

# ION RELEASE FROM RESIN MODIFIED GLASS IONOMER CEMENT IN YOUNG PERMANENT IMMATURE TEETH

## OSLOBAĐANJE JONA IZ GLAS-JONOMER CEMENATA MODIFIKOVANIH SMOLOM KOD MLADIH TRAJNIH ZUBA

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### Summary

*Anticariogenic effect of the fluoride releasing restorations is in direct correlation with the quantity of the released fluorides, but even more with the longevity of this release. Apart from fluoride, glass ionomer cements release other matrix-forming cations. From all these ions, aluminium easily forms stable complexes with fluoride ions.*

*Fuji II LC restorations were placed in young permanent immature teeth. The release of fluoride in distilled water was determined spectrophotometrically and the release of aluminium was determined by atomic absorption spectrophotometry. The ion release in hard dental tissues- enamel and dentin was analyzed by energy dispersive analysis with x-rays.*

*Fuji II LC releases fluoride and aluminium ions in distilled water. Migration of ions in hard dental tissues also exists, which suggests that the remineralization of the demineralized enamel and dentin may not be a result of the fluoride migration, but a result of the deposition of minerals in the pores of the dentin, that limits the number of diffusion pathways for the bacteria and therefore increases the caries resistance. The analysis of our samples determined high levels of fluoride, but also aluminium released from Fuji II LC, that can actually influence the determination of the fluoride release.*

**Keywords:** resin-modified glass-ionomer cement, aluminium, fluoride, ion release

### Introduction

Young permanent immature teeth have voluminous pulp, with high horns, so the possibility for artificial pulp perforation is higher than in the mature teeth. The dentin tubuli are extremely wide, with a thin layer of peritubular dentin and without any intratubular dentin.

### Kratak sadžaj

*Antikariogeni efekat ispuna koje oslobađaju fluoride u direktnoj je korelacijsi sa količinom oslobođenih fluorida, ali očito mnogo više od dugotrajnosti ovog oslobođanja. Pored fluoride, glas-jonomer cementi oslobađaju i druge jone koji sudeluju u formiraju matriksa. Između ovih jona, aluminijum najlakše formira stabilne kompleksa sa fluoridnim jonima.*

*Fuji II LC ispune bile su postavljene na mlade trajne zube. Oslobođanje fluoride u destilovanoj vodi bilo je određeno spektrofotometrijom, a aluminijuma-atomskom apsorpcionom spektrofotometrijom. Migracija jona u tvrdim zubnim tkivima-gledi i dentinu analizirali smo energetskom disperzivnom analizom sa x-zracima.*

*Fuji II LC oslobođava fluoride i aluminijum u destilованoj vodi. Rezultati ukazuju da migracija jona u tvrdim zubnim tkivima postoji, tj. da remineralizacija demineralizovanog emajla i dentina ne moraju biti rezultat fluoridne migracije, nego rezultat depozicije minerala u porama dentina, što limitira broj difuzionih putanja bakterija i kao rezultat toga nastaje pojačanje otpornosti na karijes.*

**Zaključci:** Analiza primeraka pokazuje da se oslobođa fluorid iz glas-jonomer cementa modifikovan smolom; ali u isto vreme i aluminijum, koji može uticati na određivanje koncentracije fluorida u vodenim medijima.

**Ključne reči:** glas-jonomer cement modifikovan smolom, aluminijum, fluorid, oslobođanje jona

### Uvod

Mladi trajni zubi ili zubi sa nezavršenim rastom korena imaju voluminoznu pulpu, sa visokim rogovima, pa je verovatnoća artificijelne perforacije pulpe veća nego kod zrelih zuba.

When teeth appear in the mouth, the enamel acts as a semi-permeable membrane and they become susceptible to carious agents. In the same time, the immature enamel and dentin can ease the transport of certain molecules towards the pulp. This is the reason why the obturation of dentin tubuli by biocompatible materials is the essential component in the treatment of the tooth decay.

