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ABSTRACT

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THE COMPARATIVE STUDY OF PROLONGED FLUORIDE ION RELEASE IN CONTEMPORARY DENTAL ENAMEL SEALANTS

The objectives of the study was to investigate the amount of long-term fluoride ion release from three materials (sealants) – “Fissurit FX”, “Clinpro™Sealant” and “Jen-Fissufil” in laboratory condition.

Materials and methods: 4 standard samples from three modern photocomposite sealants for tooth enamel (“Fissurit FX” (VOCO), “Clinpro™Sealant” (3M™ ESPE™) and “Jen-Fissufil” (Jendental Ukraine LLC)) were polymerized according to instructions of producer and were placed in deionized water in plastic tubes. The conditions for manufacturing the samples were standardized, with the air temperature in the laboratory maintained at 22.0 ± 1.0 °C and the relative humidity at 50.0 ± 5.0%. These conditions comply with ISO 7489 standards. An amount of fluoride ions was studied using the electronic ionometer on 1st, 2nd, 3rd, 8th, 15th, 22nd and 32nd day of exposition in the deionized water tacked from plastic tube. Throughout the study duration, the tubes containing the samples were stored in a thermostat at a temperature of 37.00 ± 0.50°C

Results: The all three enamel sealants selected for the study released fluoride ions for an extended period after polymerization, up to 30 days. The highest level of fluoride ion emission in distilled water occured on the next day after polymerization. This phenomenon can be attributed to the ongoing polymerization-depolymerization reactions within the material itself (composite conversion). “Fissurit FX” samples showed the highest F^- release: 4.80 µg/cm² – day 1, and stabilized at 0.45 µg/cm² by day 14. Day 14 to 30 – from 0.13 to 0.17 µg/cm². “Jen-Fissufil” released 3.54 µg/cm² on 1st day, 0.74 µg/cm² on 7th, and 0.26 – 0.44 µg/cm² afterwards. “Clinpro™Sealant” – 1.84 µg/cm² of F^- on 1st day, 0.58 on 2nd, 0.53 µg/cm² on 3rd. From 21st day – from 0.20 to 0.21 µg/cm².

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Comparison of the levels of fluoride ion release revealed a consistent pattern of element release within a month after polymerization for all materials: "Jen-Fissufil" exhibited the highest release, followed by "Clinpro™Sealant" and "Fissurit FX" with the lowest release. This trend was consistent throughout the month, except for the first day when "Fissurit FX" exhibited the highest release of fluoride ions.

Conclusions: The enamel dental composite sealants "Fissurit FX", "Clinpro™Sealant" and "Jen-Fissufil" are capable of emitting fluoride ions after use on enamel. The highest level of emission in distilled water occurs 24 hrs after polymerization, the process lasts more than 30 days in less amounts.

Keywords: teeth, caries, prevention, fluoride ion, sealant, composites.

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ПОРІВНЯЛЬНЕ ДОСЛІДЖЕННЯ ТРИВАЛОГО ВИВІЛЬНЕННЯ ІОНІВ ФТОРУ У СУЧАСНИХ СТОМАТОЛОГІЧНИХ ГЕРМЕТИКІВ ДЛЯ ЕМАЛІ ЗУБІВ

Мета дослідження: дослідити обсяги тривалого виділення іонів фтору з трьох стоматологічних фотокомпозитних матеріалів для герметизації фісур та ямок (стоматологічних герметиків) – «Fissurit FX», «Clinpro™Sealant» і «Jen-Fissufil» в лабораторних умовах.

Матеріали та методи. Відповідно до інструкцій виробника було підготовлено 4 стандартні полімеризовані зразки з кожного з трьох сучасних фотокомпозитних стоматологічних герметиків («Fissurit FX» (VOCO), «Clinpro™Sealant» (3M™ ESPE™) та «Jen-Fissufil» (ТОВ «Джендентал Україна»)) та поміщено в пластикові пробірки з дистильованою водою. Умови виготовлення зразків матеріалу були стандартизовані, температура повітря в лабораторії підтримувалась 22,0 ± 1,0 °C, відносна вологість 50,0 ± 5,0 %, що відповідає стандартам ISO 7489. Кількість виділених фторид-йонів досліджували за допомогою електронного іонометра на 1-шу, 2-гу, 3-тю, 8-му, 15-ту, 22-гу і 32-гу добу експозиції в дистильованій воді, відібраній із пластикової пробірки. Протягом усього часу дослідження пробірки зі зразками зберігалися в термостаті при температурі 37,00 ± 0,50 °C.

