# Micro-computed tomography evaluation of volumetric polymerization shrinkage and degree of conversion of composites cured by various light power outputs

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This study evaluated the influence of different light-curing modes on the volumetric polymerization shrinkage and degree of conversion of a composite resin at different locations using micro-computed tomography and Fourier transform infrared spectroscopy (FTIR). Specimens were divided into 4 groups based on the light-curing mode used (Bluephase 20i): 1 —High (1,200 mW/cm<sup>2</sup>); 2 —Low (650 mW/cm<sup>2</sup>); 3 —Soft-start (650–1,200 mW/cm<sup>2</sup>); and 4 —Turbo (2,000 mW/cm<sup>2</sup>). Degree of conversion was calculated by the measurement of the peak absorbance height of the uncured and cured materials at the specific wavenumbers, and was performed by FTIR 48 h after curing resin samples. Degree of conversion was analyzed using two-way ANOVA. No significant differences were observed independent of the region of the restoration investigated (p>0.05). Different curing modes did not influence volumetric shrinkage neither degree of conversion of class I composite resin restorations.

Keywords: Shrinkage, Composite, Polymerization, Light-curing, Computed-tomography

# INTRODUCTION

Over the years, composite resins have become the most used material for direct restoration of anterior and posterior teeth<sup>1)</sup>. Clinical success of these restorations depends on many factors, and their physical properties play an important role when longevity of restorations is evaluated<sup>1)</sup>. The main properties of composite resins have been investigated, such as hardness, elastic modulus, fracture and diametral tensile strength, shrinkage stress, volumetric polymerization shrinkage and degree of conversion<sup>2,3)</sup>.

The shrinkage stress is directly related to the degree of conversion of the composite resin<sup>4,5)</sup> and the volumetric polymerization shrinkage is an inherent characteristic of composite resins due to their formulation. The effect of polymerization shrinkage and stress generated at the resin-tooth interface can lead to marginal sealing loss due to gap formation, which may evolve to recurrent caries<sup>6,7)</sup>.

A resin-based composite typically consists of an organic matrix and inorganic filler particles bonded by silane coupling agents<sup>8)</sup>. Bis-GMA is used as the main monomer and presents low shrinkage and high viscosity<sup>9)</sup>. Low molecular weight monomers are added to the organic matrix to reduce the viscosity of composite<sup>8)</sup>. However, disadvantages have been related to diluent monomers, because they undergo

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higher polymerization shrinkage than those high molecular weight monomers<sup>10</sup>. Since intermolecular distance decreases as polymerization shrinkage occurs, volumetric reduction can range from 1.5 to  $5\%^{11}$ . Thus, monomer molecular characteristics and inorganic filler content influence on polymerization shrinkage. In addition, other factors such as cavity preparation or C-factor, elastic modulus of the material<sup>12</sup> and light irradiance delivered by curing unit<sup>9,13</sup> can also affect the polymerization shrinkage stress.

As photoinitiators and light curing units may alter the final outcome of restorations, these variables are under active investigation in an attempt to minimize potentially deleterious effects on polymerization and clinical reliability<sup>14)</sup>. The incident irradiance from curing units has been studied, as stress development occurs when the material behavior transitions from viscous to viscoelastic into elastic phase<sup>4</sup>. Despite this fact, it has been suggested that high irradiance can improve depth of cure and, therefore, improving the mechanical properties of composites. It has been reported that higher shrinkage stress is generated with high irradiances relative to lower ones, consequently leading to greater gap formation<sup>15,16</sup>). Moreover, it has been suggested that lower irradiance may prolong the pre-gel phase, allowing the material to flow more and resulting in lower stress during this stage, without affecting the degree of conversion<sup>13,16</sup>). It was also reported that the reduction of irradiance delivered by curing unit from 650 to 250 mW/cm<sup>2</sup> decreased the shrinkage stress<sup>4</sup>; on the

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other hand energy density of the polymerization device is a crucial factor for resin composite polymerization, together with the thickness of the material, the duration of the exposure as well as the interaction between these three factors. Besides these items, the composition of the monomer and the photoinitiation system play an important role on the polymerization and subsequently the amount of residual monomer<sup>17</sup>.

