

Evaluation of Relationship between Ion Release Dynamics and Chemical Changes of Five Fluoride Release Restorative Materials

SUMMARY

Background/Aim: The role of ions in prevention of dental caries has been demonstrated in numerous studies. The aim of this study was *to investigate* the amount of *released ions* from different restorative materials using ion selective electrode (ISE) and inductively coupled plasma-optical emission *spectrometry* (ICP-OES) and assess *the chemical changes in the material using energy dispersive spectroscopy (EDS)*. **Material and Methods:** Six plates were left in the contact with deionised water which was replaced everyday during the first 10 days, and on every tenth day for a period of 90 days. Concentrations of sodium, silicium, strontium, calcium, aluminium and phosphorus ions were analysed using inductively coupled plasma-optical emission *spectrometry* and fluoride ions were assessed using ion-selective electrode. Energy dispersive spectroscopy was utilized to study the weight percentages of elements through investigated materials. **Results:** Significant decrease in fluoride release has been observed between all tested materials during first four days ($p < 0.05$). Apart from sodium, cumulative amount of released fluoride, silicium, strontium, calcium, aluminium and phosphorus ions was $t^{1/2}$ dependent. Mean weight percentage of fluoride was the highest in GC Equia, while the highest weight percentage of strontium was observed in case of GC Fuji IX and GC Fuji II. **Conclusions:** The pull of strontium was obvious in the middle of investigated materials GC Fuji IX, CG Equia and Ketac N100 may enhance the fluoride release, which may have crucial role in caries prevention.

Key words: Fluoride Release, Glass-Ionomer Cement, Ion Release, Conventional Glass-Ionomer Cement, Resin Modified Glass-Ionomer Cement

Bojana Ilić¹, Dejan Marković², Biljana Dojčinović³, Bojan Petrović⁴

¹ Vinca Institute of Nuclear Sciences, Mike P. Alasa 12-14, 11001 Belgrade, Serbia

² School of Dental Medicine, University of Belgrade, Dr. Subotica 11, 11000 Belgrade, Serbia

³ Institute of Chemistry, Technology and Metallurgy, University of Belgrade, Belgrade, Serbia

⁴ Department of Dental Medicine, Faculty of Medicine, University of Novi Sad, Novi Sad, Serbia

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Introduction

Beyond doubt, the impact of ions, especially fluoride ions, in dental caries prevention has been proven^{1,2}. Enamel remineralization may be encouraged by high fluoride amount at the surface of the tooth³. Fluoride may interact with the elements of dental pellicle and thereby interfere with the dental plaque formation⁴. Furthermore, a high amount of fluoride may be the reason for bacterial metabolism and growth obstruction⁵.

Glass-ionomer cements are biomaterials which can release fluoride at a constant concentration over a prolonged time period⁶. In addition to conventional

and resin-modified glass-ionomer cements, there are other restorative materials that can release fluoride, such as composites, compomers, giomers and amalgams⁶. Difference in the fluoride release may be the result of different chemical composition of the materials, mechanism of their setting, amount of fluoride in the material, fluoride origin and the pH of the surrounding medium^{6,7}.

So far, three mechanisms of fluoride release from glass-ionomer cements into the aqueous solutions have been proposed. The first one comprises sudden release of ions during the first 24 h from the outer layers of the material - short-term reaction, diffusion through pores and

micro-fracture and finally gradual diffusion through the bulk cement-long-term reaction^{6,8}. This can be clarified by the fact that loosely bound water and ions in porous glass-ionomer cements can be exchanged with elements from the surrounding medium by passive diffusion^{9,10}.

Glass-ionomer cements may also release a variety of matrix-forming ions, i.e. calcium, sodium, strontium and aluminium^{7,11}. Similarly to fluoride release, the sudden burst release of matrix-forming ions during the first 24 h is also present^{7,11,12}. Apart from fluoride, the mechanisms of matrix-forming ions release are not well reported, and neither are possible chemical changes in the material.

The aim of this study was to investigate the amount of released ions from different dental restorative materials into deionised water using ion selective electrode (ISE) and inductively coupled plasma-optical emission spectrometry (ICP-OES) and to assess the chemical changes in the material using energy dispersive spectroscopy (EDS).