In this context, glass ionomer cements are materials of choice when we need to treat teeth with incomplete apex closure. Conventional glass ionomers have certain disadvantages, as long setting time, moisture sensitivity, drying after setting and brittleness. Resin modified glass ionomer cements are improved by the addition of monomer ingredients based on resins. They allow longer working time and controlled setting time, fast development of strength and lower sensitivity to environmental moisture changes and can be finished and polished immediately after setting<sup>1</sup>.

Anticariogenic effect of the fluoride releasing restorations is in direct correlation with the quantity of the released fluorides, but obviously even more with the longevity of this release<sup>2,3</sup>. Also, in the presence of fluorides in the oral cavity (for ex. after professional dental treatment) these materials can be recharged with fluoride<sup>3,4</sup>.

Apart from fluoride, glass ionomer cements release other matrix-forming cations<sup>3,5</sup>, as aluminium, calcium, silicon, sodium etc.<sup>6,7</sup>. From all these ions, aluminium easily forms stable complexes with fluoride ions. As the ion selective electrode is the most frequently used method for fluoride determination<sup>2,7-12</sup>, the complexation of fluoride with aluminium interferes with the determination of the fluoride concentration when this electrode is used, since it reacts only to ionic form of fluoride, but not to fluoride complexes.

We performed this study in order to gain information on the release of ions from Fuji II LC resin modified glass ionomer cement in the surrounding tissues in young permanent immature teeth. Therefore, we analyzed the fluoride and aluminium release in distilled water and ion release in hard dental tissues- enamel and dentin.

Dentinski tubuli ekstremno širok, sa tankim slojem peritubularnog dentina i bez imalo intratubularnog dentina. Kad se zubi pojave u ustima, gled se ponaša kao semipermeabilna membrana i zubi postaju podložni karioznim noksima. Istovremeno, nezrela gled i dentin mogu da olakšaju transport određenih molekula ka pulpi zuba. Iz ovih razloga, opturacija dentinskih tubula biokompatibilnim materijalima predstavlja esencijalnu komponentu u tretmanu zubnog karijesa.

U ovom kontekstu, glas-jonomer cementi su materijal izbora u slučajevima kada treba tretirati zube sa nezavršenim rastom korena. Konvencionalni glas-jonomer cementi stradaju od izvesnih nedostataka, kao što je dugo vreme vezivanja, osetljivost na vlažnost, isušivanje posle stvrđnjavanja i krtost. Glas-jonomer cementi modifikovani smolom usavršeni su dodatkom monomernih sastojaka na bazi smole. Oni obezbeđuju duže vreme rada i kontrolisano vreme vezivanja, brz razvitak snage i manju osetljivost na promene vlažnosti sredine, a mogu biti finirani i polirani odmah posle vezivanja<sup>1</sup>.

Antikariogeni efekat ispuna koje oslobođaju fluoride u direktnoj je korelaciji sa količinom oslobođenih fluorida, ali očigledno mnogo više sa dugotrajnošću ovog oslobođanja<sup>2,3</sup>. Isto tako, u prisustvu fluorida u oralnoj sredini (npr. nakon profesionalnog stomatološkog tretmana), ovi materijali mogu biti ponovo napunjeni fluoridima<sup>4,6</sup>.

Pored fluorida, glas-jonomer cementi oslobođaju i druge katjone, koji sudeluju u formiranju matriksa<sup>3,5</sup>, kao što je aluminijum, kalcijum, silicijum, natrijum itd.<sup>6,7</sup>. Između ovih jona, aluminijum najlakše formira stabilne komplekse sa fluoridnim jonima. Poznato je da je jon selektivna elektroda najčešće upotrebljavan metod za određivanje oslobođanja fluorida<sup>2,7-12</sup>; u tom slučaju, kompleksiranje aluminijuma sa fluoridom interferira sa determinacijom fluoridne koncentracije, kada se ona upotrebljava, pošto elektroda reaguje samo na jonsku formu fluora, a ne i na fluoridne komplekse.

