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Is there correlation between polymerization shrinkage, gap formation, and void in bulk fill composites? A μCT study

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Abstract: This *in vitro* study aimed to evaluate the volume of polymerization shrinkage (VS), gap (VG), and void (VV) using computerized microtomography (μCT) in bulk fill resin composites and conventional class I restorations, and to establish a correlation between these factors. Class I cavities (4 x 5 x 4 mm), C-factor = 4.2, were performed on caries-free human third molars and randomly divided into five groups (n = 6): FSI (Filtek Supreme XTE incremental insertion); FSS [(Filtek Supreme XTE single insertion(SI)]; TBF [(Tetric Bulk Fill: SI and manual filling (MF)]; SFM (Sonic Fill: SI/MF); and SFS (SonicFill: SI and sonic filling). The teeth were scanned and analyzed by μCT at T0, after filling the cavity with resin, and at T1, after polymerization for VG and VV, and for VS (T1-T0). There was statistically significant difference in VS in μCT for the FSI and FSS groups and between SFS and FSS as well as some difference in VV for FSI and bulk fill resin composites and no difference in VG between the conventional technique and bulk fill composites. Bulk fill resin composites presented similar VS and gap formation to those of incrementally inserted conventional resin composites. There is a moderate and weak positive correlation between polymerization shrinkage and gap formation and void, respectively. The final gap formation was more dependent on the initial gap than on polymerization shrinkage or void volume.

Keywords: Polymerization; X-ray Microtomography; Composite Resins; Imaging, Three-Dimensional.

Introduction

Resin composite (RC) performance has been the focus of many studies, mainly because of polymerization shrinkage and stress.[1] There is ample evidence that the stress resulting from polymerization shrinkage in RCs may have deleterious effects such as marginal infiltration, cuspal deflection, dental cracking, reduction in bond strength, low mechanical properties, and gap formation.[2] The control of these clinically relevant effects is of fundamental importance. Major efforts have been put into the improvement and development of materials and techniques that use new polymerization strategies, and also into the control of their effects on the tooth/restoration interface.[1,3]

During the polymerization of RCs, double and simple bonds of carbon monomers form polymer chains with covalent bonds, resulting in 1.5 to 5% of volumetric shrinkage.[4] When polymerization shrinkage is larger than the bond strength, it leads to the formation of marginal cracks, and such cracks may range from 1.67 to 5.68% of the total restoration volume.[5] It should be noted that polymerization shrinkage is greatly influenced by the configuration factor (C-factor). Cavities with a higher C-factor may have lower internal adaptation of the restorative material.[6] The incremental filling technique has been recommended for decreasing polymerization shrinkage; however, it is very difficult to prove that the incremental technique needs to be maintained to reduce shrinkage effects.[7] Increasing the number of increments causes higher stress on the remaining tooth structure and at the tooth/restoration interface, as well as high post-gel shrinkage and/or elastic modulus values;[8] in addition, the type of composite and filling technique affects the mechanical properties of large restorations.[9]

In recent years, bulk fill RCs have been proposed to decrease polymerization shrinkage and gap formation in the pulp wall,[10] inserted in a single thick layer (4–5 mm).[11,12] RCs seem to have low polymerization shrinkage and a small percentage of voids,[13] besides large clinical acceptance,[14,15] since they simplify the restorative process.[11] Another factor that should be considered is the sonic insertion of RCs as a way to reduce the size and number of spaces/voids in the material, as proposed by some manufacturers.[16]

Despite the great number of studies on polymerization shrinkage, the presence of voids and spaces in the restoration is a negative aspect that has been widely overlooked in the literature.[16] Voids and spaces may accelerate material deterioration, resulting in marginal infiltration and discoloration, higher wear, and lower flexural strength.[17] These spaces between the increments can be added during the manufacturing process or during RC insertion.[13,18] Thus, it is recommended that handling of the material be minimal to prevent air from entering the matrix, which will end up forming voids and decreasing longevity.[18]

