

Influence of Specimen Dimensions on Nominal Polymerization Contraction Stress of a Dental Composite

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The objective of the present study was to verify the influence of specimen dimensions on polymerization contraction stress of a self-cure dental composite and investigate the influence of confinement (expressed by the ratio between the bonded and unbonded area of the composite, or 'C factor') and volume of the specimen on stress values. The composite was inserted between the flat surfaces of two glass rods attached to a universal testing machine. Specimen dimensions were defined using glass rods with different diameters (2.5, 5, or 8 mm) and adjusting the distance between them (0.63, 0.83, 1.25 or 2.5 mm). An extensometer was used to keep specimen height constant. Force development was monitored for 30 min and the maximum value was used to calculate nominal stress (MPa). System deformation (compliance) was estimated in order to calculate stress values on an ideally rigid situation. Data were analyzed by ANOVA/Tukey test ($\alpha=0.05$) and regression analysis. The interaction was significant ($p<0.001$). Differences in nominal stress for different heights were verified only for 5-mm and 8-mm diameter specimens. In general, lower heights produced higher stress values. Regression analysis using all the collected data showed a linear correlation between stress and 'C factor'. However, non-linear relationships were found when stress was plotted against 'C factor' or volume selecting specimens with similar same diameter. It was concluded that specimen dimensions influenced test results. However, neither 'C factor' nor volume can be considered reliable predictors of contraction stress values.

Keywords: Contraction stress, dental composite, mechanical testing

Introduction

Stresses developed during the polymerization of dental composites are frequently associated with the failure of the tooth/composite adhesive joint, jeopardizing the clinical longevity of the restoration (Hilton, 2002; van Dijken, 2003). Several authors have investigated the different factors affecting stress development (Feilzer et al., 1987; Versluis et al., 1998; Davidson and Feilzer, 1999; Versluis and Tantbirojn, 1999), while others have been evaluating restorative strategies with the purpose of reducing stress magnitude (Choi et al., 2000; Condon and Ferracane, 2002; Braga et al., 2003).

Contraction stress values can be accessed by finite elements analysis (Laughlin et al., 2002; Barink et al. 2003), photoelastic analysis (Ernst et al. 2003; Kinomoto et al., 2003) and, more frequently, using an experimental set-up known as "tensilometer" (Feilzer et al., 1987; Alster et al., 1997 a,b; Miguel and de la Macorra, 2001; Condon and Ferracane, 2002). The use of a tensilometer to determine contraction stress was introduced in Dentistry by Bowen (1967). After a study by Feilzer et al. (1987), this method began to be used more frequently. Briefly, the experimental set-up consists of two metal or glass rods attached to opposite clamps of a universal testing machine. The composite is inserted between the opposing flat surfaces of the rods and the axial force generated by its polymerization shrinkage is monitored for a pre-determined time interval. Force values are divided by the cross-section area of rods in order to obtain nominal stress.

The crescent use of this method raised several questions and controversy among researchers. One recurrent point of dispute is regarding the influence of the deformation (compliance) of the testing set-up on force development. The experimental set-up is not ideally rigid and the elongation of the components plus the approximation of the opposite rods upon force development could reduce the values registered by the load cell. Compliance can be minimized by adding a feedback system (e.g., an extensometer) to the assembly. Its primary function is to detect any approximation

between the rods during composite contraction and command the cross-head to move in the opposite direction, maintaining the initial height of the specimen (Feilzer et al. 1987, Alster et al. 1997 a,b). By doing so, the extensometer minimizes compliance by excluding any deformation that takes place beyond its fixation points. However, some deformation of the rods still occurs within the extensometer attachments that could influence stress values.

Another important question is related to the dimensions of the specimen. So far, there is no standardization or agreement among authors regarding this matter. As with most mechanical tests, specimen dimensions influence results and may preclude an accurate comparison among different materials. A parameter usually reported in contraction stress studies is the ratio between bonded and unbonded surface of the specimen, known as 'C factor' (Feilzer et al., 1987). The results reported by different authors seem to indicate a direct relationship between stress and confinement of the specimen expressed by its 'C factor' (Bowen, 1967; Feilzer et al., 1990; Alster et al., 1997a). This index does not take the volume of the sample into consideration. In other words, it is possible to obtain specimens with different volumes and same 'C factor'. The above-mentioned study (Feilzer et al., 1987) did not report any relationship between volume and stress magnitude using a low-compliance assembly. However, studies using less rigid set-ups (e.g., without a feedback system) have shown some relationship between volume and contraction stress (Bouschlicher et al., 1997; Miguel and de la Macorra, 2001; Watts et al., 2003).

