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Otklon kvržica i postojanost adhezivne veze kod restauracija s nisko-kontrahirajućim kompozitom u distalnom području

Cuspal Deflection and Adhesive Interface Integrity of Low Shrinking Posterior Composite Restorations

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Sažetak
Kad je riječ o restaurativnim zahvatima u distalnom području trenuačno su u svim istraživanjima u središtu pozornosti nisko-kontrahirajući kompoziti. U vezi s tim obećavaju materijali dobiveni siloranskim kemijskim postupkom s prstenastim monomerima i kondenzabilni dimetakrilati s visokim postotkom punila. **Materijali i metode:** Na ekstrahiranim gornjim premolarima (n=10) promatran je otklon kvržica nakon svjetlosne polimerizacije transduserom s razlikovanjem direktne struje (DCDT-om) uz čuvanje adhezivne veze elektronskim mikroskopom (SEM-om) i dvama restorativnim sustavima. To su Filtek™ Silorane/Silorane System Adhesive (3M ESPE) i Premise™ Packable /OptiBond FL (KERR). Podaci su analizirani dvostranim *t-testom*. **Rezultati:** Vrijednost $p < 0,05$ postavljena je kao statistički značajna granica. Filtek™ Silorane može smanjiti otklon kvržica uzrokovan polimerizacijskom kontrakcijom, ali sumnja se može li se održati adhezivna veza. **Zaključak:** Premise™ Packable pokazao je veći otklon kvržica tijekom polimerizacije, ali i bolju adhezivnu vezu.

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Uvod

Većina smolastih kompozita (RBCs-a) koji se upotrebljavaju u reataurativnoj dentalnoj medicini imaju zajedničku osnovu – polimeriziraju slobodne radikale metakrilata. Taj postupak mijenja volumen materijala, što omogućuje napetost na spoju restauracija-zub poznatu pod nazivom "polimerizacijski stres zbog kontrakcije" (1). Takav stres nastaje u kompozitnoj masi te se prenosi na adhezivnu vezu (2) i zubnu površinu, što stvara otklon kvržica (3) te pukotine u okolnoj caklini i dentinu (4). Sve to pacijent doživljava kao postoperativnu preosjetljivost (5). Polimerizacijski stres može kompromitirati vezu zuba i restauracije te završiti bakterijskim mikrocurenjem (6) i na kraju rubnim obojenjem, sekundarnim karijesom, upalom zubne pulpe i nekrozom (7).

Introduction

The majority of resin–based composites (RBCs) used in restorative dentistry have their common basis in the free-radical polymerisation of methacrylates. Polymerisation process of RBCs exhibits a volumetric contraction of the material, which generates strain in the complex tooth-restoration known as polymerisation shrinkage stress (1). Polymerisation shrinkage stress developed in the resin composite restoration passes through the adhesive interface (2) to the dental structure, generating cuspal deflection (3) and cracks in the surrounding dentin and enamel (4) experienced by the patient as post-operative sensitivity (5). Polymerisation shrinkage stress may compromise the synergism of the bond at the tooth-restoration interface leading to bacterial microleakage

Polimerizacijske kontrakcije smanjuju se uglavnom na dva načina:

1. smanjivanjem reakcijske površine po jedinici volumena;
2. smanjivanjem kontrakcije korištenjem različitih vrsta smole.

Gustoća reaktivnog područja po volumnoj jedinici može se smanjiti na dva načina – ako se poveća molekularna težina prema reaktivnoj skupini i ako se poveća udjel punila. Povećanje udjela punila ima određena oganičenja. Naime, sustavi s vrlo visokim udjelom punila, poput kondenzirajućih kompozita ili materijala s optimiziranim udjelom punila i do 82 posto uz dodatak nanočestica, imaju vrijednosti kontrakcije od 1,7 posto volumena. Istaknimo da se udjel punila više ne može povećati jer preostala količina smole ne može osigurati kemijsko-fizikalnu vezu i inkorporaciju čestica punila te vlažiti njihovu veću količinu (8).

Kako bi se prebrodili problemi s polimerizacijskom kontrakcijom, stručnjaci odabiru različite tehnike, uključujući i sredstva za vezivanje s dentinom (9), nisko-kontrahirajuće kompozitne materijale (10), premaze (*eng.lineri*) od staklenih ionomera (11) i različite načine postavljanja (12). No, ni jedna od tih tehnika ne može potpuno ukloniti naprezanje. Naravno, najsigurniji način da se ono izbjegne jest korištenje smola bez kontrakcije. Odnedavno se na tržištu mogu nabaviti nisko-kontrahirajući kompoziti pod komercijalnim nazivom Filtek™ Silorane (3M-ESPE, Seefeld, Njemačka). Ime "silorane" kombinacija je gradivnih kemijskih elemenata i oksirana sadržanih u preparatu. Mreža silorana stvara se polimerizacijom kationskih otvaranja prstenova cikloalifatskih oksiranskih monomera koji pritom omogućuju nisku kontrakciju i napetost. Najvažnija je razlika u tome da se metakrilati polimeriziraju preko međuspojeva radikala, a oksirani preko kationskih međuspojeva (13). Kemijski postupak otvaranja prstenova smole volumno smanjuje kontrakciju kompozita manje od jedan posto (8).

Budući da se kompoziti tijekom polimerizacije kontrahiraju, naprezanja na adhezivnom spoju kompozit-zub i u zubu ovisna su o obliku kaviteta, veličini, C-faktoru, modulu elastičnosti zuba i kompozita te o brzini i stupnju polimerizacije (14). Ti su čimbenici međuovisni i složeno djeluju prenoseći polimerizacijsku kontrakciju u naprezanje zuba. Uklanjanje zuba klinički je važan ishod i razuman pokazatelj drugog utjecaja polimerizacijske kontrakcije, kao što je, primjerice, naprezanje spojnog područja (15). U dosadašnjim istraživanjima, u usporedbi s metakrilatnim kompozitima, otkriveno je bolje rubno prianjanje i smanjena količina mikrocurenja te manji otklon kvržica kada su se stručnjaci koristili materijalima na siloranskoj osnovi(16-18).

