Pre-heating of high-viscosity bulk-fill resin composites: Effects on shrinkage force and monomer conversion

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ABSTRACT

Objectives: To investigate the influence of pre-heating of high-viscosity bulk-fill composite materials on their degree of conversion and shrinkage force formation.
Methods: Four bulk-fill composite materials (Tetric EvoCeram Bulk Fill—TECBF, x-tra fil—XF, QuixFil—QF, SonicFill—SF) and one conventional nano-hybrid resin composite (Tetric EvoCeram—TEC) were used. The test materials were either kept at room temperature or pre-heated to 68 °C by means of a commercial heating device, before being photoactivated with a LED curing unit for 20 s at 1170 mW/cm². Shrinkage forces ($n = 5$) of 1.5-mm-thick specimens were recorded in real-time for 15 min inside a temperature-controlled chamber at 25 °C (simulating intraoral temperature after rubber dam application) with a custom-made stress analyzer. Degree of conversion ($n = 5$) was determined at the bottom of equally thick (1.5 mm) specimens using Fourier transform infrared spectroscopy. Data were analyzed with Student's t-test, ANOVA and Tukey's HSD post-hoc test ($\alpha = 0.05$).
Results: Composite pre-heating significantly increased the degree of conversion of TECBF, but had no effect on monomer conversion of the other materials investigated. For each of the test materials, pre-heated composite generated significantly lower shrinkage forces than room-temperature composite. At both temperature levels, TECBF created the significantly highest shrinkage forces, and QF caused significantly higher shrinkage forces than both XF and TEC.
Conclusions: Both the composite material and the pre-cure temperature affect shrinkage force formation. Pre-heating of bulk-fill and conventional restorative composites prior to photoactivation decreases polymerization-induced shrinkage forces without compromising the degree of conversion.
Clinical significance: Composite pre-heating significantly reduces shrinkage force formation of high-viscosity bulk-fill and conventional resin composites, while maintaining or increasing the degree of monomer conversion, dependent upon the specific composite material used.

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1. Introduction

Increasing the temperature of uncured restorative resin composites has gained popularity among dental practitioners as a way to improve material handling characteristics during placement in a cavity preparation. Pre-heating composites prior to photoactivation generally decreases their viscosity [1], which has been shown to enhance marginal adaptation [2] and reduce microleakage [3] due to improved wetting of cavity walls. Furthermore, increased polymerization temperature enhances both radical and monomer mobility resulting in higher overall conversion [4–7], which in turn may promote improved physical and mechanical properties of pre-heated composites, such as higher surface hardness, and enhanced flexural and diametral tensile strength [8,9].

However, the higher double bond conversion of pre-heated composites is also accompanied by increased volumetric shrinkage [10,11], which might lead to greater shrinkage stress development during polymerization. Shrinkage stress in composite restorations is the result of polymerization contraction taking place under confinement, due to bonding to cavity walls, and has been implicated as a causative factor for a series of clinical complications including interfacial debonding, post-operative sensitivity, cuspal deflection, and enamel fracture [12,13]. Yet, shrinkage stress is not only a function of the composite's volumetric shrinkage, and thus of its actual dimensional change during polymer network

formation, but is also determined by the material's time-dependent visco-elastic behavior, characterized by its flow capacity in the early stages of the curing reaction and by the elastic modulus acquired during polymerization [14,15]. Therefore, theoretically, the use of pre-heated composite may not increase polymerization-induced shrinkage forces, if the reduction in viscosity due to heating would allow for increased viscous flow and polymer chain relaxation, and thus increased stress relief compared to room-temperature composite, compensating the effect of the higher volume contraction. However, experimental data on the impact of composite pre-heating on shrinkage stress development is scarce. A previous study revealed higher stress values at increased composite temperatures, but only tested one single brand of composite material [16]. Since reaction kinetics and factors such as thermal conductivity and specific heat capacity differ depending on material composition [17,18], the effect of pre-heating on shrinkage force formation might vary between different composite materials.

Recently, a new category of resin-based composite materials, so-called 'bulk-fill' resin composites, has been introduced to the market in an attempt to simplify and expedite the restoration process. According to the manufacturers, these materials can be adequately photopolymerized in thick layers up to 4 or even 5 mm, which has been confirmed for the majority of bulk-fill composites in studies using infrared spectroscopy [13,19,20] and/or microhardness depth profiles [19–21], and been attributed to increased light transmittance of these materials [22]. Owing to differences in rheological properties and application techniques, bulk-fill resin composites are further classified in low-viscosity (flowable) and high-viscosity (sculptable) material types. Pre-heating high-viscosity bulk-fill composites might be an interesting approach to provide a transient viscosity reduction comparable to that of a flowable composite without sacrificing the benefits of superior mechanical properties associated with highly filled resin composites [23]. Any potential gain in material characteristics due to pre-heating should, however, not be achieved at the expense of increased shrinkage stress development. This is especially true for bulk-fill resin composites, given that these materials are generally placed in large volume without incremental layering, resulting in unfavorable configuration factors [12,24]. To date, no information is available in the literature on the effect of increasing pre-cure temperature on shrinkage force formation and double bond conversion of high-viscosity bulk-fill resin composites.

Moreover, studies that have shown optimization in monomer conversion upon pre-heating generally maintained the composite temperature constant during experimentation [4–7]. Clinically, however, heated composite cools rapidly once removed from the pre-heating device and inserted into a tooth preparation [25]. In vivo temperature measurements revealed that when a composite material is pre-heated to 60 °C, the actual composite temperature after placement, at the moment of photoactivation, is reduced to around 36 to 38 °C [26]. Therefore, it is important to evaluate the effect of pre-heating under a non-isothermal condition, where the composite temperature achieved after pre-heating is not stabilized, in order to simulate a clinically realistic scenario.

