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Short-Term Fluoride Release from Ion-Releasing Dental Materials

Kratkotrajno oslobađanje fluorida iz dentalnih materijala koji otpuštaju ione

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Abstract

Objective: To compare short-term release of fluoride ions from ion-releasing dental restorative materials. **Material and methods:** Seven experimental groups were prepared using the following six different materials: alkasite (Cention Forte), resin-modified glass ionomer cement (Fuji II LC), bioactive composite (ACTIVA BioACTIVE-RESTORATIVE), fluoride-containing nano-hybrid composite (Luminos UN), coat-free glass hybrid (EQUIA Forte HT), coat-applied glass hybrid (EQUIA Forte HT), and glass ionomer cement (Fuji IX). A total of 40 samples for each group (n=40) were prepared in Teflon molds (8 mm x 2 mm) and placed in polyethylene vials with 5 ml of deionized water. Fluoride release was measured after 6, 24, 48 hours, and after 5 weeks using an ion-selective electrode. The results were expressed in mg/l and the data were statistically analyzed using ANOVA. **Results:** Significant differences in fluoride release were observed within the first 6 hours (ANOVA $p<0.001$). EQUIA Forte HT had the highest release, while the other materials showed no significant differences. After 24 hours, EQUIA Forte HT ($p<0.001$) and Luminos UN ($p<0.05$) exhibited significantly higher releases, compared to other tested materials. EQUIA Forte HT maintained the highest release at 48 hours ($p<0.001$), followed by Cention Forte ($p<0.05$) and Luminos UN ($p<0.05$). All material pairs showed significant differences in fluoride release at 5 weeks ($p<0.001$). **Conclusion:** Coat-free EQUIA Forte HT had the overall highest fluoride release, while Cention Forte demonstrated the greatest increase over time. ACTIVA BioACTIVE-RESTORATIVE exhibited the lowest fluoride release in this study.

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Introduction

Fluoride is one of the best known anti-cariogenic remedies (1). It acts through several mechanisms, most notably by replacing hydroxyl ions in the hydroxyapatite crystal and forming more acid-resistant fluorapatite (2), influencing bacterial metabolism (3), and affecting the formation of biofilm near the restorations (4). Such desirable properties prompted the use of fluoride as an active ingredient in various dental products, in order to prevent caries formation (5).

Glass ionomer cements (GICs) are the most widely used and critically acclaimed fluoride-containing restorative materials (6). They are characterized by their excellent potential for fluoride release, which helps in preventing enamel demineralization, promotes remineralization and reduces plaque growth (6, 7). As cements, GICs set through an acid-base reaction, during which fluoride ions that are not a part of

Uvod

Fluor je jedno od najpoznatijih sredstava za suzbijanje zubnog karijesa (1). Način antikariogenog djelovanja opisan je na temelju nekoliko mehanizama, ponajprije zamjenom hidroksilnih iona u kristalu hidroksiapatita i stvaranjem fluorapatita otpornijeg na kiselinu (2), utjecajem na bakterijski metabolizam (3) i utjecajem na stvaranje biofilma u blizini ispuna (4). Takva poželjna svojstva potaknula su upotrebu fluorida kao aktivnog sastojka u raznim dentalnim proizvodima kako bi se spriječio nastanak karijesa (5).

Staklenoionomerni cementi (GIC) najčešće su korišteni i hvaljeni restaurativni materijali koji sadržavaju fluor (6). Karakterizira ih izvrstan potencijal otpuštanja fluorida koji pomaže u sprječavanju demineralizacije cakline, potiče remineralizaciju i smanjuje rast i formiranje novoga plaka (6, 7). Kao cement GIC-i se vežu kiselo-baznom reakcijom tijekom matrix formation, are released, but can also be absorbed into the cement. Thus, GICs can serve as fluoride reservoirs, keeping the fluoride amount around the tooth relatively stable (8 - 11). Despite their beneficial anti-cariogenic effect, GICs failed to become long-term restorative materials, due to their poor mechanical properties, such as low compressive strength, wear resistance and elastic modulus (12, 13). To overcome those issues, different materials combining preferable features of fluoride-releasing GICs, and mechanically resilient resin-based composites were introduced. Resin-modified glass ionomer cements (RM-GICs) consist of GIC base, with the addition of water-soluble methacrylate monomers, with boosting their mechanical features in mind (14). However, such modifications did not offer the expected results. Therefore, a major improvement of GICs was recently introduced; a glass hybrid (GH). This material is improved by adding more reactive fluoroaluminosilicate (FAS) glass fillers, which are smaller than the main glass fillers, together with higher-molecular weight polyacrylic acid molecules. FAS fillers release more metal ions, improving the crosslinking of polyacrylic acid, which enchases the physical properties (15, 16). Additionally, GHs should be used in combination with self-adhesive resin-based coating material, which further brushes up their properties. An alkasite, often listed as a subgroup of composites, is a recently introduced restorative material. It is capable of releasing fluoride, calcium, and hydroxyl ions, resulting in anti-cariogenic effect (17). Resin-based composite materials, which already possess favorable mechanical and esthetic properties, have been modified by the addition of fluorides and different hybrid versions are nowadays available on the market (18).

In order for fluoride-containing products to be efficient in combating caries development, fluoride ions need to be released (17). Therefore, fluoride release (FR), which is triggered by hydrophilic or ionic environment causing the transport of fluoride ions in and out of the material, is a crucial feature of such products (19). It is influenced by different factors, including the composition and permeability of the material, storage conditions, surface characteristics and curing technique (20 – 22). Although there are numerous commercially available products which are considered to have beneficial effects of fluoride actions, a large number of them are not sufficiently investigated. Considering the importance of FR for achieving the advertised anti-cariogenic effect, the aim of this study was to determine and compare the amount of released fluoride ions from different types of restorative dental materials. The null hypothesis is that there are no differences in FR among the tested materials.

