Influence of Curing Mode with a LED Unit on Polymerization Contraction Kinetics and Degree of Conversion of Dental Resin–Based Materials

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ABSTRACT

Purpose: The purpose of this study was to evaluate the influence of photopolymerization mode with a light emitting diode (LED) lamp on the curing contraction kinetics and degree of conversion of 3 resin-based restorative materials.

Methods: The curing contraction kinetics of Admira (ADM), Filtek P60 (P60), and Filtek Flow (FLO) were measured by the glass slide method. The materials were exposed to light from a 1,000 mW/cm⁻² power LED lamp (Elipar Freelight 2) in 3 modes: 2 continuous modes of 20 and 40 seconds (C20 and C40), and 1 exponential mode (E20; 5 seconds of exponential power increase followed by 15 seconds of maximum intensity). The degree of conversion (DG) was measured for each of the materials, and each of the modes by Fourier transformed infra-red spectrometry.

Results: P60 had the significantly lowest final contraction and FLO the highest among all light exposure modes. The C20 and C40 modes did not produce any difference in contraction or degree of conversion. The E20 mode led to a significant slowing of contraction speed combined with greater final contraction.

Conclusions: Use of a LED lamp (1,000 mW/cm2) in continuous mode reduces the exposure time by half for identical curing shrinkage and degree of conversion. (J Dent Child 2009;76:149-55)

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The use of composite resin-based materials and adhesive techniques now forms part of the range of everyday treatments. The improvement in the mechanical and esthetic properties of these materials has greatly widened their indications over recent years. During photopolymerization, however, a major problem continues to be establishment of shorter covalent C-C bonds than what the Van De Waals bonds present initially between the monomers.¹ This results in contraction of the restoration volume, and the forces produced in the material, adhesive system and hard tooth tissues are potentially damaging to the material-dental tissue interface zone.² Conversion is also incomplete, and many monomers which have not reacted remain within the polymer network, damaging the mechanical qualities of the closure and being potentially toxic to surrounding tissues.³⁻⁸

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Table 1. Materials Used in This Study					
Material	Туре	Main components*			
Admira (Voco, Cuxhaven, Germany)	Ormocer (organically modified ceramic)	Monomers: Bis-GMA, di-UDMA, TEGDMA Fillers (78% weight, 56% volume): Ba-Al-B-silicate glass (90% - particle size range=0.7 μm), SiO2 (10%) 3-dimensionally curing organic-inorganic copolymers			
Filtek P60 (3M ESPE Dental Products, St. Paul, Minn)	Resin-based com- posite	Monomers: Bis-GMA, UDMA, Bis-EMA Fillers (81% weight, 61% volume): ZrSiO4 (particle size range=0.01-3.5 µm)			
Filtek Flow (3M ESPE Dental Products)	Resin-based com- posite	Monomers: Bis-GMA, TEGDMA Fillers (47% vol.): ZrSiO4 (particle size range=0.01-6.0 μm; average particle size=1.5 μm)			

* Bis-GMA=Bisphenol-A diglycidylether dimethacrylate; UDMA=urethane dimethacrylate; TEGDMA=triethyleneglycol dimethacrylate; Bis-EMA=Bisphenol-A polyethylene glycol diether dimethacrylate

Two phases are seen during polymerization. The first is a short, pregel phase during which the material is still able to flow and permits dissipation of forces.⁹ After the gel point, flow ceases and the rigid polymer network is subjected to contraction forces.^{10,11} Reducing these contractions and curing forces, therefore, remains a challenge to preserve the key closure/tooth zone.

Two major approaches are open to resolve this: firstly formulation of new composites¹²⁻¹⁵ and secondly changing how existing materials are used¹⁴⁻¹⁵. For the latter approach, placement of resin in increments has first been evaluated and recommended to reduce polymerization shrinkage stresses created at the restorative material/tooth interface.¹⁶⁻¹⁹ Then, the influence of the type and/or mode of photopolymerization has been thought to be of interest. The ideal situation would be to combine a high degree of monomer conversion into polymers for mechanical and compatibility reasons, with reduced curing shrinkage. These 2 factors, however, are closely linked, as polymerization contraction depends on the conversion of acrylic double bonds into covalent bonds.