Material and Methods

Preparation of the specimens

Five materials were used in this study (Table 1). Materials were prepared according to the manufacturers' instructions. Capsules of GC Fuji IX, GC Equia and GC Fuji II LC were activated, set in the amalgamator and then mixed for 10 sec at high speed (Silamat device, Vivadent, Schaan, Liechtenstein). Immediately after

mixing, materials were placed into teflon moulds (5 mm in diameter and 5 mm high) using a suitable applicator. Two pastes of 3M Ketac N100 were mixed for 20 sec and placed into teflon moulds. 3M F2000 Compomer was put directly into teflon moulds using disposable cannula. After that, light-curing materials (3M F2000 Compomer, GC Fuji II, 3M Ketac N100) were polymerized on both sides with use of LED lamp (GC G-Light, GC Int., Tokyo-Japan) for 40 sec. GC Fuji IX and GC Equia were left to rest for 10 min to enable initial chemical reaction. Six plates consisting of ten moulds of each material were then incubated at 37°C in 95% humid environment for 24 h to ensure complete setting of the material. Plates were left in the contact with 20 ml of deionised water (pH=5.76±0.51), which was replaced every day during the first 10 days, and on every tenth day for a period of 90 days.

Ion measurements

The concentrations of ions in the investigated materials were detected after total dissolution in the acid, while the concentrations of ions in the liquid extract were determined directly by inductively coupled plasma-optical emission spectrometry analysis -ICP-OES (n=3). ICP-OES analysis was performed using Thermo Scientific iCAP 6500 Duo ICP (Thermo Fisher Scientific, Cambridge, United Kingdom). Quantification of ions in the solution was assessed at the adequate emission wavelengths of light. The release of fluoride ions into deionised water was determined by a fluoride ion-selective electrode (ISE DC219-F, Mettler Toledo Inc., Columbus-OH, USA) [13].

Table 1. Materials used in the study

Material	Composition	Manufacturer		Lot No.
Fuji IX	Alumino-fluoro-silicate glass, polyacrylic acid, distilled water, polybasic carboxylic acid	GC Int, Tokyo-Japan	Conventional glass-ionomer cements	1211121
Fuji II LC	Alumino-fluoro-silicate glass, methylmethacrylate, distilled water, camphorquinone	GC Int, Tokyo-Japan	Resin modified glass-ionomer cements	1107277
Equia	Polyacrylic acid, aluminosilicate glass, distilled water	GC Int, Tokyo-Japan	Conventional glass-ionomer cements	1103122
Ketac N100	Fluoroaluminosilicate glass, Vitrebond copolymer, methacrylate-modified polyalkenoic acid, HEMA	3M Dental Products, St. Paul-USA	Resin modified glass-ionomer cements	238779
Compomer F2000	Fluoroaluminosilicate glass, carboxylate dimethacrylate, glyceryldimethacrylate, poly(vinylpyrrolidone), camphorquinone	3M Dental Products, St. Paul-USA	Compomer	19970904

Energy dispersive spectroscopy (EDS)

Energy dispersive spectroscopy (EDS, JEOL, JSM-5300, Tokyo, Japan) was utilized to study the weight percentages of elements through the investigated

materials. Samples were sliced longitudinally and then transferred into the chamber of the instrument and observed at a voltage of 30 kV. Measurements were performed at three points, near the edge facing the

distilled water (1st), near the opposite edge (3rd) and in the middle of the sample (2nd). Each EDS analysis was performed 5 times at each point.

Statistical analysis

Data analysis was assessed using ANOVA Repeated Measures test, post hoc Tukey's test. The level of significance was determined at $p < 0.05$ and the data were processed using statistical software IBM SPSS 20.

Results

The investigated materials' mean weights were as follows: GC Fuji II=3.72±0.51g, GC Fuji IX=3.22±0.71g, GC Equia=3.67±0.91g, 3M Ketac N100=3.85±0.41g and 3M Compomer F2000=3.20±0.68 g. Area of moulds exposed to the deionised water was 7.85 cm² per plate for each material. Table 2. presents chemical composition of tested materials used in the study after their set-up. The exact concentrations of fluoride in the tested materials after their set up were not detected due to the use of HF in the process of materials' preparation.