U ovom istraživanju željelo se testirati jesu li razlike u polimerizacijskoj reakciji RBC-a na osnovi silorana povoljnije u odnosu prema kondenzabilnim RBC-ima na osnovi metakrilata i rezultiraju li manjim otklonom kvržica tijekom polimerizacije te zatvaraju li bolje rubno područje na granici zub/restauracija nakon što su šest mjeseci bili uronjeni u vodu.

(6) and ultimately to marginal discoloration, secondary caries, pulpal inflammation or necrosis (7).

There are two main strategies to reduce polymerisation shrinkage:

1. reduction of reactive sites per volume unit;
2. reduction of shrinkage using different types of resin.

The density of reactive sites per volume unit can be reduced principally in two ways: a. by increasing the molecular weight per reactive group; b. by increasing the filler load. The increased filler load also finds its limitation at a certain level. Very highly filled systems like packable posterior composites or materials with optimized filler load of up to 82% by addition of nano particles reveal shrinkage values down to 1.7% vol. However, also the filler load cannot be further increased when the consequently reduced amount of resin cannot any longer provide for the chemophysical incorporation of the filler particles, and for the wetting of the increased filler (8).

To overcome problems associated to polymerisation shrinkage, investigators have advocated several techniques, including the use of dentinal bonding agents (9), low shrinkage resin composite (10), glass-ionomer cement liners (11), and different application techniques (12). None of these techniques can eliminate the stress completely. Naturally, the surest way to avoid shrinkage stress is to use non-shrinking resins. Recently, a low-shrinking composite, commercialized as Filtek™ Silorane (3M-ESPE, Seefeld, Germany), was introduced. The name Silorane derives from the combination of its chemical building blocks siloxanes and oxiranes. The network of Siloranes is generated by the cationic ring opening polymerisation of the cycloaliphatic oxirane moieties, which stand for their low shrinkage and low polymerisation stress. The most important difference is that methacrylates are cured by radical intermediates and oxiranes polymerize via cationic intermediates (13).The ring-opening chemistry of the resin reduces shrinkage of the composite below 1 vol% (8).

As the composite polymerizes and shrinks, the stresses that develop at the tooth-composite adhesive interface and within the tooth itself depend upon the cavity shape, size, C-factor, modulus of the tooth, the developing modulus of the composite, and the rate of polymerisation (14). These factors combine and interact simultaneously in complex ways, translating polymerisation shrinkage into tooth stresses. The deflection of the tooth is a clinically significant outcome, as well as being a reasonable indicator of other effects of polymerisation shrinkage such as interface stress (15). Previous studies revealed higher marginal adaptation and reduced microleakage formation and lower cuspal deflection when silorane-based materials were used compared to methacrylate composites (16-18).

In the current study, the tested hypothesis was that differences in polymerisation reaction of the silorane-based RBC would result in decreased cuspal deflection during polymerisation and increased cavity adaptation at the tooth/restoration adhesive interface after 6 months water immersion compared to a packable methacrylate RBC.

Materijali i metode

Za ovo istraživanje odabrana su dva dobro poznata nisko-kontrahirajuća kompozita za distalna područja (LSPCs) - Filtek™ Silorane (3M ESPE) kao jedini komercijalni kompozit na osnovi silorana te Premise™ Packable (KERR) kao kondenzirajući dimetakrilatni kompozit. Opsežan opis tih dvaju procijenjenih materijala je u Tablici 1.

Materials and Methods

For the current study two well known low-shrinking posterior composites (LSPCs) were selected, Filtek™ Silorane (3M ESPE) as the only commercial available silorane-based composite, and Premise™ Packable (KERR), as a packable dimethacrylate-based composite. A detailed description of the two LSPCs assesed is given in Table 1.

Tablica 1. Kompoziti korišteni u istraživanju
Table 1 The resin composites used in the current study

Proizvod • Product	Proizvođač • Manufacturer	Tip • Type	Organska matrica • Resin Matrix	Punilo • Filler
Filtek™ Silorane Low Shrink Posterior Restorative (Lot#4712 Shade A3)	3M ESPE	Mikro-hibrid • Micro-hybrid	3,4-Epoxycyclohexylethylcyclopolymethylsiloxane, bis-3,4-Epoxycyclohexylethylphenylmethylsilane	Silanizirani quartz • Silanized quartz Yttriumfluoride • Yttriumfluoride 0,1 -2,0 µm 76wt%
Premise™ Packable Tri-Modal Composite Restorative (Lot#3098526 Shade A3)	KERR	Nano-hibrid • Nano-hybrid	Bisphenol A diglycidylmethacrylate (BisGMA) Triethyleneglycoldimethacrylate (TEGDMA) Ethoxylated-BisGMA	Barium Glass 0,4µm Prepolimerizirane čestice • PrePolymerised Particles 30-50µm Koloidne Si nanočestice • Colloidal Si nanoparticles 0,02µm 85,75% wt

Odabir zuba

Za analizu otklona krvžica odabrano je dvadeset gornjih premolara ekstrahiranih iz ortodontskih razloga. Vizualno su bili bez karijesa, hipoplastičnih defekata ili napuklina. Zubi su očišćeni u tekućoj vodi - ručnim su skelerima skinute meke i tvrde naslage, te su do korištenja spremljeni u 0,9-postotni NaCl s 0,02 posto natrijevih azida na temperaturi od 4°C. Maksimalna buko-palatinalna širina (BPW) izmjerena je digitalnom pomičnom mjerkom s točnošću od 10µm (PowerFix, PagetTrading Ltd, London, Velika Britanija). Vrijednost BPW-a rabila se za podjelu zuba u dvije skupine po deset, a njegova srednja vrijednost u skupini nije odstupala više od pet posto (Tablica 2.). Nakon te podjele zubi su se čuvali u destiliranoj vodi na temperaturi od 23 ± 1°C, osim kada je eksperimentalni postupak zahtijevao izolaciju od vlage. Svaki je zub bio fiksiran s krunom prema gore i okomito postavljen uzdužnom osovinom u smolu (Duracryl Plus, Spofa Dental, Čehoslovačka) stavljenu u kubične kalupe od 15 milimetara s centralno postavljenim otvorom od 12 milimetara u promjeru, izrađene od nehrđajućeg čelika. Smola se protezala dva milimetra od cementno-caklinske granice (ACJ-a).