Based on these considerations, the aim of the present study was to investigate the influence of composite pre-heating on shrinkage force development and monomer conversion of high-viscosity bulk-fill materials and a conventional nano-hybrid resin composite under a clinically relevant non-isothermal condition. The null hypothesis was that pre-heating does not affect polymerization-induced shrinkage forces and double bond conversion of the composite materials.

2. Materials and methods

Four bulk-fill composite materials [Tetric EvoCeram Bulk Fill (Ivoclar Vivadent, Schaan, Liechtenstein), x-tra fil (VOCO, Cuxhaven, Germany), QuixFil (Dentsply DeTrey, Konstanz, Germany), SonicFill (Kerr, Orange, CA, USA)] and one conventional nano-hybrid resin composite [Tetric EvoCeram (Ivoclar Vivadent)] were assessed in this study. Details of the test materials are presented in Table 1. Composite pre-heating was performed using a commercially available heating device (Calset; AdDent, Danbury, CT, USA) preset to 68 °C. A standardized volume of test material (42 mm³) was applied into the center well of the heating device and maintained in place for 5 min with the lightproof device lid on. The 5-min heating time was chosen based on a pilot study which revealed that after 5 min of heating, the resin composite materials attained the preset temperature of the heating device, as measured with a T-type thermocouple (Z2-T-2 M; Labfacility, Hanau, Germany). Room-temperature composite (23 ± 0.5 °C) was used as the control. Photoactivation was performed for 20 s with a LED light-curing unit (Bluephase G2; Ivoclar Vivadent) operated in High-Intensity Mode and equipped with a parallel-walled light guide with a 10-mm diameter light emission window. Output

Table 1
Manufacturers' information about the resin composite materials used in the study.

Material	Composition	Filler size (μm)	Filler content (wt%/vol%)	Shade	Lot no.	Manufacturer
Tetric EvoCeram Bulk Fill	Resin: Bis-GMA, Bis-EMA, UDMA Filler: Barium glass, ytterbium trifluoride, mixed oxide, prepolymer	0.04–3 (mean: 0.55)	81/61	IVA	P73694	Ivoclar Vivadent, Schaan, Liechtenstein
x-tra fil	Resin: Bis-GMA, UDMA, TEGDMA Filler: Barium boron aluminum silicate glass	0.05–10 (mean: 3)	86/70	Universal	1225566	VOCO, Cuxhaven, Germany
QuixFil	Resin: Bis-EMA, UDMA, TEGDMA, TMPTMA Filler: Silanated strontium aluminum sodium fluoride phosphate silicate glass	1–10	86/66	Universal	1109001331	Dentsply DeTrey, Konstanz, Germany
SonicFill	Resin: Bis-GMA, Bis-EMA, TEGDMA Filler: Silanated barium boron aluminum silicate glass, silica	Not indicated	83.5/66	A2	3688724	Kerr, Orange, CA, USA
Tetric EvoCeram	Resin: Bis-GMA, Bis-EMA, UDMA Filler: Barium glass, ytterbium trifluoride, mixed oxide, prepolymer	0.04–3 (mean: 0.55)	76/55	A2	R23586	Ivoclar Vivadent, Schaan, Liechtenstein

Bis-GMA: bisphenol-A-glycidyldimethacrylate; Bis-EMA: ethoxylated bisphenol-A-dimethacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; TMPTMA: trimethylolpropane trimethacrylate.

irradiance of the light source (1170 mW/cm^2) was measured using a calibrated FieldMaxII-TO power meter in combination with a PM2 thermopile sensor (Coherent, Santa Clara, CA, USA), and verified periodically during the experiments. The time between removing the composite material from the heating device and start of light polymerization was 40 s in all tests. Specimen preparation and testing was performed inside a temperature-controlled chamber set at 25 °C.

2.1. Shrinkage force

Measurements of polymerization shrinkage force were performed using a custom-made stress analyzer (Fig. 1) as previously described in the literature [27–29]. Briefly, the upper part of the apparatus consisted of a semi-rigid load cell (PM 11-K; Mettler, Greifensee, Switzerland; instrument compliance: 0.4 μm/N), to which a metal cylinder was screwed. Resin composite (42 mm^3) either at room temperature or pre-heated to 68 °C was applied to the front edge of the cylinder. The material was compressed to a thickness of 1.5 mm, and a surface area of 28 mm^2 at the top and at the bottom of the specimen (corresponding to a ratio of bonded to unbonded surface area, i.e., C-factor, of 2.0), by means of a glass plate attached to the base of the device. To improve adhesion, the surfaces of the metal cylinder and of the glass plate were sandblasted with 50-μm Al$_2$O$_3$ and primed or silanized (Mono-bond Plus; Ivoclar Vivadent). Photoactivation was performed through the glass plate, via a recess in the lower frame, at a standardized distance of 1 mm from the test material. The forces generated during polymerization shrinkage were detected by means of the load cell at a sampling frequency of 5 Hz, and continuously recorded over a period of 15 min from the initiation of photoactivation. Data were transferred real-time to the attached computer (Macintosh IIfx; Apple Computer, Cupertino, CA, USA) via an A/D converter using custom-made software. Five measurements were conducted for each combination of composite material and pre-cure temperature.

