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Influence of Ambient Temperature and Light-curing Moment on Polymerization Shrinkage and Strength of Resin Composite Cements

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Clinical Relevance

Polymerization shrinkage at 37°C with light application 5 minutes after mixing ranges from 4.0% to 5.8%. Light application performed as soon as possible after placing a restoration minimizes cement shrinkage.

ABSTRACT

Objective: The purpose of this study was to establish a clinically appropriate light-curing moment for resin composite cements while achieving the highest indirect tensile strength and lowest polymerization shrinkage.

Methods and Materials: Polymerization shrinkage of seven resin composite cements (Multilink Automix, Multilink Speed Cem, RelyX Ultimate, RelyX Unicem 2 Automix, Panavia V5, Panavia SA plus, VITA Adiva F-Cem) was measured at ambient temperatures of 23°C and 37°C. Testing was done for autopolymerized and light-cured specimens after light application at either 1, 5, or 10 minutes after mixing. Indirect tensile strength of all cements was measured after 24 hours of storage at temperatures of 23°C and 37°C, for autopolymerized and light-cured specimens after light application 1, 5, or 10 minutes after mixing. To illustrate filler size and microstructures, SEM images of all cements were captured. Statistical analysis was performed with one-way ANOVA followed by *post hoc* Fisher LSD test (α=0.05).

Results: Final polymerization shrinkage of the resin composite cements ranged from 3.2% to 7.0%. An increase in temperature from 23°C to 37°C as well as the light-curing moment resulted in material dependent effects on the polymerization shrinkage and indirect tensile

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strength of the cements. Polymerization shrinkage of the cements did not correlate with the indirect tensile strength of the cement in the respective groups. Highest indirect tensile strengths were observed for the materials containing a homogeneous distribution of fillers with a size of about 1 μm (Multilink Automix, Panavia V5, VITA Adiva F-Cem).

Conclusion: The magnitude of the effect of light-curing moment and temperature increase on polymerization shrinkage and indirect tensile strength of resin composite cements is material dependent and cannot be generalized.

INTRODUCTION

The increased use of esthetic ceramic materials in dentistry requires the application of resin-based luting cement to bond a restoration to the tooth structure. Resin-based composite materials are generally superior to conventional cements in providing higher strength and low margin wear.[1-3] Their polymerization shrinkage may nevertheless lead to microleakage.[4]

The polymerization of dual-cured resin composite cements is catalyzed by a chemically (autopolymerization) and photo-activated (light-curing) initiator. The polymerization reaction starts with the mixing of base and catalyst paste, thus activating the chemical initiator. Hence, the processing time is limited. Photo initiation allows the polymerization reaction to advance at the time a restoration is correctly placed and cement excess is removed. However, areas under an opaque restoration not reached by the light may not polymerize as well as dual-cured areas. Most cement materials reveal a higher degree of conversion by dual-curing compared with autopolymerization.[5-7]

Shrinkage occurs during the crosslinking of polymer chains, thus creating polymerization stress between tooth structure and resin composite cement.[8] In the early stage of polymerization, resin composite cement reveals high viscosity and is therefore able to relax developing stress. After a short time or after light application, the material becomes rigid and is unable to deform, hence, stress starts to increase.[9,10] The magnitude of volumetric shrinkage is determined by the amount and volume of fillers as well as the composition and the degree of conversion of the resin matrix.[10] Filler particle content for resin composite cement is typically around 50 vol%.[11] Shrinkage strain for different resin cements is reported to range from 1.77% to 5.29%.[12] Shrinkage values should be considered approximate as they vary with the extent of the polymerization reaction.[11] The application of light to dual-cured resin composite cements increases their degree of conversion but not necessarily their shrinkage strain.[12] The degree of conversion is, however, material related; some systems are significantly more dependent on light-activation than others.[5,13-17] Due to a slower reaction rate and incorporation of porosity, autopolymerized resin composite cements may develop less shrinkage stress.[18-20] A dual-curing mode, however, improves bond strength,[21] flexural strength, compressive strength, indirect tensile strength, elastic modulus, and hardness.[3,22,23]

An increase in temperature from 23°C to 37°C may also affect the polymerization process. For light-cured resin composites, a more rapid stress build-up—meaning an increased shrinkage-stress rate—was reported when the temperature was increased.[10] Because of the increased temperature, viscosity may also decrease, resulting in additional monomer conversion[24-26] and thus in a higher degree of conversion.[26]

It has been speculated that a delay in light-activation of dual-cured composite resins enhances their properties by allowing the autopolymerization initiators to react to some extent before being entrapped by photo-activated polymeric chains.[27-29] An ideal balance between autopolymerization and time of light-activation is yet to be determined.[29] To our knowledge, there are no studies that report how polymerization shrinkage of resin composite cements is affected by the light-curing moment (time of light application) and temperature increase and how these factors affect the materials' strength.

The purpose of this study was therefore to establish a clinically appropriate light-curing moment while achieving highest indirect tensile strength and lowest polymerization shrinkage. Hypotheses were that 1) an increase in temperature results in higher polymerization shrinkage and higher indirect tensile strength of resin composite cements and that 2) the light-curing moment of resin composite cements does not affect polymerization shrinkage or indirect tensile strength.