Considering the reduced number of studies evaluating the influence of specimen dimensions on contraction stress development, as well as the discrepancies found among authors regarding the influence of confinement and sample volume on stress values, the objective of this study was to verify the influence of specimen diameter and height (and its derivatives, namely, volume and 'C factor') on composite contraction stress. The intended contribution is to provide guidelines that might help standardize the testing procedure.

Nomenclature

Δl_a = Adhesive deformation mm

Δl_v = Glass deformation mm

- Δl_c = Composite deformation mm
 A_c = Cross-section area of the composite mm²
 A_{v1} = Cross-section area of the machined region of the glass rod mm²
 A_{v2} = Cross-section area of the non-machined region of the glass rod mm²
 f = Load N
 C_a = Compliance of the adhesive mm/N
 C_v = Compliance of the glass mm/N
 D_c = Composite elongation mm/mm
 E_a = Young's modulus of the adhesive MPa
 E_c = Young's modulus of the composite MPa
 E_v = Young's modulus of the glass MPa
 L_0 = Initial length of the assembly mm
 L_1^0 = Length of the non-machined segment of the glass rod before deformation mm
 L_2^0 = Length of the machined segment of the glass rod before deformation mm
 L_3^0 = Adhesive layer thickness before deformation mm
 L_4^0 = Height of the composite before contraction mm
 L_1 = Length of the assembly after composite contraction mm
 L_4^1 = Length of the composite before deformation mm
 L_1^2 = Length of the non-machined segment of the glass rod after deformation mm
 L_2^2 = Length of the machined segment of the glass rod after deformation mm
 L_3^2 = Adhesive layer thickness after deformation mm
 L_4^2 = Length of the composite after deformation mm
 T = Nominal contraction stress of composite MPa

Materials and Methods

Determination of Experimental Stress Contraction

The method used for contraction stress evaluation was similar to those used by several authors (Feilzer et al., 1987; Alster et al., 1997a,b; Choi et al., 2000). Glass rods (Pyrex) with 50 mm length and diameter of 6 or 8 mm were used. The 6-mm diameter rods had one of their flat surfaces machined in a computer-assisted lathe (Compact 5 CNC – EMCO, Hallein, Austria) to obtain a cylindrical surface 2 mm in length and 2.5 or 5 mm in diameter. The flat surface of the machined end and one of the flat surfaces of the 8-mm diameter rods were finished with 180 grit sandpaper, sandblasted with alumina (250 μ m), silane-treated (3M ESPE, St. Paul, MN, EUA, lote 9LT) and coated with a layer of Bis-GMA/TEGDMA-based adhesive (Scotchbond Multi-Usu Plus, 3M ESPE, batch 2MU), light-cured for 30 s.

After surface preparation, two rods were attached to the opposite clamps of a universal testing machine (Instron 5565, Canton, Massachusetts, EUA). Specimen height (0.63 mm, 0.83 mm, 1.25 mm or 2.5 mm) was determined by adjusting the distance between the glass rods. Therefore, 12 experimental groups were defined (n=3). Each testing condition, with the respective 'C factor' and volume, is shown in Tab. 1.

Table 1. 'C factor' and volume (mm³) of experimental conditions.

		Height (mm)				
		0.63	0.83	1.25	2.5	
Diameter (mm)	2.5	'C factor'	2	1.5	1	0.5
		Volume	3.1	4.1	6.1	12.3
	5	'C factor'	4	3	2	1
		Volume	12.3	16.4	24.5	49
8	'C factor'	6.3	4.8	3.2	1.6	
	Volume	31.7	41.7	62.8	125.6	

A commercial chemically-activated dental composite was used (Adaptic, Dentsply Ind. e Com., Rio de Janeiro, Brasil, batch 1781). The composite was mixed for 20 s on a paper pad with a plastic spatula, and placed between the adhesive-coated surfaces of the glass rods. An extensometer (Instron), attached to glass rods to detect any approximation between them, commanded the cross-head of the testing machine to restore the initial distance between extensometer clamps, with 0.1 μ m accuracy (Fig. 1).

