

## Measurement of ion elution from dental ceramics

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### Abstract

Dental ceramics are generally considered to be chemically inert materials. However, characteristics of one dental ceramic are not necessarily present in another dental ceramic material. The aim of this study was to test the elution of sodium, potassium, magnesium, silicon and aluminium ions from feldspathic, alumina and glass–ceramics after 16 h of immersion in the acetic acid solution, according to ISO Standard 6872 method and to assess their potential toxicity. Elution was determined using the atomic absorption spectrophotometer (AAS) and UV–vis spectrophotometer. The highest leaching of the ions was shown for the feldspathic ceramic specimens and the lowest for the glass ceramic specimens. The release of the ions from the analyzed specimens did not correspond to their composition declared by the manufacturers. It can be concluded that the measured release of ions is not significant and correlates well with the data from the literature linked with glass corrosion, that is, silicate-based dental ceramics.

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### 1. Introduction

Dental ceramic materials are considered chemically most inert reconstructive materials. Different kinds of ceramics possess different chemical properties. Because of this, inertness of a specific dental ceramic material cannot be generalized. Very frequently on the market appear new ceramic materials that need to be tested *in vitro* and clinically. Similarly, the development of new testing methods is needed to complete the existing knowledge of dental ceramics chemical stability.

Degradation which takes place in the oral cavity due to mechanical, chemical or chemical and mechanical influences, made these characteristics questionable. Influences of mentioned factors present in the oral cavity are inseparable from each other. White *et al.*<sup>1</sup> showed that investigations of individual influences are relevant, and that the sum of their

individual influences does not differ significantly from their simultaneous action.

If chemical influences are considered, oral cavity is a very aggressive environment for restorative materials. Firstly, saliva pH values constantly change depending on the food-intake, amount of plaque present, saliva composition and the stomach acidity. Besides, aqueous solutions have been proved to lower dental ceramics resistance to fracture. Corrosive processes, except from deteriorating mechanical properties, heighten the surface roughness and plaque adhesion of some ceramic materials, cause wear of the antagonistic teeth or restorative materials and change the colour of dental ceramics, thereby compromising the aesthetic appearance of ceramic restorations.<sup>2–4</sup> Surface roughness can cause changes in the interactions of the clinical restoration and the biologic environment. Reduction of chemical stability induces further elution of inorganic ions from the surface of dental ceramic materials. These ions are potentially toxic (e.g. lithium from lithium–glass ceramics).

There are numerous *in vitro* studies investigating chemical degradation of reconstructive materials. Main factors

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influencing stability of dental materials were type and composition of the solution, as well as duration of immersion.<sup>5–10</sup> Large differences in ion leaching have been found between saliva and distilled water, thereby casting doubt on the clinical relevance of material testing performed in distilled water.<sup>7–9</sup> Acids, like hydrochloric and acetic, have been used for the chemical stability testing. Advantages of acid over artificial saliva or distilled water, lies in faster material degradation which offers long-term prediction of dental ceramics stability. Besides, use of acids allows testing for situations when pH is lowered in the oral cavity. It is well known that pH values in the oral cavity vary considerably. That is the reason why ISO standard 6872<sup>1</sup> uses 4% acetic acid for the chemical stability testing of dental ceramics.

A whole array of different material chemical stability testing methods exists. European ISO standard<sup>11</sup> or American ADA<sup>12</sup> standard are cited as standard tests for chemical stability. These standards measure specimen total weight loss after immersion in acetic acid over a certain period of time.

