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Stupanj konverzije i temperaturni porast kod kompozita polimeriziranih LED-uređajima različitog intenziteta

Degree of Conversion and Temperature Increase During Composite Polymerisation with LED Units of Different Intensity

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Sažetak

LED-uredaji za polimerizaciju kompozitnih materijala gotovo su potpuno zamjenili halogene polimerizatore. Njihov glavni nedostatak bio je porast temperature tijekom postupka. Raniji LED-uredaji postizali su neznatan temperaturni porast, a uredaji novije generacije, zbog visokog intenziteta svjetla, ostvaruju viši stupanj konverzije, ali dovode u pitanje porast temperature tijekom polimerizacije kompozita. **Svrha rada:** bila je izmjeriti stupanj konverzije i temperaturni porast u uzorku kompozitnog materijala polimeriziranog programima različitog intenziteta četiriju LED-uredaja. **Materijal i postupci:** U istraživanju su korišteni: Bluephase, Bluephase C5, Bluephase C8 i Bluephase 16i (svi uredaji - Vivadent, Schaan, Liechtenstein). Stupanj konverzije mјeren je spektrofotometrom FTIR, a za mјerenje temperature rabljen je termometar ELV Pyroscan (Leer, Njemačka). **Rezultati:** Najviši stupanj konverzije postignut je kod polimerizacije uzoraka kompozitnog materijala soft-start programom (SOFT) Bluephase 16i uredaja, a najniži kod polimerizacije uzoraka Bluephase C5 uredajem. Najviši temperaturni porast zabilježen je tijekom polimerizacije uzoraka programom visokog intenziteta (HIGH) i SOFT programom Bluephase 16i, a najniži u slučaju polimerizacije programom niskog intenziteta (LOW) Bluephase C8 polimerizatora. **Zaključak:** Prema dobivenim rezultatima može se zaključiti da veći stupanj konverzije vodi prema većem porastu temperature, bez obzira na vrstu kompozitnog materijala.

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Adresa za dopisivanje

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Uvod

Stupanj konverzije te temperaturni porast tijekom polimerizacije svjetlosno stvrdnjavajućih dentalnih materijala važni su parametri u procjeni učinkovitosti pojedinog uređaja za fotopolimeriza-

Introduction

Important parameters for assessing the efficiency of different curing units are the degree of conversion and increase in temperature during photopolymerization of light cured dental materials. Both

ciju. Oba parametra ovise o intenzitetu svjetla emitiranog iz fotopolimerizatora, spektru valnih duljina te trajanju osvjetljavanja.

Dosad najčešće rabljeni uređaji za fotopolimerizaciju kompozitnih materijala - halogene žarulje, danas su gotovo potpuno zamijenjene diodnim (LED) uređajima za polimerizaciju. Halogeni uređaji proizvode svjetlo žarenjem tungstenske niti. Veći dio tako stvorene energije manifestira se kao toplina, a vrlo mali postotak energije emitira se kao svjetlo potrebno za fotopolimerizaciju. Tijekom vremena jakost svjetla iz halogenog polimerizatora opada zbog više čimbenika, kao što su fluktuacije u volatži, uništenje bulbusa, uništenje ili prljavi filtri, kontaminacija izlaznog optičkog voda dentalnim materijalom, oštećenje neodgovarajućim postupkom čišćenja i održavanja samog uređaja. Prednost tih uređaja je u tome što emitirani spektar pokriva apsorpcijski spektar, ne samo kamforkinona nego i ostalih fotoinicijatora koji mogu biti u kompozitnoj smoli ili ostalim fotopolimerizirajućim restaurativnim materijalima.

LED-uređaji proizvode svjetlost elektroluminiscencijom, čime se proizvodi više učinkovitog svjetla uz minimalnu proizvodnju topline. Osim toga, za razliku od halogenih žarulja koje primarno proizvode bijelo svjetlo koje filtriranjem daje plavu boju potrebnu za polimerizaciju, ti uređaji odmah proizvode plavo svjetlo, pa se tako izbjegava uporaba filtera. Vijek trajanja im je nekoliko tisuća sati, za razliku od halogenih uređaja kojima se trajnost procjenjuje na 30 do 50 sati (1-4). LED-uređaji razvijali su se tijekom triju generacija te se u svakoj nastojao povećati intenzitet emitiranog svjetla uz što manji porast temperature.

Kao što su halogeni polimerizatori tijekom godina prilagođeni različitim polimerizacijskim programima zbog što potpunije polimerizacije i manjeg temperaturnog porasta, ista tendencija nastavljena je i s najnovijim LED-uređajima.

Ranije generacije imale su nešto niži stupanj konverzije (49 do 55%) s manjim temperaturnim porastom (1-3°C) u odnosu prema halogenim uređajima (1-3). Novije generacije LED-uređaja s većim izborom polimerizacijskih programa, jačim postignutim intenzitetom te većim stupnjem konverzije, u pitanje dovode porast temperature tijekom stvrđnjavanja kompozitnog materijala. Ranije generacije diodnih uređaja emitirale su uži svjetlosni spektar - on se kretao u sklopu apsorpcijskog spektra kamforkinona (oko 468 nm). Kasniji diodni uređaji proširuju spektar na 460 do 490 nm, a najnovije generacije

mentioned parameters are dependent on the light intensity emitted from the curing unit, spectrum of emitted wavelength as well as illumination time.