Teeth selection

Twenty upper premolars, extracted for orthodontic purposes that were free on visual examination from caries, hypoplastic defects or cracks, were selected for cuspal deflection analysis. The teeth were cleaned in tap water, with calculus and soft tissue deposits being removed with a hand scaler and then stored in 0.9% NaCl containing 0.02% sodium azide at 4°C until used. The maximum bucco-palatal width (BPW) for each tooth was measured with an electronic digital caliper accurate to 10 µm (PowerFix, PagetTrading Ltd, London, UK). The BPW dimensions were used to distribute the teeth into two groups of ten teeth and the mean BPW of the teeth between groups varied by no more than 5% (Table 2).

Following distribution into groups, teeth were stored in distilled water at 23 ± 1°C except when aspects of the experimental procedure required isolation from moisture. Each tooth was fixed, crown uppermost and long axis vertical using a chemically cured resin (Duracryl Plus, Spofa Dental, CZ) in a cubic stainless steel mould with dimensions of 15 mm, which had a central cylindrical hole of 12 mm diameter. The resin extended to within 2 mm of the cementoenamel junction (CEJ).

Tablica 2. Dimenzije premolara (mm) korištenih u istraživanju naglašavaju da nije bilo razlike između skupina zuba koji su opskrbljivani
Table 2 Dimensions of the premolar teeth (mm) used in the current study highlighting no differences between the groups of teeth to be restored

Skupina • Group	BPW (mm)		MDW (mm)	
	Srednja vrijednost • Mean	S. D.	Srednja vrijednost • Mean	S. D.
Filtek™ Silorane	9.41	0.25	6.27	0.17
Premise™ Packable	9.42	0.19	6.28	0.13

Preparacija kaviteta

Standardizirani mezio-okluzalno-distalni (MOD-i) kaviteti preparirani su uz pomoć dijamantnog svrdla srednje velikoga zrna (FG837F014, Meisinger, Njemačka) i vodenim hlađenjem, te turbinskom bušilicom s velikim brojem okretaja. Promjer aproksimalnog kaviteta prepariran je do dvije trećine BPW-a širine zuba s dva milimetra širokom gingivnom stijenkom te milimetar iznad caklinsko-cementnog spojišta na cervikalnom dijelu aproksimalnih kaviteta. Okluzalni istmus je prepariran na polovicu vrijednosti BPW-a te tri i pol milimetra duboko od vrška palatinalne kvržice. Kavitetno površinski rub obrađen je do 90° te su zaobljeni svi unutarnji linijski kutovi. Daljnja ujednačenost u preparaciji postignuta je paralelnom preparacijom vestibularne i palatinalne stijenke kaviteta u skladu s navedenim postupkom (17, 19-22).

Restorativni postupak i procjena ogiba kvržica

Dvije skupine zuba nadograđene su u "porcijama" u kombinaciji s njihovim adhezivnim sustavom i slijedeći upute proizvođača. Premolari iz skupine Premise™ Packabla restaurirani su uz pomoć Premise™ Packabla u kombinaciji s trokomponentnim adhezivnim sustavom OptiBond FL (Kerr Corp., Orange, CA, SAD). Zubi su 15 sekundi bili jetkani 37,5-postotnom ortofosfornom kiselinom u gelu (Kerr Corp., Orange, CA, SAD), zatim 15 sekundi ispirani vodenim sprejem te tri sekunde izloženi laganom sušenju na zraku. Na zub je nakon toga 15 sekundi laganim pokretima utrljan OptiBond FL Prime, a sloj se stanjivao komprimiranim zrakom pet sekundi kako bi posvuda bio iste debljine. Zatim je nanesen OptiBond FL Adhesive, ostavljen je 15 sekundi te zatim raspuhivan zrakom pet sekundi i svjetlosno polimeriziran 20 sekundi svjetiljkom Demi LED light-curing system (Kerr Corp., Orange, CA, SADd) kod koje se koristi tehnologija periodičnog pomaka razine snage, tj. svake sekunde njezina izlazna snaga iznosi od 1100 mW/cm^2 do najviše 1330 mW/cm^2, a valne duljine od 450 do 470 nm. Izlazna snaga svjetiljke za polimerizaciju mjerila se nakon svake restauracije ručnim radiometrom Kerr LED. MOD-kaviteti iz skupine Filtek™ Silorana restaurirani su uz pomoć Filtek™ Silorane kompozita i Silorane System Adhesiva, koji je samojetkajući dvokomponentni adhezivni sustav na osnovi metakrilata. Na zub je 15 sekundi crnom mikročetkicom bio nanesen samojetkajući Primer Silorane System Adhesive, zatim je slijedilo pet sekundi laganog ispuhavanja te 20 sekundi svjetlosne polimerizacije. Nakon sloja Primera nanesen je Silorane System Adhesive Bond zelenom mikročetkicom te pet sekudni ispuhivan i 20 sekundi polimeriziran.

Nanošenje kompozita u "porcijama" u objema skupinama sastojalo se od horizontalnih slojeva debljine oko 1 milimetar, stavljenih na dno MOD-kaviteta i lagane vibriracije kako bi se omogućila adaptacija na stijenke kaviteta. Zatim je uneseno u kavitet sljedećih osam "porcija" trokutastog oblika oko dva milimetra debljine - za svaki aproksimalni dio kaviteta po tri te dva za okluzalni. Restauracija kaviteta počela je mezijalnim, slijedio je distalni i na kraju okluzalni kavitet. Nakon adhezivnog postupka na kavitetu zuba, te prije polimerizacije kompozitnog materijala, bukalne i tijekom toga postupka i palatinalne kvržice prepariranih zuba, postavljene su u blizini receptora

Cavity preparation

Standardised mesio-occluso-distal (MOD) cavities were prepared using a medium-grained diamond bur (FG837F014, Meisinger, Germany) in a water-cooled high-speed turbine. The BPW of the proximal box of each cavity was prepared to two-thirds of the BPW of the tooth, 2 mm gingival wall depth and 1 mm above the CEJ at the cervical aspect of the proximal boxes. The occlusal isthmus was prepared to half the BPW having a depth standardised to 3.5 mm from the tip of the palatal cusp. The cavosurface margins were prepared at 90° and all internal line angles were rounded. Further consistency in cavity preparation was ensured by parallel preparation of the facial and palatal walls of the cavity in accordance with a previously reported procedure (17, 19-22).

