**MATERIALE PLASTICE**
*https://revmaterialeplastice.ro*
*https://doi.org/10.37358/Mat.Plast.1964*

# Assessment of Water Sorption, Solubility and Interface Properties for Two Different Pit and Fissure Sealants

**ALEXANDRINA MUNTEAN[1], SORINA SAVA[2]*, CODRUTA SAROSI[3], MARIOARA MOLDOVAN[3], MARIANA PACURAR[4], ADA GABRIELA DELEAN[5]**
[1]Iuliu Hatieganu University of Medicine and Pharmacy, Department of Paediatric Dentistry, 31 A. Iancu Str., 400083, Cluj-Napoca, Romania
[2]Iuliu Hatieganu University of Medicine and Pharmacy, Department of Prosthodontics and Dental Materials, 15 V. Babes Str., 400012, Cluj-Napoca, Romania
[3]Babes-Bolyai University, Department of Polymer Composites, Raluca Ripan Chemistry Research Institute, Cluj-Napoca, Romania
[4]University of Medicine and Pharmacy Science and Technology G.E Palade, Department of Orthodontics, 38 Gh. Marinescu Str., Tg. Mures, Romania
[5]Iuliu Hatieganu University of Medicine and Pharmacy, Department of Conservative Dentistry, 33 Motilor Str., 400001, Cluj-Napoca, Romania

***Abstract:*** *The purpose of modern dental medicine is to prevent dental caries and promote minimally invasive techniques at the expense of invasive methods. Dental sealants are largely recommended for occlusal surfaces protection, but concerns are related about microleakage at material-enamel interface. This in vitro study aimed to investigate the association between chemical structure, sealing capacity and marginal infiltration for 2 fluoride F containing commercially available resin-based sealants: Pit&Fissure® Sealant (DMP) and Fissurit FX® (Voco) in order to achieve a better description and ensure adequate material selection in every day practice. An optimal resin-based sealant should mimic the structural, physical and mechanical characteristics of enamel. The main difference between the sealing materials tested is based on the amounts of inorganic filler and on the various shapes of the particles in the filler. Pit&Fisure® Sealant express less attachment at enamel interface, higher sorption and greater water solubility, when compared to Fissurit FX®. The better sealing capacity of Fissurit FX® can be described by the homogeneity of the material and the very small size of the inorganic fillers. Dental materials properties used as pit and fissure sealants are sensitive to mechanical, thermic and chemical stress from oral cavity. Regular dental check-up can detect the presence and integrity of sealant and ensure preventive effect in time.*

***Keywords:*** *sealant, sealant-enamel interface, sorption, solubility*

## 1. Introduction

Access to modern sources of information, concern for an adequate general health as well as promotion of oral and dental health with particular references to dental aesthetics, have led to a higher demand from patients and increase the acceptability of the decay prophylactic methods, especially pit and fissures sealants.

During childhood, in order to reduce caries risk, appropriate diet, hygiene and use of fluoride are recommended as an integrative part for individual oral health. The prevention of dental caries, immediately after teeth eruption, for posterior teeth, is achieved predominantly by the sealing materials applied in pits and fissures [1-6]. Caries developed in pits and fissures are most common in children, when compared to smooth surfaces, because permanent immature teeth, may have fragile occlusal surfaces [7]. Negative details of dental crown, permits food retention, entail salivary clearance and allow the initiation of the caries process, unless prophylactic measures are taken [2, 3, 8].

Occlusal surfaces caries prophylaxis is a continuous concern, and over time has experienced several adaptations in line with dental materials improvement. The acid etching technique and the advance of the adhesive systems contributed to anti-caries outcome of dental materials.

**email: andreea.sava@umfcluj.ro*

Dental sealants are part of a specific category of dental materials and have the following main characteristics: simplicity and speed in handling; increased fluidity to be able to easily penetrate the negative details of the occlusal surfaces; expression of the adhesion phenomenon using non-invasive or minimally invasive procedures at the enamel surface; fast setting reaction in the oral cavity temperature and humidity conditions; mechanical and thermal properties comparable to those of dental enamel; resistant in the oral environment; favourable aesthetic aspect, in order to facilitates the acceptance by the patient; easy detection during clinical examinations; not to cause occlusal interference; not to affect the surrounding soft tissues or other systems in the human body; not to cause allergic reactions [9, 10].

