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Post-gel and Total Shrinkage Stress of Conventional and Bulk-fill Resin Composites in Endodontically-treated Molars

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Clinical Relevance

The clinician should consider the polymerization shrinkage stress when selecting a composite resin for posterior restorations. The use of post-gel shrinkage values should guide the selection of a composite resin for posterior teeth.

SUMMARY

Objectives: The objective of this study was to evaluate the effect of the method used for calculation of polymerization shrinkage, total or post-gel, on the shrinkage stress of conventional and bulk-fill composite resins for restoring endodontically treated teeth using finite element analysis.

Methods and Materials: Four composite resins were tested for post-gel shrinkage (P-Shr) by the strain-gauge test and total shrinkage (T-Shr) using an optical method (n=10). Two conventional composite resins, Filtek Z350 XT (3M-ESPE; Z350) and TPH3 Spectrum (Dentsply; TPH3) and two bulk-fill composite resins. Filtek Bulk-Fill Posterior (3M-ESPE; POST) SureFil SDR flow (Dentsply; SDR) were tested. Elastic modulus (E), diametral tensile strength

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(DTS), and compressive strength (CS) were also determined (n=10). The residual shrinkage stress was evaluated by finite element analysis with four restorative techniques: incremental with Z350 and TPH3; SDR/TPH3 (two bulk increments of 4 mm and two occlusal increments); and two bulk increments of 5 mm for POST. Data for P-Shr, T-Shr, E, DTS, and CS were analyzed by analysis of variance and Tukey's test (α=0.05), and residual shrinkage was analyzed quantitatively and qualitatively by the modified von Mises criteria.

Results: SDR had the lowest CS values, POST and TPH3 had similar and intermediate values, and Z350 had the highest CS. TPH3 and Z350 had similar DTS values and values higher than SDR. Z350 and POST had higher P-Shr, and SDR had lower T-Shr. T-Shr resulted in higher shrinkage stress than P-Shr values. SDR/TPH3 resulted in higher shrinkage stress when using T-Shr and lower values when using the P-Shr value.

Conclusion: T-Shr resulted in higher stress in the enamel and in root dentin close to the pulp chamber than P-Shr values. The selection of the T-Shr or P-Shr changed the ranking of the shrinkage stress of the tested composite resin.

INTRODUCTION

Composite resin materials are widely used in restorative dentistry for restoring posterior vital and endodontically-treated teeth.[1,2] During the polymerization reaction, resin undergoes a transition from a predominantly viscous state to a basically solid state due to a conversion of monomer molecules in polymer chains.[3,4] Volumetric shrinkage is an inherent characteristic of resin-based materials.[5,6] Several factors, such as mechanical properties, chemical composition, and the remaining tooth structure, determine the shrinkage stress,[4,7-9] restorative technique, and curing method.[10] This residual stress may result in propagation of enamel cracks, cuspal deflection,[1,11,12] marginal loss, secondary caries, and postoperative sensitivity.[7]

Various restorative protocols, such as the incremental technique,[13] gradual polymerization,[14] and larger increments,[4,8] have been advocated to reduce the shrinkage stress. Recent trends are focused on modifications of the resin matrix, mainly for developing materials with reduced polymerization shrinkage and consequently the shrinkage stress.[15] Bulk-fill composite resins allow application of increments from 4- to 5-mm thickness,[1,16,17] with a high degree of conversion in all increments, reduced shrinkage, and shrinkage stress. Also, they are commercially available in low or high viscosity.[12,18] The viscosity and composition of the bulk-fill composite resin may interfere with the performance of the material during shrinkage measurements.[1,19] Bulk-fill composite resins have shown better or similar performance to the conventional materials in clinical trials and laboratory studies in terms of volumetric shrinkage, polymerization stress, cusp deflection, and marginal quality.[20]

When the material is light activated, the polymerization process develops, and the volumetric reduction causes shrinkage of the material.[6] However, the relationship between shrinkage and stress is not that simple because not all shrinkage causes stress.[6,19,21] Residual stresses will only be generated when the composite material can no longer relax in a timely manner; not all polymerization shrinkage causes shrinkage stresses.[21] It is important to distinguish between total shrinkage and the shrinkage that truly causes stresses, called the "post-gel shrinkage."[6,22] However, the manufacturers continue to inform the clinicians and propagate information regarding different materials using total shrinkage values. This information could be used for creating artificial benefit regarding specific composite resins that can present different rankings regarding total and post-gel shrinkage and total shrinkage reported values.