2.2. Degree of conversion

For degree of conversion measurements, a Fourier transform infrared spectrometer (Spectrum GX; PerkinElmer, Beaconsfield, UK) was used. Resin composite (42 mm^3) either at room temperature or pre-heated to 68 °C was compressed between two 1-mm-thick glass plates using spacers of 1.5 mm. Similar to the setup for shrinkage force measurements, one of the glass plates was previously sandblasted (50-μm Al$_2$O$_3$) and silanized (Mono-bond Plus; Ivoclar Vivadent). Photoactivation was performed at a standardized distance of 1 mm from the test material by placing the tip of the curing unit in contact with the sandblasted and silanized glass plate covering the top surface of the specimen. Five specimens were prepared for each experimental condition and stored dry and in the dark for 15 min at 25 °C. Following previous studies [30,31], thin chips of composite were removed with a scalpel from the non-irradiated composite surface (bottom surface) and pulverized into a fine powder. The composite powder was mixed with spectroscopically pure potassium bromide (KBr; Merck, Darmstadt, Germany) and pressed into a 10-mm diameter pellet using a hydraulic press (Specac, Orpington, Kent, UK) with a load of 2.5 tons. Composite-KBr pellets were also prepared from the uncured material. The pellets were placed into a holder attachment in the optical compartment of the spectrometer for analysis. Infrared spectra were recorded in transmission mode in the 4000–400 cm^{-1} wave number range, and then converted into the absorbance mode. A total of 20 scans per specimen were measured at a resolution of 4 cm^{-1}. Using a standard baseline technique [32], the absorbance intensities (AI; peak heights) of the aliphatic C=C stretching vibrations at 1638 cm^{-1} and aromatic C . . . C stretching vibrations (internal standard) at 1610 cm^{-1} were determined for both the cured and uncured composites, and the degree of conversion (DC) was calculated according to the following equation:

$$DC\ (\%) = \left[1 - \frac{[AI(1638\ cm^{-1})/AI(1610\ cm^{-1})]_{cured}}{[AI(1638\ cm^{-1})/AI(1610\ cm^{-1})]_{uncured}} \right] \times 100$$
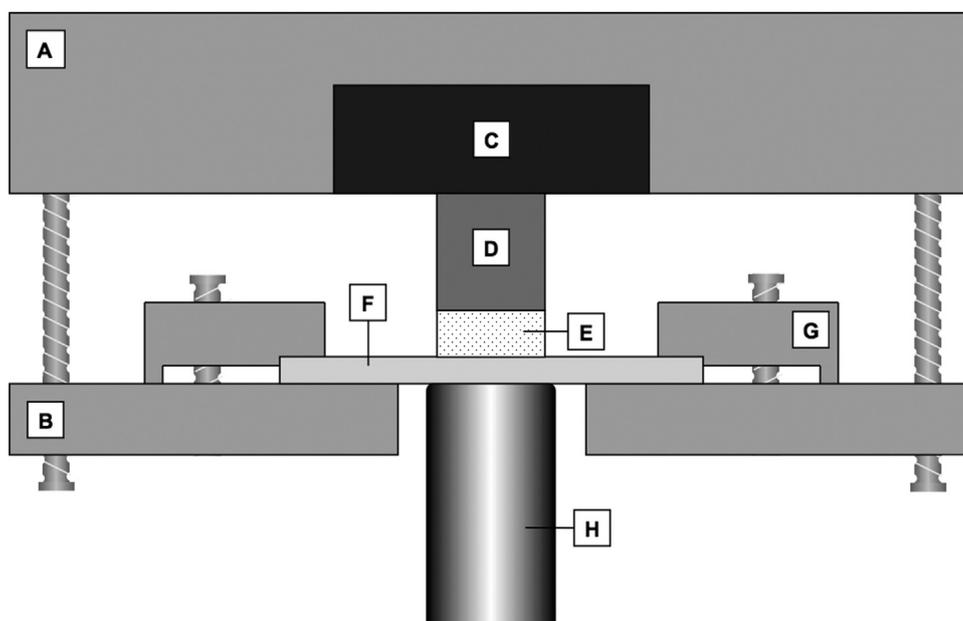

Fig. 1. Diagram of the measuring device for shrinkage force. A: upper part of measuring device; B: lower part of measuring device; C: load cell; D: metal cylinder; E: composite specimen; F: glass plate; G: holder of glass plate; H: curing light tip.

2.3. Statistical analysis

After confirming the validity of the assumption of normality by means of the Kolmogorov–Smirnov and Shapiro–Wilk tests, shrinkage force and degree of conversion values at 15 min after the start of photoactivation were analyzed using two-way analysis of variance (ANOVA) with the two factors being pre-cure temperature and composite material. For each material separately, unpaired two-sample Student's t-tests were performed to identify differences in test parameters between pre-heated and room-temperature resin composite. In addition, one-way ANOVA was applied at each pre-cure temperature for comparison of both shrinkage force and degree of conversion between the composite materials. Tukey's HSD post-hoc test was used to identify pairwise differences. All statistical testing was conducted at a pre-set global significance level of $\alpha = 0.05$ (SPSS Version 20; SPSS, Chicago, IL, USA).

3. Results

Fig. 2 shows the development of shrinkage force for each tested composite material and pre-cure temperature as a function of time. The shrinkage force values obtained at the end of the 15-min observation period are presented in Table 2. Two-way ANOVA revealed significant differences in shrinkage force due to both composite material ($p < 0.001$) and pre-cure temperature ($p < 0.001$), but no significant interaction term ($p = 0.783$). Pre-heated composite generated significantly lower shrinkage forces than room-temperature composite, irrespective of the test material. At both temperature levels, Tetric EvoCeram Bulk Fill created the significantly highest shrinkage forces, and QuixFil caused significantly higher shrinkage forces than both x-tra fil and Tetric EvoCeram.

