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Ich bedanke mich bei den unten aufgeführten Kolleginnen und Kollegen für ihre wertvolle Mitarbeit, die sie im vergangenen Jahr geleistet haben.

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Photoelastic determination of polymerization shrinkage stress in low-shrinkage resin composites

Keywords: polymerization shrinkage stress, low-shrinkage resin composites, photoelastic examination

Summary Low-shrinkage resin composites are in the focus of research in posterior resin composite restoratives. The aim of the study was to examine the polymerization shrinkage stress of new composites (Venus Diamond/Heraeus Kulzer; SDR/DENTSPLY) and an experimental low-shrinkage resin composite (Ormocer/VOCO) in comparison to established low-shrinkage resin composites (Filtek Silorane/3M ESPE; els/Saremco; Filtek Supreme XT/3M ESPE; Clearfil Majesty Posterior/Kuraray). Cylindrical cavities (\varnothing 4 mm) in Araldit-B epoxy resin plates (40×40×4 mm) were pretreated with the Rocotec system to ensure bonding of the resin composites. The resin composite specimens (n=10) were exposed to light for 60 s with a QTH curing device (Translux energy, Heraeus Kulzer, Germany). The samples were stored dark and dry (23 °C). Polymerization shrinkage stress data (MPa) 4 min and

24 h post exposure were calculated based on the diameter of the first-order isochromatic rings, obtained from the Araldit plates. The statistical analysis of the obtained data was carried out with the Wilcoxon test ($p=0.05$). After 24 h, the following mean stress values and standard deviations were obtained: Venus Diamond 3.4 ± 0.27 MPa; SDR 3.3 ± 0.26 MPa; exp. Ormocer 4.0 ± 0.18 MPa; Filtek Silorane 2.8 ± 0.19 MPa; els 2.5 ± 0.09 MPa; Filtek Supreme XT 6.0 ± 0.20 MPa; and Clearfil Majesty Posterior 5.6 ± 0.15 MPa. For all materials, higher polymerization stress values were recorded after 24 h. All differences in the shrinkage data obtained after 24 h were statistically significant ($p<0.05$) except Venus Diamond/SDR. Venus Diamond, els and SDR showed shrinkage data closer to that of Filtek Silorane.

Introduction

Today, tooth-colored composites have established themselves worldwide as the restorative material of choice. Yet despite constant development, the continued existence of polymerization shrinkage still causes problems in the bond between the dental hard tissues and the composite resin.

Polymerization shrinkage is understood as the neutral volumetric reaction of composite resins during curing. It is based on the shortening of the intermolecular distance during polymerization, resulting in a strong polymer network. The German industrial standard DIN and the international standard

ISO determine polymerization shrinkage norms according to the Archimedes principle with a test specimen in water (TANI ET AL. 1985, RUEGGERBERG & TAMARESELY 1995), which yields values in percent (%). However, since this method does not test composite resin specimens bonded in a cavity, it is not particularly clinically relevant. As soon as a composite resin specimen is luted in a cavity – as in clinical use – an entirely different situation results: complete volumetric shrinkage as in the Archimedes test is not possible. The composite resin generates contraction stresses which affect the cavity margin; in the clinical situation, these stresses are responsible for the composite pulling away from the margin, creating a marginal gap.

This detachment of the composite resin from the dental hard tissues can result in postoperative sensitivity, enamel cracking, recurrent caries, marginal discoloration and ultimately the failure of the restoration (YAMAZAKI ET AL. 2006). The polymerization contraction stresses do not, however, correspond to the percent shrinkage values provided by the Archimedes test method. The difference consists in including the composite resin's inherent elasticity (modulus of elasticity) in the determination of the polymerization shrinkage force. Thus, the polymerization shrinkage force can be defined as the product of polymerization shrinkage and the material's modulus of elasticity (ERNST ET AL. 2004), and is therefore given in MPa or N/mm². Using a composite resin with a low modulus of elasticity, i.e., greater elastic properties, can compensate for polymerization shrinkage to a certain extent. The stresses impinging upon the cavity margin are reduced. Using a composite material with an identical volumetric shrinkage (polymerization shrinkage) but a higher modulus of elasticity (i.e., it is stiffer) subjects the adhesive bond to greater stress. Because all in vitro tests are ultimately intended to simulate the clinical situation, they should approximate real clinical demands as closely as possible. For this reason, the polymerization contraction forces actually stressing the cavity margin are much more clinically significant than the determination of volumetric shrinkage (polymerization shrinkage).

