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Volumetric polymerization shrinkage and its comparison to internal adaptation in bulk fill and conventional composites: A μCT and OCT *in vitro* analysis

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ABSTRACT

Objective. To quantify the volumetric polymerization shrinkage (VPS) of different conventional and bulk fill resin composites, through micro-computed tomography (μCT), and qualitative comparison of gap formation through optical coherence tomography (OCT).

Methods. Box-shaped class I cavities were prepared in 30 third-molars and divided into 5 groups (n = 6): G1- Filtek Z100 (Z100); G2- Tetric Evoceram Bulk Fill (TEC); G3- Tetric EvoFlow Bulk fill (TEF); G4- Filtek Bulk fill (FBU); and G5- Filtek Bulk fill Flowable (FBF). All groups were treated with Adper Single Bond Plus adhesive and light cured (Bluephase 20i). Each tooth was scanned three times using a μCT apparatus: after cavity preparation (empty scan); after cavity filling (uncured scan) and after light curing of the restorations (cured scan). The μCT images were imported into a three-dimensional rendering software, and volumetric polymerization shrinkage percentage was calculated (%) for each sample. In the same images, interfacial gaps in the pulpal floor were qualitatively evaluated. After μCT evaluation, the pulpal floor from each tooth was polished until a thin tooth structure was obtained and OCT images were obtained by scanning the pulpal portion. Gap formation was observed and qualitatively compared to the μCT images.

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Results. VPS means ranged from 2.31 to 3.96% for the studied resin composites. The bulk fill materials, either high viscosity or flowable, were not statistically different from each other ($p > 0.05$). The conventional resin composite Z100 presented statistically higher VPS than both high viscosity bulk fill materials studied ($p < 0.05$), although it was statistically similar to the flowable bulk fill materials studied ($p > 0.05$). Both μCT and OCT methodologies enabled gap formation visualization, and images from both technologies could be associated. Gap formation was mostly observed for G1-Z100, G4-FBU, and G5-FBF. VPS% and pulpal gap formation could not be completely associated with each other for all groups and samples. Voids were observed in most of the resin composite fillings, and most VPS were observed in the occlusal area of the samples.

Significance. Volumetric polymerization shrinkage was material-dependent, although bulk fill materials did not differ from each other. Both μCT and OCT enabled interfacial pulpal gap formation visualization. VPS and gap formation cannot be completely associated with one another.

1. Introduction

Resin-based dental composite composition and technology has greatly improved to meet aesthetic and functional demands since it was first introduced more than 60 years ago, and is considered a reliable restorative material in dentistry [1]. Advances in resin composites have promoted the development of bulk fill resin composites, which according to manufacturers can be used in a single increment of 4–5 mm depth, due to modifications in their organic matrix, initiator, and filler content [2,3]. Such composites are available in two types: flowable and high viscosity, and many studies have compared both of them [4–8].

Despite their dramatic improvements, volumetric polymerization shrinkage (VPS) and interfacial gap formation are still an issue when it comes to the longevity of resin composite restorations [9,10]. VPS is an unavoidable characteristic generated during polymerization due to distance reduction between monomer molecules as a result of short covalent bonds, reducing the overall free volume within the monomer structure and producing densely packed polymer molecules [11]. Depending on the magnitude of the VPS, contraction stresses can be generated, which can affect the adhesive interface between the tooth and restoration [7,12]. When the stress generated exceeds the strength of the bond between the adhesive and the tooth, interfacial gaps can be formed [13,14]. It has previously been proven that polymerization shrinkage also plays a role in stress development and consequent gap formation around cavity margins [15]. Furthermore, gap formation and volumetric polymerization shrinkage that leads to an interruption of the restoration margin, associated with patient factors such as cariogenic biofilm might result in treatment failure in some cases [16]. Thus, their detection and evaluation are important both from clinical and *in vitro* research points of view [17].

Incremental techniques have been suggested to compensate for the polymerization shrinkage of composites by reducing the stresses developed within the tooth-restoration system [18,19]. However, their disadvantages regard the possibility of trapping voids between layers and the time required to place the final restoration [20]. Bulk fill composites simplify the restorative procedure by reducing the number of increments and thus the final curing time [3]. It has been shown that flowable bulk fill composites present comparable or lower volumetric polymerization shrinkage when compared to a conventional flowable resin composite [6], which was also observed for high viscosity bulk materials [4].