Результати: усі три стоматологічні герметики, відібрані для дослідження, виділяли іони фтору протягом тривалого періоду після полімеризації, до 30 днів. Найвищий рівень емісії фторид-іонів у дистильованій воді спостерігався на наступний день після полімеризації. Це явище можна пояснити реакціями полімеризації-деполімеризації, що відбуваються в самому матеріалі (конверсія композиту). Зразки матеріалу «Fissurit FX» показали найвище вивільнення F^-: 4,80 мкг/см² – 1 день, і стабілізувалося на 0,45 мкг/см² на 14 день. З 14 по 30 день – від 0,13 до 0,17 мкг/см². «Jen-Fissufil» виділив 3,54 мкг/см² на 1-й день, 0,74 мкг/см² на 7-й і 0,26–0,44 мкг/см² після.

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"Clinpro™Sealant" – 1,84 мкг/см² F- в 1-й день, 0,58 у 2-й, 0,53 мкг/см² у 3-й день. З 21 дня – від 0,20 до 0,21 мкг/см². Порівняння рівнів емісії йонів фтору виявило постійну картину вивільнення елемента протягом місяця після полімеризації для всіх матеріалів: «Jen-Fissufil» продемонстрував найвище виділення, за ним йдуть «Clinpro™Sealant» і «Fissurit FX» з найнижчим рівнем виділенням. Ця тенденція зберігалася протягом усього місяця, за винятком першого дня, коли «Fissurit FX» продемонстрував найбільшу емісію іонів фтору.

Висновки: стоматологічні фотокомпозитні герметики для фісур та ямок зубів «Fissurit FX», «Clinpro™Sealant» і «Jen-Fissufil» здатні виділяти іони фтору протягом тривалого часу після нанесення на емаль зуба та полімеризації. Найвищий рівень емісії в дистильованій воді спостерігається через 24 години після полімеризації, процес триває понад 30 днів у менших обсягах.

Ключові слова: зуби, карієс, профілактика, іон фтору, герметик, композити.

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INTRODUCTION / ВСТУП

In the current stage of dental science and practice, fissure sealing represents a vital method for primary prevention of dental caries. It is often employed in conjunction with other dental disease prevention techniques. The fundamental principle behind this procedure involves the hermetic sealing of fissures and pits on the tooth enamel. This creates a mechanical barrier that prevents the infiltration of microorganisms and halts the progression of caries during its early stages. Numerous clinical studies have corroborated the significance of pit and fissure sealing, revealing that it accounts for approximately 90.0% of all cases of caries in permanent chewing teeth and 44.0% of cases in deciduous teeth among children and adolescents [1–4].

Long-term clinical and population studies have consistently demonstrated that the application of dental filling materials as sealants on caries-resistant areas of permanent molars in children and adolescents' results in a significant reduction of carious lesions on occlusal surfaces, with a remarkable 76.0% decrease observed within 2–3 years. These findings even suggest that sealants are more effective in comparison to the localized use of fluoride varnishes. In general, clinical guidelines advocating the use of sealants for pits and fissures on chewing teeth are widely recognized as effective and beneficial. However, it is also important to emphasize the need for regular monitoring of the integrity of the sealant layer on the teeth and its renewal when necessary [5–7].

Contemporary dental sealants are materials that, after polymerization (curing) on the tooth surface, have the capability to release fluoride. In practical dentistry, the use of fluoride is primarily driven by its ability to modify the principal compound found in the hard tissues of teeth – calcium hydroxyapatite. This modification results in the conversion of calcium hydroxyapatite into fluorapatite, which forms a more stable and resistant structure, less susceptible to demineralization (the loss of calcium ions). Early versions of fluoride-releasing sealants were typically comprised of glassy cements. These materials are capable of creating a robust fluoride reservoir on the enamel surface, releasing fluoride ions over an extended period [6, 7].