Polymerization shrinkage and/or gap formation has been evaluated in the literature by means of different destructive tests,[19,20,21,22,23] hindering a more detailed analysis of the resin composite body before and after polymerization. Computerized microtomography (μCT) has been used to quantify and evaluate polymerization shrinkage,[22,23] in order to examine the tooth/restoration interface, as well as other changes in the material. This type of analysis eliminates the need for cuts and stresses to the tooth, unlike other methods, such as scanning electron microscopy (SEM).[19] μCT is a safe and nondestructive method that can analyze the material in 3D,[24] but it is infrequently used to quantify spaces and voids in restorative materials.[16]

Few studies have shown the behavior of manually and sonically inserted bulk fill RCs towards polymerization shrinkage, gap, and void formation in large class I restorations. Thus, this *in vitro* study aimed to correlate polymerization shrinkage, gap, and void in high C-factor cavities restored with conventional composites and bulk fill, using μCT for the analysis. The following null hypotheses were tested: a) there is no difference in the volume of polymerization shrinkage (VS), gap (VG), and void (VV) between bulk fill and conventional RCs; and b) there is no correlation between polymerization shrinkage, gap, and void in bulk fill and conventional RCs.

Methodology

This study was approved by the local Human Research Ethics Committee (process no. 1708531).

Sample preparation

Thirty caries-free, recently extracted human third molars were previously selected by crown size using a digital caliper (Mitutoyo Co., Tokyo, Japan), and cleaned and stored in thymol at 0.5%. Subsequently, prophylaxis and storage of the teeth were carried out in distilled water at 37 ± 1ºC (24 h). Standard class I cavities (4 x 5 x 4 mm) with a high C-factor (C = 4.2) were made with diamond tips No. 1090 and 1014 (KG SORENSEN, Cotia, Brazil) at high rotation under refrigeration. The diamond tips had a standardized active tip and a vertical stop to provide equal depth

during preparation of the wells. At the end of the preparation, the cavities were measured with a digital caliper. The diamond tips were replaced after every five cavity preparations.[23] The live internal angles were rounded with tip 1014[21] in order to facilitate material adaptation and force dissipation. After cavity preparation, the teeth were randomly divided to the following groups (n = 6 each): group 1 = FSI (Filtek Supreme XTE - incremental insertion); group 2 = FSS (Filtek Supreme XTE - single insertion); group 3 = TBF (Tetric Bulk Fill – manual filling); group 4 = SFM (SonicFill - manual filling); and group 5 = SFS (SonicFill – sonic filling). The restorative materials and their respective insertion/filling information and techniques are described in Table 1.

Restorative procedure

After preparation of the samples, enamel and dentin were etched with phosphoric acid at 37% (FGM, Joinville, SC, Brazil) for 30 and 15 s, respectively, followed by rinsing for 20 s. Excess water was removed with thin absorbent paper, followed by the application

Table 1. Technique and materials used in the study.

Code	Material/Manufacturer	Composition	Batch No.	Insertion	Technique
FSI	Filtek Supreme XTE / 3M ESPE, St. Paul, MN, USA	Non-agglomerated silica nanoparticles (20 nm), non-agglomerated zirconia (4 to 11 nm). 78.5 wt% and 63.3 vol%. Matrix: Bis-GMA UDMA, TEGDMA, PEGDMA and bis-EMA	387672	3 increments (≅1.3 mm each)	II/MF
FSS	Filtek Supreme XTE / 3M ESPE, St. Paul, MN, USA	Non-agglomerated silica nanoparticles (20 nm), non-agglomerated zirconia (4 to 11 nm). 78.5 wt% and 63.3 vol%. Matrix: Bis-GMA UDMA, TEGDMA, PEGDMA and bis-EMA	387672	Single increment (4 mm)	SI/ MF
TBF	Tetric Bulk Fill / Ivoclar Vivadent, Schaan, Liechtenstein, GE	Barium aluminum silicate glass with two different mean particle sizes, an isofiller. Ytterbium fluoride and spherical mixed oxide), Ivocerin initiator, 79-81 wt%, 61 vol% and 17vol% "Isofillers". Matrix: BisGMA, BisEMA, UDMA	U03089	Single increment (4 mm)	SI/ MF
SFM	SonicFill / Kerr, Orange, CA, USA	Barium glass, silicon dioxide (5-10%), oxide, chemicals (10-30%), MPS (10-30%), silicon dioxide, EBPDMA (1-5%), bisphenol A bis (2-hydroxy-3-methacryloxypropyl) ether (1-5%), and TEGDMA (1-5%) (Filler 83.5 wt%)	5560135	Single increment (4 mm)	SI/ MF
SFS	SonicFill / Kerr, Orange, CA, USA	Barium glass, silicon dioxide (5-10%), oxide, chemicals (10-30%), MPS (10-30%), silicon dioxide, EBPDMA (1-5%), bisphenol A bis (2-hydroxy-3-methacryloxypropyl) ether (1-5%), and TEGDMA (1-5%) (Filler 83.5 wt%)	5560135	Single increment (4 mm)	SI/ SF
	Adper Single bond 2, 3M ESPE, St. Paul, MN, USA	Bis-GMA, HEMA, dimethacrylates, photoinitiator, methacrylate functional copolymer of polyacrylic and polyitaconic acids, 10% by weight of 5 nanometer-diameter spherical silica particles, water, ethanol	N677700	Apply two consecutive coats of adhesive to the tooth surface with gentle agitation for 15 seconds; gently air thin; light cure for 10 seconds	