Dental sealants are a safe and effective way to prevent tooth decay. There are a considerable number of sealing materials, resin-based or glass ionomer cements, available on the market, at relatively comparable prices, and it is often difficult to make the right choice for a particular clinical situation [10, 11].

The purpose of modern dental medicine is to avoid dental caries and promote minimally invasive techniques at the expense of invasive methods. The active involvement and patient compliance in respect of dental caries preventive algorithm are difficult factors to quantify especially for children's, which is why dental sealants must benefit from a proper characterization using in vivo and in vitro studies.

The aim of this in vitro study was to investigate the association between chemical structure, sealing capacity, water sorption and solubility, for 2 fluoride F containing commercially available resin-based sealants: Pit&Fissure® Sealant (DMP) and Fissurit FX®(Voco).

## 2. Materials and methods

### 2.1. Materials composition

We evaluate 2 resin-based dental sealants: Pit & Fissure® (DMP) and Fissurit FX® (Voco), and the main features are presented in Table 1.

**Table 1.** Dental material characteristics

| Sample | Parameter | | |
|---|---|---|---|
| | Organic matrix | Fillers | Manufacturer |
| **Pit& Fissure Sealant** | Methacrylate Ester Monomers | Opaque and radiopaque particles Fluoride compounds | DMP Hellenic Dental Store Markopoulo Industrial Zone, 190 03, Greece |
| **Fissurit FX** | Bis-GMA* -UDMA** (91%) | Borosilicate glass Fluoride- NaF 3% | VOCO GmbH 27472 Cuxhaven Germany |

*Bis-GMA -bisphenol A glycol dimethacrylate; **UDMA-urethane dimethacrylate

Pit & Fissure® Sealant (DMP) sealant is radio-opaque, with fluoride-controlled release and photo-chemical polymerization system, designed for temporary or permanent teeth. It is used in preventive dentistry and fluoride constituent provides additional protection against decay. The material has, according to manufacturer, ideal consistency that allows penetration in pits and fissures and ensure efficient sealing [11].

Fissurit FX® (Voco) is a sodium fluoride sealing material, with a photochemical polymerization system and a high filling content (> 50%), indicated for conventional sealing and minimally invasive restorative techniques on permanent or temporary teeth. Fissurit FX has 55% micro-filler and is widely used mainly because of its easy handling [12]. It contains fluoride, Bis-GMA, di methacrylate di urethane and benzotriazole derivates.

### 2.2. Sealant enamel interface assessment

Sealant enamel interface was assessed using 20 first permanent molars, caries free under inspection in standard dental office conditions, extracted for orthodontic purposes. Once selected, the teeth were divided into two groups, according to dental material used as sealant: Pit & Fissure® Sealant (DMP) (N=10) and Fissurit FX® (Voco) (N=10).

Sealants were applied with the respect of the protocol recommended by manufacturer, as follow: professional cleaning, etching with 37% phosphoric acid, washing, drying, sealant application, light-curing for 20 s (with Optilux 501curing unit - Kerr Corp.), occlusion check.

After sealant application, teeth were kept in artificial saliva for 14 days, at 37°C, for aging. All teeth were embedded in acrylic resin, left for the next day, and then sliced into 1 mm strips using a sample cutter (IsoMet1000). Enamel-sealant interface assessment was made using Scanning Electron Microscope (SEM) (Inspect S-Fei Company) and all sample were evaluated by the same operator, to reduce inter human variation

