MICRO-COMPUTED TOMOGRAPHIC ASSESSMENT OF THE INFLUENCE OF LIGHT-CURING MODES ON INTERNAL VOID FORMATION IN BULK-FILL COMPOSITES

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ABSTRACT

Introduction: Polymerization reactions in a new generation bulk-fill composites carried out in a short time with high irradiation, raise concerns about curing processes. With micro-computed tomographic evaluation, it is possible to investigate polymerization shrinkage, and subsequent gap and void formation in dental materials.

Objectives: The aim of this study was to evaluate the void formation in bulk-fill composites light-cured with different modes using micro-computed tomography.

Material and methods: Class I preparations were made in 25 molars that were randomly divided into sub-groups, according to resin composite and curing mode used: Tetric EvoCeram (TEC)*high power mode, TEC*turbo mode, Tetric PowerFill (TPF)*high power mode, TPF*turbo mode, and TPF*3s mode. Each tooth was scanned at two time intervals: pre- and post-cure.

Results: After light-curing, a significant increase in the total volume of internal void was noted for both composites cured with high power mode compared with pre-cure. The difference between the sub-groups at post-cure was also significant. While TEC exhibited similar values in terms of different curing modes, turbo and 3s modes caused a significant difference in TPF group, and the lowest void percentage was detected in 3s mode.

Conclusions: Internal void formation results from an interplay of different factors, including composition of materials and curing modes.

Key words: bulk-fill resin composite, light-curing mode, micro-computed tomography, void formation.

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INTRODUCTION

Resin composite formulations, with the help of technological innovations, have considerably advanced since their introduction into dentistry field, resulting in restorative materials that meet functional and aesthetic requirements of patients [1]. Despite the developments in physico-mechanical characteristics, volumetric polymerization shrinkage (VPS) and deriving shrinkage stresses at the cavity walls upon polymerization, are inherent limitations of dental resin composites [2]. VPS is caused by the conversion of monomer molecules into the polymer structure accomplished by the reduction of 0.3 to 0.4 nm long van der Waals spaces into 0.15 nm long covalent bonds [3, 4]. As usually outlined in the content of polymerization-related investigations, shrinkage stresses at the tooth-restoration interface may lead to unfavorable clinical outcomes, such as marginal/

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internal gap formation, micro-leakage, discoloration, post-operative sensitivity, secondary caries, pulpal changes, and ultimately restoration loss [5, 6]. To overcome the afore-mentioned problems related to VPS and accompanying shrinkage stresses, incremental layering techniques have been introduced, where small amounts of resin composites are applied and each increment is photo-activated individually [7]. However, an incremental technique may be substantial in terms of limitation of polymerization stress and ensuring sufficient light penetration, its drawbacks are void formation between layers and extended working time, which can also increase the risk of contamination during a restoration process [8].

In recent years, following the demand for simplified restorative practices, bulk-fill composites with novel monomer content and improved curing properties, which can be placed and light-cured up to 4 or 5 mm without layering, have gained popularity among dental practitioners [7, 9]. Various prominent characteristics of bulk-fill composites include short curing time owing to new photo-initiators, and enhancement of translucency based on diminished filler amounts and raised filler size [10]. Moreover, bulk-fill composites are reported to cause lower polymerization shrinkage stresses through the incorporation of stress-relievers, with a reduced risk of gap formation at the restoration/ tooth interface compared with conventional counterparts [3, 10].

Light-curing units (LCUs) and related variables play a fundamental role in the qualification of restoration [2, 4]. In a systematic review and meta-analysis study evaluating the polymerization shrinkage stress of resin-based dental materials in terms of photo-activation protocols, the strategies that can be applied to reduce polymerization shrinkage stresses are as follows: 1. Modification of light intensity or total energy applied to the material; 2. Use of alternative polymerization sources; and 3. Use of alternative photo-activation modes [11]. There is a widespread agreement that activation with higher irradiances in a short interval can decrease the pre-gel phase, so that the restorative material flows less, leading to internal and interfacial stresses [12]. To the best of our knowledge, there is no study in the literature evaluating the effect of polymerization modes that is an effective factor in the pre-gel phase on the void formation in restorative materials. It is worth mentioning that given the significance of their role during the polymerization process, dentists should comprehensively question the characteristics and technical details of LCUs in order to not jeopardize patients' health and the success of restoration.

