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## Dimenzijske promjene staklenoionomernih cementa i giomera tijekom stvrdnjivanja materijala

### *Dimensional Changes of Glass Ionomers and a Giomer during the Setting Time*

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#### Sažetak

**Svrha:** Cilj ovoga rada bio je postupkom digitalne laserske interferometrije odrediti dimenzijske promjene konvencionalnih staklenoionomernih cementa, smolom modificiranog staklenoionomernog cementa i giomera tijekom stvrdnjivanja materijala. Također se želio odrediti utjecaj različitih programa (*visoki, postupni, niski*) polimerizacijskog uređaja proizvedenog na temelju svjetlećih dioda (LED) na dimenzijske promjene svjetlom polimerizirajućih materijala. **Materijali i postupci:** Određeno je linearno skupljanje konvencionalnih staklenoionomernih cementa (K SIC) Fuji IX Extra (F9E), Fuji IX Fast (F9F), Ketac Molar Aplicap (KM) i Ketac Molar Quick Aplicap (KMQ), zatim smolom modificiranog staklenoionomernog cementa (SM SIC) Fuji II LC (F2LC) te giomera Beautifil II (B2). Svi ispitani materijali bili su nijanse A3, a svi staklenoionomerni cementi kapsulirani. Uzorci diskoidnog oblika ( $n = 10$ ,  $d = 10$  mm,  $h = 0,85$  mm) pripremljeni su prema uputama proizvođača za svaki ispitani materijal i svaki polimerizacijski program (za svjetlom polimerizirajuće materijale). Svjetlosno polimerizirajući uzorci polimerizirani su LED uređajem za polimerizaciju (Bluephase G2, Ivoclar-Vivadent, Schaan, Lihtenštajn). Dimenzijske promjene tijekom stvrdnjivanja zabilježene su u stvarnom vremenu. Rezultati su analizirani ANOVA-om, a Tukeyjev post hoc test korišten je za višestruke usporedbe ( $\alpha < 1\%$ ). **Rezultati:** Svi ispitani materijali imali su početnu ekspanziju i naknadno skupljanje. KM i KMQ značajno su se manje skupljali od SM SIC-a polimeriziranog bilo kojim od triju polimerizacijskih programa. B2 se manje skupljao od F2LC-a. **Zaključak:** Stupanj polimerizacijskog skupljanja SM SIC-a može utjecati na trajnost restauracija.

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#### Ključne riječi

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#### Uvod

Suvremena restaurativna dentalna medicina još je suočena s problemom rubne pukotine. Adhezivna tehnologija, tj. izravno kemijsko vezanje i/ili mikromehanička adhezija na zubnu strukturu, nije riješila problem rubne pukotine i posljedičnog mikropropuštanja (1, 2). Fenomen mikropropuštanja može uzrokovati ozbiljne probleme kao što su poslijeoperacijska osjetljivost, prodiranje bakterija, što uzrokuje sekundarni karijes i upalnu reakciju pulpe, te promjenu boje zuba i mogući gubitak restauracije (3).

Dimenzijske promjene materijala tijekom stvrdnjivanja mijenjaju njegovu strukturu i mogu utjecati na adheziju, što uvelike pridonosi stvaranju rubne pukotine i mikropropuštanja (3 – 5). Sličnost između dimenzijskih promjena koje se pojavljuju pri stvrdnjivanju restaurativnih materijala i toplinske ekspanzije zuba može biti važna u smanjenju rubne pukotine (6). Prema podacima iz literature, dimenzijske promje-

#### Introduction

Modern restorative dentistry is still faced with the problem of marginal gap formation. Adhesive technology, *i.e.* direct chemical bonding and/or micromechanical adhesion to tooth structure, did not solve the issue of the marginal gap and consequent microleakage (1, 2). The phenomenon of microleakage may lead to serious problems such as postoperative sensitivity, penetration of bacteria, which causes secondary caries and inflammatory reaction of the pulp, tooth discoloration, and eventually loss of the restoration (3).

Dimensional changes of materials during setting change the structure of the material and may influence the adhesion thereby greatly contributing to creating the marginal gap followed by microleakage (3-5). Similarity in dimensional changes occurring during the setting of restorative materials and thermal expansion of the tooth may play an important role in marginal gap minimization (6). According to the liter-

ne važan su čimbenik za dugotrajnost restauracija (7 – 10). Stres koji nastaje zbog dimenzijskih promjena ovisi o stupnju skupljanja i modulu elastičnosti materijala (11, 12).

Polimerizacija kompozitnih smola može se podijeliti na pred-gel i poslije-gel fazu. Tijekom pred-gel faze molekule monomera mogu se kretati unutar polimerne mreže te se tako smanjuje polimerizacijski stres. Međutim, u poslije-gel fazi monomeri su imobilizirani porastom viskoznosti polimerne matrice, što rezultira velikim stresom u samom zubu i na adhezivnoj površini (12, 13). Postupna polimerizacija materijala preporuka je za smanjenje polimerizacijskog stresa i posljedničkog mikropropuštanja (14).

Staklenoionomerni cementi (SIC) hidrofilni su materijali koji imaju široku kliničku primjenu zahvaljujući kemijskoj adheziji na tvrda zubna tkiva, otpuštanju fluorida i biokompatibilnosti (1). Danas je komercijalno dostupno mnogo SIC-ova za kliničku upotrebu, a uzajamno se razlikuju prema sastavu praha, tekućine ili obiju komponenata. Sastav SIC-ova optimiziran je za specifične kliničke indikacije. Prema mehanizmu stvrdnjavanja mogu se razlikovati dvije glavne skupine – konvencionalni SIC (K SIC) (stvrdnjava se kiselolužnatom reakcijom) i smolom modificirani SIC (SM SIC) (stvrdnjava se kiselolužnatom reakcijom i polimerizacijom smole). Reakcija stvrdnjavanja SM SIC-a složenija je jer se obje reakcije događaju istodobno te tako utječu jedna na drugu (15).

