

Polymerization shrinkage stress of contemporary dental composites: Comparison of two measurement methods

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The aim of this study was to compare two testing methodologies employed for assessing the polymerization shrinkage stress of dental resin composites. Ten commercial resin composites were investigated (EverX Posterior & Flow; G-aenial Anterior, Posterior, A'CHORD & Universal Injectable; Filtek One Bulk Fill & Universal Restorative; SDR flow+ and Aura Bulk Fill). Photoelastic and contraction forces measurement methods were performed. The slope of the linear trendline and C-factor of specimens were calculated. The shrinkage stress values (range between: 6.4–13.4 MPa) obtained by the photoelastic method were higher for all resin composites than the values obtained by contraction forces measurements (range between 1.2–4.8 MPa). However, there was a strong linear correlation between these methods ($r=0.8$). The use of both investigated methods revealed important information about the shrinkage behavior of the restorative resin composites.

Keywords: Shrinkage stress, Contraction forces, Photoelastic, Resin composite materials

INTRODUCTION

The most common disease in the world is untreated caries of permanent teeth according to the research conducted by the Global Burden of Disease Study (2017)¹. It is worth emphasizing that this disease can be largely prevented, but unfortunately, its prevalence has slightly improved over the last 30 years due to many reasons². In order to rebuild damaged tooth tissues, a number of different dental materials are used, such as glass ionomers, compomers, giomers and composites. Currently, light-cured resin composite materials are the most frequently chosen by dentists.

Despite the many advantages, these dental materials are also not without disadvantages. Under light stimulation, a polymerization reaction is initiated in a resin composite. The monomers bond to each other and a reduction of the intermolecular distances takes place causing the material's volume shrinkage. A tensile stress at the restoration interface is created due to the polymerization shrinkage of the resin composite^{3,4}. The polymerization shrinkage stress may cause debonding of the restoration and lead to secondary caries or tooth fracture⁵. Therefore, in dentistry research on material's polymerization shrinkage and the consequences of this phenomenon is a very important topic⁶. Consequently, methods for evaluating the effects of polymerization in dental materials are essential.

The methods which investigate the effects of the polymerization process in restoration can be divided into two main groups. Firsts are methods for determining shrinkage changes in the material and seconds are techniques for stresses evaluation at the restoration

interface. Several strategies have been presented in the literature in an attempt to measure polymerization shrinkage and shrinkage stress^{7,8}. The most popular methods of shrinkage stress evaluation are collected and briefly described in Table 1⁹⁻¹⁶. In photoelastic method, optical fringes created in specific resins are analyzed to determine stress distribution. Contraction forces measurement methods are currently the most popular methods for measuring shrinkage stresses. These methods use various apparatuses (*e.g.* universal testing machine, Bioman device and controlled-compliance apparatus developed by Sakaguchi *et al.*¹⁷) for measuring contraction force generated by the polymerizing material. Stress distribution can be also simulated by finite element analysis using computer models. However this method requires tooth model and the input of materials data (elastic moduli, Poisson's ratios, and shrinkage strain)¹⁸.

However, the precise measurement method remains a topic of ongoing debate. Thus, the aim of this study was to compare two testing methods, which are used to evaluate polymerization shrinkage stress: the photoelastic method and contraction forces measurements. Linear correlation between investigated methods were analyzed. The following null hypothesis was tested: there would be no significant difference in polymerization shrinkage stress among the tested resin composite materials and methods.

MATERIALS AND METHODS

In this study ten commercial composites were investigated. The compositions and descriptions of the

Table 1 The most widely used methods for evaluating polymerization shrinkage stress

Method	Description	References
Contraction forces measurement	In this method, some apparatus (<i>e.g.</i> universal testing machine) allows to measure the force generated by the polymerizing material placed in a special testing configuration.	9,10)
Photoelastic analysis	In this technique, a special stress-sensitive material (<i>e.g.</i> epoxy plate) is used. Polymerization process of dental material generates stress in plate and tension patterns can be observed and analyzed. Shrinkage stress can be calculated from obtained data.	11–13)
Finite element analysis	This method uses specialized computer software for simulation and analysis of shrinkage stresses. This software is based on mathematical modeling and analyzed phenomena is evaluate using system of algebraic equations.	14–16)

materials are presented in Table 2.

