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Fluoride-Releasing Restorative Materials: The Effect of a Resinous Coat on Ion Release

Utjecaj smolastog premaza na otpuštanje iona iz restaurativnih materijala koji otpuštaju fluoridne ione

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Abstract
Objective: To determine the effect of two adhesive systems and a glass ionomer coating resin on fluoride release and concurrent pH changes over a period of 168 days. **Material and methods:** Four restorative materials were investigated: a giomer Beautiful II, an "alkasite" material Cention, a conventional composite Filtek Z250, and a glass ionomer cement Fuji IX Extra. Light-cured composite specimens were coated using G-aenial Bond and Clearfil Universal Bond Quick. Glass ionomer specimens were coated using GC Fuji Coat LC. Uncoated specimens were used as references. Quantitative fluoride release and pH changes were measured after 1 h, 24 h, 2 days, 7 days, 28 days, 84 days, and 168 days. **Results:** The cumulative fluoride release after 168 days increased for uncoated specimens in the following order: Filtek Z250 < Beautifil II < Cention < Fuji IX Extra. A comparatively lower fluoride release was measured for the composites coated with Clearfil Universal Bond Quick, with cumulative values after 168 days increasing in the following order: Filtek Z250 < Beautifil II < Cention. The composites coated with G-aenial Bond showed lower fluoride release compared to the uncoated specimens, with cumulative values increasing in the following order: Filtek Z250 < Beautifil II < Cention. The composites coated with G-aenial Bond showed pH values in the acidic range (4.4-5.7) after 1 h and 24 h. **Conclusion:** Fluoride release varied among the investigated restorative materials and depended on the use of dental adhesives and coatings. The pH of all materials, coating types and time points varied.

Introduction

Glass ionomer cements (GICs), giomers, and composites are capable of releasing fluoride ions; therefore, they are used in restorative dental medicine for temporary and permanent fillings (1). Fluoride ions have been incorporated into dental materials. They have been shown to produce a remineralizing effect and reduce cariogenic potential by acting on the growth and metabolism of *S. mutans (*1-4).

The release of fluoride ions from dental materials depends on factors related to an individual material and factors related to oral environment. The characteristics of materials such as composition, filler content, powder/liquid ratio, mixing process, and surface exposed to the aqueous medium affect the release of fluoride ions. Environmental factors include the pH and composition of the immersion medium and application of a dental adhesive system or coat (1, 5-10).

Previous studies showed a negative effect of a dental adhesive system or coat on the quantity of released fluoride ions.

Uvod

Staklenoionomerni cementi (SIC), giomeri i kompoziti oslobađaju fluoridne ione i zato se primijenjuju u restaurativnoj dentalnoj medicini za privremene i trajne ispune (1). Pokazalo se da dodatak fluoridnih iona u zubne materijale remineralizacijski djeluje te smanjuje kariogeni potencijal bakterija što utječe na rast i metabolizam *S. mutans* (1 – 4).

Otpuštanje fluoridnih iona iz dentalnih materijala ovisi o čimbenicima koji su vezani za svaki materijal te onima povezanima s oralnim okolišem. Karakteristike materijala kao što su sastav, sadržaj punila, omjer prah – tekućina, postupak miješanja i površina izložena vodenom mediju utječu na otpuštanje fluoridnih iona. Čimbenici okoliša uključuju pH i sastav imerzijskog medija te primjenu dentinskoga adhezijskog sustava ili premaza za staklenoionomerne cemente (1,5 – 10).

U ranijim ispitivanjima autori su istaknuli negativan učinak dentinskih adhezijskih sustava ili premaza na ukupnu količinu otpuštenih fluoridnih iona jer se smatra da tvore hi-

This has been attributed to the adhesive system or coat forming a hydrophobic barrier and thereby diminishing the diffusion of fluoride ions. This effect was more pronounced in resin composites than in GICs, which depended on type of the adhesive system or coat (7, 11-13).

Apart from the well-known classes of restorative materials that are capable of releasing fluoride ions (GICs and giomers), a novel "alkasite" restorative material has recently been introduced to the dental market. This bioactive material is capable of releasing remineralizing calcium and fluoride ions and can also neutralize acid by releasing OH⁻ ions. It is compositionally similar to the group of composite materials and is designed to be used with or without an adhesive system, depending on the operator's preferences (14, 15).

The effect of contemporary universal adhesives on fluoride release from remineralizing resin composite materials has not been investigated up to date. Therefore, the aim of this study was to compare fluoride release from specimens of restorative materials that were either coated or uncoated with a resinous layer of an adhesive system (for composite materials) and coat (for a GIC). Additionally, the effects of a resinous layer on the pH changes of the immersion medium were investigated. The first null hypothesis was that there would be no difference in fluoride ion release and pH changes between coated and uncoated specimens. The second null hypothesis was that the release of fluoride ions and pH changes of the immersion medium did not differ among the investigated restorative materials.

Material and methods

Restorative materials, adhesive systems, and a glass ionomer coat investigated in this study are presented in Table 1. Six specimens were prepared for each experimental group (n = 6).

Specimens of the composite materials were prepared using cylindrical Teflon molds with a diameter of 6 mm and a height of 2 mm. The molds were placed on a polyethylene terephthalate (PET) foil, filled with uncured material and covered with another layer of PET foil (16). The excess material was removed and specimens were polymerized using an LED curing unit (Bluephase G2, Ivoclar Vivadent, Schaan, Liechtenstein) with a nominal intensity of 1200 mW/cm² for 20 s on each side. Specimens of the GIC were cast into the mold described above, covered with PET films, and left to set for 6 min according to manufacturer's instructions (17). To ensure that the specimens were completely surrounded by an aqueous medium, a plastic thread incorporated within each specimen was used to hang the specimens from the cap of the vial.

The schematic overview of the study design is presented in Figure 1. Each sample was immersed separately in a plastic vial containing 5 ml of deionized water at 37 °C and evaluated after 0, 1, 2, 7, 28, 84, and 168 days. At each time interval, the specimens were removed from the immersion medium, placed in 5 ml of new deionized water, and stored in the incubator at 37 °C until the next time point. The pH of the immersion medium was measured using the pH meter MP 220 (Mettler Toledo, Columbus, Ohio) and the InLab Ex-

drofobnu barijeru i time umanjuju difuziju fluoridnih iona. Učinak je bio izraženiji u smolastim kompozitima negoli u staklenoionomernim cementima te je ovisio o vrsti adhezijskog sustava ili premaza (7, 11 – 13).

Uz dosad poznate restaurativne materijale koji mogu otpuštati fluoridne ione (SIC i giomeri), na stomatološkom tržištu nedavno je predstavljen i jedan novi. Taj bioaktivni materijal otpušta remineralizirajuće ione kalcija i fluorida, a također može neutralizirati kiselinu otpuštanjem OH⁻ iona. Sastavom je sličan skupini kompozitnih materijala i namijenjen je za upotrebu s dentinskim adhezijskim sustavom ili bez njega, ovisno o željama operatera (14, 15).