Evaluation of gap formation may be performed by conventional methods, such as microscopic assessment [21]. This method requires multiple sectioning of the samples, surface polishing and immersion into a staining solution [22]. In an attempt to elucidate problems related to destructive analyses, more sophisticated and nondestructive methods, that are also suitable to volumetric polymerization shrinkage assessments, are being studied nowadays facilitating the characterization of resin composites. [4–7,9,12,17,23–36].

Optical Coherence Tomography (OCT) is a nondestructive and noninvasive technique that requires neither specimen preparation and coating nor vacuum conditions [17]. OCT applies near-infrared light for cross-sectional scanning to produce 2D and 3D tomograms [28], and evaluation of marginal adaptation, voids and gap formation of different adhesive and composite resin systems has been studied with this method [7,8,27,28,30–33,37]. The OCT image is reconstructed based on the reflection of the coherent light beam from the structure [32]. When the light of the apparatus passes through two materials with different refractive indexes, a portion of the light is reflected, and if a gap is present, which means that there is air or water at the interface, the difference of the refractive indices can be detected [32]. Specimens with poor marginal sealing show images with bright clusters at the cavity wall, indicating interfacial gaps, while tight margins do not show such increased scattering [38].

Micro-Computed Tomography (μCT) obtains three-dimensional (3D) structures images of small objects with a high level of spatial resolution in a non-destructive matter [5,6]. The use of μCT allows non-destructive 2D and 3D imaging, and the possibility to analyze the material behavior inside a given geometric configuration, such as a tooth cavity [5,6,39]. This methodology has demonstrated its efficacy in the assessment and visualization of polymerization shrinkage vectors [23,40], volumetric shrinkage changes within a restoration and cementation [4–6,24,41], correlation between the polymerization shrinkage strain/stress and the internal adaptation generated by tooth stress [26,39], and correlation between digital and analogical natural tooth morphology measurements, showing a concordance correlation between methods of 97% [42], within others.

In both described methodologies, the specimens remain intact and can be used several times without damage to the structures. Several studies have pointed out the importance of OCT in detecting gaps in resin composite restorations [7,8,17,30–33,37], as well as the μCT on showing volumetric polymerization shrinkage and gap formation [4–6,41]. However, few studies had shown a comparison between volumetric polymerization shrinkage and interfacial gaps through μCT and OCT [32]. Thus, the aim of this study is to quantify the volumetric polymerization shrinkage of different conventional and bulk fill resin composites, through μCT, and qualitatively associate these results with interfacial pulpal gap formation through μCT and OCT. The working hypotheses tested were that: (1) different composites would result in different VPS and gap formation; (2) VPS and gap formation findings could be associated; and (3) both μCT and OCT methodologies would enable the qualitative observation of the gap formation.

2. Materials and methods

Thirty freshly extracted sound human third molars were obtained according to protocols approved by the University Ethical Committee. The sample size was determined according to previous studies, which used the same methodology [3–5]. Teeth had their roots cut and their cusps flattened until a regular flat surface was reached, using MetaServ® 250 twin grinder-polisher (Buehler, Lake Bluff, IL, USA), and box-shaped class I cavities (4 mm length, 4 mm wide, and 2.5 mm depth for Z100 group or 4 mm depth for the other groups) were prepared using a diamond bur (AD20 Occlusal Reduction Bur, Code 845-022, Strauss & Co., Palm Coast, FL, USA). The bur was replaced after every five cavity preparations, and the final cavity preparation was then checked with a digital caliper. All teeth were maintained in distilled water at room temperature (25 °C) before and after preparation procedures. After that, teeth were cleaned with a pumice slurry and divided into 5 different groups: G1- Filtek Z100 (Z100 - 3M Oral Care, St. Paul, MN, USA); G2- Tetric Evoceram Bulk Fill (TEC - Ivoclar Vivadent, Schaan, Liechtenstein); G3- Tetric EvoFlow Bulk fill (TEF - Ivoclar Vivadent); G4- Filtek Bulk fill (FBU - 3M Oral Care); and G5- Filtek Bulk fill Flowable (FBF - 3M Oral Care). All groups were treated with the Adper Single Bond Plus adhesive (3M Oral Care), according to manufacturer's instructions. A schematic depicting the step-by-step of this study is shown in Fig. 1. All restorative steps were performed by a specialized single operator.