Halogen curing units, the most commonly used curing units for composite materials photopolymerization, are nowadays replaced almost completely by diode curing units (LED). Halogen curing units produce light by heating of the tungsten filament. A high amount of produced energy is manifested as heat, while a very low percentage is emitted as light appropriate for curing. Light intensity emitted from halogen curing unit decreases over time. This is caused by different factors like fluctuation in line voltage, deterioration of the light bulb, deterioration of the reflector or filter, contamination of the light guide, effects of disinfection procedures on the transmission of light through the light guide and malfunction of the photoconductive fibers in the light guide. This type of curing lights emit broad spectrum of wavelengths which cover not only camphorquinone, but other photoinitiators present in other light cured dental materials as well.

LED curing units use electroluminescence to produce light, which produces more useful light with minimal production of heat. Also, these curing units produce primarily blue light as compared to halogen units. Halogen units produce white light which is filtered in blue light. Therefore, LED does not require any filters. LEDs last for thousands of hours, whereas a conventional halogen light bulb lasts for only 30 to 50 hours (1-4). LEDs were developed through three generations with the aim to increase the light intensity without a significant increase in temperature.

The tendency of modifying the halogen curing units with different polymerization modes, in order to make more complete polymerizations with a lower increase in temperature, was continued with the latest LED curing units.

Previous generations of LED curing units made slightly lower degrees of conversion (49-55%), resulting in lower temperature increase (1-3°C) in comparison to halogen curing units (1-3). Newer generations of LED curing units offer a wide range of polymerization programs, a higher intensity, and consequently a higher degree of conversion. However, they also endanger the temperature rise during the hardening of composite material. Earlier generations of LED curing units emitted narrower light spectra, ranging in absorption spectrum of camforquinone (around 468 nm). Later generations of diode curing units are widening the spectra from

od 430 do 490 nm (4). Nekorisne niže valne duljine štetno utječu na oči terapeuta, a više valne duljine nepotrebno zagrijavaju kako kompozitni materijal tako i samo zubno tkivo (5,6).

Visok stupanj konverzije osigurava dobra fizičko-mehanička svojstva kompozitnog materijala ili ispuna, a posljedica je veće polimerizacijsko skupljanje i polimerizacijski stres (7,8). Viši stupanj konverzije obično je veći kod jačeg intenziteta polimerizacijskog svjetla te duže polimerizacije, no najbolje i jedno i drugo prati veći temperaturni porast (9). S druge strane, nedovoljna jakost svjetla i nedovarajuće vrijeme osvjetljavanja mogu rezultirati lošijom polimerizacijom te nepotpunom konverzijom monomera, posebice u dubljim dijelovima kaviteta. Posljedica je otpuštanje monomera u smoli, što može štetno utjecati na vitalno pulpno tkivo (9-12).

Kako bi se ublažile negativne posljedice polimerizacije kompozita, a istodobno osigurala dugotrajnost ispuna, danas je predmet mnogih studija primjena polimerizacijskih programa različitog intenziteta i različitog vremena osvjetljavanja, ovisno o kliničkoj situaciji.

Svrha ovog rada bila je odrediti stupanj konverzije i temperaturni porast tijekom polimerizacije uzorka kompozitnog materijala polimerizacijskim programima različitog intenziteta četiriju LED-uređaja.

Materijal i postupci

Mjerenje stupnja konverzije

Za ispitivanje stupnja konverzije rabljeni su kompozitni materijali Tetric Ceram (boja A3 – exp. 2008-03, lot. G06853) (TCA3) i Tetric EvoCeram (boja A2 – exp. 2010-01, lot. J04088) (TECA3) (oba materijala: Vivadent, Schaan, Liechtenstein). Uzorci kompozitnih materijala polimerizirani su programom Bluephase soft-start (SOFT), Bluephase C5, Bluephase C8 (program visokog intenziteta - HIGH, niskog intenziteta - LOW i SOFT program) i Bluephase 16i (programi HIGH, LOW i SOFT) LED-uređajem (svi Vivadent, Schaan, Liechtenstein). U Tablici 1. prikazani su jakost i vrijeme polimerizacije za pojedini uređaj i polimerizacijski program.

Pripremljeno je bilo ukupno 96 uzoraka - po 48 za svaki materijal; po 6 uzoraka od svakog materijala za svaki polimerizacijski program.

460 to 490 nm, while the latest generations have 430 to 490 nm spectra (4). Useless lower wavelengths have harmful effect on clinician's eyes, and higher wavelengths result in unnecessary heating of composite material and dental tissue as well (5,6).

A high degree of conversion ensures good physical-mechanical properties of composite material, or composite resin filling, but on the other hand, it leads to higher polymerization shrinkage and polymerization stress (7,8). The degree of conversion is usually higher with a high intensity polymerization program and longer polymerization time is used. Unfortunately, both factors are responsible for higher temperature rise (9). However, insufficient light intensity accompanied with insufficient light curing time, can lead to poor polymerization and incomplete conversion of monomer, especially in deeper parts of the cavity. Consequently, non-reacted monomer present in the resin can be released, which can be harmful for vital pulp tissue (9-12).

In order to minimize the negative consequences of composite polymerization and, at the same time, to ensure the longevity of composite filling, the use of polymerization programs with different light intensities and different curing times depending on the clinical situation, is the object of many present studies.

The purpose of this study was to determine the degree of conversion and the temperature rise in composite materials sample polymerized by curing modes of different light intensities from four LED curing units.