Since measurement of the shrinkage in a cavity has proven to be technically difficult, several indirect methods to evaluate the effect of polymerization shrinkage have been described in the literature<sup>7,18,19)</sup>. The most used method to evaluate gap formation was dye penetration, which involved the sectioning of sample, being a destructive method that allows limited further material characterization<sup>20)</sup>. Other methods include optical instruments, fiber-optic sensing method, mercury dilatometer, electromagnetic balance and video imaging<sup>12)</sup>.

Contemporary 2D and 3D imaging methods have been used to analyze in a non-invasive and non-destructive way the behavior of materials inside a standardized cavity, such as finite element analysis<sup>18,21,22</sup>, optical coherence tomography<sup>7,23</sup>, and micro-computed tomography ( $\mu$ CT)<sup>19,22</sup>. These methods have been widely described in literature, due to reliable and significant results<sup>7,18,24,25</sup>.

The aim of this study was to evaluate and quantify the volumetric polymerization shrinkage of a composite resin at three locations of a class I cavity and its degree of conversion when composite resin restorations were light activated with different irradiance modes of a light curing unit. The hypothesis tested was that different curing modes would generate different volumetric polymerization shrinkage and degrees of conversion of the composite resin.

## MATERIALS AND METHODS

#### Specimen preparation

Materials composition, manufacturers and batch numbers are described in Table 1. Twenty-four tooth models with standardized class I cavity (2.0 mm depth×4 mm length×4 mm wide) were fabricated with acrylic resin (Ortho-Jet, Lang Dental Manufacturing, Wheeling, IL, USA), simulating a previous cavity designed in a natural tooth [33,34]. The replicas were obtained by silicone impressions made with Express Light Body (3M ESPE, St. Paul, MN, USA). Polymethyl metacrylate (PMMA) cavities containing the class I cavity were divided in 4 groups (*n*=6), according to four curing modes provide by Bluephase 20i LED-polywave curing unit (Ivoclar Vivadent, Schaan, Liechtenstein): 1 —High power (1,200 mW/cm<sup>2</sup>); 2 —Low power (650 mW/cm<sup>2</sup>); 3 —Soft-start (650–1,200 mW/cm<sup>2</sup>); and 4 —Turbo (2,000 mW/cm<sup>2</sup>).

#### Filling procedures

Acid etching (Total Etch acid, Ivoclar Vivadent) was applied into entire cavity for 30 s, rinsed with water and air-dried. Afterward, cavities were bonded with Adhese Universal (Ivoclar Vivadent) followed by oil-free air blowing to evaporate remaining solvent and lightactivated for 10 s with Bluephase 20i (Ivoclar Vivadent) in Low power intensity mode. Afterwards, cavities were restored with the composite resin Tetric EvoCeram (A2 shade, Ivoclar Vivadent) with one increment (bulkfilling) and placed in a dark vial in order to prevent any light source contact and unwanted polymerization. Afterwards, specimens were placed in the  $\mu$ CT ( $\mu$ CT40, Scanco Medical, Basserdorf, Switzerland) for the first scan (uncured). Light-activation was performed according to the assigned groups for 20 s. The curing unit was kept constant at a standardized position and distance, which was 1 mm from the cavity. Since the Turbo mode is programmed to emit light for 5 s, it was used four times consecutively in order to achieve the 20 s curing time. After the light-activation, the restored samples were inserted back into the  $\mu$ CT holder for the second scanning (cured).

### $\mu CT$ analysis

Each sample was scanned twice using  $\mu$ CT as previously described, before and after light-activation<sup>18)</sup>. The  $\mu$ CT was calibrated with a phantom standard at 70 kVp/ BH 200 mgHA/cm. The operating condition for the  $\mu$ CT device was: energy (70 kVp–114  $\mu$ A) with a resolution of 16  $\mu$ m/slice, using a sample holder of 16.5 mm. The average of the total number of slices per sample was ~300, with an average scan time of 25 min per sample. Acquired  $\mu$ CT data were imported into a workstation and evaluated with Amira software (version 5.5.2, VSG,

Table 1 Composition and filler loading, manufacturers and batch numbers of the materials

Material	Batch no.	Composition Bis-GMA, Benzotriazol, Triethyleneglycol dimethacrylate, Urethane dimethacrylate, Ethoxulated Bis-EMA, Barium glass filler, Ytterbium fluoride, Mixed Oxide, Prepolymers, Additives, Catalysts, Stabilizers and Pigments (filler loading: 53–55 wt%)	
Tetric EvoCeram	R84511		
Adhese Universal	T15773	Methacrylates, Water, Ethanol, Highly Dispersed Silicone Dioxide, Initiators and Stabilizers.	
Total etch	T38412	Phosphoric acid (37 wt%), Thickening agent, Water.	

