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Properties of Composite Materials Used for Bracket Bonding

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The purpose of this study was to evaluate *in vitro* the shear bond strength to enamel, flexural strength, flexural modulus, and contraction stress of one orthodontic composite and two flowable composites. Orthodontic brackets were bonded to 45 human maxillary premolars with the composites Transbond XT, Filtek Z-350 flow and Opallis flow and tested for shear bond strength. For measurement of flexural strength and flexural modulus, specimens were fabricated and tested under flexion. For the contraction stress test, cylindrical specimens were tested and an extensometer determined the height of the specimens. The data were subjected to one-way ANOVA and Tukey's test (α=0.05). The shear bond strength values were significantly lower ($p<0.05$) for the flowable composites compared with the orthodontic composite. For the flexural strength, no statistically significant difference was found among the composites ($p>0.05$) while the flexural modulus was significantly higher ($p<0.05$) for Transbond XT than for Filtek Z-350 flow and Opallis flow. The orthodontic composite presented significantly lower contraction stress values than the flowable composites ($p<0.05$). The light-activated orthodontic composite material presented higher flexural modulus and shear bond strength and lower contraction stress than both flowable composites.

Key Words: orthodontic brackets, composite resins, shear strength, mechanical stress.

Introduction

Several factors might affect the bond strength of bracket to enamel, leading to debonding, such as acid etching and drying time, adhesive application mode and time and photoactivation time (1). Composite photoactivation time is particularly important because underpolymerization may result in early bracket debonding (2).

Chemically activated resin composites have been widely used in Orthodontics. These composites require mixing of two pastes, which could induce incorporation of air bubbles into the material. Other disadvantages include longer working time, slower polymerization reaction and lower mechanical properties because the incorporation of oxygen in the mass inhibits the polymerization (3). For these reasons, light-activated orthodontic composite materials have been ever more frequently used for bracket bonding to dental enamel (4). These materials are very similar to the composite resins used in restorative dentistry (5), which has led to the indication of flowable composites for bracket bonding instead of orthodontic composites (6-10). The high fluidity of flowable composites could be an advantage for bracket bonding for allowing a better adaptation in areas of anchorage and regions of demineralized enamel (11). In addition, flowable composites are usually less expensive than orthodontic composites (9) and their low modulus of elasticity could act as an "elastic layer" (12), preventing stress concentration at the tooth/bracket interface during light-activation and allowing a better dissipation of the stresses generated during occlusal movements (13).

Although not being frequently cited in studies evaluating orthodontic bracket bonding, the cavity configuration factor, or C-factor, is extremely high due to the limited number of flow-free faces (14). This may be responsible for the high stress at the adhesive interface, which may contribute directly to bracket debonding, as occurs in composite resin (or resin material) restorations in anterior and posterior teeth. To the best of our knowledge, there is only one study (15) in which the authors used simulation by finite element analysis to evaluate, among other factors, the effect of the modulus of elasticity of the cement film on the stresses generated at the bonded interface. In spite of demonstrating that the modulus of elasticity had little influence on stress generation, this factor had great impact on stress distribution within the bonded interface. Therefore, one could suggest that the modulus of elasticity and resultant polymerization stress during the polymerization procedure may be related to the bond between brackets and enamel. As far as it could be acknowledged, no study has so far addressed experimentally these properties in bonded orthodontic brackets. It is also worth mentioning that several studies have analyzed only the bond strength of flowable composites associated with bracket bonding and the results are controversial (6-10).

Thus, the aim this study was to evaluate the shear bond strength to enamel, flexural strength, flexural modulus and contraction stress of two flowable composites and one orthodontic composite.

Material and Methods

Tooth Selection and Bonding Technique

After approval of the local Ethics Committee (Protocol #23115003621/2010-29), 45 sound human maxillary premolars were selected and embedded in acrylic resin (Jet; Clássico Produtos Odontológicos, São Paulo, SP, Brazil) inside PVC cylinders. The buccal surface was positioned perpendicular to the bottom of the PVC cylinders in such a way that the bonding surface would be parallel to the force applied during the shear strength test. The test surface was cleaned with a superfine pumice (SS White, Rio de Janeiro, RJ, Brazil) and water slurry in Robinson brushes (Microdont, São Paulo, SP, Brazil) mounted in a low-speed handpiece (Dabi Atlante, Ribeirão Preto, SP, Brazil) for 10 s, followed by washing with water/air spray for 10 s and air drying.

