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Effect of Rapid Polymerization on Water Sorption and Solubility of Bulk-fill Composites

Utjecaj brze polimerizacije na apsorpciju vode i topljivost bulk-fill *kompozita*

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Abstract

Objectives: The aim of the study was to examine the impact of rapid high-intensity polymerization on water sorption and solubility of a new generation of bulk-fill composite materials. **Materials and methods:** Five materials were tested: a conventional composite Filtek Z250 (3M, St. Paul, USA) and four bulk-fill composites, Filtek One Bulk Fill (3M), Tetric PowerFill (Ivoclar Vivadent, Schaan, Liechtenstein), Tetric PowerFlow (Ivoclar Vivadent), SDR Plus (Dentsply, Konstanz, Germany). Composite specimens with a 9-mm diameter and a 2-mm height (n = 6) were polymerized with a high-intensity curing unit (Bluephase® PowerCure, Ivoclar Vivadent). The control group was polymerized for a total of 40 s on both sides (1193 mW / cm²) and 3s group for 3 s on one side (3053 mW/cm²). Water sorption and solubility were measured by ISO 4049 method up to 30 days of immersion. The results were statistically analyzed using one-way ANOVA with Tukey post-hoc correction. Different polymerization protocols for the same material were compared by t-test (p < 0.05). **Results:** The 3s polymerization protocol increased the solubility of all materials. Filtek One Bulk Fill showed the highest water sorption and solubility values, and Tetric PowerFlow proved to be a stable material with low values of tested parameters. During immersion, a saturation of specimens was achieved in low-viscosity bulk-fill composites within two weeks, while in other materials it was not achieved within 30 days, or more, following the immersion. **Conclusions:** Rapid polymerization caused an increase in solubility, which could affect the biocompatibility of the investigated materials not intended for the 3s curing. SDR Plus and Filtek One Bulk Fill should not be polymerized with rapid high-intensity curing due to increased solubility that exceeds ISO 4049:2009 limits.

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Introduction

To meet ever-increasing demands for a better management of restorations with conventional composites, it is essential for dentists to have simpler clinical procedures with reduced working time. For this purpose, bulk-fill composites have been introduced in the dental market as a new category of composite resins. Unlike a conventional composite, a bulk-fill composite allows for placement of a composite layer up to 4 to 5 mm thick in the cavity (1).

In addition to the placement of bulk-fill composites in thick layers, rapid polymerization with extremely high light intensity has recently been re-popularized to accelerate the clinical procedures of composite materials (2, 3). The concept of rapid polymerization is based on the principle of exposure reciprocity, which means the higher the light intensity, the less time is required for polymerization of the material,

Uvod

Da bi se zadovoljili sve veći zahtjevi za bolju izradu restauracija od konvencionalnih kompozita, stomatolozima su prijeko potrebni jednostavniji klinički postupci sa skraćenim vremenom rada. U tu svrhu na dentalnom su tržištu predstavljeni *bulk-fill* kompoziti kao nova kategorija smolastih kompozita. Za razliku od konvencionalnog kompozita, *bulk-fill* kompozit omogućuje postaviti u kavitet kompozit debljine od 4 do 5 mm (1).

Uz postavljanje *bulk-fill* kompozita u debelim slojevima, posljednjih se godina ponovno popularizira brza polimerizacija svjetlom iznimno visokoga intenziteta radi ubrzavanja kliničkoga rada s kompozitnim materijalima (2,3). Koncept brze polimerizacije temelji se na načelu *exposure reciprocity*, odnosno što je veći intenzitet svjetlosti, to je manje vremena potrebno za polimerizaciju materijala i obratno, zato što uku-

and vice versa, because the total delivered energy required for a satisfactory polymerization remains the same (4). This concept of rapid polymerization dominated in the 1980s when plasma lamps gained their popularity. Plasma lamps could emit extremely high-intensity light, but only for a short period of up to 5 seconds (5). Higher light intensity means that more photons were emitted which could, in turn, simultaneously activate a higher amount of photoinitiator molecules and ensure faster polymerization (6, 7). In the conventional composites, a myriad of initiation nuclei create a polymer network that rapidly reaches the gel point. At the post-gel stage, numerous monomers remain immobilized in the long-chain polymer network and the final polymerization of conventional composite remains significantly lower (8). The unpolymerized monomers with restricted mobility can form bonds within the same chain, so-called cyclization. Due to cyclization, the degree of conversion sometimes attains equally high values as with slower polymerization with lower radiation intensity. Still, the mechanical properties will be weaker and the polymerization stress higher (9). Because of this, many investigations have shown that the high-intensity polymerization concept is not sustainable for conventional composites (2, 3, 5, 8,-10).

Recently, this concept has regained the interest of scientists due to the fundamental change in the chemical composition of the organic matrix of specific bulk-fill composites. The composite Tetric PowerFill (Ivoclar Vivadent, Schaan, Liechtenstein) is at the forefront. It has incorporated ß-allyl sulfone reagent, allowing for addition-fragmentation chain transfer (AFCT) (2, 11). The aforementioned reagent allows for a step-growth polymerization process and a typical radical polymerization that forms a more homogeneous polymer network than the free radical reaction alone. After initiation and addition of the AFCT reagent, the AFCT reagent terminates the polymerization on one end of the polymer chain and simultaneously creates new sulphonyl radicals that further activate other monomers. The end result is a stable short kinetic chain network (2, 11). The fact that new initiation sites occur after the illumination has ended supports the rapid high-intensity curing. In that way, the material is customized to the concept of rapid polymerization.

Given that composite materials are used for direct composite restorations and constantly exposed to moist medium, it is essential to investigate the impact and consequences of water sorption on the properties of composite materials. Water causes hydrolysis of ester bonds in polymer networks in resin composites (12). In poorly polymerized resin composites, unreacted monomers could leach out of the material facilitated by water ingress and pose threats to the biocompatibility of the material (5, 7, 13). In oral environment, the salivary esterase can even promote enzymatic monomer degradation (14, 15), possibly causing genotoxic, cytotoxic or allergic reactions to the oral mucosa (16-20). Besides, absorbed water can lead to hydrolytic degradation of resin matrix-filler bond and the silane-filler bond which can deteriorate mechanical properties (21). The amount of absorbed water and solubility of composite is conditioned by the type and amount of composite matrix, amount of filler, and de-

pna predana energija potrebna za zadovoljavajuću polimerizaciju ostaje ista (4). Taj koncept polimerizacije dominirao je 1980-ih godina kada su bile popularne plazma-svjetiljke. One su mogle osloboditi svjetlost iznimno visoke energije, no samo kratko, tj. do 5 sekundi (5). Viši intenzitet svjetlosti znači da se emitiralo više fotona koji istodobno mogu aktivirati više molekula fotoinicijatora te sukladno tomu osigurati bržu polimerizaciju (6, 7). Kod konvencionalnih kompozita mnogobrojni nukleusi iz kojih se inicira polimerizacija stvaraju polimernu mrežu koja brzo prelazi gel-točku. U sljedećoj fazi (postgel faza) u dugolančanoj polimernoj mreži ostaju imobilizirani mnogobrojni nepolimerizirani monomeri pa je konačna polimerizacija konvencionalnih kompozita često značajno smanjena (8). Nepolimerizirani monomeri zbog limitirane pokretljivosti katkad stvaraju veze unutar istoga lanca, što se zove ciklizacija. Zbog toga stupanj polimerizacije može ostati jednako visok kao u slučaju sporije polimerizacije slabijim intenzitetom zračenja. Ipak, mehanička svojstva bit će oslabljena, a polimerizacijsko naprezanje više (9). Zbog toga se u mnogim istraživanjima ističe da koncept polimerizacije visokoga intenziteta nije održiv kada je riječ o konvencionalnim kompozitima (2, 3, 5, 8 – 10).