Giomeri su materijali proizvedeni na temelju smole te su prema osnovnom sastavu slični kompozitnim materijalima, ali za razliku od kompozita kao punilo sadržavaju staklenoionomernu komponentu koja je već reagirala, nazvanu prethodno reagirano staklo (PRG). PRG punila dobivaju se potpunom ili djelomičnom reakcijom staklenoionomernog praška s polikarboksilnim kiselinama (16, 17).

Dimenzije promjena koje nastaju zbog kontrakcije materijala mogu se opisati kao linearna i volumna kontrakcija (18). Za određivanje volumne kontrakcije najčešće se koriste metode vodenog ili živina dilatometra (3, 4, 18 – 23).

Watts i Cash koristili su se indirektnom metodom, metodom *odstupajućeg diska* koja osigurava ponovljivost mjerenja polimerizacijskog skupljanja. Izračunali su volumnu kontrakciju u post-gel fazi iz linearnog pomaka diska koji je smješten na mjedenom prstenu (24). Grajower i Guelmann odredili su dimenzijsku promjenu SIC-a s pomoću linearnog pretvornika (25). U većini studija izmjerene su ukupne dimenzijske promjene koje se događaju tijekom stvrdnjavanja materijala (3, 22, 25). U ovom istraživanju prvi je put korištena metoda digitalne laserske interferometrije kako bi se odredila dimenzijska promjena staklenoionomernih cementa i giomera u realnom vremenu tijekom stvrdnjavanja materijala koje preporučuje proizvođač. Ta je metoda pouzdana i već dokazana u praćenju dinamičkih promjena pri mjerenju skupljanja kompozitnih materijala (26 – 28).

Cilj ovoga rada bio je odrediti linearne dimenzijske promjene K SIC-ova, SM SIC-a i giomera u stvarnom vremenu stvrdnjavanja materijala te procijeniti utjecaj triju različitih polimerizacijskih programa polimerizacijskog uređaja temeljenog na svjetlećim diodama (LED) na dimenzijske promjene SM SIC-ova i giomera.

ature, dimensional changes are an important factor for restoration longevity (7-10). The stress resulting from dimensional changes is dependent on the extent of curing shrinkage and elastic modulus (11, 12).

A total resin composite polymerization can be divided into pre- and post-gel phases. During the pre-gel phase, monomer molecules have the ability to move within the polymer network, thus compensating polymerization stress. However, in the post-gel phase, monomers become immobilized in the polymeric matrix of rising viscosity, generating a large amount of stress in the tooth structure and at the tooth-material interface (12, 13). Gradual polymerization of the material is recommended to reduce the shrinkage stress and consequent microleakage (14).

Glass ionomer cements (GICs) are hydrophilic materials with widespread clinical use due to the chemical adhesion to tooth structure, fluoride release and biocompatibility (1). Today, there are a large numbers of commercially available GICs for clinical use with compositional variations regarding powder, liquid or both components. Different compositions of GICs are optimized for specific clinical purposes. Two main groups of GICs can be distinguished, considering the setting mechanism: conventional GIC (CGICs) (setting through an acid-base reaction) and resin-modified GIC (RM GIC) (setting through an acid-base reaction and the simultaneous polymerization of the resinous part). The setting reaction of RM GIC is more complex because both reactions occur concurrently thus influencing each other (15).

Giomers are resin-based materials with the basic composition similar to that of resin composites, which contain already reacted glass ionomer component named pre-reacted glass (PRG) particles. The PRG fillers are produced from the complete or partial reaction of ion-leachable glasses and polyalkenoic acids (16, 17).

Dimensional changes due to curing contraction can be described as a linear and volumetric contraction (18). Most common methods for determining the volumetric curing contraction are based on measurements using water or mercury dilatometers (3, 4, 18-23). Watts and Cash used indirect method, the “deflecting disc” method, which ensures reproducible measurements of polymerization shrinkage. They calculated the volumetric contraction from the post-gel linear displacement of a deflecting disc resting on a brass ring (24). Grajower and Guelmann determined the dimensional change of GIC by means of linear displacement transducer (25). In a large number of previous studies, the total dimensional changes that occur during the setting were measured (3,22,25). In this study, for the first time, the digital laser interferometry method was used to determine dimensional changes of glass ionomer cements and giomer in real time during the setting period, as recommended by respective manufacturers. This method has previously been shown to be reliable for monitoring dynamic processes such as shrinkage of resin composites (26-28).

The aim of this study was to evaluate linear dimensional changes of CGICs a RM GIC, and a giomer during the setting time and to assess the influence of three different curing modes of a LED curing unit on dimensional changes of RM

Testirane su sljedeće istraživačke hipoteze:

1. na dimenzijske promjene utjecat će vrsta materijala (giomer i SM SIC imat će sličan stupanj skupljanja, a K SIC će imati niži stupanj skupljanja od SM SIC-a i giomera)
2. polimerizacijski programi utjecat će na dimenzije promjene svjetlom polimerizirajućih materijala.

## Materijali i metode

### Odabir materijala

U istraživanje su uključena četiri konvencionalna SIC-a (K SIC) – Fuji IX Extra (F9E), Fuji IX Fast (F9F), Ketac Molar Aplicap (KM), Ketac Molar Quick (KMQ), zatim svjetlom polimerizirajući smolom modificirani SIC (SM SIC) Fuji II LC (F2LC) i giomer Beautifil II (B2). Svi ispitivani materijali navedeni u tablici 1. bili su nijanse A3, a svi SIC-ovi bili su pakirani u kapsulama.

GIC and giomer.

Research hypotheses tested were:

1. Dimensional changes will be affected by the material type (giomer and RM GIC are expected to show a similar degree of shrinkage, while CGICs are expected to show a lower degree of shrinkage than RM GICs and giomer).
2. Dimensional changes of light-curable materials will be influenced by curing modes.

## Materials and methods

### Materials selection

This study evaluated four conventional GIC (CGICs): Fuji IX Extra (F9E), Fuji IX Fast (F9F), Ketac Molar Aplicap (KM), and Ketac Molar Quick (KMQ), a light-curable resin-modified GIC (RM GIC): Fuji II LC (F2LC) and a giomer: Beautifil II (B2). All tested materials, listed in Table 1, were of shade A3, while all of the GICs were encapsulated.