Material and Methods

The degree of conversion measurement

Composite materials used for analyzing the degree of conversion measurement were: Tetric Ceram (shade A3 – exp. 2008-03, lot. G06853) (TCA3) and Tetric EvoCeram (shade A2 – exp. 2010-01, lot. J04088) (TECA3) - (both Vivadent, Schaan, Liechtenstein). The composite resin samples were polymerized with Bluephase (soft-start polymerization mode (SOFT)), Bluephase C5, Bluephase C8 (high intensity mode (HIGH), low intensity (LOW) and SOFT mode) and Bluephase 16i (HIGH, LOW and SOFT mode) LED curing unit (all units Vivadent, Schaan, Liechtenstein). Table 1 presents intensity and polymerization time for each and polymerization mode used in the experiment.

A total of 96 samples were made, 48 for each material; 6 samples from each material were for each polymerization mode.

Tablica 1. LED-polimerizatori rabljeni u eksperimentu za polimerizaciju uzoraka kompozitnih materijala
Table 1 LED curing units used in the experiment for polymerization of composite resin samples

Polimerizacijski program • Polymerization mode	LED polimerizator/ LED curing unit			
	Bluephase	Bluephase C5	Bluephase C8	Bluephase 16i
Program visokog intenziteta • High intensity mode (HIGH)	-	-	800 mW/cm ² 20 s	1600 mW/cm ² 10 s
Program niskog intenziteta • Low intensity mode (LOW)	-	500 mW/cm ² 40 s	650 mW/cm ² 30 s	650 mW/cm ² 30 s
Soft-start polimerizacijski program • Soft-start polymerization mode (SOFT)	600 mW/cm ² prvih 5 s, 1100 mW/cm ² sljedećih 25 s • 600 mW/cm ² first 5 s, 1100 mW/cm ² next 25 s	-	650 mW/cm ² prvih 5 s, 800 mW/cm ² sljedećih 25 s • 650 mW/cm ² first 5 s, 800 mW/cm ² next 25 s	650 mW/cm ² prvih 5 s, 1600 mW/cm ² sljedećih 10 s • 650 mW/cm ² first 5 s, 1600 mW/cm ² 10 s

Uzorci kompozitnog materijala za mjerjenje stupnja konverzije pripremljeni su bili na sljedeći način: na foliju Mylar veličine 3x3 cm stavljen je uzorak nepolimeriziranoga kompozitnog materijala težine 40 mg, te je prekriven drugom folijom iste veličine. Zatim je sve stavljeno između dviju okruglih ploča od inoksa promjera 2 cm i stlačeno u ručnoj preši pod tlakom od 10⁷ Pa do debljine 0,1 mm. Tako pripremljen uzorak kompozitnog materijala polimeriziran je izvorom svjetla priljubljenog uz gornju foliju Mylar. Uzorci su mjereni nakon polimerizacije i odvajanja od folije.

Stupanj konverzije mјeren je FT-IR spektrometrom (Perkin-Elmer, model 2000, Beaconsfield, Ujedinjeno Kraljevstvo) u transmisijskom modu neposredno nakon polimerizacije uzorka. FT-IR spektri snimani su na sobnoj temperaturi (RT) u IR spektru od 4000 do 400 cm⁻¹, s rezulucijom 4 cm⁻¹ i 20 skenova po uzorku. Prosječno 2 mg nepolimeriziranog uzorka razrijedjeno je u ~100 mg spektroskopski čistog KBr-a te sprešano u male pastile koristeći se standardnom prešom s pritiskom od 5 t/cm². Za obradu spektra uzorka koristio se IRDM (IR Data Manager) program. Spektri su prebačeni u apsorpcijski mod te je stupanj konverzije bio određen standardnom metodom prema Rueggebergu (13):

$$\% \text{ konverzije} = (1 - (\text{polimerizirani uzorak} / \text{nepolimerizirani uzorak})) \times 100\%.$$

Mјerenje temperaturnog porasta

Za mјerenja temperature koristio se tvornički plastični kalup u obliku kruga promjera 2 cm i de-

The composite resin samples for the degree of conversion measurement were prepared as follows: a sample of unpolymerized composite material, 40 mg in weight, was placed on a Mylar sheet 3x3 cm in size, and covered with another Mylar sheet of the same size. After that, the sample was placed between two round inox plates, 2 cm in diameter, and pressed in a hand press at 10⁷ Pa to 0,1 mm thickness. Inox plates were used to keep the sample in the same position. After pressure applied in hand press, the inox plates were removed and the blue light source was applied on the upper Mylar sheet of the unpolymerized sample and polymerized. The samples were measured after polymerization and separation from the Mylar sheet.

The degree of conversion of the composites used in this study was measured using a FT-IR spectrometer (Perkin-Elmer, model 2000, Beaconsfield, UK) operating in transmittance mode immediately after the end of polymerization of the resin sample with curing device. The FT-IR spectra were taken at room temperature (RT) in the IR range 4000 - 400 cm⁻¹, with the resolution of 4 cm⁻¹ and 20 scans per sample. Approximately 2 mg of uncured samples were diluted in ~100 mg of spectroscopically pure KBr matrix in agate mortar and then pressed into small discs using a standard press with the pressure of 5 t/cm². The IRDM (IR Data Manager) program was used to process the obtained spectra. The spectra were converted into absorbance mode and the degree of conversion was determined by the standard method by Rueggeberg (13):

$$\% \text{ conversion} = (1 - (\text{polymerized sample} / \text{unpolymerized sample})) \times 100\%.$$

The temperature rise measurement

For the temperature measurement, a prefabricated plastic mould circular in shape, 2 cm in diameter

bljine 4 mm. U sredini je bio napravljen četvrtasti otvor veličine 4x4x4 mm, otvoren s gornje i s donje strane kalupa. Otvor je ispunjen kompozitnim materijalom te je na donju površinu uzorka kroz otvor na donjoj strani kalupa prislonjen termometar (ELV Pyroscan, Infrared Thermometer PF 1000, PS 140/ PS 300, Leer, Njemačka), a na gornju izvor svjetla. Osjetljivost rabljenog termometra iznosila je od -20°C do +300°C.

