Effects of Curing Modes on the Microhardness of Resin-Modified Glass Ionomer Cements

Utjecaj načina polimerizacije na mikrotvrdoću staklenoionomernih cemenata modificiranih smolom

Abstract

Objective: This study evaluated the effects of curing modes on surface microhardness of visible light-cured resin-modified glass ionomer cements (VLC RMGIC) and a gionomer after different storage periods in comparison to auto-cured resin-modified glass ionomer cements (AC RMGIC). Materials and Methods: The following materials were used: VLC RMIC: Fuji II LC Improved, Photac Fil Quick Aplicap, AC RMGIC: Fuji Plus, Fuji VIII LC and Gionomer: Beautifil II. The measurements of microhardness were performed using a Vickers test (100 g loads were applied for 10 s) in the following time intervals: immediately after the recommended cure and after 1, 7 and 14 days of immersion in distilled water. Five samples (d=4 mm, h=2 mm) were prepared for each combination of curing mode and tested material. Results: After 14 days, an improvement of microhardness was evident in all tested materials. The full factorial ANOVA identified a highly significant (p<0.001) effect of the factors "material", "time" and "curing mode" ("low", "soft", "high") for the light-cured materials Beautifil II, Fuji II LC and Photac Fil Quick. There was a statistically significant difference in the microhardness between different material types (Beautifil II>Fuji II LC>Photac Fil Quick>Plus>Fuji VIII) and curing modes (low<soft<high). Conclusions: Material type had the greatest impact on microhardness, followed by the factor of time, while curing modes showed a considerably smaller influence on microhardness of the light-cured materials.

Introduction

After almost 45 years of clinical use glass ionomer cements (GICs) have undergone some changes aiming to improve their properties, handling characteristics, efficacy and longevity of restorations. One of important changes is hydrophilic resin monomer incorporation to GIC, resulting in resin-modified glass ionomer cements (RMGIC) (1).

This modification was an attempt to overcome moisture sensitivity and low physical properties of conventional GIC while still maintaining unique properties of GIC such as chemical adhesion to tooth structure and fluoride release (2).

Basically, RMGICs consist of ion-leachable glass, water-soluble polymeric acids, polymerizable organic monomers with appropriate initiation system and water (3). Nowadays the available RMGIC products contain different combinations of methacrylate monomers, among which 2-hydroxyethylmethacrylate is (HEMA) the most commonly used (4-7).

Uvod

Tijekom otprilike 45 godina kliničke uporabe, staklenoionomerni cementi (SIC) podvrgnuti su odredenim promjenama radi poboljšanja njihovih svojstava, lakšeg rukovanja, učinkovitosti i trajnosti restauracije. Jedna od važnijih promena je uvođenje hidrofилног смоластог monomera u SIC, što je rezultiralo SIC-om (SMSIC-om) modificiranim smolom (1).

Tom modificacijom pokuša se riješiti problem osjetljivosti na vlagu i loša fizikalna svojstva konvencionalnog SIC-a, uz istodobno zadržavanje njegovih jedinstvenih svojstava poput kemijske adhezije za zub i otpuštanjafluorida (2).

U osnovi se SMSIC-ovi sastoje od topljivih čestica staklenih polimernih kiselina topljivih u vodi, polymerizirajućih organskih monomerov sa odgovarajućim inicijatorom polimerizacija te vode (2). Danas dostupni SMSIC-ovi sadržavaju različite kombinacije metakrilnih monomerov, među kojima se najčešće koristi 2-hidroksietilmetakrilat (HEMA) (4 – 7).
Resin monomer incorporation modifies the chemistry of the GIC setting reaction (8). The fundamental acid-base reaction begins as soon as the powder (base) and liquid (acid) are combined, forming the network of polysalts. This reaction lasts approximately several minutes, although further maturation continues over extended times (9). Acid-base reaction is supplemented by free-radical mediated polymerization of methacrylate monomers, whereas both reactions occur concurrently, if not disturbed. Monomer polymerization can be induced chemically or photo-chemically depending on the initiator system used. Thus, RMGICs available on the market can be designated as dual cure (acid-base reaction + monomer light-cure or acid-base reaction + monomer auto-cure) or triple cure (acid-base reaction + monomer light-cure + monomer auto-cure) materials.

The set of RMGIC has a complicated structure of inter-penetrating networks of poly (HEMA) and polyacrylate salts in which the unreacted (10), remaining parts of glass particles are embedded.

At an early stage of the setting process, there is a competitive nature of network-forming reactions (11,12) and a sensitive balance exists between these two processes (12).

The setting reaction of light-cured RMGIC (VLC RMGIC) involves a rapid polymerization reaction of the resinous component and a slower acid-base reaction, both of which mutually interact (13). Visible light command set of RMGIC has an advantage in clinical procedure, because polymerized monomer immediately protects the acid-base reaction from the problems of water balance and stabilizes the setting cement thus allowing the continuation of clinical procedure.

It is important to distinguish RMGICs from the materials that resemble GICs, namely polyacid modified resin composite (3) and gionmers that are compositionally more similar to light cured resin composites. Gionmers contain surface reaction type of pre-reacted glass-ionomer (S-PRG) fillers (14) obtained by reacting acid-reactive fluoride-containing glass with polyacids in the presence of water (15) and are capable of fluoride release/recharge in a wet environment.

RMGICs represent an established class of restorative materials with good clinical performance. Nevertheless, potential cytotoxicity may occur from the presence of the residual monomer (16). Palmer et al. (17) investigated HEMA release from Fuji Lining LC, Vitremer, Vitrebond and Fuji II LC and found different material behavior with regards to the effect of curing time.