Zahvaljujući temeljitoj promjeni u kemijskom sastavu organske matrice određenih *bulk-fill* kompozita, nedavno je taj koncept ponovno privukao pozornost znanstvenika. Predvodnik je Tetric Power Fill (Ivoclar Vivadent, Schaan, Lihtenštajn). Taj materijal ima uklopljen reagens ß-alil sulfon koji omogućuje reverzibilni adicijsko-fragmentacijski prijenos lanaca (2, 11). Taj reagens omogućuje skokoviti način polimerizacije uz tradicionalnu radikalsku polimerizaciju i stvara homogeniju strukturu kompozita u usporedbi sa samo slobodnom radikalskom reakcijom polimerizacije. Nakon inicijacije i aktivacije AFCT reagensa, AFCT reagens terminira polimerizaciju na jednom kraju polimernoga lanca i istodobno stvara nove sulfonilne radikale koji dalje aktiviraju druge monomere. Krajnji rezultat je stabilna mreža kratkih kinetičkih lanaca (2, 11). Činjenica da se stvaraju nova mjesta za inicijaciju polimerizacije nakon završetka iluminacije podržava brzu polimerizaciju visokim intenzitetom. Na taj je način materijal prilagođen konceptu brze polimerizacije vrlo visokoga intenziteta.

S obzirom na to da se kompozitni materijali rade za izradu direktnih kompozitnih restauracija u kojima su neprestano izloženi vlažnome mediju, bitno je istražiti utjecaj i posljedice apsorpcije vode na njihova svojstva. Voda uzrokuje hidrolizu esterskih veza polimerne mreže smolastih kompozita (12). U slabo polimeriziranim kompozitima neizreagirani monomeri mogu iscuriti iz materijala zbog olakšanoga prodiranja vode i prijetnja su biokompatibilnosti materijala (5, 7, 13). U oralnome mediju salivarne esteraze mogu potaknuti enzimsku degradaciju monomera (14, 15), što može prouzročiti genotoksične, citotoksične ili alergijske reakcije oralne mukoze (16 – 20). Uz to apsorbirana voda može potaknuti hidrolitičku razgradnju veze smola – punilo i veze silan – punilo, što može pogoršati mehanička svojstva (21). Količina apsorbirane vode i topljivost kompozita uvjetovani su vrstom i količinom kompozitne smole, količinom punila i stupnjem konverzije monomera u polimer (22, 23). Apsorpcija vode

gree of conversion of monomer into the polymer (22, 23). Water sorption is a diffusion-controlled process mainly related to the organic part of the composite materials, and it is lower for the highly filled materials (24). Therefore, the water sorption characteristics of dental composite materials are vital for a better understanding and the appraisal of behavior of a material in human mouth.

Free volume theory and interaction theory are two theories used to explain water sorption. Free volume theory is explained by water sorption in free spaces of the polymer network. In contrast, interaction theory is defined by chemical bonds formed between water and the hydrophilic ionic groups of the polymer chain (25). Most of the restorative dental materials are sensitive to water sorption and solubility. Comparing water sorption with other restorative materials, composites exhibited the lowest amount of absorbed water (0.17%), followed by compomers (1.1%) with the resin-modified glass-ionomer cements in the first place with the highest values (6%) (26).

Apart from Tetric PowerFill, the manufacturer also claims that Tetric PowerFlow (Ivoclar Vivadent) could be polymerized by rapid polymerization. Rapid polymerization was not successful in the past due to a decrease in the crosslinking density of the polymer network. So far, the influence of this type of polymerization on water sorption and solubility has not been investigated on contemporary composites, hence this study aimed to investigate the influence of rapid polymerization during 3 s with a high-intensity curing unit on water sorption and solubility of bulk-fill composites and to compare it with standard polymerization.

Material and methods

Material

Five materials were tested (Table 1): two high-viscosity bulk-fill materials: Tetric PowerFill and Filtek One Bulk Fill Restorative (3M, St. Paul, USA); and two low-viscosity bulk-fill materials: Tetric PowerFlow SDR Plus Bulk Fill Flowable, and one conventional composite: Filtek Z250 (3M).

Dimensions of composite specimens (n = 6) for water sorption and solubility were 9 mm in diameter and 2 mm in height. Polymerization was carried out using the fourth-generation light-emitting diode curing unit (Bluephase Power-Cure, Ivoclar Vivadent) with one of two modes of polymerization (Figure 1):
1. during 3 seconds on one side with average values of radiant exitance 3053 (± 4.71) mW/cm^2 or
2. 20 seconds on both sides with average values of radiant exitance 1193 (± 4.71) mW/cm^2.

Light intensity was measured three times with a built-in radiometer of the curing unit (Bluephase PowerCure, Ivoclar Vivadent) before specimen production to calculate the average intensity value.

Water sorption and solubility

After polymerization and before placing specimens in a desiccator, the edges of specimens were gently ground on abrasive paper (Silicon carbide grinding paper, wet or dry,

difuzijski je kontrolirani proces koji je vezan za organski dio kompozitnih materijala pa je obično smanjena kod materijala koji su više punjeni (24). Zato su karakteristike apsorpcije vode kompozitnih materijala od vitalnog značenja za bolje razumijevanje i procjenu ponašanja materijala u ljudskim ustima.

Teorija slobodnoga volumena i teorija interakcije dvije su teorije kojima se objašnjava apsorpcija vode. Teorija slobodnoga volumena objašnjava se apsorpcijom vode u slobodne prostore polimerne mreže, a teorija interakcije stvaranjem kemijske veze između vode i hidrofilnih ionskih skupina polimernoga lanca (25). Većina restaurativnih dentalnih materijala podložna je apsorpciji vode i topljivosti. Uspoređujući apsorpciju vode kompozita s ostalim restaurativnim materijalima, kompoziti su pokazali najmanju količinu apsorbirane vode (0,17 %), slijede kompomeri (1,1 %), pa smolom modificirani staklenoionomerni cementi koji su pokazali najviše rezultate (6 %) (26).