Critical factors such as the proportion of filler particles and the monomer chemistry of the composite resin (SATTERTHWAITE ET AL. 2009, AW & NICHOLLS 2001) additionally influence both the shrinkage and the contraction force, and present approaches for further development. Although a higher percentage of fillers associated with a minimized matrix component can reduce the composite resin's polymerization shrinkage over (AW & NICHOLLS 2001, PEARSON & BOUSCHLICHER 2001), this does not automatically imply lower polymerization stress, because as a result of the reduced matrix component, the filling material's elasticity decreases, thus rendering it stiffer. Diminished volumetric shrinkage based on an increased percentage of filler particles often results in the same polymerization stress, because the lower shrinkage is compensated by a higher modulus of elasticity (SCHATTENBERG ET AL. 2007). Another means of decreasing shrinkage forces by reducing internal tensions is a modified light-curing technique called "soft-start polymerization" (CHAN ET AL. 2008, HARDAN ET AL. 2008, KANG ET AL. 2007, ERNST ET AL. 2000). Braga et al. found a lower degree of conversion with a decrease in shrinkage force (BRAGA ET AL. 2005). Furthermore, the literature contains a number of studies on the influence of cavity geometry and filling technique on polymerization shrinkage stress (ALOMARI ET AL. 2007, JEDRYCHOWSKI ET AL. 2001, DAUVILLIER ET AL. 2000). However, because the latter two variables essentially failed to offer clinically relevant material improvements in terms of reduced shrinkage forces, it was necessary to develop new monomers and co-monomers as alternatives to conventional bis-GMA in the hope of enabling substantial reductions in polymerization shrinkage stress (KIM ET AL. 2004, ATAI & WATTS 2006).

Current research on low-shrinkage materials now focuses on different approaches using ring-opening monomers (Silorane/3M ESPE), high filler content plus reduced modulus of elasticity (Venus Diamond/Heraeus Kulzer), special polymerization modulators which are intended to effect a low-shrinkage structure of the polymer network (SDR/Dentsply), or the thorough elimination of particularly high-shrinkage monomers based on conventional composite chemistry (els/Saremco) (DUARTE ET AL. 2009, ILIE & HICKEL 2009, PAPADOGIANNIS ET AL. 2009).

Various methods exist for measuring polymerization shrinkage; many are very elaborate and sensitive to temperature fluctuations, storage time, or room temperature, all of which can substantially influence the test results (BAUSCH ET AL. 1982). For instance, one such method uses dilatometers, which determine polymerization shrinkage according to the volumetric change of a liquid surrounding the test material (BAUSCH ET AL. 1982). A special form of the dilatometer is the frequently employed "modified dilatometer" (KLEVERLAAN & FEILZER 2005, DE GEE ET AL. 1993), which is not a suitable method for testing light-curing composites due to the lack of transparency and the use of mercury (WATTS & CASH 1991). The present study employed the photoelastic method to measure shrinkage force (KINOMOTO ET AL. 2000). By utilizing a special stress-sensitive embedding material, the tension lines produced in the embedding material by the shrinkage of the restorative composite resin bonded to it can be examined to determine the shrinkage stress (ERNST ET AL. 2004).

Given the constant development of low-shrinkage composites, such as Silorane mentioned above, the question arose as to the actual contraction force of experimental and already commercially available, established composite resin materials. The following null hypotheses were therefore posed:

1. Compared to conventional composite resins, the new and experimental composites demonstrate significantly lower polymerization shrinkage stress.
2. Highly filled low-shrinkage composites are unable to significantly reduce contraction forces.

