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Contraction stress related to composite inorganic content

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ABSTRACT

Objectives. The role of inorganic content on physical properties of resin composites is well known. However, its influence on polymerization stress development has not been established. The aim of this investigation was to evaluate the influence of inorganic fraction on polymerization stress and its determinants, namely, volumetric shrinkage, elastic modulus and degree of conversion.

Methods. Eight experimental composites containing 1:1 BisGMA (bisphenylglycidyl dimethacrylate):TEGDMA (triethylene glycol dimethacrylate) (in mol) and barium glass at increasing concentrations from 25 to 60 vol.% (5% increments) were tested. Stress was determined in a universal test machine using acrylic as bonding substrate. Nominal polymerization stress was obtained diving the maximum load by the cross-surface area. Shrinkage was measured using a water picnometer. Elastic modulus was obtained by three-point flexural test. Degree of conversion was determined by FT-Raman spectroscopy.

Results. Polymerization stress and shrinkage showed inverse relationships with filler content ($R^2 = 0.965$ and $R^2 = 0.966$, respectively). Elastic modulus presented a direct correlation with inorganic content ($R^2 = 0.984$). Degree of conversion did not vary significantly. Polymerization stress showed a strong direct correlation with shrinkage ($R^2 = 0.982$) and inverse with elastic modulus ($R^2 = 0.966$).

Significance. High inorganic contents were associated with low polymerization stress values, which can be explained by the reduced volumetric shrinkage presented by heavily filled composites.

1. Introduction

The inorganic content of resin composites is a key determinant for their mechanical behavior. Several studies have correlated the filler fraction with fracture strength [1], elastic modulus [2] and wear resistance [3]. Also, increased filler content has been associated with lower volumetric shrinkage, as it reduces the volume of organic matrix present in the material [4]. However, negative effects on composite degree of conversion attributed to the filler have been reported, caused by mobility restrictions imposed on the reactive species [5] and by light scattering [6]. Also, an increased risk of crack propagation and wear was verified in heavily filled composites due to lower compressive stresses exerted by the polymeric matrix on the filler particles as a result of dissimilar coefficients of thermal expansion [7].

Volumetric shrinkage, elastic modulus and degree of conversion have been shown to modulate polymerization stress development [8–10]. Therefore, it is reasonable to expect that the latter will be significantly affected by the composite inorganic content. In fact, a direct relationship between poly-

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merization stress and filler fraction has been described for commercial composites [11,12]. These results were obtained in a testing system with very low compliance and were somewhat surprising, as they imply an inverse relationship between shrinkage and stress. Moreover, in both studies, the effect of the inorganic content was not isolated because of differences in organic content among the composites tested.

More recently, several studies have measured polymerization stress using non-rigid systems based on the fact that teeth can undergo relatively high elastic and viscoelastic deformation [13–17]. In fact, a better correlation between polymerization stress and *in vitro* microleakage of composite restorations was found when stress data were originated from a high compliance system compared to a rigid system [18]. Also using a high compliance system, a direct correlation between polymerization stress and shrinkage was found with composites containing different monomer mixtures and similar inorganic content [10].

Therefore, the purposes of the present study were: (1) to evaluate the influence of filler fraction of experimental composites on the polymerization stress and its determinants, namely, degree of conversion, volumetric shrinkage and elastic modulus and (2) to investigate the association between polymerization stress and the other variables.

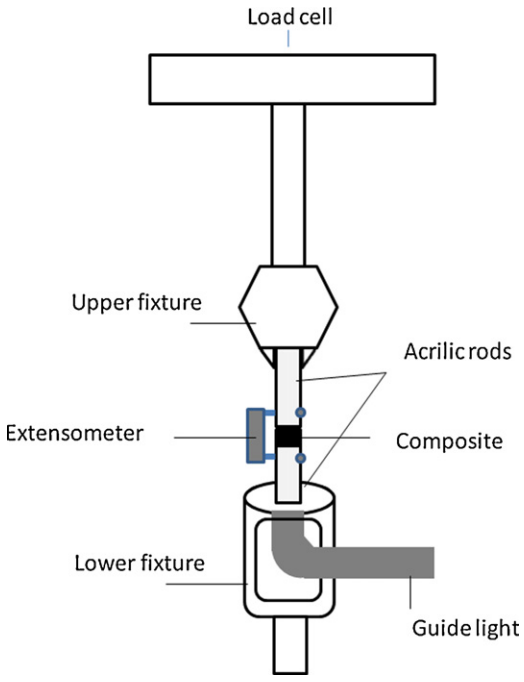

Fig. 1 – Polymerization stress test setup.