Osim Tetric Power Filla, proizvođač također tvrdi da se Tetric Power Flow (Ivoclar Vivadent) može polimerizirati brzom polimerizacijom. Brza polimerizacija u prošlosti nije bila uspješna zbog smanjenja gustoće umrežavanja polimerne mreže. Utjecaj te vrste polimerizacije na apsorpciju vode i topljivost dosad nije istražen na suvremenim kompozitima, stoga je cilj ovoga istraživanja bio ispitati utjecaj brze polimerizacije tijekom 3 sekunde s polimerizacijskom svjetiljkom visokoga intenziteta na apsorpciju vode i topljivost *bulk-fill* kompozita i usporediti ih sa standardnom polimerizacijom.

Materijal i postupci

Materijali

Ispitalo se pet kompozitnih materijala (tablica 1.), od toga dva visokoviskozna *bulk-fill* materijala – Tetric Powerfill i Filtek One Bulk Fill Restorative (3M, St. Paul, SAD), dva niskoviskozna *bulk-fill* materijala – Tetric PowerFlow i SDR Plus Bulk Fill Flowable (Dentsply, Konstanz, Germany) te jedan konvencionalni kompozit – Filtek Z250 (3M).

Kompozitni uzorci za apsorpciju vode i topljivost (n = 6) bili su sljedećih dimenzija: promjer 9 mm i visina 2 mm. Polimerizacija je obavljena svjetiljkom s diodama četvrte generacije (Bluephase PowerCure, Ivoclar Vivadent) s jednim od dvaju načina polimerizacije (slika 1.):
1. tijekom 3 sekunde s jedne strane s vrijednostima prosječnoga emitiranoga zračenja od 3053 (± 4,71) mW/cm^{2}ili
2. 20 sekunda obostrano s vrijednostima prosječnoga emitiranoga zračenja od 1193 (± 4,71) mW/cm^2.

Mjerenje intenziteta svjetlosti provedeno je tri puta radiometrom ugrađenim u postolje svjetiljke (Bluephase Power-Cure, Ivoclar Vivadent) prije izrade uzoraka da bi se izračunala srednja vrijednost intenziteta.

Apsorpcija vode i topljivost

Poslije polimerizacije i prije postavljanja uzoraka u eksikator, njihovi su rubovi nježno ispolirani na abrazivnom papiru (Silicon carbide grinding paper, wet or dry, Grit500/

Table 1   The composition of the materials used in the research according to the manufacturer's instructions and the recommended layer thickness.
Tablica 1.   Sastav materijala korištenih u istraživanju prema uputama proizvođača i preporučena debljina sloja

Material • Materijal	Manufacturer • Naziv proizvođača [Ec rep]; {Lot (i) exp}	Organic matrix • Organska matrica	Inorganic filler • Anorgansko punilo	Filler particle size • Veličina čestica punila	Weight / volume content of fillers • Maseni / volumni udio punila (%)	Recommended layer thickness • Preporučena debljina sloja
3M™ Filtek™ Z250 Universal Restorative	3M ESPE Dental Products; St. Paul, MN, SAD {NC44979 27.7.2023.}	UDMA, Bis-GMA, Bis-EMA, TEGDMA	zirconia/silica	0,01-3,5 μm	-/ 60	from 2 to 2.5 mm • od 2 do 2,5 mm
Tetric® PowerFill IVA	Ivoclar Vivadent AG; Schaan, Lihtenštajn {X56571 14.9.2022.}	monomer matrix - dimethacrilates (w = 20 - 21 %)	barium glass, ytterbium trifluoride and copolymers (w = 79 - 80 %)	40 nm – 3 μm	76 - 77 / 53 - 54	up to 4 mm • do 4 mm
3M™ Filtek™ One Bulk Fill Restorative A2	3M ESPE Dental Products; St. Paul, MN, SAD {NA61219 28.5.2022.}	AUDMA, UDMA, 1,12-dodecaane-DMA	non-aggl./non-aggr. silica f.	20 nm	˷ 76,5 / ˷ 58,5	up to 5 mm • do 5 mm
			non-aggl./non-aggr. zirconia f.	4 - 11 nm		
			aggr. zirconia/silica cluster f.	silica: 20 nm zirconia: 4 - 11 nm		
			ytterbium trifluoride f. (aggl. particles)	100 nm		
SDR® Plus Bulk Fill Flowable U	Dentsply Caulk; Milford, DE, SAD [Dentsply DeTrey GmbH; Konstanz, Njemačka] {00028647 12.8.2022.}	resin matrix – modified UDMA, TEGDMA, dimethacrylate resin and trimethacrylate resin	silanated barium-alumino-fluoro-borosilicate glass, silanated strontium alumino-fluoro-silicate glass, surface treated fume silicas, ytterbium fluoride, synthetic inorganic iron oxide pigments and titanium dioxide	-	70,5 / 47,4	up to 4mm • do 4mm
Tetric® PowerFlow IVA	Ivoclar Vivadent AG; Schaan, Lihtenštajn {Y15023 09.4.2021./ Z00V4H 20.7.2022.}	monomer matrix - dymethacrilates (w = 28 %)	barium glass, ytterbium trifluoride and copolymers (w = 71 %)	0,1 μm – 30 μm	68,2 / 46,4	up to 4 mm • do 4 mm

non-aggl.: non-agglomerated, non-aggr.: non-aggregated, aggr.: aggregated, aggl.: agglomerated, f.: filler, DMA: dimethacrylate, UDMA: urethane-dimethacrylate • neagl. – neaglomerirano, neagr. – neagregirano, agr. – agregirano, agl. – aglomerirane, p. – punilo, DMA – dimetakrilat, UDMA – uretan-dimetakrilat, TEGDMA – trietilenglikol-dimetakrilat
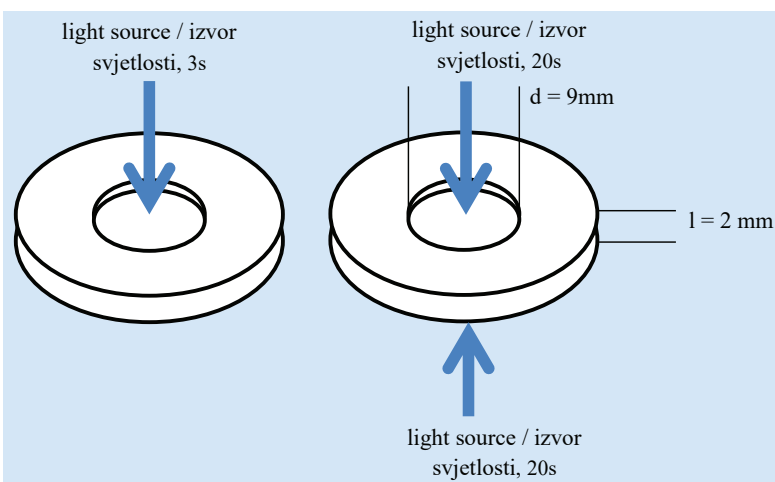
Figure 1   Comparison of 3s polymerization (left) and control groups of specimens (right)
Slika 1.   Usporedba trosekundne polimerizacije (lijevo) i kontrolne skupine uzoraka (desno)

