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An *in vitro* examination of fluoride ions release from selected materials – resin-modified glass-ionomer cement (Vitremer) and nanohybrid composite material (Tetric EvoCeram)

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The aim of this study was to examine a short-term fluoride ions release from selected materials – resin-modified glass ionomer –Vitremer (3M ESPE) and nanohybrid universal composite – Tetric EvoCeram (IvoclarVivadent). Release of fluoride ions [μg/mm^2/h] from Tetric EvoCeram and Vitremer into nine environments (artificial saliva – AS, deionized water and 0.9% NaCl) differing in composition of the solution and pH was determined. Six samples were prepared for each solution. In the short-term study, the measurements were taken after 1, 3, 24, 48, 72 and 168 hours. The cumulative values as well as levels of fluoride ions released at concrete time intervals were compared. Within 7 days (168 hours), both materials showed variable levels of fluoride ions release. The highest value of fluoride ions release from nanohybrid Tetric EvoCeram material was reported in deionized water (8) after 24 hours (1.550 ± 0.014 [μg/mm^2/h]) and the lowest value was read in the artificial saliva AS pH 7.5 (5) after 1 hour (0.022 ± 0.001 [μg/mm^2/h]). What's more, the highest value of F^- release from Vitremer was found in deionized water (8) after 168 hours of immersion (24.021 ± 2.280 [μg/mm^2/h]) and the lowest value was in the artificial saliva AS (without Ca^{2+}) pH 4.5 (6) (0.303 ± 0.249 [μg/mm^2/h]) after 168 hours. Cumulated release of F^- after 7 days was notably higher from resin- modified glass ionomer material – Vitremer in all artificial saliva solutions (1–7) which imitated the environment of oral cavity. Therefore, we can assume that Vitremer has better remineralization potential and it may constitute a more effective method of tooth decay prevention.

Key words: *fluoride ions release, nanohybrid composites, microhardness, resin-modified glass ionomers, tooth decay prevention*

1. Introduction

Oral diseases are one of the most widespread conditions globally. They constitute significant economic and health difficulties by diminishing quality of life for affected people. The most prevalent and substantial diseases are: caries, periodontal diseases, cancers of lips as well as oral cavity malignancies [27]. In Figure 1A, common oral conditions are presented.

Tooth decay is one of the most common noncommunicable diseases worldwide. It affects about 50% of children across the globe [22]. Despite the fact that oral conditions are in many cases preventable, they occur with a high frequency in low-income and middle-income countries as a reflection of social and economic inequalities [27]. Staszczyk et al. [36] in their

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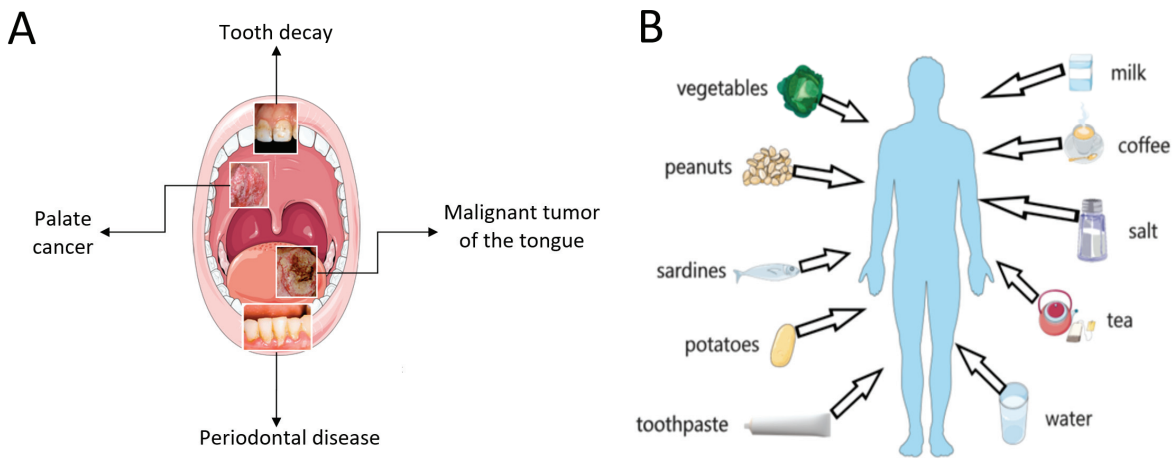

Fig. 1. A) Diagram showing the most common diseases of the oral cavity, B) The sources of fluorides for the human organism

study examined prevalence and severity of caries in the dentition of 5-, 7-, 12-year old patients from the province of Małopolskie. In the group of 12-year-olds, they observed statistically significant higher prevalence of caries among the residents of rural areas – 92.31%, whereas prevalence of tooth decay among residents of cities was determined as 72.73%.

According to WHO, dental decay is a local pathological process, which leads to decalcification of the enamel and dentin, then decomposition of hard tissues of the tooth and, as a consequence, formation of a dental cavity. Oral diseases constitute a major health problem, causing discomfort, defacement and sometimes death [22]. Caries prevention knowledge, healthy behaviour and socioeconomic conditions are the factors responsible for initiation and progression of dental caries [27]. It is commonly known that prevention is better than cure. Treatment of tooth decay is repeatedly more intricate and expensive. Early symptoms of caries as well as habits leading to tooth decay can be easily detected by pediatricians or physicians. It is essential to maintain good oral hygiene and a healthy diet with reduced amount of sugar to prevent oral diseases [22]. Fluoride, an essential element for humans, can be ingested through the food we eat or the air we breathe. While rare, it can also permeate the skin's layers and enter the body. Approximately half of the fluoride ingested is eliminated from the body within 24 hours, with most of it being excreted in urine. However, it can also be found in sweat, saliva, milk and feces [16], [19], [34]. In Figure 1B, fluoride supply sources are presented.

As a general point, it should be stressed that fluorides play a major role in tooth decay prevention. Fluoride ions (F^- ions) can influence caries processes in several ways, related to local and systemic effects. [19]. Fluorides may act topically and systemically. We may differentiate several ways F^- ions affect the carious process. Fluorides reduce acid production by bacteria due to the inhibition of enolase, which is a glycolytic enzyme that turns 2-P-glycerate to P-enolpyruvate (PEP). The reduction of PEP causes retardation of sugar transport. Moreover, fluoride ions inhibit the membrane bound, proton pumping H+/ATPase and, therefore, they affect appropriate functioning of bacteria. Thus, they limit the growth of microorganisms in dental plaque [11]. Furthermore, constant supply of low fluoride values in dental plaque/saliva/dental interference are considered as the most valuable in the caries prevention [32]. Persistent exposure to low fluorides concentration leads to remineralization of enamel and inhibition of demineralization of hard dental tissues [35]. The remineralization process of initial caries is expedited by the attendance of fluorides, but also requires calcium and phosphate in the saliva [5]. Therefore, dental restorative materials, which release fluoride ions may be used as tooth decay prevention [7], [8], [15], [17], [19], [31], [40]. Glass ionomer cements and composites are one of those materials with proven abilities of fluoride ions release [4], [23], [30], [33]. In our study, Vitremer, as a representative of resin-modified glass ionomers, showed notably higher cumulated release of F^- than Tetric EvoCeram in all nine (1–9) studied solutions. However, it should be noted that carried out studies show that levels of fluoride ions released from glass ionomers varies due to the category of those materials – conventional and resin-modified [12], [13]. Compared to standard resin-modified glass ionomer cements, Vitremer demonstrates 3 types of curing – apart from acid – base reaction, we may also distinguish unique light and dark free radical cure reactions. In contrast to Vitremer, Tetric EvoCeram as nanohybrid composite material, shows only light-curing polymerization. Nanosized materials are both natural and syn-

thetic materials with the size of components <100 nm in at least one dimension [18], [24]. Magnetic, electrical and optical properties of nanomaterials are altered. Nanocomposites have similar or even better finishing, matching of the shades, hardness and flexural strength than conventional composites [2]. Nanotechnology offers a wide range of improvements in both prevention and treatment of oral conditions – hypersensitivity and dental remineralization [24].

The aim of the study was to appraise the fluoride ions released from selected materials – resin-modified glass ionomer cement (Vitremer) and nanohybrid composite material (Tetric EvoCeram) over a period 1 week (168 hours) into nine environments – differing in composition and pH of the solution (1 – artificial saliva pH 4.5; 2 – artificial saliva pH 5.5; 3 – artificial saliva pH 6.0; 4 – artificial saliva pH 7.0; 5 – artificial saliva pH 7.5; 6 – artificial saliva without Ca^{2+} pH 4.5; 7 – artificial saliva without Ca^{2+} pH 5.5; 8 – deionized water; 9 – saline solution – 0.9% sol. NaCl). Artificial saliva imitated natural conditions of the oral cavity. Deionized water and 0.9% sol. NaCl were used to compare the release of fluoride ions in neutral conditions.