2. Materials and methods

2.1. *Composite formulations*

Eight composites were formulated containing equivalent molar concentrations of BisGMA (bisphenylglycidyl dimethacrylate or 2,2-bis[4-(2-hydroxy-3-methylacryloxypropoxy)-phenyl]propane, Esstech, Essington, PA, USA) and TEGDMA (triethylene glycol dimethacrylate or 2-methyl-2-propenoic acid, Esstech). The weight percentage equivalent of 1:1 BisGMA:TEGDMA molar ratio is approximately 1.8:1 BisGMA:TEGDMA. The photoinitiator system was composed of camphorquinone (Sigma–Aldrich Inc., St. Louis, MO, EUA) and 2-(dimethylamino)ethyl methacrylate (Sigma–Aldrich), 2 mol.% each. Inhibitor (butylated hydroxytoluene or 2,6-di-tetra-butyl-4-methylphenol, Sigma–Aldrich) was also added at 0.2 mol.%. Silanated barium glass particles with irregular shape (average size: 2 μm; density: 2.75 g/cm^3, Vigodent, Brazil) were added to the resin mixture at different ratios, from 25 to 60 vol.% (45–74 wt.%), in 5 vol.% increments. Composites were mixed by hand, in an environment without any artificial light, and stored for 2 days in the dark at room temperature prior to use.

2.2. *Polymerization stress*

The polymerization stress test ($n = 5$) used poly(methyl methacrylate) rods (radius, 3 mm and length, 13 or 28 mm) as bonding substrate for the composite, attached to a universal testing machine (Instron 5565, Canton, MA, USA). They had one of their flat surfaces roughened with #180-grit sandpaper, treated with methyl methacrylate for 10 s and coated with unfilled resin (Scotchbond Multipurpose Plus, 3M ESPE, St. Paul, MN, USA), photoactivated with 12 J/cm^2. A diagram of the testing assembly is shown in Fig. 1. The shorter rod had the opposite surface polished. The 28-mm rod was clamped to an upper chuck, connected to the load cell. On the lower fixture, the 13-mm rod was clamped to a stainless steel attachment with a slot allowing the positioning of the light guide in contact with its polished surface. The composite (1 mm thick) was inserted between the treated surfaces, resulting in specimens with a C-factor equaling 3. A quartz–tungsten–halogen light curing unit (VIP Junior, Bisco, Schaumburg, IL, USA) with an 8 mm diameter tip was used to photoactivate the composite with the light guide positioned in contact with the polished surface of the short rod. The light was transmitted through the rod, reaching the composite with an irradiance of 570 mW/cm^2. This value was accessed by placing a 13-mm PMMA rod on the light sensor of a dental radiometer (model 100, Demetron Res. Corp., Orange, CA, USA) and shinning the curing light. Because the rod cross-sectional area was smaller than the area of the sensor, the reading was corrected by multiplying it by 1.4 (*i.e.*, ratio between the area of the sensor and the cross-sectional area of the rod). A 32 s exposure was used, resulting in a radiant exposure of approximately 18 J/cm^2. Specimen height was kept constant within a 0.1 μm resolution with the use of an extensometer (model 2630-101, Instron). The extensometer was clamped to the rods in a way that its gauge length, 10 mm, was distributed 6 mm along the upper rod, 1 mm along the composite and 3 mm along the lower rod. Contraction force development was followed for 15 min and maximum nominal stress was calculated by dividing the maximum contraction force by the area of the rod.

2.3. *Volumetric shrinkage*

Volumetric shrinkage ($n = 8$) was determined using a water picnometer (5 cm^3, Brand Gbmh, Wertheim, Germany). The