**Tablica 1.** Materijali korišteni u istraživanju  
**Table 1** Materials investigated in the study

Materijal • Material	Kod • Code	Vrsta materijala • Type of material	Proizvođač • Manufacturer	LOT • LOT	Primjena • Application
Fuji IX EXTRA	F9E	K SIC • CGIC	GC Corporation, Tokio, Japan	1009205	Restaurativni • Restorative
Fuji IX FAST	F9F	K SIC • CGIC	GC Corporation, Tokio, Japan	1005251	Restaurativni • Restorative
Ketac Molar Aplicap	KM	K SIC • CGIC	3M ESPE, Seefeld, Njemačka • Germany	425688	Restaurativni • Restorative
Ketac Molar Quick Aplicap	KMQ	K SIC • CGIC	3M ESPE, Seefeld, Njemačka • Germany	416807	Restaurativni • Restorative
Fuji II LC <sup>a</sup>	F2LC	SM SIC • RM GIC	GC Corporation, Tokio, Japan	1009277	Restaurativni • Restorative
Beautifil II <sup>a</sup>	B2	Giomer • Giomer	Shofu Inc., Kjoto, Japan	041008	Restaurativni • Restorative

<sup>a</sup>svjetlom polimerizirajući materijali • Light-curable materials

### Priprema uzoraka

Pripremljeno je 10 diskoidnih uzoraka ( $d = 10$  mm,  $h = 0,85$  mm) za svaki testirani materijal (i polimerizacijski program za svjetlom polimerizirajuće materijale). Kapsulirani materijali (F9E, F9F, KM, KMQ i F2LC) pripremljeni su u skladu s uputama proizvođača. Odgovarajuća količina materijala istisnuta je iz kapsule na film od polietilen-tereftalata (PET) u kalup od nehrđajućeg čelika visine 1 mm, prekrivena je drugim PET filmom i pritisnuta ravnom pločicom od nehrđajućeg čelika. Ukupna debljina uzorka (0,85 mm) rezultat je oduzimanja debljine dvaju PET filmova od debljine prstenastog kalupa (1 mm). Nekapsulirani materijal (B2) nanesen je na PET film instrumentom 5/6 prema Heidemannu. Priprema uzoraka obavljena je u tamnoj sobi s upaljenim crvenim svjetlom kako bi se izbjegao polimerizacijski utjecaj ambijentalnog svjetla na svjetlom polimerizirajuće materijale. Za svjetlom aktiviranu polimerizaciju korišten je LED uređaj (Bluephase G2, Ivoclar-Vivadent, Schaan, Lihtenštajn). Uzorci su osvijetljeni po 20 sekunda jednim od polimerizacijskih programa – *visokim* ( $1100$  mW / cm<sup>2</sup>), *postupnim* ( $650 - 1100$  mW / cm<sup>2</sup>) i *niskim* ( $650$  mW / cm<sup>2</sup>).

### Specimen preparation

10 discoid specimens ( $d=10$  mm,  $h=0.85$  mm) were prepared for each tested material (and curing mode, for light-curable materials), 10 discoid specimens ( $d=10$  mm,  $h=0.85$  mm) were prepared. The encapsulated materials (F9E, F9F, KM, KMQ and F2LC) were mixed according to the manufacturer's instructions. An adequate amount of material was extruded onto a polyethylene terephthalate (PET) film in a stainless steel ring mold of 1 mm height, covered with another sheet of PET film and compressed using a flat stainless steel plate. The total thickness of the specimen (0.85 mm) was the result of subtracting the thickness of two PET sheets from the thickness of the ring mold (1 mm). The non-encapsulated material (B2) was applied onto the PET film using a spatula. Specimen preparation was performed in the dark room with red light to avoid curing effect of ambient light on light-curable materials. Light-curable materials were cured using LED curing unit (Bluephase G2, Ivoclar-Vivadent, Schaan, Liechtenstein) for 20 s with one of the following light-curing modes: "high" ( $1100$  mW/cm<sup>2</sup>), "soft" ( $650 - 1100$  mW/cm<sup>2</sup>) and "low" ( $650$  mW/cm<sup>2</sup>).

## Digitalna laserska interferometrija

Promjene dimenzija izračunate su iz eksperimentalnih mjerenja dobivenih s pomoću digitalnog laserskog interferometrijskog uređaja shematski prikazanog na slici 1. Diskoidni uzorci bili su s obje strane pokriveni PET folijama i umetnuti između dviju staklenih ploča. Gornja staklena ploča imala je reflektirajuću površinu i djelovala je kao zrcalo. Nosač uzorka bio je oblikovan tako da omogući fotopolimerizaciju uzorka s jedne strane i istodobno mjerenje promjene debljine uzorka s druge strane. Mikrometar je postavljen kako bi se omogućila ručna kompenzacija pomicanja gornje površine uzorka koja je prekrivena zrcalom. Upotrijebljen je na kraju svakog mjerenja kako bi se utvrdila konačna vrijednost linearnog skupljanja uzorka.

Snop svjetla koji izlazi iz He-Ne lasera (Spectra Physics, snaga = 25 mW, valna duljina = 632,8 nm) prvo je proširen i kolimiran, a zatim usmjeren na ploču nosača uzorka. Ploča tu upadnu svjetlost reflektira s pomoću dvaju ravnih zrcala, jednim smještenim na gornju površinu uzorka i drugim izvan uzorka, čime se formiraju dvije zrake svjetlosti – objektivna i referentna. Obje zrake su zatim pod malim kutom usmjerene na CCD senzor od 752 × 582 piksela, omogućujući brzinu snimanja od 25 sličica u sekundi. Dobivene slike interferencijskih pruga nose faznu informaciju uzorka. Za vremenski ovisne dimenzijske promjene položaj pruga također se mijenja u vremenu, a tražene vrijednosti promjene dimenzije računaju se numerički. Za SIC-ove sva su mjerenja počela približno 45 sekunda nakon završetka miješanja kapsula, a trajala su do kraja vremena stvrdnjavanja koje su odredili odgovarajući proizvođači (za F9F, F9E, KM, KMQ do 6 min. i za F2LC do 5 min.). U slučaju giomera (B2), mjerenja su počela istodobno s početkom svjetlosne polimerizacije i trajala su pet minuta. Interferogrami snimljeni tijekom navedenog vremena analizirani su računalnim programom pripremljenim u LabVIEW-u 2011. (National Instruments, Austin, Texas, SAD) prema već opisanom postupku (26).