Photoelastic investigation of shrinkage stress

Photoelastic method was described previously¹⁹⁾. Briefly, transparent and photosensitive plates (4 mm thickness) made of epoxy resin (Epidian 53, Organika-Sarzyna, Nowa Sarzyna, Poland) were used in photoelastic evaluation of shrinkage stress. The orifices (3 mm in diameter) were prepared in resin plates and then sandblasted with a 50- μ m grain corundum Cobra (Renfert, Hilzingen, Germany). Bonding system (Single Bond Universal, 3M ESPE, St. Paul, MN, USA) was applied and cured 10 s with the LED curing light (Elipar S10, 3M ESPE). The orifices were filled with tested material in one increment and cure from both sites for 20 s (Elipar S10, 3M ESPE). Five specimens were prepared for each material.

Twenty-four hours after the start of photopolymerization photoelastic images were visualized in circular transmission polariscope FL200 (Gunt, Hamburg, Germany) and then registered by digital camera (Canon EOS 5D Mark II, Canon, Tokyo, Japan), both in parallel and perpendicular orientation of filter polarization planes (Fig. 1).

Shrinkage stress was calculated based on quantity and diameter of the isochromatic fringes from photoelastic images analysis. Stress investigations through photoelasticity are based on modified Timoshenko equations²⁰⁾. The principal equation to calculate shrinkage stress:

$$\sigma_r - \sigma_\theta = \kappa_s \cdot m \quad (\text{Equation 1})$$

Where: $\sigma_r - \sigma_\theta$; principal stress difference (radial and circumferential stresses), κ_s ; photoelastic model constant for a specific plate thickness, m ; number of isochromes.

Configuration factor (C-factor) of specimens was calculated as the ratio of bonded composite area ($2\pi rh$) to unbonded area ($2\pi r^2$), where r is the radius of the orifices in the resin plate (1.5 mm) and h is the height of composite cylindrical specimens (4 mm).

Shrinkage stress evaluation by contraction forces measurements

Glass fiber reinforced composite (FRC) rods with 4 mm diameter and 4 cm length had one of their flat surfaces ground with 320 grit silicon carbide sandpaper. Two FRC rods were attached tightly to a universal testing machine with capacity of load-cell 2,500 N (model LRX, Lloyd Instruments, Fareham, England). The height between the rod was set at 1.5 mm. Composite was applied between the glass surfaces without an additional surface treatment of FRC rods. Two light units (Elipar S10, 3M ESPE) were used simultaneously for 20 s with the tips in close contact with the material specimen from both sides (Fig. 2).

Contraction forces were monitored for 5 min at the room temperature (22°C) from start of curing. The maximum forces were converted to stress according to Equation 2 by dividing by the cross-section area of the FRC rod surface. Five specimens were tested for each composite.

$$\delta = \frac{F}{A} \quad (\text{Equation 2})$$

Where: F ; maximum force obtained during measurements [N], A ; cross-section area of the FRC rod surface [mm^2].

In addition, the slope of the linear trendline was determined, adjusted to the initial fragment (the curing process) of the polymerization shrinkage-stress curve. Initial points from the graphs were selected that showed a straight-line Stress-Time relationship. Next a scatter chart of data points was created and a trend line with their equation was added. Linear fitting ($y=mx+b$) of the data points provides the slope (m) in MPa/s.

C-factor of specimens was calculated as the ratio of bonded composite area ($2\pi r^2$) to unbonded area ($2\pi rh$), where r is the radius of the glass fiber reinforced composite rod (2 mm) and h is the height of composite cylindrical specimens (1.5 mm).