Material and methods

This study was performed using protocols approved by the Ethics Committee of the University of Zagreb, School of Dental Medicine, approval number 05-PA-4-7-X-I-1/2020.

Six commercially available fluoride-containing restorative dental materials were used in this study. They are listed in Table 1 along with their composition provided by the manufacturers.

koje se oslobađaju ioni fluorida koji nisu dio formiranja matriksa, ali se također mogu apsorbirati u cement. Zato mogu poslužiti kao spremnik fluorida, održavajući količinu fluorida oko zuba razmjerno stabilnom (8 – 11). Unatoč njihovu blagotvornom antikariogenom učinku, GIC-i nisu uspjeli postati dugotrajni restaurativni materijal zbog loših mehaničkih svojstava kao što su niska tlačna čvrstoća, otpornost na trošenje i modul elastičnosti (12, 13). Kako bi se prevladali ti problemi, uvedeni su različiti materijali koji kombiniraju poželjna svojsta GIC-a koji otpuštaju fluor i mehanički otpornih kompozita na bazi smole. Staklenoionomerni cementi modificirani smolom (RM-GIC) sastoje se od GIC baze uz dodatak vodotopivih metakrilatnih monomera sa svrhom da se poboljšaju njihova mehanička svojstva (14). No takve promjene nisu dale očekivane rezultate. Zato je nedavno predstavljeno značajno poboljšanje GIC-ova – staklohibridi (GH). Taj je materijal poboljšan dodavanjem više reaktivnih fluoroaluminosilikatnih (FAS) staklenih punila koja su manja od glavnih staklenih punila, zajedno s molekulama poliakrilne kiseline veće molekularne težine. FAS punila oslobađaju više metalnih iona i poboljšavaju umrežavanje poliakrilne kiseline, čime se pojačavaju fizikalna svojstva (15, 16). Dodatno, GH-i bi se trebali koristiti u kombinaciji sa samoljepljivim premaznim materijalom na bazi smole koji dodatno poboljšava njihova svojstva. Alkazit, često naveden kao podskupina kompozita, nedavno je uveden kao restauracijski materijal. Svojstvo mu je otpuštanje iona fluorida, kalcija i hidroksila, što rezultira antikariogenim učinkom (17). Kompozitni materijali na bazi smola, koji već imaju povoljna mehanička i estetska svojstva, modificirani su dodatkom fluorida te su danas na tržištu dostupne različite hibridne inačice (18).

Da bi proizvodi koji sadržavaju fluor bili učinkoviti u borbi protiv karijesa, potrebno je otpuštanje iona fluorida (17). Zato je otpuštanje fluorida (FR), koje je potaknuto hidrofilnom ili ionskom okolinom koja uzrokuje transport fluoridnih iona u materijal i izvan njega, ključna značajka takvih proizvoda (19). Na njega utječu različiti čimbenici, uključujući sastav i propusnost materijala, uvjete skladištenja, karakteristike površine i tehniku stvrdnjavanja (20 – 22). Kao i broj komercijalno dostupnih proizvoda za koje se tvrdi da imaju korisne učinke djelovanja fluorida, mnogi od njih nisu dovoljno istraženi. S obzirom na važnost FR-a za postizanje oglašenoga antikariogenoga učinka, cilj ovog istraživanja bio je utvrditi i usporediti količinu otpuštenih fluoridnih iona iz različitih vrsta restaurativnih dentalnih materijala. Nulta hipoteza glasila je da neće biti razlika u FR-u među ispitanim materijalima.

Materijal i metode

Istraživanje je provedeno prema protokolima koje je odobrilo Etičko povjerenstvo Stomatološkog fakulteta Sveučilišta u Zagrebu (broj odobrenja 05-PA-4-7-X-I-1/2020.).

U ovoj studiji korišteno je šest komercijalno dostupnih restaurativnih stomatoloških materijala koji sadržavaju fluor. Uz sastav, koji navode proizvođači, navedeni su u tablici 1.

Table 1 Type and composition of the materials used in the study.
Tablica 1. Vrste i sastavi materijala korištenih u istraživanju

Materials • Materijal	Type • Vrsta	Composition • Sastav	Manufacturer • Proizvođač
Luminos UN	Fluoride-containing nano hybrid composite • Nanohibridni kompozit s fluorom	Bis-GMA/TEGDMA resin, multifunctional filler (including micronized fluoroboroaluminosilicate glass) • Bis-GMA/TEGDMA, multifunkcionalna punila (uključujući mikronizirano fluorboroalumosilikatno staklo)	UnoDent Ltd, Witham, United Kingdom • Ujedinjeno Kraljevstvo
Cention Forte	Alkasite (Resin composite with reactive glass fillers) • Alkazit (smolasti kompozit s reaktivnim česticama stakla)	UDMA, DCP, Aromatic aliphatic-UDMA, PEG-400 DMA Barium aluminium silicate glass, Ytterbium trifluoride, Isofiller, Calcium barium aluminium fluorosilicate glass, calciumfluoro silicate glass •	Ivoclar Vivadent, Schaan, Liechtenstein • Lihtenštajn
Fuji IX GP Extra (Fuji IX)	Conventional glass ionomer material • Konvencionalni staklenoionomerni cement	Liquid Distilled water, Polyacrylic acid, Powder: Fluoroaluminosilicate glass. •	GC Corporation, Tokyo, Japan
ACTIVA BioACTIVE-RESTORATIVE (Activa)	Bioactive composite • Bioaktivni kompozit	Blend of diurethane and other methacrylates with modified polyacrylic acid, silica, amorphous, sodium fluoride •	Pulpdent Corporation, Watertown, MA, USA • SAD
Fuji II LC	Resin modified glass ionomer material • Smolom modificirani staklenoionomerni cement	Fluroaluminisilicate glass/ Liquid destiled water, polyacrylic acid, HEMA, UDMA, Comphorquinone •	GC Corporation, Tokyo, Japan
EQUIA Forte HT, without coat	Glass hybrid • Staklohibridni materijal	Powder: fluoroaluminosilicate glass, polyacrylic acid, iron oxide Liquid: polybasic carboxylic acid, water •	GC Corporation, Tokyo, Japan
EQUIA Forte HT, with coat	Glass hybrid, covered with resin-based coat • Staklohibridni material sa zaštitnim smolastim premazom	Coat: silica fillers, multifunctional monomers. Powder and liquid same as EQUIA Forte HT. •	GC Corporation, Tokyo, Japan