Shrinkage stresses cannot be measured directly; the use of finite element analysis (FEA) as a computational numerical analysis is considered the most comprehensive method to calculate the complex condition of the stress inside materials and structures.[23] Therefore, the aim of this study was to evaluate the effect of the method used for calculation of polymerization shrinkage on the shrinkage stress of conventional and bulk-fill composite resins for restoring endodontically-treated teeth. The null hypothesis was that there would be no difference in residual shrinkage stress when using the total shrinkage (T-Shr) or post-gel shrinkage (P-Shr) values.

METHODS AND MATERIALS

Study Design

Four commercial materials were used in this study, including two conventional composite resins, TPH3 Spectrum (Dentsply, Konstanz, Germany; TPH3) and Filtek Z350XT (3M ESPE, St Paul, MN, USA;

Table 1: *Composition of Resin Composites (Manufacturer Information)*

Material	CODE	Shade	Composite Resin Type	Increment Size and Light Activation Time	Organic Matrix	Filler	Filler % wt/vol	Manufacturer
TPH3 Spectrum	TPH3	A2	Nanohybrid	2.0 mm, 20 s	BisGMA and BisEMA Dimethacrylate	Boro-silicate/ aluminum/barium glass and silica	75.5/57.1	Dentsply
Filtek Z350 XT	Z350	A2	Nanofilled	2.0 mm, 20 s	BisGMA, BisEMA, UDMA, TEGDMA	Silica and zirconia nanofillers, agglomerated zirconia-silica nanoclusters	82/60	3M-ESPE
Surefil SDR flow	SDR	A2	Bulk-fill flowable	4.0 mm, 40 s	UDMA, BISGMA, EBPADMA, Procrylat resin	Silane treated ceramic and YbF3	68/44	Dentsply
Filtek Bulk-fill posterior	POST	A1	Bulk-fill regular paste	5.0 mm, 10 s occlusal, 10 s buccal, and 10 s palatal	AUDMA, UDDMA, UDMA. EDMAB	Silica, zirconia and YbF3	76.5/58.4	3M-ESPE

Abbreviations: BisEMA, bisphenol A polyethylene glycol diether dimethacrylate; BisGMA, bisphenol A diglycidylmethaycrylate; EBPADMA, ethoxylated bisphenol A dimethacrylate; EDMAB, ethyl-4-dimethylaminobenzoate; TEGDMA, triethyleneglycol dimethacrylate; UDDDMA, dodecanediol dimethacrylate; UDMA, urethane dimethacrylate; YbF3: ytterbium fluoride.

Z350), and two bulk-fill composite resins, the flowable viscosity Bulk Fill Surefil SDR Flow (Dentsply; SDR) and the regular viscosity Filtek Bulk Fill Posterior (3M ESPE; POST). The composition was described according to manufacturer's information (Table 1). All resin composites were tested for P-Shr and T-Shr. Shrinkage stresses were analyzed using FEA.

P-Shr

P-Shr was determined for all tested composite resins (n=10) using the strain-gauge method.[22] The materials were shaped as a dome with 2.8 ± 0.4 mm in diameter and 1.7 ± 0.2 mm in height on top of a biaxial strain gauge (CEA-06-032WT-120, Measurements Group, Raleigh, NC, USA) that measured shrinkage strains in two perpendicular directions (x and y axes). A strain conditioner (ADS2000, Lynx, Tecnologia Eletrônica, São Paulo, Brazil) converted electrical resistance changes in the strain gauge to voltage changes through a quarter-bridge circuit with an internal reference resistance. The strain values measured along the two axes were averaged because the material properties were homogeneous and isotropic on a macro scale. All materials were light-cured using a light-emitting diode (LED) unit Bluephase G2 (Ivoclar Vivadent, Schaan, Liechtenstein) with an intensity of 1600 mW/cm^2 checked by a using Marc Resin Calibrator (BlueLight, Halifax, Canada) with the light tip held at a 1-mm distance from the surface of the composite and monitored for 10 minutes. The mean shrinkage strain was used as linear P-Shr shrinkage input for the FEA and could be converted to the volumetric percentage by multiplying by 3 and 100%.