Grit500/P1000, Buehler, Lake Bluff, Illinois, SAD) to remove overhangs and ensure that the same volumes and surfaces of all the specimens were maintained. Their mass was daily measured using an analytical balance (MS NewClassic, Mettler Toledo AG, Switzerland) until the daily difference in weight dropped below 0.1 mg, meaning that the equilibrium of the specimen mass had been established. The measured value indicated the initial mass of the specimen (m_1). Subsequently, each specimen was immersed in a conical bottom plastic container filled with 4mL of distilled water and stored in the dark in an incubator at $37 \pm 1°$ C. Specimens were weighed after 1, 2, 3, 7, 14, 21, 28 and 30 days of immersion ($m_2(t)$, where t stood for the interval from the beginning of immersion). During the immersion measurements, specimens were taken out from the water. According to the instructions of ISO standard 4049, each specimen was blotted with cellulose pads on both sides until it was free from visible moisture and waved in the air for 15 seconds. After immersion, specimens were again stored in a desiccator and weighed until they established a mass equilibrium (mass difference between the two subsequent measurements was below 0.1 mg) that denoted final mass (m_3). The total time of desorption was 48 days. Water sorption and solubility were calculated by the following formulas and presented in absolute values in grams:

$$\text{water sorption} = (m_2(eq) - m_3)/V \ (\mu g/mm^3)$$
$$\text{solubility} = (m_1 - m_3)/V \ (\mu g/mm^3),$$

where $m_2(eq)$ represents mass equilibrium.

Statistical analysis

Water sorption and solubility values were compared using a mixed-model ANOVA that included two between-subject factors (''material type`` and ''polymerization protocol``), as well as one within-subject factor (''time point``) that represented repeated observations. As statistically significant interactions of all three factors were identified, instead of interpreting the main effects overall, separate analyses were performed for each factor separately, at fixed levels of the remaining two factors (27,28). This led to using one-way ANOVA with Tukey's correction for the comparison among materials, t-test for independent observation for the comparison between two curing protocols, and repeated- measurements ANOVA with Bonferroni's corrections for the comparison among time points. Using the results of statistical comparisons among different time points obtained by repeated-measurements ANOVA with Bonferroni correction, the ''plateau`` of stable values was defined as the absence of statistically significant differences between at least two successive time points (29,30). For each test, p-values below 0.05 were considered statistically significant. The statistical analysis was performed using SPSS (version 25, IBM, Armonk, NY, USA).

Results

Filtek One Bulk Fill exhibited the highest water sorption in both groups of specimens, while Tetric PowerFlow exhibited the lowest water sorption in both groups compared to other materials. The polymerization method affected the water

P1000, Buehler, Lake Bluff, Illinois, SAD) da bi se uklonili prevjesi i osigurao isti volumen te površina svih uzoraka. Masa uzoraka svaki dan je mjerena na analitičkoj vagi (MS105 NewClassic, Mettler-Toledo AG, Švicarska) sve dok razlika između masa nije pala ispod 0,1 mg, tj. dok se nije uspostavila ravnoteža mase uzorka. Izmjerena vrijednost označavala je početnu masu uzorka (m_1). Zatim je svaki uzorak bio uronjen u plastičnu posudu s koničnim dnom i 4 mL destilirane vode te su zatim sve odložene u tamni inkubator temperature 37 ± 1 °C. Uzorci su bili izvagani poslije 1., 2., 3., 7., 14., 21., 28. i 30. dana imerzije ($m_2(t)$ – t označava interval od početka imerzije). Tijekom mjerenja imerzije mase uzorci su izvađeni iz vode. Prema uputama ISO standarda 4049, svaki uzorak je obrisan i s jedne i s druge strane na staničevini sve dok više nije bilo vidljive vlage, a poslije toga su bili sušeni 15 sekundi mahanjem u zraku. Nakon završetka imerzije uzorci su bili ponovno smješteni u eksikator te su bili vagani sve dok nisu postigli konstantnu masu (razlika mase između mjerenja manja od 0,1 mg) koja je označavala završnu masu (m_3). Ukupno vrijeme desorpcije bilo je 48 dana. Apsorpcija vode i topljivost bile su dobivene iz sljedećih formula:

$$\text{apsorpcija vode} = (m_2(eq) - m_3)/V \ (\mu g/mm^3)$$
$$\text{topljivost} = (m_1 - m_3)/V \ (\mu g/mm^3),$$

gdje je $m_2(eq)$ masa pri ekvilibriju.

Statistička analiza

Vrijednosti apsorpcije vode i topljivosti uspoređene su korištenjem mješovitoga modela ANOVA-e koji je uključivao dva čimbenika između subjekta („vrsta materijala“ i „protokol polimerizacije“) i jedan unutar subjekta („vremenska točka“) koji je predstavljao ponovljena opažanja. Kako su identificirane statistički značajne interakcije svih triju čimbenika, umjesto tumačenja glavnih učinaka u cjelini, provedene su zasebne analize za svaki čimbenik na fiksnim razinama preostalih dvaju čimbenika (27, 28). To je dovelo do korištenja jednosmjerne ANOVA-e uz Tukeyjevu korekciju za usporedbu između materijala, t-test za neovisno promatranje različitih protokola polimerizacije i ANOVA-e ponovljenih mjerenja s Bonferonijevom korekcijom za usporedbu između vremenskih točaka. Koristeći se rezultatima statističke usporedbe između različitih vremenskih točaka dobivenih ANOVA-om ponovljenih mjerenja s Bonferonijevom korekcijom, „plato“ stabilnih vrijednosti definiran je kao izostanak statistički značajnih razlika između najmanje dviju uzastopnih vremenskih točaka (29, 30). Za sve testove statistički značajnima smatrane su p-vrijednosti manje od 0,05. Statistička analiza provedena je u programu SPSS (verzija 25, IBM, Armonk, NY, SAD).

Rezultati

Filtek One Bulk Fill pokazao je najveću apsorpciju vode u obje skupine uzoraka, a Tetric Power Flow pokazao je najmanju apsorpciju u obje skupine u odnosu prema ostalim materijalima. Način polimerizacije utjecao je na apsorpciju vode

sorption of all materials except Tetric PowerFlow and SDR Plus. For Filtek One Bulk Fill, Filtek Z250 and Tetric Power-Fill, water sorption was higher in the control group of specimens when they were polymerized in the standard manner (Figure 2).

The solubility was higher for all specimens polymerized with the 3s protocol. SDR Plus specimens polymerized by standard protocol exhibited the highest solubility for this group of specimens, while Filtek One Bulk Fill and SDR Plus exhibited the highest solubility for the 3s group. Negative solubility values were established by the conventional control material Filtek Z250 for both polymerization groups and Tetric PowerFill for the control group of specimens polymerized by the standard protocol (Figure 3).