2.1. μCT evaluation

Each tooth underwent three scans, performed with a μCT apparatus (μCT40, Scanco Medical, AG, Basserdorf, Switzerland) by a μCT trained operator. The apparatus was calibrated using a phantom standard at 70 kVp/BH 200 mgHA/cm. The operating condition for the μCT device used was 70 kVp–114 microamperes with a resolution giving 16 μm/slice. The average of the total number of slices was approximately 250, and the average scan time was 28 min. Peak kilovoltage and resolution of the system can greatly affect the acquisition of the images, thus, these parameters were determined following previous studies [4,6].

The first scan was performed after cavity preparation (empty scan). After that, all teeth were etched with phosphoric acid (Ultra-etch 35%, Ultradent Products, South Jordan, UT, USA) for 30 s in enamel and 15 s in dentin, rinsed for 20 s with water and dried with a thin absorbent paper; adhesive system was applied according to manufacturer's instructions, light cured and restorations were performed with their assigned resin composites. All resin composites were applied in bulk, and since Z100 is a material that needs an incremental technique, its depth was reduced to 2.5 mm instead of 4 mm used for other materials, as it was observed a correlated degree of conversion for this material and bulk fill materials [43]. Before light curing the restorations, a second scan was performed (uncured scan), and when it was finished, teeth were removed from the μCT apparatus and individually light cured. After light curing the resin composites, teeth underwent a third scan (cured scan). In order to avoid unwanted curing of the resin composites, the μCT apparatus was covered with dark plastic, avoiding contact with any light source during all the scans. All restorations, as well as the adhesive system, were light-cured with a polywave light-curing unit for 20 s (High power, 1.200 mw/cm^2, Bluephase 20i, Ivoclar Vivadent, Schaan, Liechtenstein).

Finally, μCT data were imported into a workstation and evaluated with Amira software (version 5.5.2, VSG, Burlington, MA, USA). The three scans were superimposed in the software ("superimposition" tool), and both uncured and cured scans were subtracted from the empty scan, isolating the restoration. This procedure was performed to avoid scattering and possible noise between tooth structures and the resin composites due to their similar radiodensity [4,5,40]. After that, resin composite volumes were quantified in both uncured and cured scans, allowing the volumetric polymerization shrinkage (VPS) to be calculated as a percentage. Another subtraction was conducted for the uncured minus cured scans for imaging of resin composite's shrinkage. Using the same software, interfacial gaps in the pulpal floor were also characterized on the 3D images with green opaque color (Fig.1).

Data from VPS analysis were checked for normality using the Shapiro–Wilk test ($p > 0.05$) and after that, analyzed by one-way analysis of variance (ANOVA) at 5% level of significance and Tukey post hoc tests, using IBM SPSS software (IBM, Armonk, NY, USA).

2.2. OCT

After μCT evaluation, optical coherence tomography was performed by a trained OCT operator using an OCT device (OCT930SR, Thorlabs Inc, Newton, NJ, USA); its light source emitted a central wavelength of 930 nm and 2 mW of optical power. In order to allow the visualization of the interfacial gaps in the pulpal floor, considering OCT's limited image depth [14],