2. Materials and methods

Material study consists of 2 different materials: Tetric EvoCeram and Vitremer. Tetric EvoCeram (Ivoclar -Vivadent, Schaan, Liechtenstein) is a radiopaque, light-curing, nanohybrid composite that has 79–80% content of inorganic fillers (by weight) or 60–61% (by volume) for the Bleach shades, for all the other shades it has 75–76 wt. % or 53–55 vol. %. The size of particles of the inorganic fillers oscillates between 40 and 3000 nm (the average size is 550 nm). Dimethacrylates constitute the monomer of inorganic matrix (17–18% by weight). The remaining fillers are barium glass, ytterbium trifluoride, copolymers (82–83% by weight) and mixed oxide. Additional ingredients – stabilizers, initiators, additives and pigments constitute less than 1% by weight. Tetric EvoCeram is indicated for anterior restorations (Classes III*, IV*), restorations in the posterior region (Classes I* and II*), veneering of discoloured anterior teeth, extended fissure sealing in premolars and molars, root erosion, as well as cervical caries (*Black's Classification of Dental Caries).

Vitremer (Core Buildup/Restorative, 3M ESPE, Neuss, Germany) is a Tri-Cure Glass Ionomer System. Vitremer core buildup/restorative consists of powder and liquid components. Powder is composed of fluoroaluminosilicate (FAS) glass, microencapsulated potassium persulfate and slight amounts of pigments. The glass ionomer liquid consists of aqueous polycarboxylic acid, which is modified with methacrylate groups, copolymer, water, photoinitiators and HEMA. The composition of Vitremer liquid is similar to Vitrebond™ Liquid. However, they have different concentrations of the constituents.

Vitremer is recommended to use with a special primer, which is composed of Vitrebond copolymer, ethanol, photoinitiators and HEMA. It is applied for 30 seconds and then light cured for 20 seconds. The main function of the primer is to wet the tooth surface and to modify the smear layer. To improve esthetics of the restoration it is also recommended to use the Vitremer finishing gloss, which is a light cure dental resin.

According to manufacturer's studies, Vitremer shows three separate curing processes:

1. Acid-base reaction (between the FAS glass and the acid groups of the polymer).
2. Free radical light cure reaction (between HEMA and methacrylate groups of the polymer).
3. Free radical dark cure reaction (it is commenced by a water-activated redox catalyst and it provides a proper set of the material where light does not percolate).

Vitremer may be utilize as an esthetic Class III* and V* restorations, restorations of cervical erosion and abrasion lesions. It can be also used in deciduous teeth in Class I* and II* restorations, as a temporary repair of fractured teeth and to fill defects and undercut areas in crown preparations (*Black's Classification of Dental Caries).

The cylinder-shaped samples were prepared in a special form of a pellet, 2 mm thick and 5 mm in diameter. The mass of the samples was about 0.5 g. Specimens were cured with a use of the Bluephase Style 20i lamp (IvoclarVivadent, Schaan, Liechtenstein), which is suitable for the polymerization of all light-curing dental materials, emitting light in the wavelength range of 385–515 nm. Figure 2A presents the Teflon form and Fig. 2B shows the mold filled with prepared dental materials.

Tests of physical properties such as hardness and stiffness were carried out using the CSM MicroCombi Tester using a Vickers (HV) indenter. Each of the samples was cold-inked in Durakryl Plus (Spofa Dental, Czech Republic) acrylic adhesive and measured. During the measurement, the indenter was introduced perpendicularly to the surface of the samples with a force of 400 mN, deforming them for a period of 10 s. The meas urement made it possible to determine such parameters as hardness (HV), instrumental Young's mod-

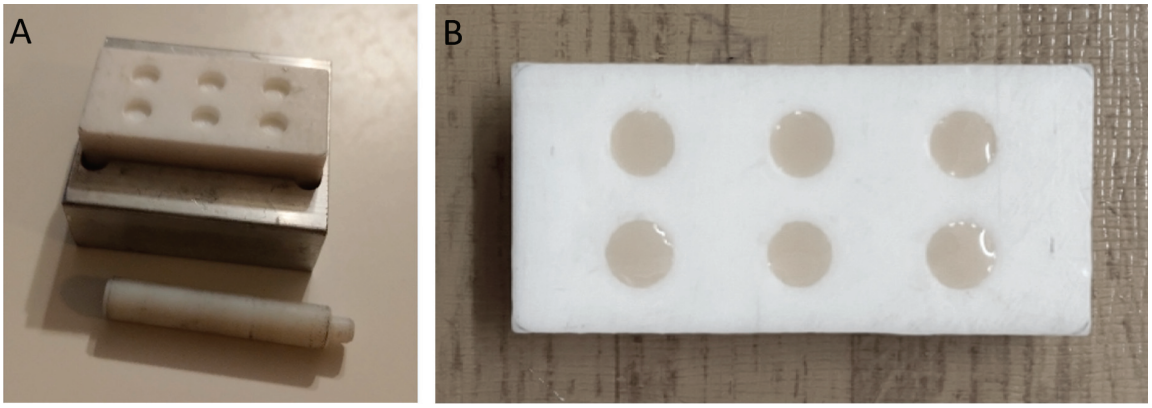

Fig. 2. A) The Polytetrafluoroethylene (PTFE) mold used to prepare cylindrical measuring samples, B) with samples

ulus (EIT), elastic and plastic energy determined in accordance with the method proposed by Oliver and Pharr.

Fluoride release test

Afterwards, they were immersed in the studied solutions (artificial saliva, differing in the composition and pH, deionized water and 0.9% NaCl sol.) and left in closed containers at 37 °C without mixing. The temperature of our studied solutions imitated the average normal body temperature. Six samples were prepared for each solution. Each sample was examined three times and an average value was calculated based on the three results. The release of fluoride ions from Tetric EvoCeram and Vitremer into nine environments was examined for 7 days (168 hours) at specific time intervals with a use of an ORION 9609 ion-selective electrode (Thermo Fisher Scientific Co., Waltham, MA, USA) connected to a pH/ion meter CPI-551 Elmetron microcomputer. The system was calibrated before each consecutive examination. The measurement was repeated three times and the mean value was established. Fluoride ions release was tested at the following time intervals: 1, 3, 24, 48, 72, 168 hours. The cumulative values as well as the levels of F^- released at specific time intervals were determined. The samples that were submerged in the solution and sealed were depicted in Fig. 3.

Statistical analyses were conducted using Statistica v.13.3 (Tibco Software Inc. CA, USA). All experiments were done six times and descriptive data were expressed as the mean and standard deviation (±SD). Comparisons of continuous data between more than

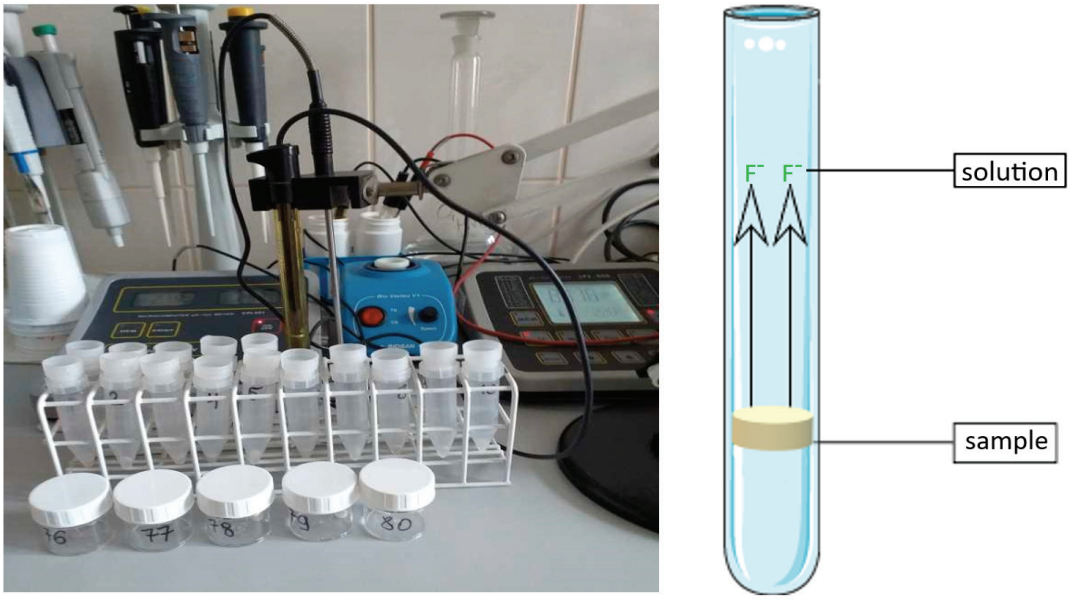

Fig. 3. Laboratory stand for measuring the release of fluorine (F^-) in liquids with different pH values, together with measuring samples

two dependent groups were performed by ANOVA analysis for dependent samples and Tukey's post-hoc test. Correlation between time of incubation and release of fluoride ions from study materials was calculated by Pearson's test. A 2-tailed *p*-value of $p < 0.05$ was considered statistically significant.