Izveli smo ovu studiju da dobijemo informaciju za oslobođanje jona iz Fuji II LC glas-jonomer cementa modifikovanog smolom u susednim tkivima kod zuba sa nesvršenim rastom korena. U tom pogledu, analizirali smo oslobođanje fluorida i aluminijuma u destilovanoj vodi i oslobođanje jona u tvrdim zubnim tkivima-gledi i dentinu.

## **Material and methods:**

Class V cavities were prepared on 14 freshly extracted human young permanent immature teeth. Teeth were conditioned with GC Cavity Conditioner and Fuji II LC resin modified glass ionomer cement restorations were places according to the manufacturer's instructions.

Teeth were stored in distilled water for 1 month. Then, they were cut longitudinally by half and the cut surfaces were placed on the bottom of plastic moulds (Buehler®, USA, Batch No. 20-8180) with 32 mm internal diameter. The moulds were filled with resin (Epo-Thin, Buehler®, USA, Batch No. 20-8140-032) and polymerized in the vacuum dessicator for 1 hour. Polymerization continued on room temperature for additional 24 hours. The sample preparation finished by grinding with different sizes of carborundum grits up to 1 µm diamond. The samples were carbon-coated (Model S105, Edwards Co., UK) and examined with JEOL JSM 5310LV Scanning Electron Microscope on 350x magnification in backscattered electron mode (20 kV accelerating voltage and 15 mm working distance). Two linescans on each tooth were performed (one in enamel and one in dentin) by Energy Dispersive Analysis with x-rays on ISIS 300 Systems (Oxford Instruments Co., UK). Linescans were approximately 200 µm length represented the elemental distribution of the following elements: O, Na, Al, P, Sr, C, F, Mg, Si i Ca. Since there is superpositioning of SiK $\alpha$  i SrL, scanning was performed on the SrK $\alpha$  line.

The distilled water where the samples were stored was used for the determination of the fluoride and aluminium. First, we determined the level of aluminium by flame atomic absorption spectrophotometry, as follows: we created a calibration curve, determined the level of aluminium and calculated the concentration by the formula:  $Al \text{ ppm} = 100a$ , where  $a$  is the concentration of aluminium in 10ml based on the calibration curve. Determination of the fluoride was done after the determination of aluminium by spectrophotometry. The procedure consisted of:

1. Creation of a calibration curve- by preparation of series of standards from 0,00 to 1,40 ppm by dissolution of a certain volume in standard solution of fluoride.

## **Materijal i metodi**

Kaviteti Klase V bili su preparirani na 14 sveže ekstrahiranih mladih trajnih zuba. Po uputstvima proizvođača, zubi su bili kondicionirani sa GC Cavity Conditioner-om i postavili smo ispune od glas-jonomer cementa modifikovanog smolom Fuji II LC (GC Co., Japan).

Zubi su bili stavljeni u destilovanu vodu u periodu od 1 meseca. Nakon toga, zubi su bili isećeni na polovinu i isećene površine bile su postavljene na dno plastičnih modli (Buehler®, USA, Batch No. 20-8180) sa 32 mm unutrašnjeg dijametra. Modle su bile ispunjene smolom (Epo-Thin, Buehler®, USA, Batch No. 20-8140-032) i polimerizovane u vakuum-eksikatoru 1h. Proces polimerizacije produžio se na sobnoj temperaturi još 24h. Priprema primeraka završila je struganjem sa različitim dimenzijama karborundumskim šmirglama do 1 µm dijamanta. Primerci su bili pokriveni ugljikom (Model S105, Edwards Co., UK) i ispitivani sa JEOL JSM 5310LV Scanning Electron Microscope sa uvećanjem od 350x i pozadinskom elektronskom metodom (20 kV akceleraciona voltaža i 15 mm radna udaljenost). Izrađena su po dva skena za svaki zub (jedan u emajlu i jedan u dentinu) korišćenjem energetske disperzivne analize x-zracima (EDX) na ISIS 300 Systems (Oxford Instruments Co., UK). Linijski skenovi sa približnom dužinom od 200 µm predstavljali su elementarnu distribuciju sledećih elemenata: O, Na, Al, P, Sr, C, F, Mg, Si i Ca. Budući da postoji preklapanje SiK $\alpha$  i SrL, za skeniranje je bila izabrana SrK $\alpha$  linija.