T-Shr

T-Shr was calculated using the optical method.[5] Each resin sample (n=10) was shaped as a dome with 2.8 ± 0.4 mm in diameter and 1.7 ± 0.2 mm in height on a platform made of addition silicone (Express XT–Light body, 3M ESPE) in a blue color, for a better contrast between resin and surface. Each sample was slightly rounded and placed on the silicone platform. The platform was placed under a microscope coupled to a camera (SZX16 & UC30, Olympus, Tokyo, Japan) so that the images could be captured in a standardized way. Once positioned, a photograph was taken before photoactivation. The light from the microscope was attached only to capture the images to avoid premature polymerization of the compound through the optical microscope. Immediately after the first photograph and before polymerization of the material, the sample was light-cured for 40 seconds using the Bluephase G2 (Ivoclar Vivadent). After polymerization, the samples were monitored, and photographs were taken after 10 and 60 minutes of the initial light activation. All procedures were performed at room temperature (22°C). ImageJ software (National Institutes of Health, Bethesda, MD, USA), available free of charge on the internet, was used to evaluate pre- and post-polymerization photos. The brightness of the image was adjusted to determine the maximum contrast between the resin sample and the silicone

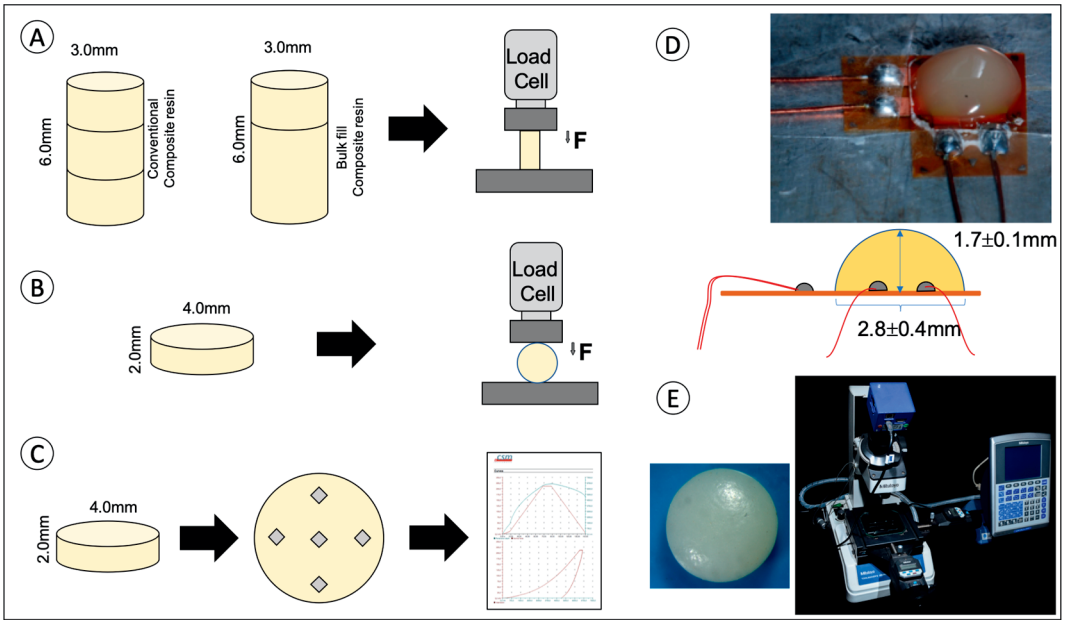

Figure 1. *Schematic diagram of sample preparation and measurement methodology of mechanical properties recorded experimentally; (A) CS, (B) DTS, (C) E, (D) P-Shr, (E) T-Shr.*

plate. Using the tool "wand tool," the contour of the sample was defined to obtain the total area. Through the photograph of the total area of the samples before and after polymerization, it was possible to calculate the volume change before and after polymerization.