To examine the surface before and after fluoride release, scanning electron microscopy (SEM) investigation was performed on FEI Nova NanoSEM 230 microscope. The magnification was set to 2500×.

3. Results

Physicochemical assay results

Microhardness tests in which hardness, stiffness and deformation energy values were determined were carried out on the included samples ($N = 8$), for which 3 measurements were made.

The obtained results indicate that Vitremer is characterized by both a higher value of HV hardness and stiffness (EIT) (Fig. 4, Table 1). Nevertheless, this material has a statistically significant lower energy value in the elastic section.

Tetric EvoCeram

Exhibition of fluoride ions from Tetric EvoCeram into nine environments – artificial saliva with different pH and composition, deionized water and saline solution is presented in Table 2.

The highest F^- ions release from Tetric EvoCeram was read in deionized water (8) after 24 hours and it was 1.550 ± 0.014 [µg/mm^2/h] whereas the lowest value was found in artificial saliva (AS) pH 7.5 (5) after 1 hour and it was 0.022 ± 0.001 [µg/mm^2/h]. Among the solutions of artificial saliva with Ca^{2+} (1–5), the highest level of fluoride ions was determined in pH 6.0 (3) after 1 hour (1.208 ± 0.107 [µg/mm^2/h]). The lowest value among the solutions of artificial saliva (1–5) was found in pH 7.5 (5) after 1 hour (0.022 ± 0.001 [µg/mm^2/h]).

Taking into consideration solutions of artificial saliva (AS) without Ca^{2+} ions (6, 7), the highest F^- ions was

Table 1. Comparison of mean values of the microhardness (HV), measured and Young's modulus (EIT) for Tetric EvoCeram and Vitremer. Descriptive data were presented as mean ± standard deviation (±SD)

Time [hours]	HV	HIT [MPa]	EIT [GPa]	W_elastic [nJ]	W_plastic [nJ]	W_total [nJ]
Tetric EvoCeram	52.69 ± 3.37	582.11 ± 39.57	12.36 ± 0.49	313.9 ± 9.52	638.20 ± 29.50	952.1 ± 31.62
Vitremer	54.36 ± 2.21	564.91 ± 34.10	16.79 ± 0.44	223.8 ± 3.98	810.6 ± 30.14	1036.0 ± 28.93

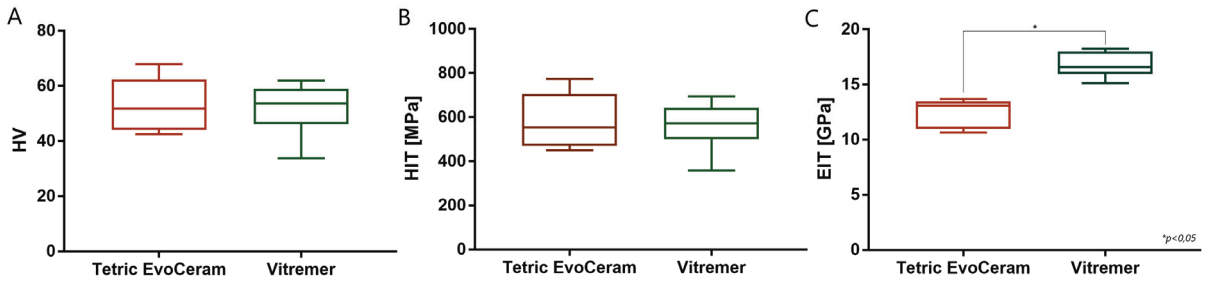

Fig. 4. Comparison of A) hardness (HV), B) instrumental hardness (HIT) and C) instrumental Young's modulus values obtained for Tetric EvoCeram and Vitremer materials; $p < 0.05$

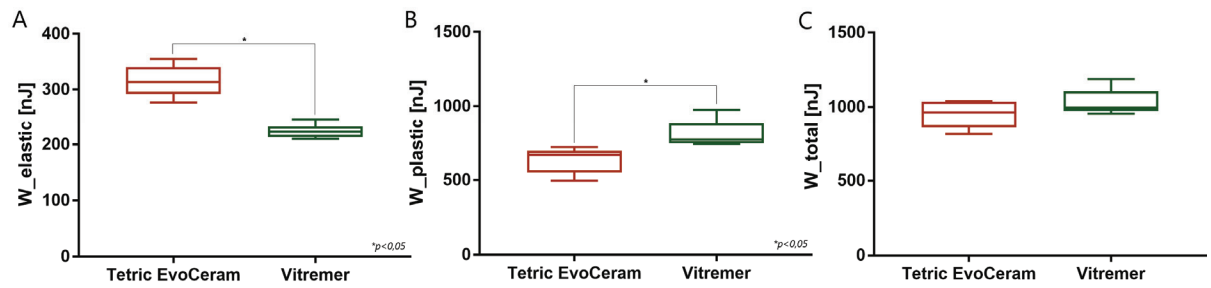

Fig. 5. Comparison of elastic (W_elastic), plastic (W_plastic) and total values of deformation energy (W_total) obtained for Tetric EvoCeram and Vitremer materials; $p < 0.05$

Table 2. Release of fluoride ions [μg/mm^2/h] from Tetric EvoCeram into nine environments differing in composition of the solution and pH. Six samples were prepared for each solution. Descriptive data were presented as mean ± standard deviation (±SD)

Time [hours]	AS pH 4.5 (1) [μg/mm^2/h]	AS pH 5.5 (2) [μg/mm^2/h]	AS pH 6.0 (3) [μg/mm^2/h]	AS pH 7.0 (4) [μg/mm^2/h]	AS pH 7.5 (5) [μg/mm^2/h]	AS–Ca^{2+} pH 4.5 (6) [μg/mm^2/h]	AS–Ca^{2+} pH 5.5 (7) [μg/mm^2/h]	Deionized H_2O (8) [μg/mm^2/h]	0.9% NaCl (9) [μg/mm^2/h]
1	0.660 ± 0.089	0.783 ± 0.084	1.208 ± 0.107	0.450 ± 0.119	0.022 ± 0.001	0.298 ± 0.064	0.105 ± 0.020	0.357 ± 0.021	0.436 ± 0.030
3	0.565 ± 0.047	0.758 ± 0.058	0.855 ± 0.029	0.548 ± 0.084	0.303 ± 0.021	0.200 ± 0.052	0.456 ± 0.268	0.658 ± 0.035	0.581 ± 0.056
24	0.523 ± 0.076	0.845 ± 0.084	0.607 ± 0.115	0.463 ± 0.026	0.187 ± 0.022	0.363 ± 0.010	0.221 ± 0.077	1.550 ± 0.014	0.988 ± 0.460
48	0.570 ± 0.023	0.518 ± 0.208	0.237 ± 0.048	0.420 ± 0.054	0.197 ± 0.017	0.200 ± 0.044	0.333 ± 0.070	1.221 ± 0.039	0.940 ± 0.199
72	0.933 ± 0.177	1.013 ± 0.352	0.378 ± 0.041	0.335 ± 0.031	0.181 ± 0.007	0.193 ± 0.031	0.328 ± 0.102	1.203 ± 0.112	0.481 ± 0.117
168	0.360 ± 0.022	0.978 ± 0.277	0.552 ± 0.711	0.477 ± 0.088	0.136 ± 0.046	0.235 ± 0.044	0.171 ± 0.028	1.295 ± 0.165	1.250 ± 0.014
Mean ± SD	0.601 ± 0.195	0.816 ± 0.254	0.639 ± 0.425	0.449 ± 0.088	0.171 ± 0.087	0.248 ± 0.075	0.269 ± 0.165	1.047 ± 0.420	0.779 ± 0.361
p-value (ANOVA for dependent samples)	<0.0001	0.0041	<0.0001	0.0014	<0.0001	<0.0001	0.0005	<0.0001	<0.0001
post-hoc Tukey's test	$p < 0.05^{\#}$ for: 1 vs. 72 1 vs. 168 3 vs. 72 3 vs. 168 24 vs. 72 24 vs. 168 48 vs. 72 48 vs. 168 72 vs. 168	$p < 0.05^{\#}$ for: 48 vs. 72 48 vs. 168	$p < 0.05^{\#}$ for: 1 vs. 24 1 vs. 48 1 vs. 72 1 vs. 168	$p < 0.05^{\#}$ for: 3 vs. 72 72 vs. 168	$p < 0.05^{\#}$ for: 1 vs. all groups 3 vs. all groups 24 vs. 168 48 vs. 168 72 vs. 168	$p < 0.05^{\#}$ for: 1 vs. 3 1 vs. 48 1 vs. 72 3 vs. 24 24 vs. 48 24 vs. 72 24 vs. 168	$p < 0.05^{\#}$ for: 1 vs. 3 1 vs. 48 1 vs. 72 3 vs. 24 3 vs. 168	$p < 0.05^{\#}$ for: 1 vs. all groups 3 vs. all groups 24 vs. all groups	$p < 0.05^{\#}$ for: 1 vs. 24 1 vs. 48 1 vs. 168 3 vs. 24 3 vs. 168 24 vs. 72 72 vs. 168

AS – artificial saliva, AS – Ca^{2+} (artificial saliva without Ca^{2+}), $^{\#}$ – groups were numbered according to time of incubation.

read in pH 5.5 (7) after 3 hours (0.456 ± 0.268 [μg/mm^2/h]), the lowest in pH 5.5 (7) after 1 hour (0.105 ± 0.020 [μg/mm^2/h]). In deionized water (8) the highest amount of F^- ions was determined after 24 hours (1.550 ± 0.014 [μg/mm^2/h]), whereas the lowest after 1 hour (0.357 ± 0.021 [μg/mm^2/h]).