METHODS AND MATERIALS

Polymerization shrinkage of four dual-curing adhesive and three self-adhesive resin composite cements (Table 1, 2) was measured. The shrinkage of

Table 1: *Cement Materials Used*

Name	Manufacturer	Type	Monomers	Fillers	Lot.No.
Multilink Automix	Ivoclar Vivadent	Adhesive resin composite cement	Base paste: Bis-GMA, HEMA, 2-dimethylaminoethyl methacrylate Catalyst paste: ethyoxylated bisphenol A dimethacrylate, UDMA, HEMA	40 vol% - Barium glass - Ytterbium trifluoride - Spheroid mixed oxide Particle size: 0.25–3.0 μm	T33131
Multilink Speed CEM	Ivoclar Vivadent	Self-adhesive resin composite cement	Base paste: UDMA, TEGDMA, polyethylene glycol dimethacrylate Catalyst paste: polyethylene glycol dimethacrylate, TEGDMA, Methacrylated phosphoric acid ester, UDMA	40 vol% - Barium glass - Ytterbium trifluoride Particle size: 0.1-7 μm	U49017
RelyX Ultimate	3M ESPE	Adhesive resin composite cement	Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers Catalyst paste: methacrylate monomers	43 vol% - Silanated fillers - Alkaline (basic) fillers Particle size: 13 μm	595287
RelyX Unicem 2 Automix	3M ESPE	Self-adhesive resin composite cement	Base paste: phosphoric acid modified methacrylate monomers, bifunctional methacrylate Catalyst paste: methacrylate monomers	43 vol% - Alkaline (basic) fillers - Silanated fillers Particle size: 12.5 μm	594259
Panavia V5	Kuraray	Adhesive resin composite cement	Paste A: Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate, Hydrophilic aliphatic dimethacrylate Paste B: Bis-GMA, Hydrophobic aromatic dimethacrylate, Hydrophilic aliphatic dimethacrylate	38 vol% - Silanated barium glass filler - Silanated fluoroalminosilicate glass filler - Colloidal silica - Silanated aluminum oxide filler Particle size: 0.01-12 μm	720007
Panavia SA plus	Kuraray	Self-adhesive resin composite cement	Paste A: 10-MDP, Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate, HEMA Paste B: Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate	40 vol% - Silanated barium glass filler - Silanated colloidal silica Particle size: 0.02-20 μm	5F0046
VITA Adiva F-Cem	VITA Zahnfabrik	Adhesive resin composite Cement	Methacrylates	41 vol% -Inorganic fillers Particle size: 0.05-1 μm	17601812

Abbreviations: 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA: bisphenol A-glycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethyleneglycol dimethacrylate.

autopolymerized specimens was recorded at temperatures of 23°C (23a) and 37°C (37a). A temperature of 23°C was chosen as the room temperature at which most *in vitro* studies are performed and 37°C represents the intraoral temperature. Light-curing was performed for cements at 23°C and 37°C 1 minute after mixing (23L1, 37L1) and at 37°C after 5 minutes (37L5) and after 10 minutes (37L10). Measurements were recorded at 23°C for 12 hours and at 37°C for 2 hours for all cements until their shrinkage process was completed. To evaluate the influence of temperature and curing mode on the strength of the cements, the indirect tensile strength of all cements was measured after air storage at either 23°C or 37°C. The specimens were autopolymerized or light-cured after 1, 5, or 10 minutes. To illustrate filler size and structures, SEM images of all cements were captured.

Table 2: *Group Identifiers for the respective Cements*

MLA	Multilink Automix
MSC	Multilink Speed CEM
RUL	RelyX Ultimate
RUN	RelyX Unicem 2 Automix
PV5	Panavia V5
PSA	Panavia SA plus
VAF	VITA Adiva F-Cem

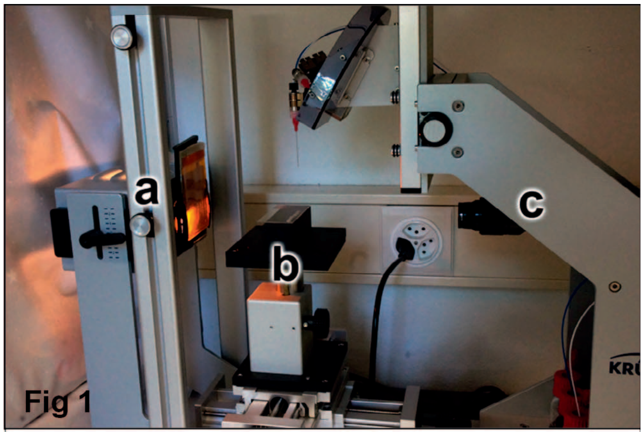

Figure 1. *Polymerization shrinkage was recorded with a drop-shape analyzer. (a) Light source with orange foil to prevent inadvertent light-activation of the cement. (b) Adjustable recording stage in which the cement was placed on a glass slide. (c) Optical system.*

Figure 2. *Cement drop image of a randomly selected specimen before and after shrinkage (PSA 23a specimen 5 [6.22% shrinkage]).*

Polymerization Shrinkage

Polymerization shrinkage data was recorded with a drop shape analyzer (DSA) (DSA305, Krüss, Hamburg, Germany). The intended use of a DSA is to determine the contact angle of a drop of liquid on a solid surface. A drop is automatically placed on the substrate on an adjustable stage and images of the silhouette are captured with the unit's camera, by which the contact angle of the drop can be measured (Advance, 1.2.0.1, Krüss) (Figure 1). To determine the polymerization shrinkage of the cements, the DSA images were obtained within a programmed time frame. The images were then exported and the area of the drops was measured digitally (Leica Application Suite, 4.7.0, Leica Microsystems, Heerbrugg, Switzerland) (Figure 2). Previously, spherical volume calculation of a two-dimensional image determined the cement's volume and consequently its shrinkage.[30] Isotropic shrinkage of the specimens was assumed. To correctly perform a volume calculation from a two-dimensional image, a perfectly semispherical cement specimen has to be produced. Since this was not possible, two-dimensional analysis of the cement area displayed in the image was chosen. To interpret the results, it must be considered that the shrinkage ratio between a semispherical volume ($V=2\pi r^3)/3$) and a semicircular area ($A=\pi r^2)/2$) was not proportional to the radius. The actual polymerization shrinkage of the volume was slightly higher than it appears in the area measurement. The difference between the shrinkage ratio of volume and area decreases with increasing radius. Therefore, to eliminate bias, similar cement drops with identical volume had to be produced.