Materials and Methods

For photoelastic measurement, the composite resins (Tab. I) were applied into a 4-mm-diameter hole drilled stress-free in an Araldit-B carrier plate made of heat-curing epoxy resin (40×40×4 mm, Tiedemann & Betz GmbH, Garmisch-Partenkirchen, Germany). The drilled surfaces of the plate were tribochemically silicated (Rocatec, ESPE-Sil, Visio-Bond, 3M ESPE, St. Paul/MN, USA) to ensure adhesive bonding of the composite to the carrier material. Both the light-curing adhesive (Visio-Bond, 3M ESPE) and the composite resin to be tested were light-cured using a halogen light-curing unit (Translux Energy, Heraeus Kulzer, Hanau, Deutschland) in standard mode (60 s, 550 mW/cm²) with direct contact. To prevent the formation of an oxygen inhibition layer, the adhesive and the composite resin were covered with a piece of

Tab. I Materials used in the photoelastic examination of polymerization shrinkage stress

Composite	Manufacturer	Lot number
Venus Diamond	Heraeus Kulzer, Hanau, Germany	010021RMK1
SDR	DENTSPLY Caulk, Milford, DE, USA	FPF-01-40-2
Exp.Ormocer	VOCO, Cuxhaven Germany	V35694
Filtek Silorane	3M ESPE St. Paul, MN, USA	7Aj
els	Saremco, Rebstein, Switzerland	11.2011-52
Filtek Supreme XT	3M ESPE St. Paul, MN, USA	7LF
Clearfil Majesty Posterior	Kuraray, Tokyo, Japan	0001D

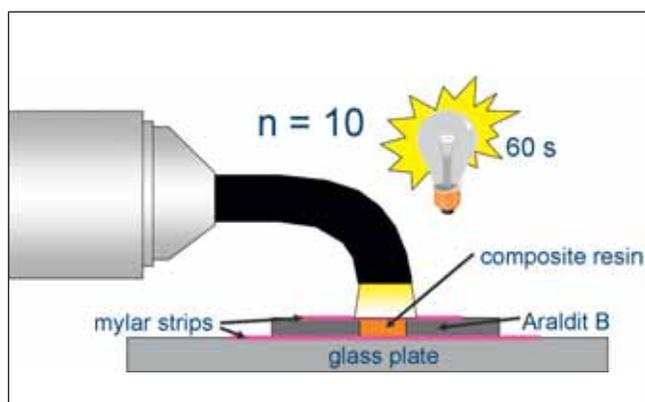


Fig. 1 Schematic visualization of composite resin samples designed for photoelastic examination between two mylar strips light-cured for 60 s from both sides.

mylar tape (Fig. 1). The composite specimens were polymerized from both sides (2×60 s).

By using Araldit-B carrier plates with a low photoelastic constant ($f_e = 10.5 \text{ N/mm}$) and a module of elasticity of 3400 MPa, material stress can be visualized using isochromatic rings (Fig. 2).

Immediately after polymerization at time point $t = 0$ (after 4 minutes) and at time point $t = 24$ (after 24 hours), the specimens ($n = 10$) were measured at a photoelasticity workstation (Fig. 3). Between individual measurements, the specimens were wrapped in conventional aluminum foil and stored dry at a constant room temperature of 23 °C. Stable storage conditions were ensured in an air-conditioned room with constant monitoring of the temperature. Through the filters present in the measurement device (Linus Photonics GmbH, Göttingen, Germany) constant light and dark fields as shown in Fig. 2 were attained for the microscopic examination (Stemi 2000-C, Zeiss, Göttingen, Germany). Illumination was provided by a fluorescent light (KL 1500 LCD, Zeiss) with a color temperature of 3000 Kelvin beneath the filter array.

Images acquired with a CCD camera (color compact camera Teli CS-5260 DP, 752×582 pxls) were transferred to the connected computer. Using a special computer program (Matrox Inspector, Rauscher GmbH, Olching, Germany), the isochromatic rings were made visible; subsequently, a cross-hair cursor (Version 3.0, Matrox Electronic System Ltd., Rauscher GmbH) (Fig. 2) was employed to determine the diameter in pixels.