Pripremljen je bio isti broj uzoraka kao i za mjerjenje stupnja konverzije. Temperatura za svaki uzorak očitavala se na početku i kraju iluminacije, a razlika između početne i krajnje vrijednosti koristila se za daljnju statističku obradu.

Za statističku obradu podataka koristila se dekskriptivna statistika, jednosmjerna analiza varijance (ANOVA), post hoc Tukeyev test i t-test sparenih uzoraka.

Rezultati

Rezultati mjerjenja stupnja konverzije

Rezultati mjerena stupnja konverzije prikazani su u Tablicama 2. i 3.

ANOVA je pokazala statistički znatne razlike u stupnju konverzije kod obaju kompozitnih materijala, ovisno o vrsti polimerizatora i polimerizacijskog programa ($p<0,001$). U Tablici 3. su rezultati Tukeyeva post-hoc testa za razlike u stupnjevima konverzije kod osvjetljavanja različitim polimerizacijskim programima za materijale TC i TEC.

Najviši stupanj konverzije postignut je kod obaju kompozitnih materijala tijekom polimerizacije uzorka SOFT programom Bluephase 16i uređaja (TC 68,60%, TEC 64,55%), a najniži stupanj zabilježen je kod polimerizacije uzorka Bluephase C5 uređajem (TC 58,87%, TEC 57,31%).

Paired samples T-test upozorio je da postoje statistički velike razlike u stupnju konverzije između obaju kompozita osvjetljavanih istim svjetiljkama. Za Bluephase C8 HIGH $p=0,010$, LOW $p=0,015$; SOFT $p=0,004$, za Bluephase 16i uređaj HIGH $p<0,001$, LOW $p<0,001$, SOFT $p<0,001$, te za Bluephase C5 $p=0,035$ TEC ima statistički znatno niže stupnjeve konverzije od TC-a, a samo kod polimerizacije programom Bluephase SOFT nema razlika u stupnju konverzije između tih dviju vrsta kompozita.

and 4 mm thick, was used. In the center of the circle a quadrangular opening 4x4x4 mm was made, opened from upper and lower side of the mould. The opening was filled with composite material. A thermometer (ELV Pyroscan, Infrared Thermometer PF 1000, PS 140/ PS 300, Leer, Germany) attached on the lower sample surface through the opening in the mould, and a light source on the upper surface in the same manner. The thermometer sensitivity range used was from -20°C to +300°C.

The same number of samples as for the degree of conversion measurement were prepared. Temperature was recorded in the beginning and in the end of illumination for each sample. The difference between incipient and terminal value was used for further statistical analysis.

For statistical analysis of data descriptive statistical methods were used, a one-way variance analysis (ANOVA), post hoc Tukey test and paired samples t-test.

Results

Results of the degree of conversion measurements

Results of the degree of conversion measurements are shown in Table 2 and 3.

ANOVA showed statistically significant differences in the degree of conversion in composite materials depending on the polymerization device, as well as on the polymerization mode ($p<0,001$). In Table 3 the results of Tukey post-hoc test for differences in degrees of conversion for TC and TEC composite material illuminated with curing modes of different intensity are given.

The highest degree of conversion was achieved in the case of polymerization of both composite material samples with SOFT mode of Bluephase 16i curing unit (TC 68,60%, TEC 64,55%), while the lowest degree of conversion was recorded in the case of polymerization with Bluephase C5 curing unit (TC 58,87%, TEC 57,31%).

Paired samples T-test showed statistically significant differences in the degree of conversion between both composite materials illuminated with the same light sources: for Bluephase C8 HIGH $p=0,010$, LOW $p=0,015$; SOFT $p=0,004$, for Bluephase 16i curing unit HIGH $p<0,001$, LOW $p<0,001$, SOFT $p<0,001$, and for Bluephase C5 $p=0,035$. TEC has a statistically significant lower degree of conversion than TC, and only in the case of polymerization using Bluephase SOFT mode. No statistical significant difference in the degree of conversion between tested composite materials was found. With all oth-

Tablica 2. Rezultati mjerjenja stupnja konverzije
Table 2 Results of the degree of conversion measurements

Kompozitni materijal • Composite material	LED polimerizator • LED curing unit	Stupanj konverzije (srednja vrijednost) • Degree of conversion (mean value) (%)	Std.Dev.	p*
TC	Bluephase C8 HIGH	65,00	0,96	<0,001
	Bluephase C8 LOW	62,63	1,43	
	Bluephase C8 SOFT	65,51	1,75	
	Bluephase 16i HIGH	67,90	0,69	
	Bluephase 16i LOW	67,87	0,77	
	Bluephase 16i SOFT	68,59	0,42	
	Bluephase C5	58,87	2,86	
	Bluephase SOFT	63,06	3,48	
TEC	Bluephase C8 HIGH	61,53	2,74	<0,001
	Bluephase C8 LOW	60,43	0,70	
	Bluephase C8 SOFT	60,24	1,42	
	Bluephase 16i HIGH	61,44	0,45	
	Bluephase 16i LOW	62,32	1,25	
	Bluephase 16i SOFT	64,55	0,46	
	Bluephase C5	57,31	3,19	
	Bluephase SOFT	60,31	0,62	