Compressive Strength and Diametral Tensile Strength

Compressive strength (CS) and diametral tensile strength (DTS; n=10) of the composite resins were calculated (Figure 1A and B, respectively). For characterization of the mechanical properties, the sample preparation for CS and DTS was performed using the maximum thickness recommended for each material and did not represent exactly the same protocol simulated in FEA. The composite resin was inserted into a cylindrical polytetrafluoroethylene mold for the CS test (6 mm height, 3 mm diameter) or the DTS test (2 mm height, 4 mm diameter). The specimens for the CS test made with bulk-fill composites were polymerized with 4.0 mm for the first increment and 2.0 mm for second increment. For conventional composites, the specimens were polymerized in three 2.0-mm increments using Bluephase G2 (Ivoclar Vivadent). The specimens were stored in distilled water for 24 hours at 37°C and afterward were submitted to CS and DTS testing in a universal testing machine (DL2000, EMIC) at a crosshead speed of 0.5 mm/min until failure occurred. CS values were calculated by dividing the fracture load (F) by the cross-sectional area and converting it into MPa. DTS values were calculated using the following equation: $DTS = 2F/\pi dt$, where d is the specimen diameter, and t is the height of the specimen. DTS and CS values were converted into MPa.

Elastic Modulus

For elastic modulus (E) measurements, disc-shaped specimens similar to DTS samples (2 mm height, 4 mm diameter) were prepared.[1] Using a Vickers indenter (CSM Micro-Hardness Tester, CSM Instruments, Peseux, Switzerland), indentations were made at the surface of the finished and polished composite resin surface. The indentations were carried out with controlled force, whereby the test load was increased or decreased at a constant speed ranging between 0 and 500 mN in 20-second intervals. The maximum force of 500 mN was held for five seconds. The load and the penetration depth of the indenter were continuously measured during the load-unload cycle. The universal hardness was defined as the applied force divided by the apparent area of the indentation at the maximum force. The indentation modulus, comparable to the material's E, was calculated from the slope of the tangent of the indentation depth curve at the maximum force.

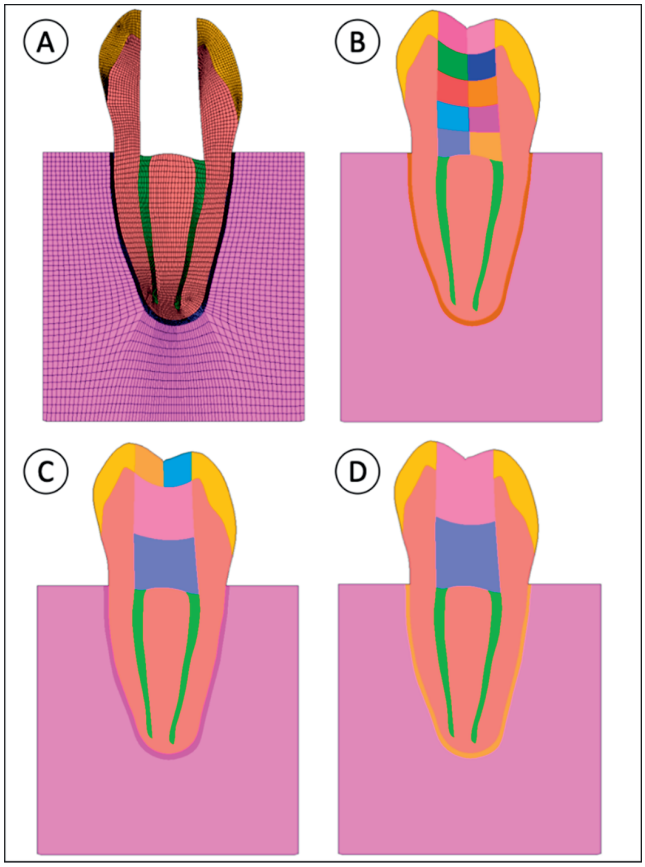

Figure 2. *Two-dimensional FEA model generation. (A) Principal model: mesh of endodontically treated tooth with a MOD preparation. (B) Model simulating incremental filling technique using Z350 or TPH3, resulting in 10 increments. (C) Model simulating SDR flowable bulk fill used for restoring the dentin (two increments of 4.0 mm) and two occlusal increments of TPH3 replacing the enamel (2.0 mm), resulting in four increments. (D) Model simulating POST replacing the dentin and enamel (two increments of 5.0 mm), resulting in two increments.*