## Statistička analiza

Linearne dimenzijske promjene dobivene digitalnom laserskom interferometrijom raščlanjene su analizom varijance (ANOVA-om) i Tukeyjevim post hoc testom za višestruke usporedbe ( $\alpha = 0,01$ ).

## Rezultati

Svi testirani K SIC-ovi pokazali su početnu ekspanziju koju je slijedilo skupljanje materijala (slika 2.). KM se značajno manje skupljao od F9E-a i F9F-a ( $p < 0,01$ ), a KMQ se značajno manje skupljao od F9F-a ( $p < 0,01$ ).

Najviše se skupio F9F, a ostali materijali sljedećim redoslijedom – F9E, KMQ i KM.

SM SIC i giomer također su imali početnu ekspanziju i naknadno skupljanje u svim trima načinima polimerizacije (slika 2.). Razlike između polimerizacijskih programa bile su statistički značajne samo za B2 između *visokog* i *niskog* polimerizacijskog programa. U tim su slučajevima linearne di-

## Digital laser interferometry

Dimensional changes were calculated from experimental measurements obtained by using digital laser interferometry device schematically shown in Figure 1. The discoid specimens were covered on both sides with PET foils and sandwiched between two glass plates. The upper glass plate had a reflective surface and acted as a mirror. The specimen carrier was designed to allow photo polymerization of the specimen from one side and simultaneous measurements of thickness variation of the specimen from the other side. A micrometer was mounted to enable manual compensation of the displacement of the upper surface of the specimen, which was covered by a mirror. The micrometer was used at the end of each measurement to verify the final value for linear shrinkage of the specimen.

The beam emerging from the He-Ne laser (Spectra Physics, power = 25 mW, wavelength = 632.8 nm) was first expanded and collimated and then steered onto the specimen carrier plate. The plate reflected incident light by means of two flat mirrors, one placed on the upper surface of the specimen and the other outside of the specimen, thus forming two beams, the object beam and the reference beam. Subsequently, both beams were directed at a small angle to the CCD sensor with 752×582 pixels allowing the recording rate of 25 frames per second. The resulting interference fringe patterns carried the specimen phase information. For the time-dependent dimensional changes of the specimen, the location of fringes was also time-dependent and the required values were evaluated numerically. For GICs, all of the measurements started approximately 45 s after the end of mixing until the end of the setting time specified by respective manufacturers (for F9F, F9E, KM, KMQ up to 6 min and for F2LC up to 5 min). In the case of giomer (B2), measurements started concurrently with the initiation of light curing and lasted for 5 min. The interferograms recorded during the aforementioned time were analyzed using a custom-made computer program prepared in LabVIEW 2011 (National Instruments, Austin, Texas, USA) according to the previously described procedure (26).

## Statistical Analysis

The linear dimensional changes obtained using digital laser interferometry were analyzed by analysis of variance (ANOVA), and Tukey post hoc test for multiple comparisons ( $\alpha = 0.01$ ).

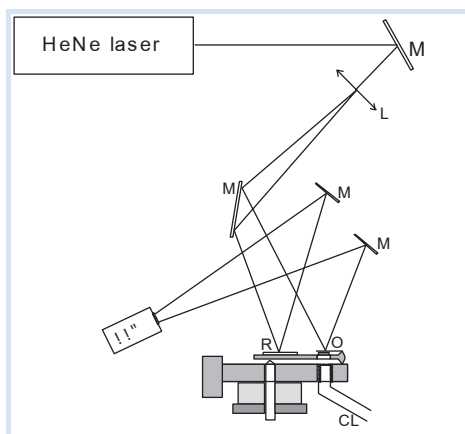
## Results

All of the tested CGICs showed an initial setting expansion, followed by shrinkage (Figure 2). KM showed significantly lower shrinkage than F9E and F9F ( $p < 0.01$ ), while KMQ showed significantly lower shrinkage than F9F ( $p < 0.01$ ).

The largest curing shrinkage had F9F followed by F9E, KMQ and KM.

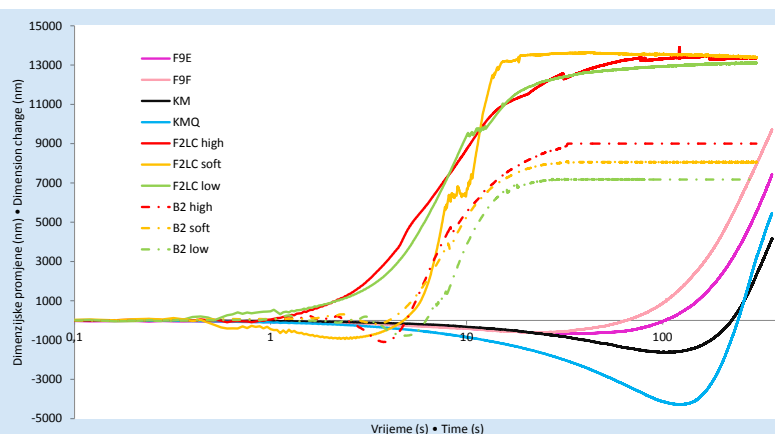
RM GIC and giomer showed an initial setting expansion and a subsequent setting shrinkage in all three curing modes (Figure 2). The differences among curing modes were statistically significant only in B2 between the “high”





**Slika 1.** Shematski prikaz interferometrijskog eksperimentalnog postava; M - zrcalo; L – leća; R – referentno zrcalo; O – objektno zrcalo; CL – polimerizacijsko svjetlo

**Figure 1** Schematic illustration of the interferometric experimental setup. M, mirror; L, lens; R, reference mirror; O, object mirror; CL, curing light



**Slika 2.** Dimenzijske promjene različitih K SIC-ova tijekom stvrdnjavanja i dimenzijske promjene SM SIC/giomera polimeriziranih različitim polimerizacijskim programima polimerizacijskog uređaja Bluephase G2 LED

**Figure 2** Dimensional changes of different CGICs during the setting and dimensional changes of RM GIC/giomer polymerized with different curing modes of Bluephase G2 LED curing unit

menzijske promjene bile značajno veće za *visoki* polimerizacijski program (B2  $1,15 \pm 0,23$  %) u usporedbi s *niskim* (B2  $0,80 \pm 0,17$  %) ( $p < 0,01$ ) (tablica 3.).