Micro-computed tomography (μ-CT) with its unprecedented level of detail in three dimensions, has gained wide acceptance in dental science and technology [13]. This method utilizes differences in X-ray attenuation to form visual image of a specimen, determined by varied tones of gray and luminosity levels [14]. Due to non-destructive nature of μ-CT technique and ability to precisely analyze a specimen's structure at macro- and micro-scale, regardless of the shape or size of sample, it stands out among several in vitro test methods introduced for the assessment of polymerization shrinkage, and subsequent gap and void formation in dental materials [15, 16].

OBJECTIVES

This research attempted to characterize the void formation in two bulk-fill composites light-cured with different polymerization modes in standardized class I cavity configuration using μ-CT. The null hypothesis was that polymerization modes did not impact the void formation in tested composites.

MATERIALS AND METHODS

SAMPLE SIZE CALCULATION

According to Hirata *et al.* study [17] and using one-way analysis of variance (ANOVA), G*Power software (version 3.1, Heinrich-Heine-Universität Düsseldorf, Düsseldorf, Germany), with $\alpha = 0.05$, power $(1 - \beta) = 0.95$, effect size = 0.984, and common standard deviation of 1.967, the total sample size required for void volume analysis was 25. Therefore, the experiment was conducted with $n = 5$ for each composite*polymerization mode sub-group.

TOOTH SELECTION AND CAVITY PREPARATION

Twenty-five caries-free human molars extracted due to periodontal or prosthetic reasons were used in this study. Teeth were cleaned from hard and soft tissue deposits and stored in a saline solution with 0.5% thymol at 4°C until further use. Standard class I cavities (4.0 mm depth × 5.0 mm mesiodistal length × 4.0 mm buccolingual width) were prepared with round and fissure diamond dental burs (G&Z Instrumente GmbH, Lustenau, Austria) attached to high-speed air-turbine under water-cooling. During cavity preparation, dimensions were frequently verified with a periodontal probe. Dental burs were replaced every five cavity preparations. Teeth were randomly assigned for each combination of the variables (composite*polymerization mode), resulting in 5 sub-groups and a total of 25 specimens, as presented in Figure 1. Two paste-like bulk-fill resin composites, including Tetric EvoCeram Bulk Fill (TEC) and Tetric PowerFill (TPF) as well as different modes of a LED LCU (Bluephase PowerCure, Ivoclar Vivadent) fourth-generation technology, were utilized in this study (Table 1).