Over the past three decades, as dental filling material technology has advanced, the incorporation of fluoride into restorative materials has become a topic of considerable interest among researchers and clinicians. These innovations enable the use of modern restorative materials as a sustained source of low-level fluoride release into the teeth over an extended period. These materials, often referred to as "smart" or "intelligent" dental materials, were designed with the aim of mitigating the development of secondary caries and counteracting high acidity on the enamel surface, particularly in patients at a high risk of dental caries [8–10].

Therefore, as of today, enamel sealants based on composite resins capable of fluoride release exhibit superior outcomes in caries prevention compared to glass-composite cements in clinical practice. The latter materials are less resilient when exposed to prolonged chewing pressure. Consequently, the majority of dental materials used for sealing fissures and pits are composites or compomers (hyomers).

Considering that the volume and duration of fluoride ion release play a pivotal role in dental caries prevention, the ongoing development and enhancement of such materials stand as significant priorities within both practical and theoretical dentistry [11, 10, 7, 8].

Aim of this study was to investigate the amount of long-term fluoride ion release from three materials (sealants) – “Fissurit FX”, “Clinpro™Sealant” and “Jen-Fissufil”.

Materials and methods

In this study, three modern photocomposite sealants for tooth enamel were useed: “Fissurit FX” (VOCO) – lot 2004713, “Clinpro™Sealant” (3M™ ESPE™) – lot N902702, and “Jen-Fissufil” (Jendental Ukraine LLC) – lot FSE100345.

From each material, four standard samples, referred to as "tablets," were created. These tablets had a diameter of 1.0 cm and a height of 0.1 cm. They were polymerized using a dental cord-less photopolymerizer “Lumeon GP”, for 20 seconds while utilizing a fluoroplastic mold. Each specimen featured a 0.05 cm diameter hole in the center, through which a polypropylene dental floss was threaded for proper fixation in tube. This setup allowed us to suspend the material sample in the test humid environment (deionized water). The conditions for manufacturing the samples were standardized, with the air temperature in the laboratory maintained at 22.0 ± 1.0 °C and the relative humidity at 50.0 ± 5.0%. These conditions align with ISO 7489 standards. Following polymerization, the samples were placed in plastic tubes and filled with distilled (deionized) water. Throughout the study duration, the tubes containing the samples were stored in a thermostat at a temperature of 37.00 ± 0.50 °C. Water samples were collected from the tubes after 24 hours of exposure and replaced with fresh water. This process was repeated every 24 hours, spanning three days, and subsequently on the 8th, 15th, 22nd, and 32nd day from the commencement of the experiment.

The collected water samples, obtained after exposure of the material were analyzed for fluoride ions using an electronic ionometer known as "Ionomer pX-150MI" (Russian Federation). Prior to each measurement, the ionometer underwent calibration using a TISAB (Total Ionic Strength Adjustment Buffer) standard solution designed for the determination of fluoride ions (Fig. 1). Each measurement had a duration of 3.0 minutes. The measurement multiplicity is 3, followed by the calculation of the mean value [3,7].