Specimen preparation

Disc-shaped specimens of each material were prepared using Teflon molds (8 mm wide, 2 mm thick). For each tested material, 40 samples were made (n=40). All tested materials, except for Luminos UN and Activa, were in encapsulated form and were mixed according to the manufacturer's instructions using Silver Mix capsule mixer (GC Corporation Tokyo, Japan). To avoid any air trapping, polyester strips were placed on both sides of the molds, with material being gently compressed using glass plates. Light curable materials (Luminos UN, Activa, Fuji II LC, Cention Forte) were cured for 40 seconds using the light cure unit Woodpecker LED-C (Guilin Tucano Medical Apparatus and Instruments Limited Company, Guilin, China), curing light output: 850 W/cm^2 wavelength: 420 nm-480 nm. The rest of the samples made from self-curing materials were left to set for 1 hour before taking them out of the moulds. In addition, the samples for coated EQUIA Forte HT were covered with coat after material setting. The coat was applied for 10 seconds using a brush, air-dried for 5 seconds and light-cured for 10 seconds.

Priprema uzoraka

Uzorci od svakog materijala u obliku diska pripremljeni su s pomoću teflonskih kalupa (širina 8 mm, debljina 2 mm). Za svaki ispitivani materijal napravljeno je 40 uzoraka (n = 40). Svi ispitani materijali, osim Luminos UN-a i Activa, bili su u kapsuliranom obliku i miješani su prema uputama proizvođača korištenjem Silver Mix miksera za kapsule (GC Corporation, Tokio, Japan). Kako bi se izbjeglo zadržavanje zraka, trake od poliestera postavljene su na obje strane kalupa, a materijal je lagano potisnut s pomoću staklenih pločica. Materijali koji se polimeriziraju svjetlom (Luminos UN, Activa, Fuji II LC, Cention Forte) polimerizirani su 40 sekunda svjetiljkom Woodpecker LED-C (Guilin Tucano Medical Apparatus and Instruments Limited Company, Guilin, Kina). Snaga polimerizirajućeg svjetla bila je: 850 W/cm^2, valna duljina: 420 nm do 480 nm. Ostali uzorci izrađeni od samostvrdnjavajućih materijala ostavljeni su da se stvrdnu 1 sat prije vađenja iz kalupa. Osim toga, uzorci materijala EQUIA Forte HT s premazom prekriveni su premazom nakon stvrdnjavanja materijala. Sloj je nanošen 10 sekunda četkicom, sušen na zraku 5 sekunda i polimeriziran svjetlom 10 sekunda.

Fluoride release measurement

The samples were individually placed in polyethylene vials (12 mm x 38 mm) (Laboroprema, Zagreb, Croatia) and 5 ml of deionized water was added. They were then left to rest at 37°C in a cooled incubator ES 120 (NÜVE, Ankara, Turkey) for 6 hours. The concentrations of fluoride released into the water were measured after 6 h, 24 h, 48 h and 5 weeks. Deionized water was replaced after every testing procedure. For the measurements, each disk was removed from the water, dried on filter paper and immediately immersed in 5 mL fresh deionized water for further measurements. The fluoride concentrations in the water samples were measured using an ionoselective electrode (F800 DIN, Xylem Analytics Germany, Weilheim, Germany) connected to an ion analyzer (inoLab Multi 9630 DS; Xylem Analytics Germany, Weilheim, Germany). The ion selective electrode was prepared for analysis using WTW outer chamber filling solution ELY/BR/503. The ISE was then calibrated using standards of 50, 100 and 200 mg/l. Once the standard checks had passed quality control, the samples were analyzed by removing the sample and adding 5 ml of TISAB II solution (Total Ionic Strength Adjustment Buffer; Merck KGaA, Darmstadt, Germany). FR from each sample was measured three times and expressed in mg/L (ppm F–).

Statistical analysis

Cumulative values for individual time points were compared using analysis of variance with Scheffe post-hoc test, and differences in release rate were analyzed using one-way analysis of variance with one dependent measurement (release rate per time point) and one independent (material). The significance level was set at 0.05, and the analysis was conducted using SPSS for Windows 22.0 (IBM, Armonk, NY, USA).

Results

The results of fluoride release measurement are shown in Table 2.