Forty-five standard metal Edgewise brackets for premolars were used (Abzil, São José do Rio Preto, SP, Brazil; base area = 12.06 mm^2). The teeth were divided into 3 groups (n=15) according to the type of resin tested: Transbond XT (orthodontic composite), Filtek Z-350 flow (flowable composite; 3M/ESPE, St. Paul, MN, USA) and Opallis flow (flowable composite; FGM, Joinville, SC, Brazil). The compositions and application mode are presented in Table 1.

Light activation was performed with a halogen light-curing unit (Optilux 501; Kerr, Orange, CA, USA) with light intensity of 450 mW/cm^2 on the mesial and distal faces, with curing time of 20 s for each proximal face. The test specimens were kept at 37º C for 24 h.

Shear Bond Strength Test

The shear bond strength test was performed in a universal test machine (model 3342; Instron Corp., Canton, MA, USA) at a crosshead speed of 0.5 mm/min. The test specimens were placed in a tensile device (Odeme Biotechnology, Joaçaba, SC, Brazil) so that a chisel would produce a force falling on the tooth/bracket interface in the occlusal/gingival direction, creating a shear stress. The load necessary to debond the bracket was recorded in N and the bond strength was expressed in MPa by dividing the load at failure in N by the surface area of the bracket in square millimeters (mm).

Adhesive Remnant Index (ARI)

After the shear bond strength test, the fractured surface of each test specimen was evaluated under a stereoscopic loupe (Kozo Optical and Electronic Instrument Co., Ltd.) at 10× magnification to quantify the ARI scores that range from 0 to 3, where: 0: No adhesive adhered to enamel; 1: less than half of the adhesive adhered to enamel; 2: Over half of the adhesive adhered to enamel; 3: The entire adhesive is adhered to enamel, including the impression of the bracket mesh.

Flexural Strength Test

Rectangular specimens (10x2x1 mm) were fabricated by filling a stainless steel split mold (Odeme Biotechnology) onto a glass slab with one increment of composite resin using a metallic spatula. The resin was covered with another

Table 1. Materials used in the study*

Material	Composition	Application mode
Condac (FGM, Joinville, SC, Brazil)	37% phosphoric acid	1 Acid etching (30 s)
Primer Tranbond XT (3M Unitek, Monrovia, CA, USA)	TEGDMA, Bis-GMA, and camphorquinone	2 Washing (30 s)
		3 Drying with an air stream (15 s)
Tranbond XT (3M Unitek, Monrovia, CA, USA)	Bis-GMA, silane, n-dimethylbenzocaine, phosphorus hexafluoride, 77% by weight of inorganic filler (silica)	4. Application of primer/adhesive (15-20 s)
Single Bond 2 (3M/ESPE, St Paul, MN, USA)	Ethanol, Bis-GMA, filler treated with silane, 2-hydroxyl methacrylate (2-hydroxirtilmethacrilate), glycerol 1, 3-dimethacrylate, copolymer of itaconic acid and diurethane dimethacrylate.	5. Air drying (15 s) at a distance of 20 cm
		6. Light curing (10 s)
Opallis Flow A2 (FGM, Joinville, SC, Brazil)	Bis-GMA, TEGDMA, Bis-EMA, 72% by weight of inorganic filler (barium-aluminum silicate and silicon dioxide)	7. Application of resin on bracket base
Filtek Z-350 Flow A2 (3M/ESPE, St Paul, MN, USA)	Bis-GMA, TEGDMA, Bis-EMA, 65% by weight of inorganic filler (silica and zirconium)	8. Light activation (40 s) 450 mW/cm^2 Energy dose: 18 J/cm^2

*Composition of materials according to information obtained from the manufacturers.

glass slab and gently pressed against the mold to extrude excess material. The entire cavity was filled with the same materials used for bonding brackets. Ten specimens were made with each material, for a total of 30 specimens (n=10). Light activation was performed for 40 s using a halogen light-curing unit (Optilux 501) with an energy density of 18 J/cm^2. The specimens were stored in water for 24 h at 37^o C immediately after the test.

Three-point flexural bending was performed in the same universal testing machine (model 3342; Instron Corp.) at a crosshead speed of 0.5 mm/min. The flexural strength was calculated using the following equation:

$$\sigma = \frac{3Fl}{2bh^2},$$

where σ is the flexural strength (MPa), F is the load necessary for fracture, l is the distance between the supports (6 mm) and b and h are the test specimen's width and height (mm), respectively .