Residual Stress Calculation: FEA

To calculate T-Shr and P-Shr residual shrinkage stresses, a two-dimensional (2D) finite element simulation was carried out simulating a mesial-occlusal-distal restoration of an endodontically-treated molar as described in a prior study.[2] The geometric model (Figure 2A) was based on a digitized buccolingual cross section of an endodontically-treated molar embedded in an acrylic resin cylinder with a simulated periodontal ligament (PDL). The coordinates of points were obtained using public domain ImageJ software (ImageJ 1.48, National Institutes of Health). The mechanical properties used were as follows: enamel, E = 84 GPa and Poisson's ratio (η) = 0.30[24]; dentin, E = 18 GPa and η = 0.23[23]; polystyrene resin, E = 13.5 GPa and η = 0.31; and polyether, E = 0.05 and η = 0.45.[24] For the restorative composite resins, the E was experimentally determined and is shown in Table 2. The Poisson's ratio was chosen to be the same for all composites at 0.24.[23] It was assumed a plane stress condition for the composites and a plane strain condition for the tooth cross sections, PDL, and acrylic resin cylinder.

The FEA was performed using MSC.Mentat (preprocessor and postprocessor) and MSC.Marc (solver) software (MSC Software, Santa Ana, CA, USA). One FEA model was generated for each of the four restorative protocols of the experimental study (Figure 2A). The Z350 and TPH3 restorations were built and cured in six increments (Figure 2B and C, respectively). The bulk-fill flowable, SDR, was placed in two increments up to 4 mm and covered with TPH3 built with two increments of 2 mm thickness on the occlusal surface (Figure 2D). The bulk-fill regular paste, POST, was used in two increments of up to 5.0 mm (Figure 2D). Polymerization shrinkage was simulated by thermal analogy. The temperature was reduced by 1°C, and the linear shrinkage value P-Shr or T-Shr was entered as the coefficient of linear thermal expansion. Nodal displacements were the constraint in the x and y directions at the bottom and lateral surfaces of the support cylinder. Modified von Mises equivalent stress (mvm) was used to express the stress conditions, using the ratio of the CS and DTS strengths. The CS and DTS used for calculating the ratios for the restorative materials are shown in Table 2 and were calculated using the experimentally determined CS and DTS values. The CS and DTS strengths for enamel were 384.0 and 10.3 MPa and for dentin were 297.0 and 98.7 MPa, respectively.[23] The mean values of the 10% highest mvm stresses were determined for the enamel and

Table 2: *Experimentally Determined Mean (and SD) Volumetric P-Shr, volumetric T-Shr, CS, DTS, and E.*[a]

Composite Resin	P-Shr (%)	T-Shr (%)	CS (MPa)	DTS (MPa)	E (GPa)
Filtek Z350 XT (Z350)	0.7 ± 0.1 C	2.6 ± 0.2 B	255.5 ± 30.4 A	47.5 ± 6.5 A	18.5 ± 2.0 A
TPH3 Spectrum (TPH3)	0.5 ± 0.0 B	2.5 ± 1.3 AB	164.4 ± 25.3 B	49.9 ± 7.2 A	15.6 ± 1.4 B
Filtek Bulk Fill Posterior (POST)	0.7 ± 0.1 C	2.1 ± 0.3 A	169.3 ± 15.8 B	42.4 ± 6.3 AB	12.8 ± 1.7 BC
Surefil SDR flow (SDR)	0.4 ± 0.1 A	3.9 ± 0.8 C	121.6 ± 13.4 C	39.4 ± 5.7 B	10.1 ± 0.9 C

[a] *Different letters mean significant difference between composite resins for each mechanical property provided by Tukey test ($p<0.05$).*

dentin structures when simulating the T-Shr and P-Shr. Fortran custom-made subroutines were generated for obtaining mvm stresses values from enamel–composite and dentin–composite interfaces.