Destilovana voda, koja je bila upotrebljena za smeštanje primeraka, bila je upotrebljena za određivanje oslobađanja fluorida i aluminijuma. Prvo, odredili smo nivo aluminijuma pomoću plamene atomske apsorpcione spektrofotometrije, na sledeći način: kreirali smo kalibracionu krivu, determinisali nivo aluminijuma, i tada proračunali koncentraciju, upotrebivši formulu  $ppm \text{ Al} = 100a$ , gde  $a$  pretstavlja koncentraciju aluminijuma u 10ml na osnovi kalibracione krive. Determinacija fluorida izvedena je posle određivanja aluminijuma, pomoću spektrofotometrije. Procedura se sastojala od sledećih:

1. Kreiranje kalibracione krive – pripremanja serije standarda od 0,00 do 1,40 ppm, razređivanjem određenog volumena sa standardnim rastvorom fluorida.

2. Determination- starts with distillation, then 50ml of the distillate was mixed with 10ml SPADNS and acidic circonyl. The absorbance was read on the spectrophotometer. If the concentration was above 3ppm, then the reading was delayed for 3 hours to allow "selfcorrection".

3. Calculation- by the formula  $F \text{ ppm} = 50A/V$ , where A is ppm of fluoride measured by the spectrophotometer, and V ml of the sample.

## Results

The results for the fluoride and aluminium release in distilled water (Table 1.) prove that significant quantities of aluminum are released

2. Određivanje: počinjemo destilacijom, a onda 50 ml destilata pomešamo sa 10 ml SPADNS (acidni cirkonil). Posle toga čitamo apsorbanciju na spektrofotometru. Ako je koncentracija aluminijuma iznad 3 ppm, odlažemo čitanje za 3 sata, da obezbedimo „samokorekciju“.

3. Kalkulacija: pomoću formule  $F \text{ ppm} = 50A/V$ , gde je A ppm fluorida izmerenog pomoću spektrofotometrije, a V je ml primerka.

## Rezultati

Dobiveni rezultati za oslobođanje fluorida i aluminijuma u destilovanoj vodi (Tabela 1.) dokazuju da su zнатне količine aluminijuma

*Table 1. Aluminium and fluoride release from Fuji II LC in distilled water after 1 month  
Tabela 1. Oslobođanje aluminijuma i fluoride iz ispuna Fuji II LC u destiliranoj vodi posle 1 meseca*

ion jon	N (samples) N (zubi)	min (ppm) min (ppm)	max (ppm) max (ppm)	X (ppm) X (ppm)
FLUORIDE FLUORID	14	0,2	0,7	0,595
ALUMINIUM ALUMINIJUM	14	2,1	4,8	3,527

in the distilled water (3,527 ppm), and also fluoride with medium concentration of 0,595 ppm.

After one month of storage of the teeth samples with Fuji II LC restorations there was no migration of ions in the enamel (Figure 1.), but we found increase in the level of all examined ions in dentin adjacent to the interface with the restoration; except Ca and  $\text{PO}_4^-$ , which were decreased (Figure 2.).

## Discussion

Number of studies indicated the possible fluoride migration from glass ionomer cements into the tooth structure. It has been documented that low fluoride concentrations can lead towards remineralization of dentin<sup>2,14</sup>. Incipient carious lesions were found to be remineralized and even hypermineralized. Our results are in correlation with these findings. Namely, in this case the migration should be even easier, because we performed our experiments on young permanent immature teeth, which enamel and

oslobodene u destilovanoj vodi (3,527 ppm), a isto tako i fluoride sa srednjom koncentracijom od 0,595 ppm.