The results of the degree of conversion measurements at 15 min after the start of photoactivation (endpoint of the observation period for shrinkage force) are displayed in Fig. 3. Two-way ANOVA revealed that the composite material ($p < 0.001$) and the interaction of pre-cure temperature and composite material ($p = 0.001$) significantly affected monomer conversion. Composite pre-heating significantly increased the degree of conversion of Tetric EvoCeram Bulk Fill ($p = 0.006$), but had no effect on monomer conversion of the other materials investigated. SonicFill attained the significantly highest degree of conversion, irrespective of the pre-cure temperature.

4. Discussion

The first part of the null hypothesis, that composite pre-heating would not affect shrinkage force formation, was rejected: increasing composite temperature prior to photoactivation significantly reduced polymerization-induced shrinkage forces of both the bulk-fill and conventional resin composites under investigation. Shrinkage forces are generated when polymerization contraction is obstructed and the developing polymer network loses its ability to re-arrange, due to mobility restrictions, and thereby to macroscopically and microscopically accommodate the reduction in volume by plastic deformation [14,15]. Raising the temperature of resin composites reduces system viscosity and improves molecular mobility as a result of higher thermal energy, facilitating polymer chain segmental movement and postponing the onset of vitrification to a later stage of the polymerization process [33,34]. Given that prior to vitrification, emerging shrinkage stresses can be partially relieved by (pre-gel) viscous flow and polymer chain relaxation [35], the greater initial compliance and prolonged stress relief opportunity of pre-heated composites might explain their lower shrinkage force formation. Moreover, using a strain gauge method, some reduction in post-gel shrinkage was observed when increasing composite pre-cure temperature [36], indicating that the fraction of total volumetric shrinkage taking place after acquisition of measurable network rigidity, and thus being responsible for stress development, is decreased relative to room-temperature composite. In the light of the present results, the previously observed enhanced marginal adaptation and reduced microleakage of pre-heated composites [2,3] might therefore not only be attributed to improved wetting of cavity walls and better handling characteristics [37], but might also be a result of the lower shrinkage forces generated at elevated pre-cure composite temperature. Indeed, a significant correlation
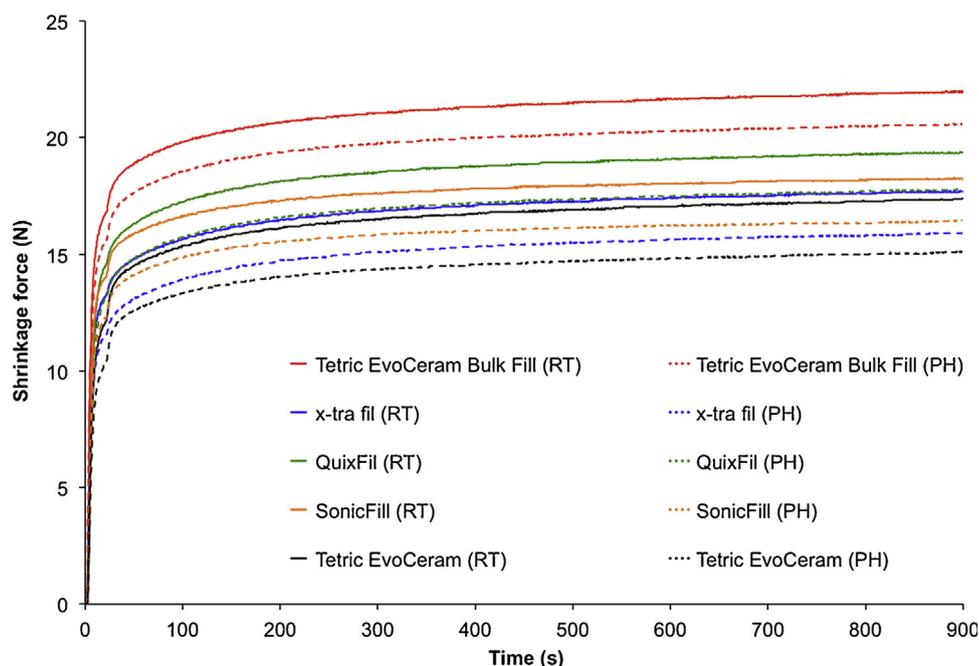

Fig. 2. Mean shrinkage force curves of the pre-heated (PH) and room-temperature (RT) composite materials as a function of time ($n = 5$).

Table 2
Mean (±standard deviation) shrinkage force (in N) of the pre-heated and room-temperature composite materials at 15 min after the start of irradiation ($n = 5$).

Group	Material				
	Tetric EvoCeram Bulk Fill	x-tra fil	QuixFil	SonicFill	Tetric EvoCeram
Room temperature	22.0 (0.4) A, a	17.7 (1.0) A, c	19.4 (0.7) A, b	18.3 (1.4) A, bc	17.4 (0.7) A, c
Pre-heated	20.6 (0.5) B, a	15.9 (0.7) B, c	17.8 (0.8) B, b	16.5 (0.8) B, bc	15.1 (0.8) B, c

