Review Article

Analytical methods for the measurement of polymerization kinetics and stresses of dental resin-based composites: A review

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ABSTRACT

Resin-based composites are commonly used restorative materials in dentistry. Such tooth-colored restorations can adhere to the dental tissues. One drawback is that the polymerization shrinkage and induced stresses during the curing procedure is an inherent property of resin composite materials that might impair their performance. This review focuses on the significant developments of laboratory tools in the measurement of polymerization shrinkage and stresses of dental resin-based materials during polymerization. An electronic search of publications from January 1977 to July 2016 was made using ScienceDirect, PubMed, Medline, and Google Scholar databases. The search included only English-language articles. Only studies that performed laboratory methods to evaluate the amount of the polymerization shrinkage and/or stresses of dental resin-based materials during polymerization were selected. The results indicated that various techniques have been introduced with different mechanical/physical bases. Besides, there are factors that may contribute the differences between the various methods in measuring the amount of shrinkages and stresses of resin composites. The search for an ideal and standard apparatus for measuring shrinkage stress and volumetric polymerization shrinkage of resin-based materials in dentistry is still required. Researchers and clinicians must be aware of differences between analytical methods to make proper interpretation and indications of each technique relevant to a clinical situation.

Key Words: Analytical procedure, polymerization, resin composite, shrinkage, stress

INTRODUCTION

Despite recent advances in new restorative materials, all resin-based composites exhibit a reduction in volume related to their polymerization reaction. Considering that these materials are bonded to tooth cavities, this volume contraction generates internal stress, which in turn compromises the mechanical and chemical stability of the restorative materials and may decrease marginal adaptation.[1]

It is of great importance to minimize the interfacial stresses during polymerization. This is because the contraction of the restorative material may cause debonding at the adhesive interface, postoperative sensitivity, marginal discoloration, recurrent caries, fracture of margins, and finally loss of the restoration.[2,3] The value of these stresses can be related to the material composition as well as the

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restorative technique and degree of conversion (DC). One of the issues associated with the restorative procedure is cavity configuration factor or C-factor that is directly related to stress and is defined as restoration's bonded-to-unbonded ratio.[4] Increasing C-value enhances the rate of shrinkage stress that leads to a decreasing flow capacity. The higher the C-factor, the less free surfaces there will be, and thus, there is little chance for the dental composite to flow and adapt changes in volume.[4,5] It has been revealed that the higher C-factor affected the interface sealing and made largest gap formation.[6]

Several techniques have been proposed to minimize the potential stress generation. For instance, a photo-activation with an initial low irradiance is followed by an exposure with higher irradiance known as "soft start method."[7] Reduced irradiation intensity within the early stages of polymerization is assumed to let stress relaxation occur before vitrification.[8] There is one variation for this technique named "pulse-delay technique" in which an interval is considered between the two pulses. In this technique, after the initial curing, it would permit to continue the polymerization in the darkness at slower rates and then followed by the irradiation at higher irradiance to ensure acceptable mechanical properties and DC.[9] Progress has been made in the conventional bisphenol A-glycidyl methacrylate (Bis-GMA)-based resin composites through the addition of new monomers such as urethane dimethacrylate (UDMA), ethoxylated bisphenol-A dimethacrylate, combined with superior fillers to generate low-shrinkage restorative materials. This is one way that has been employed by the manufacturers to reduce the shrinkage stress of dental composites without degrading the mechanical properties.[3]

Advances in the structure of methacrylate monomer have been made to deal with the shortcomings of the common composites; however, several solutions have been introduced by changing the reaction mechanism as well. This could be either by altering the active center from radical to cationic, changing the nature of the network, using ring opening species, or even by inducing phase separation.[8]

Shrinkage of resin-based dental materials has been measured by various methods in the literature. Magnitude of shrinkage strain value of a certain material is dependent on and varies with the measurement technique.[10] Besides, values obtained for any single method of measurement may vary between operators. Therefore, comparisons between published data are quite difficult while researches are being carried out in different laboratories by different operators and equipment. Each developed method for calculating the shrinkage that occurs during polymerization relies on the different physical principles for measurement.[10,11]

This study reviews the significant developments of laboratory tools in the measurement of the polymerization shrinkage and stresses of resin composite restorative materials during polymerization. Various methods have been used for evaluating the polymerization kinetics of dental resin composites. These methods can be divided into two groups: (1) measurement of shrinkage-strain and (2) measurement of shrinkage-stress. In this overview, we hope that readers better understand the process of shrinkage measurement, the factors that may contribute to the differences between the various methods in measuring the shrinkages, and also derive a rational basis for comparison.

MEASUREMENT OF SHRINKAGE-STRAIN

The majority of the methods used for evaluating polymerization shrinkage of resin composites can be divided into two groups. some methods are able to evaluate the change in density and volume (dilatometer and pycnometer and buoyancy method), and other methods utilize linear measurements (linometer, interferometer, thermo-mechanical, and optical analyses).[12]

Some of these methods involve direct contact with the specimens such as linear vertical displacement transducer (LVDT), dilatometers, pycnometers, and strain gauges. These methods may create stresses and additional deformations, therefore altering the measurement because the material is a viscous fluid at the beginning of the polymerization and does not resist even to small loads. The noncontact techniques such as three-dimensional (3D) microtomography, optical coherence tomography (OCT), and digital image correlation (DIC) have also been introduced.[12] The shrinkage kinetics of the composite materials can be characterized along the light-irradiation process using methods such as "bonded disk" method. These experimental approaches precisely measured the free strain and defined the polymerization kinetics.[13]

Dilatometry

Dilatometry is the most commonly used method for measuring the polymerization shrinkage. In this method, a nonreacting liquid such as mercury surrounds the composite sample during the curing period. Monitoring the mercury level in a capillary tube enables an operator to measure the volumetric shrinkage (VS) related to polymerization of the sample. Thus, the magnitude of shrinkage or "total shrinkage" is recorded in this way considering that the shrinkage is monitored during the curing period from the pre- to post-gel phases.[10]

In 2002, Oberholzer et al.[14] introduced an electronically controlled mercury dilatometer which measured the changes in volume of the sample every 0.5 s. In this design, the specimen is put into a sample holder with a specified volume. Then, the assembly is put into a glass beaker filled with distilled water and sealed. This beaker is coupled to a tube filled with mercury that connected to a calibrated capillary. Oberholzer's modified design was able to measure the volumetric displacements in dental materials every 0.5 s, with a measurement error of <0.027% in any phase, and an average coefficient of variation of <1.22% could be obtained. Compared with the overall shrinkage percentage in dental resins, measurement error was at an acceptable level for comparative experimental work. The apparatus is particularly appropriate for the study of polymerization shrinkage in the light-cured dental resin composites.[14]

In this dilatometer, instead of manual viewing, the change of the mercury level in the capillary could be measured electronically. In addition, the measurements in the electronic dilatometer are made regardless of shape and size of the specimen. However, sensitivity to thermal variations is one of the disadvantages for this method.[14,15]

Furthermore, Mulder et al.[15] utilized this design to determine the volumetric change of four bulk-fill flowable composites and Z250 composite as a standard control. They found that all the bulk fill flowable composites revealed higher volumetric alterations than that of Z250. The composite with the highest filler content (Z250) displayed the lowest shrinkage (1.13%), however, this was not observed for the flowable composites.