*ANOVA; TC - Tetric Ceram; TEC - Tetric EvoCeram

Tablica 3. Rezultati Tukeyeva post-hoc testa za razlike u stupnjevima konverzije kod osvjetljavanja raznim izvorima svjetla za materijale TC i TEC

Table 3 Results of Tukey post-hoc test for differences in degree of conversion for TC and TEC composite materials polymerized with curing units of different light intensity

	Bluephase C8 HIGH	Bluephase C8 LOW	Bluephase C8 SOFT	Bluephase 16i HIGH	Bluephase 16i LOW	Bluephase 16i SOFT	Bluephase C5	Bluephase SOFT
Bluephase C8 HIGH						TC#, TEC#	TC*, TEC#	
Bluephase C8 LOW				TC*	TC*	TC*, TEC#	TC#	
Bluephase C8 SOFT						TEC#	TC*	
Bluephase 16i HIGH		TC*				TEC#	TC*, TEC#	TC*
Bluephase 16i LOW		TC*					TC*, TEC*	TC*
Bluephase 16i SOFT	TC#, TEC#	TC*, TEC#	TEC#	TEC#			TC*, TEC*	TC*, TEC*
Bluephase C5	TC*, TEC#	TC#	TC*	TC*, TEC#	TC*, TEC*	TC*, TEC*		TC#
Bluephase SOFT				TC*	TC*	TC*, TEC*	TC*	

Tukey post-hoc test *p≤0,001, #p<0,05; TC - Tetric Ceram; TEC - Tetric EvoCeram

Rezultati mjerjenja temperaturnog porasta

Rezultati mjerjenja temperature prikazani su u Tablicama 4. i 5.

Najviši temperaturni porast zabilježen je tijekom polimerizacije uzoraka uređajem Bluephase 16i, i to

er polymerization modes there are statistically significant differences in the degree of conversion between tested composite materials.

Results of temperature measurements

Results of temperature measurements are shown in Table 4 and 5.

The highest temperature rise was recorded in the case of polymerization of composite resin sam-

Tablica 4. Rezultati mjerjenja temperature

Table 4 Results of temperature measurements

Kompozitni materijal • Composite material	LED polimerizator • LED curing unit	Temperatura (sr.vrijednost) • Temperature (mean value) (°C)	Std. Dev.	p*
TC	Bluephase C8 HIGH	7,25	,33	<0,001
	Bluephase C8 LOW	6,15	,28	
	Bluephase C8 SOFT	7,53	,49	
	Bluephase 16i HIGH	8,11	1,26	
	Bluephase 16i LOW	6,71	,68	
	Bluephase 16i SOFT	8,35	,48	
	Bluephase SOFT	7,85	,47	
	Bluephase C5	7,05	1,08	
TEC	Bluephase C8 HIGH	6,80	,62	<0,001
	Bluephase C8 LOW	4,98	,63	
	Bluephase C8 SOFT	6,58	,11	
	Bluephase 16i HIGH	8,10	1,24	
	Bluephase 16i LOW	6,88	,43	
	Bluephase 16i SOFT	6,33	,70	
	Bluephase SOFT	6,38	,68	
	Bluephase C5	5,08	,62	

*ANOVA; TC - Tetric Ceram; TEC - Tetric EvoCeram

Tablica 5. Rezultati Tukeyeva post-hoc testa za razlike u temperaturi kod osvjetljavanja raznim izvorima svjetla za materijale TC i TEC

Table 5 Results of Tukey post-hoc test for differences in degree of conversion for TC and TEC composite materials polymerized with curing units of different light intensity

	Bluephse C8 HIGH	Bluephase C8 LOW	Bluephase C8 SOFT	Bluephase 16i HIGH	Bluephase 16i LOW	Bluephase 16i SOFT	Bluephase SOFT	Bluephase C5
Bluephase C8 HIGH		TEC#						TEC#
Bluephase C8 LOW	TEC#		TEC#	TC#, TEC*	TEC#	TC*	TC#	
Bluephase C8 SOFT		TEC#		TEC#				TEC#
Bluephase 16i HIGH		TC#, TEC*	TEC#			TEC#	TEC#	TEC#
Bluephase 16i LOW		TEC#				TC#		TEC#
Bluephase 16i SOFT		TC*		TEC#	TC#			
Bluephase SOFT		TC#		TEC#				
Bluephase C5	TEC#		TEC#	TEC#	TEC#			

Tukey post-hoc test *p≤0,001, #p<0,05; TC - Tetric Ceram; TEC - Tetric EvoCeram

za TC s programom SOFT (8,35 °C), a za TEC s programom HIGH (8,10 °C). Najniži porast temperature postignut je kod polimerizacije programom LOW Bluephase C8 polimerizatora za oba kompozitna materijala (TC 4,98 °C, TEC 6,15 °C).

ANOVA je istaknula statistički velike razlike u porastu temperature kod materijala TC, ovisno o vrsti svjetla (p<0,001). U Tablici 5. navedene su stati-

ples with Bluephase 16i curing unit, especially for TC using SOFT mode (8,35 °C), and for TEC using HIGH mode (8,10 °C). The lowest temperature rise was achieved using LOW polymerization mode of Bluephase C8 curing unit for both composite materials (TC 4,98 °C, TEC 6,15 °C).