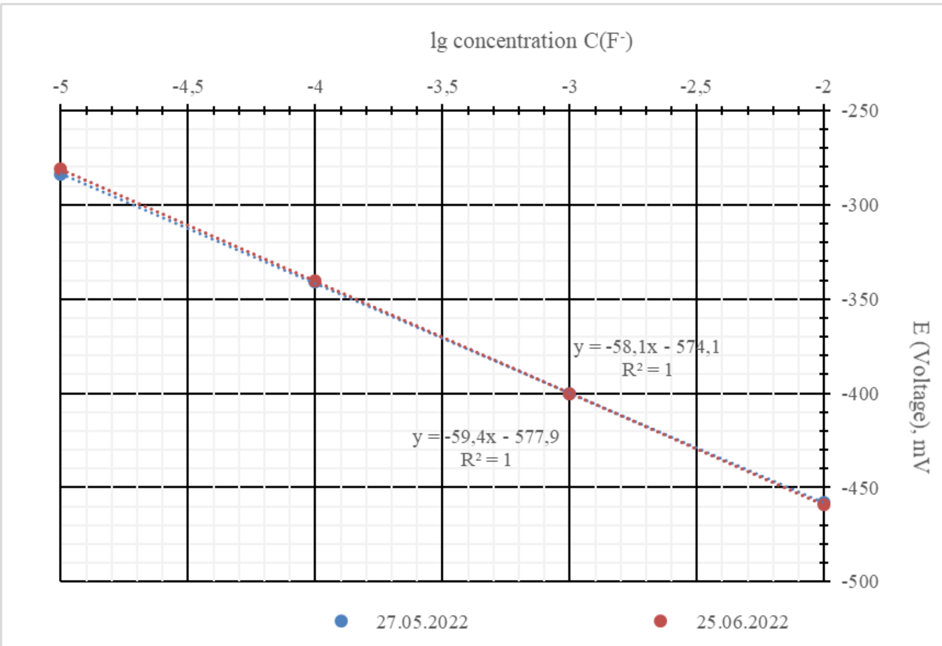

Figure 1: Calibration chart for ionometer adjustment

The concentration of fluoride ions in the solution (C) was determined using the next formula:

$$C = \frac{C(F-)\times V}{1000\times MF-} / S \times 10^6$$

where:

C (F-) – concentration of fluoride ions in the solution as calculated through ionometry.

V – the volume of the test solution (8.00 cm^3).

MF– the molar mass of fluor (19).

S – the area of the sample "tablet" (2.11 cm^2).

The obtained results were recorded in Microsoft Excel 2016 spreadsheets, where the data was also statistically processed using the descriptive statistic methods.

Results and discussion

The calculations demonstrated that all three enamel sealants selected for the study released fluoride ions for an extended period after polymerization, up to 30 days, as determined by the scope of this study. In general, the highest level of fluoride release by the materials occurred within the initial day following their photopolymerization. This phenomenon can be attributed to the ongoing polymerization-depolymerization reactions within the material itself (composite conversion). Consequently, under such conditions, the release of fluoride ions into the surrounding environment readily occurred. Starting from the third day of observation, the diffusion rate progressively decreased in all three materials.

In the case of "Fissurit FX" material, the highest release of fluoride ions among all the materials under study was recorded during the first day following polymerization, amounting to 4.80 µg/cm². This value sharply declined to 0.45 µg/cm² the subsequent day and gradually decreased further, stabilizing at relatively constant values from the 14th day onward from the commencement of the study (Table 1). Between the 14th and 30th days of observation, the level of fluoride ion release ranged from 0.13 to 0.17 µg/cm².

Table 1 – Results of ionometry and calculation of fluoride ion release by samples of "Fissurit FX" photocomposite sealant

Days	E (voltage on the ionometer), mV	lg C (F)$^-$	C(F^-) (concentration)	Emissions F^-, µg/cm^2
1	-331,50	-4,18	$6{,}67\times10^{-5}$	4,80
2	-271,75	-5,20	$6{,}25\times10^{-6}$	0,45
3	-262,75	-5,36	$4{,}3\times10^{-6}$	0,31
7	-256,00	-5,48	$3{,}35\times10^{-6}$	0,24
14	-246,50	-5,64	$2{,}30\times10^{-6}$	0,17
21	-239,75	-5,75	$1{,}76\times10^{-6}$	0,13
30	-243,25	-5,69	$2{,}02\times10^{-6}$	0,15

The study of the "Clinpro™Sealant" material showed that during the first day, this sealant also released the maximum amount of fluoride ions – 1.84 µg/cm^2, but the gradient of decrease was lower – to 0.58 and 0.53 µg/cm^2 on the second and third days. A stable level of F^- ion release was observed from day 21 – 0.20–0.21 µg/cm^2, which was higher than in the previous material (Table 2). Similar results were obtained by C.C. Lai et al. (2021) who determined the level of fluoride ion release using ion chromatography and found that "Clinpro™Sealant" released 2.61 µg/cm² of F- on the first day after polymerization. Cumulatively, for the first three days it released 5.16 µg/cm², while in our study, for the first three days, a total of 3.05 µg/cm² was released [6].