Posle jednog meseca smeštaja zubnih primeraka ispunjenih sa Fuji II LC u destilovanoj vodi, ne postoji migracija jona u emajlu (Slika 1.), ali dolazi do povećanja nivoa svih ispitivanih jona u blizini interfejsa sa ispunom, osim  $\text{Ca}^{2+}$  i  $\text{PO}_4^-$ , koji su smanjeni (Slika 2.).

## Diskusija

Veliki broj studija sugerisu moguću fluoridnu migraciju iz glas-jonomer cementnih puštenja u zubnu strukturu. Efekti fluorida na dentinu otkrili su da niske fluoridne koncentracije mogu voditi ka remineralizaciji dentina<sup>2,14</sup>. Utvrđeno je da početne kariozne lezije remineralizuju, pa čak i hipermineralizuju. Naši rezultati poklapaju se sa ovim nalazima. U našem slučaju bi migracija trebalo da bude lakša pošto su ova ispitivanja rađena na mladim trajnim Zubima, čiji emajl i dentin, kao što smo napomen-

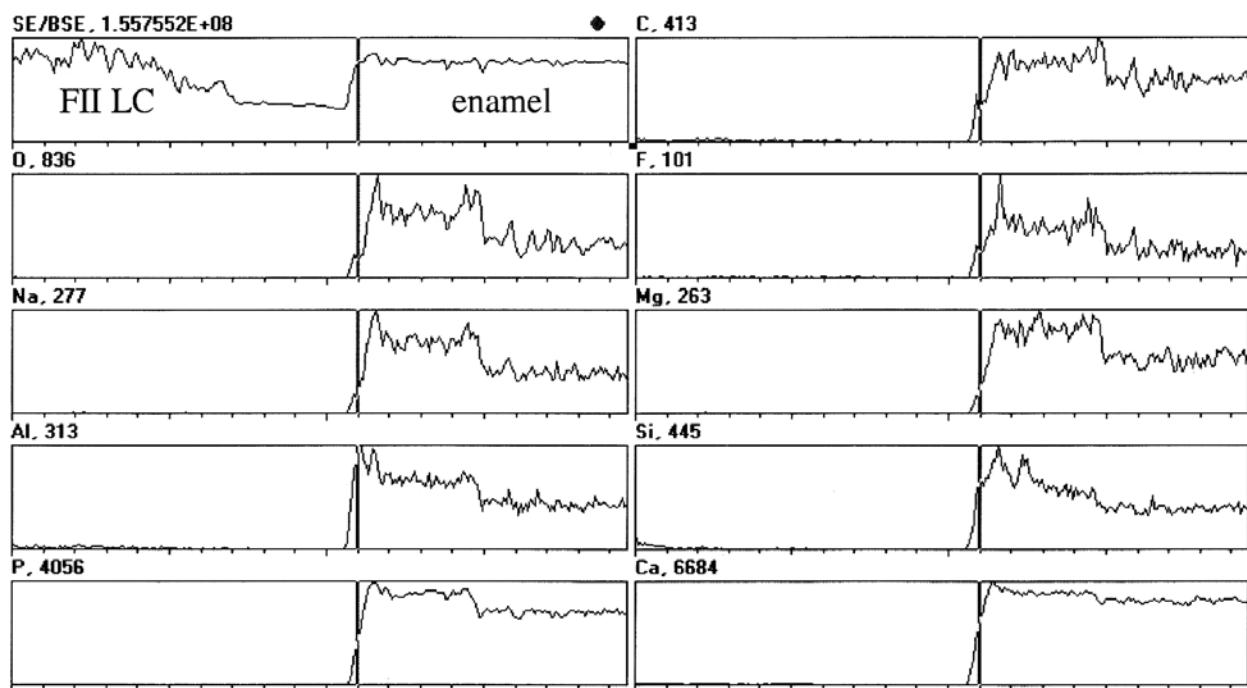


Figure 1. Ion release in enamel after 1 month of placement of Fuji II LC restorations