ANOVA showed statistically significant differences in temperature rise for TC composite material

stički znatne razlike između pojedinih izvora svjetla.

Kompozitni materijal TEC pokazuje također statistički velike razlike u utjecajima polimerizacijskih programa na temperaturni porast ($p<0,001$), a razlike su češće i veće nego kod TC-a (Tablica 5.).

Rasprava

Polimerizacijski programi visokog intenziteta danas su zanimljivi zbog uštede vremena. No, pokazalo se da oni - ako je vrijeme osvjetljavanja kratko - dovode do dostačnog ili čak nižeg stupnja konverzije i lošijih fizičko-mehaničkih svojstava u odnosu prema kompozitnom materijalu polimeriziranom srednjim intenzitetom i dužim osvjetljavanjem (14).

Rezultati ispitivanja pokazali su najveći stupanj konverzije kod uzoraka kompozitnog materijala polimeriziranog SOFT programom Bluephase 16i uređaja (68,59% za TC), što se i moglo očekivati, jer je najveći intenzitet dulje primijenjen. Najniži stupanj konverzije dobiven je tijekom polimerizacije uređajem najnižeg intenziteta i primijenjene energije - Bluephase C5 (57,31% za TEC). Ne uzimajući u obzir vrstu kompozitnog materijala, nego samo polimerizacijski program i intenzitet, iste rezultate stupnja konverzije nalazimo i kod drugih autora (4,5).

Osim svojstava izvora svjetla, na kvalitetu polimerizacije znatno utječu sastav kompozitnog materijala te vrsta fotoinicijatora. Kamforkinon (468 nm) je najčešći fotoinicijator u sastavu kompozitnih materijala. Osim njega, fenil propan dion i lucirin (aktiviraju se na nižim valnim duljinama od 410 do 430 nm) također mogu biti u sastavu kompozitnih materijala, posebice tzv. "bleaching" boja i transparentne nijanse (15). Kod takvih materijala upitna je polimerizacija nekim starijim LED-uređajima s užim spektrom emitiranih valnih duljina, čime je bila onemogućena aktivacija spomenutih fotoinicijatora. To bi moglo biti objašnjenje za niži stupanj konverzije kod polimerizacije TEC kompozitnog materijala, bez obzira na to o kojem se izvoru svjetla i polimerizacijskog programa radi. Naime, TEC prema uputama proizvođača, u svojem sastavu, osim kamforkinona, ima i lucirin kao koinicijator.

Energija emitirana iz polimerizatora ima isti utjecaj na temperaturni porast kao što ga ima i na stupanj konverzije. Prema stajalištu Loneya i Pricea (10), razlika u energiji znatno utječe na tempera-

depending on the curing unit used ($p<0,001$). In Table 5 statistical significant differences between curing units, i.e. polymerization programs, are given.

Composite material TEC also shows statistically significant differences in temperature rise influenced by polymerization modes ($p<0,001$), and the differences are more common and greater than in TC (Table 5).

Discussion

The desire to cure composite materials faster has lead to a growing interest in polymerization modes of high intensity. However, it has been shown that these modes with short curing time lead to inadequate or even lower degrees of conversion and poorer physical-mechanical characteristics compared to composite materials polymerized with medium intensity and longer curing times (14).

The results of this study have confirmed that the highest degree of conversion was in composite resin samples polymerized with SOFT mode of Bluephase 16i curing unit (68,59% for TC), because the highest intensity was applied in a longer time. The lowest degree of conversion was achieved in the case of composite resin samples polymerized with a curing unit of low intensity, or low applied energy - Bluephase C5 (57,31% for TEC). When only considering the polymerization mode and light intensity, not the type of composite material, the same results of degree of conversion are found in studies by other authors (4,5).

Besides the light source characteristics, the composition of composite material and the type of photo initiator, also significantly influence the polymerization quality. Camforquinone (468 nm) is the most common photo initiator in composite materials. However, phenyl propane dion and lucirin, which are activated at lower wavelengths (410-430 nm), can also be found in composite materials, especially in bleaching and transparent shades (15). For these materials, polymerization with LED curing units of earlier generation is questionable, due to narrower spectra of the emitted wavelengths, which enables activation of the mentioned photo initiators. This could be the explanation for lower degree of conversion of TEC composite material polymerization, regardless of the light source or polymerization mode. Besides camforquinone, TEC has lucirin as a co-initiator, as instructed by the manufacturer.

Energy emitted from the curing unit has the same influence on temperature rise as it has on the degree of conversion. According to Loney and Price (10)

turni porast, ovisno o primijenjenom polimerizacijskom programu.

Rezultati ovog ispitivanja pokazali su povezanost stupnja konverzije i temperaturnog porasta - najveći temperaturni porast zabilježen je kod polimerizacije SOFT i HIGH polimerizacijskim programom Bluephase 16i, uređaja kod kojeg je postignut i najveći stupanj konverzije. Isto tako, najniži stupanj konverzije i najniža temperatura postignuti su tijekom polimerizacije uzoraka kompozitnog materijala uređajima najnižeg intenziteta - Bluephase C5 te LOW programom Bluephase C8 uređaja. Zanimljivo je istaknuti isti način ponašanja SOFT i HIGH polimerizacijskog programa, bez obzira na to o kojoj se vrsti svjetla ili točnije, intenziteta radi. Tako se, na primjer, analizirajući rezultate postignute polimerizatorom Bluephase C8 za SOFT i HIGH program, može izvesti isti zaključak kao i za SOFT i HIGH program Bluephase 16i uređaja - kako za temperaturu tako i za stupanj konverzije. Rezultati ovog ispitivanja slažu se s rezultatima mjerjenja temperature Aguiara i njegovih suradnika (16) - oni su također dobili najveći porast temperature tijekom polimerizacije uzoraka SOFT programom. Razlog za to su, osim jakosti polimerizatora, i produljeno vrijeme osvjetljavanja iako je taj polimerizacijski program prema istraživanjima nekih autora, pokazao najbolje rezultate za ublažavanje polimerizacijskog skupljanja i stresa nastalog zbog skupljanja (7,16-20).