Utjecaj suvremenih univerzalnih dentinskih adhezijskih sustava na otpuštanje fluoridnih iona iz remineralizirajućih kompozitnih materijala do danas nije istražen. Zato je cilj ove studije bio usporediti otpuštanje fluoridnih iona iz nepremazanih uzoraka i uzoraka restaurativnih materijala tretiranih smolastim slojem adhezijskog sustava (za kompozitne materijale) i premazom (za SIC-e). Također su istraživani učinci smolastoga sloja na promjene pH vrijednosti imerzijskog medija. Prva nulta hipoteza bila je da neće biti razlike u otpuštanju fluoridnih iona i promjeni pH između premazanih i nepremazanih uzoraka. Druga nulta hipoteza glasila je da se otpuštanje fluoridnih iona i promjene pH imerzijskog medija ne razlikuju među istraživanim restaurativnim materijalima.
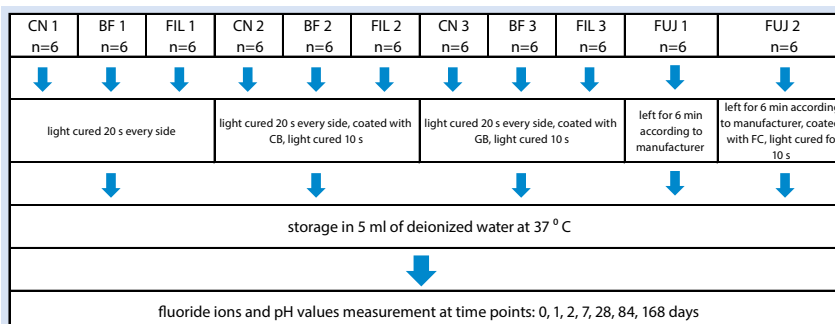
Materijali i postupci

Restaurativni materijali, dentinski adhezijski sustavi i staklenoionomerni premaz istraživani u ovoj studiji navedeni su u tablici 1. Za svaku skupinu bilo je pripremljeno šest uzoraka (n = 6).

Uzorci kompozitnih materijala pripremljeni su s pomoću cilindričnih teflonskih kalupa promjera 6 mm i visine 2 mm. Kalupi su postavljeni na polietilen-tereftalatnu foliju (PET), napunjeni materijalom i prekriveni drugim slojem PET folije (16). Višak materijala je uklonjen, a uzorci su polimerizirani LED (engl. *light emitting diode*) polimerizacijskom svjetiljkom Bluephase G2 (Ivoclar Vivadent, Schaan, Lihtenštajn) nominalnog intenziteta 1200m W/cm² u trajanju od 20 sekunda sa svake strane. Uzorci SIC-a stavljeni su u opisani kalup, prekriveni PET folijama i ostavljeni šest minuta da se stvrdnu, prema uputama proizvođača (17). Kako bi se osiguralo da su uzorci potpuno okruženi vodenim medijem, korišten je plastični konac inkorporiran u svaki uzorak, a s pomoću kojega su uzorci visjeli s čepa bočice.

Shematski prikaz dizajna studije nalazi se na slici 1. Svaki uzorak bio je zasebno uronjen u plastičnu bočicu koja je sadržavala 5 mL deionizirane vode na temperaturi od 37 °C i ispitivan nakon 0, 1, 2, 7, 28, 84 i 168 dana. Pri svakom vremenskom intervalu uzorci su uklonjeni iz imerzijskog sredstva, zatim stavljeni u 5 mL nove deionizirane vode i pohranjeni u inkubator na 37 °C do sljedeće vremenske točke. pH vrijednost imerzijskog sredstva mjerila se pH metrom MP 220 (Mettler Toledo, Columbus, Ohio, SAD) i elektrodom InLab Expert Pro pH (Mettler Toledo, Columbus, Ohio, SAD).

Table 1   Materials used in this study
Tablica 1. Materijali korišteni u istraživanju

Material class • Vrsta materijala	Commercial name (abbreviation) • Komercijalni naziv (kratica)	Composition • Sastav	Manufacturer • Proizvođač	Shade/LOT No. • Nijansa/LOT broj
Giomer	Beautifil II (BF)	S-PRG (surface pre-reacted glass ionomer) Nano fillers 83.3 wt% • Nano punila 83,3 wt %	Shofu Dental GmbH, Ratingen, Germany • Njemačka	A2/051829
Alkasite • Alkasit	Cention (CN)	Filler: calcium fluorosilicate glass, Ba-Al silicate glass, Ca-Ba-Al fluorosilicate glass, Ytterbium trifluoride, Isofiller • Punila: kalcijevo fluorosilikatno staklo, Ba-Al silikatno staklo, Ca-Ba-Al fluorosilikatno staklo, iterbijev trifluorid, *Isofiller* Monomers: urethane dimethacrylate UDMA, Tricyclodecan-dimethanol dimethacrylate DCP, Aromatic-aliphatic-UDMA, Polyethylene glycol 400 dimethacrylate PEG-400 DMA • Monomeri: uretan-dimetakrilat UDMA, Triciklodekan-dimetanol dimetakrilat DCP, aromatski-alifatski-UDMA, Polietilen-glikol 400 dimetakrilat PEG-400 DMA Initiator system: hydroperoxide, Ivocerin and acyl phosphine oxide • Inicijatorski sustav: hidroperoksid, Ivocerin i acil-fosfin oksid	Ivoclar Vivadent, Schaan, Liechtenstein • Lihtenštajn	A2/XL7102
Glass ionomer • Stakleni ionomer	Fuji IX Extra (FUJ)	Liquid: 5-10% polybasic carboxylic acid • Tekućina: 5 – 10 % polibazična karboksilna kiselina Powder: glass, oxide • Prašak: staklo, oksidi	GC Europe, Leuven, Belgium • Belgija	A3/1801171
Conventional composite • Konvencionalni kompozit	Filtek Z250 (FIL)	Matrix: Bisphenol A diglycidyl ether dimethacrylate (BIS-GMA) and triethylene glycol dimethacrylate (TEGDMA), UDMA • Matrica: Bisfenol A diglicidil dimetakrilat (BIS-GMA) i trietilen-glikol dimetakrilat (TEGDMA), UDMA Filler: zirconia and silica particles 78.5 wt %, 60% vol. • Punilo: čestice cirkonija i silicijeva dioksida 78,5 wt %, 60 % vol.	3M Deutschland GmbH, Neuss, Germany • Njemačka	A2/N984652
Universal adhesive • Univerzalni adhezivni sustav	G-aenial Bond (GB)	acetone 25-50% • aceton 25 – 50 % dimethacrylate 10-20% • dimetakrilat 10 – 20 % phosphoric acid ester monomer 5-10% • monomer estera fosforne kiseline 5 –10 % dimethacrylate component 1-5% • dimetakrilatna komponenta 1 – 5 % photoinitiator 1-5% • fotoinicijator 1 – 5 % butylated hydroxytoluene (BHT) < 0.5% • butilirani hidroksitoluen (BHT) < 0,5 %	GC Europe, Leuven, Belgium • Belgija	1811281
Universal fluoride-releasing adhesive • Univerzalni adhezivni sustav koji otpušta fluoride	Clearfil Universal Bond Quick (CB)	BIS-GMA 10-25%, ethanol 10-25%, 2-hydroxyethyl methacrylate 2.5-10%, 10-Methacryloyloxydecyl dihydrogen phosphate, hydrophilic amide monomers, colloidal silica, silane coupling agent, sodium fluoride, dl-Camphorquinone, water • BIS-GMA 10 – 25 %, etanol 10 – 25 %, 2-hidrosietil metakrilat 2,5 –10 %, 10-metakriloiloksidecil dihidrogen-fosfat, hidrofilni amidni monomeri, koloidni silicijev dioksid, silansko vezno sredstvo, natrijev fluorid, dl-kamforkinon, voda	Kuraray Europe, Hattersheim am Main, Germany • Hattersheim na Majni, Njemačka	3L0108
Glass ionomer coat • Staklenoionomerni premaz	GC Fuji Coat LC (FC)	methyl methacrylate (MMA) 25-50% • metil-metakrilat (MMA) 25 – 50 % photoinitiator 1-5% • fotoinicijator 1 – 5 % butylated hydroxytoluene (BHT) < 1% • butilirani hidroksitoluen (BHT) < 1 %	GC Europe, Leuven, Belgium • Belgija	1804021