In the saline solution – 0.9% NaCl (9) exhibition of fluoride ions was the highest after 168 hours (1.250 ± 0.014 [μg/mm^2/h]), the lowest level was read after 1 hour (0.436 ± 0.030 [μg/mm^2/h]). Taking into account the content of fluoride ions released from the samples incubated in all seven artificial saliva solutions (1–7), the highest value was noted in artificial saliva (AS) pH 6.0 (3) (1.208 ± 0.107 [μg/mm^2/h]) after 1 hour, while, on the contrary, the lowest value was found in artificial saliva (AS) pH 7.5 (5) (0.022 ± 0.001 [μg/mm^2/h]) after 1 hour.

The highest mean ± standard deviation (SD) was determined in deionized water (8) (1.047 ± 0.420 [μg/mm^2/h]). Moreover, the lowest mean ± standard deviation (SD) was observed in the artificial saliva pH 7.5 (5) (0.171 ± 0.087 [μg/mm^2/h]). Mean values ± standard deviation (SD) in the solutions of artificial saliva AS –Ca^{2+} pH 4.5 (6) (0.248 ± 0.075 [μg/mm^2/h]) and artificial saliva AS – Ca^{2+} pH 5.5 (7) (0.269 ± 0.165 [μg/mm^2/h]) were comparable.

The *p*-value (ANOVA for dependent samples) in the case of the artificial saliva AS pH 5.5 (2) was estimated as 0.0041, for the artificial saliva AS pH 7.0 (4) it was 0.0014 and for the artificial saliva AS – Ca^{2+} pH 5.5 (7) it was 0.0005. In contrast to the samples included in solutions mentioned above, *p*-value for the samples in the rest tested environments (1, 3, 5, 6, 8, 9) was determined as <0.0001, however the results were not considered as statistically significant.

Cumulated exhibition of fluoride ions from Tetric EvoCeram is presented in Table 3 and Fig. 6.

Table 3. Cumulated release of fluoride ions (CRFI) [µg/mm^2] from Tetric EvoCeram into nine environments differing in composition of the solution and pH. Six samples were prepared for each solution. Descriptive data were presented as mean ± standard deviation (±SD)

Time [hours]	AS pH 4.5 (1) [µg/mm^2]	AS pH 5.5 (2) [µg/mm^2]	AS pH 6.0 (3) [µg/mm^2]	AS pH 7.0 (4) [µg/mm^2]	AS pH 7.5 (5) [µg/mm^2]	AS–Ca^{2+} pH 4.5 (6) [µg/mm^2]	AS–Ca^{2+} pH 5.5 (7) [µg/mm^2]	Deionized H_2O (8) [µg/mm^2]	0.9% NaCl (9) [µg/mm^2]
1	0.660 ± 0.089	0.783 ± 0.084	1.208 ± 0.107	0.450 ± 0.119	0.022 ± 0.001	0.298 ± 0.064	0.105 ± 0.020	0.357 ± 0.021	0.436 ± 0.030
3	1.225 ± 0.136	1.542 ± 0.142	2.063 ± 0.136	0.998 ± 0.203	0.325 ± 0.022	0.498 ± 0.116	0.562 ± 0.288	1.015 ± 0.056	1.018 ± 0.086
24	1.748 ± 0.212	2.387 ± 0.226	2.670 ± 0.251	1.462 ± 0.229	0.512 ± 0.044	0.862 ± 0.126	0.783 ± 0.365	2.565 ± 0.070	2.007 ± 0.546
48	2.317 ± 0.235	2.905 ± 0.434	2.907 ± 0.299	1.882 ± 0.283	0.709 ± 0.061	1.062 ± 0.170	1.117 ± 0.435	3.787 ± 0.109	2.947 ± 0.745
72	3.250 ± 0.412	3.918 ± 0.786	3.285 ± 0.340	2.217 ± 0.314	0.890 ± 0.068	1.255 ± 0.201	1.445 ± 0.537	4.990 ± 0.221	3.428 ± 0.862
168	3.610 ± 0.434	4.897 ± 1.063	3.837 ± 1.051	2.693 ± 0.402	1.027 ± 0.114	1.490 ± 0.245	1.617 ± 0.565	6.285 ± 0.386	4.678 ± 0.876
Correlation (Pearson test)	$r = 0.897$, $p = 0.015$	$r = 0.930$, $p = 0.007$	$r = 0.863$, $p = 0.027$	$r = 0.895$, $p = 0.016$	$r = 0.859$, $p = 0.028$	$r = 0.890$, $p = 0.017$	$r = 0.863$, $p = 0.027$	$r = 0.919$, $p = 0.009$	$r = 0.932$, $p = 0.007$

AS – artificial saliva, AS – Ca^{2+} (artificial saliva without Ca^{2+}).

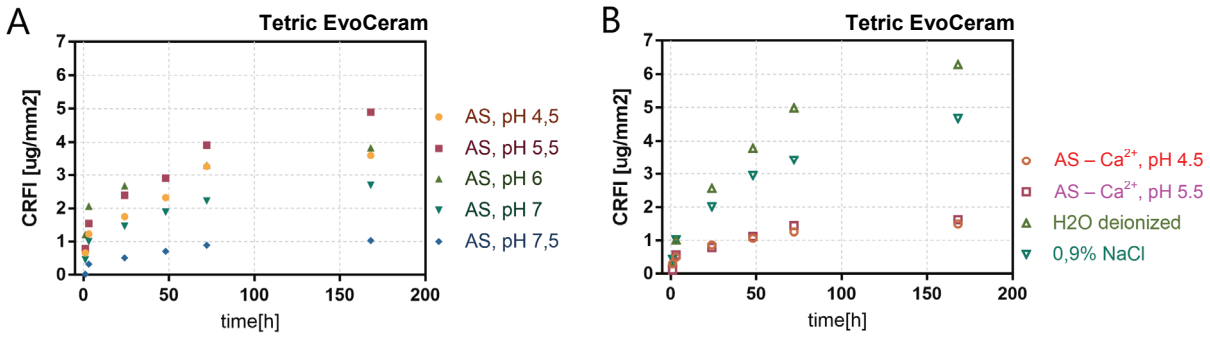

Fig. 6. Cumulated release of fluoride ions (CRFI) [µg/mm^2] from Tetric EvoCeram into nine different solutions. Points represent means of measurements in time periods. AS – artificial saliva

The highest level of cumulative release from the Tetric EvoCeram samples incubated in all nine environments (1–9) was observed in deionized water (8) (6.285 ± 0.386 [µg/mm^2]) after 168 hours. The lowest level of cumulative release from the samples immersed in all nine solutions (1–9) was found in artificial saliva (AS) pH 7.5 (5) (0.022 ± 0.001 [µg/mm^2]) after 1 hour.

The limit values of cumulative fluoride ions release from Tetric EvoCeram were presented in the following sequence:

- After 1 hour, the highest cumulative release was read in artificial saliva (AS) pH 6.0 (3) (1.208 ± 0.107 [µg/mm^2]) whereas the lowest was noted in artificial saliva (AS) pH 7.5 (5) (0.022 ± 0.001 [µg/mm^2]);
- After 3 hours, the highest cumulative release was observed in artificial saliva (AS) pH 6.0 (3) (2.063 ± 0.136 [µg/mm^2]), the lowest value was read in artificial saliva (AS) pH 7.5 (5) (0.325 ± 0.022 [µg/mm^2]);
- After 24 hours, the highest cumulative release was obtained in artificial saliva (AS) pH 6.0 (3) (2.670 ± 0.251 [µg/mm^2]), the lowest was found in artificial saliva (AS) pH 7.5 (5) (0.512 ± 0.044 [µg/mm^2]);
- After 48 hours, the highest cumulative release was noticed in deionized water (8) (3.787 ± 0.109 [µg/mm^2]), the lowest was found in artificial saliva (AS) pH 7.5 (5) (0.709 ± 0.061 [µg/mm^2]);
- After 72 hours, the highest cumulative release was observed in deionized water (8) (4.990 ± 0.221 [µg/mm^2]), the lowest was in artificial saliva (AS) pH 7.5 (5) (0.890 ± 0.068 [µg/mm^2]);
- After 168 hours, the highest cumulative release was obtained in deionized water (8) (6.285 ± 0.386 [µg/mm^2]), the lowest was in artificial saliva (AS) pH 7.5 (5) (1.027 ± 0.114 [µg/mm^2]).