The marginal stress was calculated using the photoelastic equation (ROHRBACH 1989, SCHATTENBERG ET AL. 2007):

$$\delta = (\sigma_1 - \sigma_2) C d / \lambda_2$$

- δ Isochromatic fringe order
- C Material constant ($10^{-6} \text{ mm s}^2/\text{kg}$)
- λ Wavelength of light (nm)
- d Specimen thickness (mm)
- $\sigma_1 - \sigma_2$ Difference of principal stresses

Because the parameters f_e ($=10.5 \text{ N/mm}$), D ($= 4 \text{ mm}$), and D_i ($= 4 \text{ mm}$) remained constant in this study, the equation can be summarized as follows, given the constant k (ROHRBACH 1989, SCHATTENBERG ET AL. 2007):

$$\sigma_0 = k \delta_x D_x^2$$

The visible isochromatic rings are numbered in ascending order from the outside to the inside (Fig. 2). As in previous studies, first-order isochromatic rings were chosen for measurement, in order to obtain comparable results and low standard deviation

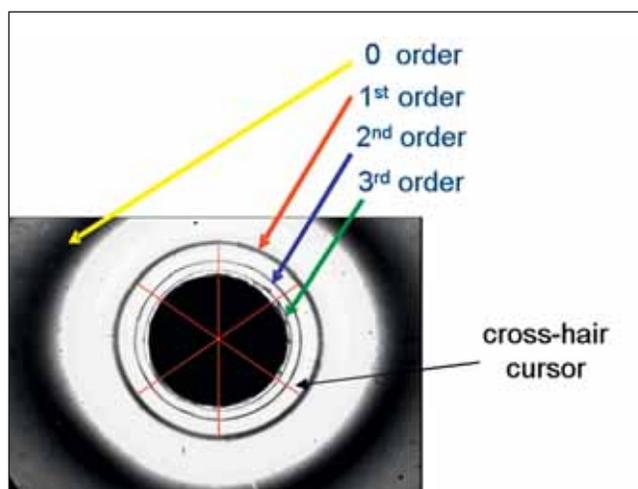


Fig. 2 Visualization of the isochromatic rings and determination of the ring diameter, sketched with the crosshair cursor.

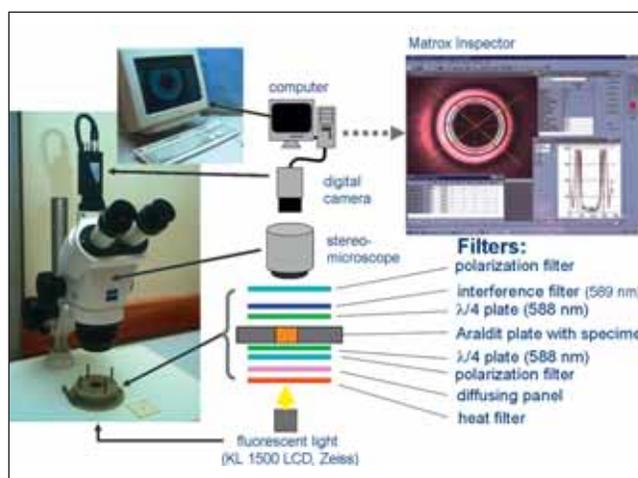


Fig. 3 Experimental setup of the photoelasticity workstation for obtaining photoelastic images.

(ROHRBACH 1989, HECHT 1989, SCHATTENBERG 2007). A partial or complete detachment of the restorative material was obvious in the photoelastic image as an interruption of continuity. These specimens were discarded and not included in the analysis.

The data were statistically analyzed using the two-sided Wilcoxon test at a significance level of 0.05.

Results

In this study, the polymerization shrinkage stress of the composite resins tested (Tab. I) was examined first after 4 minutes and again after 24 hours. Immediately following polymerization at $t = 0$ (4 min), the average polymerization shrinkage stress values in MPa \pm standard deviation were: 2.5 ± 0.20 for Venus Diamond, 2.2 ± 0.17 for SDR, 2.4 ± 0.18 for the experimental Ormocer (Voco), 2.6 ± 0.16 for Filtek Silorane, 1.9 ± 0.09 for els, 4.4 ± 0.12 for Filtek Supreme XT, and 4.7 ± 0.12 for Clearfil Majesty Posterior. The restorative materials exhibited an increase in polymerization shrinkage stress after 24 hours. The average shrinkage force values in MPa \pm standard deviation at $t = 24$ (24 h) were: 3.4 ± 0.27 for Venus Diamond, 3.3 ± 0.26 for SDR, 4.0 ± 0.18 for the experimental Ormocer, 2.8 ± 0.19 for Filtek Silorane, 2.5 ± 0.09 for els, 6.0 ± 0.20 MPa for Filtek Supreme XT, and 5.6 ± 0.15 for Clearfil Majesty Posterior.