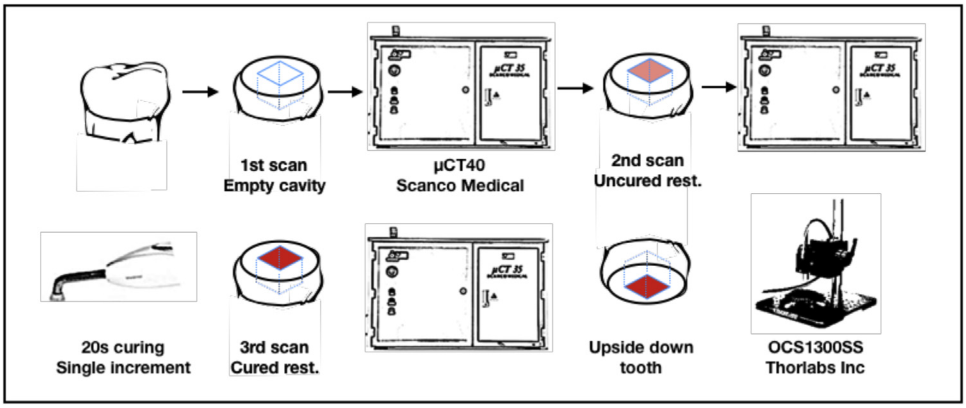

Fig. 1 – Schematic depicting step-by-step of this study: cavity preparation, followed by the first µCT scan; cavity was filled with resin composite, second scan was taken; restoration was light cured for 20 s, third scan was performed; restoration/tooth system was turned upside down for gaps observation in the OCT. Images acquired in the µCT scans were observed in a software (Amira 5.5.2). (For interpretation of the references to colour in the text, the reader is referred to the web version of this article.)

in this case about 0.7 mm (with an axial resolution of 0.4 µm and lateral resolution of 6.0 µm), teeth were prepared before the scan. The pulpal floor from each tooth was polished until a thin tooth structure was obtained, without reaching the cavity and the resin composite. Then, teeth were positioned on the worktable with the pulpal floor facing up, while the occlusal portion was positioned facing bottom, as observed in Fig. 1. OCT scanning was performed along all the pulpal surface of the polished tooth looking for interfacial gaps; movements were standardized (from top to bottom, and from left to right), double-checking. Cross-sectional images of 2000 × 512 pixels (corresponding to 2.5 × 0.7 mm) were obtained using SR Scan program (Thorlabs Inc, Newton, NJ, USA), with parameters of 2000 A-Scan (Columns) for a width of 2.5 mm (Range), by analyzing the interference pattern based on the low coherence light scattered from the sample [44]. Gap interfaces were observed by changes in the signal intensity (high scattering area) at the interface of resin and dentin, which appeared as brighter pixels since when light traverses the interface through two different media, it undergoes refraction as well as partial reflection [37]. This procedure was performed without cutting or destroying the samples.

Representative images selected by the operator (interface pulpal gap presence: image with the highest scattering area, interface pulpal gap absence: image with non-high scattering area) were saved and compared with the 3D reconstruction image of the µCT, in order to compare pulpal interface gap detection and proportions.

3. Results

Table 1 summarizes the means and standard deviation of VPS from the different groups. A significant statistical difference was found among groups, where VPS means ranged from 2.31 to 3.96%. The bulk fill materials, either high viscosity or flowable, were not statistically different from each other ($p > 0.05$); G2-TEC was similar to G3-TEF ($p = 0.827$), G4-FBU ($p = 1.000$) and G5-FBF ($p = 0.063$); G3-TEF was similar to G4-FBU ($p = 0.912$) and G5-FBF ($p = 0.412$); and G4-FBU was also similar to G5-FBF ($p = 0.097$). Regarding the conventional resin composite Z100, it presented statistically higher VPS than both high viscosity bulk fill materials studied (TEC: $p = 0.005$; FBU: $p = 0.008$), although it was statistically similar to the flowable bulk fill materials studied (TEF: $p = 0.056$; FBF: $p = 0.797$). Qualitative 3D reconstructions depicted voids in most of the resin composite fillings, and most of the volumetric polymerization shrinkage was observed at the top of the samples (occlusal), and part on the pulpal floor, observed as a green opaque line (Figs. 2 and 3).

Table 1 – Means (% ± SE) of volumetric shrinkage determined for each resin composite material, observed through µCT.