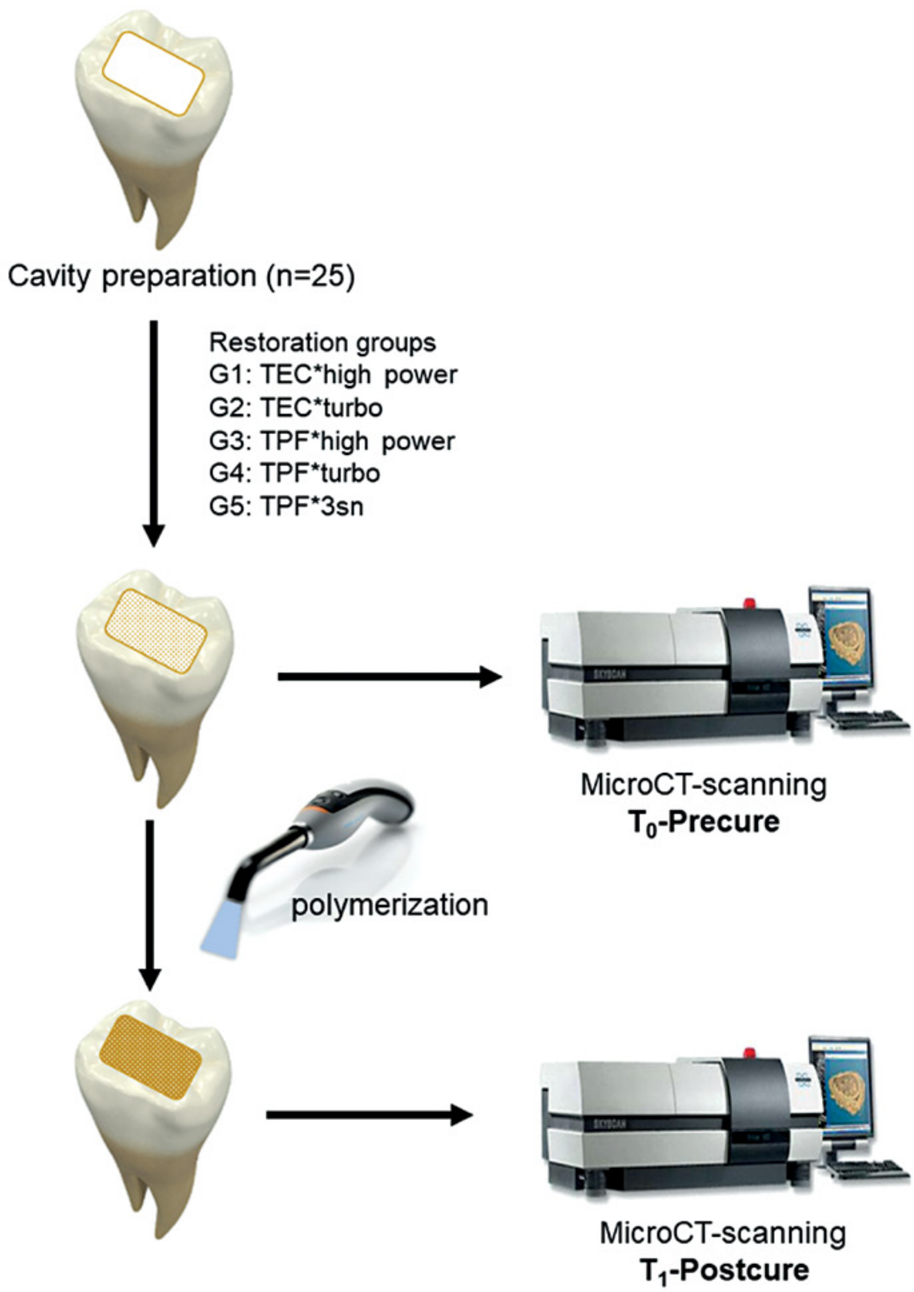


FIGURE 1. Schematic illustration of the study design

RESTORATIVES PROCEDURES

A universal adhesive system (Adhese Universal, Ivoclar Vivadent) was applied in self-etch mode and cured with a LED LCU (Bluephase PowerCure, Ivoclar Vivadent) for 10 seconds (irradiance of 1,200 mW/cm^2), according to the manufacturer's instructions. After light-curing of adhesive, the cavities were restored using bulk filling technique/manual insertion with the resin composites assigned to their group, which were packaged in syringes. The restored teeth were preserved from light contact in a dark plastic tube and placed in μ-CT. A specialized operator (O.G.Y.) applied all cavity preparations and restorative steps.

μ-CT SCANNING AND ANALYSIS

Teeth were scanned using a high-resolution, desktop μ-CT system (Bruker SkyScan 1172, Kontich, Belgium) at two time intervals: T_0 – pre-cure and T_1 – post-cure. Operating condition for the μ-CT device was as follows: 100 kVp, 100 mA, 0.5 mm Al/Cu filter, 10.2 μm pixel size, and rotation in 0.5 steps. Air calibration of the detector was executed prior to each scan to minimize ring