B2 je imao niže skupljanje od F2LC-a, ali je značajno niže skupljanje B2 u usporedbi s F2LC-om izmjereno za *niski* polimerizacijski program ( $p < 0,01$ ) (tablica 3.). Uspoređujući dimenzijske promjene K SIC-a i SM SIC-a, značajna razlika utvrđena je samo usporedbom KM-a i KMQ-a s F2LC-om – KM i KMQ imali su značajno niže skupljanje od F2LC-a polimeriziranog bilo kojim od triju polimerizacijskih programa (slika 2.).

Ekspanzija K SIC-a trajala je dulje od ekspanzije SM SIC-a. Vrijeme ekspanzije iznosilo je KM  $4,0 \pm 0,7$  min., KMQ  $3,4 \pm 0,8$  min., F9F  $2,0 \pm 0,4$  min. i F9E  $0,8 \pm 0,3$  min. F2LC imao je ekspanziju tijekom početnih  $0,1 \pm 0,06$  minuta, a B2 tijekom  $0,1 \pm 0,03$  minuta svjetlosne polimerizacije.

and “low” curing mode. In these cases, the linear dimensional changes were significantly higher for the “high” curing mode (B2  $1.15 \pm 0.23\%$ ) compared to the “low” mode (B2  $0.80 \pm 0.17\%$ ), ( $p < 0.01$ ), (Table 3).

B2 showed lower shrinkage than F2LC, but significantly lower shrinkage B2 compared to F2LC was measured for “low” curing mode ( $p < 0.01$ ) (Table 3). If we compare dimensional changes of CGICs and RM GIC, the significance was only demonstrated by comparing KM and KMQ with F2LC: KM and KMQ had a significantly lower setting shrinkage than F2LC polymerized with any of the three curing modes (Figure 2).

Expansion of CGICs lasted longer than the expansion of RM GIC. The KM expanded for  $4.0 \pm 0.7$  min, KMQ expanded  $3.4 \pm 0.8$  min, F9F expanded through  $2.0 \pm 0.4$  min and F9E  $0.8 \pm 0.3$  min. F2LC expanded through the initial  $0.1 \pm 0.06$  min and B2 expanded during  $0.1 \pm 0.03$  min of light curing.

**Tablica 2.** Srednje vrijednosti i standardna odstupanja linearnog skupljanja K SIC-ova; ista mala slova označuju statistički homogene skupine unutar stupca

**Table 2.** Mean values and standard deviations of linear setting shrinkage of CGICs. Same lowercase letters denote statistically homogeneous groups within a column

Materijal • Material	Skupljanje (%) • Shrinkage (%)	
	Sr. v. • Mean	St. dev. • St. dev.
F9E	1.22 <sup>a,c</sup>	0.38
F9F	1.32 <sup>a</sup>	0.57
KM	0.47 <sup>b</sup>	0.13
KMQ	0.62 <sup>b,c</sup>	0.33

**Tablica 3.** Srednje vrijednosti i standardna odstupanja linearnog skupljanja B i F2LC-a osvjetljenih različitim polimerizacijskim programima; ista mala slova u nizu označuju statistički homogene skupine za usporedbu između polimerizacijskih programa; ista velika slova označuju statistički homogene skupine unutar stupca

**Table 3.** Mean values and standard deviations of linear setting shrinkage of B2 and F2LC illuminated using different curing modes. Same lowercase letters within a row represent statistically homogeneous groups for the comparison among curing modes. Same uppercase letters denote statistically homogeneous groups within a column

Materijal • Material	Skupljanje (%) • Shrinkage (%)					
	Visoki • High		Postupni • Soft		Niski • Low	
	Sr. v. • Mean	St. dev.	Sr. v. • Mean	St. dev.	Sr. v. • Mean	St. dev.
B2	1.15 <sup>a,A</sup>	0.23	0.99 <sup>a,b,A</sup>	0.23	0.80 <sup>b,A</sup>	0.17
F2LC	1.59 <sup>a,A</sup>	0.40	1.65 <sup>a,A</sup>	0.34	1.62 <sup>a,B</sup>	0.35

## Rasprava

Razmatrajući suvremene SIC-ove – brojnost, raznolikost i njihovu sve češću primjenu u širokom spektru kliničkih indikacija u smislu neizbježnog skupljanja materijala sa svim mogućim posljedicama – nameće se važnost novih spoznaja o njihovim dimenzijskim promjenama. Neki istraživači navode da se K SIC-ovi mogu smatrati materijalima s niskim skupljanjem (3, 19), a drugi pak ističu da se SIC-ovii mogu značajno skupljati u usporedbi čak sa skupljanjem kompozitnih materijala (19).

U literaturi su opisane različite metode mjerenja dimenzijskih promjena SIC-ova. Unatoč njihovim specifičnostima, za većinu je zajednička procjena ukupnih dimenzijskih promjena bez mogućnosti njihova praćenja u stvarnom vremenu (3, 4, 18 – 25).