Burlington, MA, USA). The total volumetric shrinkage as well as the shrinkage on the top, bottom and sides of the cavity were evaluated. The measurement of the cavity is required to superimpose the two subsequent scans (uncured and cured) with the Amira software tool called "superimposition", which provides a perfect arrangement of both images, and exempting the presence of a reference mark in the stub during µCT scanning. By doing this, we avoid the scattering and possible noise between both structures, due to its similar radio density. The "superimposition" tool has been previously used in other studies<sup>24)</sup> and is trustable, once it is done automatically by the software and allows correlating one scan to another, which ends up making sure all the cavities are at the same position and allows measuring volumetric shrinkage at different locations.

Two registered images were subjected to Boolean operations (registered  $\mu$ CT data minus  $\mu$ CT data at cavity preparation) to enable isolation and quantification of the composite restoration's volume. The cropped volume was automatically labeled with the "segmentation editor" command and subsequently reconstructed in a 3D manner. The "Material Statistics" command computed the volume changes. From these measurements, the volumetric loss following polymerization shrinkage was calculated as percentages<sup>24</sup>. Data was analyzed by one-way analysis of variance (ANOVA) at the 5% level of significance using IBM SPSS software (IBM, Armonk, NY, USA).

#### Degree of conversion (DC%)

Specimens (5 mm diameter; 2 mm height) were prepared by packing the composite material in opaque molds between two 0.5 mm thickness glass slides.

Spectra of composite specimens polymeric matrix were recorded by a Fourier transform infrared spectroscopy (FTIR) spectrometer (Spotlight 400N FT-NIR, Perkim Elmer, Weiterstadt, Germany) equipped with an attenuated total reflectance crystal (ATR). FTIR spectra of the cured specimens were registered 48 h after dry storage at 37°C in the top and bottom surfaces. Data were acquired under the following conditions: 2,000–650 cm<sup>-1</sup> wavenumber range, 4 cm<sup>-1</sup> resolution and 40 scans co addition.

The DC% was calculated from the ratio of the height of the absorbance peak of the aliphatic bond C=C (1,636 cm<sup>-1</sup>) relative to that of the aromatic C…C bond (1,608 cm<sup>-1</sup>) which are not affected by the polymerization process. Equation (1) was utilized:

$$\text{%DC}=100 \times [1 - (\frac{\text{Ap}(\text{c=c}) \times \text{Am}(\text{c} \cdots \text{c})}{\text{Am}(\text{c=c}) \times \text{Ap}(\text{c} \cdots \text{c})})]$$
(1)

Where Am is the peak absorbance height of the uncured and Ap of the cured material at the specific wavenumbers. Data were analyzed by a two-way analysis of variance (ANOVA) at the 5% level of significance using IBM SPSS software (IBM).

### Light curing unit spectrum

In order to characterize the light source used in this experiment, according to each curing mode, a spectrometer (USB2000+, Ocean Optics, Dunedin, FL, USA) connected to a 6" integrating sphere (USS 060 SF, Labsphere, Sutton, NH, USA) was used. The curing unit was turned on for each curing mode and data were



Fig. 1 Charts representing Bluephase 20i curing modes.
A: Spectrum of High Power mode; B: Spectrum of Low Power mode; C: Spectrum of Soft-start mode (red line: initial irradiance, and blue line: second step of irradiance);
D: Spectrum of Turbo mode.

obtained by software (Spectra Suite v5.1, Ocean Optics). From the light source, a spectrum of each curing mode was obtained according to different wavelength ranges: 350–550, 350–425 and 425–550 nm (Fig. 1).

#### RESULTS

Table 2 shows the volumetric polymerization shrinkage for each curing mode and the main location where the shrinkage occurred. No significant difference was noted between curing modes (p>0.05), independent of the location. Figure 2 shows the volumetric polymerization shrinkage for curing modes.

Figure 3 shows the percentage obtained for DC%. Figure 3 also shows a representation of DC% between different light curing modes. No significant difference was noted between curing modes (p>0.05), independent of the location.