TABLE 1. Specifications of tested resin composites and light-curing unit

Material	Polymer matrix	Filler (wt.%/ vol.%)		Manufacturer
Tetric® PowerFill (TPF) (Lot No.: X48022)	Bis-GMA, Bis-EMA, UDMA, PBPA, DCP, β-allyl sulfone	Barium glass, ytterbium trifluoride, mixed oxide, copolymer (79 wt.%, –)		Ivoclar Vivadent AG, Schaan, Liechtenstein
Tetric® EvoCeram® Bulk Fill (TEC) (Lot No.: Z0032W)	Bis-GMA, Bis-EMA, UDMA (20-21 wt.%)	Barium glass, ytterbium trifluoride, mixed oxide, copolymer (76-77 wt.%, 53-54 vol.%)		
LED curing unit	Curing program: light intensity	Curing time	Wavelength range	
Bluephase® PowerCure	3 seconds cure mode 3,050 mW/ cm² ± 10%	3 seconds	385-515 nm	
	Turbo mode 2,100 mW/cm² ± 10%	5 seconds		
	High power mode 1,200 mW/cm² ± 10%	10 seconds		

Bis-GMA – bisphenol A-diglycidyl dimethacrylate; Bis-EMA – ethoxylated bisphenol A dimethacrylate; UDMA – urethane dimethacrylate; PBPA – propoxylated bisphenol A dimethacrylate; DCP – tricyclodecane dimethanol dimethacrylate

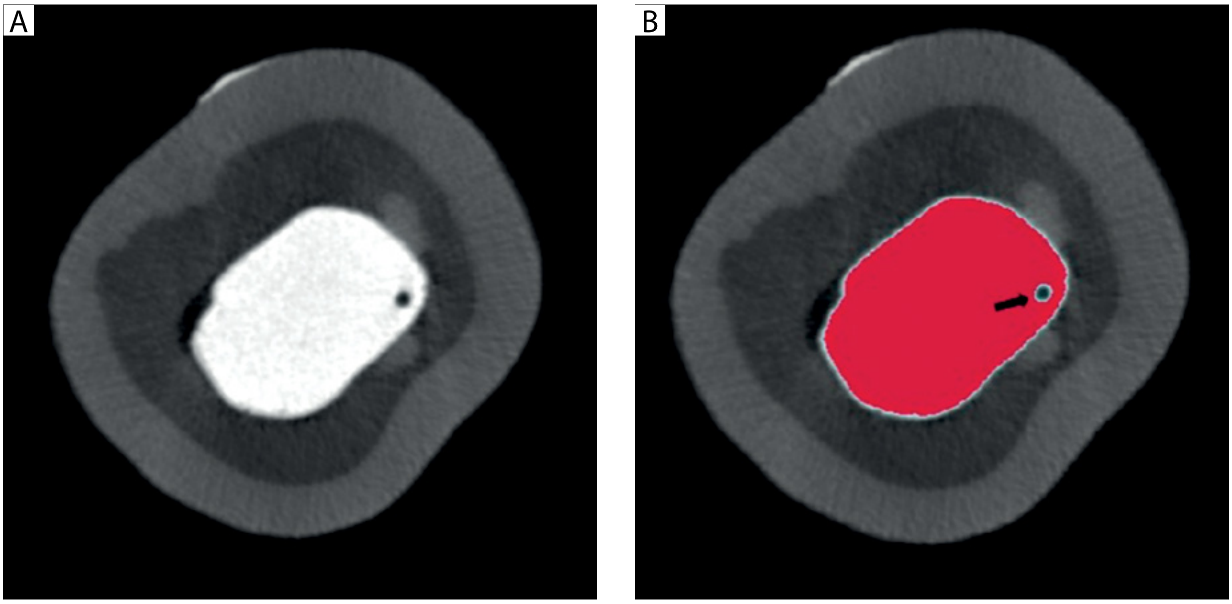


FIGURE 2. A) Resin composite and voids in an axially reconstructed μ-CT image. **B**) ROI determination to calculate internal voids in restoration (red indicates ROI area; arrow shows internal voids)

artifacts. Each sample was rotated 360° in an integration time of 5 minutes, and the average of total number of slices was approximately 600. Cross-sectional images were imported into a 3D visualization software package (NRecon version 1.6.10.4, SkyScan). Ring artifact correction and smoothing for reconstruction parameters were fixed to zero, and beam artifact correction was set to 40%. Contrast limits were applied following SkyScan instructions. CT analyzer software (CTAn version 1.17.7.2, SkyScan) was used for quantitative measurements of specimens. Reconstructed images were also further processed for visualization by CTVox software (version 3.3.0, SkyScan).