Mjerenja u uređaju digitalne laserske interferometrije počela su odmah nakon pripreme uzorka i trajala su do kraja stvrdnjavanja materijala. Priprema uzoraka trajala je otprilike 45 sekunda nakon završetka miješanja kapsula. U početnoj fazi stvrdnjavanja za sve je materijale zabilježena faza ekspanzije materijala (slika 2.). Stoga je moguće pretpostaviti da je ekspanzija postojala i tijekom pripremanja uzorka, dakle 45 sekunda prije početka interferometrijskih mjerenja. Ekspanzija KM-a trajala je  $4,0 \pm 0,7$  min, a KMQ-a  $3,4 \pm 0,8$  min., no oba su materijala pokazala manje vrijednosti skupljanja u usporedbi sa skupljanjem drugih SIC-ova. Preostala dva K SIC-a (F9F i F9E) imala su ekspanziju od  $2,0 \pm 0,4$  min., odnosno  $0,8 \pm 0,3$  minuta. SM SIC i giomer (F2LC i B2) također su imali laganu ekspanziju tijekom osvjetljivanja. Iz krivulja na slici 2. vidljivo je da se K SIC-ovi šire između prvih  $0,8 \pm 0,3$  minuta do  $4,0 \pm 0,7$  minuta, a SM SIC i giomeri šire se tijekom početnih 10 sekunda svjetlosne polimerizacije. Može se pretpostaviti da je inicijalno širenje materijala koji sadržavaju smolastu komponentu ograničeno polimerizacijom monomera i umreživanjem.

Bilo bi korisno kada bi ekspanzija, koja se pojavljuje rano tijekom stvrdnjavanja, trajala dulje jer bi se tako moglo smanjiti vrijeme skupljanja i kompenzirati negativne učinke skupljanja (14). Kao što je već spomenuto, K SIC-ovi imali su veću i dulju ekspanziju u usporedbi s ekspanzijom SM SIC-ova. Početno širenje F2LC-a i B2 bilo je manje u usporedbi s njihovim skupljanjem (slika 2.). Unatoč uočenoj ekspanziji, zbog velike varijabilnosti između uzoraka u ranoj fazi mjerenja nije je bilo moguće precizno izmjeriti. Ta rana ekspanzija također je zabilježena u studiji Bryanta i Mahlera (19), ali nije određena zbog nestabilnosti materijala u ranoj fazi procesa stvrdnjavanja.

Dosadašnje studije potvrdile su da dimenzijske promjene materijala tijekom stvrdnjavanja ovise o njegovu kemijskom sastavu i o eksperimentalnim uvjetima (3). Budući da K SIC-ovi ne sadržavaju smolaste monomere, mogu se smatrati sporostvrdnjavajućim materijalima, što stvara manji stres na adhezivnoj površini između zuba i restauracije (19). No dodatak smole mijenja način stvrdnjavanja SM SIC-a, što rezultira većim skupljanjem u odnosu prema K SIC-u (21, 29 – 31).

U ovom istraživanju vrijednosti linearnog skupljanja K SIC-ova bile su manje u usporedbi sa svjetlosno polimerizira-

## Discussion

A large range of contemporary GICs and their diverse and growing spectrum of clinical indications, considered in light of unavoidable setting shrinkage with all possible consequences, point to the importance of expanding the knowledge about their dimensional changes. The CGICs can be considered as low-shrinkage materials (3,19). However, some researchers recognized the GICs as materials capable of reaching significant shrinkage values, comparable even to those of resin composite materials (19).

In the literature, various methods of measuring dimensional changes of GIC have been described. Despite some specific differences that are pertinent to each method, the evaluation of the total dimensional changes that occur during the setting can be considered common to most of them (3, 4, 18-25).

The measurements in the digital laser interferometry device started immediately the specimen preparation and lasted throughout the setting time. Specimen preparation lasted approximately 45 s after the capsule mixing had been completed. In the initial phase of the setting, expansion was identified in all materials (Figure 2). It is thus possible to assume that materials also expanded slightly during the specimen preparation, which lasted for 45 s before the interferometric measurements started. KM expanded during  $4.0 \pm 0.7$  min, while KMQ expanded for  $3.4 \pm 0.8$  min; however, both materials showed lower shrinkage values compared to those of the other GICs. The remaining two CGICs (F9F and F9E) expanded through  $2.0 \pm 0.4$  min and  $0.8 \pm 0.3$  min, respectively. The RM GIC and giomer (F2LC and B2) also exhibited a slight expansion during illumination. From the curves in Figure 2., one can observe that CGICs expanded between first  $0.8 \pm 0.3$  min to  $4.0 \pm 0.7$  min, while RM GIC and giomers expanded through the initial 10 seconds of light curing. It can be assumed that the initial expansion in materials containing the resinous component is limited by monomer polymerization and crosslinking.

It would be beneficial if the expansion, occurring early in the curing period, would last longer because it may compensate for the negative effects of shrinkage and reduce the shrinkage time (14). As mentioned earlier, the CGICs had a higher expansion which lasted longer compared to that of RM GIC. The initial expansion of F2LC and B2 was lower compared to the following shrinkage (Figure 2). Despite these findings, it was not possible to precisely quantify the expansion because of high variation in the expansion of specimens in the early stage of measuring. This early expansion was also noticed in the Bryant and Mahler study (19), but was not assessed because of the material instability in early stage of the setting process (19).

A number of previous studies have confirmed that dimensional changes of materials during setting depended upon both the chemical composition and experimental conditions (3). Because of the resin-free structure of the original GICs, they can be considered as slow-setting materials that produce less stress at the tooth-restoration interface (19). However, the addition of the resin changes the way of the

nim materijalima i iznosile su između 0,47 % i 1,32 %, vrijednosti linearnog skupljanja SM SIC-ova bile su između 1,11 % i 1,65 %, a giomera između 0,80 % i 1,15 %. KM se značajno manje skupljao u usporedbi s F9E-om i F9F-om. KMQ također se manje skupljao u usporedbi s F9E-om i F9F-om, no statistička značajnost postojala je samo u usporedbi KMQ-a s F9F-om. Manje skupljanje KM-a i KMQ-a u usporedbi s F9E-om i F9F-om može se pripisati veličini čestica praška. Prosječna veličina čestica u KM-u i KMQ-u manja je (4,7  $\mu\text{m}$  i 6,6  $\mu\text{m}$ ) u usporedbi s česticama praška u F9E-u i F9F-u (14,9  $\mu\text{m}$  i 7,13  $\mu\text{m}$ ) (32, 33). Manje čestice znače manje skupljanje zbog manjeg razmaka između njih i razmjerno manjeg volumena matriksa (34). Neki autori navode da veći omjer praha i tekućine smanjuje skupljanje jer dio čestica praška ne reagira u matrici visokog viskoziteta (19). To je suprotno našim rezultatima jer su se F9E i F9F, koji su formulirani s većim omjerom praška i tekućine (F9F 3,6 g: 1 g, F9E 4,0 g: 1,2 g) (33), snažnije skupljali od KM-a i KMQ-a koji imaju manji omjer prašak/tekućina (3,4 g KM: 1 g i KMQ 3,4 g: 1 g). No uspoređujući KM i KMQ, koji su imali isti omjer praška i tekućine (3,4 g: 1g), ali različitu veličinu čestica (KM 4,7  $\mu\text{m}$  i KMQ 6,6  $\mu\text{m}$ ), može se zaključiti da veličina čestica ima veći utjecaj na skupljanje negoli omjer prašak/tekućina.