Bis-EMA: Bisphenol-A polyethylene glycol diether dimethacrylate; Bis-GMA: Bisphenol-A diglycidyl ether dimethacrylate; EBPDMA: Ethoxylated Bisphenol-A-dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; PEGDMA: polyethylene glycol dimethacrylate; UDMA: Urethane dimethacrylate, MPS: 3-(trimethoxysilyl) propyl methacrylate; HEMA: 2-Hydroxyethyl methacrylate; II: Incremental insertion; SI: single insertion; MF: manual filling; SF: sonic filling.

of Adper Single Bond 2 adhesive (3M, ESPE, ST Paul, USA) according to the manufacturer's instructions and then light-cured for 10 s with a high-power LED polymerization apparatus (Bluephase, Ivoclar Vivadent AG, Austria). The samples were protected from any light sources in dark plastic vials and placed in the microtomograph for scanning and volume quantification before light curing. In the microtomograph, there was no light incidence once Skyscan 1176 (V. 1.1.10, Skyscan, Kontich, Antwerp, Belgium) operational protocol uses a dark environment. Resin composite material was inserted in each of the groups as described in Table 1, and condensation was performed with a cosmedent SP2 spatula (Cosmedent, Chicago, USA). All increments were light-cured for 40 s with 1200 mW/cm^2.[25] The same operator performed all the restorative procedures. The specimens were then stored in distilled water and kept in an oven at 37°C ± 1°C for 24 h.

Computerized microtomography (μCT)

Computerized microtomography was used to analyze the restored cavities. Each tooth was scanned twice: at T0 – after insertion of the RC material, and at T1 – after the final cure. The microtomograph used a power of 90 Kvp and 275 microamperes with a resolution of 17.48 μm (Cu filter = 0.1 mm). The total number of slices averaged 250, with an average reading time close to 28 min.

The μCT data were then imported into a workstation and evaluated with the NRecon software (Version, 1.6.10.4-2015, Skyscan, Kontich, Antwerp, Belgium). The threshold was standardized by an average of the base algorithm for the components of the control group, thus eliminating any bias between the samples. The images were standardized using the DataViewer (V.1.5.2.4) software and the analyses were performed through the 3D analysis tool from CTAn (CT-Analyser software v1.10.1.0; Skyscan, Kontich, Belgium) based on the volume of black spaces (void spaces) present in the volume of interest (VOI), which consisted of the summation of all 2D images within the region of interest (ROI). In the CTVol software, the two scan images were superimposed, allowing us to get a good arrangement. All calculations were performed using the VOI obtained from the ROI centered on the delimitations of the restorative material (Figure 1).

The initial reading (T0) was considered "reference," and the final reading (T1) was considered a "target" for the geometric alignment of the images. The reference and target images were analyzed individually, and shrinkage, gap, and void were determined by the difference between the reference and target samples. The volume of polymerization shrinkage, gap, and void was calculated through the analysis of the anatomical structure of the restoration and was expressed as percentage.[24]

Sample size calculation

The data were analyzed by the STATA 14 (College Station, USA) software. Sample size, taking into account inter-group variation (147.2) and intra-group variation (104.8), as reported elsewhere,[26] resulted in four specimens per group with a statistical power of 80% and an alpha of 5%.