Measurement of specific ions release can determine process of the dental ceramic degradation more precise than measurement of the total weight loss of specimens. Toxicity of the released components is considered insignificant, because of the chemical inertness and low abrasivity of the ceramics during the masticatory function.<sup>2</sup> However, there are instances where the influence on the body is possible. For example, it can occur when broken inlay, onlay or crown parts are accidentally swallowed, also in irradiated patients with lowered salivary flow where fluorides are administered to prevent tooth demineralization. Namely, there are studies showing rapid ceramic surface deterioration in the presence of acidic fluorides.<sup>13,14</sup> If the influences of specific ions are analyzed, aluminium can be toxic in patients with renal insufficiency. There are also controversial data suggesting a role for aluminium in the development of the Alzheimer disease, as well as certain other neurodegenerative disorders of unknown etiology.<sup>15</sup> Calcium is not toxic, but its oxide in pulverized form can mechanically damage lungs and promote lung disease emergence.<sup>16</sup> Other elements like magnesium, potassium, sodium or fluorine are present during corrosion or deglutition in extremely low concentrations and their toxicity is not significant.

The aim of the study was to measure elution of sodium, potassium, magnesium, silicon and aluminium ions from different dental ceramics and to assess their potential toxicity.

## 2. Experimental procedure

Four different dental ceramic materials were tested in this investigation (Table 1). Ten platelet-formed samples with dimensions 10 mm × 10 mm × 2 mm for each material were made according to manufacturers' instructions. For IPS-Classic and Vitadur Alpha samples, a Plexiglas mould was utilized to condensate the ceramic material. After condensation the samples were put on the foil and transferred to

standard furnace for prepared manufactured using wax samples and heat-pressuring method. All samples were glazed over the whole surface to imitate the actual restorations from dental laboratories as close as possible.

Chemical composition of the tested ceramic materials, according to manufacturers declarations are shown in Table 2.

Chemicals used for the chemical stability testing are listed in Table 3.

### 2.1. Instruments used for chemical stability measurements

- (1) SPECTROPHOTOMETER UV–vis, COLEMAN 55, PERKIN ELMER (Norwalk, USA) used for determination of Si<sup>4+</sup> and Al<sup>3+</sup> ions.
- (2) ATOMIC ABSORPTION SPECTROPHOTOMETER (AAS), AA-6800, SHIMADZU (Kyoto, Japan) used for determination of Na<sup>+</sup>, K<sup>+</sup> and Mg<sup>2+</sup> ions.

### 2.2. Conditions of measurements

- Wave length of Mg: 285.2 nm
  - Wave length of K: 766.5 nm
  - Wave length of Na: 589.0 nm
  - Hollow cathode lamp.
- (3) Thermostatic shaking assembly, INNOVA 4080 INCUBATOR-SHAKER (Herisau, Switzerland) used for shaking the samples in 4% CH<sub>3</sub>COOH at temperature of 80 °C.
  - (4) Sterilizer, Instrumentaria (Zagreb, Croatia) used for drying the samples.
  - (5) UltraSonic Bath Model 1510 DTH, Electron Microscopy Sciences (Hatfield, USA) used for cleaning the samples in distilled water.

### 2.3. Chemical stability

Samples were washed with distilled water in the ultrasonic bath (ISO 3696), dried in the sterilizer at 150 ± 5 °C for 4 h. After that, every sample was transferred to a plastic polypropylene (PP) flask with 4% 25.0 mL acetic acid (CH<sub>3</sub>COOH). Samples were completely immersed in the solution. Afterwards, flasks were transferred to thermostatic shaking assembly at 80 °C, 200 rpm, for 16 h. Then, the samples were removed from flasks. The measurement of mass concentrations of eluted Na<sup>+</sup>, K<sup>+</sup>, and Mg<sup>2+</sup> ions using AAS, and Si<sup>4+</sup> and Al<sup>3+</sup> ions using UV–vis spectrophotometer from immersion solutions was performed.