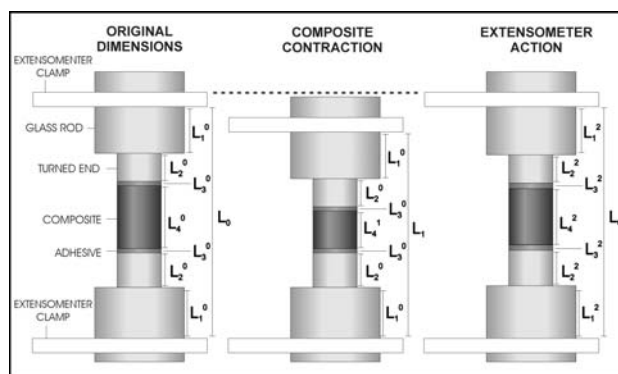


Figure 1. Schematic diagram of the testing system, showing the two imaginary stages of the test considered for calculating the stress values corrected taking into account material compliance.

Contraction stress development was monitored for 30 min, at controlled temperature of 37 \pm 1 $^{\circ}$ C, and the maximum load (N) was registered. During polymerization, shrinkage forces cause elongation of the glass contained within the extensometer clamps and also of the adhesive layer. When specimens with different dimensions are compared, glass elongation represents an important source of experimental error, because it varies according to the specimen dimensions and, consequently, its elongation is also different. The thickness of the adhesive layer, on the other hand, does not vary according to the sample dimensions.

Contraction force value was corrected according to Young's modulus, dimensions of the glass rod, adhesive layer and the composite itself. Though polymerization contraction and specimen elongation triggered by the extensometer occurred continuously, two distinct stages will be assumed for explaining the equations below: 1) Free contraction of the composite in the axial direction and 2) Deformation of the parts commanded by the extensometer. A schematic diagram of these stages is shown in Fig. 1.

Contraction stress was computed using the following equations

$$T = T_c + T_x \quad (1)$$

where T is the corrected contraction stress, T_c is the experimental contraction stress and T_x is the hypothetical stress necessary to elongate the composite to original dimension, defined as

$$T_x = E_c D_c \quad (2)$$

where $E_c = 13,000$ MPa is the Young's modulus of the composite (Laughlin et al., 2002). Composite deformation, D_c , is calculated by

$$D_c = \frac{L_4^0 - L_4^2}{L_4^2} \quad (3)$$

L_4^2 can be obtained taking into account glass and adhesive elongation, where

$$[(L_1^0 + L_2^0 + \Delta l_v) 2] + [(L_3^0 + \Delta l_a) 2] + L_4^2 = L_0 \quad (4)$$

where $L_0 = 10$ mm is the initial and final length of the assembly (i.e., the distance between the clamps of the extensometer) and $L_3^0 = 0.036$ mm is the thickness of the adhesive layer before deformation (Choi et al., 2000).

Glass elongation, Δl_v , depends on glass compliance, C_v , and maximum load, f , according to

$$\Delta l_v = C_v f \tag{5}$$

where f is the maximum contraction force and C_v is the glass compliance (Alster et al., 1997a; Schroeder 2003).

$$C_v = \frac{L_1^0}{A_{v1} E_v} + \frac{L_2^0}{A_{v2} E_v} \tag{6}$$

Glass compliance values (mm/N) are shown in Tab. 2. The adopted Young's modulus of the glass is 64,000 MPa (Laughlin et al., 2002)

Table 2. Glass compliance values (x 10⁻⁵ mm/N) for experimental conditions.

Diameter (mm)	Height (mm)				
	0.63	0.83	1.25	2.5	
2.5	1.57	1.56	1.53	1.47	
5	0.61	0.60	0.58	0.51	
8	0.29	0.28	0.27	0.23	

Adhesive elongation Δl_a depends on adhesive compliance C_a and maximum tensile force f

$$\Delta l_a = C_a f \tag{7}$$

where the adhesive compliance is

$$C_a = \frac{L_3^0}{A_{v1} E_a} \tag{8}$$

and $E_a = 4,780$ MPa is the Young's modulus of the adhesive (Choi et al., 2000).