Statistical analysis

The Shapiro-Wilk test indicated that, among the three evaluated outcomes, polymerization shrinkage and void values were not normally distributed. These data were analyzed using the Kruskal-Wallis test, followed by Dunn's post-hoc test. ANOVA was used for the gap volume, followed by Tukey's test. The correlation between the volume of shrinkage, void, and gap was obtained by Spearman's rank correlation coefficient. A multivariable linear regression was performed to model the relationship between the final gap (dependent variable) and the initial gap,

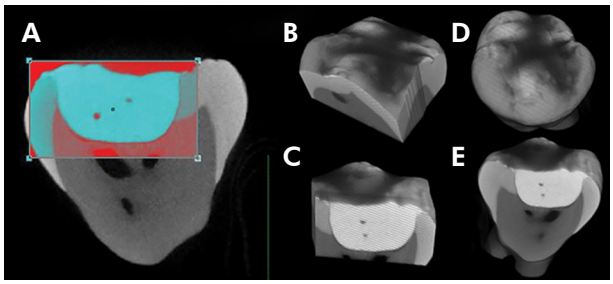

Figure 1. 3D reconstruction of cavity filled with resin composites. Illustrative 2D slice showing the region of interest - ROI (Figure A - red square). Restoration at different angles (B, C, D, and E).

VS and VV (independent variables). In all analyses, the significance level was set as $p < 0.05$.

Results

The results of the μCT analysis were expressed as a percentage of volumetric polymerization shrinkage, gap, and void.

Volume of polymerization shrinkage (VS)

Unlike the evaluation of the volume of gap (VG) and void (VV), the analysis of volumetric polymerization shrinkage (VS) took into account the difference in the volume of RCs in each time period (T1-T0) as that represents the shrinkage of the studied material. All of the RCs exhibited polymerization shrinkage (SFI < TBF and FSI < SFM < FSS). A statistically significant difference was observed in the volume of polymerization shrinkage between FSI and FSS ($p = 0.03$) and between SFS and FSS ($p = 0.01$) (Table 2).

Volume of gap (VG)

Gap volume was measured at nine different points from the pulp wall to the restoration (Figure 2), and the sum of these points determined the total volume of gap per tooth at two different moments: VG0 and VG1. The analysis of the gap and void volume on two different occasions sought to show the presence of gap after RC resin composite insertion and after polymerization of the material. Note that the difference between the final and initial times only shows the size of the gap increase rather than its real volume before and after polymerization.

The presence of gap at the tooth/restoration interface was observed in all groups before (VG0) and after (VG1) light curing (Table 2 and Figure 3). Group 4 (SFM) presented greater gap formation before and after light curing. There was no significant statistical difference in the presence of gap between the groups on the two occasions: before (Kruskal-Wallis, $p = 0.43$) and after light curing ($p = 0.64$) (Table 2).

Volume of void (VV)

The void volume was considered for all spaces, voids, and porosity observed in the body of the material before (VV0) and after light curing (VV1). The groups treated with conventional RCs were those with the lowest void volume. The analysis of VV was similar to that of VG and showed a statistically significant difference at VV0 between FSI and TBF ($p < 0.001$), FSI and SFM ($p= 0.008$), FSI and SFS ($p = 0.003$), and FSS and TBF ($p = 0.01$). In the same way, some difference in RCs was observed at VV1 for FSI and TBF ($p = 0.001$), FSI and SFM ($p = 0.02$), and FSI and SFS ($p = 0.01$).

Correlation between the volume of shrinkage, gap, and void

A moderate positive correlation ($p = 0.003$, $r = 0.538$) was observed between the volume of shrinkage (%VS) and the volume of the final gap (%VG1) (Figure 4A),

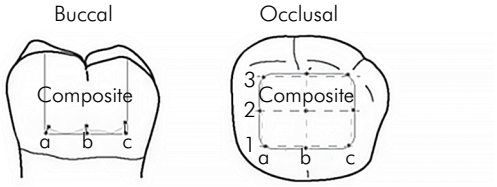

Figure 2. Analysis of gaps at the resin/tooth interface. *Point of gap measurement by region.