Restorative procedure and cuspal deflection assessment

The two groups of teeth were incrementally restored in conjunction with their corresponding bonding systems, following manufacturer's instructions. Premolars assigned to Group Premise™ Packable were incrementally restored with Premise™ Packable in conjunction with a three-step etch & rinse dental bonding system, OptiBond FL (Kerr Corp., Orange, CA, USA). The teeth were etched with 37,5 % phosphoric acid gel (Kerr Corp., Orange, CA, USA) for 15 s, rinsed with water spray for 15 seconds and dried with a gentle air stream for 3 seconds. OptiBond FL Prime was applied for 15 seconds with a light brushing motion and air-thinned for 5 s with compressed air to achieve a visibly uniform layer. OptiBond FL Adhesive was applied for 15 seconds, air-thinned for 5 s and light-cured for 20 seconds using Demi LED light-curing system (Kerr Corp., Orange, CA, USA) that employs the power of Periodic Level Shifting technology, shifting per second the output intensity from 1100 mW/cm^2 to a peak of 1330 mW/cm^2 with a wavelength of 450 to 470 nm. The light curing unit output was measured after each restoration using a Kerr LED hand-held radiometer. The MOD cavities of Group Filtek™ Silorane were restored with Filtek™ Silorane composite and Silorane System Adhesive, a methacrylate-based two step self-etch bonding system. Silorane System Adhesive Self-Etch Primer was applied for 15 seconds with black microbrush, followed by 5 seconds gentle air dispersion and 20 seconds of light curing. Silorane System Adhesive Bond was afterwards applied with green microbrush, followed by 5 seconds gentle air dispersion and 20 seconds of light curing.

The incremental composite restorations of the two groups of teeth consisted of a horizontal increment of approximately 1 mm, applied on the floor of the mesio-occluso-distal (MOD) cavity and lightly vibrated to allow adaptation to the cavity walls, and eight triangular-shaped increments of approximately 2 mm thickness, three for each proximal box and two for the occlusal cavity. The cavity was initially restored with the mesial proximal box followed by the distal and occlusal boxes, respectively. Following the adhesive procedure of the cavity surfaces, and before and during polymerization of the composite material, the buccal and palatal cusps of the prepared teeth were approximated to the receptors of two direct current differen-

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dvaju diferencijalnih vodiča direktne struje (DCDT-a) i linearno stavljene mjerne kazaljke gauge (Twin Channel Analogue Gauge Unit: Mercer 122L, Thomas Mercer Ltd, Hertfordshire, Velika Britanija) u skladu s postupkom korištenim u prijašnjim istraživanjima (17, 20-22). Svaka "porcija" bila je osvijetljena 20 sekundi Demi LED light-curing unitom (LCU-om) usmjerenim dva milimetra iznad kvržica.

Zabilježene su početne vrijednosti te je slijedilo mjernje otklona kvržica u realnom vremenu od 320 sekundi, istodobno s početkom svjetlosne polimerizacije za svaku "porciju" kompozita. Otklon kvržica zabilježen u posljednjoj sekundi promatranja (ukupno vrijeme 320 sekundi) smatrao se najboljim odrazom polimerizacijske kontrakcije u preostalom zubu te se utjecaj toplinskog širenja u tom trenutku smatrao minimalnim. Dobiveni podaci za bukalnu i palatinalnu kvržicu kombinirani su, te je tako dobiven ukupan otklon za svaki zub. Izračunane su srednje vrijednosti i standardna devijacija (SD). Podaci su analizirani dvostranim *t-testom* na statističkom programu Stata 11C software (StataCorp LP, Texas, SAD, verzija 2009.). Vrijednost $p < 0,05$ uzeta je kao granica statističke značajnosti.

Procjena adhezivnog sloja

Nakon što su mjesec dana bili uronjeni u dvostruko destiliranu vodu visoke čistoće u svjetlosno nepropusnom spremniku na temperaturi od 37°C, dva premolara iz obje skupine izabrana su slučajnim odabirom (n=2). Zubi su razrezani u buko-palatinalnom smjeru mikrotoma s vodenim hlađenjem (Low Speed Saw - Mitsubishi Ltd, Odjel za materijale Fakuleta za fiziku Sveučilišta u Bukureštu, Rumunjska) i dobiveni su rezovi od milimetra u središnjem području okluzalne površine. Nakon što su osušeni, pripremljeni uzorci položeni su na aluminijske nosače i prekriveni zlatnom prašinom. Ispitivani su raznim povećanjima i nagibima na *Zeissovu Evo 50* elektronskom mikroskopu pri EHT= 10 kV. Za svaku restauraciju kvalitativno je analiziran adhezivni sloj na dnu kaviteta.

Rezultati

Procjena otklona kvržica

Srednje vrijednosti i SD izmjerenih BPW-a širina zuba između ispitivanih skupina nisu bili statistički značajni (Tablica 2.). Oba materijala uzrokovala su ogib kvržica prema sličnom slijedu u veličini mikrometara. Za analizu podataka kombiniran je individualni ogib bukalnih i palatinalnih kvržica za svaki zub. Korišten je svaki podatak ogiba bukalne i palatinalne kvržice dobiven za svaki zub/"porciju". Ogib kvržica nakon restauracije s Filtek™ Siloranom bio je statistički znatno niži nego srednja vrijednost ogiba kvržica izmjerena na zubima restauriranima s Premise™ Packablom ($p < 0,001$ - rezultati uključuju standardnu devijaciju SD-a).