### 2.3. Sorption and solubility assessment

The water sorption and solubility of the pit and fissure sealant was conducted according to ISO 4049/2019. Light cured (for 20 s) disk specimens (diameter 15.0 ± 0.1 mm, thickness 1.0 ± 0.1 mm) obtained using a Teflon mould and Optilux 501curing unit (Kerr Corp.) were stored in a desiccator maintained at (37 ± 1)$^o$C. We prepared 40 specimens (20 for each evaluated material) divided equally for sorption and solubility assessment. After 22 h, the specimens were transferred and stored in a desiccator, at (23 ± 1) $^o$C, for 2 h and then weighed in a digital balance (XS105, Mettler-Toledo AG, Greifensee, Switzerland) with an accuracy of 0.1 mg until a constant mass (**mL**) was obtained. The values were used to calculate the volume (**V**) of each sample (in mm$^3$). The specimens were then individually immersed in distilled water at 37 ± 1$^o$C for 21 days. After 7, 14 and 21 days they are removed from distilled water with tweezers, wiped with cellulose paper, dried in air for 15 s and weighed for mass (**m2**). They must be weighed 1 min after removal from solution. Each specimen was stored in a desiccator and reweighed daily until a constant dry mass (**m3**) was obtained.

The sorption and solubility were calculated using the following equations:

**SP = (m2 − m3)/V** and **S = (m1 − m3)/V**, where **SP** is the test material's absorption (µg/mm$^3$) and **S** is its solubility (µg/mm$^3$).

## 3. Results and discussions

*3.1. Sealant enamel interface assessment*

In Figure 1 (a, b, c) Pit & Fissure® Sealant (DMP) penetration in pits and fissures is presented at different magnification. We notice sealant presence in occlusal surface negative details, as a result of dental material physical characteristics (Figure 1a) and a slight separation of the dental material at sealant enamel interface (Figure 1b). Detachment can be explained either by the insufficient adhesion of the sealant to the enamel micro retention created after etching, the inaccuracies during sealing technique or by separating the material when slicing the tooth.

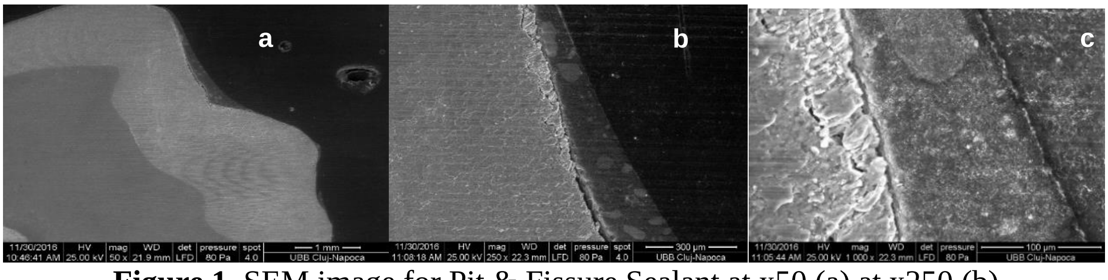


**Figure.1.** SEM image for Pit & Fissure Sealant at x50 (a) at x250 (b) and at x1000 (c) magnification

In Figure 1b, at x 250 magnification, we observed in the sealing material structure, inorganic particles, and we assume that their volume is related with inconstant penetration of dental material into enamel micro retentions. In Figure 1c the structure of the material can be observed more accurate and we notice large particles (inorganic phase, fluoride F) and we can assume that the composition of the sealant may be related with an inconstant adhesion capacity (Figure 1b).

In Figure 2, at x 1000 (a) and x 2000 (b) magnification, we distinguish a relatively uniform structure for Pit & Fissure® material with dissimilar shapes of filler particles.

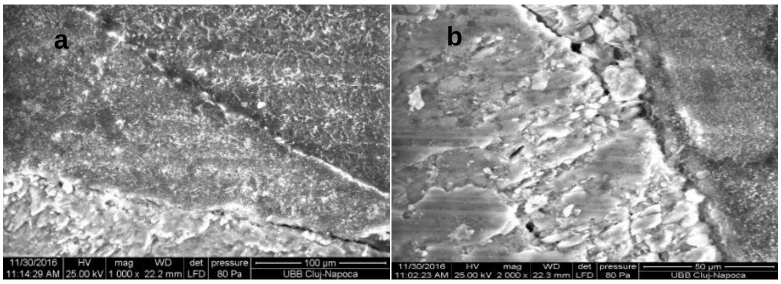


**Figure 2.** SEM image for Pit & Fissure Sealant at x1000 magnification (a) respectively x 2000 magnification (b)

Sample sealed with Fissurit FX® at x50 magnification reveal a consistent sealing; dental material appeared to be present in very narrow details of occlusal surface (Figure 3 a). At a higher magnification, we notice small cracks, and enamel discontinuity due to a subclinical stage of dental decay (Figure 3b). Continuous sealant-enamel interface confirms effective prevention and impede dental lesion development, by blocking bacteria access to fermentative substrate.