Variations in curing contraction of a given composite resin according to photopolymerization mode can, therefore, be thought of as variations in conversion.²⁰⁻²³ Various studies have shown that reducing the light energy density (defined as the intensity of light multiplied by the exposure time)²⁴ leads to identical conversion and contraction to that achieved with higher light intensity.^{20,21,23-25} Differences in the formation kinetics of the 3-dimensional network, depending on photopolymerization mode, influence the conversion and contraction of a resin-based composite material.^{24,27,28} Different light energy sources can be used for photopolymerization. LED lamps have advanced considerably over recent years. The most important part of an LED lamp is the semiconductor chip in the bulb's center. This chip has 2 regions separated by a junction. The P region is dominated by positive electrical charges, and the N region is dominated by negative charges. The junction behaves as a barrier to electron flow between N and P. With sufficient acceleration voltage, the current can pass and electrons can cross the junction to reach P. Once attracted by positive charges, they bind.

Whenever electrons bind to a positive charge, the electrical potential energy is converted into electromagnetic energy. For each binding event, a quantum of electromagnetic energy is, therefore, emitted in the form of a light photon at a frequency that is characteristic of the semiconductor material. The emitted light's color is determined by the semiconductor's chemical composition.²⁹⁻³¹ As it emits monochromatic light, almost all of the power released by an LED lamp can be transformed into light irradiation in the desired form. The irradiation can then be targeted on the absorption spectrum of camphoroquinone, which minimizes heat production and does not, as is the case with a halogen lamp, require filters to be used.

The purpose of this study was to evaluate the influence of polymerization mode with a LED lamp on contraction and contraction kinetics, and the degree of conversion of three composite resins used in conservational odontics.

METHODS

The resin-based composites used in this study are shown in Table 1.

POLYMERIZATION SHRINKAGE KINETICS

Polymerization shrinkage kinetics were measured with the deflecting disk technique first developed by Wilson in 1978 and described by Watts and Cash.³² The polymer tested was a disk-shaped specimen (1.6 x 8 mm), placed in a 1.6 mm high brass ring with an internal diameter of 15 mm, adhesively bonded onto a rigid glass microscope slide (75 x 25 x 1 mm), with a sandblasted surface. A flexible glass cover slip (0.1 mm thick) was placed on

(%volume)					
Material	Curing mode	Shrinkage means % (SD)			
ADM	C20	2.62 (±0.04) ª			
	C40	2,56 (±0.05) ^a			
	E20	2.77 (±0.03) ^b			
P60	C20	1.73 (±0.03) °			
	C40	1.77 (±0.03) °			
	E20	$2.00 (\pm 0.01)^{d}$			
FLO	C20	3.89 (±0.03) °			
	C40	3.90 (±0.02) °			
	E20	3.98 (±0.05) ^f			

Results with the same superscript letter are not statistically different. the ring rim in contact with the test material, and a centrally aligned linear vertical displacement transducer (LVTD GTX 2500, RDP Electronics, Wolverhampton, UK) was placed carefully in contact with the cover slip's upper surface.

The room temperature was maintained at 23±1°C with a relative humidity of 50%. Using a LED lamp (Elipar Freelight, 3M ESPE Dental Products, St. Paul, Minn), polymerization was initiated by the guide light from below the unset disk specimen. Five samples of each material were cured for 20 seconds (C20) and 40 seconds (C40) in conventional continuous mode and for 20 seconds in exponential mode (E20; 5 seconds of ramped activation and 15 seconds at maximum power). Light activation energy was controlled to ensure a value of 1,000 mW/cm² with a Power Max 500A radiometer (Molectron Detector, Inc, Portland, Ore). For each specimen, the cover slip was attracted axially downwards as shrinkage took place. Cover slip displacement was recorded over the time. According to Watts and Cash,32 as displacement of the disk upper surface is uniform, measurements at the center are representative of the whole system. Cover slip displacement was recorded every 500 ms for a total time of 180 seconds.