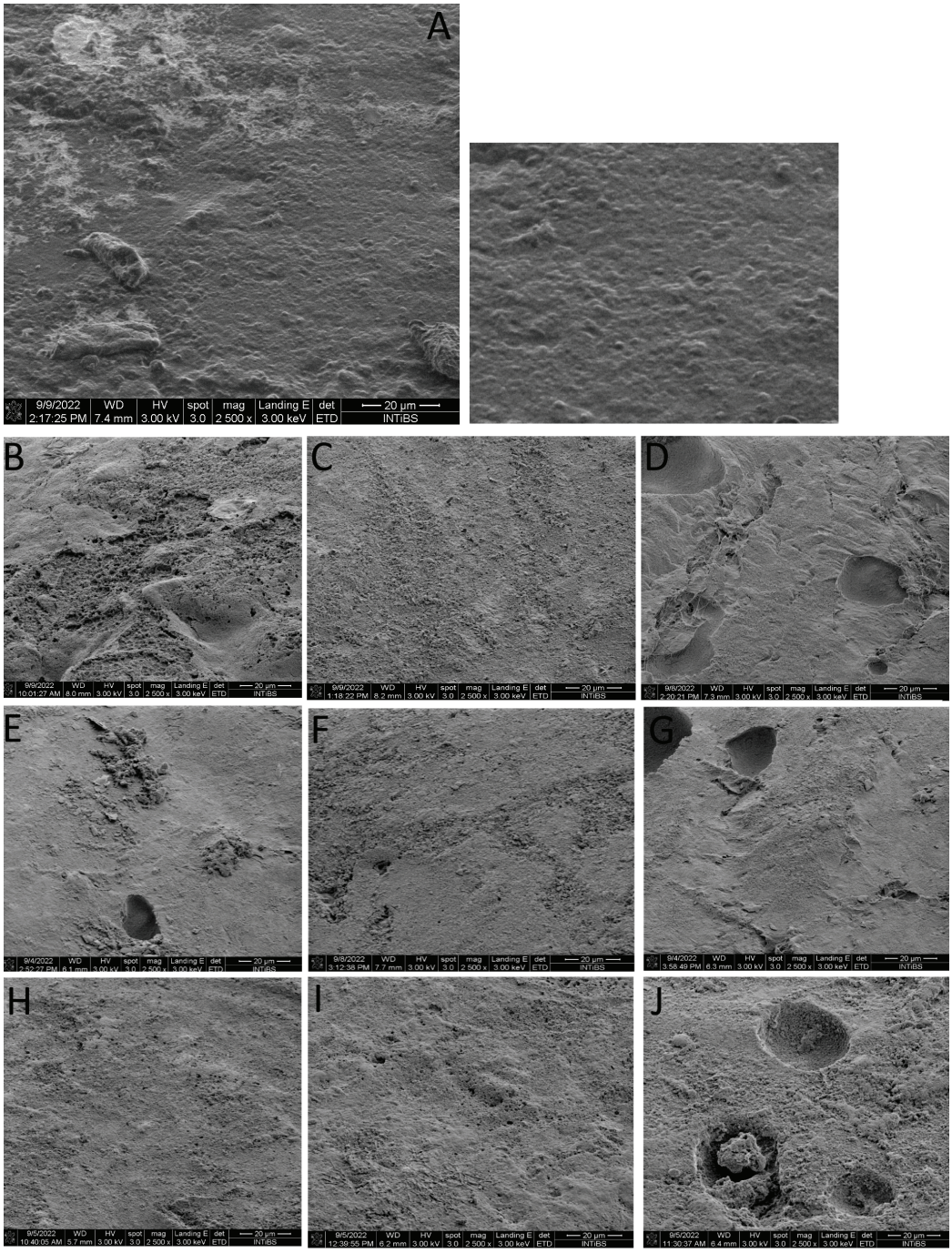

Fig. 7. SEM micrographs of A) pure Tetric EvoCeram sample and after immersion in B) H_2O, C) NaCl, D) AS pH 4.5, E) AS – Ca^{2+} pH 4.5, F) AS pH 5.5, G) AS – Ca^{2+} pH 5.5, H) AS pH 6.0, I) AS pH 7.0 and J). AS pH 7.5

Taking all seven artificial saliva solutions (1–7) into consideration, the highest level of cumulative fluoride ions exhibition was observed in pH 5.5 (2) (4.897 ± 1.063 [μg/mm^2]) after 168 hours. On the other hand, among all artificial saliva solutions (1–7), the lowest value of cumulative F^- ions release was determined in pH 7.5 (5) (0.022 ± 0.001 [μg/mm^2]) after 1 hour.

Pearson's correlation between time of incubation and values of fluoride ions released from samples in all examined solutions was positive, however it was not considered as statistically significant. The Pearson's correlation coefficient (r) was comparable in the case of samples from all tested solutions (it was approximately 0.9).

The SEM micrographs of pure Tetric EvoCeram sample and after immersion in different solutions are presented in Fig. 7. It is noticeable that fluoride release causes degradation of the surface mostly by creating craters and canals.

Vitremer

Exhibition of fluoride ions from Vitremer into nine environments – artificial saliva with different pH and composition, deionized water and saline solution is presented in Table 4.

The highest F^- ions release from Vitremer was in deionized water (8) after 168 hours and it was 24.021 ± 2.280 [μg/mm^2/h], whereas the lowest value was found in artificial saliva, pH 4.5 without Ca^{2+} ions (6) after 168 hours and it was 0.303± 0.249 [μg/mm^2/h]. Among the solutions of artificial saliva with Ca^{2+} (1–5), the highest level of fluoride ions was determined in pH 7.5 (5) after 3 hours (15.210 ± 2.648 [μg/mm^2/h]), the lowest value was found in pH 4.5 (1) after 168 hours (2.540 ± 0.740 [μg/mm^2/h]).

Taking the solutions of artificial saliva (AS) without Ca^{2+} ions (6,7) into account the highest level F^- ions was read in pH 4.5 (6) after 3 hours (5.266 ± 0.243 [μg/mm^2/h]), whereas the lowest in pH 4.5 (6) after 168 hours (0.303 ± 0.249 [μg/mm^2/h]). In deionized water (8) the highest amount of F^- ions was determined after 168 hours (24.021 ± 2.280 [μg/mm^2/h]) whereas the lowest after 1 hour (1.420 ± 0.099 [μg/mm^2/h]).

In the saline solution – 0.9% w/v NaCl (9), exhibition of fluoride ions was the highest after 24 hours (20.050 ± 1.763 [μg/mm²/h]), the lowest level was read after 3 hours (1.358 ± 0.015 [μg/mm^2/h]). Taking into account the content of fluoride ions released from the samples incubated in all seven artificial saliva solutions (1–7), the highest value was noted in artificial

Table 4. Release of fluoride ions [μg/mm^2/h] from Vitremer into nine environments differing in composition of the solution and pH. Six samples were prepared for each solution