The values expressed in Table 2 represent the arithmetic mean of three measurements, specifically for each material and time point. The data for the time points were given cumulatively. For methodological reasons, samples the values of which deviated by more than 2.5 standard deviations from the distribution average were excluded from the analy-

Mjerenje oslobađanja fluorida

Uzorci su pojedinačno stavljeni u polietilenske bočice (12 mm x 38 mm) (Laboroprema, Zagreb, Hrvatska) te je dodano 5 mL deionizirane vode. Uzorci su zatim ostavljeni šest sati u inkubatoru ES 120 (NÜVE, Ankara, Turska) na 37 °C. Koncentracije fluorida ispuštene u vodu mjerene su poslije 6, 24 i 48 sati te 5 tjedana. Deionizirana voda zamijenjena je poslije svakog mjerenja. Za mjerenja je svaki disk izvađen iz vode, osušen na filtar-papiru i odmah uronjen u 5 mL svježe deionizirane vode za daljnja mjerenja. Koncentracije fluorida u uzorcima vode mjerene su s pomoću ionoselektivne elektrode (ISE) (F800 DIN, Xylem Analytics Njemačka, Weilheim, Njemačka) spojene na ionski analizator (inoLab Multi 9630 DS; Xylem Analytics Njemačka, Weilheim, Njemačka). ISE je pripremljena za analizu korištenjem WTW otopine za punjenje vanjske komore ELY/BR/503 te je zatim kalibrirana korištenjem standarda od 50, 100 i 200 mg/L. Nakon što su standardne provjere prošle kontrolu kvalitete, uzorci su analizirani uklanjanjem uzorka i dodavanjem 5 mL otopine TISAB II (Total Ionic Strength Adjustment Buffer; Merck KGaA, Darmstadt, Njemačka). Oslobađanje fluorida iz svakog uzorka izmjereno je tri puta i izraženo u mg/L (ppm F–).

Statistička analiza

Kumulativne vrijednosti za pojedinačne vremenske točke uspoređene su s pomoću analize varijance Scheffeovim post-hoc testom, a razlike u stopi otpuštanja analizirane su s pomoću jednosmjerne analize varijance s jednim ovisnim mjerenjem (stopa otpuštanja po vremenskoj točki) i jednim neovisnim (za svaki materijal). Razina značajnosti postavljena je na 0,05, a analiza je provedena u SPSS-u za Windows 22.0 (IBM, Armonk, NY, SAD).

Rezultati

Rezultati mjerenja oslobađanja fluorida prikazani su u tablici 2.

Vrijednosti izražene u tablici 2. aritmetička je sredina triju mjerenja, posebno za svaki materijal i vremensku točku. Podatci za vremenske točke dani su kumulativno. Iz metodoloških razloga iz analize su isključeni uzorci čije vrijednosti odstupaju više od 2,5 standardne devijacije od prosjeka distribucije. Na taj način eliminirano je ukupno 17 uzoraka: Ac-

Table 2 Fluoride release (mg/l) and SD values after 6 hours, 24 hours, 48 hours and 5 weeks ($p<0.001$).
Tablica 2. Oslobađanje fluorida (mg/L) i SD vrijednosti poslije 6 sati, 24 sata, 48 sati i 5 tjedana ($p < 0,001$).

	6 hours • 6 sati	24 hours • 24 sata	48 hours • 48 sati	5 weeks • 5 tjedana
Activa	0.0027±0.0019	0.0042±0.0019	0.0055±0.0022	0.8706±0.0969
Cention Forte	0.0234±0.0203	0.4412±0.1929	9.2491±2.5479	12.6301±2.6305
EQUIA Forte HT	8.0410±3.4183	11.4452±4.0049	13.0840±4.1424	19.5236±4.1696
EQUIA Forte HT with coating • s premazom	0.7451±0.7158	0.7699±0.7267	0.7860±0.7331	4.9574±1.1639
Fuji II LC	0.1977±0.0901	0.2154±0.0904	0.23340±0.0917	1.8351±0.5204
Fuji IX	0.7657±0.0842	1.1433±0.1265	1.2887±0.1458	9.6002±0.8396
Luminos UN	0.5653±0.1704	1.8870±0.4938	3.0663±1.5647	3.1369±1.5658

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sis. A total of 17 samples were eliminated in this way: Activa (2 samples), Cention Forte (2 samples), EQUIA Forte HT without coat (2 samples), EQUIA Forte HT with coat (4 samples), Luminos UN (3 samples), Fuji II LC (3 samples) and Fuji IX (1 sample).

In the first 6 hours, there was a statistically significant difference in the amount of fluoride release between the tested materials (ANOVA p<0.001). EQUIA Forte HT scored significantly higher than the other materials, all of which do not differ significantly, compared to each other. After 24 hours, there was again a statistically significant difference in fluoride among the materials (p<0.001). This time, both EQUIA Forte HT (p<0.001) and Luminos UN (p<0.05) had statistically significantly higher score than other tested materials. In the first 48 hours, EQUIA Forte HT again scored statistically significantly higher than other materials (p<0.001), followed by Cention Forte (p<0.05) and Luminos UN (p<0.05), with other materials having statistically significantly lower values. In 5 weeks, there were statistically significant differences in the amount of fluoride released between all pairs of materials (p<0.001).

The rate of change of release was different among materials (repeated measures ANOVA for concentration variables per hour, p<0.001). The release rate of EQUIA Forte HT decreased over time, while for Cention Forte it increased between 24h and 48h, and again decreased in 48h and 5 weeks. The rates for coated EQUIA Forte HT, Fuji IX and Luminos UN differed from the other materials, but not from each other; in all three cases there was a slight continuous decrease of fluoride release rates. Hourly release rates are shown in Figure 1.

tiva (2 uzorka), Cention Forte (2 uzorka), EQUIA Forte HT bez premaza (2 uzorka), EQUIA Forte HT s premazom (4 uzorka), Luminos UN (3 uzorka), Fuji II LC (3 uzorka) i Fuji IX (1 uzorak).