Table 2. Chemical composition of tested materials (mg/g)

Elements	Materials				
	GC Fuji II	3M Ketac	GC Equia	3M Compomer	GC Fuji IX
Al	109.94	46.83	160.63	124.65	146.71
Ca	0.45	0.51	0.57	0.34	0.97
Fe	0.32	0.13	0.15	0.23	0.41
K	0.36	0.22	0.03	0.01	0.39
Mg	0.04	0.03	0.01	0.06	0.04
Na	4.04	7.60	57.81	25.24	12.50
P	11.04	8.48	35.88	26.21	29.70
Si	113.81	122.36	78.87	122.96	8.99
Sr	141.65	73.85	116.10	105.56	149.50
Zn	0.001	20.85	0.002	0.02	0.001

Initial fluoride release was in the following order: GC Fuji IX > 3M Ketac N100 > GC Equia > GC Fuji II > 3M Compomer F2000 (Figure 1). A significant decrease in fluoride release has been observed between all tested materials during the first four days ($p < 0.05$, Figure 1). Figures 2 to 7 present the cumulative amounts of sodium, silicium, strontium, calcium, aluminium and phosphorus ions released by the materials included in the study.

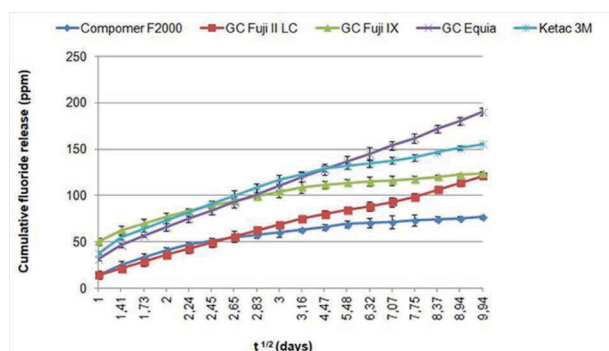


Figure 1. Cumulative fluoride release by investigated materials into deionised water

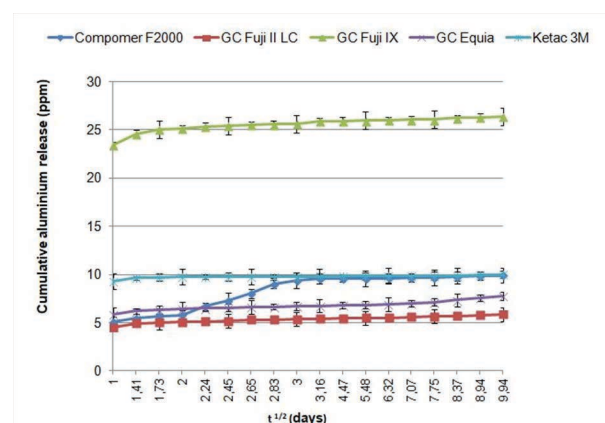


Figure 2. Cumulative aluminium release by investigated materials into deionised water

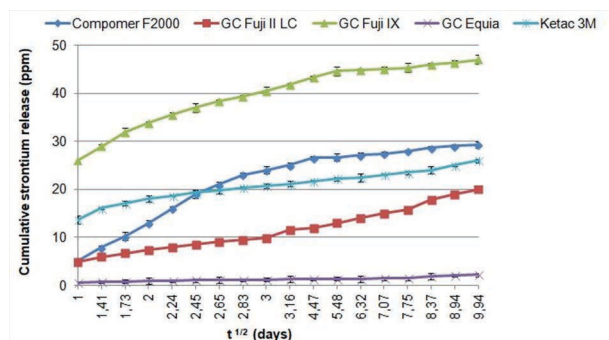


Figure 3. Cumulative strontium release by investigated materials into deionised water

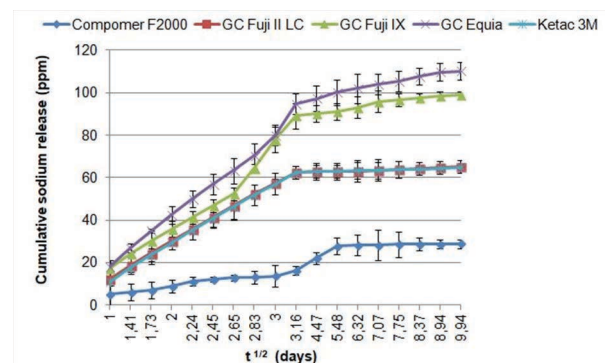


Figure 4. Cumulative sodium release by investigated materials into deionised water