Time [hours]	AS pH 4.5 (1) [μg/mm^2/h]	AS pH 5.5 (2) [μg/mm^2/h]	AS pH 6.0 (3) [μg/mm^2/h]	AS pH 7.0 (4) [μg/mm^2/h]	AS pH 7.5 (5) [μg/mm^2/h]	AS – Ca^{2+} pH 4.5 (6) [μg/mm^2/h]	AS – Ca^{2+} pH 5.5 (7) [μg/mm^2/h]	Deionized H_2O (8) [μg/mm^2/h]	0.9% NaCl (9) [μg/mm^2/h]
1	5.366 ± 0.049	7.446 ± 0.485	7.452 ± 0.877	9.093 ± 0.973	13.663 ± 1.904	4.660 ± 0.242	3.860 ± 0.072	1.420 ± 0.099	5.856 ± 0.629
3	6.310 ± 0.494	11.163 ± 2.001	11.913 ± 0.478	13.813 ± 1.952	15.210 ± 2.648	5.266 ± 0.243	3.908 ± 0.063	4.636 ± 0.362	1.358 ± 0.015
24	3.440 ± 0.248	6.341 ± 0.922	6.770 ± 0.028	7.568 ± 1.558	6.391 ± 0.050	2.845 ± 0.067	1.140 ± 0.208	15.798 ± 0.771	20.050 ± 1.763
48	2.843 ± 0.148	2.811 ± 0.155	5.185 ± 0.334	5.273 ± 0.728	6.648 ± 0.224	1.656 ± 0.078	1.170 ± 0.094	12.915 ± 0.073	15.215 ± 2.504
72	2.955 ± 0.131	3.355 ± 0.071	4.695 ± 0.046	5.398 ± 0.907	6.653 ± 0.226	1.018 ± 0.150	0.325 ± 0.047	10.420 ± 0.233	14.638 ± 2.203
168	2.540 ± 0.740	4.048 ± 0.887	3.660 ± 0.038	5.110 ± 0.575	8.318 ± 0.282	0.303 ± 0.249	0.461 ± 0.047	24.021 ± 2.280	14.876 ± 2.460
Mean± SD[#]	3.909 ± 1.479	5.861 ± 3.060	6.612 ± 2.752	7.709 ± 3.328	9.480 ± 3.845	2.625 ± 1.863	1.810 ± 1.523	11.535 ± 7.545	11.999 ± 6.658
p-value (ANOVA for dependent samples)	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001	<0.0001
post-hoc Tukey's test	$p < 0.05$[#] for: 1 vs. 3 1 vs. 24 1 vs. 48 1 vs. 72 1 vs. 168 3 vs. 24 3 vs. 48 3 vs. 72 3 vs. 168 24 vs. 168	$p < 0.05$[#] for: 1 vs. 3 1 vs. 48 1 vs. 72 1 vs. 168 3 vs. 24 3 vs. 48 3 vs. 72 3 vs. 168 24 vs. 48 24 vs. 72 24 vs. 168	$p < 0.05$[#] for: 1 vs. 3 1 vs. 48 1 vs. 72 1 vs. 168 3 vs. 24 3 vs. 48 3 vs. 72 3 vs. 168 24 vs. 48 24 vs. 72 24 vs. 168 48 vs. 168 72 vs. 168	$p < 0.05$[#] for: 1 vs. 3 1 vs. 48 1 vs. 72 1 vs. 168 3 vs. 24 3 vs. 48 3 vs. 72 3 vs. 168 24 vs. 48 24 vs. 72 24 vs. 168	$p < 0.05$[#] for: 1 vs. 24 1 vs. 48 1 vs. 72 1 vs. 168 3 vs. 24 3 vs. 48 3 vs. 72 3 vs. 168	$p < 0.05$[#] for: all intergroups comparisons	$p < 0.05$[#] for: 1 vs. 24 1 vs. 48 1 vs. 72 1 vs. 168 3 vs. 24 3 vs. 48 3 vs. 72 3 vs. 168 24 vs. 72 24 vs. 168 48 vs. 72 48 vs. 168	$p < 0.05$[#] for: all intergroups comparisons	$p < 0.05$[#] for: 1 vs. 3 1 vs. 24 1 vs. 48 1 vs. 72 1 vs. 168 3 vs. 24 3 vs. 48 3 vs. 72 3 vs. 168 24 vs. 48 24 vs. 72 24 vs. 168

AS – artificial saliva, AS – Ca^{2+} (artificial saliva without Ca^{2+}), [#] – groups were numbered according to time of incubation.

Table 5. Cumulated release of fluoride ions [µg/mm^2] from Vitremer into nine environments differing in composition of the solution and pH. Six samples were prepared for each solution. Descriptive data were presented as mean ± standard deviation (±SD)

Time [hours]	AS pH 4.5 (1) [µg/mm^2]	AS pH 5.5 (2) [µg/mm^2]	AS pH 6.0 (3) [µg/mm^2]	AS pH 7.0 (4) [µg/mm^2]	AS pH 7.5 (5) [µg/mm^2]	AS – Ca^{2+} pH 4.5 (6) [µg/mm^2]	AS – Ca^{2+} pH 5.5 (7) [µg/mm^2]	Deionized H_2O (8) [µg/mm^2]	0.9% NaCl (9) [µg/mm^2]
1	5.366 ± 0.049	7.446 ± 0.485	7.452 ± 0.877	9.093 ± 0.973	13.663 ± 1.904	4.660 ± 0.242	3.860 ± 0.072	1.420 ± 0.099	5.856 ± 0.629
3	11.677 ± 0.543	18.610 ± 2.486	19.365 ± 1.355	22.907 ± 2.925	28.873 ± 4.552	9.927 ± 0.485	7.768 ± 0.135	6.057 ± 0.461	7.215 ± 0.644
24	15.117 ± 0.791	24.952 ± 3.408	26.135 ± 1.383	30.475 ± 4.483	35.265 ± 4.602	12.772 ± 0.552	8.908 ± 0.343	21.855 ± 1.232	27.265 ± 2.407
48	17.960 ± 0.939	27.763 ± 3.563	31.320 ± 1.717	35.748 ± 5.211	41.913 ± 4.826	14.428 ± 0.630	10.078 ± 0.437	34.770 ± 1.305	42.480 ± 4.911
72	20.915 ± 1.070	31.118 ± 3.634	36.015 ± 1.763	41.147 ± 6.118	48.567 ± 5.052	15.447 ± 0.780	10.403 ± 0.484	45.190 ± 1.538	57.118 ± 7.114
168	23.455 ± 1.810	35.167 ± 4.521	39.675 ± 1.801	46.257 ± 6.693	56.885 ± 5.334	15.750 ± 1.029	10.865 ± 0.531	69.212 ± 3.818	71.995 ± 9.574
Correlation (Pearson test)	$r = 0.842$, $p = 0.035^*$	$r = 0.804$, $p = 0.054$	$r = 0.816$, $p = 0.048^*$	$r = 0.826$, $p = 0.043^*$	$r = 0.867$, $p = 0.025^*$	$r = 0.711$, $p = 0.113$	$r = 0.695$, $p = 0.125$	$r = 0.963$, $p = 0.002^*$	$r = 0.924$, $p = 0.008^*$

AS – artificial saliva, AS – Ca^{2+} (artificial saliva without Ca^{2+}), * – statistically significant.

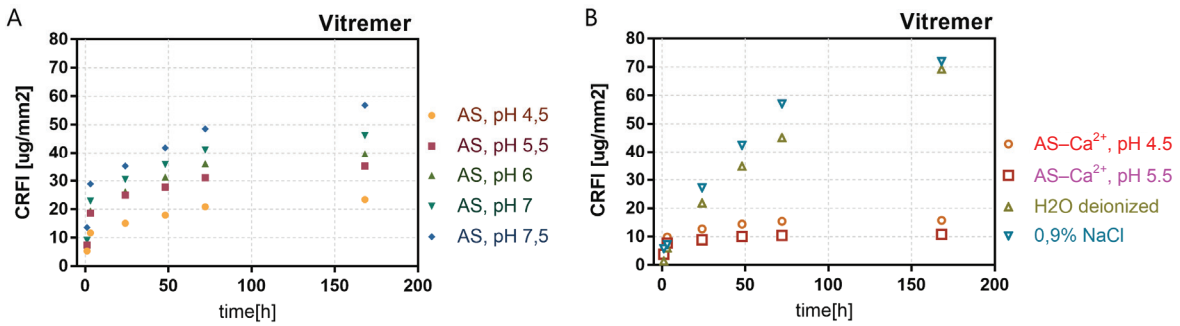

Fig. 8. Cumulated release of fluoride ions (CRFI) [µg/mm^2] from Vitremer into nine different solutions. Points represent means of measurements in time periods. AS – artificial saliva

saliva (AS) pH 7.5 (5) (15.210 ± 2.648 [µg/mm^2/h]) after 3 hours, while on the contrary the lowest value was found in artificial saliva (AS) – Ca^{2+} pH 4.5 (6) (0.303 ± 0.249 [µg/mm^2/h]) after 168 hours.

The highest mean ± standard deviation (SD) was estimated in saline solution 0.9% w/v NaCl (9) (11.999 ± 6.658 [µg/mm^2/h]), whereas the lowest mean was calculated in artificial saliva AS – Ca^{2+} pH 5.5 (7) (1.810 ± 1.523 [µg/mm^2/h]). Similar mean values were observed in deionized water (8) (11.535 ± 7.545 [µg/mm^2/h]) and saline solution 0.9% NaCl (9) (11.999 ± 6.658 [µg/mm^2/h]). The *p*-value (ANOVA for dependent samples) was determined as <0.0001 in case of samples from all examined solutions, however it was not considered as statistically significant.

Cumulated release of fluoride ions from Vitremer is presented in Table 5 and Fig. 8.