The shrinkage-strain, $\boldsymbol{\epsilon}(t),$ was expressed as a percentage, according to the equation:

$\varepsilon(t)_{\text{wool}} = 100 \mathrm{x} \Delta L/L_{o}$

In this equation, L_0 is the initial specimen light, and ΔL is the cover slip displacement in μ m. Shrinkage kine-tics were calculated from the curves obtained for the 0- to 3-second, 3- to 10-second, 10- to 20-second,

Material	Curing mode	Polymerization kinetics between :					
		0-3s	3-10s	10-20s	20-40s	40-60s	
ADM	C20	0.281 (±0.021)	0.131 (±0.001)	0.041 (±0.001)	0.011 (±4.10 ⁻⁴)	0.004 (±1.10-	
	C40	0.294 (±0.012)	0.118 (±0.002)	0.036 (±0.001)	0.012 (±1.10 ⁻⁴)	0.005 (±1.10 ⁻	
	E20	0.167 (±0.009)	0.182 (±0.007)	0.048 (±0.003)	0.013 (±0.002)	0.004 (±6.10-	
P60	C20	0.210 (±0.003)	0.077 (±0.002)	0.025 (±0.001)	0.007 (±3.10 ⁻⁴)	0.003 (±2.10	
	C40	0.202 (±0.02)	0.081 (±0.008)	0.025 (±0.001)	$0.008 (\pm 5.10^{-4})$	0.004 (±2.10 ⁻	
	E20	0.094 (±0.02)	0.142 (±0.001)	0.034 (±0.004)	0.009 (±0.001)	0.003 (±2.10	
FLO	C20	0.228 (±0.016)	0.251 (±0.003)	0.084 (±0.001)	0.017 (±0.001)	0.005 (±1.10	
	C40	0.249 (±0.025)	0.241 (±0.008)	0.075 (±0.003)	0.020 (±0.001)	0.007 (±3.10 ⁻	
	E20	0.067 (±0.026)	0.294 (±0.007)	0.103 (±0.006)	0.021 (±0.002)	0.006 (±3.10	

Polymerization contraction kinetics are significantly different for each material (p<.0001).

Polymerization contraction kinetics are significantly different for the 0–3s, 3–10s and 10–20s periods (p< 0.05); the 20–40s and 40–60s periods are not different.

Polymerization contraction is significantly slower in E20 mode in the first 0–3s period (p< 0.05) for all the materials; the p-lymerization rate is higher in E20 mode for all the materials during the 3–10s and 10–20s periods (p< 0.05); the polymerization rate for each material is identical regardless of curing mode from the 20th second onwards.

Table 4. Degree of conversion mean (SD) (%)

Curing mode	Degree of conversion (SD)
C20	57.3 (±5.0) ^{a, b}
C40	57.5 (±5.4) ^{a, b}
E20	58.3 (±3.7) ^{a, b}
C20	54.9 (±4.6) ^b
C40	54.7 (±3.8) ^b
E20	57.0 (±4.0) ^b
C20	60.3 (±3.6) ª
C40	60.7 (±3.5) ª
E20	61.1 (±3.1) ^a

Results with the same superscript letter are not statistically different.

20- to 40-second, and 40- to 60-second periods. Data were analyzed statistically by analysis of variance (ANOVA) with Student-Newman-Keuls post hoc set test at the P<.05 level.

MEASUREMENTS OF CONVERSION DEGREE

Fourier transformed infrared spectroscopy (**FTIR**) was the proven method^{21,33,34} used in this case to measure the degree of conversion (**DC**) of the test materials.

Five samples of polymerized composite resin were prepared using the same method and the same photopolymerization modes, as described previously. 2 mg of each of the materials were taken with scalpel from deep within the sample immediately after polymerization and were then mixed with KBR (Spectra-Tech, Inc, Oak Ridge, Tenn). Pellets were prepared with a die (Specac 13-mm evocable pellet die, Specac, Inc, Smyrna, Ga) and paper inserts with a 10-mm internal diameter (Spectra-Tech Paper inserts, Spectra-Tech, Inc). The same technique was used to prepare 3 pellets of nonpolymerized material. The FTIR spectra of cured and uncured samples were recorded in transmission mode using a Nicolet protege 460 spectrometer (Nicolet, Inc, Madison, Wis). A total of 32 scans were measured at a resolution of 4 cm⁻¹. The DC was calculated on a relative basis from the aliphatic C-C peak at 1,637 cm⁻¹ and aromatic C-C at 1,608 cm⁻¹ from the cured (C) and uncured (U) samples, according to the following equation:

$$DC_{o} = (1 - C/U) \times 100$$

Data were analyzed statistically by ANOVA with Student-Newman-Keuls post hoc set test at the P<.05 level.