Statistical Analysis

The P-Shr, T-Shr, E, DTS, and CS data were tested for normal distribution (Shapiro-Wilk) and equality of variances (Levene's test), followed by parametric statistical tests. One-way analysis of variance (ANOVA) was performed for each mechanical property. Multiple comparisons were made using Tukey's test. All tests used an $\alpha=0.05$ significance level, and all analyses were carried out with the statistical package Sigma Plot version 13.1 (Systat Software, Sao Jose, CA, USA). The modified von Mises stresses values were analyzed qualitatively.

RESULTS

P-Shr and T-Shr (%) of Resin Composites

The P-Shr and T-Shr mean values and standard deviations (SDs) of the four composites are shown in Table 2. ANOVA showed a significant difference between the resin composites ($p<0.05$). Tukey's test showed that POST and Z350 had the highest P-Shr values, and SDR and TPH3 had lower P-Shr values (Table 2). SDR showed higher T-Shr values than did other resin composites (Table 2). Z350 and TPH3 had intermediate values, and POST had the lowest T-Shr values.

DTS (MPa), CS (MPa), and E (GPa) of Resin Composites

The DTS, CS, and E mean values and SDs of the four composites are shown in Table 2. ANOVA showed a significant difference between the resin composites ($p<0.05$). Tukey's test showed that Z350 had the highest CS values, and SDR had lower Shr values (Table 2). TPH3 had the highest DTS values, but they were not significantly higher than Z350 or POST, and SDR had the lowest values. Regarding E results, SDR had significantly lower values than Z350 and TPH3; Z350 had higher value than all other resin composites.

Shrinkage Stress (MPa) FEA Results

Shrinkage stresses, expressed by modified von Mises values, generated by all tested restorative techniques are shown in Table 3 and Figures 3 and 4. TPH3 and Z350 groups showed the highest stress values at enamel and dentin structures at T-Shr and P-Shr (Table 3). The models generated with T-Shr values (Figure 3) resulted in a substantial increase of shrinkage stress compared with models with P-Shr values, irrespective of the restorative technique.

Table 3: *The 10% Highest Mean (SD) Values of Modified von Mises Stress (MPa) Extracted From Enamel and Dentin Structures Calculated Using T-Shr and P-Shr of Conventional and Bulk-fill Composite Resins.*

Composite Resins	T-Shr		P-Shr	
	Enamel	Dentin	Enamel	Dentin
Z350XT	209.3 (50.9)	53.9 (26.0)	55.3 (13.5)	14.3 (6.9)
TPH	187.2 (46.1)	49.3 (23.8)	38.4 (9.5)	10.1 (4.9)
SDR/TPH	156.1 (15.9)	36.9 (15.1)	23.1 (2.0)	4.3 (1.8)
POST	68.4 (10.8)	23.5 (8.6)	22.5 (3.5)	7.7 (2.8)

T-Shr and P-Shr shrinkage-interface stress values for the different resin techniques are presented in Figure 4. SDR/TPH3 had the highest difference between the stress generated on the enamel–composite and dentin–composite interface when using T-Shr and P-Shr (Figure 4C). The T-Shr values generated elevated peak stress values at the limits of the increments and also at the transition of the tooth structures.

DISCUSSION

The selection of direct restorative material with high performance and durability has led to the development of new materials. The change from the use of the incremental technique to the bulk-filling method may be a result of the simplified restorative technique that reduces the possibility of errors, has fewer operative steps, and requires less clinical

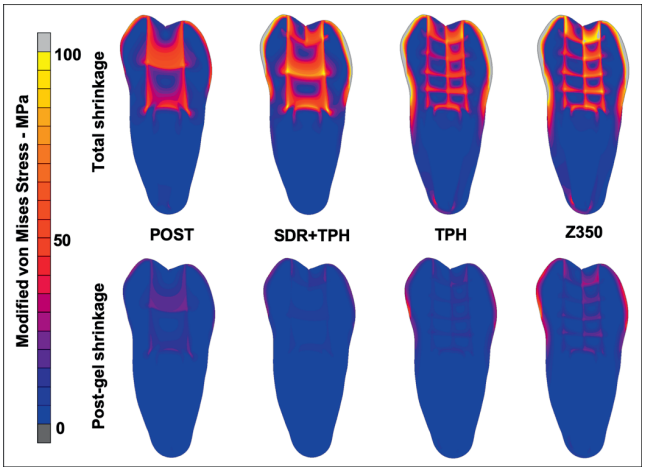

Figure 3. *Modified von Mises shrinkage stress (MPa) distribution for the different restorative materials and techniques after polymerization using T-Shr values and P-Shr values.*