Apart from the potential cytotoxicity, it is very important to achieve an adequate polymerization of the resinous component in order to ensure basic mechanical properties and longevity of restorations (18). Polymerization effectiveness is influenced by the type of light curing unit (LCU), its irradiance and exposure time (18).

Nowadays, different types of LCUs are being used for the polymerization of light activated resinous materials (20), and halogen and light emitting diode light curing units (LED LCU) are most commonly used „third generation” of LED LCUs which generates multiple wavelengths. The „third generation” of LED LCUs which generates multiple wavelengths Uvođenje smolastog monomera modificira kemijski proces SIC-ove reakcije stvrdnjavanja (8). Osnovna kiselolužnata reakcija počinje čim se prah (lužina) i tekućina (kiselina) povežu, formirajući mrežu polisalca. Ta reakcija traje nekoliko minuta, iako se maturacija nastavlja (9). Kiselolužnata reakcija dopunjena je polimerizacijom metakrilatnih monomera posredovanom slobodnim radikalima, a obje reakcije događaju se istodobno, ako nisu ometane. Polimerizacija monomera može biti inducirana kemijski ili fotokemijski, ovisno o primijenjenom iniciatorskom sustavu. Stoga SMSIC-ovi koji su dostupni na tržištu mogu biti dvostruko stvrdjavajući (kiselolužnata reakcija + svjetlosna polimerizacija monomera ili kiselolužnata reakcija + samostvrdnjavanje monomera) ili trostruko stvrdjavajući (kiselolužnata reakcija + svjetlosna polimerizacija monomera + samostvrdnjavanje monomera).

Stvrdnuti SMSIC ima složenu strukturu interpenetriranih mreža pol(HEMA) i polakrilatnih soli u kojima su smješteni nereagirani (10) preostali dijelovi čestica stakla.

U ranoj fazi procesa stvrdnjavanja prisutan je kompetitivni odnos između reakcija stvaranja mreža (11,12) i postoji osjetljiva ravnoteža između tih dvaju procesa (12).

Reakcija stvrdnjavanja svjetlom polimerizirajućih SMSIC-a (SP SMSIC) uključuje brzu reakciju polimerizacije smolaste komponente i spori kiselolužnata reakciju, a obje uzajamno djeluju (13). Svjetlosna polimerizacija SMSIC-a ima prednost u kliničkom radu, zato što polimerizirani monomer odmah štiti kiselolužnata reakciju od problema s ravnovesom vode i stabilizira stvrdjavajući cement, što omogućuje nastavak kliničkog postupka.

Važno je razlikovati SMSIC-ove od materijala koji sliče SIC-ovima, odnosno od polikiselolinom modificiranih kompozitnih smola (2) i gionmersa koji su prema sastavu više slični svjetlom polimerizirajućim smolastim kompozitima. Giomeri sadržavaju površinski tip već reagiranih staklenionomerskih (S-PRG) čestica punila (14) dobivenih reakcijom reakcionoga fluoridnog stakla s polikiselolinama uz prisutnost vode (15) te mogu otpuštati/primati fluoride u vlažnim uvjetima. SMSIC-ovi su dobro poznati restaurativni materijali s dobrim kliničkim učincima. Unatoč tomu može se pojaviti citotoksčnost zbog rezidualnog monomera (16), Palmer i suradnici (17) istraživali su otpuštanje HEMA-e iz Fuji Lining LC-a, Vitremera, Vitrebonda i Fuji II LC-a te otkrili različito ponašanje materijala u odnosu prema trajanju polimerizacije.

Uz potencijalnu citotoksčnost, iznimno je važno postići odgovarajuću polimerizaciju smolaste komponente kako bi se postigla osnovna mehanička svojstva i dugotrajnost restauracije (18). Na učinkovitost polimerizacije utječu vrsta polimerizacijskog uređaja (PU), snaga i valna duljina. Na učinkovitost i dugotrajnost restauracije utječe i tip polimerizacijskog uređaja (PU) i dijelov snage i valne duljine (LED). Posebno je učinkovita treća generacija LED uređaja koji generiraju višestruke valne duljine, poprilično učinkovite za polimerizaciju bilo koje vrste dentalnih restaurativnih materijala (20).
is particularly effective for polymerization of any type of dental restorative material (20).

Multiple studies have compared the effectiveness of the halogen and LED LCU with similar (600 mW/cm²) (21), or different light intensities (halogen 350 mW/cm², LED 1400 mW/cm², LED 1100 mW/cm²) (22), and with different light intensity and exposure time (halogen 700 mW/cm², LED 500 mW/cm²; 20 s and 40 s) on the microhardness (MH) of RMGIC (23). Microhardness (MH) value was dependent on the light used to cure them (21,22). In relation to the curing time, similar MH results were obtained with halogen and LED 40 s time exposure, while a significant difference was found between halogen 40 s and LED 20 s (23). Parisay et al. (24) also compared halogen and LED curing units with different light intensity (halogen 600 mW/cm², LED 700 mW/cm²), and exposure time (20 s, 30 s, 40 s) on top and bottom MH. The top surface MH was higher in all experimental groups. An increase in both the top and the bottom MH was observed when exposure time was increased. In the halogen group, there were no significant differences between 20, 30 and 40 s, while in the LED group, 40 s curing yielded significantly better results than 20 and 30 s.

Also, there are studies that compared MH of resin-modified versus conventional GIC with regards to depth of cure and post-irradiation hardness (25-30).