The highest level of cumulative release from the Vitremer samples incubated in all nine environments (1–9) was observed in saline solution 0.9% NaCl (9) (71.995 ± 9.574 [µg/mm^2]) after 168 hours. The lowest level of cumulative release from the samples immersed in all nine solutions (1–9) was found in deionized water (8) (1.420 ± 0.099 [µg/mm^2]) after 1 hour.

The limit values of cumulative fluoride ions releases from Vitremer were calculated as follows:

- After 1 hour, the highest cumulative release was read in artificial saliva (AS) pH 7.5 (5) (13.663 ± 1.904 [µg/mm^2]) wheras the lowest was noted in deionized water (8) (1.420 ± 0.099 [µg/mm^2]);

- After 3 hours, the highest cumulative release was observed in artificial saliva (AS) pH 7.5 (5) (28.873 ± 4.552 [μg/mm^2]), the lowest value was read in de-ionized water (8) (6.057 ± 0.461 [μg/mm^2]);
- After 24 hours, the highest cumulative release was obtained in artificial saliva (AS) pH 7.5 (5) (35.265 ± 4.602 [μg/mm^2]), the lowest was found in artificial saliva AS – Ca^{2+} pH 5.5 (7) (8.908 ± 0.343 [μg/mm^2]);
- After 48 hours, the highest cumulative release was noticed in saline solution 0.9% NaCl (9) (42.480 ± 4.911 [μg/mm^2]), the lowest was found in artificial saliva AS – Ca^{2+}+ pH 5.5 (7) (10.078 ± 0.437 [μg/mm^2]);
- After 72 hours, the highest cumulative release was observed in saline solution 0.9% NaCl (9) (57.118 ± 7.114 [μg/mm^2]), the lowest was in artificial saliva AS – Ca^{2+} pH 5.5 (7) (10.403 ± 0.484 [μg/mm^2]);

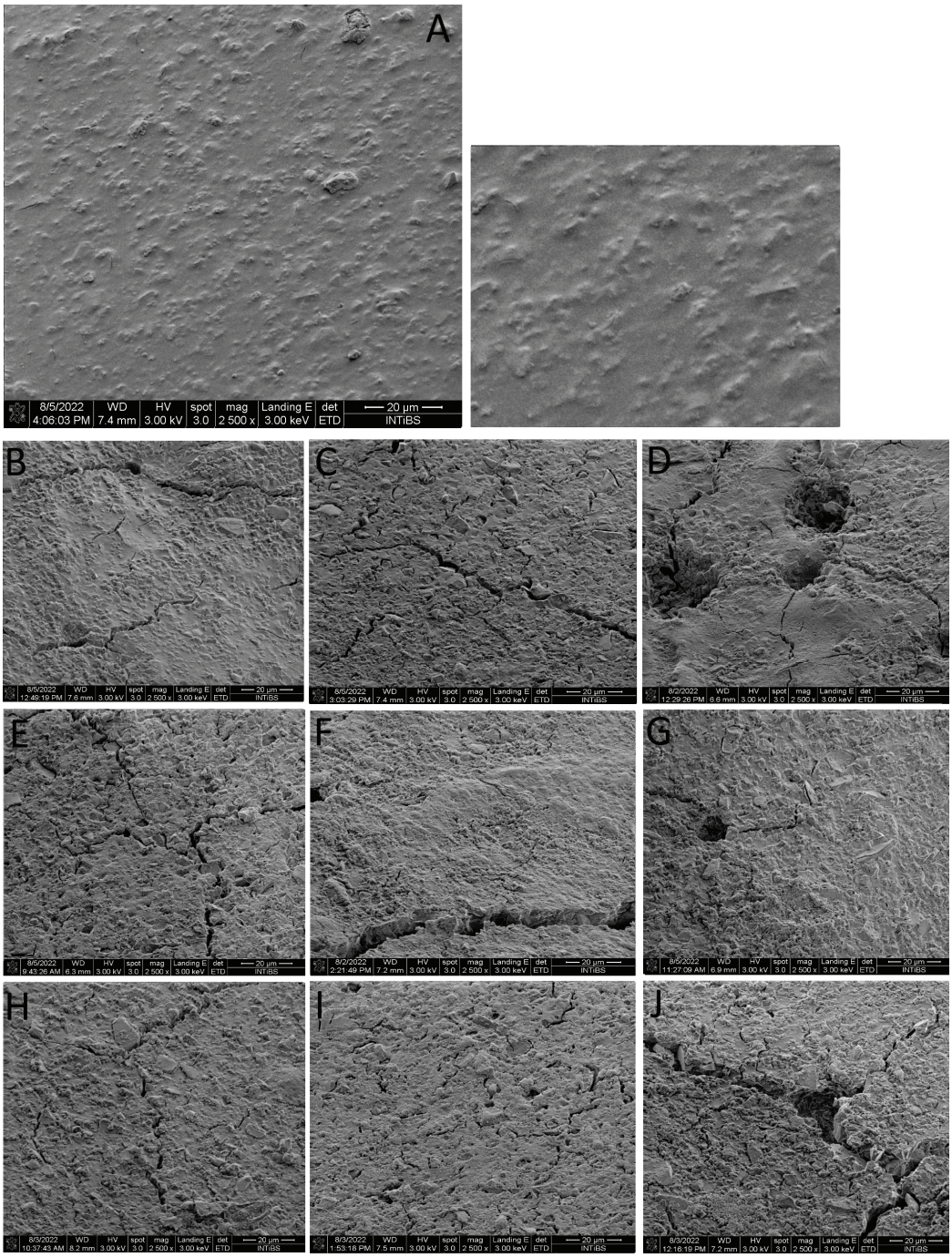

Fig. 9. SEM micrographs of A) pure Vitremer sample and after immersion in B) H_2O, C) NaCl, D) AS pH 4.5, E) AS – Ca^{2+} pH 4.5, F) AS pH 5.5, G) AS – Ca^{2+} pH 5.5, H) AS pH 6.0, I) AS pH 7.0, and J) AS pH 7.5

- After 168 hours, the highest cumulative release was obtained in saline solution 0.9% NaCl (9) (71.995 ± 9.574 [μg/mm^2]), the lowest was in artificial saliva AS – Ca^{2+} pH 5.5 (7) (10.865 ± 0.531 [μg/mm^2]).

Taking all seven artificial saliva solutions (1–7) into consideration, the highest level of cumulative fluoride ions exhibition was observed in pH 7.5 (5) (56.885 ± 5.334 [μg/mm^2]) after 168 hours. On the other hand, among all artificial saliva solutions (1–7), the lowest value of cumulative F^- ions release was determined in pH 5.5 – Ca^{2+} (7) (3.860 ± 0.072 [μg/mm^2]) after 1 hour.

Pearson's correlation between time of incubation and values of fluoride ions released from samples in all examined solutions was positive. In the solution of artificial saliva – AS pH 4.5 (1) (p = 0.035), artificial saliva – AS pH 6.0 (3) (p = 0.048), artificial saliva – AS pH 7.0 (4) (p = 0.043), artificial saliva AS pH 7.5 (5) (p = 0.025), deionized water (8) (p = 0.002), 0.9% sol. NaCl (9) (p = 0.008), Pearson's correlation was considered as statistically significant. The highest Pearson correlation coefficient (r) was estimated in deionized water (8) (r = 0.963) and it indicated strong correlation between the time of incubation and amount of released fluoride ions.

The SEM micrographs of pure Vitremer sample and after immersion in different solutions are presented in Fig. 9. It is noticeable that fluoride release causes degradation of the surface mostly by creating craters, canals and cracks.

4. Discussion

It is commonly known that dentistry as a branch of medicine is constantly developing. Rapid scientific and technological advances contribute to the transformation of healthcare. Scientists are searching for materials that have the best mechanical and physicochemical properties, adequate to oral cavity conditions.

Saliva constitutes one of the main elements of the oral cavity, where many reactions take place in the natural surroundings. Human saliva, as a physiological fluid, affects the maintenance of proper oral hygiene. In addition, it participates in the supply of health-promoting substances and nutrients that have an essential impact on the whole organism. It is not possible to create artificial saliva with identical formula to the natural one, due to many variable factors and interactions between all constituents [21], [29].

In this study, the choice of *in vitro* model was dictated by several reasons. First of all, the use of natural human saliva is demanding as a result of the lack of stability outside the oral cavity and bacterial colonization. What is more, diet, age, sex, diseases, pharmacologic agents may also affect the composition of saliva. Additionally, the presence of other ions from human saliva could alter the measurements of F^- from tested materials. Therefore, the utilization of artificial saliva in our study is explained by the desire to obtain stable conditions. The recipe of artificial saliva is based on average tested samples of natural saliva [18]. [24]. In our own study, to prepare 1 litre of artificial saliva, the following components were used: 0.4 g of NaCl; 0.4 g of KCl; 0.908 g of $CaCl_2 \cdot 2H_2O$; 0.78 g of $NaH_2PO_4 \cdot 2H_2O$; 0,005 g of $Na_2S \cdot 9H_2O$.