Figure 4 shows that specimens of Tetric PowerFlow and SDR Plus materials reached the plateau of mass change dur-

svih materijala, osim na Tetric Power Flow i SDR Plus. Kad je riječ o Filtek One Bulk Fillu, Filtek Z250 i Tetric Power Fillu, apsorpcija vode bila je veća u kontrolnoj skupini uzoraka polimeriziranih standardnim načinom (slika 2.).

Topljivost materijala veća je za sve skupine uzoraka koji su polimerizirani trosekundnim protokolom. SDR Plus uzorci polimerizirani standardnim protokolom pokazali su najveću topljivost za tu skupinu uzoraka, a Filtek One Bulk Fill i SDR Plus pokazali su najveću topljivost za trosekundnu skupinu. Negativne vrijednosti topljivosti pokazao je konvencionalni kontrolni materijal Filtek Z250 u obje polimerizacijske skupine te Tetric Power Fill za kontrolnu skupinu uzoraka polimeriziranu standardnim protokolom (slika 3.).

Na slici 4. su uzorci materijala Tetric Power Flow i SDR Plus koji su postigli „plato" promjene mase tijekom imerzije već nakon dva tjedna, dok drugi materijali to nisu postigli u


Figure 2 Comparison of water sorption between control group of polymerized specimens and 3s polymerized specimens. The same letters (20 s uppercase, 3 s lowercase) indicate statistically homogeneous groups within the illumination protocol. Statistically similar pairs between groups (20 s vs. 3 s) are indicated by square brackets. Error bars show ±1 standard deviation (due to low variability within the thickness of whiskers).

Slika 2. Usporedba apsorpcije vode između kontrolne skupine polimeriziranih uzoraka i 3 sekunde polimeriziranih uzoraka; ista slova (velika za 20 s, mala za 3 s) označavaju statistički homogene skupine unutar protokola osvjetljavanja; statistički slični parovi između skupina (20 s vs. 3 s) označeni su uglatim zagradama, stupci pogreške pokazuju ± 1 standardnu devijaciju (zbog male varijabilnosti, unutar debljine crte)
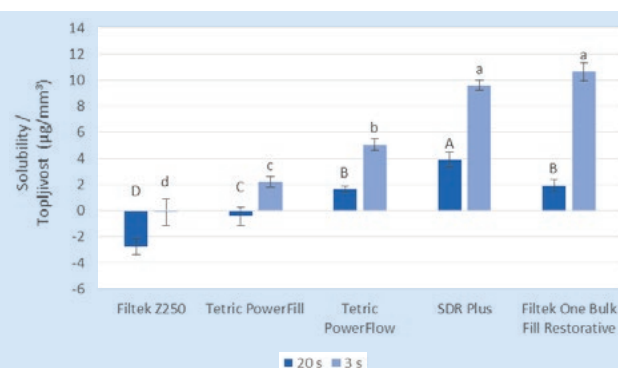
Figure 3 Comparison of solubility between control group of polymerized specimens and 3s polymerized specimens. The same letters (20 s uppercase, 3 s lowercase) indicate statistically homogeneous groups within the illumination protocol. There is a statistically significant difference when comparing the 3 s and 20 s polymerization protocols within the same material. Error bars show ±1 standard deviation (due to low variability within the thickness of whiskers).

Slika 3. Usporedba topljivosti između kontrolne skupine polimeriziranih uzoraka i 3 sekunde polimeriziranih uzoraka; ista slova (velika za 20 s, mala za 3 s) označavaju statistički homogene skupine unutar protokola osvjetljavanja; postoji statistički značajna razlika pri usporedbi trosekundnoga s i dvadesetsekundnoga polimerizacijskog protokola unutar istog materijala; stupci pogreške pokazuju ± 1 standardnu devijaciju (zbog male varijabilnosti, unutar debljine crte)
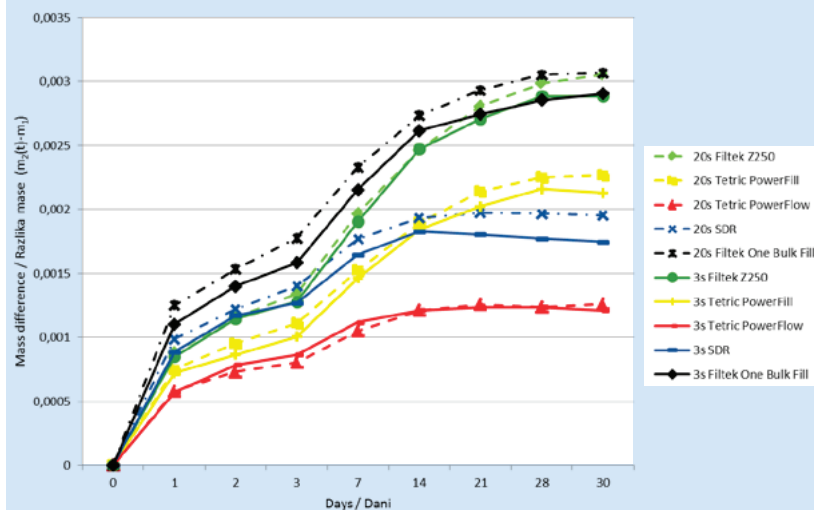
Figure 4 Difference in specimen mass change by days during specimen immersion. Dashed lines indicate specimens polymerized for 20 s, and solid lines indicate specimens polymerized for 3 s.

Slika 4. Razlika u promjeni mase uzorka po danima tijekom imerzije uzorka – isprekidane crte označavaju uzorke polimerizirane 20 s, a pune crte uzorke polimerizirane 3 s

ing their immersion after only two weeks, whereas other materials did not reach it within 30 days. The exception is *Tetric PowerFill* polymerized with 3s protocol after 28 days.

Discussion

This research examined the effect of 3s light polymerization with an intensity of approximately 3000 mW/cm² on water sorption and solubility of contemporary bulk-fill composite materials. The results showed an increased solubility for all materials polymerized with the 3s protocol. In contrast, standard polymerization of 20 s with a moderate light intensity of 1200 mW/cm² affected an increased water sorption in three of the five tested materials. The exceptions were low-viscosity materials with equal sorption values regardless of the polymerization protocol.