Dental resin cement is sticky and viscous and additionally, hardens over time. Hence, the automatic syringe of the DSA could not be used to apply a defined amount of material. Consequently, the weight of the drops was determined during application with a scale (AT261, Mettler, Greifensee, Switzerland). The auto-mix tip was set on the cement syringe and time recording was started as soon as the first drop was discarded on a piece of paper. The second drop with a mean weight of 20.0 ± 2.0 mg was placed on a glass slide. The slide was then placed on the recording stage of the DSA and the focus of the optical system was adjusted. The DSA was covered with a dark box to prevent daylight from entering the stage. An orange foil was previously positioned between the sample area and the DSA's illumination source to prevent inadvertent light-activation of the cement.

For the 12 hour measurement groups (23a, 23L1), DSA recordings started one minute after the first drop left the mixing tip. The DSA was programmed to take an image of the cement drop once every 20 seconds for 10 minutes, then every 10 minutes for 50 minutes and finally every hour for 11 hours. For the 2-hour measurement groups (37a, 37L1, 37L5, 37L10), images were captured once every 20 seconds for 10 minutes, then every 10 minutes. Images were imported into Leica software (Leica Application Suite, 4.7.0, Leica Microsystems, Wetzlar, Germany). The area of each drop was recorded. The cement area of each image was compared with the area on the initial image that was taken 1 minute after the cement first left the syringe. With this data, the percent shrinkage was calculated and graphically displayed.

Measurements of all cements were recorded for autopolymerized specimens at 23°C over 12 hours (23a) and at 37°C over 2 hours (37a) in a temperature-controlled room (23°C). For measurements at 37°C, a heating device (IPX4, Thermo Technologies, Rohrbach, Germany) with constant temperature control (TSM125 H-Tronic, Hirschau, Germany) was placed on the DSA stage. Light-curing of the cements was performed at 23°C and 37°C. At both temperatures, light-curing was initiated after 1 minute (23L11, 37L1); at 37°C, light-curing was alternatively performed after 5 minutes (37L5) and 10 minutes (37L10). Light-curing was applied with a polymerization lamp (Elipar, 3M ESPE, Seefeld, Germany) having an intensity of 1200 mW/cm^2 for 20 seconds. The intensity of the lamp was checked before each measurement using the device provided by the manufacturer. Time was counted from the first moment the cement left the syringe for the discarded drop. Five specimens were measured in each group for all cements.

Indirect Tensile Strength

Indirect tensile strength of the cements was measured on cylindrical test specimens 3 mm in height and diameter (n=10). The cement was flowed into the respective cavities of a customized Teflon mold and kept in place with a plastic foil and a glass plate on each side. The specimens were either autopolymerized or light-cured (Elipar, 3M ESPE) at the respective times (after 1, 5, or 10 minutes) and immediately stored under temperature control (CTS T-4025, Hechingen, Germany) at 23°C or 37°C, respectively. After 1 hour, the specimens were removed from the molds and stored in a dark box for another 23 hours at the respective temperature. Prior to testing, the diameter and height were determined using a digital caliper (Cal IP 67, Tesa, Renens, Switzerland). The specimens were loaded radially until fracture with a preload of 20 N and a crosshead speed of 1 mm/min (Z020, Zwick/Roell, Ulm, Germany).

The following equation was used to calculate indirect tensile strength:

$$\sigma_t = 2F/\pi dh$$

where σ_t is the indirect tensile strength; F is the fracture load; d, the specimen diameter; and h, the specimen height.

Scanning Electron Microscopy

Discs with a diameter of 15 mm and a thickness of 1 mm were manufactured for all cements using a Teflon mold. The cement was flowed into the mold cavity and kept in place with a polyester foil and a glass plate on each side. Light-curing was performed with overlapping applications of a polymerization lamp (Elipar, 3M ESPE). All specimens were then fixed (UHU plus, UHU, Bühl, Germany) on a slide and simultaneously wet-polished with silicon carbide paper P1200-4000 (Type 401319, Exakt, Norderstedt, Germany). SEM images of gold-sputtered cement structures at 1000× were captured (Philips XL30 FEG ESEM, Philips Electron Optics, Eindhoven, The Netherlands).

Statistical Analysis

Data of all tests were analyzed for normal distribution using the Shapiro-Wilk test and all data were distributed normally. Hence, one-way ANOVA followed by the *post hoc* Fisher LSD test were chosen to test for differences between groups (α=0.05). Correlation between final polymerization shrinkage and the respective indirect tensile strength of each group was investigated.

RESULTS

Polymerization Shrinkage

Polymerization shrinkage of all cements within the different groups is displayed in Figure 3. Means and standard deviations of the final polymerization shrinkage after 12 hours for 23°C and after 2 hours for 37°C for the different groups are listed in Table 3. Final shrinkage ranged from 3.2% to 7.0%.