Figure 1 Schematic representation of the study design.
Slika 1. Shematski prikaz dizajna studije

pert Pro pH electrode (Mettler Toledo, Columbus, Ohio). Prior to the pH measurement, the electrode was calibrated using standard buffer solutions at pH = 4 and pH = 7.

A volume of 4.5 ml of the medium and 0.5 ml of TISAB III buffer (Total Ionic Strength Adjustment Buffer, Thermo Fisher Scientific, Chelmsford, USA) were placed in a new beaker to determine the fluoride ions concentration. Quantitative fluoride ions release was measured using a standard ion-selective electrode Orion 9609BNWP (Thermo Fisher Scientific, Massachusetts, USA), ISO 19448: 2018. Before measurements, the ion-selective electrode was calibrated using a series of standards of known concentration in a range of 10^{-5}–10^{-2} mol/L F$^-$.

The fluid samples were placed on a RH magnetic stirrer (IKA-Werke GmbH & Co. KG, Staufen, Germany), which was set at 500 rpm without heating. A sensor was placed inside the fluid sample, which rotated at the indicated speed during the measurement. An ion-selective electrode was connected to an Expandable Ion Analyzer EA 940 (Orion Research, Beverly, USA) from which the values were read. An average of 3 readings was calculated for each sample.

Normality of distribution was verified using the Shapiro Wilk's test, and equality of variances was checked using the Levene's test. Mean values of fluoride release and pH changes of the immersion medium were compared among the combinations of materials and resinous coatings using a one-way ANOVA with Tukey post-hoc adjustment. Mean values of fluoride release and pH changes of the immersion medium for each combination of materials and resinous coatings were compared among different measurement times using repeated-measures ANOVA with Bonferroni post-hoc adjustment. For all analyses, the overall level of significance was set at 0.05. Statistical analysis was performed in SPSS 20 (IBM, Armonk, NY, USA).

Results

Fluoride release

Mean concentrations of released fluoride ions from uncoated specimens are presented in Figure 2. All uncoated groups showed a statistically significant increase in released fluoride ions over time in the following order: BF < CN < FUJ. When comparing cumulative values after 168 days, FUJ demonstrated 3 times higher values than CN, and 35 times higher values than BF.

The effect of coating systems on fluoride release is shown in Table 2. The coating of composite specimens with adhesive systems led to a statistically significant decrease in the amount of released fluoride ions. Materials treated with GB released more fluoride ions than materials treated with CB. FIL released fluoride ions only when fluoride-releasing adhesive CB was applied. Both FUJ groups showed a statistically significant increase in released fluoride ions over time, whereas uncoated specimens reached 30 times higher values compared to the specimens coated with FC.

Prije mjerenja pH, elektroda je kalibrirana s pomoću standardnih puferskih otopina pH = 4 i pH = 7.

Volumen od 4,5 mL tekućine i 0,5 mL pufera TISAB III (Total Ionic Strength Adjustment Buffer, Thermo Fisher Scientific, Chelmsford, SAD) stavljen je u novu čašicu za određivanje koncentracije fluoridnih iona. Kvantitativna količina otpuštenih fluoridnih iona mjerena je standardnom metodom ionski selektivnom elektrodom Orion 9609BNWP (Thermo Fisher Scientific, Massachusetts, SAD) propisanom normom ISO 19448:2018. Ionski selektivna elektroda se prije mjerenja baždarila standardnom metodom s pomoću razrijeđenih komercijalnih standarda koncentracija u rasponu od 10^{-5} do 10^{-2} mol/L F$^-$.

Pri mjerenju se uzorak tekućine nalazio na digitalnoj magnetskoj miješalici RH digital (IKA-Werke GmbH & Co. KG, Staufen, Njemačka) koja je bila podešena na 500 rpm (engl. *revolutions per minute*), bez grijanja miješalice. U uzorak tekućine uronjen je senzor koji se tijekom mjerenja vrtio navedenom brzinom. Ionski selektivna elektroda bila je spojena na čitač Expandable IonAnalyzer EA 940 (Orion Research, Beverly, SAD) s kojega su se očitavale vrijednosti. Za svaki uzorak tekućine očitale su se tri vrijednosti te izračunao njihov prosjek.

Normalnost distribucije rezultata provjerena je Shapiro-Wilksovim testom, a jednakost varijanci Leveneovim testom. Prosječne vrijednosti otpuštanja fluoridnih iona i promjene pH vrijednosti imerzijskog sredstva uspoređivane su među kombinacijama materijala i smolastih premaza korištenjem jednosmjerne ANOVA-e s Tukeyjevom post-hoc korekcijom. Prosječne vrijednosti otpuštanja fluoridnih iona i promjene pH imerzijskog sredstva za svaku kombinaciju materijala i smolastih premaza uspoređivane su između različitih vremenskih intervala korištenjem ponovljenih mjerenja ANOVA-e s Bonferronijevom post-hoc korekcijom. Za sve analize je razina značajnosti bila postavljena na 0,05. Statistička analiza provedena je u SPSS 20 programu (IBM, Armonk, NY, SAD).

Rezultati

Otpuštanje fluoridnih iona

Srednje vrijednosti koncentracija otpuštenih fluoridnih iona iz restaurativnih materijala bez premaza prikazane su na slici 2. Svi restaurativni materijali bez premaza pokazali su statistički značajan porast otpuštenih fluoridnih iona tijekom vremena sljedećim redoslijedom: BF < CN < FUJ. Uspoređujući kumulativne vrijednosti nakon 168 dana, FUJ je pokazao tri puta veće vrijednosti od CN-a i 35 puta veće od BF-a.

Učinak premaza na otpuštanje fluoridnih iona prikazan je u tablici 2. Premazivanje kompozitnih uzoraka dentinskim adhezijskim sustavima rezultiralo je statistički značajnim smanjenjem količine otpuštenih fluoridnih iona. Materijali tretirani GB-om otpustili su više fluoridnih iona od onih tretiranih CB-om. FIL je otpustio fluoridne ione samo kada je tretiran dentinskim adhezijskim sustavom CB. Obje FUJ skupine pokazale su statistički značajan porast otpuštenih fluoridnih iona tijekom vremena te su nepremazani uzorci dosegnuli 30 puta veće vrijednosti u usporedbi s uzorcima premazanima FC-om.

Table 2   Fluoride ions release from coated and uncoated specimens. Uppercase letters denote statistically homogeneous groups within materials subgroups (with or without coat). Lowercase letters denote statistically homogeneous materials subgroups (time points). Values are presented in ppm. Standard deviations are presented in parentheses.