	Volumetric shrinkage (%)
G1 – Filtek Z100 (Z100)	3.96 (0.30) A
G2 – Tetric EvoCeram Bulk fill (TEC)	2.31 (0.14) B
G3 – Tetric EvoFlow Bulk fill (TEF)	2.75 (0.32) AB
G4 – Filtek Bulk fill (FBU)	2.40 (0.28) B
G5 – Filtek Bulk fill Flowable (FBF)	3.50 (0.37) AB

Fig. 2 shows representative images of the equivalence within µCT and OCT images. Qualitative comparisons of µCT and OCT images depicted a correspondence within the gaps or lack of gaps observed in the pulpal floor of the restorations. Interfacial gaps were observed as bright spots in OCT images, while when a detachment of the resin composite from the tooth is observed, gaps are demonstrated as two bright lines with a black line in the middle. The lack of gaps is demonstrated as a continuous line with no bright spots. In µCT images, gaps are observed as an opaque green line. Qualitatively, for all µCT and OCT images, the highest amount of interfacial gaps in the pulpal floor were observed for G1-Z100, G4-FBU, and G5- FBF.

4. Discussion

This study evaluated the volumetric polymerization shrinkage of different conventional and bulk fill resin composites,

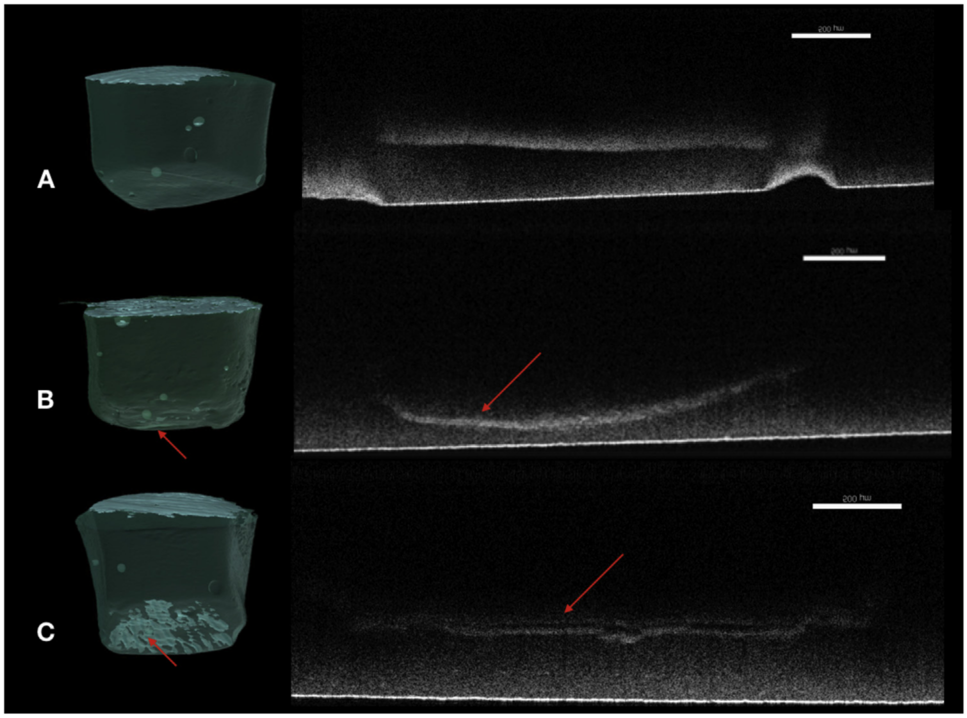

Fig. 2 – Representative images showing correlation within images obtained by μCT (left column) and OCT (right column). A: image from a gap-free sample in the pulpal floor, note the lack of gaps and volumetric shrinkage in this region in the μCT image, correlating with an absence of bright spots in the OCT image. B: image from a sample with few gaps in the pulpal floor, opaque green areas in that region demonstrates gaps in the μCT image, correlating with the bright spots in the OCT images. C: image from a sample with a great area of gap in the pulpal floor, observed in the μCT image, correlating with the OCT image showing detachment of the resin composite from the tooth (two bright lines with a black line in the middle). VPS and gap formation are showed as red arrows. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

through μCT; the correspondences of μCT and OCT images regarding interfacial gap formation observation; and the correspondences within VPS and gap formation. The first hypothesis was accepted, since different composites showed different VPS and gap formation. Volumetric polymerization shrinkage ranged from 2.31 to 3.96%, which is in accordance with previous studies [4–6]. The conventional resin composite studied showed no significant difference when compared to the flowable bulk fill composites, although it showed higher VPS than the high viscosity bulk fill composites. Bulk fill composites had shown lower volumetric shrinkage than conventional composites when the same type of consistency is compared [4,41]. However, when different consistencies are compared a composite with lower filler content, or flowable, could result in higher volumetric shrinkage than the ones with high viscosity, since it commonly presents increased monomer content, which may lead to increased VPS [45], which in turn could explain why the flowable bulk fill composites were similar to the conventional resin composite and the high viscosity bulk fill was different. Since shrinkage occurs in the course of monomer conversion to polymer, the lesser the filler content, the higher the resultant shrinkage will likely result [46].