Ranije generacije LED-uređaja imale su manji porast temperature u odnosu prema rezultatima novijih LED polimerizatora dobivenih u ovoj studiji (4,98 do 8,35 °C) (1,21). Prema istraživanjima Zacha i Cohen (22), već porast temperature od 5,5 °C može rezultirati ireverzibilnim promjenama u pulpnom tkivu.

Velik utjecaj na vitalnost pulpe, osim karakteristika polimerizatora i sastava kompozitnog materijala, ima dubina kaviteta te debljina preostalog dentina. Što je veća debljina zaostalog dentina, to je manja mogućnost termičkog oštećenja pulpnog tkiva (15,22-24).

Samo na temelju rezultata mjerjenja temperature i stupnja konverzije, teško se može zaključiti koji je od polimerizacijskih programa bolji. Za konačan zaključak potrebno je obaviti dodatna mjerjenja polimerizacijskog skupljanja i stresa koji pritom nastaju. Iz svega spomenutoga jasno je da niz parametara na koje je moguće donekle utjecati, pridonose kvaliteti i dugotrajnosti kompozitne restauracije. Na kliničaru je da - ovisno o situaciji - prilagodi vrijeme i jakost polimerizacije kako bi se uz maksima-

the energy difference has a high impact on temperature rise depending on the polymerization mode used. The results of this study have shown the connection between degree of conversion and temperature rise; the highest temperature increase and highest degree of conversion were recorded in the polymerization with SOFT and HIGH polymerization modes of the Bluephase 16i curing unit. At the same time, the lowest degree of conversion and the lowest temperature rise were achieved in polymerization of composite resin samples with curing units of lowest light intensity - Bluephase C5 and LOW mode of Bluephase C8 curing unit. It is interesting to note almost equal behavior of SOFT and HIGH polymerization programs despite the curing unit or light intensity used. For instance, observing the Bluephase C8 curing unit, the same conclusion for SOFT and HIGH program as for SOFT and HIGH programs of the Bluephase 16i unit can be made, for both temperature rise and degree of conversion. The results of this study match the results of temperature measuring reported by Aguiara et al. (16), who also recorded the highest temperature rise when SOFT polymerization mode was used for photo polymerization of composite resin. Reasons for this are: a long curing light intensity and the extended curing time. However, this polymerization mode showed the best results in decreasing polymerization shrinkage and stress produced during shrinkage (7,16-20).

Earlier generations of LED curing units recorded lower temperature rise compared to the results of recent LED curing units gain in this study (4.98 to 8.35 °C) (1,21). According to research by Zach and Cohen (22), temperature rise of 5.5 °C could already result in irreversible changes in pulp tissue.

Aside from the curing unit characteristics and the composition of the composite material, other important influences on pulp vitality are: the cavity depth and the thickness of dentine towards pulp. The thicker the dentine layer is, the lesser the chance for thermic damage of the pulp tissue (15,22-24).

Observing only the results of temperature and the degree of conversion measurement, it is difficult to conclude which polymerization program is the one with the best performance. For the final and complete conclusion, polymerization shrinkage and polymerization shrinkage stress should be performed. When all this is considered, it is clear that many relatively easy to influence parameters contribute to quality and longevity of composite resin restoration. Depending on the clinical situation,

Ian učinak konverzije postigle najmanje moguće negativne posljedice fotopolimerizacije kompozitnog materijala.

Zaključak

Rezultati ovog ispitivanja pokazali su jednako ponašanje rezultata stupnja konverzije i temperaturnog porasta kod pojedinih polimerizacijskih programa. Najveći stupanj konverzije i temperature zabilježen je kod uzoraka kompozitnih materijala polimeriziranih polimerizacijskim programom najvišeg intenziteta, a najniži kod programa najslabijeg intenziteta.

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Abstract

The appearance of LED curing units for composite materials polymerization has almost completely replaced halogen curing units. The main disadvantage of halogen curing units was a temperature rise during the polymerization. Earlier generations of LED devices made the temperature rise negligible. The newest generation of LED curing units makes a higher degree of conversion due to higher light intensity, but also brings to question the temperature rise during the composite materials polymerization. **Aim:** The purpose of this study was to measure the degree of conversion and temperature rise in composite resin samples. **Material and Methods:** The samples were polymerized by curing modes of different light intensity of four LED units: Bluephase, Bluephase C5, Bluephase C8 and Bluephase 16i (all units Vivadent, Schaan, Liechtenstein). The degree of conversion was measured by FTIR spectrophotometer, while for the temperature it was measured using the thermometer ELV Pyroscan (Leer, Germany). **Results:** The highest degree of conversion was achieved in the case of composite resin samples polymerized with soft-start mode (SOFT) of Bluephase 16i unit, while the lowest degree of conversion was achieved in the case of polymerization with Bluephase C5 curing unit. The highest temperature rise was recorded in the case of composite samples polymerization with high intensity mode (HIGH) and SOFT mode of Bluephase 16i, while the lowest in the case of polymerization with low intensity mode (LOW) of Bluephase C8 unit. **Conclusion:** According to the results of this study, it can be concluded that higher degree of conversion brings higher temperature rise, regardless of composite material used.