Tablica 2.   Otpuštanje fluoridnih iona iz premazanih i nepremazanih uzoraka. Velika slova označavaju statistički homogene skupine unutar podskupina materijala (s premazom i bez premaza). Mala slova označavaju statistički homogene podskupine materijala (vremenske točke). Vrijednosti su u ppm. Standardne devijacije prikazane su u zagradama.

Time (days) • Vrijeme (dani)	FILTEK Z250			BEAUTIFIL II			CENTION			FUJI IX EXTRA	
	Uncoated • Bez premaza	Clearfil Universal Bond Quick	G-aenial Bond	Uncoated • Bez premaza	Clearfil Universal Bond Quick	G-aenial Bond	Uncoated • Bez premaza	Clearfil Universal Bond Quick	G-aenial Bond	Uncoated • Bez premaza	GC Fuji COAT LC
0	0	0 A	0	0.05 (0.03) Aa	0.026 (0.02) Aab	0.01 (0.01) Ab	0.08 (0.02) Aa	0.004 (0.001) Ab	0.005 (0.002) Ab	2.95 (0.5) A	0.18 (0.06) A
1	0	0 A	0	0.11 (0.05) Aa	0.026 (0.02) Ab	0.013 (0.01) Ab	4.22 (0.44) Ba	0.057 (0.02) Ab	0.386 (0.2) Ab	7.95 (0.89) B	0.26 (0.1) A
2	0	0 A	0	0.14 (0.06) Aa	0.026 (0.02) Ab	0.013 (0.05) Ab	7.15 (0.64) Ca	0.071 (0.02) ABb	0.462 (0.22) Ab	9.94 (0.84) C	0.18 (0.12) A
7	0	0.0000833 A	0	0.22 (0.07) Aa	0.025 (0.02) Ab	0.016 (0.04) Ab	8.93 (0.75) Da	0.08 (0.03) ABCb	0.91 (0.33) Ac	14.5 (0.56) D	0.61 (0.23) AB
28	0	0.000695 B	0	0.6 (0.16) Ba	0.043 (0.01) Ab	0.087 (0.01) Ab	10.12 (0.99) DEa	0.23 (0.05) BCb	2.669 (0.72) Bc	22.49 (1.16) E	0.97 (0.33) BC
84	0	0.0007667 B	0	1.01 (0.26) Ca	0.08 (0.03) Bb	0.322 (0.07) Bc	10.8 (1.06) Ea	0.24 (0.05) Cb	3.088 (0.69) Bc	45.7 (2.12) F	1.18 (0.42) C
168	0	0.0007667 B	0	2.19 (0.4) Da	0.08 (0.03) Bb	1.77 (0.17) Bc	12.75 (1.07) Fa	0.80 (0.23) Db	5.072 (0.99) Cc	55.49 (3) G	1.85 (0.5) D

Table 3   pH changes in coated and uncoated groups. Uppercase letters denote statistically homogeneous groups within materials subgroups (with or without coat). Lowercase letters denote statistically homogeneous materials subgroups within time points. Values are presented in ppm. Standard deviations are presented in parentheses.

Tablica 3.   Promjene pH vrijednosti kod premazanih i nepremazanih uzoraka. Velika slova označavaju statistički homogene skupine unutar podskupina materijala (sa i bez premaza). Mala slova označavaju statistički homogene podskupine materijala (vremenske točke). Vrijednosti su predstavljene u ppm. Standardne devijacije prikazane su u zagradama.

| Time (days) • Vrijeme (dani)	FILTEK Z250			BEAUTIFIL II			CENTION			FUJI IX EXTRA	
	Uncoated • Bez premaza	Clearfil Universal Bond Quick	G-aenial Bond	Uncoated • Bez premaza	Clearfil Universal Bond Quick	G-aenial Bond	Uncoated • Bez premaza	Clearfil Universal Bond Quick	G-aenial Bond	Uncoated • Bez premaza	GC Fuji COAT LC
0	6.16 (0.08) Aa	6.41 (0.2) Ab	4.42 (0.08) Ac	7.27 (0.16) Aa	6.64 (0.33) Ab	4.75 (0.11) Ac	6.83 (0.11) ABCa	7 (0.2) ABCa	4.53 (0.16) Ab	6.47 (0.27) A	6.42 (0.08) A
1	6.26 (0.04) Aa	7.63 (0.32) Cb	5.39 (0.74) Bc	6.53 (0.06) Ba	6.61 (0.14) Aa	5.67 (0.21) Bb	6.99 (0.2) Ca	6.52 (0.19) Ab	5.73 (0.38) Bc	6.81 (0.33) AB	6.89 (0.19) B
2	7.5 (0.32) Da	7.5 (0.4) Ca	6.89 (0.23) Cb	6.57 (0.24) Ba	6.58 (0.09) Aa	6.9 (0.15) CDb	6.61 (0.15) Aa	6.92 (0.51) Aba	6.79 (0.25) Ca	7.11 (0.2) B	6.96 (0.22) B
7	6.96 (0.21) Ca	6.8 (0.15) ABab	6.64 (0.23) Cb	6.99 (0.2) Aa	6.9 (0.13) ABa	7.44 (0.27) Db	6.87 (0.26) ABCa	7.55 (0.25) CDb	7.19 (0.22) Cab	6.76 (0.1) AB	7.64 (0.29) C
28	6.96 (0.09) Ca	6.49 (0.11) Ab	6.4 (0.09) Cb	7.49 (0.18) Ca	7.03 (0.12) BCb	7.48 (0.35) Da	6.68 (0.19) ABa	7.23 (0.33) BCDa	6.85 (0.58) Ca	6.57 (0.16) A	7.79 (0.22) C
84	6.86 (0.12) BCab	6.97 (0.1) Bb	6.69 (0.15) Ca	7.48 (0.31) Ca	7.19 (0.26) BCa	7.19 (0.41) CDa	6.78 (0.1) ABCa	7.62 (0.26) Db	6.91 (0.36) Ca	6.63 (0.12) A	7.87 (0.23) C
168	6.63 (0.04) Ba	6.97 (0.09) Bb	6.74 (0.12) Ca	7.47 (0.17) Ca	7.27 (0.24) Ca	6.71 (0.55) Cb	6.91 (0.08) BCa	7.4 (0.37) BCDb	6.94 (0.12) Ca	6.56 (0.25) A	7.89 (0.15) C

pH changes

pH changes of uncoated specimens are presented in Figure 3. The pH of dental materials differed in the first measurement (1 h time point) in the following order: FIL < FUJ < CN < BF (6.16, 6.47, 6.83, and 7.26). Also, the values differed at the last measurement (168 days): FUJ < FIL < CN < BF (6.56, 6.63, 6.91, and 7.45).

pH values in coated and uncoated specimens are presented in Table 3. pH values showed growth tendency over time