Considering the depth of cure, VLC RMGIC should be placed in layers which by manufactures recommendations should not exceed 2 mm. Contrary, auto-cured RMGIC (AC RMGIC) may be placed in bulk and are clinically more convenient.

The aim of this study was to assess the MH of AC RMGICs, as well as VLC RMGICs and giomer light-cured with three different curing modes of LED unit after artificial aging for up to 14 days.

Research hypotheses tested were:

1) MH values will be affected by material type; giomer is expected to show higher MH than GICs, whereas AC and VLC RMGICs are expected to show similar MH values
2) MH values of VLC RMGICs will be influenced by three different curing modes
3) MH values will improve with artificial aging.

Material and methods

Microhardness measurements

This study evaluated two VLC RMGICs: (Fuji II LC (F2LC), Photac Fil Quick Aplicap (PFQ)), two AC RMGICs: (Fuji Plus (FP), Fuji VIII GP (FVIII)) and a giomer: (Beautifil II (B2)). All tested materials were of A3 shade and their detailed composition is given in Table 1.

For each experimental group, five cylindrical specimens (d=4 mm, h=2 mm) were prepared using Teflon molds. For encapsulated materials F2LC, PFQ, FP and FVIII, the capsules were mixed according to respective manufacturers’ instructions and then extruded into molds. B2 was applied into molds using a spatula, taking care not to incorporate air inclusions. Mold openings were covered with a polyethyl-

Mnogobrojne su studije usporedivale učinkovitost halogeni i LED polimerizacijskih uređaja sa sličnom (600 mW/cm²) (21) ili različitim jačinom svjetlosti (halogeni 350 mW/cm², LED 1400 mW/cm², LED 1100 mW/cm²) (22) te s različitim jačinom svjetlosti i vremenom izloženosti (halogeni 700 mW/cm², LED 500 mW/cm²; 20 s i 40 s) na mikrotvrđućo (MT) SMSIC-ova (23). Vrijednost mikrotvrđeće (MT-a) ovisila je o svjetlu koje je korišteno za polimerizaciju (21, 22). U odnosu prema trajanju polimerizacije, slični rezultati MT-a dobiveni su halogenim i LED polimerizacijskim uređajima uz izloženost od 40 sekunda, a znatne su razlike zabilježene između izloženosti halogenim uređajima u trajanju od 40 sekunda i LED-u u trajanju od 20 sekunda (23). Parisay i suradnici (24) također su uspoređivali halogene polimerizacijske uređaje i LED uređaje uz različitu jačinu svjetla (halogeni 600 mW/cm², LED 700 mW/cm²) i vrijeme polimerizacije (20 s, 30 s, 40 s) na MT gornje i donje površine uzorka. MT na gornjoj površini bio je viši u svim eksperimentalnim skupinama. Uz produljeno vrijeme polimerizacije, zabilježeno je povećanje MT-a na gornjoj i donjoj površini uzorka. U halogenoj skupini nije bilo značajnih razlika između 20, 30 i 40 sekunda, a u LED skupini 40-sekundna polimerizacija pokazala je znatno bolje rezultate od 20- i 30-sekundne.

I u drugim istraživanjima uspoređivan je MT halogen modifikiranih i konvencionalnih SIC-ova u odnosu prema dubini polimerizacije i tvrđeći nakon osvjetljavanja (25 – 30).

S obzirom na dubinu polimerizacije, SP SMSIC-ovi trebali bi se postavljati u slojevima koji prema preporukama proizvođača ne bi smjeli prelaziti 2 milimetra. Nasuprot tomu, samostvrdnjavajući SMSIC-ovi (SS SMSIC-ovi) mogu biti postavljeni u debelom sloju i klinički su prikladniji.

Cilj ovog istraživanja bio je ispitati mikrotvrđeću MT SMSIC-ova, te SP SMSIC-ova i giomera polimeriziranih trima različitim polimerizacijskim programima LED uređaja tijekom umjetnog starenja od 14 dana.

Ispitivale su se sljedeće hipoteze:

1) vrsta materijala utjecat će na vrijednosti MT-a, očekuje se da će giomer imati veći MT od SIC-ova, a SS i SP SMSIC-ovi trebali bi imati slične vrijednosti MT-a,
2) polimerizacijski programi utjecat će na MT SP SMSIC-ova,
3) MT materijala rast će s duljinom umjetnog starenja.

Metoda

Mjerenje mikrotvrđeće

U ovom radu ispitana su dva SP SMSIC-ova (Fuji II LC (F2LC), Photac Fil Quick Aplicap (PFQ)), dva SS SMSIC-ova (Fuji Plus (FP), Fuji VIII GP (FVIII)) i jedan giomer (Beautifil II (B2)). Svi testirani materijali bili su u nijansi A3, a njihov detaljni sastav naveden je u tablici 1.