Mostly, interfacial gaps and shrinkage were observed in the occlusal surface, and less frequently observed on the mesiodistal and buccolingual walls of restorations, agreeing with results from previous studies, that related higher shrinkage occurring in unbonded free surfaces [4–6]. Moreover, voids were presented in most of the restorations, also agreeing with previous studies [4,6,8,20].

The second hypothesis had to be rejected, since VPS and interfacial gap formation did not correspond completely. Qualitatively, the groups that showed a greater number of interfacial gaps were G1- Z100, G4- FBU and G5-FBF, demonstrated as bright spots or a line within bright spots in the OCT images, and opaque lines in the μCT images. This result was expected for the conventional resin composite, as it does not have the modern formulation of the bulk fill composites, which, in order to be cured in 4–5 mm deep, present stress relievers and polymerization modulators in their composition [47,48]. For both FBU and FBF, a modified urethane dimethacrylate was introduced in their chemistry (AUDMA – aromatic urethane dimethacrylate), which presents a high molecular weight. According to the manufacturer, by adjusting the proportions of high molecular weight monomers, those RCs could produce low polymerization shrinkage with a low modulus development, resulting in low stress [49]. However, in this study, although manufacturers claim that reduced stress is expected, such bulk fill composites presented a greater number of interfacial gaps than the other bulk fill materials, which