The amount of solubility of each composite material is determined by the amount of unreacted monomer, polymerization promoters, oligomers, filler particles, and products of degradation processes in the composite material (24, 31, 32). In this study, the rapid polymerization protocol markedly increased the solubility. The very high light intensity of 3053 mW/cm² emitted in the 3s program of the Bluephase Power-Cure used in this study released two and a half times more energy per second than the standard protocol with 1193 mW/cm². Such polymerization is much faster with the 3s program than with the standard program because a large number of free radicals that trigger a radical polymerization reaction are generated at the same time. In Tetric PowerFill, the AFCT reagent will condition the formation of short-chain polymers without much cross-linking, resulting in a more complete and more homogeneous polymer network. Therefore, its solubility values are one of the lowest in this study. For other materials, due to the increased rigidity of the polymer network, a large amount of unpolymerized monomers will remain, which dissolve and leach out of the specimen surface during prolonged exposure to water, causing increased solubility. Some studies have reported that the size of the unreacted monomer plays a role in the amount of solubility, e.g., triethylene glycol dimethacrylate (TEGDMA) is reported to have a higher and faster tendency to release from the material due to lower molecular weight (13,33). The solubility values for 3s polymerization of SDR Plus and Filtek One Bulk Fill were the highest, exceeding 7.5 μg/mm³, which is not within the recommended values by ISO 4049:2009. The high solubility values for Filtek One Bulk Fill in 3s polymerization can be explained by its reduced degree of polymerization, which was determined in a previous study by Marović et al. (2). Apart from unpolymerized monomers, it is possible that filler nanoparticles were also eluted from the surface of Filtek One Bulk Fill, thus contributing to its solubility.

Water sorption and dissolution of their individual components occur simultaneously during exposure of composite specimens to water. It is usually difficult to distinguish the effect of solubility while the specimens are still in the aqueous medium because water sorption is dominant, leading to an overall increase in mass. The exact extent of solubility could be determined only after the second drying of the ISO

roku od 30 dana. Iznimka je Tetric Power Fill polimeriziran trosekundnim protokolom nakon 28 dana.

Rasprava

Ovo istraživanje proučavalo je utjecaj trosekundne polimerizacije svjetlom intenziteta oko 3000 mW/cm² na apsorpciju vode i topljivost suvremenih *bulk-fill* kompozitnih materijala. Rezultati su pokazali da je topljivost bila povišena za sve materijale polimerizirane trosekundnim protokolom. Suprotno tomu, standardna polimerizacija od 20 sekunda svjetlom umjerenog intenziteta od 1200 mW/cm² utjecala je na povišenu apsorpciju vode kod triju od pet ispitanih materijala. Iznimke su bili niskoviskozni materijali koji su imali jednake vrijednosti apsorpcije bez obzira na polimerizacijski protokol.

Količina topljivosti pojedinoga kompozitnoga materijala uvjetovana je količinom nereagiranoga monomera, promotorima polimerizacije, oligomerima, česticama punila te produktima degradacijskih procesa u kompozitnom materijalu (24, 31, 32). Rezultati topljivosti u ovome istraživanju pokazali su iznimno povećanu topljivost brzim polimerizacijskim protokolom. Emitiranjem svjetlosti vrlo visokoga intenziteta od 3053 mW/cm², poput trosekundnoga programa Bluephase Power Cure svjetiljke rabljene u ovom istraživanju, oslobodilo se nešto više od dva i pol puta energije u sekundi u usporedbi sa standardnim protokolom od 1193 mW/cm². Takva polimerizacija mnogo je brža u trosekundnome programu nego u standardnome zato što se istodobno stvara velika količina slobodnih radikala koji pokreću radikalnu reakciju polimerizacije. U slučaju Tetric Power Filla, AFCT reagens uvjetovat će stvaranje više kratkolančanih polimera bez velikoga ukriženoga polimeriziranja te potpuniju i homogeniju polimernu mrežu. Zbog toga su njegove vrijednosti topljivosti jedne od najnižih u ovom istraživanju. Kod ostalih materijala, zbog povećane rigidnosti mreže, zaostat će veća količina nepolimeriziranih monomera koji se pri dugotrajnom izlaganju vodi otapaju i izlaze s površine uzorka uzrokujući povećanu topljivost. Autori određenih istraživanja navode da veličina nereagiranoga monomera igra ulogu u količini topljivosti, npr., za trietilenglikol-dimetakrilat (TEGDMA) navodi se da ima veću i bržu tendenciju otpuštanja iz materijala zbog manje molekularne mase (13, 33). Vrijednosti topljivosti za trosekundnu polimerizaciju materijala SDR Plus i Filtek One Bulk Fill bile su najviše – više od 7,5 μg/mm³ što nije unutar preporučenih vrijednosti prema standardu ISO 4049:2009. Visoke vrijednosti topljivosti za Filtek One Bulk Fill pri trosekundnoj polimerizaciji možemo razjasniti i njegovim smanjenim stupnjem polimerizacije koji su utvrdili Marović i suradnici (2). Osim nepolimeriziranih monomera, moguće je da su nanočestice punila također bile eluirane s površine Filtek One Bulk Filla pridonoseći tako njegovoj topljivosti.

Apsorpcija vode i otapanje pojedinih komponenata događaju se istodobno tijekom izlaganja kompozitnih uzoraka vodi. Obično je teško razaznati utjecaj topljivosti dok su uzorci u vodenome mediju zato što je apsorpcija vode dominantna, dovodeći do ukupnoga povećanja mase. Točan stupanj toplji-

4049 testing protocol. Our research unquestionably showed increased solubility after 3s polymerization for all materials, hence it is reasonable to suspect that this is the reason for the seemingly reduced water sorption in the 3s group. This led to the increased water sorption in the control group of specimens in high-viscosity materials in the statistical analysis. According to accepted theories of water diffusivity in composite materials (24), the amount of water sorption is inversely proportional to the degree of conversion because greater binding of monomers to polymer reduces the free space for water diffusion between polymer networks (31). In addition to the maximum achievable degree of conversion of individual material, it is important to study the post-cure polymerization kinetics after light illumination. Par et al. demonstrated that the long-term post-cure polymerization of the SDR continues up to 7 days, which leaves a more extended period for the release of unreacted compounds and thus higher solubility values (34). This could explain the higher solubility values of its successor SDR Plus than those of other materials tested in this study.

Filtek One Bulk Fill and Fitek Z250, as two of the most filled materials in our research, exhibited the highest water sorption for both polymerization methods. Although the high content of fillers slows down the diffusion of water, the type of fillers and the percentage of their silanization affect the final water sorption values, which have been considered in previous studies (35, 36). Nano-fillers that tend to agglomerate and silane on their large surface easily hydrolyze when exposed to water. This creates free spaces that facilitate the diffusion of water in such a polymerized composite. Our previous long-term study of water sorption of specimens polymerized by standard protocol over 90 days confirms the current results because the same sorption patterns were observed for Filtek One Bulk Fill (37, 38). In addition, Filtek One Bulk Fill also contains a long chain aromatic urethane dimethacrylate (AUDMA) with a small number of reactive groups added to reduce polymerization shrinkage. However, their polymerization leads to a lower degree of cross-linking of the polymer network. The lower density of the polymer opens up free spaces within the network, thus leaving room for water sorption according to free volume theory (14). Filtek Z250 does not contain nano-fillers, but includes a hydrophilic bisphenol A-glycidyl methacrylate (Bis-GMA) resin which, by interaction of its numerous hydroxyl groups with water, forms hydrogen bonds and enhances water sorption (35). Another reason for the increased sorption of Filtek Z250 could be TEGDMA resin residues. Although the manufacturer has replaced most of the hydrophilic resin TEGDMA with more hydrophobic resins (Table 1), its traces could still contribute to increased sorption, which was also observed in the study of Baor et al. (39).