### 2.4. Si<sup>4+</sup> elution measurement

First, 1 mL of the tested solution and 2 mL of ammonium-molibdate solution was added to a 10 mL flask and well mixed. After exactly 5 min, 2 mL of oxalic acid was added and stirred. After 1 min, 2 mL of Photo-Rexa, Kemika (Zagreb,

Table 1  
Dental ceramics samples

Name	Manufacturer	Type of ceramics
Vitadur alpha	Vita-Zahnfabrik, Bad Säckingen, Germany	Feldspathic ceramic (all-ceramic material)
IPS-Classic	Ivoclar Vivadent, Schaan, Liechtenstein	Feldspathic ceramic (metal ceramic material)
IPS-Empress 2	Ivoclar Vivadent, Schaan, Liechtenstein-layering technique	Glass-ceramic (apatite ceramic)
IPS-Empress 2	Ivoclar Vivadent, Schaan, Liechtenstein-staining technique	Glass-ceramic (lithium-disilicate ceramic)

Table 2  
Chemical composition of the samples (values shown as %)

	Vitadur alpha	IPS-Classic	IPS-Empress 2 (material for layering)	IPS-Empress 2 (material for staining)
SiO <sub>2</sub>	62.0–65.0	57.8–73.0	59.0–63.0	57.0–80.0
Al <sub>2</sub> O <sub>3</sub>	14.0–15.0	11.1–17.1	17.0–21.0	0–5.0
K <sub>2</sub> O	7.0–8.0	6.4–19.3	10.0–14.0	0–13.0
Na <sub>2</sub> O	4.0–5.0	1.9–9.6	3.5–6.5	
CaO	1.0–2.0	0.1–2.6	0.5–2.5	
MgO		0.1–1.8	0.1–1.8	0–5.0
ZrO <sub>2</sub>	0.2–0.8			
B <sub>2</sub> O <sub>3</sub>	6.0–7.0		0.1–1.0	
P <sub>2</sub> O <sub>5</sub>				0–11.0
La <sub>2</sub> O <sub>3</sub>				0.1–6.0
Li <sub>2</sub> O		0.0–0.5		11.0–19.0
ZnO				0–8.0
BaO			0.0–1.5	
CeO <sub>2</sub>			0.0–1.0	
TiO <sub>2</sub>			0.0–0.5	
Supplements			1.0–1.5	0–8.0

Croatia) was added and refill with redistilled water and well mixed. After 10 min, absorbance was measured at 640 nm.

### 2.5. Al<sup>3+</sup> elution measurement

One mL of tested solution, 2 mL of acetic buffer and 2 mL of aluminon were added to 50 mL flask. After 15 min absorbance was measured at 525 nm.

Mass concentration values for eluted ions were calculated using surface of the samples in  $\mu\text{g}/\text{cm}^2$ . Results were analyzed using descriptive statistics.

## 3. Results

Measured variations of the elution have been described by following descriptive statistics parameters: mean value, median, standard deviation, interquartile range, minimum, maximum, 25 and 75 percentiles. Mentioned parameters for

Table 3  
Chemicals

Chemicals	Manufacturer
CH <sub>3</sub> COOH conc., (NH <sub>4</sub> ) <sub>2</sub> MoO <sub>4</sub> , H <sub>2</sub> C <sub>2</sub> O <sub>4</sub> , KCN, Photo – Rex (4-(metilamino)-phenol sulphate), acetic puffer (CH <sub>3</sub> COONH <sub>4</sub> + CH <sub>3</sub> COOH), aluminon (Aurin – carbonic acid)	Kemika, Zagreb, Croatia
Standard elution Si, Al, Na, K, Mg	Merck, Darmstadt, Germany

sample groups are graphically shown on Figs. 1–4. The results for magnesium are not graphically shown, because the values lay beyond the level of detection. Fig. 5 shows the relation of the ion elution mean values for the samples.