Mean values followed by same capital letters in columns, and by same small letters in rows, are not significantly different at the 0.05 level.
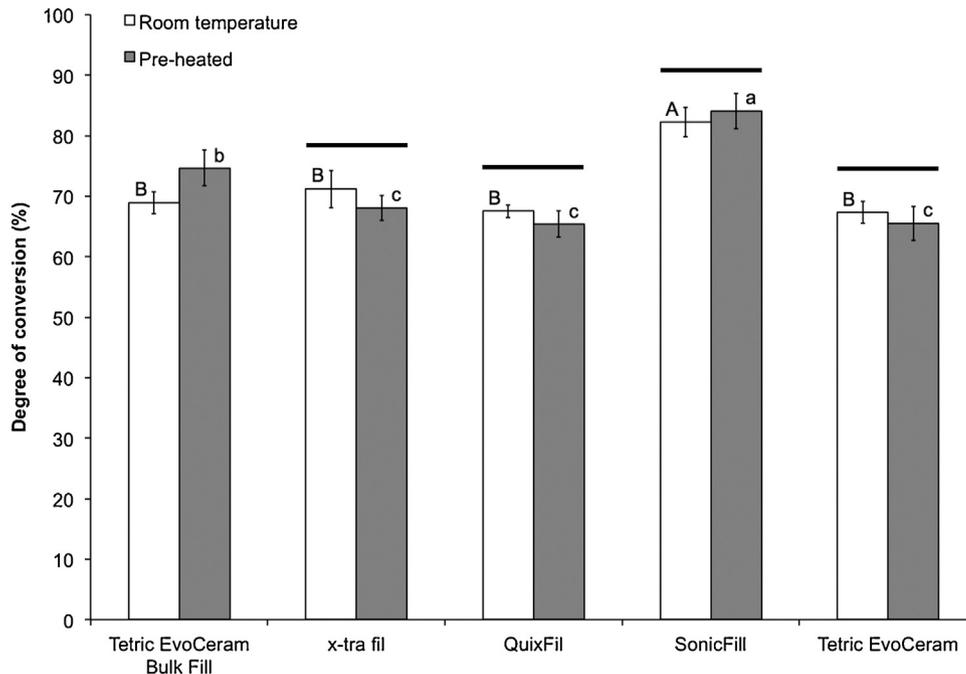
Fig. 3. Mean degree of conversion values and standard deviations (represented by error bars) of the pre-heated and room-temperature composite materials ($n = 5$). Within each composite material, groups linked with a horizontal bar are not significantly different ($p > 0.05$). Within each pre-cure temperature, groups marked with same letters (capital and lower case for room-temperature and pre-heated composites, respectively) are not significantly different ($p > 0.05$).

between polymerization shrinkage stress and interfacial integrity has been demonstrated [38–40], with correlation coefficients being higher when stress data were originated from a more compliant (semi-rigid) testing system compared to a highly rigid (i.e., near-zero compliance) system [38]. In the present investigation, a semi-rigid stress analyzer with a clinically relevant system compliance of 0.4 μm/N was used, which closely mimics compliance conditions in tooth cavities, as previously discussed [27,28].

Dental resins are subjected to post-polymerization shrinkage for up to about 24 h after photoactivation [41]. Nevertheless, real-time shrinkage stress measurements are usually limited to the first 3–30 min of the polymerization reaction, in order to shorten overall measurement duration [12,24,29,30,42]. In the present investigation, shrinkage forces were continuously recorded for 15 min from the start of irradiation, based on previous observations in our laboratory, which revealed that after 15 min, resin composites had reached about 90% of their maximum shrinkage force value after 24 h [27]. Furthermore, the shrinkage force curves of the different experimental groups run parallel to each other already about 5 min after the start of irradiation (Fig. 2). Therefore, although shrinkage force values at the end of the 15-min observation period do not reflect the ultimate values in absolute terms, the use of this methodology to determine relative differences between experimental groups is justified.

Even though bulk-fill composite materials enable the restoration build-up in 4–5 mm thick layers [43], specimen thickness in the present study was only 1.5 mm in order to allow a fair

comparison of shrinkage force formation of the bulk-fill composites with that of a conventional composite (Tetric EvoCeram). In contrast to bulk-fill composites, Tetric EvoCeram is indicated for use only in layers of max. 2-mm thickness, and would therefore not polymerize properly at 4–5-mm thickness. Furthermore, the well-established test set-up for shrinkage force measurements [27] chosen in the present study, with a C-factor of 2, might represent a more realistic configuration for Class II restorations [24] than if specimen thickness would have been increased to 4 mm, resulting in a very low C-factor of 0.75. Increasing specimen thickness in the shrinkage stress test would allow for increased (radial) stress relief by viscous flow, thus reducing the axial (measurable) stress [44].

In an attempt to minimize shrinkage stresses, bulk-fill resin composites contain features such as pre-polymer stress relievers, polymerization modulators, and modified high-molecular-weight base monomers [43]. Several studies revealed, in fact, reduced shrinkage stress formation of flowable bulk-fill materials compared with both conventional flowable and non-flowable micro- and nano-hybrid resin composites [13,27,42]. Yet, in agreement with previous reports [12,45], the findings of the present research do not confirm the favorable results of flowable bulk-fill materials in view of reduced stress development for the high-viscosity bulk-fill resin composites under investigation, given that these materials developed similar or even higher shrinkage forces than a conventional (nano-hybrid) resin composite, independent of the pre-cure temperature. The higher shrinkage forces recorded for Tetric EvoCeram Bulk Fill and QuixFil in comparison with

conventional Tetric EvoCeram, which has a similar resin composition as its bulk-fill counterpart, might be attributed to the higher filler content of the bulk-fill materials (Table 1). Higher filler content increases composite stiffness [46], which has been related to increased stress formation by reducing the material's flow capacity [47]. Pre-heating of the tested high-viscosity bulk-fill materials might therefore be a reasonable way to reduce shrinkage forces to levels below, or at least near to that of room-temperature conventional composite, particularly when bulk-filling cavities with unfavorable (high) C-factors where stresses are expected to be highest [15].