Unatoč mnoštvu objavljenih podataka o skupljanju materijala, teško je usporediti rezultate različitih studija zbog varijacija u metodologiji, različitih uvjeta ispitivanja, različitih SIC materijala i polimerizacije uređajima različitih intenziteta svjetlosti. Nekoliko je studija pokazalo da volumno skupljanje različitih SIC-ova i kompozitnih materijala varira od 2 % do 5 % (3, 18, 19, 35). To volumno skupljanje odgovara linearnom skupljanju od 0,75 % do 1,72 %, što se podudara s našim rezultatima skupljanja koji su u rasponu od 0,47 % do 1,65 %. No konkretne brojčane vrijednosti dobivene u našem radu ne mogu se izravno usporediti s onima iz drugih studija zbog jedinstvene geometrije uzoraka i uvjeta mjerenja. Glavno ograničenje našeg eksperimentalnog postavka jest da pozicioniranje diskoidnih uzoraka velikog promjera između PET folija na koje je materijal djelomično pričvršćen može djelovati kao vanjski ograničavajući čimbenik na skupljanje materijala. Štoviše, sama težina zrcala koje je postavljeno na površinu nestvrdnutog uzorka može poremetiti opseg i smjer skupljanja.

Polimerizacijski program dodatni je važan čimbenik u polimerizacijskom skupljanju svjetlom polimeriziranih materijala. Iz slike 2. vidi se da skupljanje B2 i F2LC-a ovisi o intenzitetu svjetlosti. B2 se značajno manje skupljao kada je polimeriziran *niskim* polimerizacijskim programom u usporedbi s *visokim*. Utjecaj polimerizacijskog programa bio je vidljiv i u skupljanju F2LC-a, ali razlika između polimerizacijskih programa nije bila statistički značajna. Tijekom polimerizacije B2 i F2LC-a *niskim* polimerizacijskim programom manje skupljanje je zabilježeno u fazi izlaganja svjetlu nižeg intenziteta, a porast skupljanja s povećanjem intenziteta svjetla. Za giomere zabilježeno je značajno manje skupljanje tijekom *niskog* polimerizacijskog programa u odnosu prema *visokom*. Neki autori navode da smanjeni intenzitet svjetla usporava polimerizaciju, što pridonosi smanjenju polimerizacijskog skupljanja (36).

setting reaction in the RM GICs and consequently results in a larger shrinkage compared to that of CGICs (21, 29-31).

In this study, the values of linear curing shrinkage of the CGICs were lower than those of the light-polymerized materials and ranged between 0.47% and 1.32%, while RM GIC showed linear shrinkage between 1.11% and 1.65%, and giomer had shrinkage values between 0.80% and 1.15%. KM had significantly lower shrinkage compared with F9E and F9F. In addition, KMQ showed lower shrinkage compared to F9E and F9F, but statistical significance was demonstrated only in the comparison of KMQ with F9F. Lower shrinkage of KM and KMQ compared to F9E and F9F may be attributed to the powder glass particles size. The mean particle size in KM and KMQ is lower (4.7  $\mu\text{m}$  and 6.6  $\mu\text{m}$ , respectively) compared to that in F9E and F9F (14.9  $\mu\text{m}$  and 7.13  $\mu\text{m}$ , respectively) (32,33). Small particles imply lower shrinkage due to a smaller distance between the particles and a smaller volume ratio of the matrix (34). Some authors have recognized that a higher powder/liquid ratio leads to lower shrinkage because the part of the particles remains unreacted in the high-viscosity matrix (19). This is contrary to our results because F9E and F9F, which were formulated with a greater powder/liquid ratio (F9F 3.6 g: 1 g, F9E 4.0 g: 1.2 g) (33), had higher values of shrinkage than KM and KMQ with lower powder/liquid ratio (KM 3.4 g: 1g and KMQ 3.4 g: 1 g). However, comparing KM and KMQ, which both had the same powder/liquid ratio (3.4 g: 1g), but different particle size (KM 4.7  $\mu\text{m}$  and KMQ 6.6  $\mu\text{m}$ ), it can be concluded that greater impact on curing shrinkage was exerted by the particles size than by the powder/liquid ratio.

Despite the large amount of published data about polymerization shrinkage, it is difficult to compare the results of different studies because of some variations in methodology, different testing conditions, different GIC materials investigated, and curing using different light intensity. Several studies indicated that volumetric shrinkage of different GICs and resin composites varied from 2% to 5% (3, 18, 19, 35). This volumetric shrinkage corresponds to the linear shrinkage of 0.75% to 1.72%, which coincides with our results of shrinkage in the range of 0.47% to 1.65%. However, the concrete numerical values obtained in our study cannot be directly compared to those from other reports due to unique specimen geometry and measurement conditions. The main limitation of our setup is that positioning the discoid specimens with a high diameter / height ratio between the PET foils, to which the materials partially adhered, might have acted as external constraint on the setting shrinkage. Moreover, the mere weight of the mirror positioned on the unset specimen surface might have interfered with the extent and direction of the setting shrinkage.