Jongsma and Kleverlaan[16] evaluated the effect of temperature on the contraction stress and strain of four commercial composites using tensilometer and

mercury dilatometry, respectively. The test procedure was carried out at different temperatures (23, 30, 37, and 44°C). They found that increasing the temperature can lead to the higher volume shrinkage of dental resin composites. Nevertheless, the contraction stress did not change significantly at higher than 30°C.

Yamamoto et al.[17] compared the water-filled dilatometer and laser speckle correlation methods on the different flowable resin composites and a hybrid resin composite as control. The average volume changes of the resins after 180 s ranged from ~3.3% to 4.4% for the flowable composites and from ~1.8% to 2.3% for the hybrid composite. The overall obtained data from the speckle contrasts decreased shortly after the initial light exposure started and then gradually increased. The speckle contrast results showed that the polymerization changes of the flowable resins were more than that of the obtained results by the water-filled dilatometer. The results suggested that the polymerization characteristics of flowable composites could be measured successfully using both methods.

Linometery

In 1993, de Gee et al.[18] introduced a modified linometer, which was reasonably simple, fast, and insensitive to temperature changes producing constant results. They found no significant differences between this method and dilatometry. The composite specimen was placed between the glass slide and an aluminum disk. The distance from the aluminum disk to the glass could be adjusted with a displacement transducer to select the height of the resin sample. The disk and glass were greased to avoid adhesion of the composite sample. This apparatus, however, measured only linear changes. The linear polymerization shrinkage was calculated by the following formula.

$$\text{Linear shrinkage (LS)} = \frac{\Delta L}{L + \Delta L} \times 100$$

Where specimen L is the specimen thickness after polymerization and ΔL is the recorded displacement. This technique like other techniques was based on the measuring linear shrinkage using contact displacement transducers; may have potential errors related to the effect of gravity or nonuniform shrinkage.[18]

Gonçalves et al.[19] evaluated the influence of bis-GMA/triethyleneglycol dimethacrylate (TEGDMA) (B/T) and UDMA/TEGDMA (U/T) ratios on the polymerization stress (PS) and VS. PS and VS were determined with a universal testing machine (UTM) and linometer,

respectively. Increasing the base monomer content in both series enhanced the viscosity and polymerization maximum rate (Rp_{max}) while decreased PS and VS. In addition, the UDMA-based materials showed higher DC, PS, and VS and lower viscosity. Linear shrinkage was measured via above formula and converted to VS by the following equation:[19]

$$\text{Volumetric shrinkage (VS)} = 3LS - 0.03LS^2 + 0.0001LS^3$$

Where LS represents the linear shrinkage of the specimens.

Yamasaki *et al.*[20] evaluated the "low-shrink" composite materials (Kalore, N'Durance, and Filtek P90) and control group (Esthet X HD). The VS was investigated by the linometer and the highest value was obtained for Esthet X HD, followed by N'Durance. However, Filtek P90 and Kalore presented the lowest and statistically similar extent of shrinkage.

Gas pycnometery

In 1999, Cook *et al.*[21] used a noncontact method called gas displacement pycnometer to determine the volume changes during polymerization of composite materials in a dry state. This method is not time-consuming but measures only the final quantity of shrinkage.

First, the specimen of composite paste was sandwiched between two small sheets of a thin Mylar sheet. Then, the volume of the sample plus Mylar sheets was determined in a constant room temperature (23°C ± 2°C) by a controlled gas pycnometer with a reservoir of helium gas. The volume of a specimen placed in the specimen chamber was then measured by the pressure change of helium when the pressurized expansion chamber was opened up to the specimen chamber. Then, after curing, the volume of the composite specimen plus Mylar sheets was redetermined, and the volume differences between uncured and cured specimens (ΔV) were calculated. The Mylar sheets were then removed from the specimen and the volume (V) of the cured specimen alone was determined. Quantification of the percentage shrinkage (S) can be calculated using the following formula:[21]

$$\text{Shrinkage (S)} = \frac{\Delta V}{V + \Delta V} \times 100$$

Average of at least four determinations for each material was considered as the VS.

Amore *et al.*[22] evaluated the polymerization shrinkage of three packable dental composites with different distances from the light tip to the surface of the composites (2 or 10 mm) by a gas pycnometer. They found no statistically difference in the polymerization shrinkage for the three tested composites, regardless of the materials or distances.

Similarly, Cilli *et al.*[23] evaluated the volumetric contraction of five resin composites (Durafil VS, Z100, Filtek Z250, Filtek P60, Surefil) using the gas pycnometer. They found that Filtek Z250 and Filtek P60 composites showed the least and highest percentage of shrinkage among the tested materials, respectively. In a study conducted by Maia *et al.*,[24] the polymerization shrinkage was evaluated by gas pycnometer method. They found that the Silorane-based composites presented the lowest value for shrinkage, followed by the nonflowable resin-based composites (RBCs). The lower values of shrinkage were reported for the flowable RBCs. The depth of cure and microhardness were also investigated. Their findings have shown that although the filler content is an important feature which controls the polymerization shrinkage, it is not the only factor that influences the material properties.