Promjene pH vrijednosti

Vrijednosti pH otopine kod restaurativnih materijala korištenih bez premaza prikazane su na slici 3. Vrijednosti pH među dentalnim materijalima razlikovale su se pri prvom mjerenju poslije jednoga sata prema sljedećem redoslijedu: FIL < FUJ < CN < BF (6,16, 6,47, 6,83 i 7,26). Vrijednosti su se također razlikovale pri zadnjem mjerenju nakon 168 dana: FUJ < FIL < CN < BF (6,56, 6,63, 6,91 i 7,45).
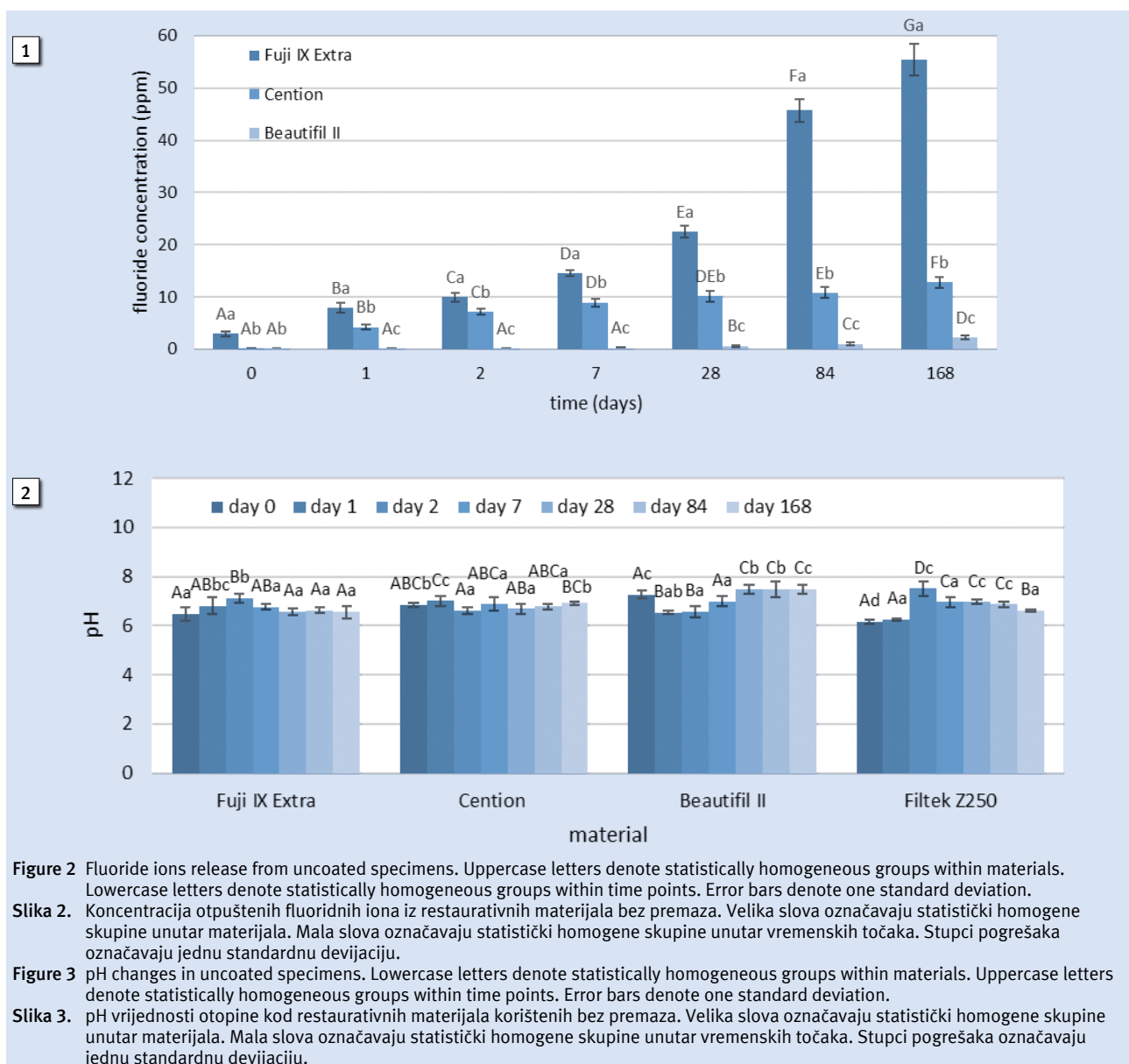
Figure 2 Fluoride ions release from uncoated specimens. Uppercase letters denote statistically homogeneous groups within materials. Lowercase letters denote statistically homogeneous groups within time points. Error bars denote one standard deviation.

Slika 2. Koncentracija otpuštenih fluoridnih iona iz restaurativnih materijala bez premaza. Velika slova označavaju statistički homogene skupine unutar materijala. Mala slova označavaju statistički homogene skupine unutar vremenskih točaka. Stupci pogrešaka označavaju jednu standardnu devijaciju.

Figure 3 pH changes in uncoated specimens. Lowercase letters denote statistically homogeneous groups within materials. Uppercase letters denote statistically homogeneous groups within time points. Error bars denote one standard deviation.

Slika 3. pH vrijednosti otopine kod restaurativnih materijala korištenih bez premaza. Velika slova označavaju statistički homogene skupine unutar materijala. Mala slova označavaju statistički homogene skupine unutar vremenskih točaka. Stupci pogrešaka označavaju jednu standardnu devijaciju.

in all tested materials. FIL and CN showed higher pH values when coated with CB than the uncoated group. BF showed the highest values in the uncoated group. FUJ showed higher values when coated with FC. GB showed lower pH values in all groups after 1 hour and 24 hours. After 1 hour, the pH values among the materials increased in the following order: FIL < CN < BF (4.42, 4.53, and 4.75). After 24 hours, the pH values increased among the materials in the following order: FIL < BF < CN (5.39, 5.67, and 5.73).

Vrijednosti pH premazanih i nepremazanih uzoraka prikazane su u tablici 3. Vrijednosti pH pokazale su tendenciju rasta tijekom vremena u svim ispitivanim materijalima. FIL i CN imali su veće vrijednosti pH kada su bili premazani CB-om u usporedbi sa skupinom bez premaza. BF je pokazao najviše vrijednosti u skupini bez premaza. FUJ je pokazao veće vrijednosti kada je korišten premaz FC. GB je pokazao niže pH vrijednosti u svim skupinama poslije jednoga sata i 24 sata. Nakon jednoga sata pH vrijednosti među materijalima povećavale su se prema sljedećem redoslijedu: FIL < CN < BF (4,42, 4,53 i 4,75). Nakon 24 sata vrijednosti pH povećale su se među materijalima sljedećim redoslijedom: FIL < BF < CN (5,39, 5,67 i 5,73).

Discussion

This study investigated fluoride release of four restorative materials and the effect of contemporary universal adhesive systems, and a coat on fluoride ions release. Also, for all coat-

Rasprava

U ovoj studiji autori su istraživali otpuštanje fluoridnih iona iz četiriju restaurativnih materijala i učinak suvremenih univerzalnih dentinskih adhezijskih sustava i premaza

ed and uncoated specimens, pH changes were evaluated. According to a statistically significant effect of the material type and resinous coating on fluoride release and pH changes, both null hypotheses were rejected.

All fluoride-releasing materials used in this study showed long-term fluoride release, as reported in several previous studies (18, 19). FUJ presented the highest cumulative values of released fluoride ions, followed by CN and BF. As expected, the conventional composite FIL did not release fluoride ions, except when its specimens were coated with the fluoride-releasing adhesive CB.