RESULTS

The mean polymerization shrinkage and polymerization shrinkage kinetics rates are shown in Tables 2 and 3. Statistically, and regardless of curing mode, P60 exhibited the least significant contraction and FLO the most. Remarkably, there was no significant difference between the C20 and C40 modes for any of the materials tested. All of the materials displayed the highest curing shrinkage in E20 mode. Polymerization contraction was significantly slower in E20 mode in the first 3-second period, and the polymerization rate was higher for all materials during the 3- to 10-second period. Curing speed for each material was identical, regardless of curing mode from the 20th second onwards.

The conversion measurement results are summarized in Table 4. No significant differences were found in DC between the 3 curing modes, regardless of the material.

DISCUSSION

Polymerization shrinkage is caused by the formation of shorter covalent bonds when the materials cure than the pre-existing Van der Waals bonds between the monomers. Reducing shrinkage, which causes stress and favors loss of cohesion between dental tissues and materials, remains a challenge.³⁵ Curing contraction is a complex process involving many interdependent factors: the number of covalent bonds formed, the nature of the initial monomers^{36,37}, the ratio, size, and type of charges¹⁰, the presence of binding agents¹³; the concentration of photoinitiators²², and the light energy supplied.²⁴

The polymerization shrinkage values of the different materials in our study fall within a range which is conventionally reported in the literature.³⁸ P60 exhibits the lowest final shrinkage. A leading factor involved in this result is its matrix composition: the Bis-EMA it contains is a monomer derived from Bis-GMA, in which some of the hydroxyl groups have been substituted.³⁹ Additionally, P60 contains UDMA, which has lower intrinsic viscosity than Bis-GMA but at higher molecular weight²⁵. The presence of these 2 combined monomers contributes to the lesser shrinkage of P6037,40. Similarly, FLO's matrix composition also partly explains its greater shrinkage. Dilution of high viscosity Bis-GMA monomers by lower viscosity, lower molecular weight TEGDMA leads to increased shrinkage. The smaller size of the TEGDMA monomers promotes molecular mobility, facilitating the formation of a large number of covalent bonds. This dilution appears essential, however, since-due to the system's low mobility-the Bis-GMA monomers are only weakly capable of forming C-C bonds.⁴¹⁻⁴³ The contraction results for ADM and its matrix containing Bis-GMA, di-UDMA, and TEGDMA are consistent with the aforementioned explanations.

The charge ratio and type of charges present in the material also play an important role. A direct relationship exists between the charge ratio and polymerization shrinkage. For equivalent composite volume, the lower the charge volume, the greater the matrix volume. As mineral loads are solids and do not contract, it is the resin which contracts because of monomer conversion and physical deformation due to the transition from gel to solid.44 FLO, with a 47% charge-volume ratio, shrinks more than P60 with a 61% ratio. In addition to charge ratio, charge size is also important. A small particle has a higher surface/volume ratio than a large particle. As a result, for the same charge volume and matrix volume, the larger the particle size the less resin is required to surround them and the higher the amount of resin between the particles. For smaller charges, the distance and mass of interparticle resin are reduced. This leads to fewer bonds and the formation of shorter chains, resulting in less contraction^{10,44}.

In addition, large particles allow light rays to better pass through the material and, thus, permit better polymerization and greater contraction.⁴⁵ These effects partly explain FLO's greater shrinkage, with its charge distribution up to 6µm compared to P60 and its larger charges of 3.5µm. ADM belongs to the ormocer (organically modified ceramic) family and has a complex composition. ADM can be thought of as being formed from an inorganic SiO₂ frame and shriveled into a ball. Onto this, polymerizable organic units with mineral charges that have an average diameter of 0.7µm, are integrated into the 3-dimensional structure, bonded through organomineral connecting parts. As charges make up 56% of the volume, the predetermined 3-dimensional network and high molecular weight of the ormocers undoubtedly explain the behavior of ADM between those of FLO and P60.

