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Influence of the bonding substrate in dental composite polymerization stress testing

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ABSTRACT

Our objective was to compare the polymerization stress (σ_{pol}) of a series of composites obtained using poly(methyl methacrylate) (PMMA) or glass as bonding substrates, and to compare the results with those from in vitro microleakage of composite restorations. The tested hypothesis was that stress values obtained in a less rigid testing system (i.e. using PMMA) would show a better relationship with microleakage data. Five dental composites were tested: Filtek Z250 (FZ), Z100 (Z1), Concept (CO), Durafill (DU) and Heliomolar (HM). σ_{pol} was determined in 1 mm high specimens inserted between two rods $(\emptyset = 5 \text{ mm})$ of either PMMA or glass. The composite elastic modulus (E) was obtained by three-point bending. σ_{pol} and *E* data were submitted to a one-way analysis of variance/Tukey test ($\alpha = 0.05$). For the microleakage test (MI), bovine incisors received cylindrical cavities (\emptyset = 5 mm, h = 2 mm), which were restored in bulk. After storage for 24 h in water, specimens were subjected to dye penetration using AgNO₃ as tracer. Specimens were sectioned twice, perpendicularly, and microleakage was measured (in millimeters) under 20× magnification. Data from MI were submitted to the Kruskal-Wallis test. Means (SD) of σ_{pol} (MPa) using glass/PMMA were FZ: 7.5(1.8)^A/2.5(0.2)^{bc}; Z1: 7.3(0.5)^A/2.8(0.3)^{ab}; CO: 6.8(1.1)^A/3.2(0.5)^a; DU: 4.5(0.7)^B/2.0(0.2)^{bc}; HM: 3.5(0.2)^B/2.3(0.3)^c. σ_{pol} obtained using PMMA rods were 34-67% lower than with glass. Means (SD) for tooth average/tooth maximum microleakage were FZ: $0.92(0.19)^{8}/1.53(0.30)^{a}$; Z1: $1.19(0.21)^{A}/1.75(0.20)^{a}$; C0: $1.26(0.25)^{A}/1.78(0.24)^{a}$; DU: $0.83(0.30)^{8}/1.75(0.20)^{A}/1.75(0.20)^{a}$; DU: $0.83(0.30)^{B}/1.75(0.20)^{A}/1.75(0$ $1.68(0.46)^{a}$; HM: $0.81(0.27)^{B}/1.64(0.54)^{a}$. The tested hypothesis was confirmed, as the composites showed the same ordering both in the polymerization stress test using PMMA rods and in the microleakage test.

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1. Introduction

Common causes for failure of composite restorations are often related to imperfections of the tooth/restoration interface (e.g. postoperative sensitivity, marginal discoloration and secondary caries) [1,2]. The interfacial stresses developed due to composite shrinkage upon polymerization are considered to be primary factors leading to the loss of bonding integrity [3]. For this reason, different research groups have focused on developing mechanical tests to quantify the polymerization stress [4–7]. A common approach is to insert the composite between two flat surfaces of glass or steel attached to the opposite fixtures of a universal testing machine. The contraction force exerted by the composite when both substrates are pulled together is recorded by the load cell. The maximum nominal stress is obtained by dividing the maximum registered load by the cross-sectional area of the rod [8–11].

Several studies have shown that the strain capacity of the testing system components has great influence on the stress values. The sum of these deformations is referred to as the system's

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compliance [8,11,12], expressed in mm N⁻¹. The higher the compliance, the higher the system's strain capacity is, and therefore the lower the recorded stress values [8,12]. Aiming at reducing the influence of these deformations, a gauge length transducer is often employed to monitor the distance between the opposite bonding substrates, providing feedback to the crosshead in order to maintain the initial distance. In this situation, the deformation of the structures located within the fixation points of the transducer still influences the value registered by the load cell.

Studies evaluating cuspal deflection as a function of the composite polymerization shrinkage suggested that the dental structure presents a relatively high strain capacity [13,14]. Determination of the compliance in a clinical situation is not feasible due the plethora of factors involved, such as variations in the elastic modulus of the dental substrate (anisotropy) and the complexity of the cavity geometry. This notwithstanding, it is likely that the use of testing systems with "near zero" compliance overestimates the stress compared to values found in strain conditions more akin to that of the dental structure.

A study comparing the stress developed by five commercial composites in a high compliance system (which used poly(methyl methacrylate)(PMMA) rods as bonding substrate) and in a low compliance





system (using glass rods) found values 53–68% lower than the former [15]. Moreover, stress reduction between both substrates was shown to be directly related to composite elastic modulus. Finite element analysis showed that the transverse stress distribution in the composite was similar for both glass and PMMA. However, longitudinal stress distribution showed a large compressive stress zone within the composite when PMMA was used. In practical terms, the use of PMMA has the advantage of significantly reducing specimen loss during testing. When glass rods are used, stress values around 9 MPa often cause specimen failure due to debonding or crack propagation in the glass near the interface with the composite [16].

A direct relationship between the stress developed by commercial composites in low compliance settings and in vitro microleakage was observed in a few studies [3,17,18]. In order to consider PMMA as an alternative to glass in the determination of polymerization stress, it is necessary to verify whether the results obtained agree with those from interfacial integrity evaluations. Therefore, the aim of this study was to determine the polymerization stress of five commercial composites with different filler levels in testing systems with high and low compliance (using, respectively, PMMA and glass as bonding substrates) and relate these results with those from a microleakage test. The null hypotheses were that for a series of resin composites: (i) the compliance of the testing system does not affect the materials' ranking; and (ii) regardless of the compliance of the testing system, stress values show a strong correlation with in vitro microleakage of composite restorations. The ultimate goal was to validate the use of PMMA rods as the bonding substrate in the polymerization stress test.