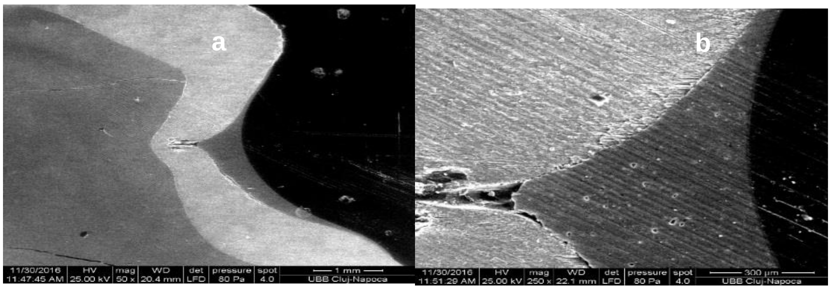


**Figure 3.** SEM image for Fissurit FX at x50 (a) respectively x250 magnification (b)

At higher magnifications (x 500, x2000), we discern an excellent sealant/ enamel interface (Figure 4a, 4b). The dental material adheres almost utterly to the enamel and has a homogenous structure, on which very small (max. 2-3 μm) inorganic particles can be observed.

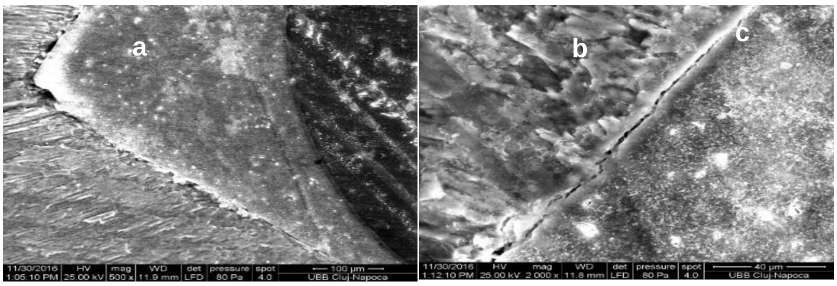


**Figure 4.** SEM image for Fissurit FX at x500 (a), x 2000 (b) magnification

Fissurit FX® structure reveal agglomerations of smaller particles with a uniform distribution of the inorganic filling in the organic matrix. We observe at the sealant / enamel interface, the penetration of the material into the micro-retentivities of the enamel. Fissurit FX® showed an extremely small detachment (2%) at x 250 magnification when compared to Pit&Fissure® Sealant (Figure 1b and 3b).

The study of the dental structures at the nano-scale level allows a better understanding of the structure and functionality of the dental surfaces [12, 13].

Permanent first molar is most susceptible for occlusal caries because of variation in occlusal fissure morphology and long eruption phase [12]. Unfavourable morphology makes these fissures difficult for salivary access and minimizes fluoride deposition, for preventive effect [13, 14] and for this reason SEM evaluation of marginal adaptation was used in order to examine sealant diffusion in profound areas of grooves and pits. Specific conditions as: porosity and viscosity of dental material, contours and geometric shapes of microparticles used as fillers are key elements in sealant composition, in order to ensure preventive effect. Oral cavity specific conditions (temperature, humidity, bacterial plaque, microorganisms) engender interactions with dental material that can modify structure with subsequent consequences on sealant effectiveness [15].

Sealing ability differs among resin-based sealants evaluated in our study. Less detachment of dental material at sealant-enamel interface express by Fissurit FX® increased the sealing ability; improved shear strength augments the longevity of sealant [7, 12].