Indirect Tensile Strength

Indirect tensile strength values are graphically displayed in Figure 4. Values at 0 minute represent autopolymerized specimens. Means and standard deviations of all specimens at 23°C and 37°C are statistically compared in Table 4. Figure 5 plots all groups that were measured for the polymerization shrinkage to the respective indirect tensile strength.

Cement Filler Composition

SEM backscatter images of the cement surfaces are displayed in Figure 6 at a magnification of 1000×. A regular distribution of small filler particles (around 1 μm in diameter) was found for MLA and VAF. (See Table 2 for full names of these and other cements used.) Slightly larger particles (up to 5 μm in diameter) were detected for PV5. RUN and RUL revealed similar filler patterns: a mixture between small- and medium-sized particles up to 10 μm. PSA and MSC contained a mixture between small, medium, and large filler particles. For MSC, particle clusters up to 20 μm were detected. Particle sizes

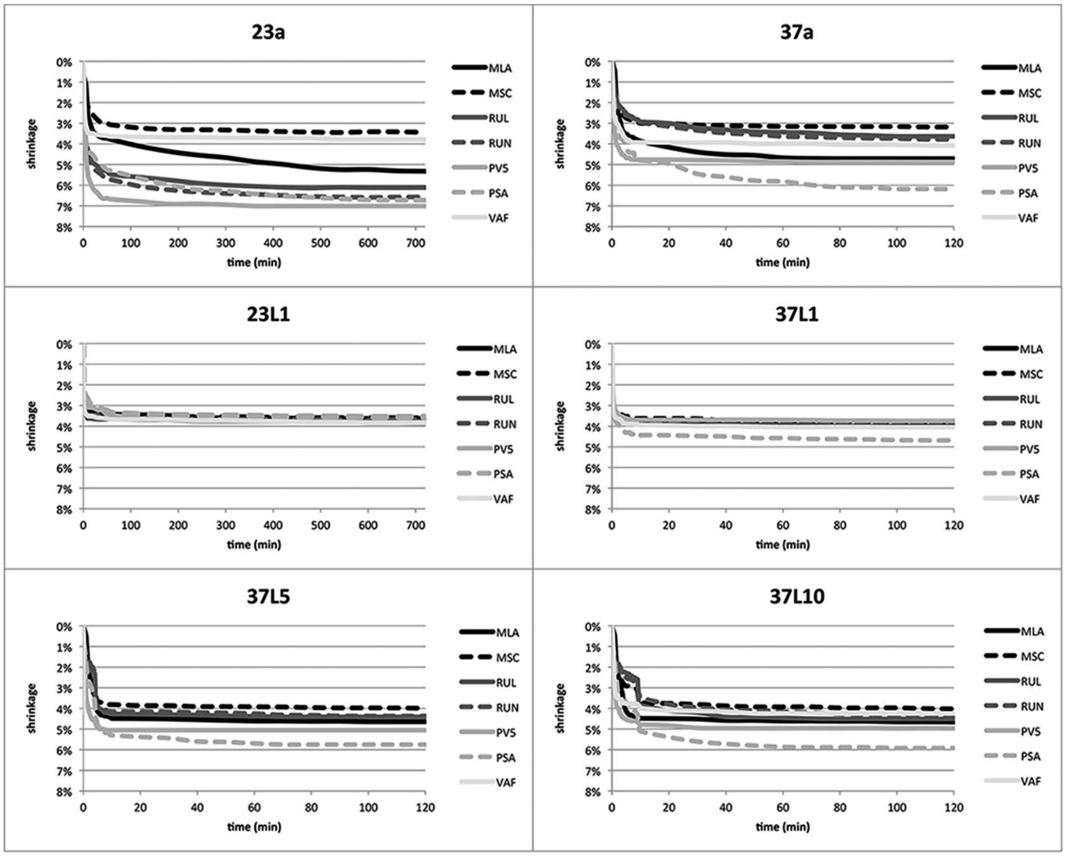

Figure 3. *Polymerization shrinkage of all cements within the different groups.*

reflected those provided by the manufacturer except for MSC, in which the filler sizes were twice the size described and PV5 that revealed rather smaller filler sizes.

DISCUSSION

The purpose of this study was to establish a clinically appropriate time of light-curing while achieving best material properties and lowest polymerization shrinkage. The first hypothesis, that an increase in temperature results in higher polymerization shrinkage and higher indirect tensile strength of resin composite cements was rejected because the increase of temperature resulted in different effects for each cement. The second hypothesis that the light-curing moment of resin composite cements does not affect polymerization shrinkage or indirect tensile strength was also rejected. Early light-curing after 1 minute was beneficial to decrease polymerization shrinkage for most cements. The effect of the light-curing moment on indirect tensile strength was also material related.

Table 3: *Means and Standard Deviations of Final Polymerization Shrinkage After 12 h for 23°C and After 2 h for 37°C of the Different Groups*[a]