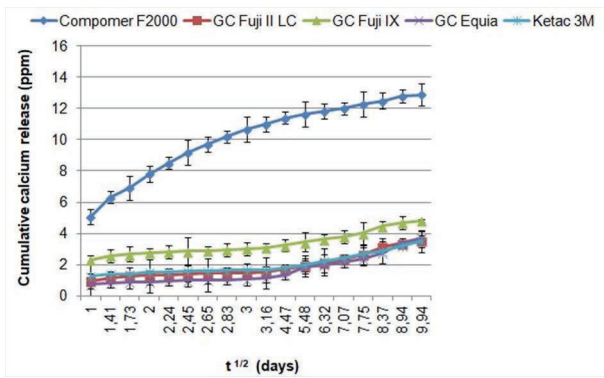


Figure 5. Cumulative calcium release by investigated materials into deionised water

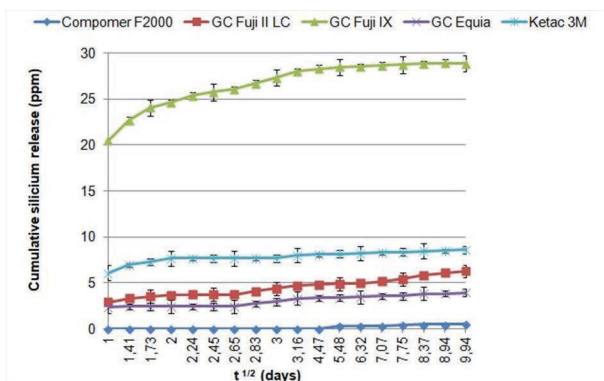


Figure 6. Cumulative silicon release by investigated materials into deionised water

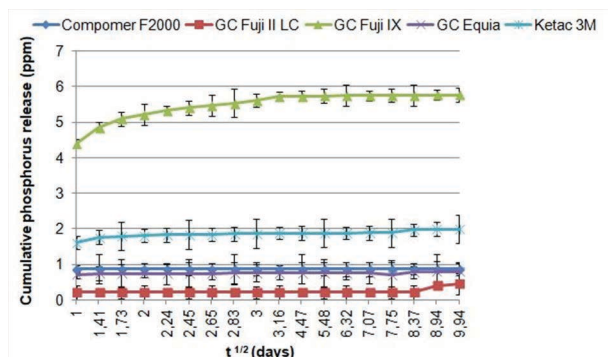


Figure 7. Cumulative phosphorus release by investigated materials into deionised water

Cumulative concentrations of released ions were plotted time - $t^{1/2}$. Cumulative concentrations of released fluoride, silicon, strontium, calcium, aluminium and phosphorus ions were $t^{1/2}$ dependent, while cumulative concentrations of sodium ion release were $t^{1/2}$ dependent after the tenth day in all cases. For all tested materials, a significant correlation between strontium and fluoride cumulative ion release was evident ($p < 0.05$, Pearson's correlation), as was as between aluminium and fluoride ions ($p < 0.01$, Pearson's correlation).

Weight percentages of the oxygen were higher than that of other elements regarding all the investigated materials (Table 3). In case of GC Equia the mean weight percentage of fluoride was the highest at all points of measurement compared to the other investigated materials and gradually decreased ($p < 0.05$) toward the surface that was in contact with the deionised water ($p < 0.05$). A higher mean weight percentage of fluoride in the middle of the specimens of other investigated glass-ionomer materials was observed but without statistically significant differences ($p > 0.05$). A statistically lower weight percentage of fluoride was noted between Compomer F2000 and other investigated materials ($p < 0.05$) at the 2nd point, while at the 3rd point the weight percentage of fluoride was statistically higher compared to GC Fuji II and Ketac N100 ($p < 0.05$). Statistically lower weight percentage of fluoride was noted comparing GC Fuji II with GC Fuji IX and Ketec N100 ($p < 0.05$) at the 2nd point, while at the 3rd point that trend was evident in contrast with GC Fuji IX and GC Equia ($p < 0.05$).

The highest weight percentage of strontium was noted in the middle of the material specimens, and especially in the case of GC Fuji IX (Table 3). A statistically significant difference between 1st and 2nd points of measurements was observed ($p < 0.05$), except in case of GC Equia ($p > 0.05$). The highest weight percentage of aluminium was noted in the middle of the material GC Fuji IX and statistically differed compared to the other investigated materials ($p < 0.05$, Table 3). According to the weight percentage, zirconium and zinc were detected only in case of Ketac N100 and phosphorus in case of GC Equia (Table 3).