The obtained data were processed using the Statistica 13.1 computer program (Statsoft, Cracow, Poland). The Shapiro-Wilk test was used to verify the normal

Table 2 Composition of investigated resin composite materials based on manufacturer's information

Material	Manufacturer	Composition	Special characteristic
EverX Posterior	GC, Tokyo, Japan	Bis-GMA, PMMA, TEGDMA, millimeter scale glass fiber filler, barium glass (filler content: 76 wt%, 57 vol%)	fiber filler
EverX Flow	GC	Bis-EMA, TEGDMA, UDMA, micrometer scale glass fiber filler, barium glass (filler content: 70 wt%, 46 vol%)	fiber filler
G-ænial Universal Injactable	GC	Bis-EMA, dimethacrylate co-monomers, UDMA, silica, barium glass, (filler content: 69 wt%/45 vol%)	highly thixotropic viscosity
G-ænial Posterior	GC	UDMA, dimethacrylate co-monomers (Bis-GMA free), Fluoroaluminosilicate glass, fumed silica, pre-polymerized fillers (silica, strontium and lanthanoid fluoride), (filler content: 77 wt%, 65 vol%)	including two types of pre-polymerised fillers (PPF), light scattering ability
G-ænial Anterior	GC	UDMA, dimethacrylate co-monomers (Bis-GMA free), silica, fumed silica, pre-polymerized fillers (silica, strontium and lanthanoid fluoride) (filler content: 73 wt%, 64 vol%)	including two types of pre-polymerised fillers (PPF), light scattering ability
G-ænial A'CHORD	GC	Bis-EMA, UDMA, TEGDMA, dimethacrylate co-monomers, silanized barium glass (300 nm), fumed silica (16 nm), silicate glass (filler content: 41 vol%)	Full-Coverage Silane coating (FSC) and high performance pulverized Cerasmart (HPC)
Filtek one Bulk Fill Restorative	3M ESPE, St. Paul, MN, USA	AFM (dynamic stress-relieving monomer), AUDMA, UDMA and DDDMA, silane treated ceramic, silane treated silica, silane treated zirconia, (filler content: 76.5 wt%/58.5 vol%)	addition-fragmentation monomers (AFM) relieve stress during polymerization, and aromatic urethane dimethacrylate (AUDMA), 3M's TRUE nanotechnology
Filtek Universal Restorative	3M ESPE	AFM (dynamic stress-relieving monomer), AUDMA, DUDMA, and DDDMA, silane treated ceramic, silane treated silica, silane treated zirconia, (filler content: 76.5 wt%/58.5 vol%)	Innovative AFM and AUDMA monomers help reduce shrinkage and relieve stress, enabling placement up to 5mm, 3M's TRUE nanotechnology
SDR flow+	Dentsply, Milford, DE, USA	proprietary modified urethane dimethacrylate resin, TEGDMA, dimethacrylate, trimethacrylate resin; silanated barium-alumino-fluoro-borosilicate glass; silanated strontium alumino-fluoro-silicate glass; surface treated fumed silicas; ytterbium fluoride; (filler content: 70.5 wt%/47.4 vol%)	low viscosity material, SDR technology
Aura Bulk Fill	SDI, Bayswater, Australia	DU-DMA, TEGDMA, Bis-EMA, Barium Alumino-borosilicate glass, silica (filler content: 81 wt%/65 vol%)	—

wt%: percentage by weight, vol%: percentage by volume, Bis-GMA: bisphenol A glycol dimethacrylate, PMMA: polymethyl methacrylate, TEGDMA: triethyleneglycol dimethacrylate, Bis-EMA: bisphenol A ethoxylated dimethacrylate, UDMA: urethane dimethacrylate, AFM: addition-fragmentation monomer, AUDMA: aromatic urethane dimethacrylate, DDDMA: 1,12-dodecane dimethacrylate, DUDMA: Diurethane dimethacrylate

distribution of the data. Homogeneity of variance was analyzed with Levene's test. On the basis of the obtained results, the Kruskal-Wallis test with multiple comparisons of mean ranks. The assumed level of

significance was $p=0.05$. Additionally, the results from two test methods were compared using the Spearman's rank-order correlation.