The lowest solubility was demonstrated by Filtek Z250 and Tetric PowerFill (40). Filtek Z250 has negative solubility values, which can be explained by strong and numerous hydrogen bonds within Bis-GMA molecules, which is why water cannot be completely removed during desorption. However, the low solubility values of Tetric PowerFill compared to other bulk-fill composites in 3s polymerization can also

vosti mogao se odrediti tek nakon drugog sušenja protokola ispitivanja ISO 4049. Naše istraživanje neupitno je pokazalo pojačanu topljivost pri trosekundnoj polimerizaciji i to za sve materijale, pa je opravdano posumnjati da je to razlog za naizgled sniženu apsorpciju vode u trosekundnoj skupini. To je u statističkoj analizi rezultiralo povišenom apsorpcijom vode u kontrolnoj skupini uzoraka kod visokoviskoznih materijala. Prema dosad prihvaćenim teorijama difuzivnosti vode kod kompozitnih materijala (24), količina apsorbirane vode obrnuto je proporcionalna stupnju konverzije jer se većim povezivanjem monomera u polimer smanjuje slobodni prostor za difuziju vode između polimerne mreže (31). Dodatno maksimalno ostvarivom stupnju konverzije pojedinog materijala bitno je proučiti i kinetiku polimerizacije koja se dugoročno naknadno događa nakon svjetlosne polimerizacije materijala. Par i suradnici uočili su dugoročno naknadnu polimerizaciju SDR materijala koja maksimalni stupanj konverzije postiže do 7 dana, što ostavlja veće razdoblje za otpuštanje nereagiranih reaktivnih spojeva kompozitnoga materijala te time veće vrijednosti topljivosti (34). To bi moglo biti objašnjenje za veće vrijednosti topljivosti njegova nasljednika – SDR Plusa od drugih ispitanih materijala u ovom istraživanju.

Filtek One Bulk Fill i Filtek Z250, kao dva najviše punjena materijala u našem istraživanju, pokazala su najveću apsorpciju vode tijekom oba načina polimerizacije. Iako visok udio punila usporava difuziju vode, vrsta punila i postotak njihove silanizacije utječe na konačne vrijednosti apsorpcije vode, što je razmatrano u prijašnjim istraživanjima (35, 36). Nanopunila imaju tendenciju prema aglomeraciji i silan na njihovoj velikoj površini lako hidrolizira pri izlaganju vodi. To stvara slobodne prostore pa je olakšana difuzija vode u tako polimeriziranom kompozitu. Naše prethodno dugoročno istraživanje apsorpcije vode uzoraka polimeriziranih standardnim protokolom tijekom 90 dana potvrđuje sadašnje rezultate jer su uočeni isti obrasci apsorpcije na Filtek One Bulk Fillu (37, 38). Uz to, Filtek One Bulk Fill također sadržava dugolančani aromatični uretan-dimetakrilat (AUDMA) s malim brojem reaktivnih skupina koji je dodan radi smanjenja polimerizacijskog skupljanja. No njihova polimerizacija potiče manje ukrižno povezivanje polimerne mreže. Manja gustoća polimera otvara slobodne prostore unutar mreže i ostavlja prostor za apsorpciju vode prema teoriji slobodnoga volumena (14). Filtek Z250 ne sadržava nanopunila, ali sadržava hidrofilnu bisfenol A-glicidil metakrilatnu (Bis-GMA) smolu koja interakcijom svojih mnogobrojnih hidroksilnih skupina s vodom stvara vodikove veze i pojačava apsorpciju vode (35). Drugi razlog za povećanu apsorpciju Filteka Z250 mogli bi biti ostatci smole TEGDMA. Iako je proizvođač većinu hidrofilne smole TEGDMA zamijenio hidrofobnijim smolama (tablica 1.), ipak bi njezini ostatci mogli pridonijeti povećanoj apsorpciji, što se razmatralo i u istraživanju Baora i suradnika (39).

Najmanju topljivost pokazali su Filtek Z250 i Tetric PowerFill (40). Filtek Z250 ima negativne vrijednosti topljivosti, što se može objasniti čvrstim vodikovim vezama s Bis-GMA-om, zbog čega se voda ne može potpuno ukloniti pri desorpciji. No niske vrijednosti topljivosti Tetric Power Filla u usporedbi s ostalim *bulk-fill* kompozitima pri trosekundnoj

be explained by step-growth polymerization using ß-allyl sulfone, the so-called AFCT, which leads to more stable polymerization in which there are no remaining unreacted monomers (2,11).

The observed water sorption curves (Figure 4) are increased at the beginning of the immersion, after which the highest level of absorbed water is reached, i.e., the highest measured mass followed by the plateau. Both low-viscosity materials in this study achieved water saturation after 7 and 14 days for both polymerization methods, while SDR Plus showed a slight decrease, which means that the process of releasing soluble components began to prevail (41, 29). Different proportions of inorganic and organic parts between high-viscosity and low-viscosity composites could play an important role in the obtained results. A higher proportion of the organic matrix creates a higher diffusion gradient of water to the specimen center, leading to faster water saturation of the material, especially if that organic matrix contains a hydrophilic resin such as TEGDMA found in SDR Plus.

The step-growth polymerization of Tetric PowerFill could explain the mass change curves during immersion (Figure 4). It is observed that Tetric PowerFill polymerized with 3s program reached a plateau after 28 days, while other tested high-viscosity materials did not achieve the plateau even after 30 days. Although Tetric PowerFill does not have a higher proportion of organic matrix than low-viscosity materials, it does have AFCT polymerization. Therefore, cross-linking of the polymer network plays a more important role in water sorption kinetics than in the final values, i.e. water sorption by volume (42).

This study was based on ISO 4049:2009 standard, hence 2 mm thick specimens were used, which enabled us to compare our results to the recommended values. For the extension of this study it would be interesting to evaluate properties of bulk-fill composite materials on 4 mm specimens, which would be more similar to clinical conditions. However, owing to this experimental design, we were able to determine that all materials in both polymerization protocols had less than 40 μg/mm^3 water sorption and satisfied the ISO recommendation. On the other hand, rapid curing of SDR Plus and Filtek One Bulk Fill exceeded the maximum acceptable value for solubility (7.5 μg/mm^3).