The graph in Fig. 5 shows that IPS-Classic ceramics samples had highest elution values for sodium ( $1.1 \mu\text{g}/\text{cm}^2$ ), silicon ( $2.2 \mu\text{g}/\text{cm}^2$ ) and aluminium ( $2.7 \mu\text{g}/\text{cm}^2$ ), while Vitadur Alpha samples showed highest elution of potassium ions ( $1.3 \mu\text{g}/\text{cm}^2$ ). Vitadur Alpha samples leached more

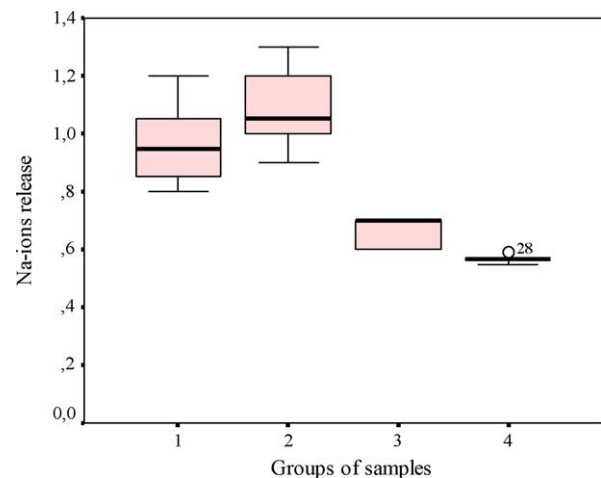


Fig. 1. Na-ions release ( $\mu\text{g}/\text{cm}^2$ ) by sample groups ((1) Vitadur alpha, (2) IPS-Classic, (3) IPS-Empress – layering technique and (4) IPS-Empress – (staining technique)). ONo = Outlier (box plots/Explore). Cases with values between 1.5 and 3 box lengths from the upper or lower edge of the box. The box length is the interquartile range. No-record number.

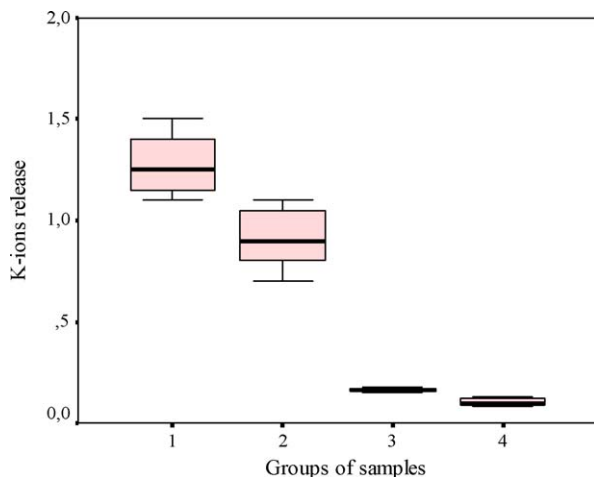


Fig. 2. K-ions release ( $\mu\text{g}/\text{cm}^2$ ) by sample groups ((1) Vitadur alpha, (2) IPS-Classic, (3) IPS-Empress – layering technique, (4) IPS-Empress – staining technique).

potassium than aluminium ions, which is not in accordance with its chemical composition (Table 2). Similarly, of all ions tested, IPS-Classic ceramics samples leached highest levels of aluminium ions, although they are present in much smaller amounts than silicon ions. IPS-Empress 2 glass-ceramics showed best results with lowest ion elution. Both IPS-Empress 2 sample groups showed sodium ion elution of  $0.6\text{--}0.7 \mu\text{g}/\text{cm}^2$ , while potassium and silicon ions were found in traces ( $0.1\text{--}0.4 \mu\text{g}/\text{cm}^2$ ). If we consider the chemical composition, results are somewhat unexpected, since glass-ceramics either have no, or have very small amounts of sodium according to the manufacturers' declaration of composition. Interestingly, glass-ceramics for layering technique did not show aluminium ion elution, while glass-ceramics for staining technique showed the highest alu-