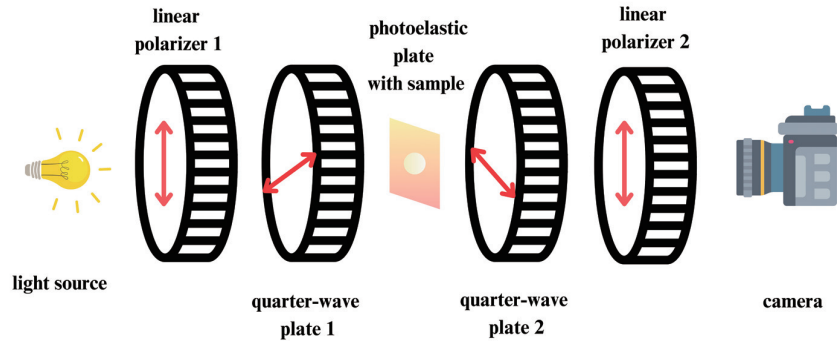


Fig. 1 Schematic of the photoelastic method of shrinkage stress assessment.

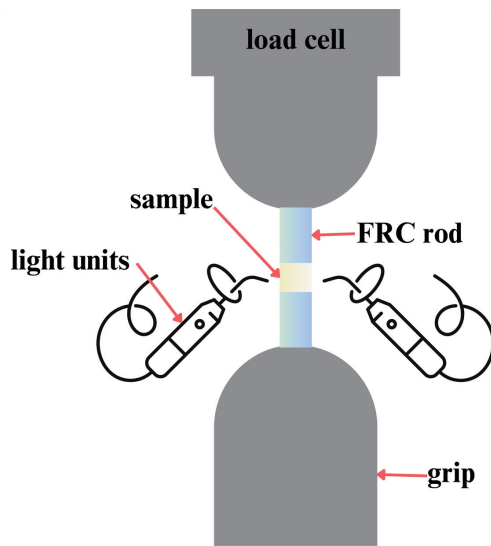


Fig. 2 Schematic of shrinkage stress evaluation by contraction forces measurements.

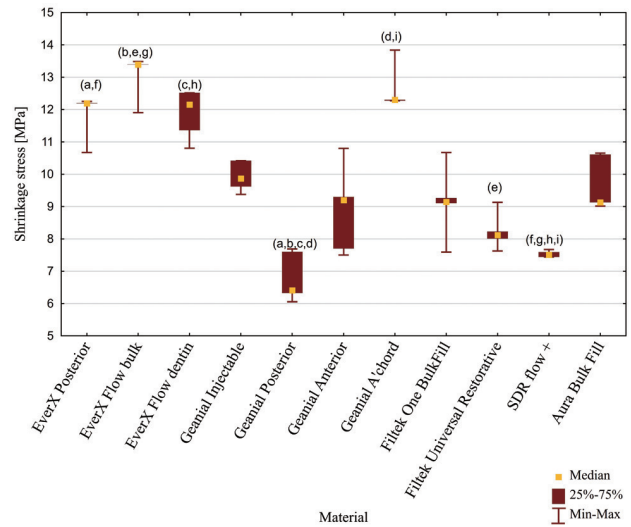


Fig. 3 Shrinkage stress of investigated resin composites using photoelastic method.

The results with the same assigned letter show a statistical difference at the level of $p \leq 0.05$. The results without the assigned letter do not show a statistical difference.

RESULTS

Photoelastic method

The results of shrinkage stress determined by photoelastic method are presented on Fig. 3. The highest shrinkage stress was observed for EverX Flow bulk (13.4 MPa) while the lowest for G-ænial Posterior (6.4 MPa).

Statistically significant differences in shrinkage stress (photoelastic method) were found for the following comparisons (multiple comparisons of mean ranks for all groups):

- G-ænial Posterior *vs.* EverX Posterior (p -value=0.02820); (b) EverX Flow bulk (p -value=0.00077); (c) EverX Flow dentin (p -value=0.01433); (d) G-ænial A'CHORD (p -value=0.00157).
- (e) Filtek Universal Restorative *vs.* (e) EverX Flow bulk (p -value=0.04514).
- (f-i) SDR flow+ *vs.* (f) EverX Posterior (p -value=0.04678); (g) EverX Flow bulk (p -value=0.00144); (h) EverX Flow dentin

- (p -value=0.02433); (i) G-ænial A'CHORD (p -value=0.00286).