Archimedes principle (buoyancy method)

Archimedes principle (buoyancy of a material in fluid) is a simple and inexpensive method that has been used to evaluate the volumetric changes by measuring density variations. When a body is immersed in a liquid, it is buoyed up by a force equivalent with the weight of the dispersed liquid. Whether a given body will float, sink, or remain static in a given fluid depends on both the weight and volume of that material. The relative density – the weight per unit volume of the body compared to that of the liquid – determines the buoyant force.[1,11]

This method includes weighing the material several times in two distinct environments of the recognized density (conventional air is used as one of the environment). Several liquids such as distilled water, mercury, silicone oil, and sodium lauryl sulfate with at least 99.0% purity can be used as the second surrounding medium.[25] Density of specimen is calculated according to the below equation:

$$\rho = \frac{m_{water}}{m_{air} - m_{water}} (\rho_{water} - \rho_{air}) + \rho_{air}$$

Where ρ is the density of the material, m_{water} is the weight in grams (g) of the specimen in water, m_{air}

is the weight in grams (g) of the specimen in air, ρ_{water} is the density of water at the exactly measured temperature according to the density table for distilled water, and ρ_{air} is the density of air that considered 0.0012 g/cm.[11]

$$\Delta V = (\frac{1}{\rho_{15min}} - \frac{1}{\rho_{uncure}})\frac{1}{\rho_{uncure}} \times 100\%$$

There are several characteristics for this method:

- Using Archimedes method, the entire volumetric change is taken directly providing 3D VS[11]
- Specimen size and geometry are not considered as a problem when applying Archimedes principles
- This method is based on weighing the specimen before and after polymerization and using the values to calculate specific gravity before determining VS
- This method is a multistep and time-consuming process and a number of variables including the presence of voids inside the specimen or air bubbles on its surface can affect the results[11,26]
- This is the only method for measuring polymerization shrinkage that has published standards for execution in ISO 17304.[25]

Lee et al.[27] examined the volumetric polymerization shrinkage of two anterior-posterior restorative hybrid composites, three posterior restorative hybrid composites, and two flowable composites using the buoyancy method. Packable composite showed the lowest, and the flowable composites showed the highest shrinkage. They found that increasing the light intensity enhanced the maximum rate of polymerization shrinkage while decreased the peak time.

Koplin et al.[1] combined the continuous buoyancy measurements and models for polymerization kinetics which appeared to be a promising approach to describe and analyze the volume- and temperature-behavior of different dental composites. They investigated the volume and thermal behavior of four dental composites with different modes of initiation. In addition, they reported that the influence of different initiation modes and light sources on the development of the volume and the temperature of the composites during the curing reaction could be evaluated.

In 2011, de Melo Monteiro et al.[11] compared the polymerization shrinkage values of different resin-based dental composites (Filtek Z250TM, Filtek Z350TM, Filtek P90TM/3M ESPE, Esthet-XTM,

TPH SpectrumTM/Dentsply, Tetric CeramTM/Ivoclar-Vivadent) using the OCT and buoyancy method. The obtained results varied with the method used. Despite numerical differences between two methods, the ranking of the materials was very similar and Filtek P90 presented the lowest shrinkage values.

Strain gauge

Strain gauges are very sensitive to linear dimensional changes. In this technique, the gauge is bonded to a substrate and the linear dimensional changes occurring in the substrate are transferred to the gauge and measured. It is interesting to note that when the substrate has a measurable modulus to induce stress on the gauge, the linear dimensional changes would transfer to the gauge. Thus, this method is applicable to measure the postgel shrinkages of composites.[28]

Sakaguchi et al.[28] have evaluated three types of resin composites (microfilled, hybrid, and posterior) for polymerization temperature rise. They also investigated the shrinkage during curing and shrinkage for various shades of theses composites using strain gauge. The posterior composite (P-50) demonstrated the lowest temperature rise and polymerization shrinkages. The shrinkage of Silux Plus (microfilled composite) dark gray was significantly lower than that of all other materials with other shades.

Shekhli[29] studied the influence of light intensity reduction and elongation of the curing time with soft start polymerization and pulse cure mode on the polymerization shrinkage by strain gauge. They found that the prolonged low-intensity pulse cure mode significantly resulted in lower and gradual post-gel polymerization shrinkage strain for all the composites tested.

In 2010, El-Korashy[30] evaluated the effect of preheating temperature of dental composite, light curing regimen (mode and duration) on the postgel shrinkage strain, and DC of a hybrid resin composite. They found that preheating of resin composite before curing significantly not only increased its DC but also increased its postgel shrinkage strain. In addition, the soft start curing mode decreased the postgel shrinkage strain of the composite without altering the DC.

The bonded–disk shrinkage–strain measurement method

This method was initially developed in 1991 by Watts and Cash.[31] It has been internationally adopted by a number of academic and industrial research laboratories.[32] A schematic picture of bonded-disk

shrinkage strain instrument is shown in Figure 1. The "bonded disk" technique has a simple design and does not require expensive instrumentation.[10] A disk-shaped unset specimen is placed upon a rigid glass plate and is sandwiched between the glass plate and a thin microscope cover-slip. In some cases, light sand-blasting of the glass plate is needed to improve adhesion to the specimen. This cover-slip diaphragm is also supported by an outer circumferential brass ring with a square cross-section. The internal diameter of the brass ring must exceed the diameter of the specimen disk[33] by monitoring the deflection of a thin glass cover-slip. Shrinkage is indirectly measurable using a linear vertical displacement transducer (LVDT) and recorded by a computer.[10] It has been reported that this geometry would ensure a certain configuration factor that controls the shrinkage direction.[33]

Other advantages are as follows:
• Shrinkage direction is determined by the configuration factor rather than by the light irradiation direction
• This technique diminishes the shrinkage in a radial direction as low as possible while maximal shrinkage happens in the vertical (axial) direction. Consequently, the VS will be equal to axial shrinkage of the material
• Thickness of specimens is low, and therefore, light penetration occurs easily. This ensures equal monomer conversion on upper and lower surfaces and consequently throughout the thickness
• Specimen diameter corresponds to light-beam diameter of curing units
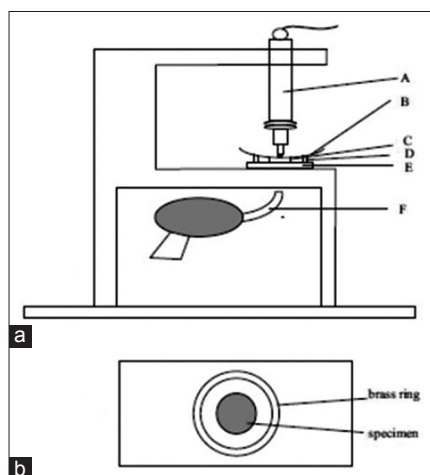

Figure 1: (a) Schematic picture of bonded-disk shrinkage strain instrument; (A) LVDT transducer, (B) cover slip, (C) disk specimen, (D) brass ring, (E) rigid glass plate, and (F) light source. (b) Disk specimen mounted on the rigid glass plate.