Otklon kvržica nakon restauracije s Filtek™ Siloranom statistički je bio mnogo niži nego srednja vrijednost otklona kvržica zabilježena kod premolara restauriranih s Premise™ Packablom ($p < 0,001$ - rezultati uključuju i SD). Dobiveni su sljedeći podaci:

- za Filtek™ Silorane - otklon kvržica: 1,12 ±0,49 (μm)^{a}, (aritm. sred ± SD);
- za Premise™ Packable - otklon kvržica: 4,03 ±1,19 (μm)^{b}, (aritm. sred ± SD),

Skupine se statistički znatno razlikuju (p<0,001).

tial transducers (DCDT) of a linear displacement measuring gauge (Twin Channel Analogue Gauge Unit: Mercer 122L, Thomas Mercer Ltd, Hertfordshire, UK) in accordance with the procedure utilised in previous studies (17, 20-22). Each increment was irradiated for 20 seconds using Demi LED light-curing unit (LCU) at a distance of 2 mm above the cusp tips. A baseline measurement was taken, followed by a measurement of cuspal deflection in real time for 320 seconds from the start of light-exposure, for each increment of composite. The cuspal deflection recorded in the last second of monitoring (total time, 320 seconds) was considered to reflect most the polymerization shrinkage stress in the remaining tooth as the thermal expansion influence on shrinkage was supposed to be minimal at this moment. The data obtained for the buccal and palatal cusps were combined to give an overall cuspal deflection for each tooth. Mean values and standard deviations (SD) of measurements were calculated. Data were analysed using a two-tailed *t*-test. Stata 11C statistical software (StataCorp LP, Texas, USA, version 2009) was used for data analysis. A *p*-value < 0.05 was considered statistically significant.

Adhesive interface assessment.

After 1 month water storage in high purity double distilled water in a lightproof container maintained at 37°C, two premolars (n=2) randomly selected from each group were sectioned bucco-palatally using a water-cooled microtome (Low Speed Saw - Mitsubishi Ltd, from Department of Materials – Faculty of Physics – University of Bucharest) in order to obtain a 1 mm thick section in the middle of the occlusal surface. After being air dried, the prepared specimens were mounted on aluminium stubs and sputter-coated with gold. Specimens were examined at a variety of magnifications and tilt angles in a *Zeiss Evo 50* scanning electron microscope at EHT= 10 kV. For each restoration the adhesive interface located at the cavity floor was qualitatively analysed.

Results

Cuspal deflection assessment

The means and standard deviations of the BPW dimensions of the teeth did not vary significantly between the tested groups (Table 2). Both materials caused cuspal displacement, following a similar cuspal deflection pattern, in the range of micrometers.The individual buccal and palatal cuspal deflections for each tooth/increment were combined for data analysis. The cuspal deflection following restoration with Filtek™ Silorane was significantly lower than the mean cuspal deflection value recorded for premolars restored with Premise™ Packable. ($p < 0.001$, results including the Standard Deviation – SD). Results obtained there were:

- for Filtek™ Silorane, cuspal deflection was: 1.12 ±0.49 (μm)^{a}, (mean ± SD);
- for Premise™ Packable, cuspal deflection was: 4.03 ±1.19 (μm)^{b}, (mean ± SD),

where groups with different superscript differ significantly (p<0.001).

Procjena adhezivnog spoja

Za dva različita korištena materijala slike SEM-a otkrile su različite stupnjeve prianjanja materijala na zub. Na mjerilu od 200µm slike nisu pokazivale očita pucanja adhezivne veze između restorativnog sustava i dentinskog dna kaviteta, bez obzira na vrstu kompozita i adhezivni sustav.

Silorane System Adhesive stvorio je deblji adhezivni hibridni sloj u usporedbi s OptiBond FL Adhesive Systemom (Slika 1a,b.).

Kod većih povećanja slike SEM-a pokazale su bolji učinak vezanja Premise™ Packable/OptiBond FL restauracija za dentin u usporedbi s Filtek™ Silorane/ Silorane System Adhesivom. Kod primjene Silorane System Adhesiva pojavile su se pukotine između Silorane System Adhesive–Primera i prepariranog dentina, s pukotinama od oko 1µm na dnu kaviteta (Slika 2a,b.).

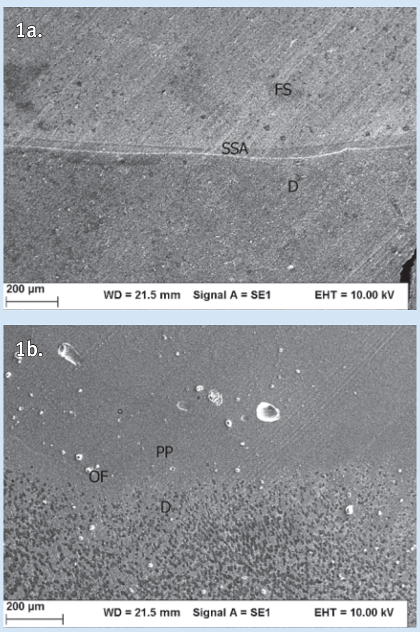
Procjena sloja Silorane System Adhesiva pokazala je postojanost veze između Silorane System Adhesive–Primera i sloja Silorane System Adhesive–Bonda. Silorane System Adhesive-Primer i Silorane System Adhesive –Bond mogu se smatrati za dva zasebna sloja jer se tako i polimeriziraju. Nije bilo nanopropuštanja između Silorane System Adhesive–

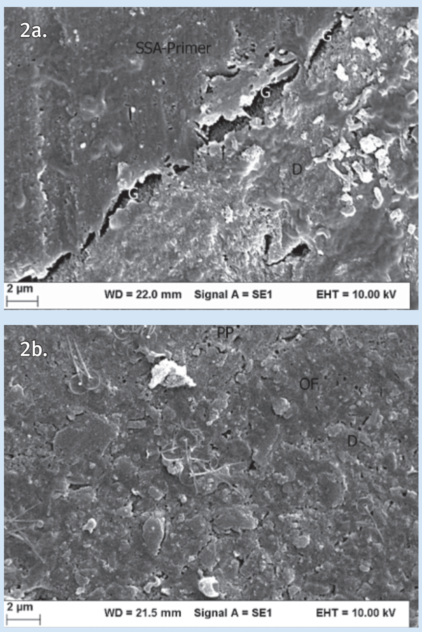
Adhesive interface assessment

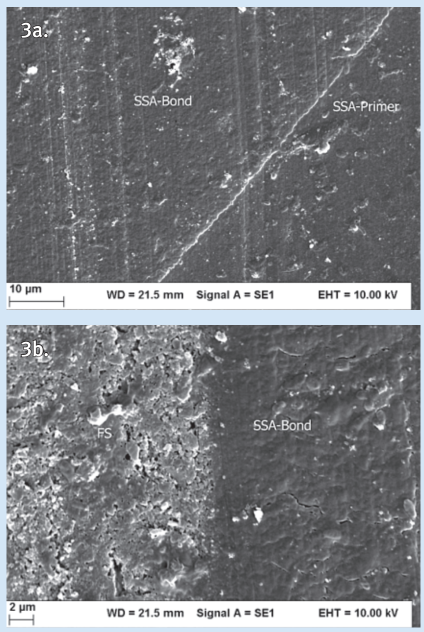
SEM images revealed different cavity adaptation patterns for the two different restorative systems. At 200µm scale, SEM images did not show any obvious adhesive failure between the restorative system and dentin cavity-floor regardless of the type of composite and associated adhesive system. The Silorane System Adhesive realized a thicker adhesive layer in comparison to the OptiBond FL Adhesive System (Fig. 1).