Table 3. Weight percentages ($MV \pm SD$) of main elements identified with EDS

Elements	GC Fuji IX			GC Fuji II LC			GC Equia			Ketac N100			Compomer F2000		
	1S	2S	3S	1S	2S	3S	1S	2S	3S	1S	2S	3S	1S	2S	3S
C	18.09±1.8	19.05±2.1	20.63±0.5	20.03±0.8	19.53±3.4	18.26±2.4	20.71±1.5	15.94±2.6	23.78±1.7	34.19±1.8	26.55±2.1	20.31±2.3	37.43±2.5	28.82±1.1	28.73±1.4
O	36.38±1.3	37.62±1.1	38.30±2.1	24.68±1.2	27.04±1.2	28.99±1.1	29.97±2.4	31.38±2.1	34.88±1.0	29.00±2.0	29.32±1.5	32.33±1.2	35.44±1.3	30.03±2.4	32.85±2.1
F	9.09±0.2	11.11±0.5	9.95±0.1	8.56±0.9	9.48±0.4	8.08±0.2	16.72±0.2	22.13±0.6	27.30±0.4	8.44±0.2	11.41±0.3	8.18±0.2	10.01±0.4	9.17±0.1	11.11±0.1
Al	11.48±1.1	14.76±1.1	9.69±0.8	12.20±0.2	10.94±0.5	11.45±0.2	11.71±0.1	11.87±0.3	10.08±0.7	4.37±0.1	6.72±0.6	4.84±0.2	7.23±0.2	8.66±0.3	8.35±0.2
Si	6.74±0.1	9.55±0.6	5.85±0.2	10.44±0.3	11.14±0.3	10.52±0.8	6.76±0.1	7.07±0.1	6.03±0.4	9.76±0.3	13.44±1.1	9.46±0.3	7.75±0.2	10.19±1.2	8.46±0.4
Sr	18.21±0.4	26.95±1.3	15.58±1.1	24.11±1.3	21.87±1.0	22.70±0.9	9.84±0.4	15.88±1.2	7.41±0.7	5.93±0.3	11.97±0.3	9.97±0.2	9.88±0.2	11.18±0.7	10.51±0.9
P	NA	NA	NA	NA	NA	NA	1.35±0.3	0.96±0.1	1.30±0.2	NA	NA	NA	NA	NA	NA
Zn	NA	NA	NA	NA	NA	NA	NA	NA	NA	3.68±0.7	4.35±0.4	NA	NA	NA	NA
Zr	NA	NA	NA	NA	NA	NA	NA	NA	NA	13.06±1.2	22.79±2.1	12.82±0.9	NA	NA	NA
Na	NA	NA	NA	NA	NA	NA	4.00±1.4	3.43±3.2	2.99±2.8	NA	NA	NA	2.26±0.3	1.95±0.2	0.96±0.1

1S- measurement point near the edge faced the distilled water, 2S- measurement point in the middle of the sample, 3S- measurement point near the opposite edge of distilled water
 NA- not available

Discussion

When they first appeared, more than thirty years ago, glass-ionomer cements found their place in everyday dental practice due to their biocompatibility, chemical bonding to dental tissues and possibility to set in a humid environment¹⁴. In addition, one of the main characteristics of these materials is the ability to release fluoride, which has a decisive role in the dental caries prevention and/or secondary caries inhibition^{1,6,7,15}.

However, the mechanism of fluoride release from glass-ionomer cements has not been fully understood¹⁶. After initial burst release during the first 24 h, constant fluoride concentrations in aqueous solutions are achieved during the first ten days¹⁷⁻²⁰. In the present study, initially released concentrations of fluoride ranged from 14-50 ppm, while cumulative fluoride release on the tenth day varied from 63 to 123 ppm depending on the investigated material, which is in accordance with the previous studies^{21,22}. In addition, the process of long-term fluoride release proved to be $t^{1/2}$ dependent, as previously described in the literature^{11,23}, which implies its gradual diffusion.