The computed compliance of the adhesive layer (mm/N) was 3.16×10^{-6} mm/N for specimens with diameter of 2.5 mm, 0.79×10^{-6} mm/N for specimens with diameter of 5 mm and 0.31×10^{-6} mm/N for specimens with diameter of 8 mm.

Statistical Analysis

Stress values were submitted to two-way ANOVA and Tukey test with a 95% confidence interval. Regression analysis for "stress x 'C factor'" and "stress x volume" was performed for the entire sample and grouping the data by diameter.

Results

ANOVA revealed a significant interaction between the main factors ($p < 0.001$). Stress averages and standard deviations are shown in Tab. 3. No significant differences between heights were detected for the smallest diameter. For the 5-mm diameter specimens, there were significant differences among specimens with different heights, particularly among 0.63 mm, 1.25 mm and 2.5 mm, and also between 0.83 mm and 2.5 mm. For the 8-mm diameter specimens, those with 0.63 mm developed statistically higher stress than the other groups, and those with 0.83 mm also present higher stress than the 2.5-mm height specimens.

Table 3. Mean and standard deviation (MPa) of stress values (values followed by the same superscripts are not statistically different, $p > 0.05$).

Diameter (mm)	Height (mm)				
	0.63	0.83	1.25	2.5	
2.5	7.9±0.5 ^{cdef}	7.5±0.4 ^{def}	6.8±0.7 ^{ef}	5.2±0.5 ^{ef}	
5	17.8±1.9 ^b	13.5±1.1 ^{bc}	9.2±0.2 ^{cde}	3.2±1.3 ^f	
8	31.5±5.4 ^a	16.7±2.5 ^b	12.5±1.9 ^{bcd}	7.6±0.8 ^{cdef}	

Regression analyses between stress and 'C factor' are displayed in Fig. 2. Considering the totality of the specimens (N=36), a positive linear correlation was found. However, regression analysis grouped by diameter detected strong non-linear correlations between the two variables. Regression analysis between stress and volume for the entire set of data showed high dispersion, without tendency for correlation. Fig. 3 shows "stress x volume" curves grouped by diameter, evidencing strong non-linear relationships between variables.

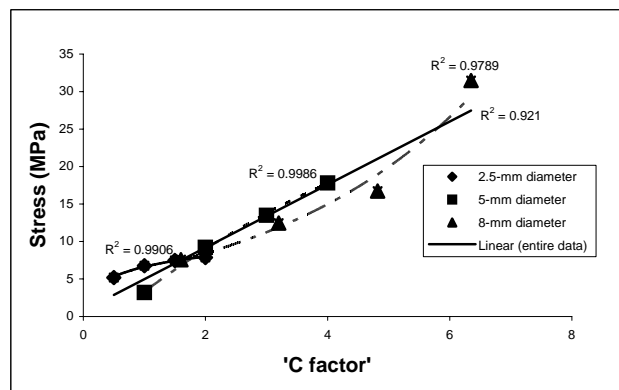


Figure 2. Regression analysis between stress and 'C factor' for different diameters (logarithm function for the 2.5-mm diameter groups, polynomial function for the 5-mm diameter groups and exponential function for the 8-mm diameter groups) and for entire data set (linear function).

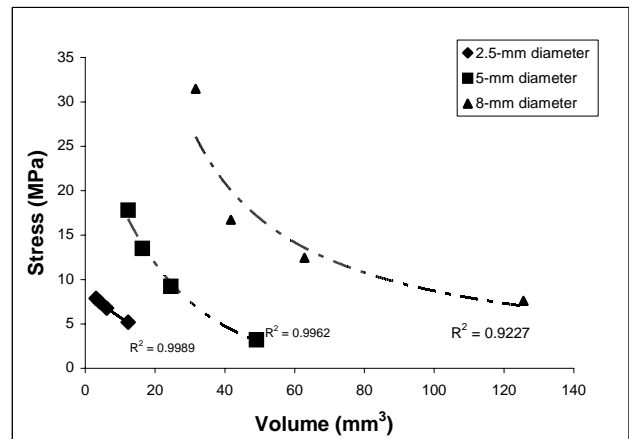


Figure 3. Regression analysis between stress and volume for different diameters (power function for the 8-mm diameter groups and exponential function for 2.5- and 5-mm diameter groups).