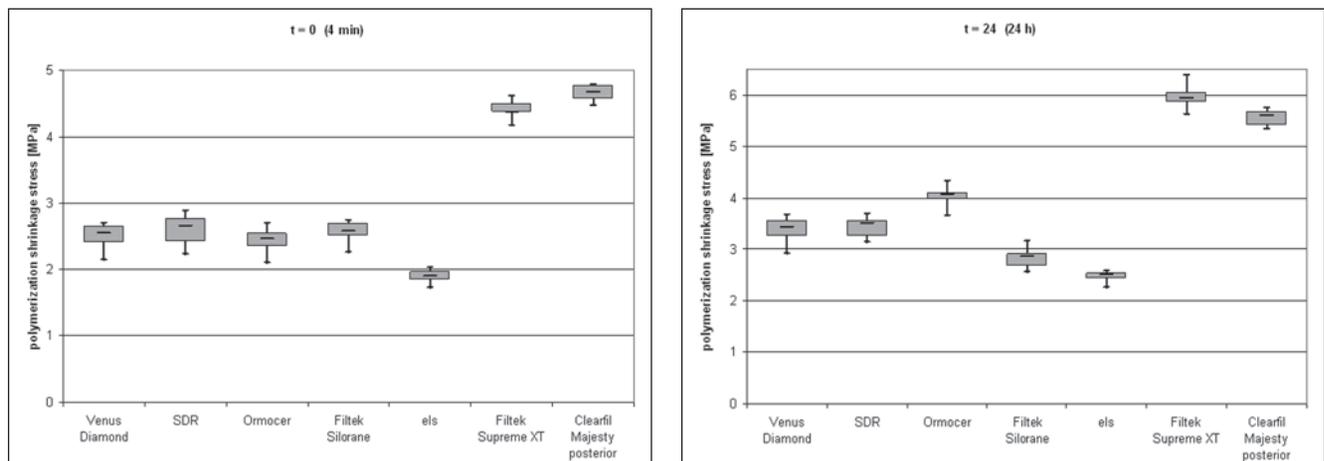


Fig. 4 and 5 Box-plots of the median polymerization shrinkage stress values for all materials obtained after 4 minutes ($t=0$) and 24 hours ($t=24$ h). Data are listed for median, minimum and maximum values, 1st and 3rd quartiles.

Tab. II Statistical evaluation of the group comparisons (Wilcoxon test) 24 h ($t=24$) after exposure, significance set at $p < 0.05$.

	Clearfil Majesty Posterior	Ormocer	Filtek Silorane	Venus Diamond	Filtek Supreme XT	SDR
els	0.005	0.005	0.008	0.005	0.005	0.005
Clearfil Majesty posterior	–	0.005	0.005	0.005	0.005	0.005
Ormocer	–	–	0.005	0.005	0.005	0.005
Filtek Silorane	–	–	–	0.005	0.005	0.005
Venus Diamond	–	–	–	–	0.005	0.721
Filtek Supreme XT	–	–	–	–	–	0.005

The medians, maximum and minimum values, and 1st and 3rd quartiles are graphically depicted in Figures 4 and 5. The results of the statistical analysis are given in Table II.

Discussion

The photoelastic method for measuring polymerization contraction forces has been described in several previous publications (KINOMOTO ET AL. 2000, ERNST ET AL. 2004, ROHRBACH 1989). This method makes it possible to demonstrate the same material properties by using industrially fabricated Araldit-B plates and unvaryingly identical pre-treatments. In contrast to that used in previous studies (SCHATTENBERG ET AL. 2007, ERNST ET AL. 2004), the new plate design with a high C-Factor (specimen thickness increased from 3 to 4 mm; diameter of test cavities reduced from 5 to 4 mm) enabled more exact differentiation of the polymerization shrinkage forces, particularly in the low-shrinkage composites.