U prvih 6 sati zabilježena je statistički značajna razlika u količini oslobođenih fluorida između ispitivanih materijala (ANOVA p < 0,001). EQUIA Forte HT postigao je znatno bolji rezultat od ostalih materijala koji se uzajamno ne razlikuju značajno. Poslije 24 sata ponovno je uočena statistički značajna razlika u količini oslobođenoga fluora između materijala (p < 0,001). Taj put su EQUIA Forte HT (p < 0,001) i Luminos UN (p < 0,05) imali statistički značajno viši rezultat od ostalih testiranih materijala. U prvih 48 sati EQUIA Forte HT ponovno je postigao statistički značajno više rezultate od ostalih materijala (p < 0,001), a slijede ga Cention Forte (p < 0,05) i Luminos UN (p < 0,05). Ostali materijali imali su statistički značajno niže vrijednosti. U 5 tjedana zabilježene su statistički značajne razlike u količini oslobođenih fluorida između svih parova materijala (p < 0,001).

Brzina promjene otpuštanja među materijalima je različita (ANOVA test po satu, p < 0,001). Brzina otpuštanja EQUIA Forte HT-a smanjuje se s vremenom, a za Cention Forte povećava se između 24 i 48 sati te ponovno smanjuje između 48 sati i 5 tjedana. Vrijednosti za EQUIA Forte HT s premazom, Fuji IX i Luminos UN razlikuju se od ostalih materijala, ali ne jedan od drugoga; u svim trima slučajevima postoji lagano kontinuirano smanjenje brzine otpuštanja fluorida. Stope oslobađanja po satu prikazane su na Slici 1.

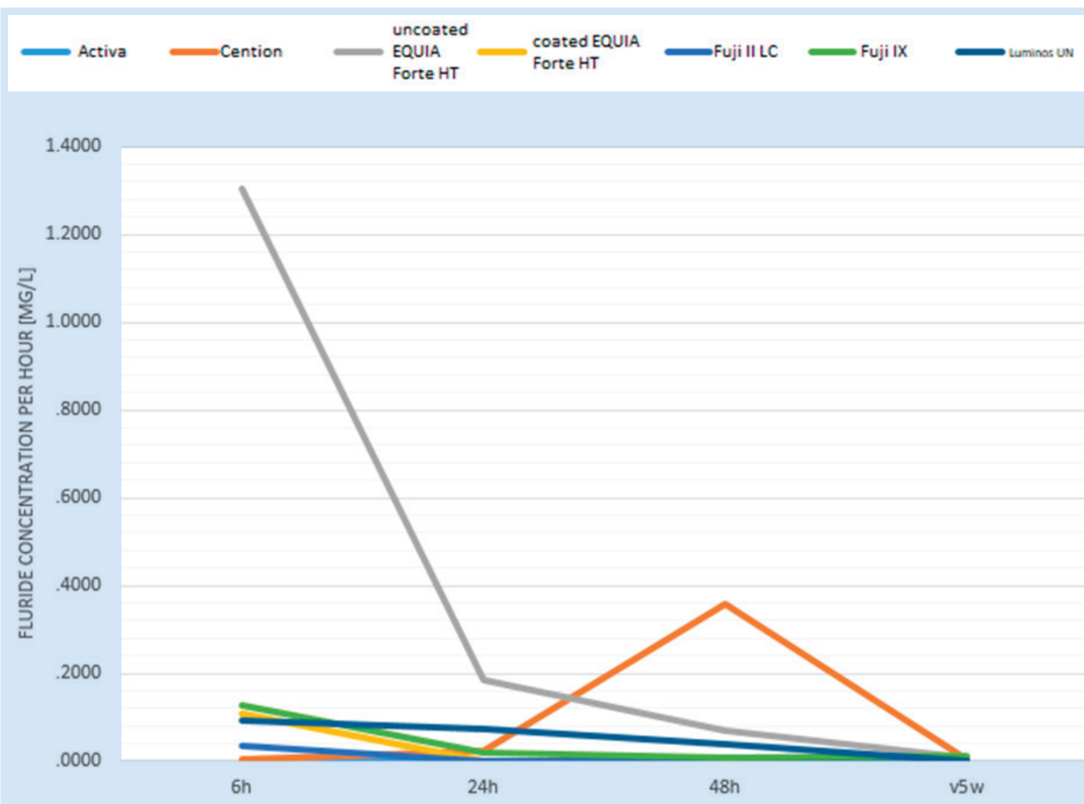

Figure 1 Hourly fluoride release rates (mg/l).

Slika 1. Otpuštanje fluorida po satu (mg/L).

Discussion

The results of this study showed that uncoated EQUIA Forte HT released the highest amount of fluorides among the tested materials in all testing periods, thus, the null-hypothesis was rejected. In addition, it was shown that FR rates of the materials varied greatly compared to each other, owing to differences in their composition.

The reason why uncoated EQUIA Forte HT, a glass hybrid material, outperformed other materials lies in its composition, as glass hybrids developed from glass ionomer cements, with several significant modifications (23). The initial 'burst effect', which is characterized by releasing high amounts of fluorides in the first 24 hours, is a well-known property of glass ionomers (24), also observed in glass hybrids. It is one of their biggest advantages, as it helps neutralizing the bacteria and promotes dentine remineralization (25). One of glass hybrid improvements is the replacement of Ca^{2+} with Sr^{2+} ions; this enhanced the fluoride release since strontium fluoride complex dissolves faster than calcium fluoride complex (26). However, it is interesting to notice that the same material, this time coated with the recommended EQUIA Coat, released much lower amounts of fluorides. This can be attributed to the fact that this type of coat is resin-based and contains nanofillers, which obstructs the gaps in the material and protects it, thus effectively reducing the ion release (27). These findings are also supported by the research of Tiwari et al. (28) and McKnight-Hanes et al. (29) who also concluded that coat application significantly decreases the fluoride release. However, the introduction of the coating brings about a notable enhancement in the mechanical properties of glass hybrids. It is important to emphasize that the improvement in mechanical properties was, in fact, the primary reason behind the application of the coating in the first place. Yet, certain volume of fluoride release is still retained. The amalgamation of the aforementioned effects highlights the multifaceted benefits that arise from the utilization of the coating. Not only does it sustain the desirable anti-cariogenic properties associated with fluoride, but it also serves as a catalyst for reinforcing the mechanical characteristics of glass hybrids. This symbiotic relationship between the coating and the material not only enhances their overall performance, but also extends their potential applications in diverse fields (30).