The degree of double bond conversion is a key material feature of dental resin composites, because it affects both physical and mechanical polymer properties as well as biocompatibility [48–51]. Previous studies indicated a significant increase in monomer conversion when dimethacrylate-based composites are photopolymerized at elevated composite temperature [4–7]. Elevating the cure temperature decreases system viscosity and improves molecular mobility, increasing collision frequency of reactive species and postponing diffusion-controlled propagation (autodeceleration), thus increasing final limiting conversion [4,7]. In the present investigation, however, only one of the five tested resin composites (Tetric EvoCeram Bulk Fill) showed a significant increase in monomer conversion upon pre-heating, whereas no difference in the degree of conversion between pre-heated and non-pre-heated groups was observed for the remaining materials. Therefore, the second part of the null hypothesis could not be rejected. In contrast to the studies that have demonstrated optimization in monomer conversion at elevated temperature [4–7], in the present investigation, the elevated composite temperature achieved after pre-heating was not maintained constant during experimentation in order to simulate a clinically relevant scenario where an isothermal curing condition cannot be realized. Once composite is pre-heated, there is a time delay between removing it from the heating device, dispensing it from a compule, placing it into a cavity preparation, contouring it, and subsequent light-curing. Preliminary investigations in our laboratory revealed that during the 40-s time interval between composite removal from the Calset device and start of photo-activation, the temperature of the pre-heated test materials decreased to between 34.9 °C (for SonicFill) and 36.4 °C (for x-tra fil). The rapid drop in composite temperature during handling might have prevented a sufficient increase in polymerization reactivity and consequent enhancement in monomer conversion, at least of four of the five materials under investigation. This is in accordance with findings of a recent study on non-isothermal polymerization of pre-heated composite that also observed no difference in the degree of conversion compared with room-temperature composite [2]. Furthermore, the same study revealed no difference in the susceptibility to softening in ethanol between pre-heated and room-temperature composite [2], suggesting similar crosslink density under non-isothermal curing conditions [52]. In addition, dental resins cured at different temperatures to similar final conversions have been reported to exhibit similar degrees of polymer network cross-linking as assessed by measurements of their glass transition temperature [53].

The effect of temperature on the extent of polymerization has been demonstrated to depend strongly on the photoinitiator system used [54]. Tetric EvoCeram Bulk Fill, the only material showing an increase in double bond conversion upon pre-heating, contains an additional photoinitiator (Ivocerin®) besides conventional camphorquinone/amine initiator systems. The addition of this germanium-based initiator with a higher quantum yield conversion compared to camphorquinone [55,56] might have affected pre-heating efficacy and contributed to the observed

increase in monomer conversion of Tetric EvoCeram Bulk Fill at elevated pre-cure temperature.

Isolation of the operative field by a rubber dam is highly recommended when placing direct adhesive restorations [57]. Plasmans et al. [58] reported an intraoral temperature of 25.1 °C around the treatment area after rubber dam application, and cavity surface temperatures during operative procedures may not be much beyond this value, at least if local anesthesia is used [59]. Consequently, and in line with previous in vitro studies [2,28], in the present investigation, specimen preparation and testing was performed inside a temperature-controlled chamber at 25 °C, which might closely match clinically relevant ambient temperature conditions in case of rubber dam placement.

5. Conclusions

Within the limitations of the present in vitro study, it can be concluded that both the composite material and the pre-cure temperature affect shrinkage force formation. Under clinically relevant (i.e., non-isothermal) conditions, composite pre-heating prior to photoactivation decreases polymerization-induced shrinkage forces of high-viscosity bulk-fill and conventional resin composites, while maintaining or increasing the degree of conversion.

Acknowledgments

References

[1] S. Lucey, C.D. Lynch, N.J. Ray, F.M. Burke, A. Hannigan, Effect of pre-heating on the viscosity and microhardness of a resin composite, J. Oral Rehabil. 37 (2010) 278–282.
[2] N.R. Froes-Salgado, L.M. Silva, Y. Kawano, C. Francci, A. Reis, A.D. Loguercio, Composite pre-heating: effects on marginal adaptation, degree of conversion and mechanical properties, Dent. Mater. 26 (2010) 908–914.
[3] W.C. Wagner, M.N. Aksu, A.M. Neme, J.B. Linger, F.E. Pink, S. Walker, Effect of pre-heating resin composite on restoration microleakage, Oper. Dent. 33 (2008) 72–78.
[4] M. Daronch, F.A. Rueggeberg, M.F. De Goes, R. Giudici, Polymerization kinetics of pre-heated composite, J. Dent. Res. 85 (2006) 38–43.
[5] M. Daronch, F.A. Rueggeberg, M.F. De Goes, Monomer conversion of pre-heated composite, J. Dent. Res. 84 (2005) 663–667.
[6] M. Trujillo, S.M. Newman, J.W. Stansbury, Use of near-IR to monitor the influence of external heating on dental composite photopolymerization, Dent. Mater. 20 (2004) 766–777.
[7] L.G. Lovell, S.M. Newman, C.N. Bowman, The effects of light intensity, temperature, and comonomer composition on the polymerization behavior of dimethacrylate dental resins, J. Dent. Res. 78 (1999) 1469–1476.
[8] S. Deb, L. Di Silvio, H.E. Mackler, B.J. Millar, Pre-warming of dental composites, Dent. Mater. 27 (2011) e51–e59.
[9] J.R. Bausch, C. de Lange, C.L. Davidson, The influence of temperature on some physical properties of dental composites, J. Oral Rehabil. 8 (1981) 309–317.
[10] U. Lohbauer, S. Zinelis, C. Rahiotis, A. Petschelt, G. Eliades, The effect of resin composite pre-heating on monomer conversion and polymerization shrinkage, Dent. Mater. 25 (2009) 514–519.
[11] R. Walter, E.J.J. Swift, H. Sheikh, J.L. Ferracane, Effects of temperature on composite resin shrinkage, Quintessence Int. 40 (2009) 843–847.
[12] R.J. Kim, Y.J. Kim, N.S. Choi, I.B. Lee, Polymerization shrinkage, modulus, and shrinkage stress related to tooth-restoration interfacial debonding in bulk-fill composites, J. Dent. 43 (2015) 430–439.
[13] D. Marovic, T.T. Tauböck, T. Attin, V. Panduric, Z. Tarle, Monomer conversion and shrinkage force kinetics of low-viscosity bulk-fill resin composites, Acta Odontol. Scand. 73 (2015) 474–480.
[14] R.R. Braga, R.Y. Ballester, J.L. Ferracane, Factors involved in the development of polymerization shrinkage stress in resin-composites: a systematic review, Dent. Mater. 21 (2005) 962–970.
[15] C.L. Davidson, A.J. Feilzer, Polymerization shrinkage and polymerization shrinkage stress in polymer-based restoratives, J. Dent. 25 (1997) 435–440.
[16] F.C. Calheiros, M. Daronch, F.A. Rueggeberg, R.R. Braga, Effect of temperature on composite polymerization stress and degree of conversion, Dent. Mater. 30 (2014) 613–618.