The type of photoelastic measurement employed here is impressive due to its low variance, which can demonstrate even the slightest differences in polymerization shrinkage stress in an experimental setup using ten specimens per restorative material. Measurements were taken 4 minutes and 24 hours after curing the specimens. The 4 minutes between polymerization and the first measurement was the time required to place the specimen in the photoelasticity workstation. For all specimens, this examination time point was set as $t = 0$. The greatest shrinkage force values (MPa) were measured at 24 h. In studies by Watts and Cash (1991) and de Gee et al. (1993), the examination time point $t = 0$ was set at 1 minute after poly-

merization. In contrast, Attin et al. (1995) measured shrinkage force after 5 minutes ($t = 0$) and 24 hours ($t = 24$). The great variation in shrinkage force results can be attributed to changes in the material composition (PRICE ET AL. 2000), i. e., to an increased proportion of filler particles and an increased diameter of the same (SATTERTHWAITE ET AL. 2009, AW & NICHOLLS 2001). The decrease in shrinkage force can be explained by a lower modulus of elasticity (DAVIDSON & DE GEE 1984); but the polymerization stress affecting the cavity walls also depends on the extent to which the material shrinks. The viscoelastic behavior of the composite resin, along with the volumetric shrinkage, are two of the most critical factors influencing shrinkage force (PFEIFER ET AL. 2008, BRAGA 2005).

The manufacturers of the composite resins examined here recommend different polymerization times. To facilitate standardization and contrary to manufacturer's instructions, the present study uniformly employed a curing time of 60 s at a light intensity of 550–600 mW/cm². Adequate curing depth of the 4-mm-thick specimens was ensured by light polymerizing from both sides of the plates (2×60 s). This length of light exposure – far above that recommended by the manufacturer – was intended to guarantee complete curing of the composite specimens.

As repeatedly shown in the literature, polymerization shrinkage primarily depends on the degree of conversion (BRAGA & FERRACANE 2002, SPINELL ET AL. 2009). The light dose fundamentally influences the polymerization shrinkage force. To deliver an adequate light dose, consisting of light intensity (J/cm²) and duration of light exposure, a curing time of 60 s per side was chosen in this study. Both Visvanathan et al. (2007) and Lopes

et al. (2008) found higher polymerization shrinkage values with higher intensity of light units (LOPES ET AL. 2008).

In all the composites tested here, both experimental and conventional, an increase in shrinkage force (MPa) was observed after 24 h. This increase was lowest in Filtek Silorane (0.2 MPa), followed by els (0.6 MPa) and Venus Diamond (0.9 MPa). The largest increase after 24 h was found for the experimental Ormocer composite (1.6 MPa) and Filtek Supreme XT (1.6 MPa); however, the baseline shrinkage value of 4.4 MPa was also quite high. This rise can be explained by continued "post-polymerization" even after the end of light irradiation, as known from the literature (YAP ET AL. 2000). After reaching this point, the contraction force remains stable and essentially no further development occurs (ATTIN ET AL. 1995). With the least increase in polymerization shrinkage force within 24 h, Filtek Silorane produced results similar to those found in numerous other studies which also obtained good results with this low-shrinkage composite in terms of shrinkage behavior and macro-, micro-, and nanomechanical properties (MIN ET AL. 2010, ILIE & HICKEL 2009, PAPADOGIANNIS ET AL. 2009, SCHATTENBERG ET AL. 2007). This was confirmed by other authors as well, who found that in contrast to the methacrylate-based materials, the silorane composites exhibited lower shrinkage forces but with comparable mechanical properties (LEPRINCE ET AL. 2010, DUARTE ET AL. 2009, WEINMANN ET AL. 2005), also regarding the occurrence of marginal gaps (BAGIS ET AL. 2009).