	23a	37a	23L1	37L1	37L5	37L10
MLA	5.3% ± 0.1%^{A,a*}	4.7% ± 0.1%^{A,b}	3.6% ± 0.3%^{A,B,C,c}	4.0% ± 0.1%^{A,B,d}	4.6% ± 0.3%^{A,b}	4.7% ± 0.3%^{A,b}
MSC	3.5% ± 0.2%^{B,a,b}	3.2% ± 0.1%^{B,b}	3.6% ± 0.2%^{A,C,a,c}	3.8% ± 0.2%^{B,C,c,d}	4.0% ± 0.1%^{B,d}	4.0% ± 0.2%^{B,d}
RUL	6.1% ± 0.5%^{C,a}	3.6% ± 0.1%^{C,b}	3.8% ± 0.1%^{A,B,C,b}	4.0% ± 0.1%^{A,B,C,b}	4.4% ± 0.2%^{A,C,c}	4.5% ± 0.3%^{C,c}
RUN	6.6% ± 0.5%^{C,D,a}	3.8% ± 0.2%^{C,D,b}	3.7% ± 0.1%^{A,B,C,b}	3.9% ± 0.1%^{A,B,C,b}	4.4% ± 0.2%^{A,C,c}	4.4% ± 0.3%^{B,C,c}
PV5	7.0% ± 0.4%^{D,E,a}	4.9% ± 0.2%^{A,b}	3.5% ± 0.3%^{C,c}	3.7% ± 0.2%^{C,c}	5.1% ± 0.2%^{D,b}	5.0% ± 0.4%^{A,b}
PSA	6.7% ± 0.7%^{C,E,a}	6.2% ± 0.3%^{E,b}	3.9% ± 0.1%^{B,c}	4.7% ± 0.2%^{D,d}	5.8% ± 0.2%^{E,e}	5.9% ± 0.1%^{D,e}
VAF	3.8% ± 01%^{B,a}	4.1% ± 0.2%^{D,b}	3.8% ± 0.2%$^{A,B.a}$	4.1% ± 0.1%^{A,b}	4.2% ± 0.2%^{B,C,b}	4.2% ± 0.3%^{B,C,b}

[a] *Superscript letters indicate statistical similar groups (vertical comparison: uppercase letters; horizontal comparison: lowercase letters).*

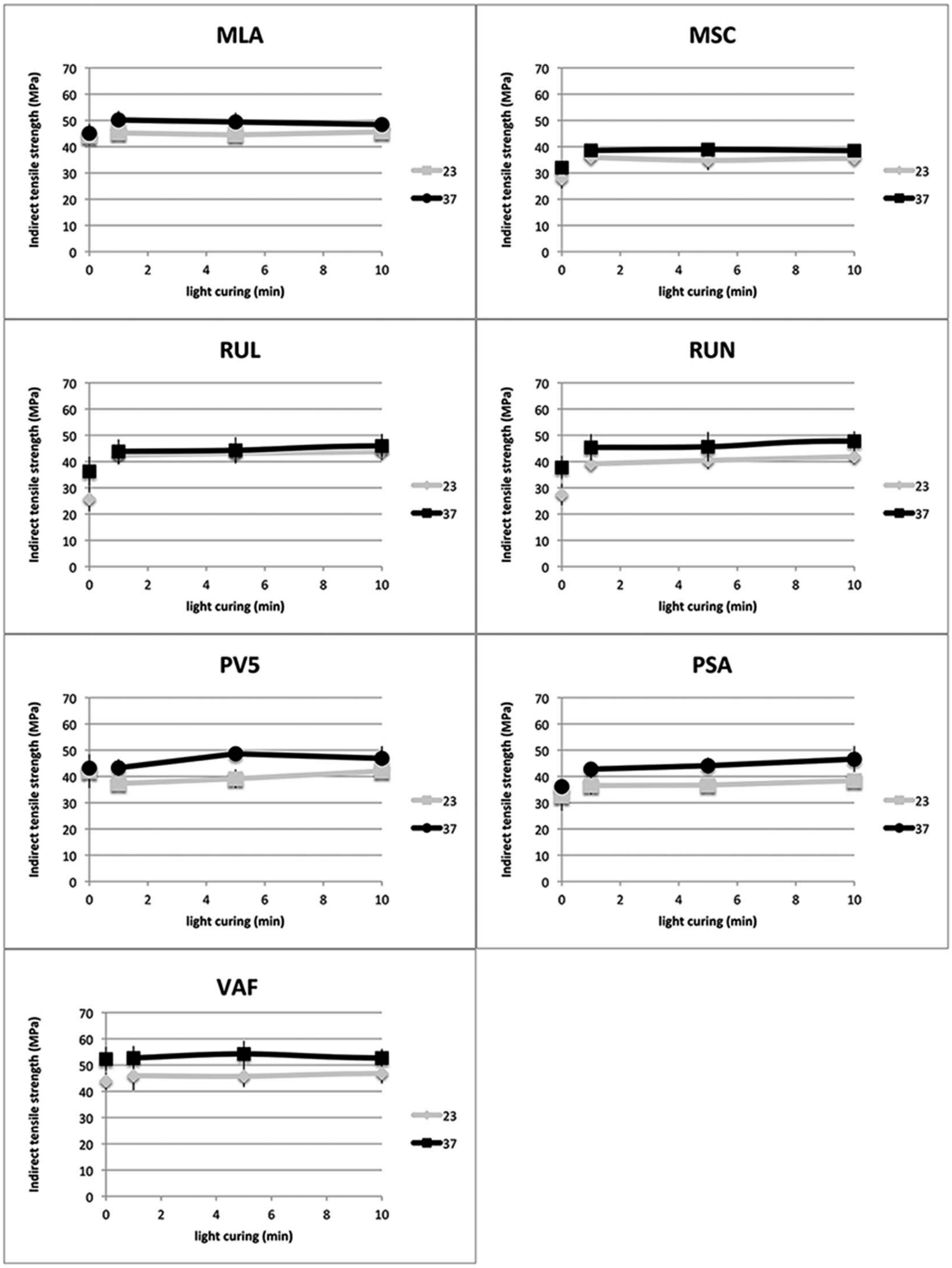

Figure 4. *Indirect tensile strength of all cements at 23°C and 37°C after autopolymerization (0 minute) or light-curing after 1, 5, or 10 minutes.*