GICs are characterized by the initial release of a large amount of fluoride ions called the burst effect which occurs within 24 h of cement setting time (20-22). According to Wiegand et al., this effect results from a reaction between glass particles and polyalkenoate acid (1).

Our study confirmed a similar burst effect in FUJ specimens, and also in the alkasite composite material CN. However, the burst effect was present only in uncoated FUJ and CN specimens. The observed burst effect found in CN could be a result of its composition. According to the respective manufacturer, this material comprises 78.4 wt% of the following inorganic fillers: barium aluminum silicate glass, ytterbium trifluoride, Isofiller (patented filler), calcium barium aluminum fluorosilicate glass, and a calcium fluorosilicate (alkaline) glass. Also, 24.6 wt% of the material is composed of alkaline (calcium fluorosilicate) glass filler, which is responsible for fluoride, hydroxide, and calcium ions release (14).

Our results are partially in contrast with those from a study of Gupta et al., which tested similar materials, Cention N (self- and light-cured) and a conventional GIC. Their results showed a time-dependent decrease in fluoride release from all tested materials, except for GIC in an acidic immersion medium. This difference between the released fluoride ions could be a result of different measuring time periods in the aforementioned study (7, 21, and 28 days). However, their results of a GIC in neutral medium releasing significantly higher amounts of fluoride ions than Cention N corroborate our results of fluoride ions release in these materials (23).

Tiskaya et al. evaluated fluoride release, pH changes, and apatite formation of two bioactive composites (Cention N and Activa). Their cumulative values of fluoride ions release were below 8 ppm after 42 days, which is lower than the values obtained in our study (30.49 ppm after 28 days). This difference could be due to different specimen geometry and different immersion media. In our study, specimens were stored in deionized water while artificial saliva was used in the aforementioned study. Specimens immersed in artificial saliva tend to show a 17–25% lower fluoride release compared to specimens immersed in water (24, 25). This is explained by a lower diffusion gradient between the material and artificial saliva compared to the diffusion gradient between the material and deionized water. Also, artificial saliva may contain components that form a pellicle on the material surface and thereby interfere with ion release, decreasing it for about 15-20% (1, 25- 27).

na otpuštanje fluoridnih iona. Također su se za sve premazane i nepremazane uzorke pratile promjene pH vrijednosti imerzijskog sredstva. Prema utvrđenom statistički značajnom učinku vrste materijala i premaza na otpuštanje fluoridnih iona i promjene pH, sve nulte hipoteze su odbačene.

Svi materijali koji otpuštaju fluoridne ione korišteni u ovoj studiji pokazali su dugotrajno otpuštanje fluorida, kako je navedeno i u nekoliko ranijih studija (18, 19). Najviše kumulativne vrijednosti otpuštenih fluoridnih iona imali su uzorci FUJ-a, zatim slijede CN i BF. Očekivano, konvencionalni kompozit FIL nije otpuštao fluoridne ione, osim u skupini premazanoj dentinskim adhezijskim sustavom CB koji ima svojstvo otpuštanja fluoridnih iona.

Staklenoionomerne cemente karakterizira početno otpuštanje velike količine fluoridnih iona pod nazivom *burst effect* koji se pojavljuje unutar 24 sata od početka vezivanja cementa (20 – 22). Prema Wiegandu i suradnicima, navedeni učinak rezultat je reakcije između čestica stakla i polialkenoatne kiseline (1).

U ovom istraživanju potvrđen je *burst effect* kod FUJ uzoraka, a i u alkasitnom kompozitnom materijalu CN. No učinak se pojavio samo u uzorcima FUJ i CN pripremljenima bez premaza. Uočen efekt pronađen u CN-u mogao bi biti rezultat njegova sastava. Kako navodi proizvođač, taj materijal sadržava 78,4 % težinskog udjela sljedećih anorganskih punila: barij-aluminijeva silikatnog stakla, iterbijeva trifluorida, Isofillera (patentirano punilo), kalcij-barij-aluminijeva fluorosilikatnog stakla i kalcijeva fluorosilikata (alkalno staklo). Također se 24,6 % težinskoga udjela materijala sastoji od alkalnoga (kalcijeva fluorosilikatnog) staklenog punila odgovornog za otpuštanje fluoridnih, hidroksilnih i kalcijevih iona (14).

Rezultati u ovoj studiji djelomično su u suprotnosti s onima iz studije Gupta i suradnika koji su testirali slične materijale, Cention N (kemijski polimerizirani i svjetlosno polimerizirani) i konvencionalni SIC. Njihovi su rezultati pokazali vremenski ovisno smanjenje otpuštanja fluoridnih iona iz svih ispitivanih materijala, osim SIC-a u kiselom imerzijskom mediju. Ta razlika između otpuštenih fluoridnih iona mogla bi biti rezultat različitih razdoblja mjerenja u spomenutoj studiji (7, 21 i 28 dana). No njihovi rezultati uzoraka SIC-a u neutralnom mediju koji otpuštaju znatno veće količine fluoridnih iona od Centiona N, potvrđuju naše rezultate o otpuštanju fluoridnih iona u tim materijalima (23).

Tiskaya i suradnici proučavali su otpuštanje fluoridnih iona, promjene pH i stvaranje apatita dvaju bioaktivnih kompozita (Cention N i Activa). Njihove kumulativne vrijednosti otpuštanja fluoridnih iona bile su manje od 8 ppm nakon 42 dana, što je niže od vrijednosti dobivenih u našem istraživanju (30,49 ppm nakon 28 dana). Ta razlika može biti posljedica različite geometrije uzorka i različitih imerzijskih medija. U našem istraživanju uzorci su bili pohranjeni u deioniziranoj vodi, a u spomenutom istraživanju korištena je umjetna slina. Uzorci uronjeni u umjetnu slinu pokazuju od 17 do 25 % niže vrijednosti otpuštenih fluoridnih iona u usporedbi s uzorcima uronjenima u deioniziranu vodu (24, 25). Smatra se da je to rezultat nižeg gradijenta difuzije između materija-

The giomer BF did not show the burst effect, which is in correspondence with the study of Yap et al. (28). The slower release of fluoride ions from BF can be attributed to its hydrophobic resinous matrix and a relatively low amount of pre-reacted glass ionomer fillers. Similar to the findings of our study, Mousavinasab et al. have also reported a higher release of fluoride ions from a GIC compared to a giomer. According to their study, the differences in fluoride ions release between those materials could be caused by a greater porosity of GIC, lack of glass ionomer matrix phase, and incorporated resin components in giomers (29).

In our study, the giomer showed the lowest cumulative fluoride release. According to a study by Colceriu Burtea et al., this finding can be explained by material characteristics where giomers include polyacrylic acid from PRG (pre-reacted glass) rather than amino acid modified polyalkenoic acid in the composition of the PRG. They also found that experimental giomers which contained hydrophilic and flexible polymer matrix based on UDMA showed higher cumulative fluoride ions release than giomers based on rigid and hydrophobic dimethacrylates (TEGDMA in BF) (1, 19, 28, 30).

The results of our study showed that the amount of released fluoride ions in all investigated materials was diminished in coated specimens, which is in line with previous studies (7, 11, 12). Surprisingly, the fluoride-releasing adhesive system reduced the amount of leached fluoride ions.