At a higher magnification, SEM images illustrated the better bonding effectiveness of Premise™ Packable /OptiBond FL restorative system to dentin compared to Filtek™ Silorane/ Silorane System Adhesive. Silorane System Adhesive exhibited adhesive failure between Silorane System Adhesive -Primer and the prepared dentin, with gaps of approximately 1µm at the cavity floor interface (Fig. 2).

SEM evaluation of the Silorane System Adhesive layers demonstrated bonding integrity between Silorane System Adhesive -Primer and Silorane System Adhesive -Bond. The Silorane System Adhesive -Primer and Silorane System Adhesive -Bond are distinguishable as two distinct layers since they are cured separately. No cohesive failure occured between Silorane System Adhesive -Bond and Filtek™ Silorane







Slika 1a,b. Slike SEM-prereza restauracija. Restauracijsko/dentinski spoj dna kaviteta kod Premise™ Packabla (a), Filtek™ Silorane restauracije (b). PP-Premise™ Packable, OF - OptiBond FL Adhesive System, FS - Filtek™ Silorane, SSA - Silorane Adhesive System, D - Dentin

Figure 1a,b SEM images of sectioned restorations. Restoration/dentin interface of the cavity floor of a Premise™ Packable (a), respectively, Filtek™ Silorane Restoration (b). PP- Premise™ Packable, OF- OptiBond FL Adhesive System, FS- Filtek™ Silorane, SSA -Silorane Adhesive System, D- Dentin.

Slika 2a,b. Slike SEM-prereza restauracija. Restauracijsko/dentinski spoj dna kaviteta kod Premise™ Packabla (a), Filtek™ Silorane restauracije (b). PP - Premise™ Packable, OF-OptiBond FL Adhesive System, FS- Filtek™ Silorane, SSA - Primer-Silorane Adhesive System Primer, D - Dentin, G - procjep

Figure 2a,b SEM images of sectioned restorations. Restoration/dentin interface of the cavity floor of a Premise™ Packable (a), respectively, Filtek™ Silorane restoration (b). PP- Premise™ Packable, OF- OptiBond FL Adhesive System, FS- Filtek™ Silorane, SSA -Primer-Silorane Adhesive System Primer, D- Dentin, G- Gap.

Slika 3a,b. Slike SEM-prereza restauracija. Restauracijsko/dentinski spoj dna kaviteta kod Filtek™ Silorane (FS) restauracije na različitim magnitudama. Isticanje slojeva Silorane System Adhesiva (SSA), tj. Silorane System Adhesive Primera (SSA -Primer) i Silorane System Adhesive Bonda (SSA-Bond) te adhezivna svojstva Silorane System Adhesive -Bonda i Filtek™ Silorane kompozita.

Figure 3a,b SEM images of the restoration/dentin interface of the cavity floor of a sectioned Filtek™ Silorane (FS) restoration at different magnitudes. Highlighting the layers of Silorane System Adhesive (SSA), namely Silorane System Adhesive Primer (SSA -Primer) and Silorane System Adhesive Bond (SSA -Bond), and the adhesive characteristics of Silorane System Adhesive -Bond to Filtek™ Silorane composite.

Bonda i Filtek™ Silorana, što dokazuje dobru polimerizacijsku kompatibilnost između difunkcionalnih metakrilata iz adheziva i siloranskih monomera u Filtek™ Silorane chemistryu (Slika 3.).

Rasprava

Za ovo su istraživanje materijali odabrani na temelju zajedničke kliničke primjene i raspona polimerizacijske kontrakcije nedavno objavljene u literaturi. Guggenberger je odredio polimerizacijsku kontrakciju 22 metakrilatna kompozita za restauracije i kompozit Filtek™ Silorane, prema njemačkim industrijskim standardima - DIN 13907:2005 (Archimedova metoda), te dobio polimerizacijsko skupljanje od 0,99%±0,07 posto za Filtek™ Silorane i 1,80±0,22 posto za Premise. To su znatno niže vrijednosti u odnosu prema ostalim ispitivanim kompozitima (23). U ovom se istraživanju proučavao ogib kvržica i adaptacija na unutrašnjost kaviteta za dva nisko-kontrahirajuća kompozita za distalna područja (LSPC) - Filtek™ Silorane - kompozit na osnovi silorana i Premise™ Packable - kondenzabilni kompozit na osnovi dimetakrilata. Rezultati potvrđuju spoznaje da se kompoziti nastavljaju mijenjati i nakon osvjetljavanja (25). Korištena DCDT-metoda bila je dobro prilagođena testiranju ogiba kvržica koje uzrokuje polimerizacijska kontrakcija kompozitnog materijala za restauraciju kaviteta. Uočena je korelacija između vrijednosti kontrakcije kompozitnih materijala i količine ogiba kvržica. Ogib kvržica zabilježen u posljednjoj sekundi promatranog razdoblja (t=320s) uzet je kao relevantna vrijednost polimerizacijske kontrakcije u preostalom zubu. Ogib kvržica u 320 sekundi za Filtek™ Silorane, koji pokazuje najniže vrijednosti kontrakcije, iznosio je 1,12 ± 0,49 μm, a za Premise™ Packable, koji se više kontrahira, bio je 4,03 ± 1,19 μm. Kompozit na bazi silorana proizveo je znatno manji ogib kvržica u usporedbi s dimetilmetakrilatnim kompozitom, što je u skladu s prijašnjim istraživanjima (17, 25). Procijenjeno je i objavljeno da (25) je ogib kvržica za 240 s od početka svjetlosne polimerizacije interferometrijskim mlazom raspršenih elektrona (ESPI-em) iznosio gotovo 3,5 μm za eksperimentalni Filtek™ Silorane (zvan Hermes), što je statistički mnogo manje od Premisa (oko 6 μm) i drugih kompozita na osnovi metakrilata. Također je, osim ogiba kvržica, procijenjeno mikrocurenje ekstrahiranih premolara restauriranih s raznim komercijalnim kompozitima na osnovi dimetilmetakrilata i onih s eksperimentalnim kompozitima na osnovi oksirana i silorana, korištenjem iste DCDT-metodologije kao i u ovom istraživanju, ali s drugom jedinicom za polimerizaciju (XL2500, 689 mW/cm^2, 3M ESPE) te slojevitom tehnikom (bez horizontalnog sloja). Uočeno je da je eksperimentalni siloranski kompozit nazvan H1, smanjio ogib kvržica (6,01± 1,8 μm) i mikrocurenje u usporedbi s kompozitima na osnovi dimetilmetakrilata - Filtek™ Z250 (16,5±3,3 μm). U ovom istraživanju razlika u ogibu kvržica nije bila toliko velika između Filtek™ Silorana i Premise™ Packabla, te između Filtek™ Silorana i Filteka™ Z250. Takav je rezultat postignut zato što Premise™ Packable kondenzirajući kompozit za distalna područja s visokim udjelom punila ima viši modul elastičnosti, niže polimeriza-

