols on adhesive interfaces, through the bond strength test and micromorphological aspects; and to verify the influence of the shape of the specimen (stick or dumbbell) on the values of bond strength, as well as its variability and stress distribution, through finite element analysis. The hypothesis of the study is that the use of different aging protocols, can alter the bond strength of the adhesive interfaces in different ways and that the shapes of the stick or dumbbell specimens can result in different stress distributions and consequently different bond strengths.

2. Materials and methods

The project was approved by the Research Ethics Committee of the School of Dentistry of the University of São Paulo (FR-318205; Protocol 14/2010). A total of 36 sound molars were ceded by the Human Teeth Bank of the same institution. The teeth were stored in sodium azide 0.02% at 4 °C until the specimens' preparation. After restorative protocol, the teeth were cut in the middle, and each part was included in one of six groups, according to Table 1 (n = 12). Three factors were evaluated in the study: specimen shape (stick or dumbbell); thermal cycling (yes or no) and mechanical cycling (method 1, method 2 or none).

The dental enamel and root were cut using a diamond disc (Buehler Diamond Wafering Blade 11-4245, Buehler Ltd., Lake Bluff, IL, USA) in a cutting machine (Isomet 1000, Buehler Ltd., Lake Bluff, IL, USA) at 200 RPM, under refrigeration. The exposed dentin surface was plain and perpendicular to the long axis of the tooth. The teeth were embedded in acrylic using rigid PVC rings with a diameter of 14 mm and height of 10 mm. The dentin surface was sanded with sandpapers of 220, 320 and 400 grit for 15 s each and 600 grit for 30 s in a polishing machine (DP- Struers, Ballerup, Denmark), to obtain a homogeneous and standardized smear layer. All the adhesive and restorative protocols were done by the same operator, at 23 °C under air humidity of 50%. The teeth were etched with 37% phosphoric acid (Dentsply, Petrópolis, RJ, Brazil) for 15 s, washed with a water/air jet for 15 s and dried with an air jet for 30 s. The adhesion area was delimited using an adhesive tape with a hole of 8 mm in diameter. The dentin was rewetted with 1.5 μL of distilled water for 20 s, and 8 μL of adhesive primer was applied (Scotchbond Multipurpose, 3 M ESPE, St. Paul, MN, EUA), left for 15 s and submitted to an air jet to evaporate the solvent. The adhesive bond (Scotchbond Multipurpose) was applied (8 μL) under shaking with a microbrush; a gentle air jet was used to make a more uniform adhesive layer. The adhesive was photoactivated with an energy density of 10 J/cm^2, using a halogen lamp Optilux 501 (Kerr, Danbury, CT, USA). The resin composite Z100 was inserted in three increments of 1.5 mm each, on the prepared surface. A mylar strip and a glass slice were placed over the last layer before photoactivation to make the surface uniform. Each increment was photoactivated with an 18 J/cm^2.

Table 1
Experimental groups evaluated.

Experimental groups	Specimen shape	Thermal cycling	Mechanical cycling
Group 1	Stick Dumbbell	Without	Without
Group 2	Stick Dumbbell	With	
Group 3	Stick Dumbbell	Without	With macro-rotation
Group 4	Stick Dumbbell	With	
Group 5	Stick Dumbbell	Without	With micro-rotation
Group 6	Stick Dumbbell	With	

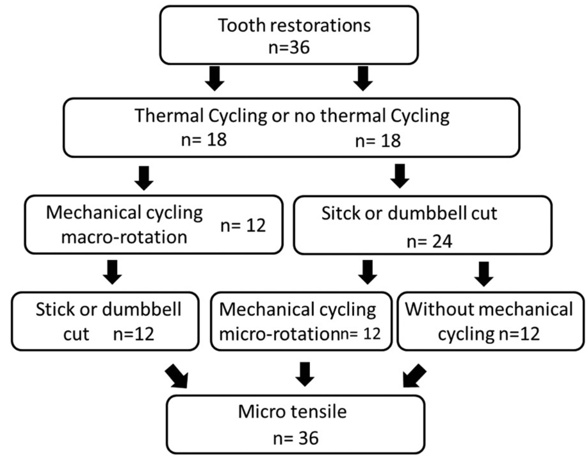

Fig. 1. Flowchart of experiments sequence.