Slika 1. Oslobođanje jona u gledi posle 1 meseca od postavljanja ispune sa Fuji II LC cementom

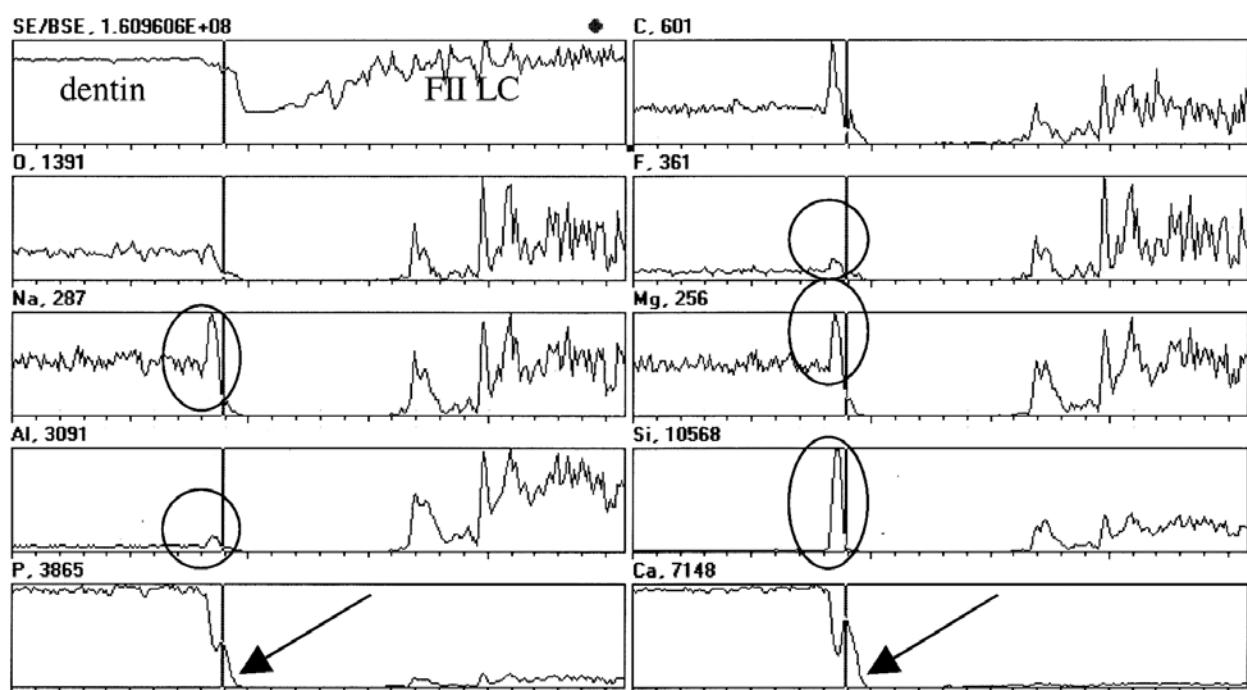


Figure 2. Ion release in dentin after 1 month of placement of Fuji II LC restorations

Slika 2. Oslobođanje jona u dentinu posle 1 meseca od postavljanja ispune sa Fuji II LC cementom

dentin can ease the transport of the fluoride. Remineralization of the demineralized enamel and dentin may not be a result of the fluoride migration, but a result of the deposition of minerals in the pores of the dentin, that limits the number of diffusion pathways for the bacteria and therefore increases the caries resistance.

uli, mogu olakšati transport fluorida. Remineralizacija demineralizovanog emajla i dentina ne moraju biti rezultati fluoridne migracije, već rezultat depozicije minerala u porama dentina koji limitira broj difuzionih putanja bakterija, i kao rezultat toga nastaje pojačanje otpornosti na karijes.