• Light-intensity and specimen temperature is controllable.[33]

The values resulted from bonded-disk method are similar to those from a mercury dilatometer. The correlation between two methods is surprising because the bonded-disk method measures the dimensional change along one axis while the dilatometer evaluates the volumetric changes. However, the results from the bonded-disk method would mostly depend on the dimensions and boundary conditions of the specimen.[10]

It has been reported that C-factor can be calculated via the following equation:[34]

$$\text{C-factor} = \frac{D}{(2 \times [h + \Delta h])}$$

Where h is the thickness of the cured specimen, Δh is measured shrinkage value by the LVDT, and D is the diameter of the sample.

There are two possible views about calculating the bonded and nonbonded surface areas considering whether the surface that is in contact with the flexible cover-slip is counted as bonded or as (free) nonbonded. Watts and Marouf[32] proposed that this surface is a free surface because the cover-slip is highly compliant. Therefore, the bonded surface area (S1) is only the lower surface while the free surfaces (S2) are above and at the periphery. The C-factor is then

$$\text{C - factor} = \frac{s_1}{s_2} = \frac{1}{(1 + 4[\frac{h}{d}])}$$

For precise determination of maximum final shrinkage-strain values, Watts and Marouf[32] proposed a high aspect ratio (7–9:1). Disk diameter of 7–9 mm and thicknesses of 1 mm of the bonded disk should be utilized and similar results with precision dilatometry may be obtained. The uniaxial strain value was reduced in diameters below 7 mm, 5 mm, or lower.

Lee et al.[34] examined four commercially available composites. The axial polymerization shrinkage and free VSs at varying C-factors by different specimen geometry were determined using "bonded-disk method" and buoyancy method, respectively. For calculation of the VS from axial shrinkage using the bonded disc method, C-factor of the specimens should be higher than 5. Because at the C-factor of 5–6 and higher, the axial shrinkage approached the true VS and reached a plateau.

Alnazzawi and Watts[35] selected six commercially available resin composites with different filler loadings. They measured simultaneously the shrinkage strain, exotherm, and coefficient of thermal expansion (CTE) with a modified bonded-disk instrument that included a temperature-monitoring apparatus. It was found that shrinkage strain, exotherm, and CTE would decrease with increasing filler loading. Furthemore, a positive correlation was found between the shrinkage strain and CTE for the materials tested.

Optical coherence tomography

OCT is a noncontact and noninvasive medical diagnostic imaging modality with a safe broadband light source and high resolution. The basic principle of OCT is analogous to computerized tomography, magnetic resonance imaging, and B-scan ultrasound which uses X-rays, spin resonance, and sound waves, respectively, unless only light is used.[36] OCT technique is able to obtain simultaneously high-resolution images of teeth and periodontal tissues without exposing the patient to ionizing radiation.[37]

OCT is based on a Michelson interferometer with a low coherence and broadband light source. This system includes three main parts: a scanning probe, a base unit, and a computer. The base unit contains the superluminescent diode light source.[11] At first, an empty cylindrical Teflon mold is scanned to ensure its accurate height. For recording the exact amount of uncured resin composite, a second scan is performed after insertion of composite. Fifteen minutes after photoactivation, the third scan is performed. Linear shrinkage is then calculated according to the following formula:

$$\text{Linear shrinkage} = \frac{RC_{0min} - RC_{15min}}{RC_{0min}} \times 100\%$$

Where $RC_{0\ min}$ is the mean composite thickness between points 1 and 2 in the unpolymerized state and $RC_{15\ min}$ is the mean composite thickness between two points in the polymerized state. Thus, it is possible to measure the linear shrinkage using OCT. The refractive index of all materials can also be calculated using the previously taken pictures.[11]

de Melo Monteiro et al.[11] have compared seven resin-based dental composites. For measuring the linear shrinkage, the thickness of the samples was measured before and after photo-curing using OCT. Polymerization shrinkage was also measured using buoyancy method of Archimedes. The results showed that polymerization shrinkage values vary with the method used. Despite numerical differences, the ranking of the resins was very similar such that Filtek P90 (a Silorane-based composite) presented the lowest shrinkage values than that of other microhybrid composites.

X-ray microcomputed tomography

X-ray microcomputed tomography (μCT) has been recently used to examine the 3D marginal adaptation of the light-cured resin composite restorations and interface of the tooth–adhesive composite. Furthermore, high-resolution μCT is able to obtain actual 3D information from the cavity during polymerization.[38]

Sun and Lin-Gibson[39] investigated the volume of dental resin composites before and after polymerization using μCT and determined the polymerization shrinkage. μCT tolerates air bubbles because they are not counted in determining the volume of composites. Moreover, μCT provides the same accuracy for different shapes and physical states. VS in this technique is examined regardless of the degree of constraint. The obtained results were in agreement with the extent of shrinkage obtained via density measurements for the same sample. In the above study, with the addition of radiopaque filler, sufficient contrast between the sample and background is acquired. Volumes of specimens were calculated by appropriate image analysis procedures.

Chiang et al.[38] studied the composite which has been traceable using glass beads, with and without dentin adhesive, and digitized with μCT. Orientation of displacement vectors in unbonded restorations were inward to the center of mass, while the bonded restorations showed two shrinkage patterns: toward one side of the tooth cavity and/or toward the top-surface of the restoration. This method enables us to visualize the real deformation vectors generated by curing contraction.

AcuVol

Accurate VS can be measured using the AcuVol by Bisco Company. This instrument utilizes a video-imaging method which allows comparison of the volumes of dental composites before and after polymerization shrinkage. The AcuVol includes a tabletop instrument that connects to a computer.[40] This technique has been shown to yield results comparable to those observed using mercury dilatometry. Its ease of usage and capability to follow

VS during the entire curing are another advantages of this technique.[26]

Tiba *et al.*[26] compared the polymerization shrinkage of three dental resin composites using two commercially available video-imaging devices (AcuVol and Drop Shape Analysis System). Statistical analysis revealed that the two imaging devices produced equivalent results for the two of materials being tested but not for the third one (Venus).