Contraction forces measurements method

The results of shrinkage stress determined by contraction forces measurements are presented on Fig. 4. The highest values of approximately 4.5 MPa were achieved by EverX materials (Posterior, Flow bulk, Flow dentin). The SDR material achieved the lowest shrinkage stress values; 1.2 MPa.

Statistically significant differences in shrinkage stress (contraction forces measurements) were found for the following comparisons (multiple comparisons of mean ranks for all groups):

- (a-c) G-ænial Anterior *vs.* (a) EverX Posterior (p -value=0.02433); (b) EverX Flow bulk (p -value=0.02433); (c) EverX Flow dentin (p -value=0.00766)

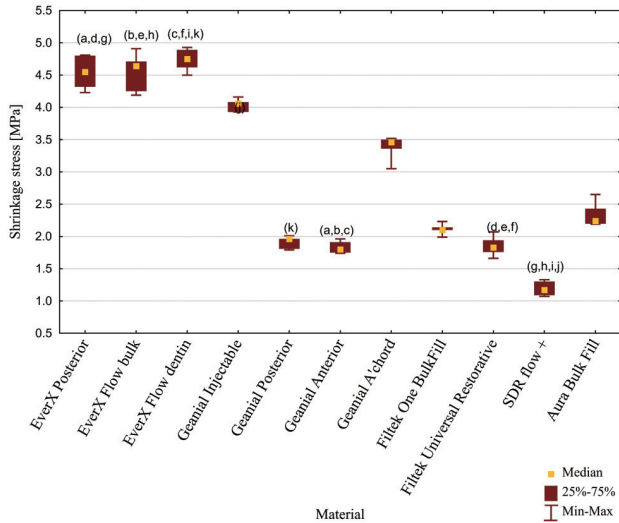


Fig. 4 Maximum shrinkage stress of investigated composites using contraction forces measurements. The results with the same assigned letter show a statistical difference at the level of $p \leq 0.05$. The results without the assigned letter do not show a statistical difference.

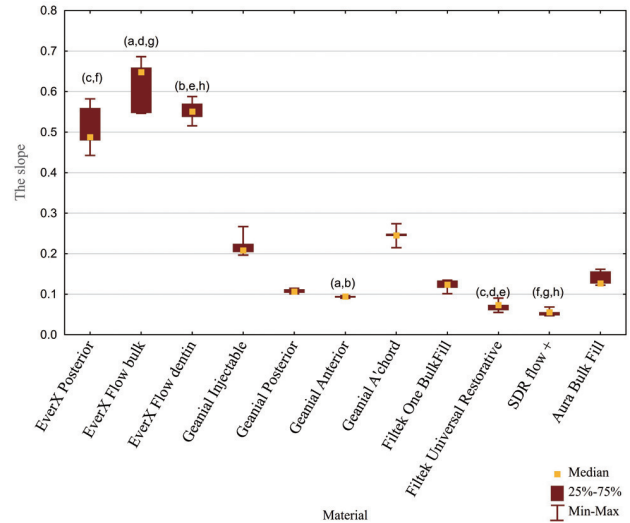


Fig. 6 The slope (absolute values) of the linear trendline, which was determined for the initial measurement points of the polymerization shrinkage-stress curve (immediately after the start of curing). The results with the same assigned letter show a statistical difference at the level of $p \leq 0.05$. The results without the assigned letter do not show a statistical difference.

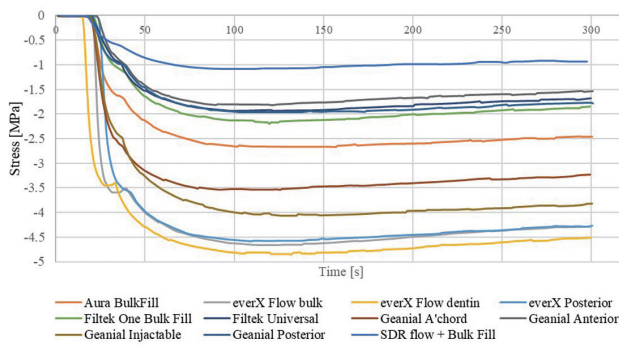


Fig. 5 Polymerization shrinkage-stress curve of tested materials.