Figure 2 shows the irradiance at different wavelengths for curing modes used in this study. Low, High and Turbo modes emitted blue and violet lights during all the period the light was on, while the Softstart mode started emitting violet light after 5 s it was turned on, and kept constant until the end of the 20 s. The High mode reached approximately 1,215 mW/cm<sup>2</sup>, while the Low mode 525 mW/cm<sup>2</sup>. The first step (lower irradiance) for Soft-start mode reached less than 300 mW/cm<sup>2</sup>, while the second step (higher irradiance after 5 s until the end of light irradiation) showed approximately 1,021 mW/cm<sup>2</sup>. The Turbo mode reached 1,535 mW/cm<sup>2</sup> (Fig. 3).

Figure 4 is a 3D representative composite resin restoration for each curing mode before and after lightactivation, exhibiting the locations of shrinkage. The highest amount of volumetric shrinkage was seen in the top of the cavity, followed by the pulpar floor and the proximal, consecutively. Similar volumetric shrinkage was obtained with different curing modes (p>0.05) at the top, bottom or sides of the cavity (Table 2).

Table 2Volumetric polymerization shrinkage for each curing mode (in %), standard deviations (SD) and the contribution of<br/>different locations of the cavity to total shrinkage (top, bottom and sides)

Curing mode	Total shrinkage (SD)	Top (SD)	Bottom (SD)	Sides (SD)
High	2.03 (0.23) A	98.84 (1.13) A	1.15 (1.13) A	0.00 (0.00) A
Low	2.06 (0.17) A	99.42 (0.60) A	0.50 (0.63) A	0.07 (0.11) A
Soft-start	2.65 (1.24) A	97.76 (1.94) A	2.19 (1.90) A	0.04 (0.10) A
Turbo	2.30 (0.20) A	99.37 (1.52) A	0.62 (1.51) A	0.00 (0.00) A

Uppercase letters compare curing modes for total shrinkage and locations (p<0.05).





Fig. 2 Volumetric polymerization shrinkage (%) of groups. Error bars reflect the Standard Error.

Fig. 3 Degree of conversion (%) for each light curing mode and location. Error bars reflect the Standard Error.



Fig. 4 Figures representing 3D reconstruction of composite restoration for each curing mode (A1–A2: High Power mode; B1–B2: Low Power mode; C1–C2: Soft-start mode and D1–D2: Turbo mode) and the composite shrinkage volume (turquoise blue color).

## DISCUSSION

Polymerization of dental composites is a complex process, in which many factors such as monomer composition, initiation systems, filler technologies, type of curing unit, composite filing technique, and the cavity preparations can influence the longevity of restorations<sup>26-29)</sup>. The control of intensity of emission of curing light units has been suggested in order to minimize the effects of shrinkage stress and increase the durability of composite restorations<sup>30</sup>). Pulse and progressive light curing techniques (Soft-start or Ramp)<sup>30</sup> are the most common settings used in order to control polymerization stress. Ramp curing is where intensity continuously increases over exposure and Soft-start is a low intensity start followed by a final cure at high intensity<sup>31</sup>). In our study the curing modes tested were: High, Low, Turbo and Soft-start<sup>32</sup>.

Based on the results of this study, the hypothesis was rejected since no significant differences in volumetric polymerization shrinkage nor degree of conversion were observed between curing modes delivered from the polywave light curing unit used, although different irradiance incidences were detected for the curing modes tested (High, Low, Soft-start, or Turbo). From a theoretical standpoint, lower shrinkage stress would be expected along with a progressive increase in irradiance level using Soft-start mode<sup>7</sup>, however, volumetric shrinkage was not reduced. 3D rendering of shrinkage, which represents gap formation also did not show any difference in a qualitative analysis. Previous studies have demonstrated that low irradiance can improve quality of marginal integrity, but some physical properties such as degree of conversion, flexural modulus, flexural strength and microhardness might be compromised<sup>33,34)</sup>. To overcome this concern, studies have suggested the utilization of the Soft-start polymerization technique, in which an initial light exposure irradiation step is performed with very low irradiance for five seconds followed by high irradiance until the end of light irradiation<sup>34)</sup>. In the present investigation, no significant difference in DC% was observed between top and bottom surfaces when the Soft-start mode was used. Related to volumetric polymerization shrinkage it has been reported that Low intensity modes result in less polymerization shrinkage than High and Soft-start modes<sup>33)</sup>, which does not agree with the present study. It is also found in the literature that Soft-start and ramping modes does not affect neither the shrinkage nor the mechanical properties, meaning a successful degree of conversion achieve<sup>13)</sup>.