Blackham *et al.*[41] evaluated the properties of newer hybrid resin composites with prepolymerized filler particles and compared them with traditional hybrids and a microfill composite. They found that the traditional hybrid composites (Esthet-X, Z250) had higher strength and modulus, hybrid composites containing prepolymerized fillers (Premise, Gradia Direct Posterior) showed more moderately strength, and the microfill composite (Durafill VS) had lower strength. Polymerization shrinkage was determined by video-imaging device (AcuVol, Bisco). It was found that Premise and Durafill VS had the lowest polymerization shrinkage. Using prepolymerized filler particles, resin composites benefit by having lower polymerization shrinkage. However, increasing esthetics and polishability of hybrid resin composites containing prepolymerized filler particles probably offset possible reduction in their strength properties.

In 2010, Lien and Vandewalle[42] used AcuVol to determine the polymerization shrinkage of dental restorative materials. They found that the Silorane-based material (Filtek LS) compared to the methacrylate-based composites had the lowest polymerization shrinkage; however, they had an overall mixed mechanical performance. Filtek LS had relatively higher flexural strength, modulus, and fracture toughness but relatively lower compressive strength and microhardness.

Digital image correlation
It is another noncontact optical method that has been used to measure the polymerization shrinkage in dental composites. This method was introduced at the University of South Carolina in 1980s and has been extensively used to measure strain, flow, and displacement in recent years.[43]

Previous methods do not help understand how and where shrinkage stress develops in the real restored teeth. In addition, they do not display how the cavity walls constrain the shrinkage of composites and would lead to the creation of shrinkage stress in

restorations.[44] The basic idea of DIC is to compare visible patterns on the sample surface from sequential images taken during the deformation of material. Thus, displacement and strain fields can be determined through tracing the movement of the visible points on a specimen's surface.[43] One of the main advantages of this method is full-field measurement which is very beneficial in observing nonuniform deformation and strain patterns.[44] Figure 2 shows schematic presentation of the images taken before and after the deformation of a specimen and procedures for showing the displacement vectors by DIC method.

Li *et al.*[45] calculated the VS using a single-camera 2D measurement on a commercial composite and compared it with the value reported by the manufacturer. After reaching its peak value, the shrinkage strain gradually decreased with increasing distance along the beam length before leveling off to a value of approximately 0.2% at a distance of 4–5 mm. The shrinkage curves with respect to the specimen depth and time were obtained. Maximum shrinkage occurred at a depth of approximately 1 mm rather than the surface. They considered oxygen inhibition layer as a possible explanation for this issue. Using an irradiance of a 180 mW/cm^2 compared to that of 450 mW/cm^2, seemed to be sufficient in achieving the maximum depth of cure for the materials tested in their study.

Chuang *et al.*[13] evaluated the influence of cavity shape and lining materials in MOD composite restorations by characterizing the polymerization shrinkage and cusp deflection using a Digital Image
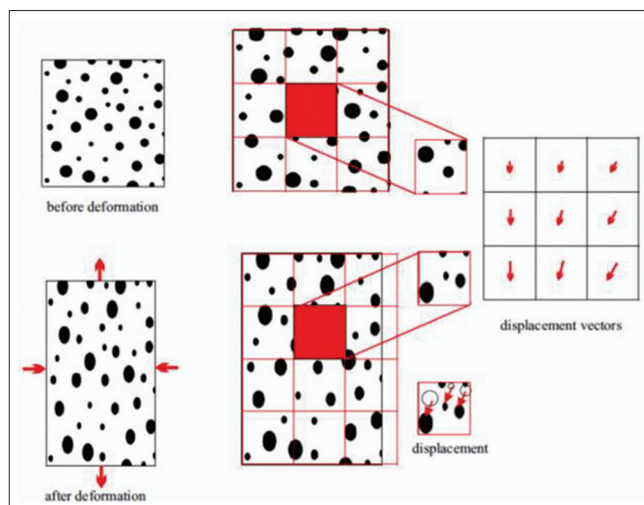

Figure 2: A schematic presentation of the images taken before and after the deformation of a specimen and procedures for showing the displacement vectors by digital image correlation method.

Correlation technique. They found that initially covered with flowable composite linings revealed greater amount of composite displacements on free and bonded surfaces compared to that of covered with glass ionomer lining and unlined groups. It was shown that there was a positive correlation between cusp deflections with cavity depth and the cusp compliance, while shrinkage in the free surfaces was dependent on the cavity width and C-factor.

In 2014, Li *et al.*[44] prepared specimens with model cavities making of cylindrical glass rods. To obtain high-contrast speckles, after filling the cavity with composites, the surfaces were sprayed with a thin layer of white paint followed by fine black charcoal powder. They took pictures both before curing and 5 min after curing. Eventually, the two pictures were correlated using the Digital Image Correction software to assess the displacement and strain distributions. They found that the resin composite showed vertically shrinkage toward the bottom of the cavity. Top center portion of the composite restorations had the largest downward displacement. Simultaneously, the composite shrunk horizontally toward its vertical midline. This shrinkage stretched the material in the proximity of the "tooth–restoration" interface. They

reported that this fact in the clinical situation may lead to cuspal deflections and high tensile strains around the composite restorations.

A summary of shrinkage strain data in the literature for several commonly used dental resin composites is provided in Table 1.

MEASUREMENT OF SHRINKAGE-STRESS

Advances in developing methods for measuring shrinkage-stress has not been fast. One of the problems is design of the specimen holder, which must become bonded in the process of measurement. It must be efficiently de-bonded to affect a subsequent measurement.[33] It has been emphasized that the shrinkage stress is not a material property and it is an outcome of multiple factors that special ways have to be used for evaluation.[47]

Shrinkage stress values can be accessed by several analytical techniques such as finite element analysis (FEA),[48-52] photoelastic analysis,[53-55] and more commonly, using an experimental setup known as "tensilometer,"[3,56-58] Bioman device,[59,60] and crack analyzing method.[46,61,62]

Table 1: Shrinkage strain data in the literature for the several dental resin composites