The process of enamel acid etching plays an important role in sealant adhesion, because creates microscopic cracks, in which the sealing material adheres more deeply. Resin-based sealing materials are heterogeneous materials with two main components: resin matrix and filler particles [7, 14]. If the sealing material has uniform composition and small inorganic filling, it will be able to penetrate more easily the created micro-cracks on the enamel surface, during the acid etching process, thus increasing the probabilities of a suitable adhesion [6]. Pitt & Fissure® Sealant material contains a hybrid filling, with inorganic particles of 50-60 μm, surrounded by particles of much smaller size.

Meerbeek et al. [11] show that the fundamental principle of adhesion to the dental substrate is based on an exchange process, while inorganic material on the tooth surface is replaced with synthetic resin. The authors establish that this process involves two phases: removing calcium phosphates through which the micro porosity is exposed to the tooth surface, material infiltration and the polymerization of the resin in the surface created micro porosity [11, 16]. Authors noticed also a chemical interaction between resin based dental material and tooth substrate components; the functional monomers and the components of the tooth substrate are responsible for these phenomena [12, 17]. Pit & Fissure® Sealant filler particles are considerably larger when compared to Fissurit FX®, but both sealants filler particles are of irregular shape. An optimal resin-based sealant should mimic the structural, physical and mechanical characteristics of enamel. The better adaptation of Fissurit FX® (Voco) to the enamel surface is due to the homogeneity of the material and the very small dimensions of the inorganic fillers (2-3μm) [17].

## 2.2. Sorption and solubility assessment

Although the ISO 4049/2019 standard recommends a test period of 7 days, in this study we extended the evaluation to 14 and then to 21 days, for a better characterisation of the time factor on absorption and solubility [18].

Sorption in distilled water expose statistically significant differences for Pitt & Fissure® in all evaluated comparatives intervals opposite to Fissurit FX® which express this phenomenon after 14 or 21 days (Table 2).

**Table 2.** Sorption evaluation for selected sealants

| Time | Pitt & Fissure | | | Fissurit | | |
|---|---|---|---|---|---|---|
| | Mean | SD | p | Mean | SD | p |
| 7 days | 4.67 | 0.28 | **0.0028** | 1.84 | 0.54 | **0.0059** |

| | | | | | | |
|---|---|---|---|---|---|---|
| 14 days | 5.94 | 0.32 | | 2.83 | 0.65 | |
| 7 days | 4.67 | 0.28 | **0.0000** | 1.84 | 0.54 | **0.0000** |
| 21 days | 15.7 | 0.54 | | 11.32 | 0.46 | |
| 14 days | 5.94 | 0.32 | **0.0000** | 2.83 | 0.65 | **0.0000** |
| 21 days | 15.7 | 0.53 | | 11.32 | 0.46 | |

Absorption depends on the type of filling, the volume of filling and the adhesion of the filling-matrix. The filling volume, which reside in the total volume of the composite material, will determine the amount of polymer and the capacity of the composite material to absorb saliva [16, 19]. In line with this, sealant resin material with a lower filler content and a higher resin matrix content has a greater saliva absorption value, element validated in our study for Pit & Fissure®.

The results showed that, there were significant differences between the sorption values after 14 and 21 days, for both dental sealants. These observations are in addition to the conclusions of other studies on the complete lack of predictability of the time period regarding the water absorption of dental materials [18].

Solubility values recorded at 7,14 and 21 days express statistically significant differences for Pitt & Fissure® (Table 3).

**Table 3.** Solubility evaluation for selected sealants.

| **Time** | **Pitt & Fissure** | | | **Fissurit** | | |
|---|---|---|---|---|---|---|
| | Mean | SD | p | Mean | SD | p |
| 7 days | -25.33 | 3.14 | 0.0032 | -15.28 | 22.14 | 0.1601 |
| 14 days | -28.16 | 2.67 | | -30.99 | 20.90 | |
| 7 days | -25.33 | 3.14 | 0.0248 | -17.55 | 2.04 | 0.2529 |
| 21 days | -21.79 | 3.25 | | -24.91 | 1.49 | |
| 14 days | -28.16 | 2.67 | 0.0033 | -30.99 | 20.90 | 0.0049 |
| 21 days | -21.79 | 3.25 | | -24.91 | 19.28 | |

Solubilization takes place by water diffusion in the polymer matrix, dissolution and dispersion of macromolecules. The insolubility of all components of a fluoride containing resin-based sealant is a basic condition for clinical success. Inorganic fillers are insoluble, but by softening and dissolving the resin surface, they remain exposed and are easily removed by external agents. The solubility of composite materials is influenced by: residual monomers, type, percentage and average size of inorganic filler particles, their surface area, silane coupling agents, the presence of air inclusions inside the composite. Some of the absorbed water act as a plasticizer and affect mechanical properties of resin-based materials. It is observed that the materials with high absorption also have high solubility values at the same measurement interval [9, 18, 19].