In the current study, during the early testing periods, some of the light cured materials, namely Activa, Fuji II LC, Cention Forte and coated EQUIA Forte HT, released somewhat lower amount of fluorides than self-cured glass hybrid and glass ionomer cement. The main contributing factor to this result is the effect of the curing method; previous research has shown that the initiation of polymerization through light exposure enhances the formation of chemical bonds, leading to an increased density of cross-linking. As a result, the permeability of the resin matrix towards fluoride ions is ultimately decreased (31, 32). While composites continued to release relatively low amounts of fluorides, Cention Forte, an alkasite material, outperformed all other tested material after 5 weeks. Singbal et al. reported similar findings (33). Initial low rates of fluoride release observed in Cention Forte could

Rasprava

Rezultati ove studije pokazali su da EQUIA Forte HT bez premaza oslobađa najveću količinu fluorida među testiranim materijalima u svim razdobljima ispitivanja i zato je nulta hipoteza odbačena. Osim toga, pokazalo se da se stope FR materijala uzajamno veoma razlikuju zbog razlika u sastavu.

Razlog zašto je EQUIA Forte HT bez premaza, staklohibridni materijal, nadmašio druge materijale, jest u njegovu sastavu jer su se staklohibridi razvili iz staklenoionomernih cemenata, uz nekoliko značajnih modifikacija (23). Početni 'burst efect', koji karakterizira otpuštanje velikih količina fluorida u prva 24 sata, dobro je poznato svojstvo staklenoionomera (24) koje je također uočeno kod staklohibrida. To im je jedna od najvećih prednosti jer pomaže neutralizirati bakterije i potiče remineralizaciju dentina (25). Jedno od poboljšanja staklohibrida jest zamjena Ca2+ ionima Sr2+/Ca^{2+} ionima SR $^{2+}$; to je povećalo otpuštanje fluorida zato što se kompleks stroncijeva fluorida otapa brže od kompleksa kalcijeva fluorida (26). No zanimljivo je primijetiti da je isti materijal, ovaj put premazan preporučenim EQUIA Coat premazom, otpuštao znatno manje količine fluorida. To se može pripisati činjenici da je ta vrsta premaza na bazi smole i sadržava nanopunila koja začepljuju praznine u materijalu i štite ga učinkovito smanjujući otpuštanje iona (27). Te rezultate podupire i istraživanje Tiwarija i suradnika (28) te McKnight-Hanesa i suradnika. (29) koji su također zaključili da nanošenje premaza značajno smanjuje otpuštanje fluorida. No, uvođenje premaza znatno poboljšava mehanička svojstava staklenih hibrida. Važno je istaknuti da je poboljšanje mehaničkih svojstava zapravo bio primarni razlog za primjenu premaza. Ipak, određeni volumen otpuštanja fluorida još je uvijek zadržan. Kombinacija gore navedenih učinaka ističe višestruke prednosti koje proizlaze iz upotrebe premaza. Ne samo da održava poželjna antikariogena svojstva povezana s fluoridom, nego služi i kao katalizator za jačanje mehaničkih svojstava staklohibrida. Taj simbiotski odnos između premaza i materijala ne samo da poboljšava njihovu ukupnu izvedbu, nego također proširuje njihovu potencijalnu primjenu u različitim područjima (30).

U ovome istraživanju, tijekom ranog razdoblja testiranja, neki od svjetlosno polimerizirajućih materijala, najviše Activa, Fuji II LC, Cention Forte i EQUIA Forte HT s premazom, otpuštali su nešto nižu količinu fluorida od samostvrdnjavajućih staklenih hibrida i staklenoionomernog cementa. Glavni čimbenik koji pridonosi tom rezultatu jest način stvrdnjavanja; prethodna istraživanja pokazala su da početak polimerizacije izlaganjem svjetlu pospješuje stvaranje kemijskih veza, što povećava gustoću križnog povezivanja. Kao rezultat toga u konačnici je smanjena propusnost matrice smole prema fluoridnim ionima (31, 32). Dok su kompoziti nastavili otpuštati razmjerno niske količine fluorida, alkalni materijal Cention Forte nadmašio je sve ostale ispitivane materijale nakon 5 tjedana. Singbal i suradnici objavili su slične nalaze (33). Početne niske stope otpuštanja fluorida uočene u Cention Forteu mogu se pripisati činjenici da punila u tom materijalu prolaze površinsku modifikaciju, što rezultira povećanom otpornošću na razgradnju i potencijalno rezultira be attributed to the fact that the fillers present in this material undergo surface modification, resulting in increased resistance to degradation and potentially leading to a release of a reduced quantity of fluoride ions (7). This suggests that there is a certain period of time needed for the material matrix to be maturated enough to release fluorides.

In the current study, Activa, a composite material, released the lowest amounts of fluorides in all testing periods. Similar findings were reported by Rifai et al. (34), who compared Activa to EQUIA Forte HT, concluding that glass hybrid material outperforms composite materials. In addition, Hokii *et al.* (35) compared the fluoride release from six different restorative materials. They also concluded that Activa releases lower amounts of fluorides compared to glass hybrids and glass ionomers. In contrast to our study, Activa released a significant amount of fluoride ions in the study conducted by Vicente *et al.* (36). The explanation for this is the exposure of the material to acidic pH of 3, 5 environments in their study, leading to a significant increase in ion release rate in certain measuring periods, meaning that Activa expresses anti-cariogenic properties in caries challenging conditions.