- (d–f) Filtek Universal Restorative *vs.* (d) EverX Posterior (p -value=0.04056); (e) EverX Flow bulk (p -value=0.04056); (f) EverX Flow dentin (p -value=0.01327)
- (g–j) SDR flow+ *vs.* (g) EverX Posterior (p -value=0,00077); (h) EverX Flow bulk (p -value=0.00077); (i) EverX Flow dentin (p -value=0,00019); (j) Geanial Injectable (p -value=0.03035)
- (k) Geanial Posterior *vs.* (k) EverX Flow dentin (p -value=0.03265).

The polymerization shrinkage-stress curve of tested materials were showed on Fig. 5.

Figure 6 shows the slope (absolute values) of the linear trendline determined for the initial measurement points of the polymerization shrinkage-stress curve. Statistically significant differences in slope values were found for the following comparisons (multiple

comparisons of mean ranks for all groups):

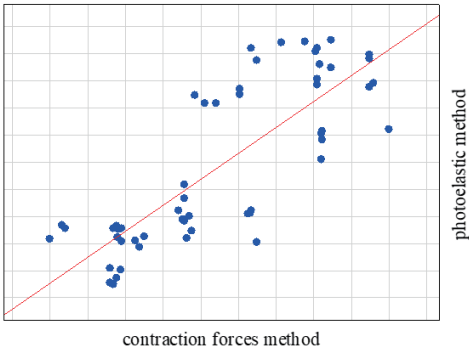
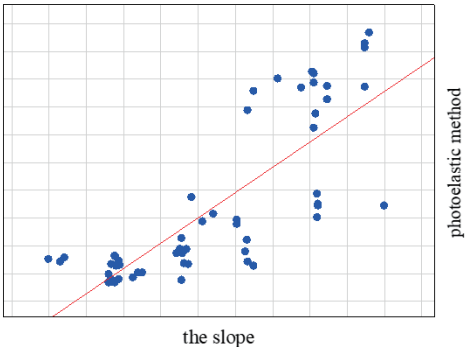
- (a, b) Geanial Anterior *vs.* (a) EverX Flow bulk (p -value=0.00898); (b) EverX Flow dentin (p -value=0.03035)
- (c–e) Filtek Universal Restorative *vs.* (c) EverX Posterior (p -value=0.01051); (d) EverX Flow bulk (p -value=0.00093); (e) EverX Flow dentin (p -value=0.00368)
- (f–h) SDR flow+ *vs.* (f) EverX Posterior (p -value=0.00242); (g) EverX Flow bulk (p -value=0.00018); (h) EverX Flow dentin (p -value=0.00077).

Results of correlation coefficient and evaluation of correlation strength between analyzed research methods were presented at Table 3. There is a strong correlation (correlation coefficient 0.8, $p < 0.001$) between photoelastic method and contraction forces measurements method.

DISCUSSION

Due to the polymerization processes of resin composites, avoiding shrinkage is impossible. This topic is still widely researched due to the development of new resin composite materials used to reconstruct lost tooth tissues. The exact values and distribution of shrinkage stresses in a dental cavity cannot be measured directly, only there are the measurements of the effect of the polymerization shrinkage on surrounding structures, which are very important to understand this phenomenon. It is essential to evaluate material's behavior and compare different testing methods with an indication of the limitations

Table 3 Results of Spearman correlation coefficient between analyzed research methods

Analyzed methods	Scatter plot	Spearman correlation coefficient	<i>p</i> -value
photoelastic method and contraction forces measurements		0.793671	0.000000
photoelastic method and the slope		0.824612	0.000000

and assumptions of each of them.

The values of shrinkage stress obtained from the two selected methods (photoelastic method and contraction forces measurements) for the tested materials are different. The values obtained by the photoelastic method are higher for all materials than the values obtained by contraction forces measurements. Thus, the null hypotheses were rejected. However, there is a strong linear correlation between these methods (Table 2). There are many factors, besides material composition, that influence the shrinkage stresses. These include the C-factor, the reconstruction method, and the curing method²¹).