In the present study, the materials coated with GB showed higher fluoride ions release than materials coated with the fluoride-releasing adhesive system CB. This could be explained by material composition. Water sorption depends on material hydrophilicity and can reduce polymer mechanical properties affecting its hygrothermal degradation and polymer hydrolysis that later forms water channels, surface erosions, and crazing that impact material permeability (31-34). High concentrations of acidic monomers contribute to hydrophilicity. GB contains 5-10% phosphoric acid ester monomers, while CB contains hydrophilic amide monomers (35, 36). Resin monomers with ester bonds are highly prone to hydrolysis in the presence of water, which could be one of the reasons for an increased fluoride ions release from materials coated with GB (37, 38). Also, some studies found that resin polarity can act as a major determinant of water uptake. An increase in polarity results in higher water sorption. Polar functional groups include OH- groups, carboxyl groups and phosphate groups, which tend to form hydrogen bonds with water. The water molecules that are „bound" by polar functional groups induce swelling and plasticization of the polymer network (31, 39-41). The GB adhesive system used in this study had dimethacrylate (10–20%) and dimethacrylate components (1-5%). Those components could partially explain an increased amount of released fluoride ions from materials coated with GB.

The hydrophilic resins used in universal adhesive systems are prone to limited monomer conversion due to phase separation which leads to degradation in an aqueous medium (38, 42). Oguri et al. showed that degree of conversion could depend on the functional monomer and photoinitiator system (43). Also, the lack of compatibility between hydropho-

la i umjetne sline u usporedbi s gradijentom difuzije između materijala i deionizirane vode. Umjetna slina također može sadržavati komponente koje tvore pelikulu na površini materijala i tako ometaju otpuštanje iona, smanjujući ga za oko 15 do 20 % (1, 25 – 27).

Giomer BF nije pokazao *burst effect*, što je u skladu sa istraživanjem Yapa i suradnika (28). Sporije otpuštanje fluoridnih iona iz BF-a može se pripisati njegovoj hidrofobnoj smolastoj matrici i razmjerno maloj količini prethodno reagiranih staklenoionomernih čestica punila. Slično našem istraživanju, Mousavinasab i suradnici također su izvijestili o većem otpuštanju fluoridnih iona iz SIC-a u usporedbi s giomerom. Prema njihovu istraživanju, razlike u otpuštanju fluoridnih iona između materijala mogle bi biti prouzročene većom poroznošću SIC-a, nedostatkom matrične faze i ugrađenim komponentama smole u giomere (29).

U našoj studiji giomer je pokazao najniže kumulativno otpuštanje fluoridnih iona, a prema studiji Colceriu Burtea i suradnika te se vrijednosti mogu objasniti karakteristikama materijala u kojima giomeri uključuju poliakrilnu kiselinu iz PRG-a, a ne modificiranu aminokiselinu polialkenoatne kiseline u sastavu PRG-a. Također su otkrili da su eksperimentalni giomeri koji su sadržavali hidrofilnu i fleksibilnu polimernu matricu na bazi UDMA-e pokazivali veće kumulativno otpuštanje fluoridnih iona od giomera na bazi krutih i hidrofobnih dimetakrilata (TEGDMA u BF-u) (1, 19, 28, 30).

Rezultati ovog istraživanja pokazali su da je količina otpuštenih fluoridnih iona u svim ispitivanim materijalima bila niža u premazanim uzorcima, što je u skladu s ranijim studijama (7, 11, 12). Iznenađuje da je dentinski adhezijski sustav koji otpušta fluoridne ione smanjio količinu otpuštenih fluoridnih iona.

U ovom istraživanju su materijali premazani GB-om pokazali veće otpuštanje fluoridnih iona od onih premazanih adhezijskim sustavom CB koji ima svojstvo otpuštanja fluoridnih iona. Dobiveni rezultat mogao bi se objasniti sastavom materijala. Sorpcija vode ovisi o hidrofilnosti materijala i može smanjiti mehanička svojstva polimera tako što utječe na njegovu higrotermalnu razgradnju i hidrolizu polimera koja poslije stvara vodene kanale, površinske erozije i pukotine koje utječu na propusnost materijala (31 – 34). Visoka koncentracija kiselih monomera pridonosi hidrofilnosti, GB sadržava od 5 do 10 % monomera estera fosforne kiseline, a CB pak monomere hidrofilnih amida (35, 36).

Monomeri smole s esterskim vezama vrlo su skloni hidrolizi u prisutnosti vode, što bi mogao biti jedan od razloga za povećano otpuštanje fluoridnih iona iz materijala premazanih adhezijskim sustavom GB (37, 38). Također je u nekim istraživanjima otkriveno da polaritet smole može djelovati kao glavna odrednica sorpcije vode. Što je smola polarnija, to je veća sorpcija vode. Polarne funkcionalne skupine uključuju OH- skupine, karboksilne skupine i fosfatne skupine koje imaju tendenciju stvaranja vodikovih veza s vodom. Molekule vode koje su „vezane" polarnim funkcionalnim skupinama izazivaju bubrenje i plastificiranje polimerne mreže (31, 39 – 41). Dentinski adhezijski sustav GB korišten u ovoj studiji u svojem sastavu ima dimetakrilat (10 – 20 %) i dimetakrilatne komponente (1 – 5 %). Te bi komponente mogle djelomič-

bic photoinitiator and hydrophilic monomers showed lower values of degree of conversion when compared with hydrophilic photoinitiator and monomers. A lower degree of conversion can affect adhesive permeability and lead to acidic monomer diffusion (44-46). CB scientific documentation declares the presence of hydrophobic photoinitiator camphorquinone and hydrophilic amide monomers which could explain lower pH values than pH values in uncoated specimens after 1 hour for BF and FIL, and after 24 hours for CN (35). A lower degree of conversion can also be speculated to have led to lower pH values for GB at time intervals 1 hour and 24 hours.

According to its respective manufacturer, CN shows a buffering ability by releasing acid-neutralizing hydroxide ions. Our results demonstrated small pH changes when CN was exposed to neutral medium, which can be compared with the studies of Gupta et al. and Tiskaya et al. (23, 24). Also, an interesting finding from our study was that the average pH level for CN was higher in the group that was coated with CB compared with the uncoated group. A similar result was present for FIL, but not for BF, and this could be due to increased reactivity when the acidic coat was applied.

FUJ also presented higher pH values when coated with FC. Given that uncoated FUJ specimens tended to decrease the pH value from neutral to acidic over time, the barrier formed in coated specimens showed a lack of that tendency.

GB showed lower pH levels than uncoated specimens and coated with CB. This can be related with a higher concentration of released fluoride ions when compared with materials coated with CB adhesive. The results of similar studies showed that the highest fluoride ions release was found in acidic medium (23, 47-49).

no objasniti povećanu količinu otpuštenih fluoridnih iona iz materijala premazanih GB-om.