Test Design

Numerous methods have been proposed to measure polymerization shrinkage kinetics; each has its disadvantages.[9,12,29,31] Results for the same material may differ between methods due to various testing parameters. Shrinkage strain for RUN (4.10 ± 0.03%) and MLA (4.65 ± 0.06%) at 23°C dual-cured have been reported in the literature but should not be compared with the area shrinkage values found in this study for these cements due to varying test setups.[12] The volumetric shrinkage of composites has been shown to be proportional to its degree of

Table 4: *Mean and Standard Deviation of Indirect Tensile Strength of All Cements at 23°C and 37°C of Autopolymerized and Light-Cured Specimens*[a]

(MPa)	**23a**	**23L1**	**23L5**	**23L10**	**37a**	**37L1**	**37L5**	**37L10**
MLA	$44.4 \pm 1.5^{A,a}$	$45.2 \pm 2.6^{A,B,a}$	$44.5 \pm 2.5^{A,a*}$	$45.6 \pm 2.4^{A,B,a,b}$	$45.1 \pm 3.6^{A,a}$	$50.2 \pm 3.4^{A,c}$	$49.4 \pm 3.5^{A,c}$	$48.4 \pm 2.7^{A,b,c}$
MSC	$27.8 \pm 3.8^{B,a}$	$35.2 \pm 1.9^{C,b,c}$	$34.8 \pm 3.7^{B,b,c}$	$35.5 \pm 1.8^{C,b,d}$	$32.0 \pm 4.6^{B,c}$	$38.6 \pm 3.1^{B,d,e}$	$38.9 \pm 2.9^{B,e}$	$38.5 \pm 2.2^{B,d,e}$
RUL	$25.7 \pm 4.7^{B,a}$	$42.4 \pm 3.4^{B,D,b}$	$43.1 \pm 3.1^{A,C,b}$	$43.8 \pm 3.2^{B,D,b}$	$36.3 \pm 5.6^{B,C,c}$	$43.9 \pm 4.6^{C,b}$	$44.3 \pm 5.0^{C,b}$	$46.0 \pm 4.5^{A,b}$
RUN	$27.4 \pm 4.1^{B,a}$	$39.1 \pm 2.3^{D,E,b}$	$40.4 \pm 3.3^{C,D,b}$	$41.9 \pm 2.8^{D,b,c}$	$37.7 \pm 4.4^{C,b}$	$45.4 \pm 5.0^{C,c,d}$	$45.6 \pm 5.6^{A,C,c,d}$	$47.8 \pm 3.8^{A,d}$
PV5	$41.9 \pm 6.3^{A,a,b}$	$37.3 \pm 2.3^{C,E,c}$	$39.1 \pm 3.6^{D,E,a,c}$	$42.0 \pm 2.6^{D,a,b}$	$43.2 \pm 5.3^{A,a,b,d}$	$43.3 \pm 3.3^{C,b,d}$	$48.6 \pm 2.7^{A,e}$	$46.9 \pm 4.6^{A,d,e}$
PSA	$32.5 \pm 5.6^{C,a}$	$36.5 \pm 3.4^{C,E,b,c}$	$36.7 \pm 2.7^{B,E,b,d}$	$38.2 \pm 2.1^{C,b,d}$	$36.1 \pm 2.7^{B,C,a,c,d}$	$42.8 \pm 2.8^{C,e}$	$44.1 \pm 3.6^{C,e,f}$	$46.4 \pm 4.4^{A,f}$
VAF	$43.9 \pm 3.2^{A,a}$	$46.0 \pm 5.6^{A,a}$	$45.7 \pm 4.1^{A,a}$	$46.8 \pm 3.8^{B,a}$	$52.3 \pm 4.6^{D,b}$	$52.7 \pm 4.5^{A,b}$	$54.2 \pm 5.0^{D,b}$	$52.7 \pm 3.4^{C,b}$

[a] *Superscript letters indicate statistical similar groups (vertical comparison: uppercase letters; horizontal comparison: lowercase letters).*

conversion.[32] The area shrinkage measurement in this study is easy to perform and allows monitoring of the shrinkage over a long time period. To interpret the results, it must be considered that the shrinkage ratio between a semispherical volume and a semicircular area is not proportional to the radius, therefore the actual polymerization shrinkage of the volume is slightly higher than it appears in the area measurement. Also, the actual polymerization shrinkage in a clinical situation may differ since the cement layer is thinner and not exposed to oxygen in most areas. The presence of oxygen at the cement surface inhibits the polymerization process.[33] Also, clinically, the cement is confined between a restoration and the tooth surface and the configuration-factor was not considered in the present study. Testing these luting materials away from the tooth structure cannot lead to solid conclusions, as the interaction between the different adhesive systems and luting cements or between the moist dentin and cements might affect their properties, hydrolytic stability, or strength.

When measuring dual-curing materials, the time of the first contact between the base and catalyst, the exact light-curing moment, as well as the starting time of the measurement must be standardized to achieve reproducible results. Since area shrinkage measurements cannot completely predict resin composite cement behavior regarding stress development,[10] further investigations are to be performed.