Luminos UN composite performed better than Activa. Yet, it was inferior to other materials. The explanation for this could lie in the fact that the water uptake/dissolution process in composite resins is less efficient in delivering fluoride compared to mechanisms observed in glass-ionomer-based materials (37). Alternatively, it could be indicative of lower fluoride concentrations in the composite formulation.

To simulate the oral environment, other studies tested the materials in various storage mediums, for instance artificial saliva or different types of acids, with different pH cycling solutions (38). However, for the purposes of the current study, deionized water was chosen as the storage medium. This selection was made because deionized water is free from fluoride traces or minerals, thus allowing for an accurate measurement of fluoride ion release. This was further considered sufficient, as it has been demonstrated that fully replicating oral conditions are exceedingly challenging due to various factors, such as the rate of saliva flow and individual habit variations (39).

While these studies offer valuable preliminary data, it is essential to recognize that the outcomes may be influenced by numerous factors such as the specific protocols employed, the concentrations of the bioactive materials tested, and the environmental conditions in which the experiments took place, therefore, experimental conditions under which they were conducted should be considered. In addition, this study investigated solely the fluoride release, while other ions, most notably calcium ions, also play a significant role in the remineralization process. By conducting additional studies, researchers can delve deeper into the mechanisms underlying fluoride ion release from these dental materials, exploring their long-term release patterns and their ability to provide sustained anti-cariogenic benefits. Moreover, investigating the potential of different coating techniques or modifications to enhance the fluoride ion release of EQUIA Forte HT and other materials could lead to the development of more effective preventive strategies in the field of dentistry. Further-

otpuštanjem smanjene količine fluoridnih iona (7). To sugerira da je potrebno određeno razdoblje da matrica materijala dovoljno sazrije i otpusti fluoride.

U ovome istraživanju je kompozitni materijal Activa ispustio najmanju količinu fluorida u svim razdobljima testiranja. O sličnom rezultatu izvijestili su Rifai i suradnici (34) koji su Activu usporedili s EQUIA Forte HT-om te zaključili da je stakleni hibrid bolji materijal od kompozitnih. Uz to, Hokii i suradnici (35) usporedili su otpuštanje fluorida iz šest različitih restaurativnih materijala te također zaključili da Activa oslobađa manje količine fluorida u usporedbi sa staklenim hibridima i staklenoionomerima. Suprotno našem istraživanju, u studiji Vicentea i suradnika (36), Activa je oslobodila značajnu količinu fluoridnih iona. Objašnjenje za to je izlaganje materijala kiseloj pH okolini od 3,5 u njihovoj studiji, što je znatno povećalo stopu otpuštanja iona u određenim razdobljima mjerenja, a to znači da Activa izražava antikariogena svojstva u stanjima izazovnima za karijes.

Kompozit Luminos UN pokazao se boljim od Active, ali je inferioran u odnosu prema drugim materijalima. Objašnjenje za to mogla bi biti činjenica da je proces unosa/otapanja vode u kompozitnim smolama manje učinkovit u otpuštanju fluorida u usporedbi s mehanizmima uočenima u materijalima na bazi staklenoionomera (37). Alternativno bi to mogao biti pokazatelj nižih koncentracija fluorida u kompozitnoj formulaciji.

Za simulaciju oralnog okruženja u drugim studijama materijali su testirani u različitim medijima za pohranjivanje, na primjer, umjetna slina ili različite vrste kiselina, s različitim pH otopinama (38). Međutim, za potrebe ove studije kao medij za pohranu odabrana je deionizirana voda. To je učinjeno zato što ne sadržava tragove fluorida ili minerala, što omogućuje točno mjerenje otpuštanja iona fluorida. To se nadalje smatralo dovoljnim jer je pokazano da je potpuna replikacija oralnih stanja iznimno zahtjevna zbog različitih čimbenika, kao što su brzina protoka sline i individualne varijacije navika (39).

Iako ovo istraživanje nudi vrijedne preliminarne podatke, bitno je prepoznati da na rezultate mogu utjecati mnogobrojni čimbenici kao što su specifični korišteni protokoli, koncentracije testiranih bioaktivnih materijala i okolišni uvjeti u kojima su se eksperimenti obavljali, te zato treba uzeti u obzir eksperimentalne uvjete u kojima su provedeni. Uz to, u ovoj je studiji istraživano samo otpuštanje fluorida, a drugi ioni, ponajprije ioni kalcija, također imaju značajnu ulogu u procesu remineralizacije. Provođenjem dodatnih studija istraživači mogu detaljnije istražiti mehanizme koji se nalaze u osnovi otpuštanja fluoridnih iona iz ovih stomatoloških materijala, istražujući njihove dugotrajne obrasce otpuštanja i njihovu sposobnost da omoguće trajne antikarijesne prednosti. Štoviše, istraživanje potencijala različitih tehnika premazivanja ili modifikacija za povećanje otpuštanja fluoridnih iona EQUIA Forte HT-a i drugih materijala moglo bi potaknuti razvoj učinkovitijih preventivnih strategija u području stomatologije. Nadalje, bilo bi vrijedno istražiti korelaciju između oslobođenih koncentracija fluoridnih iona i stvarnih kliničkih ishoda kad je riječ o prevenciji karijesa. Utvrđivanje učinkovitosti tih materijala u scenarijima stvarnoga svijemore, it would be valuable to explore the correlation between the released fluoride ion concentrations and the actual clinical outcomes in terms of caries prevention. Determining the effectiveness of these materials in real-world scenarios and evaluating their long-term clinical performance could provide a more comprehensive understanding of their practical utility and help guide clinicians in selecting the most appropriate materials for their patients.