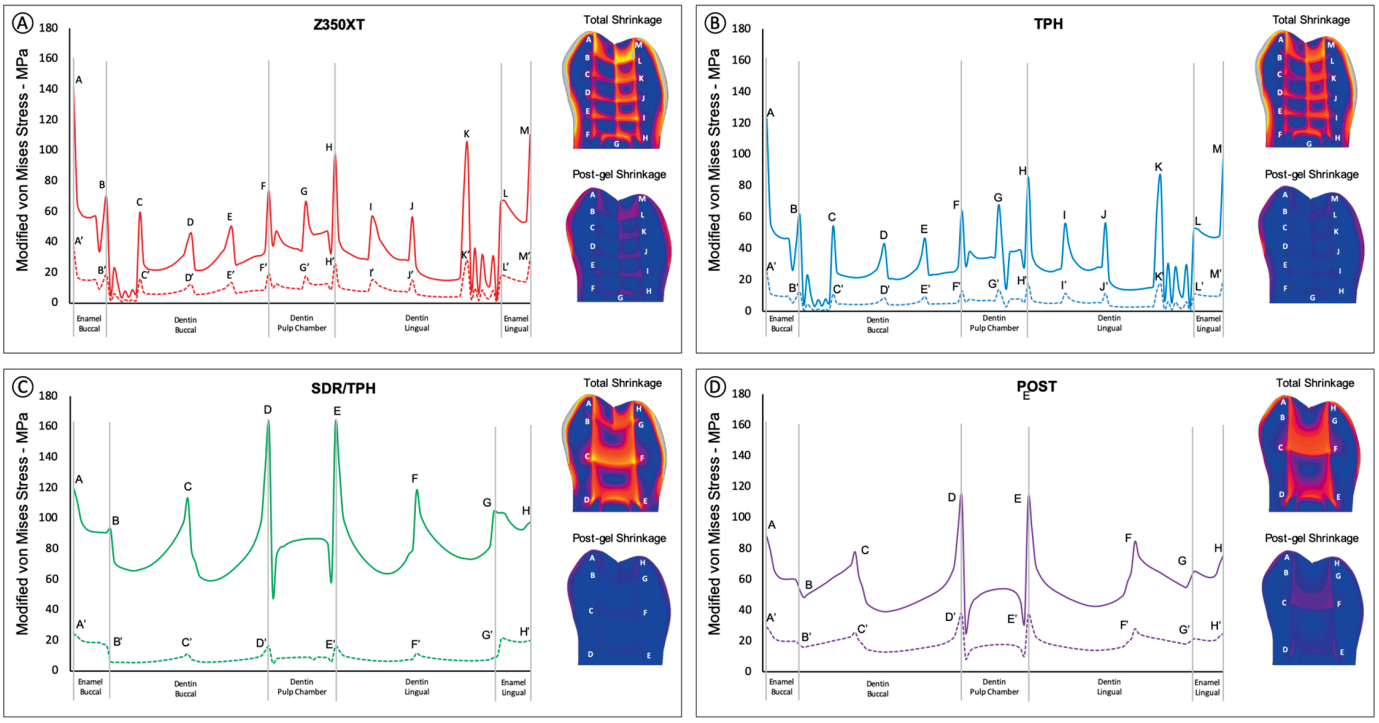

Figure 4. *T-Shr and P-Shr shrinkage modified von Mises stresses from composite–enamel and dentin–composite interfaces values for the different resin techniques.*

time.[1] The results of this study confirmed that P-Shr and T-Shr were dependent on material composition and that those values influenced residual shrinkage stress; therefore, the null hypothesis was rejected.

Volumetric shrinkage is a consequence of the polymerization of resin-based materials during formation of a polymer network, causing dimensional changes.[5] It is known that not all polymerization shrinkage causes shrinkage stresses.[19,21] The total shrinkage measures the entire amount of dimensional change of the composite during polymerization including the pre-gel stage, capable of flowing by releasing part of its tension, and P-Shr stage.[21,26,27] This fact may explain why the percentage of volumetric total shrinkage of all four tested composites in this study presented values significantly higher than percentage results for P-Shr shrinkage. Residual stresses will only be generated when the composite material cannot timely relax anymore, which happens when a more rigid polymer network structure has developed, occurring only in the P-Shr phase.