Discussion

In the present study, a self-cure composite was used to allow uniform polymerization, regardless of the specimen's dimensions (Alster et al., 1997a). The use of a light-cured material (more frequently employed in restorative dentistry) would be problematic, because light attenuation through the composite would result in undesirable differences in conversion, particularly with the thicker specimens.

Compliance values calculated in the present study are approximately 10 times higher than values related by Alster et al. (1997b). This can be explained by the fact that authors used steel rods, that has Young's modulus much higher than glass (207 GPa and 64 GPa, respectively) (Laughlin et al., 2002). Also, those authors did not use a layer of unfilled resin between the composite and the steel rods. In that study, specimens with 5.35-mm diameter showed stress values (after corrected for compliance) between 14.5 and 5.2 MPa for heights between 0.6 and 2.7 mm. These values are similar to those reported in the present study (between 17.8 and 3.2 MPa for heights between 0.63 and 2.5 mm, for 5-mm diameter).

Stress values varied significantly with specimen height for the 5- and 8-mm diameter specimens. In general, taller specimens developed lower stress values, evidencing an effect that we will call "boundary effect". For large diameters, reductions in height probably hinder the occurrence of polymerization shrinkage transversally to the long axis of the specimen, due to a large bonded area between the glass and the composite. Consequently, a higher fraction of the contraction force tends to manifest longitudinally. In fact, in the present study, a small influence of the boundary effect associated with a relatively high compliance may explain the lack of statistical differences among 2.5-mm diameter specimens.

To some extent, the 'C factor' helps the visualization of the boundary effect. According to the authors that proposed the index, a higher confinement reduces the possibility of plastic deformation during the early stages of polymerization, which is evidenced by the regression curve between stress and 'C factor' for the entire data set. However, the fact that a non-linear effect of 'C factor' on contraction stress (with higher R^2 values than displayed when using the entire data set) was verified when regression analysis was conducted with data grouped by diameter shows that 'C factor' does not fully explain contraction stress development. Moreover, it can be observed in Fig. 2 that, for low 'C factors', the distance between the experimental observation and the regression curve may represent an error close to 50 %.

The influence of composite volume on contraction stress development seems to be less evident than that of the confinement. No regression curve could be fit between volume and stress for the entire data set. Data grouped by diameter revealed non-linear correlations between the two variables. When contraction stress was plotted against "1/volume" as independent variable, linear functions were found for 2.5 mm and 5 mm diameters, while for the 8 mm diameter, the best fit was obtained with the use of an exponential function (Fig. 4). The fact that a non-linear correlation was observed with the largest diameter can be considered another evidence of boundary effect. However, the distance between the curves hinders any prediction based on sample volume.

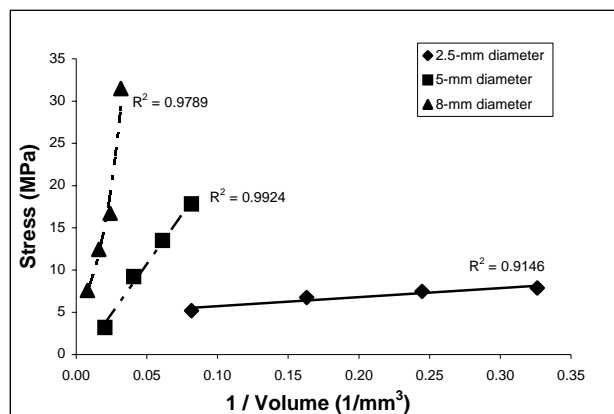


Figure 4. Regression analysis between stress and "1 / volume" index for different diameters (linear function for 2.5 and 5-mm diameters and exponential function for 8-mm diameter).

When the tensiometer is used to determine contraction stress of photoactivated composites, specimen dimensions must be defined in order to allow a homogeneous degree of conversion. Therefore, in order to avoid a gradient of conversion through the thickness of the sample, which would increase the system's compliance, specimen height should be kept around 1 mm (Lim et al., 2002). Specimens with reduced height are easier to build, which is particularly important when self-cure composites are being tested. However, it must be considered that reduced heights tend to increase the influence of boundary restraints.

In the present study, it was evident that specimen dimensions significantly influence contraction stress values. Also, the results suggest that stress values cannot be accurately predicted by 'C factor' or sample volume, because of the influence of boundary constraints defined by specimen's diameter.

Acknowledgements

The authors acknowledge the financial support by FAPESP (00/00550-0) and CAPES.

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