The data used to obtain the modulus of elasticity were taken from the flexural strength test; that is, when the test was performed, a computer coupled to the test machine used the load values, for each test specimen, corresponding to the displacement of the active tip. Each load value and corresponding displacement value was inserted in the following equation to obtain the FM value, which is the modulus of elasticity from the flexion test:

$$FM = \frac{f1.l^3}{4b.h^3.d}$$

where, f1 is the load recorded at time (1), l is the distance between the supports, b and h are the height and width of the test specimen (mm), respectively, and d is the deflexion (mm) corresponding to f1.

Polymerization Contraction Stress Test

The test was performed with poly (methyl methacrylate) - PMMA cylinders, 5 mm in diameter and 13 mm long, used as substrates for the composites. The ends of the cylinders were polished with a sequence of 600- to 4000-grit silicon carbide papers followed by 3 or ¼ µm diamond paste in soft felt polishing pad (Buehler-MetaDi; Buehler Ltd. Lake Bluff, IL, USA). A universal testing machine (Instron 5565) was used, and the shorter cylinders were fixed to the bottom clasp on the polymerization stress device, and the longer cylinders to the top clasp, with a distance of 1 mm (C factor =2.5; volume =16 mm^3) between them. After insertion of the composite, the transducer - extensometer (model 2630-101, Instron Corp.) was coupled to the cylinders to maintain the distance between them during the test.

Light activation of the composite resin was performed for 40 s using a halogen light-curing unit (Optilux 501) with an energy density of 18 J/cm^2. The test was monitored for 10 min from the beginning of light activation. Five specimens were tested for each of the flowable composites and the orthodontic composite (n=5).

Data from all tests were subjected to statistical analysis by one-way ANOVA and Tukey's test (α=0.05).

Results

The means and standard deviations of shear bond strength (MPa), flexural strength (MPa), flexural modulus (GPa) and polymerization contraction stress (MPa) of the materials are shown in Table 2.

There was no statistically significant difference among the composites for flexural strength (p>0.05). For shear bond strength, Transbond XT presented the highest values (p<0.05) and the other materials were similar to each other (p>0.05). As regards the ARI, score 1 was the most frequent in all groups, followed by score 2 (Table 3).

For the flexural modulus, Filtek Z-350 flow and Opallis flow presented the lowest values (p<0.05). For polymerization contraction stress, Transbond XT presented the lowest value, Filtek Z-350 flow the highest value (p<0.05) and Opallis flow presented an intermediate stress value, differing significantly from the other materials (p<0.05).

Table 2. Means and standard deviations of shear bond strength (MPa), flexural strength (MPa), flexural modulus (GPa) and polymerization contraction stress (MPa) of the tested materials

Composites	Bond strength	Flexural strength	Flexural modulus	Contraction stress
Tranbond XT	25.1 ± 4.4^a	152.7 ± 31.4^A	4.7 ± 2.9b	2.2 ± 0.1C
Opallis Flow A2	15.6 ± 5.8^b	140.9 ± 32.7^A	2.5 ± 0.7a	4.9 ± 0.4A
Filtek Z-350 Flow A2	16.9 ± 8.0^b	155.8 ± 30.1^A	2.2 ± 0.3a	4.3 ± 0.3B

Different lowercase or uppercase letters, either superscript or not, indicate statistically significant difference among the groups (Tukey's test, p<0.05).

Table 3. Adhesive Remnant Index (ARI) recorded in the groups

Composite	Adhesive remnant index				Fractured enamel
	0	1	2	3	
Tranbond XT	0	13	1	0	1
Opallis Flow A2	0	11	4	0	0
Filtek Z-350 Flow A2	0	13	1	0	1

0: No adhesive adhered to enamel; 1: less than half of the adhesive adhered to enamel; 2: Over half of the adhesive adhered to enamel; 3: The entire adhesive is adhered to enamel, including the impression of the bracket mesh.

Discussion

The bond strength values found in this study for flowable composites were significantly lower compared with those found for Transbond XT. Some studies have also found lower bond strength values for flowable composites when compared with an orthodontic composite (7,10,16). However, these results are controversial (6,8,9). Thus, continuous evaluations of the mechanical behavior of composites is an attempt to understand the reasons for the different results of shear bond strength tests found in the literature (17).