U svakoj ekperimentalnoj skupini pripremljeno je pet cilindričnih uzoraka (ø = 4 mm, v = 2 mm) s pomoću teflonskih kalupa. Za kapsulirane materijale F2LC, PFQ, FP i FVIII kapsule su miješane u skladu s preporukama proizvođača, a zatim je materijal u kapsulu istisnut u kalupe. B2 je u kalupe stavljen instrumentom 5/6 prema Heidemannu, pažnju pribor da se ne unese zrak. Otvore kalupa pokriveni su...
ene terephthalate (PET) film and compressed using a stainless steel slab to remove the excess material. For the light-curing materials, specimens were illuminated with a LED LCU (Bluephase G2, Ivoclar-Vivadent, Schaan, Liechtenstein) positioned immediately above the specimen. Each experimental group was illuminated with one of the following light-curing modes for 20 s: low (650 mW/cm²), soft (650-1100 mW/cm²) and high (1100 mW/cm²). Specimen preparation was performed at room temperature, while subsequent aging was simulated by the storage at 37±1°C in deionized water in an filmom od polietilentaereftalata (PET) i stisnuti pločom od nehrđajućeg čelika kako bi se uklonio višak materijala. Za materijale koji se polimeriziraju svjetlom, uzorci su polimerizirani LED polimerizacijskim uređajem (Bluephase G2, Ivoclar-Vivadent, Schaan, Liechtenstein) postavljen izravno nad uzorkom. Svaka eksperimentalna skupina osvjetljena je jednim od sljedećih programa polimerizacije tijekom 20 sekunda: niskim (650 mW/cm²), POSTUPNIM (650 – 1100 mW/cm²) i visokim (1100 mW/cm²). Priprema uzoraka obavljena je na sobnoj temperaturi, a umjetno starenje poslije toga.

### Table 1
Materials used for the measurement of Vickers microhardness

| Kod materijala | Ime proizvoda | Vrsta materijala | Proizvođač • Manufacturer | Prijama • Shade EXP LOT | Sastav • Composition |
|-----------------|---------------|-------------------|----------------------------|--------------------------|----------------------|
| F2LC            | Fuji II LC    | Smolom modificirani GIC | GC Corporation, Tokyo, Japan | A3 2018-10-26 1610278 | Tekućina: polietilena kiselina; HEMA; 2,2,4 TMHEDC; TEGDMA. Prah: fluoroalumosilikatno staklo • Liquid: poliacrylaci acid; HEMA; 2,2,4 TMHEDC; TEGDMA. Powder: fluoro-alumino-silicate glass. |
| PFQ             | Photac Fil Quick Aplicap | Smolom modificirani GIC | 3M, Seefeld, Njemačka | A3 2019-02 669852 | Tekućina: staklenionomerneni kompatibilni monomeri i oligomeri; akrilno- i maleinsko-kiselinski kopolimeri; kamforkinon; stabilizatori; voda. Prah: Na-Ca-Al-La fluosilikatno staklo; aminoaktivator • Liquid: glass ionomer compatible monomers and oligomers; acryl- and maleic-acid copolymers; camphorquinone; stabilizers; water. Powder: Na-Ca-Al-La fluosilicate glass; amine activator. |
| FVIII           | Fuji VIII GP  | Smolom modificirani GIC | GC Corporation, Tokyo, Japan | A3 2019-08-06 1708071 | Tekućina: 2-HEMA 25–50%; vinska kiselina 5–10%; 7,7,9 (ili 7,9,9)-trimetil-4,13-diokso-3,14-dioksa-5,12-diazoheksadekan-1,16-diyl bismetakrilat 1–5%; 2-hidroksi-1,3-dimetakriloksipropan 1–5%. Prah: N/A • Liquid: 2-HEMA 25–50%; tartaric acid 5–10%; 7,7,9 (or 7,9,9)-trimetil-4,13-dioxo-3,14-dioxa-5,12-diazaheksadecane-1,16-diyl bismetacrylate 1–5%; 2-Hydroxy-1,3 dimetacryloxypropane 1–5% Powder: N/A. |
| FP              | Fuji PLUS     | Smolom modificirani GIC | GC Corporation, Tokyo, Japan | A3 2019-07-19 1707201 | Tekućina: 2-HEMA 25–50%; 7,7,9 (ili 7,9,9)-trimetil-4,13-dioksa-3,14-dioxa-5,12-diazoheksadekan-1,16-diyl bismetakrilat 1–5%; 2-hidroksi-1,3-dimetakriloksipropan 1–5%. Prah: N/A • Liquid: 2-HEMA 25–50%; 7,7,9 (or 7,9,9)-trimetil-4,13-dioxo-3,14-dioxa-5,12-diazaheksadecane-1,16-diyl bismetacrylate 1–5%; 2-Hydroxy-1,3 dimetacryloxypropane 1–5% Powder: N/A. |
| B2              | Beautifil II  | Giomer, Stvrdnjavajući | Shofu Inc., Kjoto, Japan | A3 2020-04-30 051705 | Bis-GMA 7,5%; trietilen-glikol dimetakrilat 5%; alumofluoroborosilikatno staklo 7,5% Al₂O₃, DL-kamforkinon. • Bis-GMA 7,5%; Trietilen-glikol dimetacrylate 5%; Alumofluoroborosilicate glass 7,5%; Al₂O₃, DL-Camphorquinone. |

(2,2,4 TMHEDC = 2,2,4 trimetil heksametilen dicarbonat; HEMA = 2-hidroksietilmetakrilat; TEGDMA = trietilen-glikol dimetakrilat) • (2,2,4 TMHEDC = 2,2,4 trimetil hexamethylen dicarbonate; HEMA = 2-hydroxyethylmetacrylate; TEGDMA = trietilene glycol dimetacrylate)
incubator (Cultura, Ivoclar-Vivadent, Schaan, Liechtenstein) for up to 14 days.

Vickers MH was evaluated at four time intervals: immediately after specimen preparation and manufacturer recommended setting time 0 day, 1 day, 7 days and 14 days. The surfaces of aged specimens were gently blotted to remove water before the measurements. A diamond pyramid was indentated into irradiated specimen surfaces using the Leitz Miniload 2 microhardness tester (Leitz, Oberkochen, Germany) with a load of 100 g, dwell time of 10 s and 5 indentations per specimen. MH was calculated by the following equation: \(HV = \frac{1.8544xF}{d^2}\), where \(d\) is the diagonal of the indentation and \(F = m \times g\) (\(g = 9.81\) N/kg, \(m\) = load).