2.1. Specimen preparation and treatments

The restored teeth follow the treatment and preparation described in the flowchart presented in Fig. 1, according to the experimental group. All groups were stored for a similar time to avoid differences regarding hydrolytic degradation due to storage time, so the groups with no cycling treatment were maintained in distilled water during the cycling time of the other groups. The thermal cycling was performed in the teeth before the cut, with 10,000 cycles in a water bath of 5 °C–55 °C, leaving the specimens immersed for 1 min in each bath per cycle. (Nova Ética, LTDA -Brazil). The mechanical cycling with the macro-rotation, the rotation of all the restoration/teeth in one block, previously to be cut in dumbbells or sticks, was performed in the teeth using a dynamic device (Nova Ética, LTDA- Brasil) in distilled water at 37 °C. The PVC rigid rings with the teeth included were coupled to the machine and held by a jaw. A poly-acetate tip moved from the actuator and contacted the occlusal surface of the teeth generating a rotation of 30° in the clockwise direction, with a maximum load of 10 Kgf, followed by a counter-clockwise rotation until relieving the load to zero. The specimens were submitted to 500,000 cycles at a frequency of 4 Hz. The mechanical cycling with the micro-rotation (Microspecimen Former, University of Iowa, USA) was performed with the specimens already cut into stick or dumbbell shapes. The specimens were coupled in mandrels fixed to the machine, and they were turned in its main axis at 4 Hz to induce sinusoidal stress at the external surface of the interface between dentin and resin composite. During the test, the specimens were automatically sprayed with distilled water at 37 °C, and the temperature was kept constant. The specimens were tested until fracturing or reaching 100,000 cycles.

2.2. Micro-tensile test

To obtain specimens with a stick shape, the teeth were cut with a diamond disc (Buehler Diamond Wafering Blade 11-4244, Buehler, Lake Bluff, IL, EUA) at 200 RPM, under refrigeration with cuts of 2-mm-thicker, in mesial-distal and buccal-lingual directions. The base was cut perpendicular to the long axis of the teeth. The cross sectional area of the stick was of 4 mm^2. To obtain specimens in dumbbell shape, one more step was added to this protocol, the sticks were individually machined with a diamond bur (1093 FF, KG Sorensen), under refrigeration. The high rotation turbine was coupled to a device for dumbbell specimen confection (Microspecimen Former), which pinches

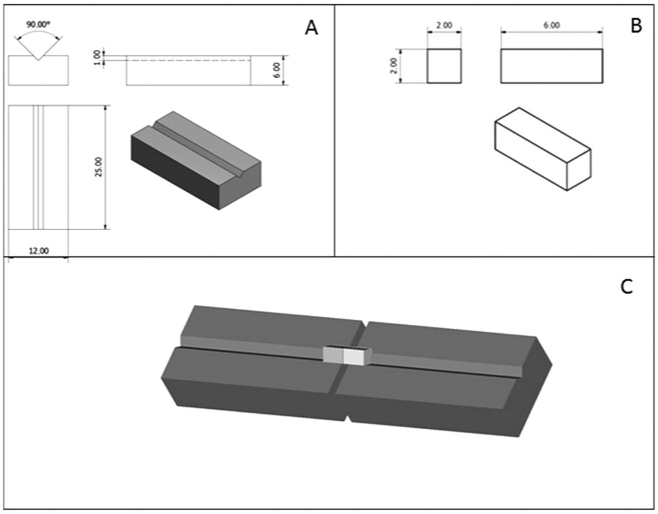

Fig. 2. Geraldeli's holder and stick shape specimen. Technical drawing of the holder (A) and of the specimen (B) and geometry of the set assembled (C).

the specimen, allowing its rotation around its long axis and its movement parallel to this long axis. The central cylindrical portion of the dumbbell shape specimen was designed with a diameter of 1 mm. The diamond bur was changed every two teeth to decrease the stress generated by the bur.