The percentage of polymerization shrinkage is influenced by the amount of organic content of the composite[28] and the percentage of inorganic content.[29] The change of a resin composite to a solid material, characterized by the development of the elastic modulus, during the polymerization results in rigid restorations and consequently residual shrinkage stresses by the effect of polymerization contraction.[1] The P-Shr of the Z350 composite resin was higher than the others due to the high content of filler in the resin matrix, which increases the modulus of elasticity and rigidity of the material[8] and tends to increase the shrinkage stress. The higher P-Shr of the POST composite resin can probably be explained by the higher polymerization ratio obtained by the modification on the modulation of the polymerization reaction in deeper areas. The lower post-gel shrinkage value of SDR could be explained by the lower viscosity that results in more flexibility and also by the amount of inorganic filler content, an attribute of flowable composites.[30] Additionally, the SDR resin has a polymerization modulator that acts at propagation of the linear and branched chains of the polymers of the resin, reducing the formation velocity of the polymer network, keeping its viscosity longer and providing lower stress compared with traditional composites.[31] Inversely, SDR presented the highest total shrinkage value, probably due to the smaller amount of filler content. The total volumetric contraction depends on factors such as the size of the charge particles and also the type of the organic matrix and concentration of monomers.[32]

The Filtek Bulk Fill Posterior had intermediate E value and the highest P-Shr values; however, the POST showed the lowest modified von Misses shrinkage stress. The smaller number of increments, only two increments of 5 mm, and consequently, only two light activations can explain this finding. The SDR/TPH3 had four increments, and TPH3 and Z350XT had 10 increments. When restoring a tooth, increasing the number of increments results in higher stresses in the remaining tooth structure and at the tooth/restoration interface.[4,8] The combination of higher elastic modulus of TPH3 and Z350XT and the significantly larger number of increments creates higher stress for both incremental filling techniques. Bulk-filling techniques reduce the increment number by using a larger volume of the composite, which results in lower residual shrinkage stress compared with the incremental filling techniques. The residual stress is known to concentrate at the cervical enamel.[1,4,33] The lower shrinkage stress observed when flowable composite resin in combination with conventional composite resin may be explained by the lower P-Shr and E values that determine higher stress relief during the polymerization reaction.[1,3] The stress presented at the root dentin close to the pulp chamber was already expected once the model simulated an endodontically-treated tooth with the pulp chamber totally filled with resin composite. Therefore, the bulk-fill resin composites show up as alternatives to restore endodontically-treated teeth with lower shrinkage stress than the incremental technique, due to the lower stress generated at severely compromised dental structures.[32] The highest values of residual shrinkage stress were verified when the total shrinkage values were simulated compared with the post-gel values. The total shrinkage considers the total volumetric shrinkage of the increment, as presented by most manufacturers; however, in reality, the shrinkage is not correlated with the volume change involved in stress.[19] The post-gel shrinkage concept has turned out to be a useful factor to develop an understanding of the development and calculation of shrinkage stresses. This study had limitations. Only a general 2D model was used. The use of a 3D model can demonstrate the stress concentration along the gingival margin. From a clinical perspective, this study highlighted that the use of a bulk-fill technique may contribute to less shrinkage stress generation on endodontically-treated molars, which are normally more susceptible to fracture and crack propagation.[34,35] It is important that the clinician be capable of differentiating the type of shrinkage when choosing a dental composite; total shrinkage does not reflect shrinkage stress behavior, which is much better characterized by post-gel shrinkage.

CONCLUSIONS

The use of total shrinkage values for finite element simulations resulted in higher stress in the enamel and in root dentin close to the pulp chamber than when post-gel shrinkage values were used. The selection of total or post-gel shrinkage changed the ranking of the shrinkage stress of the tested composite resins. The use of post-gel shrinkage values should guide the clinician in composite resin selection for posterior teeth.

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the Federal University of Uberlândia.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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