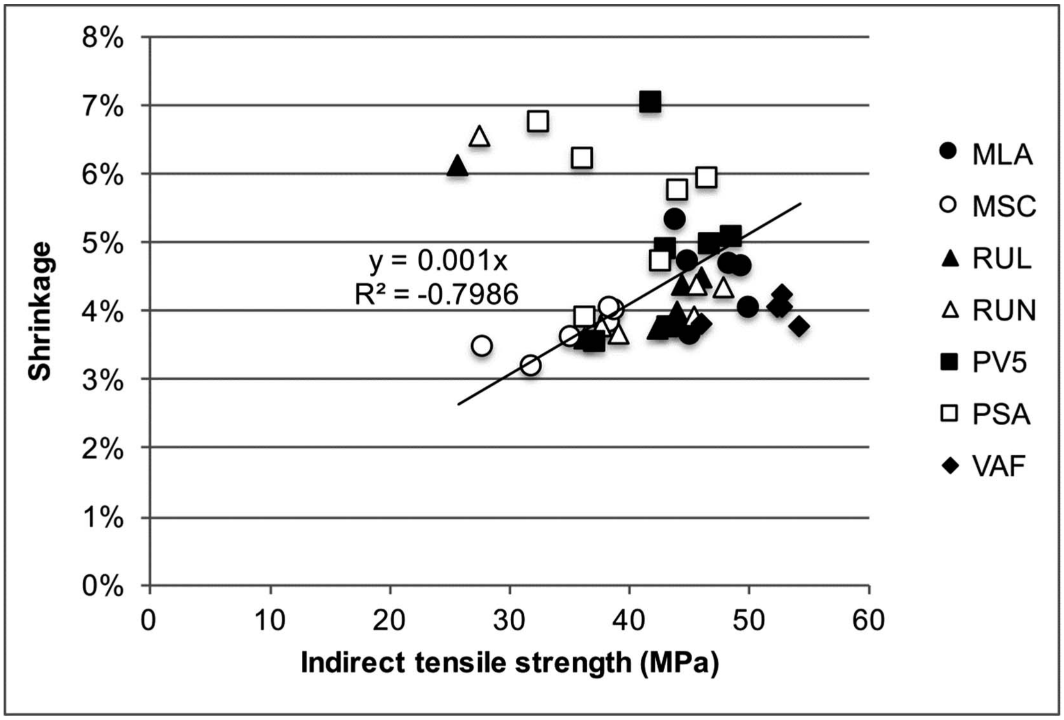

Figure 5. *Correlation between polymerization shrinkage and indirect tensile strength of the respective groups of all cements.*

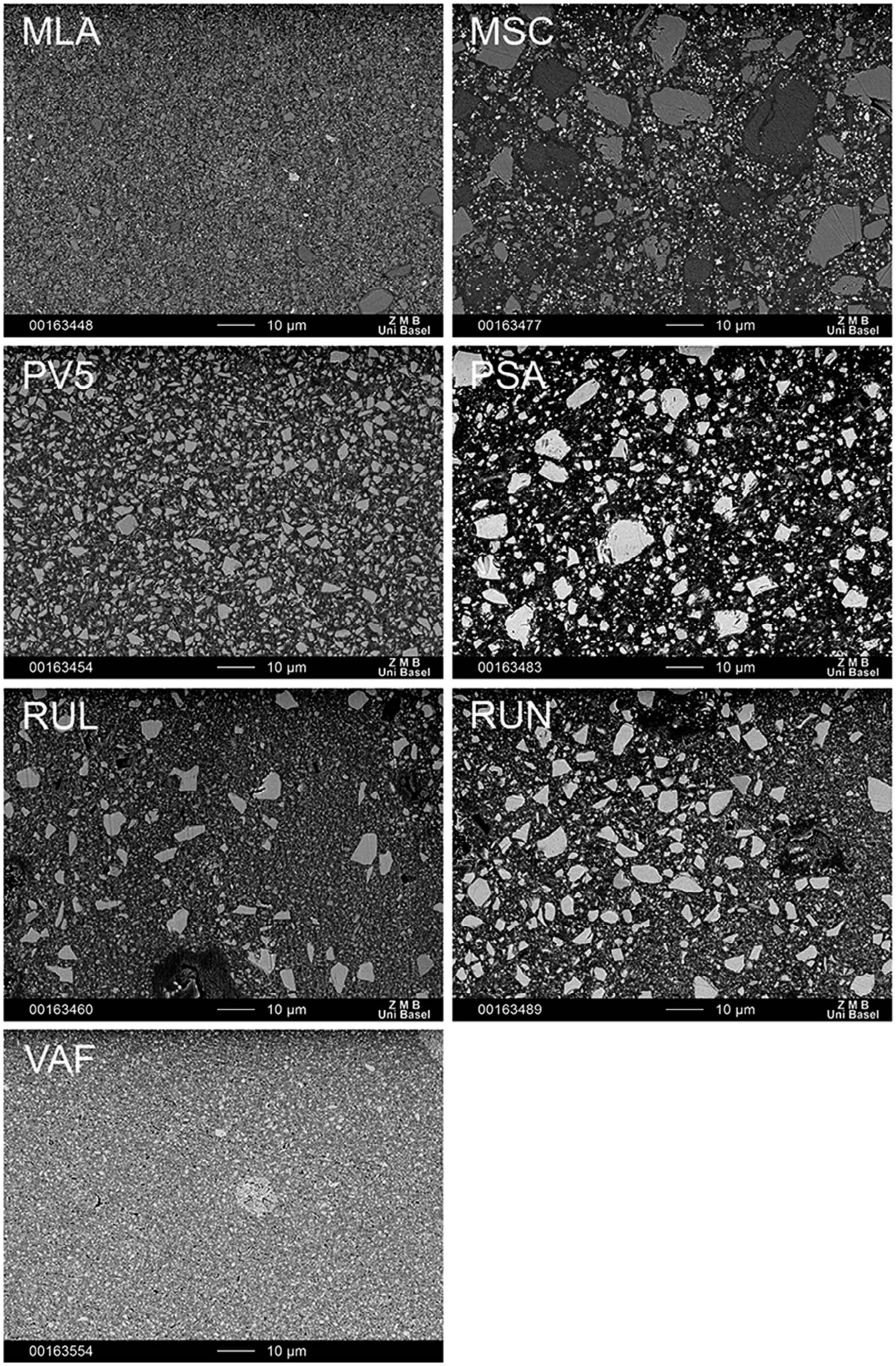

Figure 6. *SEM backscatter images of cement surfaces (1000×).*

Measuring the indirect tensile strength can be considered a standard method of screening material strengths.[34,35] The test provides reliable information on the mechanical strength of resin composites with the advantage of easy handling.[35-37]