Material	Manufacturer	Method	Percentage shrinkage (SD)	Reference
Filtek Z250	3M ESPE, St Paul, MN, USA	OCT	2.63 (0.66)	[11]
		Archimedes	2.04 (0.18)	[11]
		Dilatometry	1.034 (0.0126)	[14]
		Gas pycnometery	2.4 (0.1)	[16]
		Bonded disk	0.56 (0.3)	[23]
			1.59 (0.052)	[32]
Filtek Z350	3M ESPE, St Paul, MN, USA	OCT	1.02 (0.38)	[11]
		Archimedes	1.28 (0.06)	[11]
Z100	3M ESPE, St Paul, MN, USA	Gas pycnometery	1.97 (0.3)	[23]
		Archimedes	2.51 (0.08)	[27]
		Bonded disk	2.70 (0.03)	[34]
Filtek P60	3M ESPE, St Paul, MN, USA	Gas pycnometery	3.26 (0.4)	[23]
		Archimedes	1.92 (0.07)	[27]
		Bonded disk	2.07 (0.04)	[34]
Tetric Ceram	Ivoclar Vivadent, Schaan Liechtenstein	OCT	1.94 (0.07)	[11]
		Archimedes	0.94 (0.27)	[11]
		Archimedes	2.69 (0.09)	[27]
Heliomolar	Ivoclar Vivadent, Schaan Liechtenstein	AcuVol	2.3 (0.1)	[46]
Esthet-X	Dentsply Caulk, Germany	OCT	1.65 (0.32)	[11]
		Archimedes	1.74 (0.37)	[11]
		Linometery	2.78 (0.08)	[20]
N'Durance	Septodont, Lancaster, PA, USA	Linometery	2.24 (0.07)	[20]
Silorane Filtek P90	3M ESPE, St Paul, MN, USA	OCT	0.70 (0.01)	[11]
		Archimedes	0.88 (0.36)	[11]
		Linometery	1.76 (0.03)	[20]
GC Kalore	GC Corporation, Japan	Bonded disk	1.70 (0.03)	[35]
		AcuVol	2.0 (0.0)	[46]

OCT: Optical coherence tomography; SD: Standard Deviation

Stress analyzer: Tensilometer – (universal testing machine with an extensometer as a feedback system)

The use of a tensilometer to investigate the shrinkage stress was introduced in dentistry in 1967 by Bowen.[58] This method has been used more frequently. Briefly, the experimental setup includes two metal or glass rods. These rods are connected to the opposite clamps of an UTM. The resin composite is placed between the opposing flat surfaces of the rods, and the axial force produced by its polymerization shrinkage is monitored for a certain time interval. Force values are divided by the cross-section area of rods to calculate the nominal stresses.[63] A schematic illustration of the universal machine experimental setup (a) and stress analyzer apparatus (b) is shown in Figure 3.

The use of this method raised several controversies among researchers. The effect of the deformation (system compliance) of the testing setup on force development remains a point of argument. The elongation of the components as well as the approximation of the opposite rods in test setup during applying the force could reduce the values recorded by the load cell. System compliance can be reduced by adding a feedback system such as an extensometer to the assembly. With extensometer, any approximation between the rods during composite shrinkage is detected and the crosshead is moved in the opposite direction. Thus, the initial height of the sample maintains constant. Therefore, the extensometer minimizes compliance by eliminating any deformation that takes place beyond the fixation (attachment) points. However, some deformation of the rods still occurs within the extensometer attachments that could affect stress data.[63,64]

Witzel et al.[56] investigated the influence of specimen dimensions on the PS of a dental composite. A linear
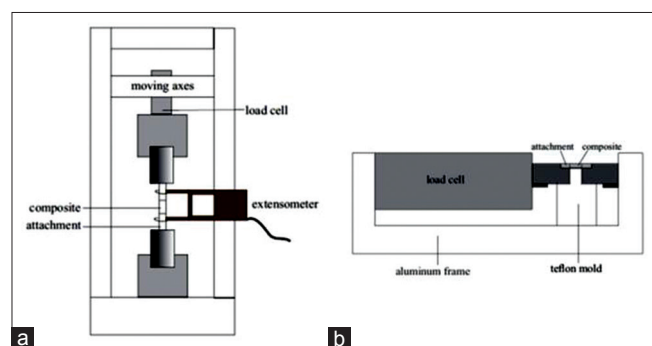

Figure 3: A schematic illustration of the universal machine experimental setup (a) and stress analyzer apparatus (b).

correlation between the PS and "C-factor" was found. Cadenaro et al.[3] evaluated the shrinkage stress of three resin composite restorative materials during photo-polymerization: a microhybrid composite (Filtek Z250); a nanofilled composite (Filtek Supreme); and a low-shrinkage composite (ÆLITE™) using a stress analyzer device. They found that Ælite LS exhibited the lowest shrinkage stress values, whereas the difference between Filtek Z250 and Filtek supreme was not statistically significant.

Another investigation[57] compared the polymerization shrinkage stress of resin composites (microhybrid, hybrid, and microfilled) photo-cured by quartz-tungsten halogen light and light-emitting diode using the UTM attached to an extensometer. The shrinkage stresses 40 s after polymerization and 10 min later were analyzed. The shrinkage stresses for all the composites were higher at 10 min than that of at 40 s regardless of the irradiation source. The microfilled composite revealed the lowest shrinkage stress value. It was reported that the light source had no effect on the shrinkage stress of hybrid and microhybrid composites, except for the microfilled composite at 10 min. Consequently, the composition of resin composite had the strongest influence on the shrinkage stresses.

Bioman shrinkage-stress measurement method

First, Bowen[58] and Hegdahl and Gjerdet[65] recorded the development of shrinkage forces with the UTM. Other researchers have used a servo-hydraulic UTM, in which careful procedures have been deployed with the aim of eliminating system-compliance to a great extent. However, the equipment was expensive and complex. Furthermore, this equipment includes construction of special attachments and couplings which tend to increase the machine-compliance requiring correction.

The Bioman shrinkage-stress device was introduced at the University of Manchester with an innovated design. This design is able to measure stress corresponding to a standardized and clinically suitable system compliance.[33,60] The system is based on a cantilever load cell fitted with a rigid clamp. The compliant end of the cantilever holds a circular steel rod. The counterface consisted of a removable rigid glass plate that is held rigidly relative to the base plate in a special clamp during measurement. The resin composite is then placed between the treated plate by sandblasting and vertical rod to form an

unset specimen disk. The composite is cured through its thickness dimension from below. Thickness of specimen corresponds to different ratios of bonded to unbonded surface areas that is equivalent of configuration factor.[60]

$$C\text{-}factor = \frac{d}{2h}$$

Where d and h are the diameter and thickness of the disk specimens, respectively.