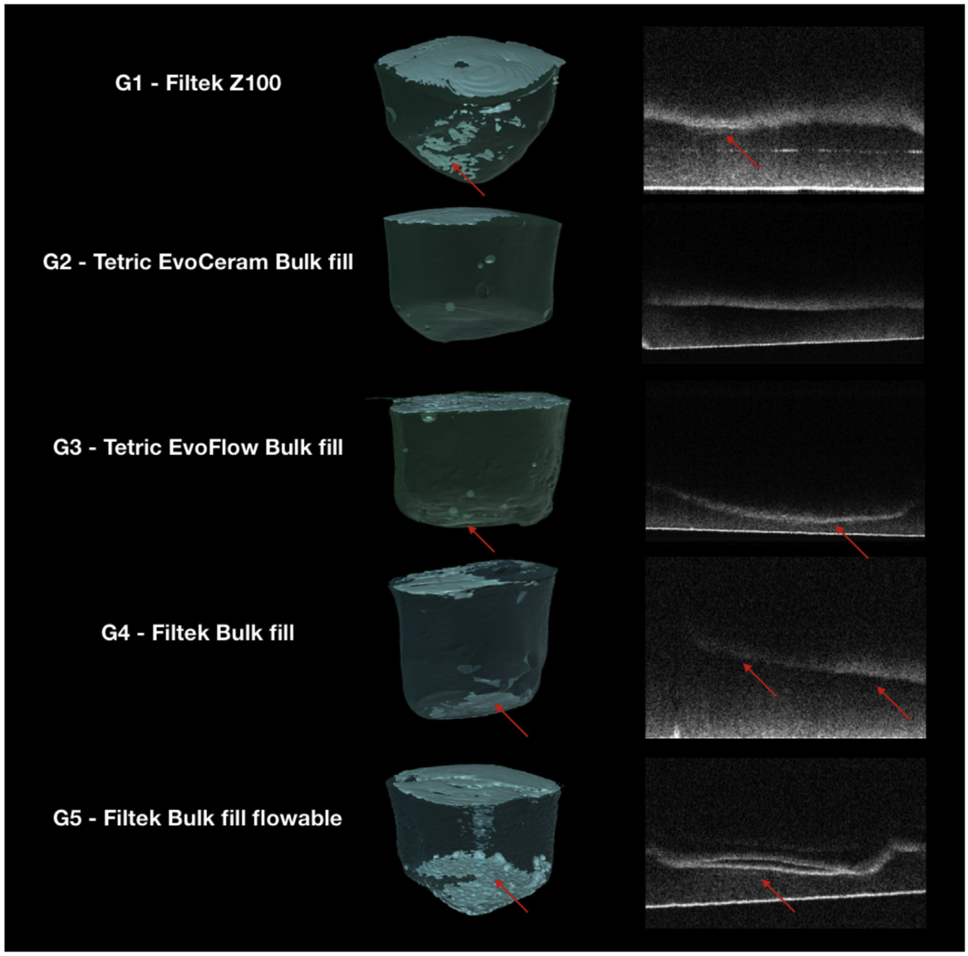

Fig. 3 – Images representing μCT 3D renderings and OCT images of groups. Transparent green areas represent the mass of the composite, while opaque green areas represent areas where volumetric polymerization shrinkage occurred, leading to gap formation in some samples. Note that most of the volumetric polymerization shrinkage is located mainly on top of the restorations, and not always the groups with higher VPS showed greater gap formation. VPS and gap formation are showed as red arrows. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

goes in accordance with previous studies [49,50]. In this study, TEC and TEF demonstrated qualitatively reduced amounts of interfacial gap formation in both μCT and OCT analysis, agreeing with the results of recent studies [49,50]. According to the manufacturer, these resin composites present a silanized filler that acts as a shrinkage stress reliever and provides a lower elastic modulus, reducing stress, and thus, interfacial gap [49].

It has been observed that, although low volumetric shrinkage does not necessarily correspond to a low polymerization stress development [51], high polymerization stress values are correlated with gap formation along the bonded tooth interface [50]. Moreover, it has been observed that reduced volumetric shrinkage does not guarantee reduced interfacial gap formation [15], thus agreeing with our results. It is important to mention that this study used class I cavities, and the C-factor can have a strong effect on internal adaptation [31]. The results of this in vitro study give useful background to the understanding of polimerization shrinkage and internal adaptation of resin composites, however, it is important to notice that the clinical significance of the gaps presence remais controvertial, as the correlation between marginal quality, clinical outcome, and postoperative sensitivity is hard to measure. Several studies relate presence of gaps in the interface with postoperative sensitivity [10] and secondary caries development [10,52,53], while some other studies state that it is more determinated by the oral enviroment [54]. To the best of our knowledge, there is still a lack of evidence regarding a sistematic correlation betweeen marginal adaptation and its clinical outcome. Thus, further clinical investigations are needed [55].

Both methodologies studied enabled the observation of presence of interfacial gap formation, thus accepting the third hypothesis. Such results agree with a previous study that showed that such technologies can be useful as non-destructive methods for evaluation of internal adaptation [32]. When compared, μCT and OCT present advantages and disadvantages. For instance, OCT permits instant non-invasive imaging with no radiation hazards [37], while μCT takes minutes to capture the images [6]. On the other hand, μCT enables the 3D observation of entire structures [6], while OCT presents a limited depth of visualization [14], A drawback of this study relates to the fact that it is nearly impossible to correlate both μCT and OCT images at the exact same location, thus, one cannot be 100% sure that what is being observed in one system is exactly the same image as in the other system. Improvements should be made in order to further correlate the exact same image from both methodologies.

5. Conclusion

Based on the results obtained by this study, the following conclusions can be made:

1 The high viscosity bulk fill resin composites used in this study showed decreased volumetric polymerization shrinkage when compared to a high viscosity conventional resin composite.
2 The flowable bulk fill resin composites used in this study showed similar volumetric polymerization shrinkage when compared to a high viscosity conventional resin composite.
3 The bulk fill resin composites studied do not show statistical differences from each other, being it high or low viscosities, regarding VPS.
4 Volumetric polymerization shrinkage and interfacial gap formation do not completely correspond between one another after qualitative evaluation.
5 The μCT and the OCT techniques used in this study showed to enable the detection of gaps in the pulpal floor of class I restorations with correspondent results, thus, both techniques can be used for such evaluation.

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