Table 2. Volume of polymerization shrinkage (VS), gap (VG), and void (VV) using μCT (mean/SD*).

Experimental groups	VS (%)	VG		VV	
		VG0 (%)	VG1(%)	VV0 (%)	VV1(%)
FSI	1.21/0.99^{b}	1.95/0.90^{a}	2.19/0.85^{a}	9.04/3.74^{a}	9.87/4.77^{a}
FSS	2.91/1.05^{a}	1.75/0.50^{a}	1.97/0.48^{a}	12.01/2.43^{ab}	13.35/3.28^{ab}
TBF	1.21/0.50^{ab}	1.80/0.67^{a}	2.30/0.75^{a}	18.23/2.27^{c}	19.27/2.39^{b}
SFM	1.69/0.59^{ab}	2.54/0.70^{a}	2.95/0.93^{a}	15.45/3.47^{bc}	16.56/3.66^{bc}
SFS	1.01/0.48^{b}	2.14/0.93^{a}	2.58/0.81^{a}	16.09/2.74^{bc}	16.75/2.88^{bc}

"0": after the insertion of the composite resin; "1": after the final cure; SD: standard deviation. *Means with the same superscript letters are not statistically different from each other ($p < 0.05$).

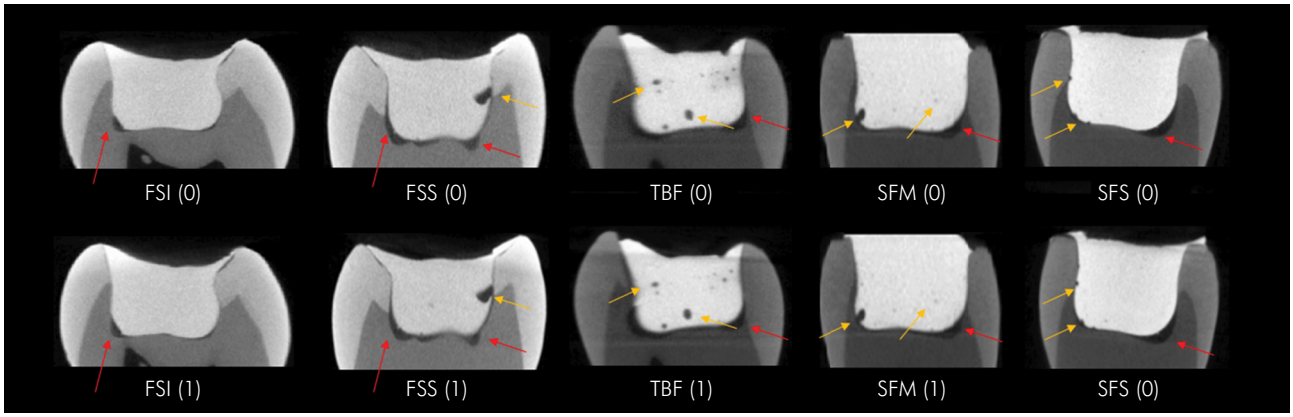

Figure 3. Presence of gaps and voids in the resins before (0) and after (1) light curing indicated by arrows in the μCT (red=gap; yellow=void). FSI: Filtek Supreme XTE - incremental insertion; FSS: Filtek Supreme XTE - single insertion; TBF: Tetric Bulk Fill; SFM: SonicFill - manual filling; SFS: SonicFill - sonic filling.

whereas there was a weak positive correlation between the volume of polymerization shrinkage (%VS) and the volume of the final void (%VV1) (Figure 4B, $p = 0.009$, $r = 0.476$).

The linear regression model explained 89% of the variation in the final gap. The initial gap was the main factor related to the final gap ($p < 0.001$). For each 1-mm^3 increase in the initial gap, the final gap increased by 0.95 mm^3, maintaining VV and VS constant.

Discussion

The results of the present study demonstrate that, regardless of the insertion and filling technique, all RCs exhibited polymerization shrinkage, gap, and void. The first null hypothesis was partially rejected because there was a difference between the volume of polymerization shrinkage for FSI and FSS and for SFS and FSS, in addition to a void for FSI and bulk fill RCs; however, no difference was found for gap volume (VG) between bulk fill and conventional RCs. The second null hypothesis was rejected because there was a positive correlation between polymerization shrinkage and gap and void in the studied RCs.