Statistical analysis

Normality of distribution and homogeneity of variances was confirmed using the Shapiro-Wilk’s and the increase of MH for each experimental group were compared using a mixed model ANOVA, which accounted for the clustering effect of the repeated measurements performed on individual specimens (31). Post-hoc multiple comparisons were made using a Tukey’s HSD correction. Partial eta-squared statistics were used to evaluate relative influences of the factors “time”, “material” and “curing mode”. Statistical software SPSS 20 (IBM, Armonk, NY, USA) was used, with the level of significance set at 0.05.

Results

Mean MH values as a function of material, aging time and curing mode are presented in Figure 1. The full factorial ANOVA which considered the light-cured materials B2, F2LC and PFQ identified a highly significant \((p < 0.001)\) effect of the factors “material”, “time” and “curing mode”, with the relative influences of individual factors represented by partial eta squared values of 0.991, 0.778 and 0.171, respectively.

The highest partial eta squared for the factor “material” indicates that the highest amount of variation of the dependent variable MH is attributable to the material type. For individual materials, MH values were in the following ranges: 70.4-74.4 for B2, 34.1-44.1 for F2LC, 26.7-41.1 for PFQ, 31.0-40.5 for FP and 27.8-38.6 for and FVIII.

The factor “time” showed a significant interaction \((p < 0.001)\) with the factor “material”, reflecting a more pronounced MH improvement with time in the group of glassionomer-based materials compared to the gionomer B2. After 14 days, the MH values for B2 improved for 2.9-5.7% compared to the immediate (0-day) values, whereas a much more extensive improvement was identified in the glassionomer-based materials: 20.9-22.4% in F2LC, 47.7-51.0% in PFQ, 30.9% in FP and 38.7% in FVIII.

Although statistically significant, the influence of the factor “curing mode” on MH was the weakest among all tested factors. This modest effect of different curing modes is visualized in Figure 1. Simulated is pohranom u deionizovanoj vodi u inkubatoru (Cultura, Ivoclar-Vivadent, Schaan, Liechtenstein) pri 37 ± 1 °C u razdoblju do 14 dana.

MT prema Vickersu mjerjen je u četiri vremenska intervala: 0. dan, tj. neposredno nakon pripreme uzorka i nakon vremena stvrdnjavanja prema preporuci proizvođača, 1. dan, 7. dan i 14. dan. Prije mjerenja s površine pohranjenih uzoraka nježno je uklonjena voda. Dijamantna piramida utisnuta je u stvrdnutu površinu uzorka primjenom uređaja za mjerenje mikrotvrdoće Leitz Miniload 2 (Leitz, Oberkochen, Njemačka) opterećenjem od 100 g, trajanjem opterećenja 10 sekunda i s 5 utisnucima po uzorku. MT je izračunat prema sljedećoj jednadžbi: \(MT = \frac{1.8544 x F}{d^2}\), pri čemu je \(d\) dijagonala utisnucera u (g = 9,81 N/kg, \(m\) = teret).

Statistička analiza

Normalnost distribucije i homogenost varijanci potvrđena je najprije Shapiro-Wilkovim testom, a zatim Leveneovim. Pretpostavka sferičnosti za ponovljena mjerenja potvrđena je Mauchlyjevim testom. Srednje vrijednosti MT-a za svaku eksperimentalnu skupinu uspoređene su mješanim modelom ANOVA-e kojim je uzet u obzir učinak grupiranja ponovljena mjerenja obavljenih na pojedinačnim uzorcima (31). Mnoge post-hoc usporedbi obavljene su Tukeyjevim HSD testom. Statistika parcijalnog eta-kvadrat testiranja koštena je za procjenu relativnih učinaka čimbenika vrijeme, materijal i polimerizacijski program. Korišten je statistički softver SPSS 20 (IBM, Armonk, NY, SAD), uz razinu značajnosti 0,05.

Rezultati

Srednje vrijednosti MT-a kao funkcije materijala, vremena starenja i polimerizacijskog programa prikazane su na slici 1. Potpuna faktorska ANOVA kojom su uzeti u obzir svjetlom polimerizirajući materijali B2, F2LC i PFQ zabilježila je iznimno značajan \((p < 0.001)\) učinak čimbenika materijal, vrijeme i polimerizacijski program, uz razmjernije utjecaje pojedinačnih čimbenika prikazanih parcijalnim eta-kvadrat vrijednostima od 0,991, 0,778 i 0,171 (tim redom).

Najviša vrijednost parcijalnog eta-kvadrata za čimbenik materijal pokazuje da se najveće varijacije ovisne varijable MT-a mogu pripisati vrsti materijala. Za pojedinačni materijal vrijednosti MT-a bile su u sljedećim intervalima: 70,4 – 74,4 za B2, 34,1 – 44,1 za F2LC, 26,7 – 41,1 za PFQ, 31,0 – 40,5 za FP i 27,8 – 38,6 za FVIII.