[17] S. Lottanti, T.T. Tauböck, M. Zehnder, Shrinkage of backfill gutta-percha upon cooling, J. Endod. 40 (2014) 721–724.

[18] M. Saitoh, S. Masutani, T. Kojima, M. Saigoh, H. Hirose, M. Nishiyama, Thermal properties of dental materials—cavity liner and pulp capping agent, Dent. Mater. J. 23 (2004) 399–405.

[19] Z. Tarle, T. Attin, D. Marovic, L. Andermatt, M. Ristic, T.T. Tauböck, Influence of irradiation time on subsurface degree of conversion and microhardness of high-viscosity bulk-fill resin composites, Clin. Oral Investig. 19 (2015) 831–840.

[20] N. Ilie, A. Kessler, J. Durner, Influence of various irradiation processes on the mechanical properties and polymerisation kinetics of bulk-fill resin based composites, J. Dent. 41 (2013) 695–702.

[21] A. Alrahlah, N. Silikas, D.C. Watts, Post-cure depth of cure of bulk fill dental resin-composites, Dent. Mater. 30 (2014) 149–154.

[22] S. Bucuta, N. Ilie, Light transmittance and micro-mechanical properties of bulk fill vs. conventional resin based composites, Clin. Oral Investig. 18 (2014) 1991–2000.

[23] R.Z. Alshali, N.A. Salim, J.D. Satterthwaite, N. Silikas, Post-irradiation hardness development, chemical softening, and thermal stability of bulk-fill and conventional resin-composites, J. Dent. 43 (2015) 209–218.

[24] A.J. Feilzer, A.J. De Gee, C.L. Davidson, Setting stress in composite resin in relation to configuration of the restoration, J. Dent. Res. 66 (1987) 1636–1639.

[25] M. Daronch, F.A. Rueggeberg, L. Moss, M.F. de Goes, Clinically relevant issues related to preheating composites, J. Esthet. Restor. Dent. 18 (2006) 340–350 discussion 351.

[26] F.A. Rueggeberg, M. Daronch, W.D. Browning, M.F. De Goes, In vivo temperature measurement: tooth preparation and restoration with preheated resin composite, J. Esthet. Restor. Dent. 22 (2010) 314–322.

[27] T.T. Tauböck, A.J. Feilzer, W. Buchalla, C.J. Kleverlaan, I. Krejci, T. Attin, Effect of modulated photo-activation on polymerization shrinkage behavior of dental restorative resin composites, Eur. J. Oral Sci. 122 (2014) 293–302.

[28] T.T. Tauböck, T. Bortolotto, W. Buchalla, T. Attin, I. Krejci, Influence of light-curing protocols on polymerization shrinkage and shrinkage force of a dual-cured core build-up resin composite, Eur. J. Oral Sci. 118 (2010) 423–429.

[29] M.M. Stavridakis, F. Lutz, W.M. Johnston, I. Krejci, Linear displacement and force induced by polymerization shrinkage of resin-based restorative materials, Am. J. Dent. 16 (2003) 431–438.

[30] L.G. Cunha, R.C. Alonso, C.S. Pfeifer, L. Correr-Sobrinho, J.L. Ferracane, M.A. Sinhoreti, Contraction stress and physical properties development of a resin-based composite irradiated using modulated curing methods at two C-factor levels, Dent. Mater. 24 (2008) 392–398.

[31] J.L. Ferracane, E.H. Greener, Fourier transform infrared analysis of degree of polymerization in unfilled resins—methods comparison, J. Dent. Res. 63 (1984) 1093–1095.

[32] R.R. Moraes, A.L. Faria-e-Silva, F.A. Ogliari, L. Correr-Sobrinho, F.F. Demarco, E. Piva, Impact of immediate and delayed light activation on self-polymerization of dual-cured dental resin luting agents, Acta Biomater. 5 (2009) 2095–2100.

[33] J.W. Stansbury, Dimethacrylate network formation and polymer property evolution as determined by the selection of monomers and curing conditions, Dent. Mater. 28 (2012) 13–22.