For the micro-tensile test, the stick shape specimens were bonded individually to a Geraldeli device (Fig. 2) using cyanoacrylate glue, which was instantaneously polymerized by a thin layer of dimethyl-p-toluidine. The dumbbell shape specimens were tested using a device named DICKS where the specimens are adapted without the use of glue (Fig. 3). For both the specimen shapes, the size of the transversal section was measured with a digital pachymeter. The micro-tensile test was performed on a Universal Test machine (Kratos, São Paulo, Brazil) at 1.0 mm/min. The Adhesive strength (MPa) was calculated dividing the rupture load for the transversal section area of the specimen.

2.3. Scanning electronic microscopy (SEM)

Three stick and three dumbbell shape specimens representative of each group were metallized with a gold coating, using a high-vacuum sputtering (MED020, Bal-Tec, Balzers, Liechtenstein) for 150 s. The images were performed in scanning electronic microscopy (Quanta 600F, FEI, Brno, República Theca) with a magnification of x 35.

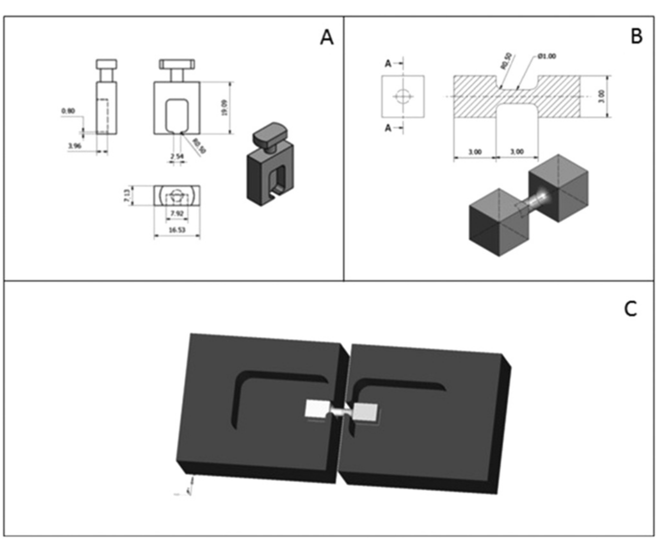

Fig. 3. Dick's holder and dumbbell shape specimen. Technical drawing of the holder (A) and of the specimen (B) and geometry of the assembled set (C).

2.4. Finite element analysis (FEA)

The specimens and the holders used in the micro-tensile test were measured and molded in CAD with the software Autodesk Inventor (Autodesk Inc., San Rafael, CA, USA). The models were exported to the software Altair Hypermesh (Altair Engineering Inc, Troy, Michigan, USA), where the meshes were generated. The meshes were processed in the program Ls-PrePost (Livermore Software Technology Corporation, Livermore, CA, USA), to determine the simulation parameters. The numeric simulation was calibrated to be processed in the finite elements program Ls-Dyna v917R4.2.1 (Livermore Software Technology Corporation). The results were visualized by the program Altair Hyperview (Altair Engineering Inc.) and exported as figures.

To compare the results of the finite elements analysis with the experimental data, the machine displacement was mimicked through a gentle change of speed from 0 to 1 mm/min, the last one being the speed used experimentally. The force values were measured in a plane near the knots in which the displacement was imposed, obtaining a force reading very similar to the one registered by the load cell in a universal test machine. Table 2 contains the elastic modulus and Poisson's coefficient of the materials present in the specimen (dentin, adhesive and resin composite) and both holders, for the stick shape specimen (aluminium) and dumbbell specimens (steel), according to Braga et al. [16].