Čimbenik vrijeme pokazao je značajnu interakciju \((p < 0,001)\) s čimbenikom materijal, odražavajući izraženije po boljanje MT-a s vremenom u skupini stakloionomernih materijala u usporedbi s gionomerom B2. Nakon 14 dana vrijednosti MT-a za B2 poboljšale su se za 2,9 do 5,7 % u usporedbi s neposrednim (0. danom) vrijednostima, a znatno veće poboljšanje zabilježeno je za stakloionomerne materijale: 20,9 – 22,4 % u F2LC-u, 47,7 – 51,0 % u PFQ-u, 30,9 % u FP-u i 38,7 % u FVIII.

Iako je statistički značajan, učinak čimbenika polimerizacijski program na MT bio je najslobodniji od svih ispitanih čimbenika. Taj skromni učinak različitih polimerizacijskih
ally observable in Figure 1 within all combinations of factors “material” and “time” as a trend of increase in the order of low < soft < high. However, due to the small effect size, the differences among curing modes were statistically significant only in B2 after 1 and 7 days and in F2LC after 14 days. In these cases, the MH appeared significantly higher for the “high” curing mode compared to the “low” mode.

**Discussion**

The aim of this study was to determine the MH of AC and VLC RMGICs, as well as a giomer and to determine a dependence of the curing type (AC/VLC) and storage time on the MH as well as a dependence of three different curing modes (“low”, “soft”, “high”) of the LED curing unit and storage time.

In previous studies, the dependence of the MH of composite materials on the applied polymerization modes of halogen curing units was demonstrated. In a study by Soh and Yap (32) the polymer structure of composite materials was curing mode dependent. Pulse delay curing mode resulted in lower crosslink density and lower Knoop hardness. In da Silva’s research (33), higher values of Knoop MH of composite materials were found by exposing the material to high polymerization mode instead of standard and gradual.

A high power LED LCU was chosen in this study since such a curing unit type is the most frequently used in current clinical practice. Hardness was evaluated after four post-irradiation time intervals, starting immediately after manufacturer recommended curing time and followed by artificial aging for 1, 7 and 14 days. For the first measurement, the same programa može se vidjeti na slici 1. kod svih kombinacija čimbenika materijal i vrijeme kao trend povećanja prema redoslijedu niski < postupni < visoki. No zbog malog učinka, razlike između polimerizacijskih programa statistički su znatno samo za B2 nakon jednog dana i 7 dana te za F2LC nakon 14 dana. U tim je slučajevima MT znatno visi za visoki polimerizacijski program u usporedbi s niskim.

**Rasprava**

Cilj ovog istraživanja bio je odrediti mikrotvrdoću SS SMSIC-ova i SP SMSIC-ova i giomera te utvrditi ovisnost MT-a o načinu stvrdnjivanja materijala (samostvrdnjavanje/svjetlosna polimerizacija) i vremenu pohrane, te ovisnost MT-a o trima različitim polimerizacijskim programima (niski, postupni, visoki) LED polimerizacijskog uređaja i vremenu pohrane.

U prijašnjim istraživanjima istaknuta je ovisnost MT-a kompozitnih materijala o primijenjenom polimerizacijskom programu halogenih polimerizacijskih uređaja. U studiji Soaha i Yap (32) polimerna struktura kompozitnih materijala bila je ovisna o polimerizacijskom programu. Program polimerizacije Pulse delay rezultirao je slabijom gustoćom polimernih mreža i slabijom tvrdoćom prema Knoopu. U da Silvinu istraživanju (33) više vrijednosti MT-a kompozitnih materijala prema Knoopu zabilježene su nakon izlaganja materijala visokom programu polimerizacije umjesto standardnog i postupnog.

U ovom radu odabrana je LED polimerizacijski uređaj jer je snage zato što se takav uređaj najčešće koristi u suvremenoj kliničkoj praksi. Tvrdoca je određena četiri puta nakon...
Microwave-radiated staklenionomernih cementa smolom modificiranih Spajic et al.

evaluate MH at earlier time points, thus providing no data LCU (LED 1100 mW/cm²), which agrees with our MH values fifteen minutes after curing were achieved by Bluephase infrared (FTIR) study of visible light-cured RMGICs Fuji reaction, thus inhibiting it (11, 36). In a Fourier-transform infrared (FTIR) study of visible light-cured RMGICs Fuji osvjetljuvanja – prvi put neposredno nakon proizvodlječeva preporučenog vremena stvrdnjivanja i zatim nakon umjetnog stvrdnjavanja od 1, 7 i 14 dana. Za prvo mjerenje uzorci nisu bili u doticaju s medijem za pohranjivanje, tako se oponaša liničku situaciju u kojoj je restauraciju izolirana od sline i vode tijekom postavljanja i svjetlosne polimerizacije. Nakon toga restauracije su trajno izložene vlažnim uvjetima tijekom njihova trajanja u usitima. Iako je poznato da MT varira kroz preporučenu debljinu sloja (23, 31), ovo istraživanje usredotočeno je na učinak različitih polimerizacijskih programa na MT površine uzorka, u skladu s ranijim istraživanjima (27).

Od svih ispitanih materijala, B2 je imao najveće vrijednosti MT-a koje su bile gotovo dva puta veće od vrijednosti ispitanih SMSIC-ova. Takav nalaz očekivao je zbog sličnosti sastava giomera kompozitnim smolama, unatoč razlici u anorganskom punitu.

MT-i B2 bili su u rasponu od 70,4 ± 2,5 do 74,4 ± 2,2, slika 1. Ti nalazi mogu se usporediti s nalazima Mobarak i sur. (22) u studiji o utjecaju različitih polimerizacijskih uređaja (halogenih 350 mW/cm², LED 1400 mW/cm², LED 1100 mW/cm²). Najveće vrijednosti mikrotvrdoće Beautifil-a (72,9 ± 0,9) čiji je sastav sličan sastavu B2, izmjerene petnaest minuta nakon stvrdnjivanja, postignute su uređajem Bluephase (LED 1100 mW/cm²), što odgovara našim vrijednostima MT-a dobivenima tijekom polimerizacije visokim programom neposredno nakon stvrdnjivanja (71,7 ± 2,4).