Transbond XT presented higher modulus of elasticity than Filtek Z-350 flow and Opallis, which seems to be a reasonable result, as the orthodontic composite has greater filler content (77%) than the flowable composites, Filtek Z-350 flow (65%) and Opallis (72%). The filler is generally responsible for the increase of the mechanical properties of the material (18). However, the increase in the quantity of filler would not have any direct relationship with the increase in bond strength of the materials, since the involved materials had a minimum intrinsic strength to bear the forces to which they were submitted during the test.

The C-factor is extremely high at the adhesive interface formed between the bracket and the dental enamel. The role of C-factor in the development of polymerization stress in composite materials was first demonstrated by Feilzer et al. (19), who described that when two rigid surfaces are united, such as the bond between dental enamel and the metal bracket, the only region responsible for release of the stresses generated by polymerization contraction, elastic deformation of the material and flow is the free part in the thin film of composite material between the enamel and bracket (20).

Therefore, the C-factor is given by the ratio between the bonded surfaces and the free surfaces, and the smaller the non-bonded surface area, the smaller the possibility for the cement material to flow, and thus the greater the polymerization stress generated at the adhesive interface (14). Considering the size of the bracket area and the approximate thickness of the cement film (more or less 0.3 mm) (14), the C-factor of a bracket bond is around 6. This means that the stress generated at the bracket-enamel interface is extremely high. Therefore, the use of materials with a lower flexural modulus may generate lower stresses and diminish the impact of polymerization on the bonded interface.

Based on the Feilzer's et al. theory (19), it was to be expected that Transbond XT, which is the material with the highest flexural modulus would also cause the highest polymerization stress values, as shown by Condon and Ferracane (21). However, the results from the polymerization contraction stress test of the composites showed that the flowable composites generated a statistically higher stress when compared with Transbond XT. Gonçalves et al (22) showed that the composite matrix had a stronger influence on polymerization stress, conversion and reaction rate, when different BisGMA:TEGDMA ratios were compared, whereas filler fraction showed a stronger influence on shrinkage and modulus. Thus, materials with a high percentage of diluent monomers of low molecular weight, such as TEGDMA, present high volumetric contraction, and consequently, high contraction stress values, due to increase of the conversion rate (23). Perhaps the presence of diluent monomers (TEGDMA and Bis-EMA) and low filler content in Filtek Z-350 flow and Opallis, may have contributed to a statistically higher contraction stress when compared with Transbond XT.

Higher ARI values are favorable for avoiding damage to the enamel, as the residue may safely be removed with suitable rotary instruments. In the present study there was higher prevalence of ARI 1 (82%) and 2 (13%) values in all groups, thus detecting a failure in the bonding to enamel, or greater retention of the adhesive material to the bracket, as shown in previous studies (1,24).

Considering the limitations of this study, it may be concluded that the light-activated orthodontic composite showed the highest shear bond strength and flexural modulus, and the lowest contraction stress values in a comparison with flowable composites.

Resumo

O objetivo desse estudo foi avaliar a resistência ao cisalhamento no esmalte, resistência flexural, módulo flexural, tensão de contração de polimerização de duas resinas *flow* e uma resina ortodôntica. Os bráquetes ortodônticos foram colados em 45 pré-molares humanos e divididos: Transbond XT, Filtek Z-350 flow, Opallis *flow* e testado para resistência ao cisalhamento (n=15). Para a resistência e módulo flexural espécimes foram confeccionados e testados sob flexão. Para o teste de tensão de contração de polimerização, espécimes cilíndricos foram confeccionados e monitorados com um extensômetro (Instron). Os dados foram submetidos aos testes ANOVA a um critério e Tukey (α=0,05) para contraste de média. (n=15). A resistência de união das resinas *flow* foram significantemente menos que o da resina ortodôntica (p>0,05). A resistência flexural não demonstrou diferença significante entre os grupos testados (p>0,05). O módulo flexural da resina ortodôntica foi significantemente maior que o grupo das resinas *flow* (p<0,05). A tensão de contração de polimerização da resina ortodôntica foi significantemente menor (p<0,05). A resina ortodôntica obteve os maiores valores de resistência de união ao cisalhamento, resistência flexural e a menor tensão de contração de polimerização quando comparada as resinas *flow* testadas.

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