In recent years, μCT has become an important tool in the analysis of polymerization shrinkage,[22,23] gap formation,[24] and void.[16] μCT can produce quantitative analyses of polymerization shrinkage when compared with conventional methods, which are qualitative or semiquantitative.[27] Also, this type of analysis does not cause stress, deterioration, cracking, or destruction

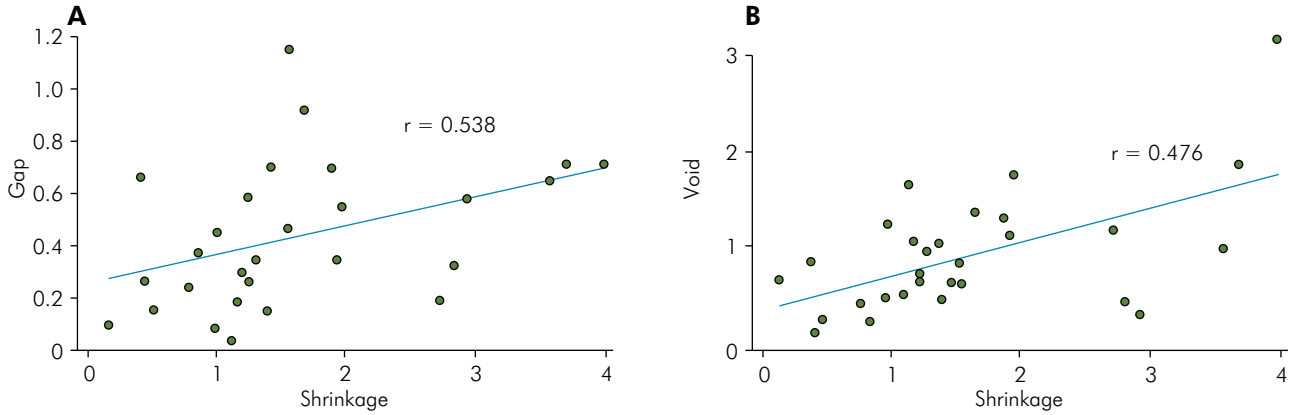

Figure 4. A – Correlation between volume of polymerization shrinkage and final gap in the pulp wall at the resin/tooth interface ($p = 0.003$). B – Correlation between volume of polymerization shrinkage and final void ($p = 0.009$).

of the sample, thus allowing for the sample to be analyzed in 3D before and after treatment without interfering in the experiment, unlike SEM[19] and other 2D methods.

The volume of polymerization shrinkage of the materials investigated in this study ranged from 1.01 to 2.91%, which is an acceptable value, according to the literature.[4] The low shrinkage values observed in the analyzed RCs may be explained by the increase in inorganic load,[28] since it is known that RCs with a lower fill load may have a shrinkage greater than those with a higher load. In addition, all methacrylate-based RCs shrink to some extent, and shrinkage can be reduced by using monomers with a high molecular weight.[29]

Higher polymerization shrinkage was observed in FSS, and this can be explained by the application of single-increment RC (4 mm) instead of the incremental filling recommended by the manufacturer (Table 1). Shrinkage in the incremental technique was similar to that of bulk fill RCs, but the incremental filling technique increases the deformation of the restored tooth, which could be a negative outcome due to higher stress on the tooth-composite structure.[7] However, in this study, μCT did not allow us to measure polymerization shrinkage stress.

Low shrinkage values were observed for SFS, and this could perhaps be explained by the modification of the charging behavior of the particles, which possibly minimizes the stress generated by polymerization shrinkage, and also by the fact that methacrylate-based resins, such as SonicFill, decrease shrinkage, to some extent, with the use of high molecular weight monomers, since the nature of the monomer determines the amount of bulk shrinkage during polymerization and the resulting stress.[29] SonicFill was not better than the manual insertion of this same resin composite or than Tetric Bulk Fill for the shrinkage volume, nor did it differ in the FSI. Benetti et al.[30] also did not observe any difference in polymerization shrinkage between SFS and TBF, but class II cavities were analyzed through a linear variable differential transformer (LVDT). On the other hand, Orlowski et al.[31] observed better results for SFS than for TBF; however, their work verified the marginal sealing of cavities and not polymerization shrinkage.