The results of this study demonstrated that location of the volumetric shrinkage did not differ as a function of curing mode, neither degree of conversion at different surfaces. Studies reported that shrinkage may occur at deeper areas of restorations<sup>21</sup>, while other investigation found that changes in the bottom of the cavity were delayed when compared to the surfaces closer to the light source<sup>33)</sup>. Clinically the bottom and lateral shrinkage generated (gap formation), represented by 3D deformation of the uncured composite, are relevant once it may result in post-operative sensitivity, internal and marginal gaps<sup>24)</sup>. In this study, volumetric shrinkage was primarily detected at the occlusal surface, little at bottom and almost inexistent at sides. No differences were observed among groups as could be speculated. Qualitative analysis based on 3D rendering of samples also showed no difference between groups.

In order to obtain a high degree of conversion and

adequate polymerization of a light cure resin based composite, the spectral output from curing unit must match the wavelength-dependent photosensitivity of the photoinitiator used in the resin material<sup>35)</sup>. Based on this fact, this study used a polywave curing unit, that emits light at two wavelengths, between 440 and 470 nm and below 420 nm, which is able to polymerize a wide range of composites, such as the one used in this study. Adequate light exposure results in a biocompatible restoration with the manufacturers' intended physical properties and clinical longevity<sup>36)</sup>.

Degree of conversion was tested for all irradiances. Directly irradiated surfaces did not show statistically higher DC% than deeper surfaces with any of the irradiances. Samples were successfully polymerized when applied in a single 2-mm thick increment (approximately DC 70%). Since the same composite material was used for all of the samples, volume filler loading and characteristics was not an influential factor, since it has been reported in the literature that different particles can affect light scattering and restrict molecular mobility<sup>27)</sup>. This result indicates that the filler and initiator systems of the tested composite resin produce adequate degree of conversion independent of the curing mode tested. It was shown that the Bluephase 20i (Ivoclar Vivadent) reaches a peak of 410 and 460 nm for all the different light curing modes, which can help explain the present results.

The present study used artificial cavities in order to standardize samples, avoiding dimensional differences and its influence on the results. Thus, substrate PMMA, dimension of cavity preparation (and volume of restorative material), type of composite, the distance and positioning of light tip, and type of light curing unit were all standardized. It is known that most curing units do not emit homogeneous light, resulting in different degree of conversion values at the composite surface and subsurface<sup>36,37</sup>. Variations on distance from the light tip and irradiance could modify volumetric shrinkage using the same material<sup>7,22,38</sup>.

Class I cavities were selected due to their common clinical use, its high C-factor value which influence polymerization shrinkage stress making it a suitable model for volumetric polymerization shrinkage measurement<sup>39,40</sup>. Immediate scan after polymerization were made once for all of composites, previous studies have shown that 90% of the shrinkage occurs within the first hours<sup>4</sup>). Also, composite resin shade affects the light penetration in the material and its polymerization<sup>4</sup>). The most commonly used shade in restorative dentistry (A2) was employed in the present investigation in an attempt to standardize the restorative procedure.

 $\mu$ CT analysis has been an important tool to quantify and evaluate not only volumetric polymerization shrinkage<sup>20,22,41)</sup>, but also to characterize the restorative material volumetric distribution such as fillers or presence of voids <sup>18,22,42,43)</sup>. This method has been shown to depict locations of composite shrinkage when the substrate adherent was uniform, revealing gap formation regions and regions of potential interfacial failure<sup>24)</sup>.

Reducing the light intensity and modifying the polymerization technique with Soft-start mode did not significantly change the volumetric polymerization shrinkage and neither affect the degree of conversion achieved. While it was expected that the different irradiances delivered would generate different degrees of conversion between curing modes thus influencing gap formation<sup>4</sup>, this was not observed in the present study.

Further studies analyzing curing modes and its influence on shrinkage and gap formation should include the dentin/enamel as a substrate for 3D analysis and different curing units.

## CONCLUSION

Based on results of this study, it was concluded that different curing modes of the same light-curing unit did not significantly influence volumetric shrinkage or degree of conversion of a composite resin.

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# CONFLICT OF INTEREST

Authors declare that they have no conflict of interest.

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