Tijekom 14 dana umjetnog starenja, vrijednosti MT-a za B2 polako su, ali kontinuirano raste. Yap i suradnici (34) ispitali su tvrdoću Beautifil-a nakon 30 dana pohranе u destiliranoj vodi i bili su MT-a dobivenima tijekom polimerizacije visokim programom neposredno nakon stvrdnjivanja (71,7 ± 2,4, slika 4). Ucitavanje MT-a u ranijim vremenom intervalima, zbog čega ne postoji podaci koji bi se mogli usporediti s našim rezultatima. SP SMSIC-ovi pokazali su sličan utjecaj polimerizacijskih programa na MT kao i B2. Statistički značajan utjecaj polimerizacijskog programa koji je dokazan omnibus-testom upućuje da je MT u pozitivnom odnosu s ukupnom energijom zračenja koju je primila površina uzoraka. Energija zračenja (J) rabiljena pri svakom programu stvrdnjivanja (nizak – 9,9 J, postupan – 14,7 J, visok – 17,5 J) odgovarala rasponu vrijednosti MT-a: niska < postupna < visoka (35).

VLC RMGICs exhibited a similar influence of curing modes on MH as it was in B2. A statistically significant effect of the curing mode identified in the omnibus test suggests that MH was positively related to the total radiant energy received by specimen surfaces. Radiant energy (J) delivered by each curing mode (low 9.9, soft 14.7, high 17.5) concurs with the ranking of MH values: low < soft < high (35). The inability of the statistical analysis to detect significant differences within each material x time combination was due to a very small effect size. When the curing mode “high” was used instead of “low”, the differences in MH amounted to about 1-3, which represents an improvement in the range of 2-7%.

A similar influence of the curing modes in the polymerization process of two different material groups tested is an interesting finding since the setting mechanism of the RMGICs involves acid-base reaction and polymerization of resinous part of the cement. Both reactions are diffusion-controlled; therefore their rates decrease as the network formation impairs the diffusion of reactive species. Since the free-radical-mediated polymerization reaction is inherently faster than the acid-base reaction, the light activation causes the resinous network formation to occur ahead of the acid-base reaction, thus inhibiting it (11, 36). In a Fourier-transform infrared (FTIR) study of visible light-cured RMGICs Fuji...
II LC and Photac Fil Quick Aplicap, the rate of the acid-base reaction was the highest during the light activation (37). These results were explained by the decline in the reaction rate of the diffusion-controlled acid-base reaction through the formation of an infinite methacrylate network (gelation) and additionally by the contribution of the heating effect which originated from the exothermic polymerization reaction, as well as from the curing unit (37).

However, our results showed that MH of VLC RMGICs continued to increase for 14 days post-irradiation. These findings correspond to Ellakuria et al. (26). They found increasing MH up to 15 days, after which MH decreased up to 12 months.

Young (38) investigated conventional GIC (Fuji IX) and RMGIC (Fuji II LC Improved) by FTIR investigation and observed two separate diffusion mechanisms for acid neutralization. The earlier-occurring, faster reaction stopped after 30 and 150 min respectively, suggesting that the water required for its progress was depleted after a certain time period. The slower reaction was also an acid-base neutralization which was triggered by absorbed water, therefore occurring with a delay. Thus, the post-irradiation increase in MH may be mainly addressed to the continuation of the acid-base reaction.

There are several materials related factors that may influence the mechanical properties of RMGIC such as resin monomer content, type of resin monomers, particle volume and size, polymeric acids molecular weight, number and size of voids and water content. From the point of MH, particle volume and size are of particular interest.

Mean particle size of F2LC and PFQ are 5.9 μm and 5.56 μm respectively (39) and even less in F2LC Improved (38).

Xie et al. (40) evaluated mechanical properties and microstructures of GICs and found that the mechanical properties were closely related to their microstructures. The Knoop hardness (KHN) obtained in their study was significantly greater for Fuji II LC Improved than for Photac Fil, as it was in our study comparing F2LC and PFQ. Based on the SEM fractography, the author suggested that in the case of Fuji II LC Improved a better indentation resistance might be attributed to homogeneously distributed small glass particles, while on the fractured surface of Photac Fil less exposed glass particles well-integrated in the resinous matrix were identified.

MH values for PFQ significantly increased from 0 d to 1 d, followed by a less pronounced trend of increase towards 14 d. This evidence might be explained by research of Kako-boura et al. (41) who used FTIR analysis and found that acid-base reaction of F2LC, Photac-Fil Aplicap (earlier formulation of PFQ) and Vitremer was significantly slowed by the light polymerization. Between the tested materials, the smallest extent of the acid-base reaction was found in Photac-Fil Aplicap. The authors attributed these findings to the majority of its polyacrylic acid added to powder component, which required more water infiltration for full reaction. This could explain significantly lower MH values measured for PFQ in our study at 0 d in all three curing modes when samples were without contact to storage media. After 1 d aging in de-smolaste mreže prije kiselo-lužnate reakcije, čime je inhibira (11, 36). U istraživanju s pomoću infracrvene Fourier-Transform (FTIR) analize svjetlom svrstavajućih SMSC-ova Fuji II LC i Photac Fil Quick Aplicap, brzina kiselo-lužnate reakcije bila je najveća tijekom aktivacije svjetlom (37). Ti su rezultati objašnjeni padom u procesu kiselo-lužnate reakcije kontroliranom difuzijom te stvaranjem mreže metakrila (gelacije) i dodatno doprinosom zagrijavanja koji se pojavljuje zbog egzogermičke reakcije polimerizacije te zagrijavanja polimerizacijskog uređaja (37).