In our short-time (7 days) study, we obtained significant differences in values of fluoride ions – released from resin-modified glass ionomer (Vitremer) and nanohybrid composite (Tetric EvoCeram). Solutions of artificial saliva imitated natural conditions of the oral cavity. The authors of our study decided to create nine solutions differing in the composition and pH, to compare the values of fluoride ions released in contrasting conditions, taking into account artificial saliva with and without Ca^{2+}. According to Bhatnagar et al. [3], fluoride ions exhibit strong affinity with positively charged calcium ions. Thus, created complexes may affect the amount of fluoride ions released from studied samples. In the authors' own study, cumulative values in solutions of artificial saliva pH 4.5 (1) and pH 5.5 (2) with Ca^{2+} were higher than values determined in solutions of artificial saliva without Ca^{2+} ions pH 4.5 (6) and pH 5.5 (7) from both tested material.

Deionized water and 0.9% NaCl sol. were used to compare values of F^- released from the samples in neutral conditions. Additionally, it should be mentioned that deionized water was a solution that eliminated potential interactions between fluoride ions and other ions. What is more, saline solution is electrolytically similar to human saliva. The temperature of all nine solutions was 37 °C and it mimicked average natural body temperature.

Our study shows that cumulative values from Vitremer samples were notably higher than from Tetric EvoCeram in all nine (1–9) tested environments. The highest level of cumulative release from the Vitremer among all nine environments (1–9) was observed in saline solution 0.9% NaCl (9) (71.995 ± 9.574 [μg/mm^2]) – after 168 hours. The lowest level of cumulative release from the samples immersed in all nine solutions (1–9) was found in deionized water (8) (1.420 ± 0.099 [μg/mm^2]) – after 1 hour. A comparative analysis of our results and results obtained by other authors is difficult due to different experimental

conditions. However, most studies show that fluoride ions release from other dental materials is higher in deionized water than in human, artificial saliva as well as saline solution [7], [15], [31]. These results may be explained by lower diffusion gradient between dental materials and saline solution or ion-enriched patient's saliva compared to gradient between materials and deionized water [39].

On the other hand, the highest level of cumulative release from the Tetric EvoCeram samples incubated in all nine environments (1–9) was observed in deionized water (8) (6.285 ± 0.386 [μg/mm^2]). The lowest level of cumulative release from the samples immersed in all nine solutions (1-9) was found in artificial saliva (AS) pH 7.5 (5) (0.022 ± 0.001 [μg/mm^2]) May et al. [23] evaluated release of fluoride ions on days 1, 7, 14, 30 (Phase I) from three restorative materials: a resin-based composite (Z100TM, 3M-ESPE), a resin-modified glass-ionomer cement (VitremerTM, 3M-ESPE) and a bioactive material (Activa Bioactive-Restorative TM, Pulpdent). Samples were immersed in deionized water. Taking all time intervals in Phase I into consideration, the greatest fluoride ions release was observed from Vitremer and the lowest from Z100. Abudawood et al. [1] examined resin-based composite (Z100), resin-modified glass ionomer cement (Vitremer) and a new experimental material, which is a self-curing resin-based composite with light curing option. Specimens were submerged in distilled water. The measurements were taken after 1, 7, 14 and 30 days and then they were exposed to 2.0% neutral sodium fluoride foam. The quantity of re-released fluoride ions were also measured at days 1, 7, 14, 30. Fluoride ions values were significantly higher from Vitremer than from the experimental material at days 1 and 7. In the authors' study, in deionized water (8) the highest values of fluoride ions in time intervals were also noted after 24 hours of examination (15.798 ± 0.771 [μg/mm^2/h]) and after 168 hours (24.021 ± 2.280 [μg/mm^2/h]).Vitremer and experimental material released notably more fluoride ions than Z100. Additionally, Donly et al. [6] conducted study on caries inhibition at restoration margins. In their reasearch, they examined *in vitro* caries inhibition of Vitremer, Cention N and and Z100. Standarized Class V preparations were placed in molars. The study showed that Vitremer had significantly less enamel and dentin demineralization than Cention N and Z 100. The research indicated that Vitremer may clinically inhibit caries at restoration margins.

Porenczuk et al. [28] compared fluoride ions values released from 3 different materials: glass-ionomer cement (3M KetacMolar, Saint Paul, MN, USA), resin-modified glass ionomer cement (ACTIVA BioActive--Restorative, Pulpdent, Watertown, MA, USA), and nanocomposite (Tetric EvoCeram, IvoclarVivadent AG, Schaan, Liechtenstein) to solutions: deionized water, artificial saliva, pH-cycling solution The study showed that the greatest amount of fluoride ions was observed from GIC specimens (20.698–54.118 ppm), significantly lower was determined from bioglass-reinforced RMGIC (from 1.236 to 15.552 ppm) and the lowest value was read from nanohybrid polymer-resin (0.370–1.148 ppm). In our own study, values of fluoride ions calculated from the samples of Tetric EvoCeram ranged between 0.022 ± 0.001 [μg/mm^2/h] to 1.550 ± 0.014 [μg/mm^2/h].

Moreover, Helvatjoglu-Antoniades et al. [14] examined release of fluoride ions in distilled water (at 37 °C) from different types of materials and luting cement. In their study of materials, they demonstrated that release of fluoride ions was notably higher from glass-ionomer materials than from composite resin – Tetric. The order of fluorides level released from the prepared samples was as follow: Miracle Mix > Fuji III, KetacCem > Fuji II LC > Ketac Silver, Compoglass F > Fissurit F, Helioseal F > Tetric (> indicates statistical significance, $P < 05$).

Naoum et al. [25] assessed the fluoride ions release and recharge from three fluoride – containing resin composites: Beautifil II, Gradia Direct X, Tetric EvoCeram and glass – ionomer cement Fuji IX Extra into deionized water (pH 6.5) and lactic acid (pH 4.0). Tetric EvoCeram demonstrated the lowest fluoride ions release and fluoride recharge from the resin composites.

Garcez et al. [9] in their research evaluated fluoride ions release from different materials (including Vitremer and Tetric Ceram) in two protocols: deionized water and pH-cycling system (demineralizing solution – pH 4.3 and remineralizing solution – pH 7.0) for 15 days. Both Vitremer and Tetric Ceram released more fluoride ions in pH-cycling solution than in deionized water. The highest mean of fluoride ions among all tested samples was calculated from Vitremer in pH-cycling (1.3212 [pg/mm^2]; SD 0.3330) on the first day of immersion.

Most often in pediatric dentistry, carious cavities are filled with the direct method using various dental materials, however, in the case of extensive carious lesions, steel crowns can be used. Sztyler et al. [37] examined physicochemical, microbial and cytotoxic properties of the stainless steel crowns used in pediatric dentistry. In their study, crowns made of stainless steel did not show any cytotoxic effect in the direct contact method. The roughness of the material was

primarily influenced by the implant's shape, but the finishing process, particularly the polishing, also played a significant role. The largest roughness values were determined in the areas of crown rubble (B1), whereas the lowest occurred in areas characterized by large, flat, easy-to-polish surfaces (M).

In our own study, we used scanning electron microscopy (SEM) to visualize the change in the structure of the material after a certain time of fluoride ions release. It is noticeable that fluoride release causes degradation of the studied materials surface mostly by creating craters and canals. Gülses et al. [10] evaluated marginal integrity of five different materials used for retrograde root filling after apicoectomy. Scanning electron microscopy (SEM) analysis was used for quantitative analysis of marginal adaptation properties of the sealers.

Dental materials, which contain fluorides exhibit differences in the release of fluoride ions and uptake characteristics. Short-term and long-term fluoride release depend on several factors: matrices, fluoride content, fillers, setting mechanisms and environmental conditions of restoratives [39]. In addition, according to Williams et. al [40] release of fluoride ions is also dependent on surface area. Differences between our results and results obtained by other researchers may be explained by the different size and shape of prepared samples.

Furthermore, the mechanisms responsible for the release of fluoride ions are diffusion and dissolution of the dental material. Diffusion is caused by a proper counter ion such as sodium or by exchange of hydroxyl groups in fluid [26], [38].

5. Conclusions

Both materials showed ability to release fluoride ions in our *in vitro*, experimental conditions. Our short-time study was conducted to analyze and contrast the level of fluoride ions released from resin-modified glass-ionomer cement (Vitremer) and nanohybrid composite material (Tetric EvoCeram). The values of fluorides differed significantly in concrete time intervals. The highest level of cumulative release from Vitremer was observed in saline solution (9), whereas from Tetric EvoCeram in deionized water (8).

In our own study, we demonstrated that Vitremer constitutes a better reservoir of fluorides and it may reduce the occurrence of recurrent caries. However, it should be proven by further clinical studies.

Acknowledgements

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