C-factor is a cavity qualification method. In this research the C-factor in photoelastic method was 8/3 and in contraction forces measurement 4/3. The more un-bonded surface area, the lower the stress value²²). If we divide the values of both research methods by the appropriate C-factor, these values are more similar to each other. However, when considering shrinkage stresses, the directions in which the stresses are tested, should be also used. In this study, methods investigated the stress generated in different directions. The photoelastic method allows to determine the values of circumferential and radial stresses generated on the border of the restoration (imitated by the orifices in the resin plate), while the second tested method determines the linear stresses. It is also worth mentioning that some

authors point out that C-factor is not so important, but the volume of the material that is polymerized^{23,24}). This explains why the stresses obtained by the photoelastic method are higher than the values obtained by the second test method. From a clinical point of view, cavity preparation is a very crucial element. Therefore, the use of free walls, in which there may be some compensation for stresses resulting from polymerization, is important.

The shrinkage stresses will depend on the composition of the material. Conventional layered resin composites, bulk-fill (designed to be placed in 4 mm or greater increments) resin composites and materials with fibrous filler were used in this research. The obtained results also allow to notice the differences between the individual materials. The highest shrinkage stress values were observed for materials with short fiber filler. Our results are consistent with those of other authors^{25,26}). On contrary, other research indicate that the fibers should reduce shrinkage stress^{3,27}).

Along the length of the filler fibers the resin is not able to shrink so in this direction original dimensions is retained. Although, the resin matrix tried to shrink in other directions. The fibrous filler may cause additional obstruct to the flow of the material near the interface during the curing process which may cause a local physical state with high stress concentration. Additionally, this materials have high modulus of elasticity and the slope value. This indicate that fiber composite's capacity for

viscous flow or its compliance decreases rapidly during polymerization (the stress increases dramatically, Fig. 4). There were no statistically significant differences between micro scale and millimeter scale fibers (EverX flow *vs.* EverX posterior), however, it is worth noting that EverX posterior (millimeter scale) had a higher filler loading than the flow material. As far as the authors are aware, there are no existing studies in the literature addressing the impact of fiber length on the shrinkage stresses of experimental dental composites. Thus, there is a clear need for research in this area. Which could suggest that EverX flow with higher filler content could achieve lower stress values. Recent studies in literature reported that the use of short fiber composites (EverX Posterior and EverX Flow) in restoring large cavities showed less prone to polymerization shrinkage-related crack formation than layered conventional resin composites^{28,29}. It seems there is lack of direct link between shrinkage stress values and clinically relevant effect like enamel crack formation²⁸, marginal microleakage³⁰, and cuspal deflection³¹.

To reduce polymerization shrinkage and minimize the stress of resin composite materials different approaches have been proposed: modification of curing process placement techniques, and new composite formulations^{32,33}.

An example of a commercially used attempt to reduce shrinkage stress is Stress Decreasing Resin (SDRTM) technology. The SDR flow+ material showed similar stresses compared to the tested materials that have a high filler content and lower stress in comparison with materials with a similar filler content (G-ænial Inj. and G-ænial A'CHORD). This is consistent with the others results^{34,35}. According to the manufacturer, this product contain novel UDMA-based monomer with high molecular weight (849 g/mol) and a polymerization modulator. Polymerization modulator is a photoactive group chemically embedded with urethane monomer, which interact with camphorquinon and controlling polymerization kinetics³⁶⁻³⁸.

In Filtek one Bulk Fill Restorative and Filtek Universal Restorative there are two innovative monomers which are claimed to reduce shrinkages stress. In the composition of these two materials there is a high-molecular-weight aromatic urethane dimethacrylate (AUDMA). The use of high molecular weight monomers causes decreases in the number of reactive groups in the resin, which reduce volumetric shrinkage. In addition, 3M uses novel approach to reduce stress generated during polymerization process —covalent adaptable networks. Briefly, network contain covalent bonds that are able to dynamically break, relax and rearrange to form new bonds under specific stimuli. During this process the stress that has been built up during the formation of the network is released³⁹. Filtek one Bulk Fill Restorative and Filtek Universal Restorative use an addition-fragmentation-chain transfer monomer (AFM) for this purpose⁴⁰. These materials had similar shrinkage stress values, and they were comparable to other materials containing no fiber filler.