In every day practice, dental material properties differ from in vitro conditions and especially resin based dental sealants express more marginal infiltration due to different in vivo conditions [19, 20]. Long-term high humidity, mechanical wear, occlusal stress, and complicated oral environments may cause the dissolution and partial loss of bioactive filler from the resin matrix of pit and fissure sealants. Moisture control and oral cavity humidity exert specific effects on sealants physical and mechanical properties [20, 21].

The main difference between the sealing materials tested is based on the amounts of inorganic filler and on the different forms of the particles in the filler, because both materials are resin-based and contain Fluor F in their composition [22, 23].

## 4. Conclusions

The chemical compounds make the difference between the materials. The filler fraction and particle size and uniformity of the filler distribution are the main determining factor affecting sealant enamel interface, in our study. The better sealing capacity of Fissurit FX® can be explained by the homogeneity of the material and the very small size of the inorganic fillers (2-3µm).

Pit&Fisure® Sealant express greater values for sorption and solubility, facts that can act on mechanical properties and subsequent on protective effect.

Dental sealants properties are sensitive to specific humidity conditions from oral cavity. Regular dental check-up can detect the presence and integrity of sealant and ensure preventive effect in time.

Sealant placement is a sensitive procedure that should be performed in a moisture-controlled environment. Maintenance is essential and the reapplication of sealants, when required, is important to maximize the effectiveness of the treatment.

**References**

1. FERRACANE, J. L., Resin Composite-State of the Art, *Dent Materials*, **27**, 2011, 29-38**.**
2. HERLE, G.P., JOSEPH, T., VARMA, B., JAYANTHI, M., Comparative evaluation of glass iono mer and resin-based fissure sealant using non-invasive and invasive techniques - A SEM and micro leakage study, *J Indian Soc Pedo Prev Dent*, **22**, 2004, 55-62.
3. CEHRELI, Z.C., GUNGOR, H.C., Quantitative microleakage evaluation of fissure sealants applied with or without a bonding agent: results after four-year water storage in vitro. *J Adhes Dent*, **5**, 2008, 379-84.
4. ALBANI, F., BALLESIO, I., CAMPANELLA, V., MANZO, G., Pit and fissure sealants: results at five and ten years, *Eur J Paediatr Dent*, **6**, 2005, 61-65.
5. GEIGER, S.B., GULAYEV, S., WEISS, E.I., Improving fissure sealant quality: mechanical preparation and filling level, *J Dent*, 2000, **28**, 407-412.
6. GUNGAR, H.C., TURGUT, M.D., ATTAR, N., ALTAY, N., Microleakage evaluation of a flowable polyacid-modified resin composite used as fissure sealant on air-abraded permanent teeth, *Oper Dent*, **28**, 3, 267- 273.
7. PARDI, V., SINHORETI, M.A., PEREIRA, A.C., AMBROSANO, G.M., MENEGHIM, M.C., In Vitro Evaluation of Microleakage of Different Materials Used as Pit-and-Fissure Sealants, *Braz DentJ*, **1**, 2006, 49-52.
8. WELBURY, R., RAADAL, M., LYGIDAKIS, N.A., EAPD guidelines for the use of pit and fissure sealants, *Eur J Paediatr Dent.*, **5**, 2004, 179-184.
9. RATNADITYA, A., KUMAR, M.G.M., AVULA, S.S., JOGENDRA, M.Z., KANDREGULA, C.R., KOPURI, R. K., Clinical evaluation of retention in hydrophobic and hydrophilic pit and fissure sealants- A two-year follow-up study, *J Young Pharm*, **7**, 2015, 171-179.
10. POGGIO, C., ANDENNA, G., CECI, M., BELTRAMI, R., COLOMBO, M., CUCCA, L., Fluo ride release and uptake abilities of different fissure sealant, *J Clin Exp Dent*, **8**, 2016, 284-289.
11. VAN MEERBEEK, B., MUNCK, J.DE., YOSHIDA, Y., INOUE, S., VARGAS, M., VIJAY, P., VAN LANDUYT, K., LAMBRECHTS, P., VANHERLE, G., Buonocore memorial lecture. Adhesion to enamel and dentin: Current status and future challenges, *Oper Dent*, **28**, 2003, 215-235.
12. FERNANDES, K.S., CHALAKKAL, P., DE ATAIDE, I.N., PAVASKAR, R., FERNANDES, P.P., SONI, H. A., A comparison between three different pit and fissure sealants with regard to marginal integrity, *J Conserv Dent*, **15**, 2012, 146-150.
13.MUNTEAN, A., MESAROS, A., FESTILA, D., MOLDOVAN, M., MESAROS, M., In Vitro Microleakage Evaluation Around Three Types of Dental Sealants Rev. Mat. Plastice, **53**, 2016, 166-170.
14. GANESH, M., SHOBHA, T., Comparative evaluation of the marginal sealing ability of Fuji VII and concise as a pit and fissure sealant, *J Contemp Dental Pract*, **8**, 2007, 10-18.
15.VEIGA, N.J., FERREIRA, P.C., CORREIA, I.J., PEREIRA, C.M., Fissure Sealants: A Review of their Importance in Preventive Dentistry, *OHDM*, **13**, 2014, 987-993.
16.BEUN, S., BAILLY C., DEVAUX J., LELOUP G., Physical mechanical and rheological characterization of resin-based pit and fissure sealants compared to flowable resin composites. *Dent Mater*, **28**, 2012, 349-359.