To mimic the test with the stick shape specimen (Fig. 2), a finite elements mesh was developed based on the square section of the stick, with 20 × 20 elements of the mean size of 0.1 mm. Along the length of the adhesive, five elements were inserted. The stick-holder interface was simplified, combining the mesh of both structures and eliminating the need to implement contact algorithms. The mesh of the holder propagates from the stick and maintains hexahedral elements, but the elements become bigger from the specimen to the fixing point of the holder to the claws to reduce the computational cost. The supports are made from aluminium and were modeled as such. The materials for all the different components were defined being elastic. For the adhesive, in special, failure criterion was inserted, to remove elements which showed stress over the admissible. Regarding the border conditions, the faces at the far end of the holder, opposite side to the specimen, were selected to apply restrictions. In the lower portion, the displacements were restricted, and in the upper portion, the displacement in the long axis of the stick/holder was applied, and perpendicular displacements were restricted.

To mimic the micro-tensile test with the dumbbell shape specimen (Fig. 3), the upper fixing claws were removed to simplify the mesh creation in finite elements analyses. The holder was modeled with 10 elements in the contact area with the specimen, and it was composed of hexahedra elements in its totality. The mesh of the dumbbell shape specimen was created maintaining hexahedral elements in its totality, and the number of elements was increased at the radius which is in contact with the holder. The adhesive layer was composed of 5 elements in thickness, in the same way as that of the stick-shape specimen. The dumbbell specimen was positioned to touch the holder surface

Table 2
Materials and elastic properties used for finite elements analysis.

Material	Elastic modulus[a] (GPa)	Poisson[a] [-]	Traction failure[a] (MPa)
Steel	210	0.3	–
Aluminum	70	0.33	–
Dentin	18	0.3	–
Resin	9	0.25	–
Adhesive	2	0.3	16

[a] Data extracted from Braga et al. [16].

Table 3
Mean and standard deviation of adhesive strength obtained in the micro-tensile test, in the different experimental conditions.

Experimental groups	Specimen shape	Adhesive strength (MPa)
Group 1	Stick	11.4 (1.9)
	Dumbbell	15.9 (5.1)
Group 2	Stick	9.9 (4.9)
	Dumbbell	15.0 (8.3)
Group 3	Stick	9.4 (1.8)
	Dumbbell	17.1 (8.0)
Group 4	Stick	10.7 (2.3)
	Dumbbell	15.7 (6.6)
Group 5	Stick	13.4 (4.3)
	Dumbbell	19.7 (5.4)
Group 6	Stick	11.9 (3.3)
	Dumbbell	13.4 (8.8)

gently. Likewise, in the stick-shape specimen, all the materials were considered elastic but the holder of the dumbbell specimen is of steel, its properties are described in Table 2. The knots of the claw-holder area, in the opposite side of the specimen, were used to apply the following border conditions: restriction in the lower holder and application of movement away from the supports; The application of a gentle displacement avoids an oscillation of results, as for the stick-shape specimen. Fig. 2 and Fig. 3 show the geometry and measures of the Geraldeli and Dicks holders respectively, and the simplified representation of the specimens in stick shape and dumbbell shape.

2.5. Statistical analysis

Data from the micro-tensile were analyzed using three-way ANOVA (specimen shape, thermal cycling, and mechanical cycling) and Tukey's test. The global significance level adopted was 5%.

3. Results

The mean and standard deviation of adhesive strength obtained in the micro-tensile test are shown in Table 3. The variance analysis indicates that the triple interaction of factors shape (S) × thermal cycling (TC) × mechanical cycling (MC) was not significant ($p = 0.698$). Also, the double interactions of factors SxTC ($p = 0.391$), TCxMC ($p = 0.477$), and SxMC ($p = 0.746$) were not significant.