The third material, "Jen-Fissufil", also released fluoride at its maximum on the first day after polymerization – 3.54 µg/cm², which was higher than "Clinpro™Sealant" but lower than «Fissurit FX". However, the intensity of fluoride release was lower in this sealant – 1.30 µg/cm², decreasing to 0.74 µg/cm² within 7 days. Subsequently, the material maintained a stable release of fluoride ions at levels ranging from 0.26 to 0.44 µg/cm² until the end of the observation period, surpassing imported analogues in this regard (Table 3).

Table 2 – Results of ionometry and calculation of fluoride ion release by samples of "Clinpro™ Sealant" photocomposite sealant

Days	E (voltage on the ionometer), mV	lg C (F)$^-$	C(F$^-$) (concentration)	Emissions F$^-$, µg/cm^2
1	-307,25	-4,59	$2{,}55\times10^{-5}$	1,84
2	-282,25	-5,02	$9{,}48\times10^{-6}$	0,68
3	-275,75	-5,14	$7{,}33\times10^{-6}$	0,53
7	-270,25	-5,23	$5{,}89\times10^{-6}$	0,42
14	-260,25	-5,40	$3{,}96\times10^{-6}$	0,29
21	-252,75	-5,53	$2{,}94\times10^{-6}$	0,21
30	-251,75	-5,55	$2{,}83\times10^{-6}$	0,20

Comparison of the levels of fluoride ion release by the three studied sealants in distilled water revealed a consistent pattern of element release within a month after polymerization for all materials: "Jen-Fissufil" exhibited the highest release, followed by "Clinpro™Sealant" and "Fissurit FX" with the lowest release. This trend was consistent throughout the month, except for the first day when "Fissurit FX" exhibited the highest release of fluoride ions (see Fig. 2). According to the literature sources, the primary source of fluoride in the majority of modern enamel dental sealants is fluoride-silicate glass (composite filler).

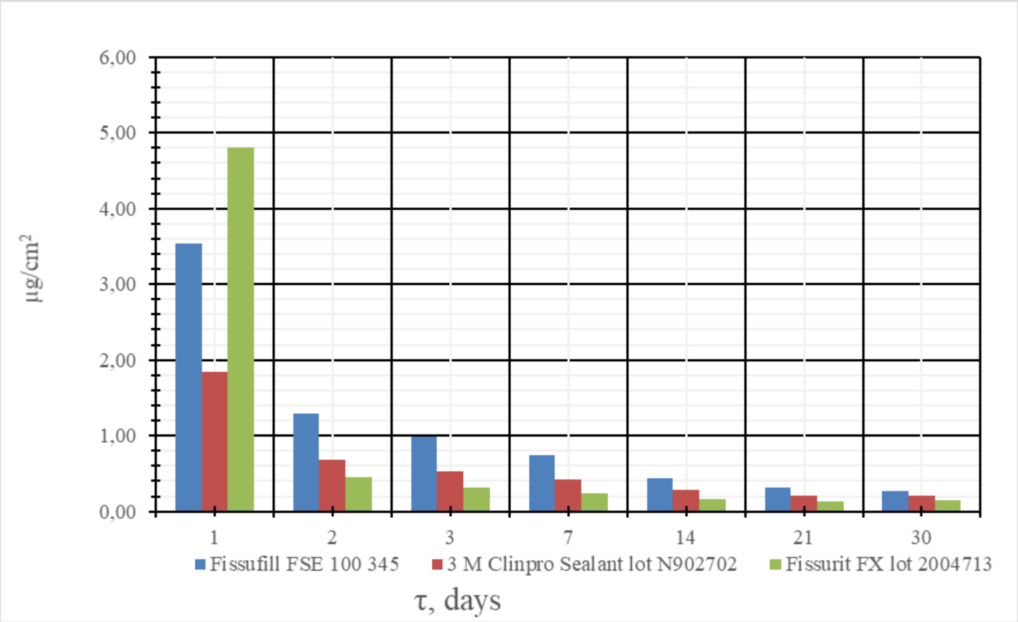

Figure 2 – Comparison of fluoride ion release by three photocomposite sealants during 30 days