which demonstrates the good compatibility for polymerisation between difunctional methacrylates of the adhesive and the silorane monomers within Filtek™ Silorane chemistry (Fig.3).

Discussion

The materials used in this study were chosen on the basis of common clinical use and a spectrum of polymerisation shrinkage values recently reported in literature. Guggenberger R, determined the polymerisation shrinkage of 22 restorative methacrylate composites and Filtek™ Silorane composite according to the German Dental Standard DIN 13907:2005 (Archimedes method) and recorded a polymerisation shrinkage of 0.99%±0.07 % for Filtek™ Silorane and 1.80±0.22 % for Premise, significantly lower values compared to the other composites investigated (23). The present study investigated the cuspal deflection and internal cavity adaptation of these two LSPCs, Filtek™ Silorane, a silorane-based composite in comparison to Premise™ Packable, a packable dimethacrylate-based composite. The results of the current study supports previous studies showing that properties of light-activated composites continue to change after irradiation stops (25). The DCDT testing method was well suited to evaluate tooth deflection induced by the polymerisation shrinkage of cavity-restoring composite materials. There was observed a correlation between shrinkage values of the composite materials and the amount of generated cuspal deflection. The cuspal deflection recorded in the last second of monitoring (time, t =320 s) was considered to reflect mostly the polymerisation shrinkage stress in the remaining tooth. Cuspal deflection at 320 s for Filtek™ Silorane, which exhibits the lowest shrinkage value, was 1.12 ± 0.49 μm, whereas Premise™ Packable, which shrinks more, was 4.03 ± 1.19 μm. The silorane-based composite caused significantly less cuspal deflection compared to the dimethacrylate–based composite, which is in agreement with previous reports (17, 25). It was evaluated (25) cuspal deflection for 240 seconds from the start of light-curing process using an electronic speckle pattern inteferometry (ESPI) and reported a value of nearly 3.5 μm for the experimental Filtek™ Silorane (named Hermes), significantly less than for Premise (approximately 6 μm) and other methacrylate-based composites. The cuspal deflection and microleakage of extracted premolar teeth restored with different commercial dimethacrylate-based composites and experimental oxirane and silorane composites was evaluated (17) as well, using the same DCDT methodology as in the present study, but another curing unit (XL2500, 689 mW/cm^2, 3M ESPE) and incremental technique (without horizontal layer). It was observed that the experimental silorane composite, named H1, produced a reduction in cuspal deflection (6.01± 1.8 μm) and decrease in microleakage compared with a dimethacrylate-based composite, Filtek™ Z250 (16.5±3.3 μm). In our study, the difference in cuspal deflection is not so high between Filtek™ Silorane and Premise™ Packable, as between Filtek™ Silorane and Filtek™ Z250, also because Premise™ Packable is the highly filled packable posterior composite on the market, thus having a higher elascijsko skupljanje (26) i stvara manji ogib kvržica u usporedbi s ostalim dimetilmetakrilatnim kompozitima. Apsolutne vrijednosti u ovom istraživanju nisu uspoređene s podacima iz drugih studija jer bi to bilo moguće samo ako bismo se koristili istim uvjetima procjene s jednakim mjerenjem ogiba kvržica, korištenim materijalom i istim uređajem za polimerizaciju (27).

U ovom istraživanju dobiveni su podaci o manjem ogibu kvržica u restauraciji s Filtek™ Siloranom u usporedbi s Premise™ Packablom, što može imati više objašnjenja. Glavno se temelji na različitim procesima polimerizacije tih dvaju LSPC-a, kationskom otvaranju prstenova kompozita na siloranskoj osnovi, što stvara manje volumetrijsko skupljanje u usporedbi s reakcijom dodavanja slobodnih radikala dvostrukih veza kompozita na osnovi dimetilmetakrilata (8). U drugim istraživanjima sa siloranima istaknuta je polimerizacija sa sporim početkom, jer kationska reakcija treba više vremena negoli mehanizam dodavanja slobodnih radikala (28), omogućujući tako da materijal teče i napetost popusti, što rezultira manjim ogibom na kvržicama. Posebna pozornost posvećena je optimalnom vezivanju i adheziji materijala jer, ako se materijal ne stvrdne potpuno, tada polimerizacijska kontrakcija i pomak kvržica mogu biti manji (28). Isti je učinak moguć s djelomičnim kidanjem adhezivnog sloja i odvajanjem kompozitnog materijala iz kaviteta (15).