All of the materials were polymerized with an LED technology lamp; its emission spectrum centered closely on camphoroquinone, together with almost no heat produced, almost unlimited bulb life, low electrical consumption and capacity to polymerize efficiently like a halogen lamp^{31,46-48} have furthered its development and use over recent years. In our study, the Elipar Freelight 2 lamp has a high power of 1,000 mW/cm². Shrinkage kinetics and final contraction are similar for all of the materials polymerized in C20 and C40 modes. Adding a further 20 seconds illumination does not alter the behavior of the materials. This has also been reported in a recent study49 and can undoubtedly be explained by the rapid formation of a rigid polymer network, which, due to molecular immobility, no longer permits new bonds to be formed.

No difference in DC was seen between any of the ma-terials polymerized by C20 and C40 modes. Hence, the relationship between DC and total light energy does not appear to be linear. This is consistent with other studies.^{20,34} The use of high light intensity was driven by the need to obtain restorations with optimal mechanical properties. This is limited, however, by the longer-term adverse biocompatibility effect due to the release of

unpolymerized groups⁵⁰ and due to contraction forces and, consequently, the potential loss of material/dental tissue cohesion.⁵¹⁻⁵³ For this reason, "softstart" photopolymerizations were designed.

We noted the major influence of illumination mode on material behavior in our study. The exponential ramp in light intensity reduced initial contraction speed (0- to 3-second period) by almost half for ADM, more than half for P60, and by almost 75% for FLO. Conversely, for the 3- to 10-second period, polymerization speed accelerated markedly in E20 mode and fell for the continuous modes. Thereafter, from 20-second illumination onwards, speeds fell for each of the materials and were very similar regardless of mode. This can be explained firstly because lower initial light intensity (0-3 seconds) allows more gradual molecular mobility over time and, resulted in, slower formation of the polymer network. This produces favorable conditions for positioning molecules within the polymer chains during their formation. It even influences the spatial configuration of the network. Low light intensity only activates few photoinitiators and, therefore, leads to only few growth centers. Consequently, polymerization is propagated mostly by addition of 1 monomer molecule after another, resulting in relatively linear polymerization.²³ The 3- to 10-second period in E20 mode is an acceleration phase, during which the polymerization rate increases, although the number of reactive monomers falls. This phase is common to the multifunctional methacrylates because the reaction proceeds in a highly restrained environment.41

In our study, E20 mode which produces a slower initial reaction was associated with a type of polymerization catch-up. Later, for all of the materials and regardless of mode, the process decelerated progressively due to the increasingly difficult chain formation as the rigidity of the system increases with time. This deceleration is not due to the number of available monomers remaining but to the inability of the radical reaction to propagate. This explains why FLO with its matrix composition and lower charge ratio, giving it greater flow capacity, exhibited a less-pronounced and longer deceleration phase.

We also note that, as has recently already been reported,⁴⁹ the final contraction and the DC were slightly greater for all of the materials in E20 mode. This indicates a nonlinear relationship between irradiation dose and polymerization. As we have described, many complex factors are involved, but it is also important to consider the effect of high light energy on the photoinitiators. Activated photoinitiators drive the polymerization reaction, although they can also stop it by binding to each other or to the activated end of a polymer chain; this is the inactivation phenomenon.²⁴ High light intensity tends to promote this mechanism.

The advantages of slower contraction of material flow over time is promoted by irradiating in exponential mode (ie, potential dissipation of contraction forces), combined with a highly charged composite material whose mecha-nical qualities are endowed with a satisfactory DC in a shorter clinical time. This appears to be a combination that may reduce damage to the dental tissue/materials interface zone. Further studies are needed to evaluate specifically the tooth/materials interface under different light-cured conditions.

CONCLUSIONS

The desire for a material that is correctly polymerized in a short clinical time—with few residual monomers, exhibiting low curing contraction, and not producing high polymerization forces—currently requires compromise. To assist clinicians in these choices, different factors should be considered. The final degree of contraction is not a decisive factor. Contraction kinetics, however, may guide the practitioner. For this, the composition of the materials and their polymerization mode play a fundamental role.

From our results, the use of a low-viscosity resin combined with a highly charged material—polymerized with a high intensity LED lamp operated in exponential mode providing a satisfactory degree of conversion appears to be a useful clinical compromise.

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