Due to current recommendations, long-term release of low doses of fluoride is recognized as more effective for the prevention of dental caries than a single application of high doses [1]. However, the ability of sealing materials to prevent enamel demineralization, which occurs due to the prolonged emission of low concentrations of fluoride ions, remains an unexplored and controversial issue. Dijkman and Arends (1992) suggested that this effect is insufficient, as enamel remineralization requires concentrations ranging from 5 to 80 ppm [11]. In contrast, J. Featherstone (1999) emphasized that 0.03–0.05 ppm of fluoride is sufficient for significant enamel remineralization [2]. Furthermore, the literature contains data regarding the additional positive effect of sealing the occlusal surfaces of teeth with fluoride-containing materials, which results in a decreased incidence of caries on the apical surfaces of chewing teeth [12].

Table 3 – Results of ionometry and calculation of fluoride ion release by "Jen-Fissufil" photocomposite sealant samples

Days	E (voltage on the ionometer), mV	lg C (F)$^-$	C(F$^-$) (concentration)	Emissions F$^-$, µg/cm^2
1	-323,83	-4,31	4,93×10^{-5}	3,54
2	-298,50	-4,74	1,80×10^{-5}	1,30
3	-291,50	-4,86	1,37×10^{-5}	0,98
7	-284,17	-4,99	1,02×10^{-5}	0,74
14	-271,17	-5,21	6,11×10^{-6}	0,44
21	-262,50	-5,36	4,33×10^{-6}	0,31
30	-258,17	-5,44	3,65×10^{-6}	0,26

In a study by Z. Raszewski (2021), fluoride ion release from experimental materials, composed of mixtures of fluoride-containing glass with polymethyl methacrylate (PMMA) (sodium fluoride 10.00%, "Kavitan Plus" glass-cement powder – 5.00 and 10.00%, and "Fritex" – 10.00%), was investigated using spectrophotometry. The study observed the maximum and prolonged release of fluoride ions from the 10.00% sodium fluoride in PMMA, with a concentration of 1.58 µg/cm², occurring two weeks after polymerization. For the mixture of 10.00% "Fritex" with PMMA, fluoride emission began at 0.23 µg/cm², increased to 1.26 µg/cm² on the 14th day, and reached its maximum on the 30th day of the study at 2.32 µg/cm² [10].

Premaraj T.S. (2017) performed a comparison of the long-term release of fluoride ions by two sealants, "ProSeal®" and "Opal®Seal™". Using a fluorometer, the study found that "ProSeal®" released 4.80 µg/cm² one day after polymerization, 3.30 µg/cm² on the third day, 1.00 µg/cm² on the 7th and 14th days, and 0.40 µg/cm² from the 21st day. "Opal®Seal™" released a smaller amount of fluoride ions during the experiment, with 1.65 µg/cm² on the first day, 0.80 µg/cm² on the third day, 0.35 µg/cm² on the 7th day, and 0.10 µg/cm² from the 21st day [13].

In studies of T.Z. Ei and Y. Shimada (2018), using ionometry, it was determined that the maximum volume of fluoride ion diffusion in a solution of artificial saliva occurred on the second day after the curing of glass-ionomer cement "Fuji VII" (25.00 µg/cm²), while "Clinpro™Sealant" showed 2.80 µg/cm² and "Teethmate F-1" exhibited 4.80 µg/cm². The level of fluoride ion release in all three materials sharply decreased on the 4th day of observation [14].