Polymerization Shrinkage and Indirect Tensile Strength

Light-curing temperature effects on polymerization shrinkage and indirect tensile strength are material related. No correlation was observed between polymerization shrinkage and indirect tensile strength of the respective groups ($y=0.001x$, $R^2=-0.7986$; Figure 5). Several factors such as filler size and material, degree of conversion, initiator system, and cement monomer effect polymerization shrinkage and strength.[10] Some cements (MSC, RUL, RUN) revealed a strong dependence of the initiator system on light-curing, indicated by the significantly lower polymerization shrinkage as well as lower indirect tensile strength of autopolymerized specimens at 37°C than that of light-cured specimens. Increased temperature decreased the polymerization shrinkage of autopolymerized specimens MLA, RUL, RUN, and PV5 hastened activation of the initiator and, therefore, also ended polymerization sooner (Figure 3). In the literature, increased temperature was associated with a higher polymerization stress rate and a higher degree of conversion for light-cured resin composites[21,38] because of increased free radical and monomer mobility.[39,40] A decrease in viscosity of the cement due to the higher temperature also results in a higher collision frequency of the unreacted active groups.[41]

For light-cured specimens at 1 minute, an increase in temperature from 23°C to 37°C also increased the polymerization shrinkage of all cements. This was probably due to the polymerization process of the specimens being in a more advanced state when the light was applied at 37°C than it was at 23°C because of the enhanced energy supply. The application of light resulted in a freezing of the system: the cement became rigid and was unable to shrink further.

Indirect tensile strength of the cements was lowest for all cements when the specimens were stored at 23°C and no light was applied. The indirect tensile strength was enhanced when either the temperature was increased or light-curing was performed. The degree of the effect was material related.

The cement providing the highest indirect tensile strength (54.3±5.0 MPa) and lowest polymerization shrinkage (4.2%±0.2%) at 37°C with light-curing after 5 minutes was VAF. In addition, MSC demonstrated a low polymerization shrinkage of 4.0 ± 0.1%, but this cement revealed the lowest indirect tensile strength of all cements (38.9±2.9 MPa). Regarding the filler size, the highest indirect tensile strengths were observed for the materials containing a homogeneous distribution of small fillers of about 1 μm (MLA, PV5, VAF).

Clinical Implications

For clinical application, the following procedures are recommended to achieve best material properties: For MLA, MSC, RUL, and RUN, light application should be performed as soon as possible within the first 5 minutes after placing the restoration. For these materials, indirect tensile strength at 37°C was not significantly influenced by the time of light application but polymerization shrinkage was smaller when the light was applied earlier. Polymerization shrinkage for these cements ranged around 4%. It has been speculated that a delay in light-activation of dual-cured resin cements would enhance their mechanical properties,[42] although no effect was observed on the bond strength of resin cements to the substrate when the light-curing was delayed for 5 minutes.[43] In the present study, a delayed light-curing of 5 minutes for PV5 and 10 minutes for PSA was beneficial and resulted in an increase in their indirect tensile strength. However, it has been found that prolonged self-curing of the cements of 10 minutes may compromise the overall degree of conversion[28] and increase water sorption.[44] These findings must be interpreted with care and should not be generalized because the present study revealed that resin composite cements differ greatly in their curing behavior. PSA revealed slow polymerization reactions according to Figure 3, especially for autopolymerization. This might have been caused by the content of 10-methacryloyloxydecyl dihydrogen phopshate (10-MDP). Although 10-MDP enhances bond strength, it also inhibits the polymerization reaction.[45] 10-MDP is an acidic monomer that interferes with the amine initiator and therefore negatively affects the cement degree of conversion[46] in both autopolymerization and dual-curing mode.[47]

If polymerization shrinkage must be decreased for PV5 or PSA, an early light application can be performed, but it might result in insufficient polymerization. PV5 and PSA displayed higher polymerization shrinkage of 4% to 6%. For VAF, light application does not increase the strength or polymerization shrinkage (4%) significantly and may be omitted. For the other cements, light application is essential to increase strength: even when performed 10 minutes after mixing, it still increased the material's strength between 4 and 11 MPa. The application of a high-strength cement such as MLA, PV5, or VAF is recommended for cementing silicate ceramics to improve the stability of the restorations.[48]

CONCLUSIONS

The magnitude of the effect of light-curing and temperature increase on polymerization shrinkage and indirect tensile strength of resin composite cements is material dependent and cannot be generalized.

The tested resin composite cements provided polymerization shrinkages of 4% to 6% at 37°C with light-curing after 5 minutes. To keep shrinkage at a minimum, light application can be performed as soon as possible within the first 5 minutes after restoration placement.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial or other personal interest of any nature or kind in any product, service and/or company that is presented in this article, except for the following: Jens Fischer is Head of the Division of Dental Materials and Engineering at University of Basel. Dr Fischer is also Head of Research & Development at VITA Zahnfabrik.

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