2. Materials and methods

Five commercially available composites were tested: Filtek Z250 (3M ESPE, St. Paul, USA), Z100 (3M ESPE, St. Paul, USA), Concept (Vigodent, Rio de Janeiro, Brazil), Durafill (Heraus Kulzer, Hanau, Germany) and Heliomolar (Ivoclar Vivadent, Schaan, Liechtenstein). Their detailed compositions as informed by the respective manufacturers are described in Table 1.

2.1. Polymerization stress test

Polymerization stress was measured using borosilicate glass or PMMA rods as bonding substrate for the composite, both with a diameter of 5 mm. The rods were sectioned in 13 and 28 mm segments. For the 13 mm PMMA rods, one of the flat surfaces was polished using 600–1200 sandpaper and felt disks with 1 μ m alumina paste (Alumina 3, ATM, Altenkirchen, Germany) to allow for light transmission during photoactivation with the highest irradiance possible. The opposite surface and one of the flat surfaces of the 28 mm rods were sandblasted with alumina (250 μ m). For the glass rods, polishing was not necessary because the surface obtained after sectioning was sufficiently smooth. For the glass rods, the sandblasted surface was coated with a silane compound (Ceramic Primer, 3M ESPE), while for the PMMA rods the surface was treated with methyl methacrylate monomer (JET Acrílico Auto Polimerizante, Artigos Odontológicos Clássico, São Paulo, Brazil).

Table 1

Composites	tested	in	the	study.	
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These surfaces then received two thin layers of an unfilled resin (Scotchbond Multipurpose Plus, bottle 3, 3M ESPE), photoactivated with 12 J cm⁻² (400 mW cm⁻² \times 30 s).

The rods were attached to the opposite fixtures of a universal testing machine (Instron 5565, Canton, MA, EUA). On the lower fixture, the 13 mm rod was fixed to a stainless steel attachment with a slot allowing the positioning of the light guide in contact with its polished surface. The 28 mm rod was attached to the upper fixture, connected to the load cell. The distance between the rods was 1 mm (cavity configuration factor (*C*) = 2.5; volume = 16 mm^3). After the insertion of the composite, an extensometer (model 2630-101, Instron) was attached to the rods in order to monitor the distance between them during the test and provide feedback to the machine's actuator to re-establish the initial distance. Therefore, the value registered by the load cell corresponded to the force necessary to maintain the initial height of the specimen in opposition to the force exerted by the shrinking composite. Photoactivation was carried out using a quartz-tungsten-halogen light-curing unit (VIP Junior, BISCO, Schaumburg, IL, USA). After propagating through the length of the 13 mm rod, the irradiance reaching the composite surface was 570 mW cm⁻². The irradiance was periodically checked with a dental radiometer (model 100, Demetron Res. Corp., Orange, CA, EUA). A 32 s exposure was used, providing a radiant exposure of approximately 18 [cm⁻². Contraction force was monitored for 5 min from the onset of photoactivation and the maximum nominal polymerization stress was calculated by dividing the maximum force value by the cross-sectional area of the rod

2.2. Elastic modulus determination

For each composite, 10 specimens were built using a $10 \times 2 \times 1$ mm stainless steel split mold. Photoactivation followed the same parameters described in the polymerization stress test. The three-point flexural test was conducted after 24 h storage in water at 37 °C in a universal testing machine (Instron 5565), with 8 mm span between supports and a cross-head speed of 0.5 mm min⁻¹. Data from the initial linear portion of the load × displacement curve was used to calculate the elastic modulus using the following formula:

$$M_{\rm f} = \frac{L \times D^3}{4 \times w \times h^3 \times d} \times 10^{-3}$$

where $M_{\rm f}$ is the elastic modulus in flexure (GPa), L is the load recorded (N), D is the span between the supports (mm), w is the width of the specimen, h is the height of the specimen and d is the displacement corresponding to L (all in mm).

2.3. Microleakage test

Bovine incisors received cylindrical cavities with a diameter of 5 mm and a depth of 2 mm on the labial surface, the enamel which was flattened using a 400 grit sandpaper (C = 2.6; volume = 32 mm³). The cavities were prepared using high-speed diamond burs and finished using cylindrical low-speed diamond burs, both under copious

Composite	Туре	Composition	Manufacturer
Filtek Z250	Hybrid	BisGMA, UDMA, BisEMA, zirconia/silica (0.19–3.3 μm, 60 vol.%)	3M ESPE, St. Paul, EUA
Z100	Hybrid	BisGMA, TEGDMA, zirconia/silica (up to 4.5 µm, 66 vol.%)	3M ESPE, St. Paul, EUA
Concept	Hybrid	BisGMA, UDMA, esther of methacrylic acid, barium and aluminum silicates ($0.001-2 \ \mu m$, 77.5 wt.%)	Vigodent S/A, Rio de Janeiro, Brazil
Durafill	Microfilled	UDMA, silica di-oxide (0.02–0.07 µm, 40 vol.%)	Heraus Kulzer GmbH, Hanau, Germany
Heliomolar	Microfilled	BisGMA, UDMA, decandiol dimethacrylate, colloidal silica (0.04–0.2 μm, 46 vol.%)	Ivoclar Vivadent, Schaan, Liechtenstein

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