Regarding the factor "specimen shape," the adhesive strength in the dumbbell shape specimen (16.1 ± 2.5 MPa) was observed to be statistically higher than that of the stick shape specimens (11.1 ± 1.9 MPa, $p < 0.0001$). However, statistical differences among the experimental groups were not observed, regarding the thermal cycling ($p = 0.200$) and the mechanical cycling ($p = 0.587$).

The specimens fractured during cutting or machining were around 2 for stick shapes and 3 for dumbbell shapes per tooth, indicating that 40% of the sticks and 60% of the dumbbell shapes specimens were lost in the preparation. The pre-failure specimens were not included in the statistical analysis. The greater loss of dumbbell shape specimen shows the difficulty in preparing these specimens. There was no loss of specimen during thermal and mechanical cycling.

Three stick shape and three dumbbell shape specimens from each group were analyzed by SEM, and all specimens presented mixed fracture with an interface very similar among the groups. Differences in degradation standard were not noticed in the groups submitted to thermal or mechanical cycling. The presence of porosity was also detected in all groups. It is important to highlight that all the dumbbell shape specimens showed a clear presence of grooves and fracture lines at the edges of the machined specimens. Representative SEM images are shown in Fig. 4 for stick and dumbbell shape specimens.

The stress data obtained in the finite elements analysis were collected for the stick shape specimen, in the area near the fixation, and for the dumbbell shape specimen, in the cylindrical area, using a "section-force" tool which displays the stress in the selected section. This type of data is more stable than the one obtained from the border conditions.

The stress distribution along the stick interface was not homogeneous; some areas showed tensile stress and others compressive stress (Fig. 5). However, the distribution stress in the dumbbell shape specimen was homogeneous along the specimen (Fig. 6).

4. Discussion

The hypothesis of the study was accepted only partially, although the specimen shape results in different stress distributions during the micro-tensile test, the aging protocols used were not able to degrade the adhesive interface and to decrease the adhesive strength.

Besides being widely used, the aging methods still do not show standardization in literature. The thermal cycling has been used in adhesive interface tests of 500 up to 10,000 cycles [17,18], in an immersion time lower than 15 s, [19] or longer, such as the 60 s. [20] The mechanical cycling has been used with 500 up to 2,000,000 cycles [20,21]. In this study, the specimens were submitted to 10,000 thermal cycles, at 5 °C and 55 °C, with 60 s of immersion time in each bath, associated or not to one of the two mechanical cycles, or 500,000 mechanical cycling in macro-rotation with 98 N; or micro-rotation cycling with 100,000 cycles. There were no significant differences among the cycled and non-cycled groups.

It is believed that in this study, the flat shape of the restoration resulted in a low C-Factor, reducing the stress generated in the interface and, consequently, the request of the adhesive interface [22]. Other authors had also observed the absence of statistical differences between thermos cycled and non-cycled groups when a restoration with low C-factor was made, but significant differences were found when the same protocol was applied in specimens with high C-factor, such as class I restorations [19]. Another factor that can influence the absence of effect of the aging protocols is the adhesive system used. This study was performed with the Scothbond Multipurpose (3M ESPE) adhesive, which is a conventional three-step adhesive, considered the gold-standard for adhesion. The use of a simplified adhesive, more susceptible to degradation, as the one-step adhesives, would possibly be able to indicate differences in adhesive strength using the same aging protocols [23]. Furthermore, the substrate can also influence the adhesive strength data directly [24], which makes data interpretation difficult and increases the variability. The experimental design of this study, using the same teeth to make stick and dumbbell shape specimens, tried to minimize this effect, but the dentin heterogeneity can also be determinant in the obtained results.