Results of numerous studies led to the conclusion that conventional glass-ionomer cements initially release the highest fluoride concentrations in the surrounding medium compared to resin modified cements^{19,24}. Results of our study are consistent with these findings since the highest level of initially released fluoride was observed in the GC Fuji IX group (50 ppm). During the experimental period, the most consistent fluoride release has been noticed in the GC Equia group (7-10 ppm, up to 90 days).

Resin modified glass-ionomer cements may release concentrations of fluoride equivalent to the conventional glass-ionomers²⁵. Concentrations of initially released fluoride by nano-ionomer 3M Ketac N100 (37 ppm) were comparable to the result obtained by Paschoal *et al.*²⁵ and they proved to be similar to conventional glass-ionomer cements tested in our study. In the next ten days the concentrations of fluoride released by 3M Ketac N100 were nearly identical to the concentrations of fluoride released by GC Equia and ranged from 8-17 ppm, as demonstrated earlier²⁵. After that period, a decrease in the amount of released fluoride was observed and it was continuously comparable to 3M Compomer F2000 and GC Fuji IX. On the other hand, concentrations of fluoride ions released by GC Fuji II LC in the first 24 h were similar to the values obtained in the case of 3M F2000 Compomer, with no evident burst effect, as also reported by Yip *et al.*²⁶, and with the smallest oscillations even after the tenth day. The differences in the results gained in our study, regarding fluoride release by two resin modified glass-ionomer cements, may be due to particle size (fluoroaluminosilicate glass), type and ratio of the added resin monomer.

All investigated glass-ionomer cements in the present study exhibited a very small level of released phosphorus ions, which were often immeasurable. Some studies demonstrated that the acid environment did

not have effect on the release of phosphorus ions, as on strontium, silicium, aluminium, calcium and sodium ion release which were evidently increased under the same conditions^{11,27}. Other studies demonstrated cumulative concentrations of released ions being, apart from sodium, t or $t^{1/2}$ dependent¹¹; however, our results showed for all tested materials that sodium exhibited $t^{1/2}$ dependent release after the 10th day. Although the content of silicium in the set materials investigated in this study was high, except in case of GC Fuji IX, its concentrations in deionised water were very low. In contrast, the release of aluminium and strontium ions into deionised water was evidently higher respecting its high content in all set materials used in the study.

Release of aluminium was the highest in the GC Fuji IX and 3M Ketac N100 groups and significantly correlated with fluoride release for all investigated materials. It may be assumed that aluminium enhances fluoride release by forming aluminofluoro complexes AlF^- and AlF_2^- ^{11,28}. Wiegand *et al.*⁶ suggested that these complexes possess anticariogenic effect but further confirmations are needed. Although it is known that aluminium is neuro-toxic and may disturb calcium homeostasis and cellular oxidation, the amounts released from glass-ionomers are still small to exert these effects⁷.

Significant correlation was observed between the release of strontium and fluoride ions in this study. Dabsie *et al.* showed noticeable incorporation of strontium into the surface layer of enamel¹². Due to this fact, it is assumed that strontium has synergistic effect with the fluoride in antibacterial activity and therefore also plays an important role in caries reduction¹².

Although the exact concentrations of fluoride in the tested materials could not be detected due to HF use, which is the limitation of this study, helpful information have been gathered regarding the chemical changes that occur in the investigated materials during the contact with the deionised water. The highest proportion of strontium and aluminium in the case of GC Fuji IX specimens fully corresponded with the highest cumulative amount of released ions. Higher fluoride content at the surface opposite to deionised water of Compomer F2000 samples compared to GC Fuji II and Ketac N100 may explain the fact that the used resin (carboxylate dimethacrylate, glyceryldimethacrylate) did not allow the diffusion of fluoride ions which is in accordance with the dynamics of its release. The pull of strontium observed in the middle of investigated materials GC Fuji IX, CG Equia and Ketac N100 may have an influence on the fluoride release enhancement.

Conclusions

The pull of strontium was obvious in the middle of investigated materials GC Fuji IX, CG Equia and Ketac

N100 may enhance the fluoride release, which may have crucial role in caries prevention. Although significant correlation was observed between releases of fluoride and strontium, and fluoride and aluminium, further investigations should be performed in order to determine the exact role of strontium and aluminium in caries prevention.

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Correspondence

Bojana Četenović

Vinca Institute of Nuclear Sciences

Belgrade, Serbia

e-mail: bojana.cetenovic@hotmail.com