An additional important factor in polymerization shrinkage of light-curable materials is the curing mode applied. From the Figure 2, it is apparent that the curing shrinkage of B2 and F2LC depended on the light intensity. B2 showed significantly less shrinkage when polymerized using the "low" curing mode compared to the "high" curing mode. The influence of the curing mode was also observable in F2LC but the difference was not statistically significant. During the light

U ovom istraživanju se F2LC mnogo više skupljao negoli B2. Moguće da velike pre-polimerizirane S-PRG čestice punila, kod kojih se kiselolužnata reakcija dogodila već tijekom proizvodnje giomera, pridonose manjem polimerizacijskom skupljanju (37). Drugi autori također su izmjerili veće skupljanje SM SIC-ova negoli kompozitnih materijala, što se slaže s našim nalazima o većem skupljanju F2LC-a u usporedbi sa skupljanjem kod giomera (38, 39).

Treba istaknuti da su uvjeti ispitivanja u našoj studiji odgovarali kliničkim uvjetima pri izradi restauracije. Mjerenje skupljanja trajalo je malo dulje od vremena preporučenog za obradu restauracija prema uputama proizvođača. Mjerali smo skupljanje materijala prije nego što se izloži slini ili vodenom mediju. U ranijim istraživanjima već je dokazan utjecaj različitih eksperimentalnih uvjeta (25). Zbog hidrofilne prirode i strukture staklenoionomernog cementa, apsorpcija vode potiče djelomičnu kompenzaciju skupljanja (21). No materijal prolazi kroz određene dimenzijske promjene prije bilo kakva kontakta sa slinom. Zato se skupljanje događa dok je cement još zaštićen od izlaganja vodi.

## Zaključak

Rezultati ovog istraživanja pokazuju da dimenzijske promjene tijekom stvrdnjavanja ovise o vrsti materijala – K SIC-ovi imali su niži stupanj skupljanja od SM SIC-a i giomera. Prva hipoteza djelomično je prihvaćena; s obzirom na usporedbu KM-a i KMQ-a s F2LC-om polimeriziranim sa svim trima polimerizacijskim programima, te usporedbu KM-a s B2 polimeriziranim *visokim* polimerizacijskim programom.

Razlike između polimerizacijskih programa bile su statistički značajne samo za B2 između *visokoga* i *niskoga* polimerizacijskog programa. Za F2LC nije zabilježena značajna razlika između triju polimerizacijskih programa.

Ekspanzija materijala tijekom početnog vremena stvrdnjavanja, što smanjuje neto iznos skupljanja, bila je veća i trajala je dulje kod K SIC-a u usporedbi sa SM SIC-om i giomerom.

## Zahvale

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## Sukob interesa

Autori ne navode sukob interesa.

curing of B2 and F2LC, the “soft” curing mode led to the lower shrinkage in the stage of exposure to the lower intensity light and increased with the increase in light intensity. For the giomer, the “low” curing mode produced significantly lower curing shrinkage than the “high” curing mode. Some authors concluded that reduced light intensity leads to slower curing and be beneficial for reducing the setting shrinkage (36). In this study, F2LC showed a more extensive shrinkage than B2. It is possible that the acid-base reaction occurring during the manufacturing of giomer resulted in large pre-polymerized S-PRG fillers that produced less curing shrinkage (37). Other authors also reported higher shrinkage of RM GIC than composite materials, which concurs with our findings of a higher shrinkage in F2LC compared to that in giomer (38, 39).

It should be emphasized that test conditions in our study corresponded to the clinical conditions of restoration placement. Measurement lasted a little bit longer than the manufacturer recommended finishing time. We measured shrinkage of material prior to their exposure to saliva or aqueous media. In some previous studies, the influence of different test condition has already been proven (25). Due to the hydrophilic nature and the structure of glass ionomer cement, water sorption leads to partial compensation of the shrinkage (21). However, the material passes through a certain extent of dimensional changes before any contact with the saliva. Thus, the setting shrinkage takes place while the cement is still protected against water exposure.

## Conclusion

The results of this study show that dimensional changes during setting depended on the material type: the CGICs showed a lower degree of shrinkage than the RM GIC and the giomer. The first hypothesis was partially accepted; considering the comparison between KM and KMQ with F2LC polymerized with all of the three curing modes, as well as the comparison between KM with B2 polymerized with the “high” curing mode.

The differences among curing modes were statistically significant only in B2 between the “high” and the “low” curing mode. There was no significant difference in F2LC between the three curing modes.

The material expansion during the initial setting time, which reduces the net amount of curing shrinkage, was higher and lasted longer in the CGICs compared to the RM GIC and the giomer.

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## Conflict of interest

None declared.



**Abstract**

**Objectives:** The aim of this study was to evaluate dimensional changes of conventional glass ionomer cements, resin-modified glass-ionomer cement, and a giomer during the setting time using digital laser interferometry. Additionally, the influence of different curing modes ("high", "soft", and "low") of a light-emitting diode (LED) curing unit on dimensional changes was evaluated. **Materials and methods:** Linear curing shrinkage of conventional glass ionomer cements (CGICs): Fuji IX Extra (F9E), Fuji IX Fast (F9F), Ketac Molar Aplicap (KM), Ketac Molar Quick Aplicap (KMQ), resin-modified glass ionomer cement (RM GIC): Fuji II LC (F2LC) and giomer: Beautifil II (B2) was analyzed. All tested materials were of shade A3, while all of the GIC were encapsulated. Discoid specimens ( $n=10$ ,  $d=10$  mm,  $h=0.85$  mm) were prepared for each tested material and each curing mode (for light-curable materials) according to the manufacturer's instructions. Light-curable specimens were cured with LED curing unit (Bluephase G2, Ivoclar-Vivadent, and Schaan, Liechtenstein). Dimensional changes during curing were recorded in real-time. The results were analyzed by ANOVA, and Tukey post hoc test was used for multiple comparisons ( $\alpha < 1\%$ ). **Results:** All tested materials showed an initial setting expansion and a subsequent setting shrinkage. KM and KMQ had significantly lower setting shrinkage than RM GIC polymerized using any of the three curing modes. B2 showed lower shrinkage compared to F2LC. **Conclusions:** The extent of curing shrinkage in RM GIC measured in this study can affect longevity of restorations.

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**Key words**

Glass Ionomer Cements; Dental Curing  
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