After the first scanning, restorations were photo-polymerized with a LED unit (Bluephase PowerCure, Ivoclar Vivadent) using their assigned curing modes (high power and turbo modes for both composites, and 3 seconds cure mode for TPF composite only), according to the manufacturer instructions. Afterwards, teeth were inserted back into the holder for the second scan, ensuring standardization with the same scanning parameters. Then, region of interest (ROI) was selected for each slice in order to determine the void volumes throughout the restoration (Figure 2). By measuring the internal void and restoration volumes in each sample, the percentage of internal void volume in relation to total restoration volume was determined.

STATISTICAL ANALYSIS

Statistical package program (SPSS version 21.0, IBM SPSS; Chicago, IL, USA) was used for data analysis. Shapiro-Wilk test was applied to determine whether the data was normally distributed. Because preliminary analysis of the data revealed heterogeneous variance between groups, one-way ANOVA test was performed followed by post-hoc Tamhane test. Results were considered significant for $p < 0.05$.

RESULTS

The mean total volume of internal void (in percentages) and standard deviation (SD) obtained with μ-CT for all tested composite*curing mode sub-groups are summarized in Table 2. The representative two-dimensional (2D) images of the specimens are visualized in Figure 3. After light-curing, a statistically significant increase in the total volume of internal voids was noted for TPF (p = 0.014) and TEC (p = 0.008) cured with high power mode compared with pre-cure. The difference between the curing mode*composite sub-groups tested at post-cure was also significant ($p < 0.001$). When the composite groups were evaluated within themselves, TEC exhibited similar values in terms of different curing modes. Turbo and 3s modes caused a significant difference in the TPF composite group, and the lowest void percentage was detected in the 3s mode group, followed by turbo mode and high power mode.

DISCUSSION

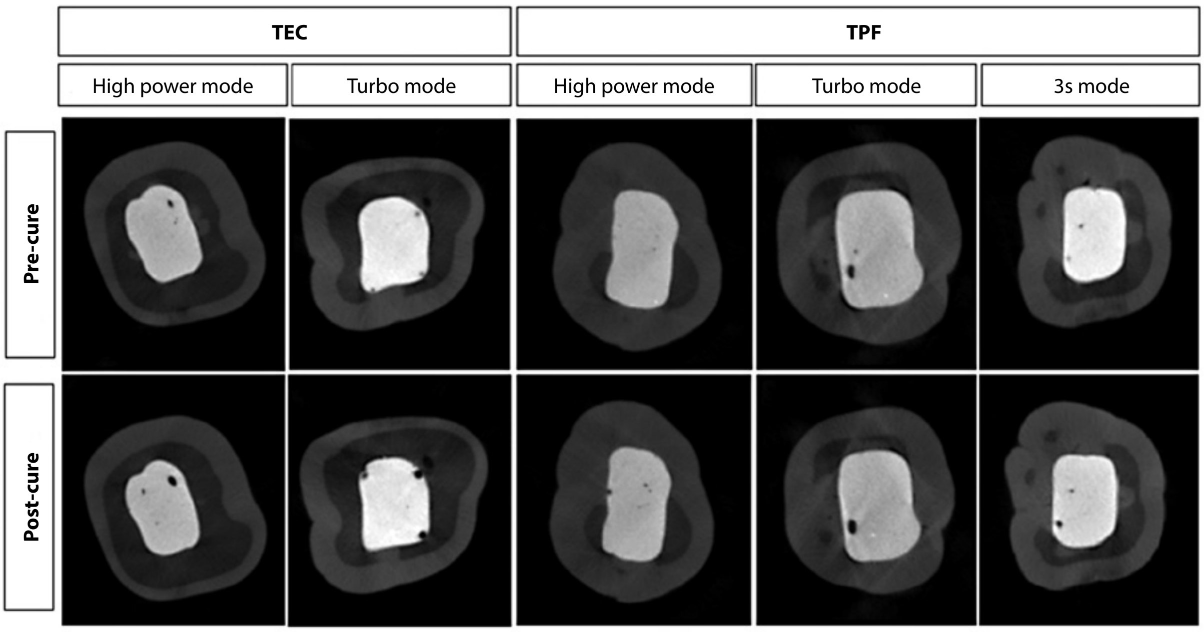
The void formation of tested composites polymerized with different curing modes was evaluated using μ-CT. In order to avoid making a difference within the tested