After amplifying the load signal from the cantilever cell, the signal is received by the computer. The registered load is then divided by the disk area to calculate the stress values in MPa.[60]

Watts *et al.*[60] tried to outline design parameters for a new methodology for the problem of simultaneous stress-kinetic measurements, especially for the light-cured materials. Because studies of free shrinkage-strain kinetics on the restoratives had begun to increase, there were very few investigations on the stress-kinetic measurements. Specimens with the thicknesses of 0.8 and 1.2 mm were made from four resin composites. Concurrent measurements were made of the end displacement of the cantilever load cell, relative to a lower glass plate retaining the specimen. Overall magnitudes of stress values were between 5 and 8 MPa. The maximum stresses created with the greater thickness correspond to lower C-factor and were significantly higher. However, the increases of stress with thickness were only moderate, in the range of 11%–15%. They claimed that their new device is a feasible system for rapid and precise measurement of stress-kinetics in the photo-cured and also self-cured resin-based materials.

Spinell *et al.*[59] determined the polymerization shrinkage-strain (S_Y) and shrinkage-stress (S_Z) of six resin cements and compared their performance with the DC data. Three self-adhesive and three nonself-adhesive resin cements were evaluated in their study. S_Y and S_Z were measured by the bonded-disk method and Bioman instrument, respectively. Then, the DC was measured by Fourier transform infrared spectroscopy–Attenuated total reflectance spectroscopy. They found that the setting reactions for the self-curing materials were considerably slower than that of dual-cured. Furthermore, dual-curing enhanced the resin-matrix conversion. Dual-curing significantly increased both the polymerization shrinkage-strain rate and shrinkage-stress rate. Therefore, the

maximum polymerization rates occurred at the earlier time-points. Only the self-curing materials had decreased shrinkage-stress magnitudes. Moreover, the self-curing systems resulted in lower DC whereas did not necessarily result in a lower S_Y (shrinkage-strain) as compared to that of dual-curing systems.

Photoelastic analysis

Photoelastic analysis is a visual measurement based on the property of some transparent materials to exhibit interference fringes when stressed in a polarized light field.[54] Photoelastic analysis was used to analyze shrinkage stress in several investigations. The internal stresses of the photoelastic material are transformed by visible light, which presents the location and the magnitude of the stress. The stress produced on inlays, onlays, posts, crowns, abutments, etc., has been analyzed through this method in the literature.[53] In this technique, a special stress-sensitive embedding material was used and the tension lines generated in the embedding material by the shrinkage of resin-based material bonded to it can be evaluated.[55] The photoelastic method is simpler than some other techniques and is suitable for evaluating the shrinkage stress. The specimens used in the photoelastic test are round and uniform permitting the variable of irregular stress distribution to be omitted.[53]

Lopes *et al.*[53] evaluated the PS generated by a Silorane-based composite by means of photoelasticity. Visual representation of the stress considering the isochromatic ring of first order was measured. In their study, two conventional composites with adhesive Single Bond 2 and also Silorane-based composite Filtek P-90 with primer and adhesive Filtek P-90 from one brand (3M ESPE) were assessed. The Silorane-based composite showed similar shrinkage stress to that of the traditional composites. However, its adhesive system displayed higher shrinkage stress than those by the etch-and-rinse 2-step adhesive. They concluded no benefit from the reduced shrinkage of the Silorane-based materials with regard to the generated stresses at the substrate interface.

Oliveira *et al.*[54] assessed stress induced in three experimental composites (with CQ/amine, PPD/amine, or both as photoinitiators) and two commercial composites (Silorane, Z250) using photoelastic analysis immediately after photo-polymerization, 24 h and 7 days later. They found that immediately after photo-polymerization, the stresses created by Silorane were similar to that of Z250. However, Z250 showed

higher stresses than that of Silorane composite 24 h and 7 days after photopolymerization. No significant difference between the experimental composites was found. All photoinitiators provided comparable stresses during polymerization.

Rullmann *et al.*[55] examined the polymerization shrinkage stress of new composites (Venus Diamond and SDR) and an experimental composite (Ormocer) in comparison to the low-shrinkage resin composites (Filtek Silorane, Filtek Supreme XT, and Clearfil Majesty Posterior) 4 min and 24 h after light exposure. The shrinkage stress was evaluated by the calculation of the diameter of the first-order isochromatic rings. Higher PS values were obtained after 24 h. Except Venus Diamond/SDR, all polymerization shrinkage data were statistically significant. Venus Diamond and SDR composites had comparable shrinkage stresses with that of Filtek Silorane.

Finite element analysis

Finite element analysis (FEA) provides a framework to combine material properties, with geometry and boundary conditions. Utilizing computational methods, investigation of dental materials behavior is possible. This modeling approach can also leads to the design of experimental studies.[48]

3D finite element simulation of the polymerization process is one of the methods to estimate the magnitudes of prestresses at the interfaces and within the materials. These simulations is also able to analyze which areas in a given material such as enamel, dentin, resin composite, and interfaces are most prone to failure and how restorations should be made to minimize prestresses areas.[49]

Versluis *et al.*[48] calculated and validated the shrinkage stresses associated with the reported tooth deformations. It was found that the shrinkage stresses were dependent on the size and configuration of the restorations. The tooth's resistance against polymerization shrinkage decreased with loss of dental hard tissue. It was concluded that removal of dental hard tissue might decrease the stiffness of the tooth. Decreasing stiffness of the supporting tooth structure could decrease the residual stresses at the interface between the restoration and tooth structure.

Barink *et al.*[49] simulated the polymerization process in a detailed 3D finite element model of an upper premolar with a cusp-coverage restoration. They found that the stresses increased rapidly

during polymerization while diminished again in the postpolymerization phase. At the interface of tooth–composite, the tensile stresses relaxed to a higher degree than the shear stresses. This finding that the stresses significantly decreased during the postpolymerization period may suggest that the mechanical loading should be limited throughout the first few hours after placing restoration.

Bicalho *et al.*[51] evaluated three commercially resin composites with three filling technique (bulk, 2.0 mm increments, and 1.0 mm increments) for restoring a molar were simulated in a 2D FEA. Postgel shrinkage was measured using the strain gauge technique to validate the results of FEA. The results showed that with increasing the number of increments and high postgel shrinkage composites and/or elastic modulus values, the higher stresses in the remaining tooth structure and interface would be expected. Further, increments of approximately 2 mm offered the best condition compared to the bulk or 1 mm increment placements.