Just as the low-shrinkage composites already on the market, the experimental Ormocer showed considerably lower shrinkage forces than the conventional material Filtek Supreme XT (LEPRINCE ET AL. 2010). The composites Venus Diamond and SDR provided similar shrinkage force results. Marchesi et al. (2010) also observed less shrinkage stress development in Venus Diamond compared to several low-shrinkage composites (MARCHESI ET AL. 2010). In a recent study on the shrinkage behavior of methacrylate-based flowable composites based on the SDR ("Smart Dentin Replacement") technology, these materials exhibited even lower shrinkage forces than the silorane or nano- and microhybrid composites examined (ILIE & HICKEL 2010). The polymerization modulator built into the structural resin backbone of the SDR composite is intended to allow the monomers to link more flexibly to form the polymer network, thereby allowing a high degree of conversion and a high network density. This is designed to compensate volumetric shrinkage and thus result in less polymerization stress. The overall lowest shrinkage stress values were exhibited by the "extra low-shrinkage" composite els (1.9 MPa at $t = 0$ and 2.5 at $t = 24$ h). This microhybrid composite showing both low shrinkage stress and low volumetric shrinkage (1.5%) owes its stress-free polymerization to a homogeneous combination of fillers and resin matrix. The composite consists of a microfine, homogeneous BaAlBSi (barium-aluminum-boron-silicate) glass powder and a small percentage of a silanized silicium dioxide which is incorporated into the polymer network during polymerization. The glass filler has an average particle size of 0.7 μm ; no particle is larger than 2.6 μm and thus contains a finer mineral filler than many other composites. Considered a low-shrinkage composite because of its high filler content,

Clearfil Majesty Posterior did not yield clinically relevantly reduced shrinkage stress values compared to conventional composite resins, due to its consequentially higher modulus of elasticity. This finding clearly demonstrates that merely increasing the filler content to reduce polymerization shrinkage does not show any potential for improving marginal integrity.

The approach to reducing shrinkage by developing new matrix chemistry, not just by trying different fillers or filler contents, opens new and wider horizons for improving the light-curing direct composite restorative materials. Other authors have also come to this conclusion in studies on low-shrinkage monomers in composite resins (EICK ET AL. 2010, VASUDEVA 2009). Various approaches – for instance ring-opening monomers (Filtek Silorane), reducing the modulus of elasticity of the entire composite while maintaining a high filler content (Venus Diamond), the integration of polymerization modulators (SDR), but also the consistent evolution of the Ormocers (e.g., the experimental Ormocer in this study) or the use of selective matrix components (els) – yield similarly good results.

Therefore, both null hypotheses posed at the beginning of this study are confirmed:

1. The new and experimental composites exhibit significantly reduced polymerization shrinkage stress compared to conventional composites.
2. The reduction of polymerization shrinkage by increasing the filler content does not facilitate a reduction of polymerization shrinkage stress.

Résumé

Le but de l'investigation était de déterminer la force de rétraction de nouveaux composites (Venus Diamond/Heraeus Kulzer, SDR/DENTSPLY) et d'un composite expérimental (Ormocer expérimental/VOCO), et de les comparer avec les composites établis d'une force de rétraction réduite (Filtek Silorane/3M ESPE, els/Saremico) ainsi qu'avec les composites bien documentés (Filtek Supreme XT/3M ESPE, Clearfil Majesty Posterior/Kuraray).

Les composites ont été insérés dans des cavités cylindriques de paillettes d'araldit B après un traitement au Rocatec des surfaces de contact. Une polymérisation de 60 s avec une lampe halogène a été effectuée (Translux Energy, Heraeus Kulzer). La force de rétraction a été mesurée immédiatement ($t = 0$) et après 24 heures ($t = 24$), après stockage des échantillons dans des conditions sèches, sombres et à température ambiante constante. L'évaluation statistique fut établie selon le test de Wilcoxon. Après 24 heures, la force de rétraction (MPa) était de $3,4 \pm 0,3$ pour Venus Diamond, $3,4 \pm 0,2$ pour SDR, $4,0 \pm 0,2$ pour l'Ormocer expérimental, $2,8 \pm 0,2$ pour Filtek Silorane, $2,5 \pm 0,1$ pour els, $6,0 \pm 0,2$ pour Filtek Supreme XT et $5,6 \pm 0,2$ pour Clearfil Majesty Posterior.

Les nouveaux composites et les composites expérimentaux d'une force de rétraction réduite montrent, après 24 heures, une réduction de près de 50% de la force de rétraction, comparés aux composites bien documentés.

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