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Key words

Composite Resin; Polymers

it is up to the practitioner to adapt polymerization time and intensity, which ensures maximal degree of conversion and minimal negative effects of composite material photopolymerization.

Conclusion

Results of this study show the same behavior for the degree of conversion and temperature rise with individual polymerization modes. The highest degree of conversion and the highest temperature rise was recorded when composite resin samples were polymerized with curing modes of the highest intensity, while the lowest results were recorded when the lowest light intensity mode was used.

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References

1. Knezevic A, Tarle Z, Meniga A, Sutalo J, Pichler G, Ristic M. Degree of conversion and temperature rise during polymerization of composite resin samples with blue diodes. *J Oral Rehabil.* 2001;28(6):586-91.
2. Knežević A, Tarle Z, Meniga A, Šutalo J, Pichler G, Ristić M. Polymerization of composite materials with blue super bright light emitting diodes. *Acta Stomatol Croat.* 1999;33(3):337-48.
3. Tarle Z, Meniga A, Knezevic A, Sutalo J, Ristic M, Pichler G. Composite conversion and temperature rise using a conventional, plasma arc and an experimental blue LED curing unit. *J Oral Rehabil.* 2002;29(7):662-7.
4. Uhl A, Sigusch BW, Jandt KD. Second generation LEDs for the polymerization of oral biomaterials. *Dent Mater.* 2004;20(1):80-7.
5. Price RB, Felix CA, Andreou P. Evaluation of the second generation of LED curing light. *J Canad Dent Assoc.* 2003;69(10):666-71.
6. Obici AC, Sinhoreti MA, Correr Sobrinho L, Goes MF, Consani S. Evaluation of depth of cure and knoop hardness in a dental composite photo-activated using different methods. *Braz Dent J.* 2004;15(3):199-203.
7. Ilie N, Felten K, Trixner K, Hickel R, Kunzelmann KH. Shrinkage behavior of a resin-based composite irradiated with modern curing units. *Dent Mater.* 2005;21(5):483-9.
8. Knezevic A, Demoli N, Tarle Z, Meniga A, Sutalo J, Pichler G. Measurement of linear polymerization contraction using digital laser interferometry. *Oper Dent.* 2005;30(3):346-52.
9. Tarle Z, Knezevic A, Demoli N, Meniga A, Sutaloa J, Unterbrink G et al. Comparison of composite curing parameters: effects of light source and curing mode on conversion, temperature rise and polymerization shrinkage. *Oper Dent.* 2006;31(2):219-26.
10. Loney RW, Price RB. Temperature transmission of high-output light-curing units through dentin. *Oper Dent.* 2001;26(5):516-20.
11. Hume WR, Gerzina TM. Bioavailability of components of resin-based materials which are applied to teeth. *Crit Rev Oral Biol Med.* 1996;7(2):172-9.
12. Gerzina TM, Hume WR. Diffusion of monomers from bonding resin composite combinations through dentine in vitro. *J Dent.* 1996;24(1-2):125-8.
13. Rueggeberg FA, Caughman WF, Curtis JW Jr, Davis HC. Factors affecting cure at depths within light-activated resin composites. *Am J Dent.* 1993;6(2):91-5.
14. Peutzfeldt A, Asmussen E. Resin composite properties and energy density of light cure. *J Dent Res.* 2005;84(7):659-62.
15. Park YJ, Chae KH, Rawls HR. Development of a new photoinitiation system for dental light-cure composite resins. *Dent Mater.* 1999;15(2):120-7.
16. Aguiar FH, Barros GK, dos Santos AJ, Ambrosano GM, Lovadino JR. Effect of polymerization modes and resin composite on the temperature rise of human dentin of different thicknesses: an in vitro study. *Oper Dent.* 2005;30(5):602-7.
17. Friedl KH, Schmalz G, Hiller KA, Märkl A. Marginal adaptation of Class V restorations with and without "softstart-polymerization". *Oper Dent.* 2000;25(1):26-32.
18. Dennison JB, Yaman P, Seir R, Hamilton JC. Effect of variable light intensity on composite shrinkage. *J Prosthet Dent.* 2000;84(5):499-505.
19. Aguiar FH, Ajudarte KF, Lovadino JR. Effect of light curing modes and filling techniques on microleakage of posterior resin composite restorations. *Oper Dent.* 2002;27(6):557-62.
20. Demoli N, Knezevic A, Tarle Z, Meniga A, Sutalo J, Pichler G. Digital interferometry for measuring of the resin composite thickness variation during blue light polymerization. *Optics Commun.* 2004;231(1-6):45-51.
21. Knežević A, Tarle Z, Meniga A, Sutalo J, Pichler G. Influence of light intensity from different curing units upon composite temperature rise. *J Oral Rehabil.* 2005;32(5):362-7.
22. Zach L, Cohen G. Pulp response to externally applied heat. *Oral Surg Oral Med Oral Pathol.* 1965;19:515-30.
23. Tjan AH, Dunn JR. Temperature rise produced by various visible light generators through dentinal barriers. *J Prosthet Dent.* 1988;59(4):433-8.
24. Camps J, Dejou J, Remusat M, About I. Factors influencing pulpal response to cavity restorations. *Dent Mater.* 2000;16(6):432-40.