17. POP, A.S., CAMPIAN, R.S., PACURAR, M., TEODORESCU, E., BUTA, O., TARMURE, V., Evaluation of Sealing Materials Adhesion to Enamel. An in vitro study., *Mater. Plast.*, **54**, 2017, 129-132.
18. YANG, S.-Y., CHOI, J.-W., KIM, K.-M., KWON, J.-S. Prevention of Secondary Caries Using Resin-Based Pit and Fissure Sealants Containing Hydrated Calcium Silicate., *Polymers*, **12,** 2020, 1200.
19. MOLDOVAN, M., BALAZSI, R., SOANCA, A., ROMAN, A., SAROSI, C., PRODAN, D., VLASSA, M., COJOCARU, I., SACELEANU, V., CRISTESCU, I., Evaluation of the degree of conversion, residual monomers and mechanical properties of some light-cured dental resin composites, *Materials*, **12**, 2019, 2109.
20. TAMAS, C., MOLDOVAN, M., PREJMEREAN, C., COLCERIU, A., FURTOS, G., VEZSENYI, L., PRODAN, D., GRECU, R., SIMON, V. Structure and properties of inorganic fillers for dental composites. *J Optoelectron Adv M.*, **7**, *2005*, 2849-2852.
21. BOBOIA, S., MOLDOVAN, M., BURDE, A., SAROŞI, C., ARDELEAN, I., ALB, C., The effects of the curing regime and the composition on the absorption, solubility and the amount of residual monomers of dental flowable composites. *J Optoelectron Adv M*., **17**, 2015, 1487-1493.
22. JUMANCA, D., GALUSCAN, A., POPESCU, L.A., GHIORGHE, A., ANDRIAN, S., SINESCU, C., Comparative Study on Available Sealing Materials, *Rev. Chim.*, **67**(2), 2016, 241-244.
23.TARMURE, V., CAMPIAN, R.S., IONESCU, E., PRODAN, D., PACURAR, M., TEODORESCU, E., POP, A.S., Structure and the Shear Bond Strength of Two Pit and Fissure Sea ants, *Mater. Plast.*, **54**, 2017, 393-395.

Manuscript received: 03.07.2020