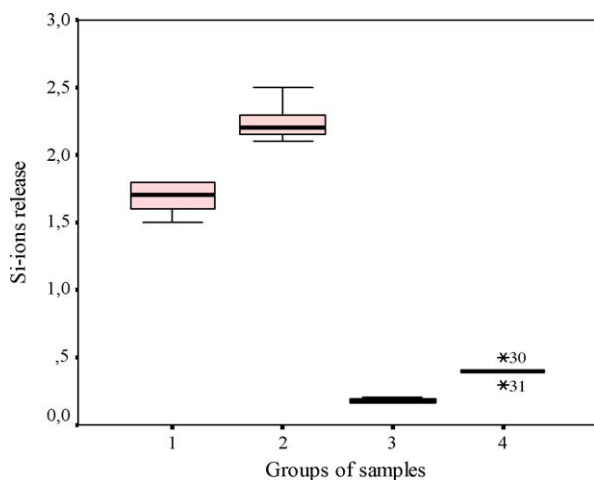


Fig. 3. Si-ions release ( $\mu\text{g}/\text{cm}^2$ ) by sample groups ((1) Vitadur alpha, (2) IPS-Classic, (3) IPS-Empress – layering technique and (4) IPS-Empress – staining technique). \*No = extremes (box plots) Cases with values more than 3 box lengths from the upper or lower edge of the box. The box length is the interquartile range. No = record number.

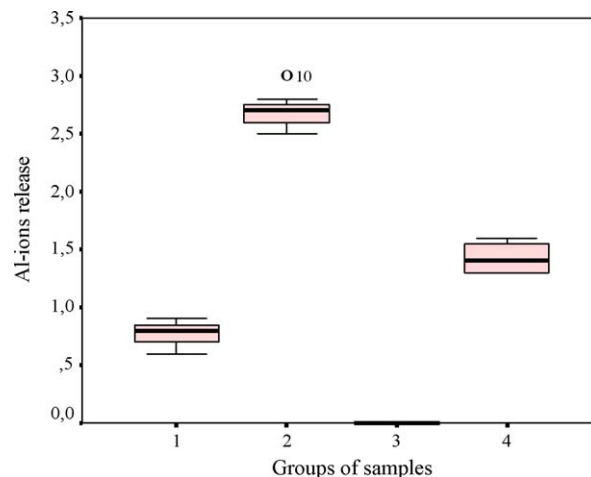


Fig. 4. Al-ions release ( $\mu\text{g}/\text{cm}^2$ ) by sample groups ((1) Vitadur alpha, (2) IPS-Classic, (3) IPS-Empress – layering technique, (4) IPS-Empress – staining technique). ONo = Outlier (box plots/explore). Cases with values between 1.5 and 3 box lengths from the upper or lower edge of the box. The box length is the interquartile range. No = record number.

minium ion elution ( $1.4 \mu\text{g}/\text{cm}^2$ ) in relation to other tested ions. These results did not correlate with composition of glass-ceramic samples (Table 2).

#### 4. Discussion

In this study we followed some propositions declared in ISO Standard 6872.<sup>11</sup> Except for this, acetic acid was used because of its pH value (pH 2.4). That pH value is similar to pH values of some refreshing drinks, juices and to values found in the dental plaque. Acetic acid is, also, the most frequently used acid for domestic purposes.<sup>3</sup> Duration of measurement and number of samples were also performed according to ISO standard. However, we did not measure the weight loss, but elution of elements after immersion in acetic acid. Živko-Babić et al.<sup>17</sup> and Jakovac et al.<sup>18</sup> conducted similar measurements on the feldspathic (IPS-Classic) and hydrothermal (DuceraGold) dental ceramic, testing their chemical stability during the 6-month immersion period in hydrochloric acid. The elution of  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$  ions, changes in pH value and loss of sample mass were measured. The elution of all ions was found to be highest during the first three months. After this period elution dropped drastically, and in case of  $\text{Mg}^{2+}$  ions stopped completely.