Rodrigues *et al.*[52] employed FEA for rectangular Class I cavity wall models and investigated the correlation of interfacial shrinkage stress at the adjoining cavity walls and C-factor. They found that the increase of the C-factor did not lead to enhancement of the calculated stress peaks in cavity walls.

Chen *et al.*[50] combined DIC and FEA to model the shrinkage behavior of a microhybrid composite under different light-curing regimens. In their study, composite fillings were placed under both unbonded and bonded conditions. They found that the shrinkage centers in the bonded cases were detected closer to the cavity floor than those in the unbonded situations and were less affected by curing regimens. The FEA results revealed that step curing may decrease the tensile stress along the cavity walls and the stress was modulated by the accumulated light energy density.

Crack analysis method

Yamamoto *et al.*[61] introduced a simple way for calculating localized PS based on the crack analysis method. Indentation cracks are created near a cavity in a brittle ceramic material that simulates dental enamel. Soda-lime glass or micaceous glass-ceramics have been used in the literature as the brittle materials.[61,62] The cracks propagated when subjected to tensile stresses, such as those stress due

to polymerization shrinkage of a composite cured in the cavity with good interfacial adhesion. The stress is calculated from the change in dimension of crack lengths and the known as fracture toughness of the brittle material. The PS of resin composite within the ceramic hole was calculated from the crack length and the fracture toughness of the glass-ceramic.[61] This method is also an appropriate approach for studying operating factors such as configuration factors, light irradiation protocols, adhesion, and cavity design.[66] Figure 4 shows a schematic diagram of crack analysis method.

Yamamoto *et al.*[61] assessed three composites (Heliomolar, Herculite XRV, Z100) having different contraction stresses using the crack analysis method. All the resin composites revealed crack propagation and the formation of contraction gaps. The obtained contraction stresses ranged from 4.2 to 7.0 MPa. Correlation between the stress values and the contraction gaps was not found. Another study conducted by Yamamoto *et al.*[62] (2014) showed that stress in the materials tested increased up to 12 h after irradiation, while the calculated stress at 24 h was two times more than the calculated stress at 30 min. The increase in elastic modulus values from 30 min to 24 h was less than the increase in stress values within the same time period.

Yamamoto *et al.*[46] in their other study compared several low-shrinkage composites with a microfill composite (Heliomolar) in terms of PS, polymerization shrinkage, and elastic modulus. PS and polymerization shrinkage were measured at 2 and 10 min after irradiation with crack analysis method and video-imaging device (AcuVol, Bisco), respectively. Among the low-shrinkage composites, two of them revealed significantly reduced PS compared to the conventional composite which has previously reported in *in vitro* tests to generate low stress. Hence, despite many factors involved in measuring PS, it can be concluded that the reduced shrinkage itself does not always produce lower stress.

A summary of PS data in the literature for several commonly used dental resin composites is provided in Table 2.

CONCLUSION

Based on the data in the literature, there are many factors that affect the polymerization kinetic of

Table 2: Polymerization stress data in the literature for the several dental resin composites

| Material	Manufacturer	Method	Polymerization stress in MPa (SD)	Reference
GC Kalore	GC Corporation, Japan	Crack analysis method	2.0 (0.7) 2 min 3.8 (0.5) 10 min 5.8 (0.4) 30 min 7.0 (0.3) 1 h 11.3 (0.7) 12 h 13.5 (1.2) 24 h	[62]
			3.3 (0.8) 2 min 4.7 (1.0) 10 min at 200 µm distance	[46]
Venus Diamond	Heraeus Kulzer GmbH, Hanau, Germany	Crack analysis method Tensilometer	0.7 (0.8) 2 min 2.3 (1.9) 10 min 4.2 (3.0) 30 min 6.5 (2.9) 1 h 11.7 (1.1) 12 h 12.6 (1.0) 24 h	[62]
			0.6 (0.8) 2 min 2.5 (2.1) 10 min at 200 µm distance	[46]
			0.5 (0.15) 40 s with feedback 0.4 (0.1) 40 s without feedback	[64]
Heliomolar	Ivoclar Vivadent, Schaan Liechtenstein	Crack analysis method	4.9 (1.3) one step curing at 570 µm distance 6.4 (0.7) One-step curing/2 min	[61] [66]
Silorane	3M ESPE, St Paul, MN, USA	Photoelastic analysis Tensilometer	3.08 (0.09) 1.31 (0.29) 40 s with feedback 0.65 (0.08) 40 s without feedback	[54] [64]
Filtek Z250	3M ESPE, St Paul, MN, USA	Tensilometer Photoelastic analysis Bioman	1.76 (0.06) 40 s with feedback 0.77 (0.1) 40 s without feedback 3.19 (0.13) 5.47 (0.25)	[64] [54] [60]

SD: Standard Deviation
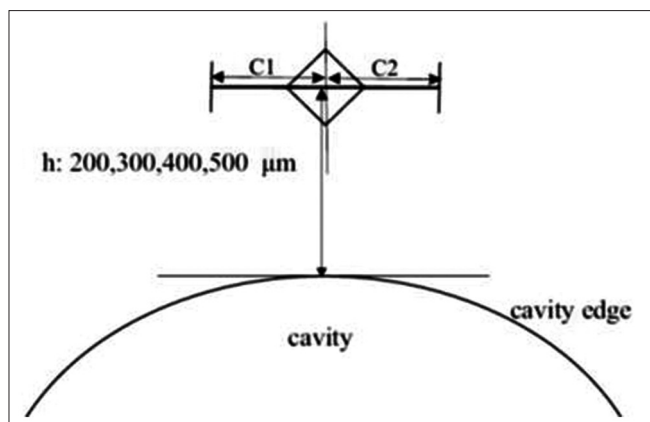
Figure 4: A schematic diagram of crack analysis method: (v) Vickers indentation (square) in glass or ceramic disk. (h) Distances between indentation center and edge of cavity. Lengths of cracks (C1 and C2) will be measured.

dental resin-based composite materials. Various methods for measuring the polymerization shrinkage and stresses of dental resin composites have been introduced, and each method has its own advantages and disadvantages. Regarding the great variations between the reported data, we might better compare the composite materials being evaluated by the same analytical method. However, the search for an ideal and standard apparatus for measuring shrinkage stress and continuous volumetric polymerization shrinkage of resin-based materials in dentistry is still required.

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Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or nonfinancial in this article.

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