No naši rezultati pokazali su da se MT SP SMSIC-a nastavila povećavati 14 dana nakon osvjetlivanja. Ta zapažanja odgovaraju rezultatima Ellakuria i suradnika (26). Oni su povećanje MT-a zabilježili do 15 dana, nakon čega se smaživala do 12 mjeseci.

Young (38) je istraživao konvencionalni SIC (Fuji IX) i SMSIC (Fuji II LC Improved) analizom FTIR i zabilježio dva odvojena mehanizma difuzije za neutralizaciju kiseline. Reakcija koja je pojavljivala ranije i bila je brža, prestala je nakon 30 i 150 minuta (tim slicecom), što sugerira da se voda koja je potrebna za njezin nastavak potrošila nakon određenog vremena. Sporija reakcija također je bila kiselo-lužnata neutralizacija koju je potaknula apsorbirana voda te se zato pojavila s odgodom. Zato se povećanje MT-a nakon osvjetlivanja uglavnom može pripisati nastavljanju kiselo-lužne reakcije.

Nekoliko je čimbenika vezanih uz vrstu materijala koji mogu utjecati na mehanička svojstva SMSIC-ova, kao što su sadržaj smolastog monomera, tip smolastih monomerima, veličina i opseg čestica, molekularna težina polimernih kiselina, broj i veličina pora te sadržaj vode. Za MT su opseg i veličina čestica od posebnog interesa.

Srednja veličina čestica F2LC i PFQ jest 5,9 μm i 5,56 μm (39), a čak i manja u F2LC Improvedu (38).

Xie i suradnici (40) ispitali su mehanička svojstva i mikrostrukturu SIC-a i ustanovili da su mehanička svojstva usko povezana s mikrostrukturom. Tvrdoća prema Knoopu (KHN), dobivena tijekom njihova istraživanja, znatno je veća za Fuji II LC Improved negoli za Photac Fil, kao i u našem istraživanju u kojem su uspoređeni F2LC i PFQ. Na osnovi SEM fraktografije, autor ističe da se u slučaju Fuji II LC Improveda veći otpor na utiskivanje može pripisati homogenom distribuiranim malim česticama stakla, a na lomnoj površini Photac Fila zabilježene su manje izložene čestice stakla dobro integrirane u smolastu matricu.

Vrijednosti MT-a za PFQ značajno su se povećale od 0. o 1. dana, nakon čega je slijedio slabiji trend povećanja prema 14. dana. Ovaj nalaz može se objasniti radom Kaboura i suradnika (41) koji su, koristeći analizom FTIR, ustanovili da je kiselo-lužnata reakcija F2LC-a, Photac-Fil Aplicapa (ranije formulacije PFQ-a) i Vitremera znatno usporena procesom polimerizacije. Među ispitanim materijalima najmanji stupanj kiselo-lužne reakcije zabilježen je za Photac-Fil Aplicap. Autori su takva zapažanja pripisali polikrtilnoj kiselinii koja je uglavnom dodana komponenti praha, što za potpunu reakciju zahtijeva infiltraciju veće količine vode. To bi moglo objasniti znatno niži vrijednosti MT-a izmjerene za PFQ u našoj studiji na 0. dan u svim trima polimerizacijskim programima kada uzorci nisu bili u doticaju s vo-
ionized water, water sorption initiated more acid-base reaction resulting in significantly increased MH. Between tested materials, including AC RMGICs, such behaviour was only observed in PFQ and this could be due to the proprietary material composition which is not fully disclosed by the manufacturer.

AC RMGICs, FP, and FVIII, although for different clinical use (luting of indirect restorations and restorative treatment, respectively) exhibited MH values in the range of VLC RMGICs. Also, the influence of aging time was similar to that observed in VLC RMGIC products. It can be assumed that despite the monomer polymerization in AC RMGICs is longer than in VLC RMGICs, monomer polymerization influenced the acid-base reaction in the same way in both material groups, resulting in a continuation of the and acid-base reaction in tested time period, which was reflected as an increase in MH.

MH values attained by different curing modes were positively related to radiant energy delivered to the specimen surface. Despite being statistically significant, the differences among curing modes can be considered negligible from the clinical standpoint. Material composition exerted a stronger effect on MH values, suggesting that the material choice had more impact on the mechanical properties than the curing mode. An improvement in MH was identified in all materials after an artificial aging of 14 days, reflecting the long-term continuation of the setting reaction.

Conclusions

All curing modes investigated can be effectively used in clinical work. Material type had more impact on microhardness than the curing mode. An improvement in MH was observed for all tested materials, including AC RMGICs, FP, and FVIII, although for different clinical use (luting of indirect restorations and restorative treatment, respectively) exhibited MH values in the range of VLC RMGICs. Also, the influence of aging time was similar to that observed in VLC RMGIC products. It can be assumed that despite the monomer polymerization in AC RMGICs is longer than in VLC RMGICs, monomer polymerization influenced the acid-base reaction in the same way in both material groups, resulting in a continuation of the and acid-base reaction in tested time period, which was reflected as an increase in MH.

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