Kako bi se provjerila adhezivna veza, uporabljen je SEM. Karakterizacija spojnog (hibridnog) sloja uz pomoć SEM-a tih dvaju nisko-kontrahirajućih kompozita za distalna područja, zalijepljena za dentin okluzalnog kaviteta, ovisila je o korištenom adhezivnom sustavu. Premolari iz skupine Premise™ Packabla u "porcijama" su restaurirani kompozitom Premise™ Packablom u kombinaciji s OptiBond FL-om, trokomponentnim adhezivnim sustavom koji se u dentalnoj medicini smatra "zlatnim standardom" kad je riječ o trajnosti veze (9). To što nije bilo pukotina kod restaurativnog sustava OptiBond FL/Premise™ Packabla, pokazuje da je stres polimerizacijske kontrakcije niži nego snaga dentinske sveze za korišteni adhezivni sustav i zubni ispun, te je održana tijekom polimerizacije i nakon nje. SEM-analiza nije otkrila nikakve pukotine u restaurativnom sustavu Silorane System Adhesive/Filtek™ Silorana, između Silorane System Adhesive-Bonda na osnovi metakrilata i siloranskog kompozita, koji su dokazali svoju kemijsku kompatibilnost. Male su pukotine ipak bile pronađene na spoju dentina i Silorane System Adhesive–Primera. Budući da je Filtek™ Silorane nisko-kontrahirajući kompozit koji je stvorio manji otklon kvržica te Silorane Adhesive System zbog svoje debljine može djelovati kao elastični pufer (29), pukotine između dentina i adhezivnog sustava nisu se mogle pripisati kontrakcijskom stresu u procesu polimerizacije, nego manjoj učinkovitosti u stvaranju veze samojetkajućeg sustava adhezije (9). Skladištenje u vodi možda je utjecalo na integritet hidrofilnog samojetkajućeg primera Silorane System Adhesive-Primera koji sadržava HEMA-u (30). U istraživanjima je, u usporedbi s metakrilatnim kompozitima, otkriveno (17) bolje rubno zatvaranje i smanjeno mikrocurenje te manje vrijednosti otklona kvržica kada su se liječnici koristili materijalima sa siloranima. Za detaljno istraživanje testiranih materijala potrebno je više fi-

tic modulus, lower polymerisation shrinkage (26) and thus lower cuspal deflection tendency in comparison to all other known dimethacrylate-based composites. The absolute values of the current studies were not statistically compared to other studies because this would be possible only if the same conditions of evaluations with the same gage of cuspal deflection, materials and light-curing system (27) were used. For the current study, the lower cuspal deflection caused by Filtek™ Silorane in comparison to Premise™ Packable, could have many explanations. The main explanation is based on the different polymerisation process of the two LSPCs, the cationic ring-opening polymerisation process of the silorane-based composite yielding a reduced volumetric shrinkage in comparison to the free-radical addition reaction of the double bonds of the dimethacrylate-based composite (8). Other studies with siloranes have reported a polymerisation reaction with a slow onset as the cation formation needs more time than a free radical formation mechanism (28), allowing time for flow of material and stress relaxation, resulting in less cuspal deflection. A special attention was payed for an optimum cure and adhesion of the materials as if the material did not cure 'completely', then less polymerisation shrinkage may have accounted for a lower cuspal displacement (28), the same effect being possible with a partial debonding of the composite material from the cavity (15).

In order to evaluate the integrity of the adhesive interface, a SEM analysis was performed. SEM interface characterization of the two low-shrinking composites bonded to the dentin of the occlusal cavity floor depended also on the type of adhesive system performance. The premolars of Group Premise™ Packable were incrementally restored with Premise™ Packable in conjunction with OptiBond FL, a three-step etch & rinse dental bonding system, considered in the dental literature as the „golden standard" adhesive system in terms of sealing durability (9). The lack of gaps for OptiBond FL/Premise™ Packable restorative system demonstrated that the polymerisation shrinkage stress of the composite was lower than the dentin bond strength of the adhesive system used and tooth-restoration interface integrity was kept during polymerisation. SEM evaluation of Silorane System Adhesive/Filtek™ Silorane restorative system did not disclose any separation between methacrylate-based Silorane System Adhesive-Bond and the silorane composite, which revealed their chemical compatibility. But small gaps were formed between dentin and Silorane System Adhesive -Primer. Since Filtek™ Silorane is a low-shrinking composite that caused lower cuspal deflection, and the Silorane Adhesive System by its high thickness layer could act as an elastic buffer (29), the gaps between dentin and adhesive system couldn't be attributed to the shrinkage stress that accompanies the polymerisation process, but to the lower efficiency of the self-etch mechanism of adhesion (9). The water storage is possible to have affected the integrity of the hydrophilic self-etch primer of Silorane System Adhesive-Primer that contains HEMA (30). Studies revealed (17) higher marginal adaptation and reduced microleakage formation and lower cuspal deflection when silorane-based materials were used compared to methacrylate composites. For a complete investigation of these materials, it is necessary to use more physicozikalno-kemijskih metoda, poput rendgenske kristalografije, nuklearne magnetske rezonancije, fluorescencije i mikroskopiranja absorpcije infracrvenim zračenjem (32).

Zaključak

Testirana hipoteza djelomice je odbačena, jer je razlika u polimeizacijskoj reakciji RBC-sustava na osnovi silorana rezultirala smanjenim ogibom kvržica, ali i promjenama na integritetu spoja zub/restauracija, u usporedbi s kondenzabilnim RBC-sustavom na osnovi dimetilmetakrilata.

chemical investigation methods, like X Ray Crystallography, Nuclear Magnetic Resonance, fluorescense and infra-red absortion microscopy (31).

Conclusions

The tested hypothesis was partially rejected since differences in polymerisation reaction of the silorane-based RBC system would result in decreased cuspal deflection, but also in the alteration of the tooth/restoration interface integrity in comparison with a packable dimethacrylate RBC system.

Abstract

Low shrinking resin composites are in the focus of research in posterior resin composite restoratives. In this context, the silorane-chemistry, incorporating ring-opening monomers and highly filled packable dimethacrylates seem to be most promising. **Material and Methods:** The goal of this study was to investigate cuspal deflection after light-curing in extracted upper premolars (n=10), using direct current differential transducers (DCDT), and adhesive interface integrity by scanning electron microscopy (SEM) evaluation, of two restorative systems: 1) Filtek™ Silorane/ Silorane System Adhesive (3M ESPE); 2) Premise™ Packable /OptiBond FL (KERR). Data were analysed using a two-tailed *t*-test. **Results:** A p-value < 0.05 was considered statistically significant. Filtek™ Silorane may reduce cuspal deflection associated to the polymerization process, but there is concern regarding durability of adhesive interface integrity. **Conclusions:** Premise™ Packable showed higher cuspal deflection and complete integrity of adhesive interface.

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