The present studies led to further investigations of the chemical stability of other dental ceramic materials, but also to modifications of the used method. Due to the differences in the medium and duration of the experiment, it is not possible to compare our results with those obtained from earlier studies, except from the fact that magnesium ions showed more inertness than other tested ions.

Results of this study showed highest ion elution levels for the IPS-Classic, feldspathic ceramic samples, while the most inert were glass-ceramic samples (IPS-Empress 2 ceramics

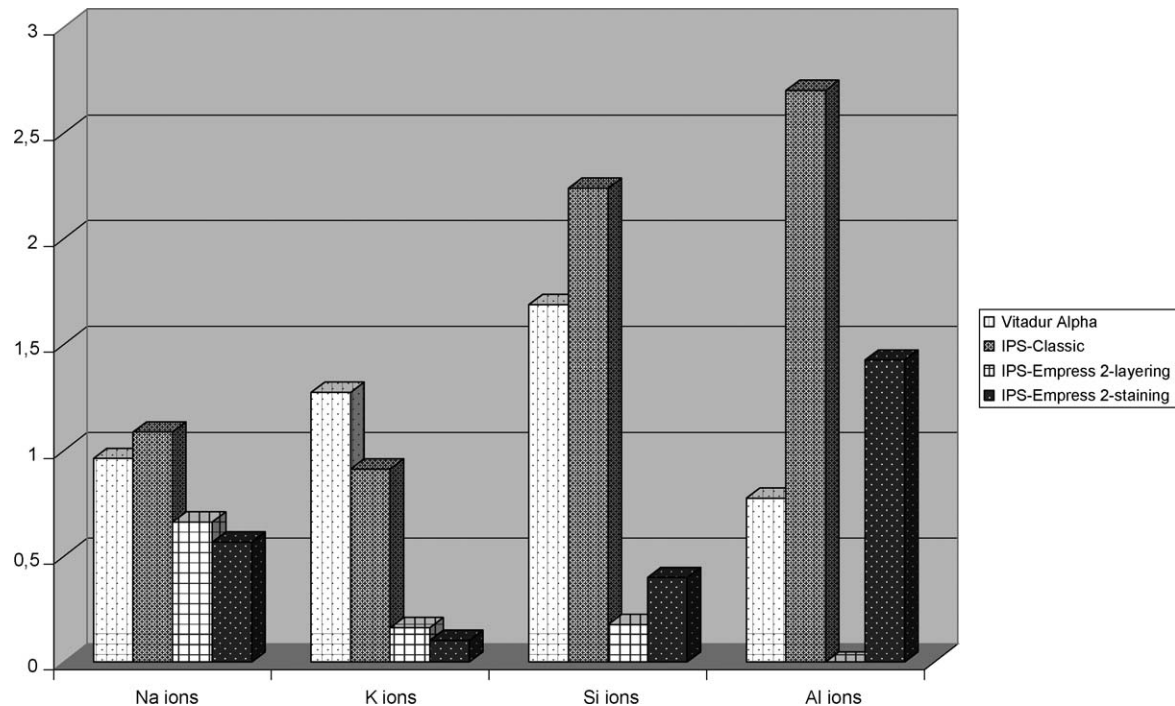


Fig. 5. Mean values of ions release ( $\mu\text{g}/\text{cm}^2$ ) by sample groups.

for layering and staining). Extent of the ion elution was not proportional to the amount of ions in the samples. Such relationship was observed for all samples tested, especially in the glass–ceramics group where both groups of samples elute relatively high amounts of sodium ions, contrary to the composition, which lists no or very low levels of sodium. Observed higher levels of alkali ions are in accordance with published data, revealing diffusion of water molecules into the glass during glass corrosion processes. Water molecules react with free oxygen ions forming hydroxyl ions, which elute together with alkali ions in order to retain the neutrality.<sup>19</sup> Compared to acid solutions, process of corrosion and ion exchange runs significantly slower in water, while corrosion of ceramics is brought through translation of mentioned metal ions into the solution. Presence of alkali oxides in the ceramics changes the chemical stability towards mineral acids. Ceramics containing CaO is the most unstable one. Degradation of dental silicate ceramic materials takes place mainly due to corrosion of silicates, or glass, through selective release of alkali ions (alkali silicate glasses) and dissolution of glass structural net. At pH values of 9 or less, alkali ions release is the dominant mechanism of degradation.<sup>2,20</sup>