TABLE 2. Mean (± SD) void volume (%) for each bulk-fill composites and curing modes

Groups	Evaluation time		*p*-value
	T_0 – pre-cure	T_1 – post-cure	
TEC*high power	0.035 ± 0.029[a,b]	0.046 ± 0.025[a,b]	0.008
TEC*turbo	0.032 ± 0.002[a]	0.047 ± 0.010[a]	0.431
TPF*high power	0.000 ± 0.000[b]	0.152 ± 0.104[a,b,c]	0.014
TPF*turbo	0.055 ± 0.023[a,b]	0.112 ± 0.005[c]	0.399
TPF*3s	0.004 ± 0.003[b]	0.018 ± 0.010[b]	0.184
p-value	< 0.001	< 0.001	

n = 5 specimens per experimental group.

Different lowercase letters indicate statistically significant differences in the same column.



TEC – Tetric EvoCeram Bulk Fill, TPF – Tetric PowerFill

FIGURE 3. Representative two-dimensional (2D) images of the specimens in axial section

parameter based on the composite type and properties, conventional resin-based composites were not included in this study as a control group. The outcomes of this study indicated that variable void percentages were detected depending on the curing modes used. Therefore, the null hypothesis was rejected.

Porosity, which can be categorized into two main divisions, such as open and closed pore systems, in accordance with the accessibility, is one of the main parameters that decrease mechanical properties of resin composite restorations, especially under fatigue loading [18]. Additionally, the presence of porosities, such as voids and gaps, may contribute to the increased water absorption capacity, thus discoloration of restoration [19]. Voids within a restoration may be caused by mechanical air entrapment inside the composite material during manufacture as well as packaging or manipulation throughout the restorative procedure [20]. In some investigations, it is specified that during the manufacturing process, filling resin composites into syringes under a vacuum ensures no voids in the material [19, 21]. According to the literature, as intensive manipulation of the composite leads to increased air entrapment through the material [22, 23], the handling of resin composites by the practitioner should be kept to a minimum [24]. Other reasons behind the presence of voids within the restorations could be a consequence of the viscosity of material, insertion technique, and the operator's experience [20]. In an attempt to eliminate discrepancies of void formation caused by the material dispensing and restorative application in the present study, all cavities were filled by a 10-year experienced single operator using bulk technique and two bulk-fill composites with similar consistency.

Apart from the afore-mentioned factors, a previous study using optical coherence tomography to perform a 3D assessment of void and gap formation in flowable resin composites emphasized that re-arrangement of the resin matrix and flow during the pre-gel phase may affect the eventual inclusion of voids [25]. The polymerization reaction of resin composites, a complex phenomenon that includes pre-gel, gel, and post-gel phases, is substantially affected by the curing modes with a variety of light output emission and time protocol, consenting to different radiant exposures [26]. Recently, a dental manufacturer (Ivoclar Vivadent) has launched a 3s PowerCure portfolio comprising high- and low-viscosity bulk-fill composite systems (Tetric PowerFill and Tetric PowerFlow), a universal adhesive (Adhese Universal), and a high irradiance LED LCU (Bluephase PowerCure). It is claimed that adequate polymerization can be achieved in a short time, about 3 seconds, in these bulk-fill composite systems using the LED LCU, with high irradiance specifically designed for them. Theoretically, the topic of polymerization with high irradiance and short/ultra-short time in clinical practices must be approached carefully with potential concerns regarding LCUs' efficacy and safety [27]. Therefore, the aim of this study was to scrutinize the factors related to the light-curing parameters, which could influence the void formation using μ-CT. With this method, internal porosity can be visualized and quantified.