Comparing monomers used in dental materials, TEGDMA, which has low molecular weight and viscosity, can produce high shrinkage stresses. This effect is due to the increased reactivity of this monomer, which causes high conversion and therefore high shrinkage. This is confirmed by the obtained results³³. EverX Posterior, EverX Flow, G-ænial A'CHORD and Aura Bulk Fill materials are characterized by higher shrinkage stress values compared to materials that do not contain this monomer.

The organic to inorganic phase ratio, type of monomers and fillers are factors that determine the shrinkage and shrinkage stress of dental resin composites. According to Kleverlaan and Feilzer⁴¹ filler amount and monomers system strongly influence on volumetric shrinkage and elastic modulus and there is reverse dependence tensile modulus-shrinkage. Similar results are visible in different paper⁴². But increase amount of filler causes higher elastic moduli in bending and may results in higher contraction stress^{43,44}.

In our study shrinkage stress decrease with increase filler content. The two materials SDR+ and EverX posterior are exceptions. This may be due to the innovative composition of SDR flow+ and in the case of EverX posterior it contains a specific fiber filler

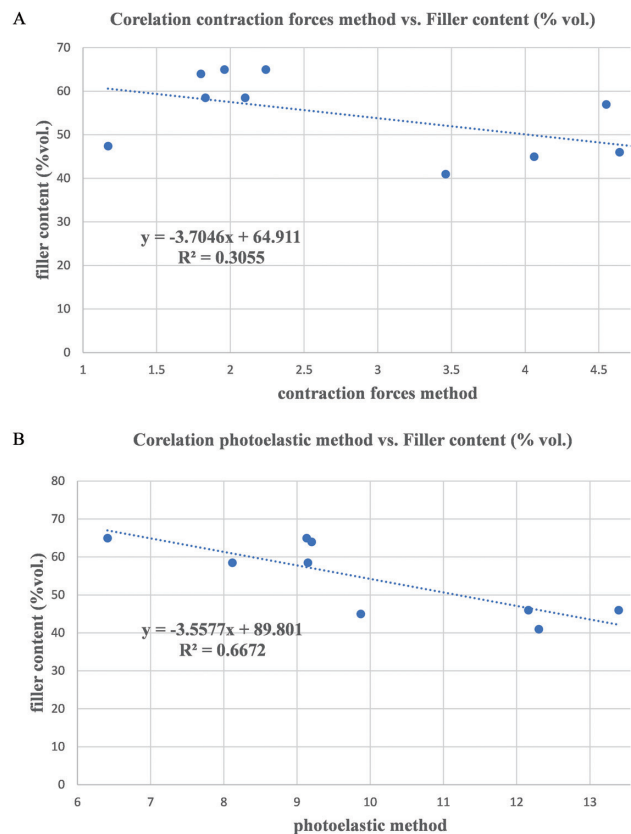


Fig. 7 Relationship between volumetric filler content and shrinkage stress investigated with: A) photoelastic method; B) contraction forces measurements.

of relatively large size compared to other materials. Excluding these materials, the strong correlation between the filler content (vol%) given by manufactures information and the shrinkage stress (elasto-optic method $r^2=0.6672$, contraction forces measurements $r^2=0.7462$) has been found (Figs. 7a, b).

It should be emphasized that shrinkage stresses cannot be determined in clinical conditions. Our methods have some limitations (rigidity of the system), inadequate imitation of cavity (regular, cylindrical samples), however, compared to the finite element methods, photoelastic method seems to be more adequate test method for contraction stress measurements because simulates cavity preparation (sandblasting, adding bond system). However both used methods are easy and reproducible, additionally, the correlation between the results indicates that despite the limitations, dental materials can be successfully characterized. In addition, it would be worth comparing how other frequently tested properties such as shrinkage (volumetric, linear), modulus of elasticity are related to each other.

CONCLUSIONS

The use of both investigated methods revealed important information about the shrinkage behavior of the restorative resin composites.

1. There is a strong correlation between the two studied research methods.
2. The filler amount influence on contraction stress. Lower amount of filler then higher contraction stress however some specific composite composition (or degree of conversion) may affect this correlation.

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