Hidrofilne smole koje se koriste u univerzalnim dentinskim adhezijskim sustavima sklone su ograničenoj konverziji monomera zbog razdvajanja faza, što potiče razgradnju u vodenom mediju (38, 42). Oguri i suradnici pokazali su da stupanj konverzije može ovisiti o funkcionalnom monomeru i sustavu fotoinicijatora (43). Nedostatak kompatibilnosti između hidrofobnog fotoinicijatora i hidrofilnih monomera također je pokazao nižu vrijednost stupnja konverzije u usporedbi s hidrofilnim fotoinicijatorom i monomerima. Niži stupanj pretvorbe može utjecati na propusnost adhezijskog sustava i rezultirati difuzijom kiseloga monomera (44 – 46). U znanstvenoj dokumentaciji o CB-u navodi se prisutnost hidrofobnog fotoinicijatora kamforkinona i hidrofilnih amidnih monomera koji bi mogli objasniti niže pH vrijednosti od pH vrijednosti na nepremazanim uzorcima nakon jednog sata za BF i FIL, te nakon 24 sata za CN (35). Također se može pretpostaviti da je niži stupanj konverzije doveo do nižih vrijednosti pH kod uzoraka premazanih GB-om u vremenskim razmacima od jednoga sata i 24 sata.

Prema proizvođaču, CN pokazuje pufersko svojstvo otpuštanjem hidroksidnih iona koji neutraliziraju kiselinu. Naši rezultati pokazali su male promjene pH kada je CN bio izložen neutralnom mediju i mogu se usporediti sa studijama Gupta i suradnika te Tiskaya i suradnika (23, 24). Također je zanimljivo otkriće u našoj studiji bilo da je prosječna razina pH za CN bila viša u skupini premazanoj CB-om negoli u onoj nepremazanoj. Sličan rezultat dobiven je za FIL, ali ne i za BF, a to bi moglo biti zbog povećane reaktivnosti kada se nanese kiseli sloj.

FUJ je također pokazao veće pH vrijednosti kod uzoraka premazanih FC- om. S obzirom na to da su uzorci FUJ-a bez premaza s vremenom smanjivali pH vrijednost s neutralnog na kiseli, barijera nastala u premazanim uzorcima onemogućila je spomenutu pojavu.

GB je pokazao nižu razinu pH vrijednosti od nepremazanih uzoraka i uzoraka premazanih CB-om. Navedeni rezultat može se povezati s većom koncentracijom otpuštenih fluoridnih iona u usporedbi s materijalima premazanima CB adhezivom. U sličnim je istraživanjima istaknuto da se najveće otpuštanje fluoridnih iona događa u kiselom mediju (23, 47 – 49).

Conclusions

The amount of released fluoride ions varied among dental materials and depended on the use of adhesive systems and coatings. The glass ionomer Fuji IX Extra showed the highest values of released fluoride ions followed by the alkasite material Cention and the giomer Beautifil II. Both adhesive systems and the coat had a diminishing effect on released fluoride ions. pH values of the immersion medium differed among materials, treatments and time points. The amount of released fluoride ions showed a growth tendency over time in all tested materials. The lowest pH values were identified in all material specimens coated with G-aenial Bond.

Zaključak

Količina otpuštenih fluoridnih iona varirala je među dentalnim materijalima i ovisila je o upotrebi dentinskih adhezijskih sustava i premaza. Stakleni ionomer Fuji IX Extra pokazao je najveće vrijednosti otpuštenih fluoridnih iona, a slijedili su ga alkasitni materijal Cention i giomer Beautifil II. Dentinski adhezijski sustavi i premaz umanjili su otpuštanje fluoridnih iona. Vrijednosti pH imerzijskog sredstva razlikovale su se između materijala, tretmana i vremenskih točaka. Količina otpuštenih fluoridnih iona pokazala je tendenciju rasta tijekom vremena u svim ispitivanim materijalima. Najniže pH vrijednosti utvrđene su u svim uzorcima materijala premazanima G-aenial Bondom.

Conflict of interest

The authors report no conflict of interest

Sukob interesa

Autori nisu bili u sukobu interesa.

Author's contribution

K.K. - research, data collection, formal analysis, interpretation, visualization, writing - original draft, approval of the final version of the article for publication; **M.P.** - concept and design, formal analysis, statistical analysis, interpretation, writing - critical review and editing, approval of the final version of the article for publication; **K.P.** - concept and design, supervision, interpretation, writing - critical review and editing, approval of the final version of the article for publication; **I.Š.** - concept and design, supervision, interpretation, writing - critical review and editing, approval of the final version of the article for publication; **Z.T.** - supervision, resources, writing - critical review and editing, approval of the final version of the article for publication

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K. K. – istraživanje, prikupljanje podataka, formalna analiza, interpretacija, vizualizacija, pisanje – originalni nacrt, odobravanje krajnje verzije članka za objavljivanje; **M. P.** – koncept i dizajn, formalna analiza, statistička analiza, interpretacija, pisanje – kritički pregled i uređivanje, odobravanje krajnje verzije članka za objavljivanje; **K. P.** – koncept i dizajn, supervizija, interpretacija, pisanje – kritički pregled i uređivanje, odobravanje krajnje verzije članka za objavljivanje; **I. Š.** – koncept i dizajn, supervizija, interpretacija, pisanje – kritički pregled i uređivanje, odobravanje krajnje verzije članka za objavljivanje; **Z. T.** – supervizija, resursi, pisanje – kritički pregled i uređivanje, odobravanje krajnje verzije članka za objavljivanje

Sažetak

Svrha rada: Cilj istraživanja bio je utvrditi učinak dvaju dentinsko adhezijskih sustava i staklenoionomerne smole na količinu otpuštenih fluoridnih iona i promjenu pH tijekom 168 dana. **Materijali i metode:** U istraživanju su korištena četiri restaurativna materijala: giomer Beautifil II, alkasitni kompozitni materijal Cention, konvencionalni kompozit Filtek Z 250 i konvencionalni staklenoionomerni cement Fuji IX Extra. Svjetlosno polimerizirani kompozitni uzorci premazani su G-aenial Bondom i adhezivom Clearfil Universal Bond Quick. Na uzorke sa staklenoionomernim cementom nanesen je premaz GC Fuji Coat LC. Uzorci bez premaza korišteni su kao referencije. Mjerenja količine otpuštenih fluoridnih iona i pH vrijednosti provedena su u sljedećim vremenskim intervalima: 1 sat, 24 sata, 2 dana, 7 dana, 28 dana, 84 dana i 168 dana. **Rezultati:** Kumulativne količine otpuštenih fluoridnih iona poslije 168 dana povećale su se na nepremazanim uzorcima sljedećim redoslijedom: Filtek Z 250 < Beautifil II < Cention < Fuji IX Extra. Kompoziti premazani Clearfil Universal Bond Quickom otpuštali su manju količinu fluoridnih iona od nepremazanih uzoraka, a kumulativne vrijednosti dosegnute nakon 168 dana povećavale su se sljedećim redoslijedom: Filtek Z 250 < Beautifil II < Cention. Kompoziti premazani G-aenial Bondom također su otpuštali manje količine fluoridnih iona u usporedbi s nepremazanim uzorcima – kumulativne vrijednosti povećavale su se sljedećim redoslijedom: Filtek Z 250 < Beautifil II < Cention. Kompoziti premazani G-aenial Bondom u svim su skupinama pokazivali pH vrijednosti u kiselom rasponu (4,4 – 5,7) u intervalima od jednoga i 24 sata. **Zaključak:** Količina otpuštenih fluoridnih iona varirala je među ispitivanim restaurativnim materijalima i ovisila je o upotrebi dentinskog adhezijskog sustava i premaza. Vrijednosti pH varirale su između materijala, tretmana i vremenskih intervala.

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