Considering the results of this study, the difference in void formation between bulk-fill composites was remarkable even before the initiation of a polymerization reaction. A relatively higher void formation rate was observed in the structure of TEC groups. This result was consistent with a study by Demirel *et al.* [22], in which the authors pointed out that the tested restorative materials were found at different void levels depending on their chemical, physical, and rheological differences. Tetric PowerFill has a nearly identical composition as its predecessor, Tetric EvoCeram Bulk Fill, with the exception of having a 3s curing setting at 3050 mW/cm^2. The difference between Tetric family members tested in this study can be assumed to originate from β-allyl sulfone in the resin matrix of Tetric PowerFill [28]. This reagent supports the addition-fragmentation chain transfer (AFCT), resulting in a change from the radical chain growth mechanism towards a step-like chain growth system [29]. In the existence of AFCT, the radical chain polymerization terminates, simultaneously creating a sulfonyl radical and a new double bond that result in shorter polymer chains, leading to a delayed gel point and more homogeneous polymerization [30]. In this study, contrary to expectations from the material, TPF composite showed lower void formation only in the 3s mode after polymerization. In a previous study, lesser gap and void formation was demonstrated in a material with a delayed gel point obtained by re-arrangements in the resin matrix structure [25]. On the other hand, considering that the TPF sub-group had the lowest percentage of voids after curing and also a lower void volume before curing, it can be assumed that these low values obtained after polymerization were not related to the mode but rather to the operator's action. Furthermore, contrary to expectations, it was revealed that the voids in the structure of both composite restorations increased with the curing process using high power mode, which was expected to lead to a longer pre-gel phase with lower irradiation. At this point, the lack of information on how curing modes affect rheological properties of resin composites was a limitation of this study.

In a previous paper, the formation of internal voids in bulk-fill composites was investigated in terms of pre-heating and sonic delivery by micro-computed tomography. It was revealed that the tested composites, except for SonicFill, indicated the highest void rates with the sonic delivery method, while the lowest void rates were obtained in other composites (VisCalor Bulk, Filtek One Bulk Fill, Tetric EvoCeram Bulk Fill, and Clearfil Majesty) using pre-heating technique [19]. In a study by Hirata *et al.* [17], it was reported that the sonic insertion technique might augment void formation during resin composite delivery, depending on the brand of restorative material. Another research topic regarding void formation and curing modes was polymerization shrinkage. In a study by Almeida Junior *et al.* [24], in a micro-computed tomographic assessment, a weak positive correlation between polymerization shrinkage and void formation has been reported. In another study analyzing the effect of shortened curing modes on bulk-fill resin composites, it was shown that volumetric polymerization shrinkage was mainly affected by the materials tested, whereas shear bond strength was influenced by the materials, polymerization modes, and thermal cycling [31]. On the other hand, it was observed that optical coherence tomography (OCT) as an alternative to μ-CT, is utilized to characterize voids in a few studies. In these studies, the tendency of void formation differed between various flowable composites [21, 25], and the use of flowable composites led to an increase in the number and percentage of void volume in restorations [20].

CONCLUSIONS

It was assumed that the varying degrees of void formation in the tested resin composites were largely related to the application technique or the operator's actions, and to a lesser extent, the polymerization modes could also be responsible. Micro-computed tomography was shown to be a useful method that allows realistic visualization of the exact volume and location of possible defects and voids in restorations. Even though void formation is not the only component impacting clinical performance, more studies are needed in view of the inadequate data on the relevance between void formation and polymerization modes.

DISCLOSURES

1. Institutional review board statement: The study was approved by the Pamukkale University Non-Interventional Clinical Research Ethics Committee, with approval number: E-60116787-020-49303.
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