Conclusions

Based on the findings of this study, we can draw several significant conclusions regarding the fluoride ion release characteristics of various dental materials. Specifically, the uncoated EQUIA Forte HT, a glass hybrid material, demonstrated a notable advantage in terms of releasing a higher concentration of fluoride ions within a short-time period compared to other materials examined in this investigation. This observation highlights the potential of EQUIA Forte HT to effectively contribute to the prevention of dental caries by delivering a substantial amount of fluorides. All other tested materials exhibited some degree of fluoride ion release, which could possibly aid in preventing the formation and progression of dental cavities. However, further research is needed to comprehensively validate anti-cariogenic effects of these materials, especially in clinical conditions.

Conflict of interest

The authors declare no conflict of interest.

Ethics statement

The study was performed in accordance with the declaration of Helsinki and was approved by the Ethics Committee of the University of Zagreb, School of Dental Medicine.

Author's contributions: **L.S.B.V.-** organized and conducted the research, **N.N.V.-** organized and conducted the research, **I.Š.-** wrote the manuscript, contributed substantially to discussion, **E.K.-** proofread the manuscript, **I.M.-** proofread the manuscript, secured financing, **S.J.K.-** participated in experimental design, proofread the manuscript, contributed substantially to discussion

ta i procjena njihove dugoročne kliničke učinkovitosti mogli bi omogućiti sveobuhvatnije razumijevanje njihove praktične korisnosti i pomoći kliničarima u odabiru najprikladnijih materijala za njihove pacijente.

Zaključak

Prema rezultatima ove studije možemo izvući nekoliko značajnih zaključaka u vezi sa značajkama otpuštanja fluoridnih iona iz različitih dentalnih materijala. Konkretno, staklohibridni materijal EQUIA Forte HT bez zaštitnoga premaza pokazao je značajnu prednost u otpuštanju veće koncentracije fluoridnih iona u kratkom razdoblju, u usporedbi s drugim materijalima testiranima u ovom istraživanju. To zapažanje ističe potencijal EQUIA Forte HT-a da učinkovito pridonese prevenciji zubnog karijesa otpuštanjem znatne količine fluorida. Svi ostali ispitani materijali pokazali su određeni stupanj otpuštanja fluoridnih iona, što bi moglo pomoći u sprječavanju stvaranja i napredovanja zubnog karijesa. No potrebna su daljnja istraživanja kako bi se sveobuhvatno potvrdili protukarijesni učinci tih materijala, posebno u kliničkim uvjetima.

Sukob interesa

Autori nisu bili u sukobu interesa.

Etička izjava

Studija je provedena u skladu s Helsinškom deklaracijom i odobrilo ju je Etičko povjerenstvo Stomatološkog fakulteta Sveučilišta u Zagrebu.

Doprinos autora: **L. S. B. V.** – provedba istraživanja; **N. N. V.** – provedba istraživanja ; **I. Š.** – priprema i pisanje teksta, znatan doprinos raspravi; **E. K.** – uređivanje teksta; **I. M.** – uređivanje teksta, financije; **S. J. K.** – dizajn studije, uređivanje teksta, znatan doprinos raspravi

Sažetak

Svrha istraživanja: Željelo se usporediti kratkotrajno oslobađanje fluoridnih iona iz dentalnih restaurativnih materijala koji otpuštaju ione. **Materijali i metode**: Pripremljeno je sedam eksperimentalnih skupina. Ispitano je šest različitih materijala: alkazit (Cention Forte), staklenoionomerni cement modificiran smolom (Fuji II LC), bioaktivni kompozit (ACTIVA BioACTIVE-RESTORATIVE), nanohibridni kompozit koji sadržava fluor (Luminos UN), staklohibridni materijal bez premaza (EQUIA Forte HT), staklohibridni materijal s premazom (EQUIA Forte HT) i staklenoionomerni cement (Fuji IX). S pomoću teflonskih kalupa (8 mm x 2 mm) pripremljeno je ukupno 40 uzoraka za svaku skupinu (n=40) Uzorci su zatim stavljeni u polietilenske bočice s 5 mL deionizirane vode. Otpuštanje fluorida mjereno je ion-selektivnom elektrodom poslije 6, 24 i 48 sati te 5 tjedana. Rezultati su izraženi u mg/L, a podatci su statistički obrađeni ANOVA testom. **Rezultati**: Značajne razlike u oslobađanju fluorida uočene su unutar prvih 6 sati (ANOVA $p < 0,001$). EQUIA Forte HT imao je najveće otpuštanje, a ostali materijali nisu pokazali značajne razlike. Poslije 24 sata pokazali su EQUIA Forte HT ($p < 0,001$) i Luminos UN ($p < 0,05$) značajno veća otpuštanja u usporedbi s drugim testiranim materijalima. EQUIA Forte HT nastavio je oslobađati najviše iona poslije 48 sati ($p < 0,001$), a slijede ga Cention Forte ($p < 0,05$) i Luminos UN ($p < 0,05$). Svi parovi materijala pokazali su značajne razlike u oslobađanju fluorida poslije 5 tjedana ($p < 0,001$). **Zaključak**: EQUIA Forte HT bez premaza imao je ukupno najveće otpuštanje fluorida, a Cention Forte pokazao je najveće povećanje tijekom vremena. ACTIVA BioACTIVE-RESTORATIVE pokazala je najniže otpuštanje fluorida u ovoj studiji.

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