Conclusions

The solubility of all of the test samples significantly increased during rapid polymerization with very high light intensity, which could be associated with the elusion of unreacted monomers and possible adverse reactions of gingival and mucosal cells. The solubility values of SDR Plus and Filtek One Bulk Fill exceeded the recommended values by ISO 4049:2009 standard, hence rapid polymerization is not recommended for those materials. The effect of rapid polymerization on water sorption was material-dependent, with the low-viscosity bulk-fill materials being insensitive to the polymerization protocol.

polimerizaciji, mogu se objasniti i skokovitom polimerizacijom s pomoću ß-alil sulfona, tzv. AFCT-a čime se postiže stabilnija polimerizacija u kojoj nema ostatnih nereagiranih monomera (2, 11).

Promatrane krivulje apsorpcije vode (slika 4.) sadržavaju povećanje na početku imerzije nakon kojega se postiže najviša razina apsorbirane vode, odnosno najviša izmjerena masa te nakon toga „plato". Oba niskoviskozna materijala u ovom istraživanju postigla su zasićenje vodom već nakon 7 dana, odnosno 14 dana za oba načina polimerizacije, a kod SDR Plusa uočen je i blagi pad, što znači da je počeo prevladavati proces otpuštanja topljivih komponenti (41, 29). Različit udio anorganskoga i organskoga dijela između visokoviskoznih i niskoviskoznih kompozita mogao bi biti važan u dobivenim rezultatima. Veći udio organske matrice stvara veći difuzijski gradijent vodi u unutrašnjost uzorka, što rezultira bržim zasićenjem materijala vodom, pogotovo ako ta organska matrica sadržava hidrofilnu smolu poput TEGDMA-e koja se nalazi u SDR Plusu.

Skokovita polimerizacija Tetric Power Filla mogla bi objasniti krivulje promjena mase tijekom imerzije (slika 4.). Uočava se da je Tetric Power Fill polimeriziran trosekundnim programom postigao „plato" nakon 28 dana, a drugi ispitani visokoviskozni materijali to nisu postigli ni nakon 30 dana. Iako Tetric Power Fill nema veći udio organske matrice poput niskoviskoznih materijala, ipak ima AFCT polimerizaciju. Zato bi umreženje polimerne mreže bilo važnije za kinetiku apsorpcije vode, nego za konačne vrijednosti apsorpcije vode, odnosno apsorpciju vode po volumenu (42).

Ovo istraživanje temeljilo se na standardu ISO 4049:2009, pa su korišteni uzorci debljine 2 mm kako bismo mogli usporediti naše rezultate s preporučenim vrijednostima. Za proširenje ovog istraživanja bilo bi zanimljivo ispitati navedena svojstva *bulk-fill* kompozitnih materijala na uzorcima debljine 4 mm koji bi bili sličniji kliničkim uvjetima. No zahvaljujući ovom eksperimentalnom dizajnu uspjeli smo utvrditi da svi materijali u oba polimerizacijska protokola imaju apsorpciju vode manju od 40 μg/mm^3 i zadovoljavaju preporuke ISO-a. S druge strane, brza polimerizacija SDR Plusa i Filtek One Bulk Filla premašuje maksimalno dopuštene vrijednosti topljivost od 7,5 μg/mm^3.

Zaključci

Topljivost svih ispitanih materijala značajno se povećala tijekom brze polimerizacije s vrlo visokim intenzitetom svjetlosti, što bi moglo biti povezano s eluzijom nereagiranih monomera i mogućim štetnim reakcijama stanica gingive i sluznice. Vrijednosti topljivosti SDR Plusa i Filtek One Bulk Filla premašile su preporučene vrijednosti prema standardu ISO 4049:2009 i zato se za te materijale ne preporučuje brza polimerizacija. Učinak brze polimerizacije na apsorpciju vode ovisio je o materijalu, pri čemu su niskoviskozni *bulk-fill* materijali bili neosjetljivi na protokol polimerizacije.

Conflict of interest

None declared

Author's contribution: D.M., M.P. and **Z.T** – conceived the study and were in charge of overall direction and planning; **N.K.** and **M.M.** – performed the experiment and analyzed the dana; **D.M., Z.T.** and **M.P.** – supervised the work; **N.K., D.M.** – wrote the manuscript and constructive feedback and editing from **M.P.** and **Z.T.** All authors discussed, reviewed, and approved the contents within the manuscript.

Sukob interesa

Autori nisu bili u sukobu interesa.

Doprinos autora: D.M., M.P. i Z.T. – osmislili su studiju i bili zaduženi za cjelokupno usmjeravanje i planiranje; **N.K. i M.M.** – izradili su eksperiment i analizirali podatke; **D.M., Z.T. i M.P.** – nadzirali su rad; **N.K. i DM.** – napisale su rukopis; **M.P., Z.T.** – konstruktivne povratne informacije i uređivanje; Svi su autori raspravljali, pregledali i odobrili sadržaj unutar rukopisa.

Sažetak

Svrha: Svrha ovoga istraživanja bila je ispitati utjecaj brze polimerizacije visokoga intenziteta na apsorpciju vode i topljivost nove generacije *bulk-fill* kompozitnih materijala. **Materijal i postupci:** Ispitano je pet materijala: konvencionalni kompozit Filtek Z250 (3M, St. Paul, SAD) i četiri *bulk-fill* kompozita – Filtek One Bulk Fill (3M), Tetric PowerFill (Ivoclar Vivadent, Schaan, Lihtenštajn), Tetric PowerFlow (Ivoclar Vivadent) i SDR Plus (Dentsply, Konstanz, Njemačka). Kompozitni uzorci promjera 9 mm i visine 2 mm (n = 6) polimerizirani su svjetlom visokoga intenziteta (Bluephase® PowerCure, Ivoclar Vivadent). Kontrolna skupina uzoraka polimerizirana je obostrano tijekom ukupno 40 sekunda (1193 mW/cm²), a trosekundna jednostrano 3 sekunde (3053 mW/cm²). Apsorpcija vode i topljivost mjereni su metodom ISO 4049 do 30. dana imerzije. Rezultati su statistički obrađeni s pomoću jednosmjerne ANOVA-e uz Tukeyevu post-hoc korekciju. Različiti protokoli polimerizacije za isti materijal usporeeni su t-testom (p < 0,05). **Rezultati:** Trosekundni protokol polimerizacije povisio je topljivost svih materijala. Filtek One Bulk Fill pokazao je značajno najviše vrijednosti u apsorpciji vode i topljivosti, a Tetric PowerFlow pokazao se stabilnim materijalom s niskim vrijednostima ispitanih parametara. Tijekom imerzije je saturacija uzoraka niskoviskoznih *bulk-fill* kompozita postignuta nakon dva tjedna, a kod ostalih materijala nije postignuta unutar 30 ili više dana imerzije uzoraka. **Zaključak:** Brza polimerizacija prouzročila je povećanje topljivosti što bi moglo utjecati na biokompatibilnost ispitanih materijala koji nisu namijenjeni trosekundnoj polimerizaciji. SDR Plus i Filtek One Bulk Fill ne bi se smjeli polimerizirati brzom polimerizacijom visokoga intenziteta zbog povećane topljivosti koja premašuje ograničenja standarda ISO 4049:2009.

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