Highest quantities of fluoride in watery media are released during the first days<sup>9</sup>, and then it decreases to almost constant level. The examinations carried out by Tay & Braden<sup>15</sup> and Veerbeck et al.<sup>16</sup> prove that fluoride release is a result of two different processes. The first process is characterized by an initial burst of fluoride from the surface<sup>9</sup>, after which the release is markedly reduced<sup>17</sup>. The first process is accompanied by a second diffusion process, when small quantities continue to be released in the surrounding medium for a long period of time. Our results confirm the previous findings.

Aluminium fluoride is one of the stable complexes which can be formed with the fluoride anions. Several different forms of aluminium fluoride with different Al/F ratios can be found. One aluminium ion in the complex structure can be surrounded by a maximum of six octahedrally placed fluoride ions. Therefore, the presence of one aluminium ion can eliminate maximum of six free fluoride ions from the solution<sup>13</sup>. Highest quantities of aluminium are released during the first days after setting<sup>12</sup>. During the maturation, aluminium release is decreased, because aluminium ions from the surface were washed out, and the rest are captured deep into the matrix<sup>13</sup>. The analysis of our samples determined high levels of aluminium released from Fuji II LC, which can actually influence the determination of the fluoride release.

## Conclusions

1. Release of ions in distilled water and hard dental tissues from Fuji II LC resin-modified glass ionomer cement placed in young permanent immature teeth exists.
2. Remineralization of the demineralized enamel and dentin must not be a result of the fluoride migration only.
3. Aluminium release in watery media can influence the determination of the fluoride release.

Najveća količina fluorida u vodenim medijima oslobođena je tokom prvih dana<sup>9</sup>, a onda opada na skoro konstantnom nivou. Ispitivanja Tay & Braden-a<sup>15</sup> i Veerbeck-a et al.<sup>16</sup> ukazuju da se kao rezultat dva različita procesa javlja proticanje fluorida. Prvi proces karakterističan je po inicijalnoj eksploziji oslobođanja fluorida sa površine<sup>9</sup>, nakon čega je oslobođanje značajno reducirano<sup>17</sup>. Drugi je propraćen sekundarnim procesom difuzije, kada male količine fluorida produžavaju oslobođanje u okolnom medijumu u dugom vremenskom periodu. Naši rezultati potvrđuju nalaze drugih autora da se fluoridi oslobođaju u destilovanoj vodi nakon jednog meseca.

Aluminijum fluorid jedan je od stabilnih kompleksa koji može biti formiran sa fluoridnim anjonima. Može biti pronađeno nekoliko različitih formi aluminijuma u različitim Al/F proporcijama. Jedan aluminijumov jon u kompleksnoj strukturi okružen je sa maksimumom od šest oktaedralno postavljenih fluoridnih jona. Ovo znači da prisustvo jednog aluminijumovog jona može eliminisati maksimum šest slobodnih fluoridnih jona iz vodenog rastvora<sup>13</sup>. Najveća količina aluminijuma oslobođava se tokom prvih nekoliko dana nakon vezivanja cementa<sup>12</sup>. Sa maturacijom, oslobođanje aluminijuma se smanjuje, pošto su aluminijumovi joni sa površine oprani, a ostatak je zarobljen duboko u matriksu<sup>13</sup>. Analizom naših primjera, može se utvrditi da postoji visok nivo oslobođanja aluminijuma, koji može uticati na determinisanje oslobođanja.

## Zaključci

1. Postoji oslobođanje jona u destilovanoj vodi i tvrdim zubnim tkivima iz Fuji II LC (glas-jonomer cementa modifikovanih smolom) postavljenim na mlade trajne zube.
2. Remineralizacija demineralizovanog emajla i dentina ne mora biti rezultat fluoridne migracije.
3. Oslobođanje aluminijuma iz glas-jonomer cementa modifikovanih smolom može uticati na određivanje koncentracije fluorida u vodenim medijima.

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