All cavities presented a gap at the interface between the RC and the pulp wall (Figure 3), and the final gap volume ranged from 1.97 to 2.95% between the groups. In this study, the sonic filling technique (SFS) did not influence the lower gap formation, because the manually inserted bulk fill RCs (SFM and TBF) did not differ statistically from the sonically inserted ones. In addition, Benetti et al.,[30] when using an LVDT and an electron microscope, did not observe any difference in polymerization shrinkage, gap formation, and polymerization depth between SFS and TBF in class II cavities. However, Kapoor et al.[10] found better adaptability and lower gap formation in the pulp wall when bulk fill RCs were used compared with conventional RCs, but the analysis was made by SEM, which may raise some doubts about final gap formation.[19]

Gap formation is a complex phenomenon and depends on the interaction of several factors.[30] Polymerization shrinkage is not the only factor involved in gap formation around the cavity edges, as some other factors, such as cuspal deflection,[32] type of cavity,[6,30] and insertion technique,[10] may also generate a gap. However, observing the presence of a gap in SEM or through other destructive tests creates uncertainty about whether the gap formed before and/or after light curing, because the preparation of the sample can cause stress and deterioration at the tooth/restoration interface, casting doubt about whether the gap occurred before or after light curing or if the failure was caused by the insertion technique or if different areas, mainly near the angles, were not properly selected in the cuts. Therefore, the analysis in μCT has become an accurate, safe, and non-destructive method for evaluating these materials in 3D.[19,24]

Both bulk fill and conventional RCs presented voids in the body of the material (Table 2), but a smaller void volume was found for conventional RCs. There was no difference in void volume among bulk fill composites. According to the literature, the handling of RCs by the operator should be minimized to prevent the formation of air bubbles.[18] Restoring cavities, especially deep ones, with 2-mm-thick increments is time-consuming and implies the risk of entrapment of air bubbles during the incremental technique.[33] However, in the present study, the

insertion/condensation of conventional RCs by the incremental technique eventually reduced the number of voids in the body of the material when compared with the sonic and manual technique of bulk fill RCs; one hypothesis is that spatulation could reduce the number of bubbles or other defects present in the matrix during the manufacture of the material.

The results of the present study indicate a moderate positive correlation between polymerization shrinkage and gap formation. In the literature,[30] there is a strong positive correlation; however, polymerization shrinkage was analyzed through LVDT and gap formation was evaluated with a visible scale in the microscope objective. This correlation, as found in the present study and in the literature, demonstrates that there is an association between polymerization shrinkage and the final gap, but other factors should be considered as well. The small difference between the final and initial gaps is proportional to the observed polymerization shrinkage. However, the presence of the final gap in the restoration seems to be associated more with insertion of the material in the cavity (initial gap) than with polymerization shrinkage. This fact had not been previously described in the literature, and the linear regression model performed in the present study explained 89% of the final gap variation, where the initial gap was the main associated factor. For each 1-mm³ increase in the initial gap, the final gap increased by 0.95 mm³, maintaining the void volume and polymerization shrinkage constant.

The correlation between void volume and polymerization shrinkage was weak (Figure 4B). The regression analysis showed that 94.5% of the void volume increased, that is, the final void volume was due to polymerization shrinkage. A possible explanation for these findings is that molecular rearrangement of monomers in a smaller space[34] and the internally generated polymerization shrinkage forces[35] would increase the gap/void around this region. However, further *in vitro* studies should be conducted to assess polymerization shrinkage, gap formation, and void in the material, taking into account factors associated with polymerization shrinkage stress in bulk fill RCs.

Conclusions

The sonic and manual insertion of bulk fill RCs in large cavities shows polymerization shrinkage and gap formation similar to those observed in conventional nanoparticle-filled RCs subjected to the incremental technique. There are moderate and weak positive correlations between polymerization shrinkage and gap formation and between polymerization shrinkage and voids, respectively, and final gap formation is more dependent on the initial gap than on polymerization shrinkage or on void volume.

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