Aluminium ions show largest variability, namely IPS-Empress 2 for layering releases no aluminium ions, while the immersion solution of IPS-Empress 2 for staining has highest levels of aluminium ions, of all ions tested. Considering inertness of samples and the fact that same glazed was used, the degree of difference is somewhat surprising, although present to some extent in the declared composition of these dental ceramic materials.

As already mentioned, it is important to study chemical degradation and corrosion products of dental ceramics due to the possible toxic effect of some elements. When the toxic effect is considered, biocompatibility of the lithium-based glass–ceramics could be questionable.<sup>2,21,22</sup> Anusavice and Zhang<sup>23</sup> studied chemical stability of three lithium-based glass–ceramics and found highest weight loss of samples at pH 11, value almost impossible to reach in vivo. From toxicological point of view, maximal  $\text{Li}^+$  release value from 28 crowns at  $80^\circ\text{C}$  equals 1.2 mg. This value is somewhat lower than the accepted daily doses acquired through food-intake of 2.0 mg.

There is a few literature data relating to toxic influence of dental ceramics on other organs, or organism in general. Persson-Sjögren and Sjögren<sup>24</sup> proved statistically significant rise in levels of insulin release from the Langerhans cells, after insertion of the Empress ceramics. Other tested dental ceramic materials failed to reveal any influence on the insulin release levels, except for Vita VMK 95 dental ceramic (VITA Zahnfabrik, Bad Säckingen, Germany), which lowered the release. The question remains which components of the IPS-Empress or Vita VMK 95 dental ceramic caused changes in insulin release levels, and what concentrations might cause such adverse effects. The danger lies in overlooking the possibility that minimal amounts of ions eluted due to chemical or mechanical wear, might adversely affect the pancreas, or other organs or tissues.

Recent study by Messer et al.<sup>25</sup> confirm the need for detailed analysis of biocompatibility for dental ceramic materials. They also showed that IPS-Empress 2 ceramics can lower



cell activity, and according to common empirical standards for dental alloys and composites, such a material would not be biologically acceptable. Further analysis of ion elution for, as yet untested ions like lithium, might provide further insight in this regard. Sundar and Amber<sup>26</sup> measured elution of sodium, potassium, calcium, and magnesium oxides from different dental ceramic materials and found the amount not significant in relation to their respective amounts ingested through food intake. Schäfer and Kapper<sup>27</sup> conducted an investigation very similar to ours, and measured ion elution levels for sodium, calcium, magnesium, aluminium, silicon and iron in 14 different dental ceramic materials. They concluded that modern ceramic systems show higher chemical stability than traditional systems and lower levels of ion elution than requested by the ISO-standard.<sup>11</sup> Our investigation included different dental ceramic materials, but the results were in accordance with their conclusions.

Measuring ions elution from different dental ceramic has to be continued and expand to other declared elements especially those with potential toxicity (Lithium, etc.) or with any other possible influence on peoples health.

## 5. Conclusions

1. The highest ion elution was shown for IPS-Classic ceramics (feldspathic ceramic), and the lowest for IPS-Empress 2 ceramics (glass–ceramic).
2. The release of the ions from the analyzed specimens did not correlate with the values of the composition declared by the manufacturer.
3. Results are in accordance with the glass corrosion and silicate-based dental ceramics theories.
4. The ion elution values were toxicologically not significant.
5. Further corrosion analysis has to be continued, regarding the eluted ions (Li<sup>+</sup> release for example) and their potential cytotoxicity.

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