In a study of D. Dionysopoulos (2016), a comparison of the release of fluoride ions (using electron ionometry) was performed for three enamel sealants: "Teethmate F-1" (Kuraray), "Fissurit FX" (Voco), "BeautiSealant" (Shofu), and glass-ionomer cement "FX-II" (Shofu). The study found that the most active fluoride ions were released by the glass-composite cement, while the performance of composite sealants showed slight differences. The maximum fluoride release was observed in all materials during the first day after the samples were manufactured. Afterward, in composite sealants, the release progressively decreased until the 4th day, where it stabilized. For "FX-II," the release was 74.00 µg/cm² on the first day, 24.00 µg/cm² on the 7th day, and 20.00 µg/cm² from the 14th day. "Teethmate F-1" released 14.00 µg/cm² on the first day, 5.00 µg/cm² on the 4th day, and dropped to 4.00 µg/cm² on the 7th and 14th days, reaching 0.50 µg/cm² by the 28th day. "Fissurit FX" released 11.00 µg/cm² on the first day, 4.00 µg/cm² on the 4th day, and decreased to 2.50 µg/cm² on the 7th and 14th days, with a further drop to 0.50 µg/cm² on the 28th day. "BeautiSealant" released 7.00 µg/cm² on the first day, 1.00 µg/cm² on the 4th, 7th, and 14th days, and dropped to 0.25 µg/cm² on the 28th day [15].

P. Kosior et al. (2017) performed similar studies, focusing on the cumulative amount of fluoride ions, on sealants "Conseal F," "Fissurit FX," "Delton Fs+," and "Admira Seal" in sodium chloride saline. Their findings showed that "Delton Fs+" exhibited the highest level of ion emission (61.91 ± 12.07 g F/mm²), while "Fissurit FX" had the lowest (28.08 ± 3.10 g F/mm²). Notably, "Fissurit FX" displayed the maximum fluoride ion emission within the first hour (4.04 µg/mm²/h), and both "Fissurit FX" and "Delton Fs+" reached their peak fluoride ion emission within 48 hours after polymerization [16].

Hence, our performed research has demonstrated the similarity among three materials, namely "Fissurit FX", "Clinpro™Sealant" and "Jen-Fissufil" – concerning the emission of fluoride ions in distilled water. These results are consistent with findings from other researching centers. Performing of such experiments in clinical settings is limited by the complexity of the measurement technique and ethical considerations. Additionally, the emission of fluoride ions may depend on the area and thickness of the sealant layer, as well as the mineral composition of the patient's oral fluid. It's also important to consider the potential for composite sealants to absorb fluoride ions when the concentration of fluoride in the oral fluid increases, such as when using fluoride toothpaste with 1450 ppm fluoride. This process may impact the duration and volume of fluoride emission by the sealant material, even with prolonged wear [15].

CONCLUSIONS / ВИСНОВКИ

In our study of fluoride ion emission by three polymerized enamel dental composite sealants "Fissurit FX", "Clinpro™Sealant", and "Jen-Fissufil" — we have made the following observations:

1. All three materials are capable of emitting fluoride ions after use.
2. The highest level of fluoride ion emission in distilled water occurs one day after polymerization.
3. "Fissurit FX» samples showed the highest release, reaching 4.80 µg/cm² on the first day, which then stabilized at 0.45 µg/cm² by day 14. From day 14 to day 30, the levels ranged from 0.13 to 0.17 µg/cm².
4. "Jen-Fissufil" released 3.54 µg/cm² on the first day, 0.74 µg/cm² on the 7th day, and subsequently maintained levels between 0.26 and 0.44 µg/cm².
5. "Clinpro™Sealant" released 1.84 µg/cm² of F- on the first day, and the release decreased to 0.58 and 0.53 µg/cm² on the 2nd and 3rd days, respectively. Stable release of F- ions was observed from the 21st day, ranging from 0.20 to 0.21 µg/cm².

Similar research should be continued at the clinical stage to clarify the levels of fluoride emissions in the human oral cavity.

PROSPECTS FOR FUTURE RESEARCH / ПЕРСПЕКТИВИ ПОДАЛЬШИХ ДОСЛІДЖЕНЬ

A process of improving the qualities and creation the new compositions of dental fissure sealants for caries prevention is perspective direction for research in organic chemistry and clinical dentistry.

CONFLICT OF INTEREST / КОНФЛІКТ ІНТЕРЕСІВ

The authors declare no conflict of interest.

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