[34] R.V. Mesquita, J. Geis-Gerstorfer, Influence of temperature on the visco-elastic properties of direct and indirect dental composite resins, Dent. Mater. 24 (2008) 623–632.

[35] H. Lu, J.W. Stansbury, C.N. Bowman, Towards the elucidation of shrinkage stress development and relaxation in dental composites, Dent. Mater. 20 (2004) 979–986.

[36] D. Tantbirojn, S. Chongvisal, D.G. Augustson, A. Versluis, Hardness and postgel shrinkage of preheated composites, Quintessence Int. 42 (2011) e51–e59.

[37] J.B. da Costa, T.J. Hilton, E.J.J. Swift, Critical appraisal: preheating composites, J. Esthet. Restor. Dent. 23 (2011) 269–275.

[38] L.C. Boaro, N.R. Froes-Salgado, V.E. Gajewski, A.A. Bicalho, A.D. Valdivia, C.J. Soares, et al., Correlation between polymerization stress and interfacial integrity of composites restorations assessed by different in vitro tests, Dent. Mater. 30 (2014) 984–992.

[39] F.C. Calheiros, F.T. Sadek, R.R. Braga, P.E. Cardoso, Polymerization contraction stress of low-shrinkage composites and its correlation with microleakage in class V restorations, J. Dent. 32 (2004) 407–412.

[40] J.L. Ferracane, J.C. Mitchem, Relationship between composite contraction stress and leakage in Class V cavities, Am. J. Dent. 16 (2003) 239–243.

[41] D. Truffier-Boutry, S. Demoustier-Champagne, J. Devaux, J.J. Biebuyck, M. Mestdagh, P. Larbanois, et al., A physico-chemical explanation of the post-polymerization shrinkage in dental resins, Dent. Mater. 22 (2006) 405–412.

[42] N. Ilie, R. Hickel, Investigations on a methacrylate-based flowable composite based on the SDR technology, Dent. Mater. 27 (2011) 348–355.

[43] J. Manhart, R. Hickel, Bulk-fill-composites. Modern application technique of direct composites for posterior teeth, Swiss Dent. J. 124 (2014) 19–37.

[44] M.F. Witzel, R.Y. Ballester, J.B. Meira, R.G. Lima, R.R. Braga, Composite shrinkage stress as a function of specimen dimensions and compliance of the testing system, Dent. Mater. 23 (2007) 204–210.

[45] J.H. Jang, S.H. Park, I.N. Hwang, Polymerization shrinkage and depth of cure of bulk-fill resin composites and highly filled flowable resin, Oper. Dent. 40 (2015) 172–180.

[46] M. Braem, W. Finger, V.E. Van Doren, P. Lambrechts, G. Vanherle, Mechanical properties and filler fraction of dental composites, Dent. Mater. 5 (1989) 346–348.

[47] J.R. Condon, J.L. Ferracane, Assessing the effect of composite formulation on polymerization stress, J. Am. Dent. Assoc. 131 (2000) 497–503.

[48] T.T. Tauböck, M. Zehnder, T. Schweizer, W.J. Stark, T. Attin, D. Mohn, Functionalizing a dentin bonding resin to become bioactive, Dent. Mater. 30 (2014) 868–875.

[49] L.C. Boaro, F. Goncalves, T.C. Guimaraes, J.L. Ferracane, C.S. Pfeifer, R.R. Braga, Sorption, solubility, shrinkage and mechanical properties of low-shrinkage commercial resin composites, Dent. Mater. 29 (2013) 398–404.

[50] T.T. Tauböck, H. Oberlin, W. Buchalla, M. Roos, T. Attin, Comparing the effectiveness of self-curing and light curing in polymerization of dual-cured core buildup materials, J. Am. Dent. Assoc. 142 (2011) 950–956.

[51] W.F. Caughman, G.B. Caughman, R.A. Shiflett, F. Rueggeberg, G.S. Schuster, Correlation of cytotoxicity, filler loading and curing time of dental composites, Biomaterials 12 (1991) 737–740.

[52] E. Asmussen, A. Peutzfeldt, Influence of pulse-delay curing on softening of polymer structures, J. Dent. Res. 80 (2001) 1570–1573.

[53] L.G. Lovell, H. Lu, J.E. Elliott, J.W. Stansbury, C.N. Bowman, The effect of cure rate on the mechanical properties of dental resins, Dent. Mater. 17 (2001) 504–511.

[54] T. Scherzer, U. Decker, The effect of temperature on the kinetics of diacrylate photopolymerizations studied by Real-time FTIR spectroscopy, Polymer 41 (2000) 7681–7690.

[55] J.G. Leprince, W.M. Palin, M.A. Hadis, J. Devaux, G. Leloup, Progress in dimethacrylate-based dental composite technology and curing efficiency, Dent. Mater. 29 (2013) 139–156.

[56] N. Moszner, U.K. Fischer, B. Ganster, R. Liska, V. Rheinberger, Benzoyl germanium derivatives as novel visible light photoinitiators for dental materials, Dent. Mater. 24 (2008) 901–907.

[57] S.D. Heintze, V. Rousson, Clinical effectiveness of direct class II restorations—a meta-analysis, J. Adhes. Dent. 14 (2012) 407–431.

[58] P.J. Plasmans, N.H. Creugers, R.J. Hermsen, M.M. Vrijhoef, Intraoral humidity during operative procedures, J. Dent. 22 (1994) 89–91.

[59] N. Hofmann, B. Hugo, B. Klaiber, Effect of irradiation type (LED or QTH) on photo-activated composite shrinkage strain kinetics, temperature rise, and hardness, Eur. J. Oral Sci. 110 (2002) 471–479.