However, the specimen shape showed statistical differences in the adhesive strength and the mean values were higher in the dumbbell shape specimens. The surface area for adhesion in the sticks was 4 mm^2, and in the dumbbells specimens was around 0.8 mm^2. Griffith's law, which associates a lower strength to a higher surface area of the specimen due to the presence of micro-defects, could explain this experimental data, however the FEA show that stress distribution can take the main role to determine adhesive strength in the samples. The dumbbell specimens are just docked in the micro-tensile holder by the notch area, and its higher adhesive strength is related to more homogeneous stress distribution during the specimen traction, according to the finite elements analysis displayed in Fig. 5 and Fig. 6. In this way, when Dicks device was used, the rupture stress data obtained dividing the force by the transversal section area was compatible with the real stress generated in the interface during the micro-tensile test. This fact allows the dumbbell shape specimen to be less sensitive to changes in the specimen dimensions as observed by Raposo et al. in a finite elements analysis study [7]. In another way, the stick shape specimens fixed in the Geraldeli holder showed more heterogeneous stress distribution, having areas with high tensile stress, with intermediary stress and even with compressive stress. Therefore, some specimen areas, such as the

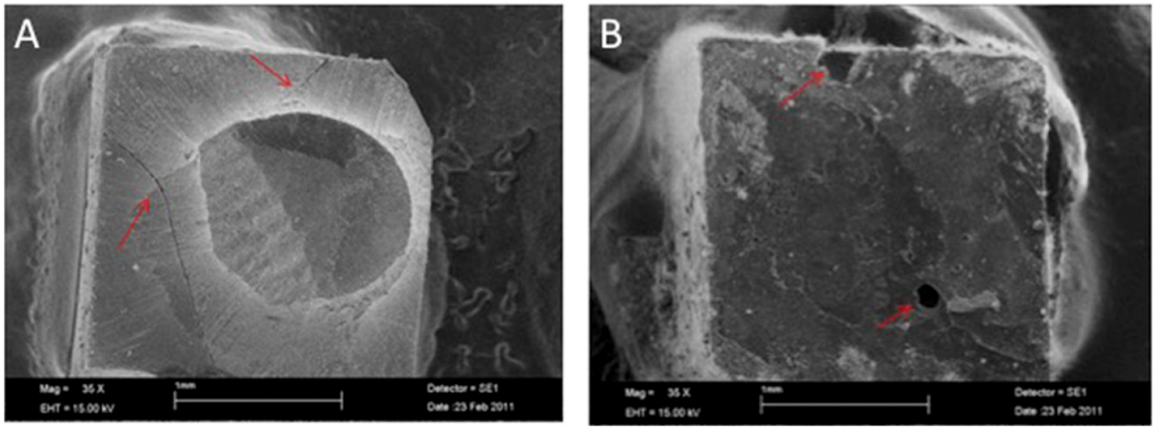

Fig. 4. SEM images of dentin in (A) the dumbbell shape specimen, headset indicates the cracks in the machined dentin; (B) the stick shape specimen, headset indicates porosities in the interface.

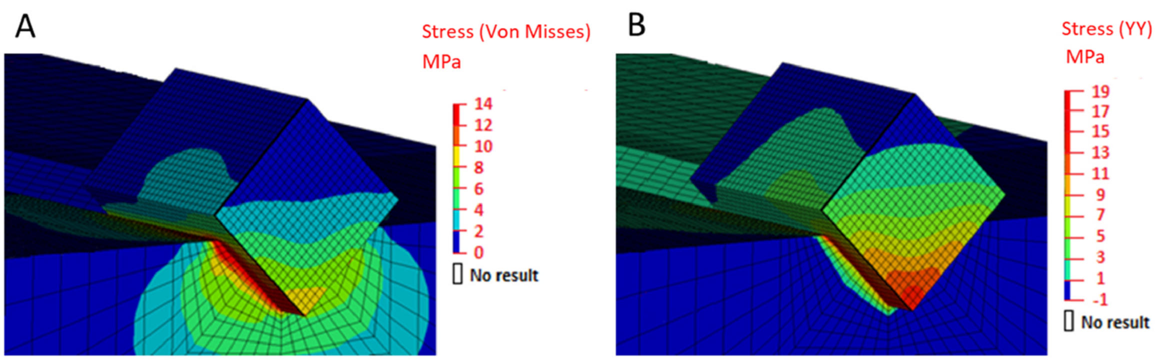

Fig. 5. Stress distribution in the stick shape specimen. (A) Von Mises stress (MPa) and (B) axial stresses (MPa, in Y-axis) of the holder and the adhesive in its interface with the resin composite.

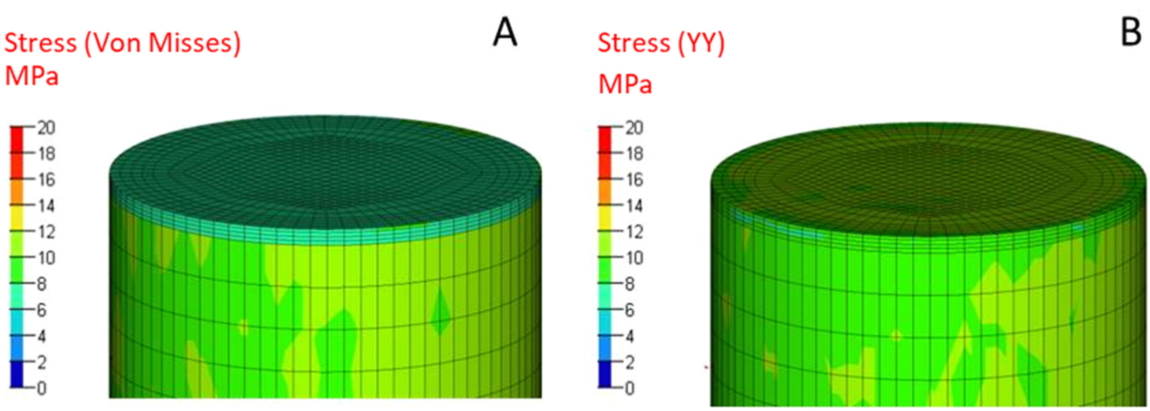

Fig. 6. Stress distribution in the dumbbell shape specimen. (A) Von Mises stress (MPa) and (B) axial stresses (MPa, in Y-axis) of the holder and the adhesive in its interface with the resin composite.

specimen area adhered to the claw, are submitted to stress higher than the adhesive interface area. The rupture stress displayed a mean stress value, which is lower than the maximum supported by the stick, given by the division of the registered force by the transversal section area. The study of Ferreira et al., observed, by finite elements analysis, that when stick shape specimens are submitted to tensile, without any holder, they concentrated the stress on the edges of adhesion area, whereas the hourglass specimens distributed the stress along the entire peripheral adhesive area [6].

However, not only the holders and specimens geometry influence the micro-tensile data, some authors have reported some disadvantages of the dumbbell or hourglass shape specimens [25], because of the higher premature failure, asymmetry in the lateral notch and high-stress induction by the diamond bur during specimen machining. In the present study, the premature failures of the dumbbell shape were higher than for the stick shape, which is by the literature [25]. These failures can be related to the difficulty to machine the specimens; cracks can be observed in SEM in the edges of machined specimens (Fig. 4), which can also help to explain the higher variability observed in this group. However, the use of a standard device to perform the machining decreases the specimens' asymmetries and decreases the stress generated by the burs, favoring to reach a higher mean of adhesive strength in this group. The diamond burs used to machine the specimen were constantly changed to avoid that burs without cut generate more stress during the specimen preparation.

5. Conclusion

Within the limitations of this study, it can be concluded that for the adhesive system used: the stick shape specimens showed significantly lower adhesive strength than the dumbbell shape due to the more homogeneous stress distribution in the latter. The thermal cycling (10,000 cycles, at 5 °C and 55 °C, 60 s in each bath) and also both the mechanical cycling, the macro-rotation (500,000 cycles- 98N- 4 Hz) and Micro-rotation (1,000,000 cycles −4 Hz) were not able to significantly reduce the adhesive strength when applied alone nor when associated.

Conflict of interest

The authors declare that there is no conflict of interest in this study.

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