STUDIU COMPARATIV PRIVIND ADEZIUNEA LA STRUCTURA DENTARĂ A COROANEI CERAMICE TOTALE CU CIMENTURI ADEZIVE DIFERITE

COMPARATIVE STUDY ON ADHESION TO THE DENTAL STRUCTURE OF THE TOTAL CERAMIC CROWN WITH DIFFERENT ADHESIVE CEMENTS

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The purpose of this study is to analyze with the help of the scanning electron microscopy and FTIR microscopy, the way in which the adhesion to the dental structure and to different types of ceramics of some adhesive materials is made. For this study, 45 free teeth were used, which were divided into 3 equal groups: group I restored with IPS E.max CAD-On ceramic crowns, Ivoclar Vivadent cemented with Maxcem Elite, Kerr; group II restored with IPS E.max Press ceramic crowns, Ivoclar Vivadent cemented with RelyX Ultimate Clicker, 3M Espe and group III restored with Novodent GS Zirconia crowns cemented with Variolink Esthetic. The teeth were embedded into the resin, sectioned and subjected to the analyses.Both the tooth-cement interface and the cement-ceramic crown interface were evaluatedwith the help of SEM and FTIR. The materials used showed a good adhesion to the dental structure, and the presence of micro-cracks was observed at the ceramic interface. Moreover, the evaluated materials showed different microbial attachment ability, the most significant adherence inhibition of the Lactobacillus acidophilus being observed in the case of cemented Zirconia crowns.

Scopul acestui studiu este de a analiza cu ajutorul microscopiei electronice și a microscopiei FTIR, modul în care se realizează aderența la structura dentară și la diferite tipuri de ceramică a unor materiale adezive. Pentru acest studiu, s-au folosit 45 de dinți extrași, care au fost împărțiți în 3 grupe egale: grupul I restaurat cu coroane ceramice IPS E.max CAD-On, Ivoclar Vivadent cimentat cu Maxcem Elite, Kerr; grupul II restaurat cu coroane ceramice IPS E.max Press, Ivoclar Vivadent cimentat cu RelyX Ultimate Clicker, 3M Espe și grupul III restaurat cu coroane Novodent GS Zirconia cimentate cu Variolink Esthetic. Dinții au fost încorporați în rășină, secționați și supuși analizelor. Atât interfața dinte-ciment, cât și interfața coroană ciment-ceramică au fost evaluate cu ajutorul SEM și FTIR. Materialele utilizate au arătat o bună aderență la structura dentară, iar prezența microfisurilor a fost observată la interfața coroanei ceramice. Mai mult, materialele evaluate au aratat o capacitate de atașare microbiană diferită, cea mai semnificativa inhibare a aderenței a Lactobacillus acidophilus fiind observată în cazul coroanelor de zirconiu cimentate.

Keywords: *dental crown, ceramics, SEM, self-adhesive cement, self-etch cement*

1. Introduction

The demand and interest of dentists and patients for non-metallic and biocompatible restorative materials are constantly growing. In this sense, all-ceramic restorations based on lithium disilicate are among the most accepted restorative treatments. The lack of metal allows these restorations to restore the natural appearance of the dental structure in terms of color and transparency, to which is added a good mechanical strength and an increased value of flexural strength.

Zirconia is a ceramic material with a very good mechanical resistance, which does not corrode over time, it is thermal insulating, biocompatible and bioinert, being very well accepted by the body, without the risk of allergic reactions. Thus, it is possible to use Zirconia in dentistry for the manufacture of endodontic posts, implants, orthodontic brackets, crowns.

The long-term success of total ceramic restorations does not only depend on their properties, but it is also influenced by the way in which they are cemented.

Polymerizable cements are a group of materials used in dentistry that include composite resins, glass ionomer cements and self-adhesive cements. They make strong connections with both the restorative material and the biological tissue on which they are applied. However, their properties are directly dependent on several factors that contribute decisively to their clinical performance: the way in which the polymerization is performed; the nature and degree of processing (conditioning) of the surface to be cemented; degree of mineralization of biological tissue etc [1-4]. Oral bacteria rapidly attach and develop biofilms on dental surfaces and foreign materials (such as orthodontic devices or dental ceramics), and they are an important factor in enamel degradation, caries, periodontal disease and degradation of foreign materials utilized in dental prosthetics [5-6].The oral biofilm (also known

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as dental plaque) formed by cariogenic microorganisms is a complex microbial community in the mouth. Microorganisms such as *Streptococcus mutans, Lactobacillus sp., Bifibobacterium sp., Candida albicans, Actinomyces sp* and *Bifidobacterium sp* are the most investigated ethiologies of caries and degradation of materials developed for oral use [7, 8].

In order to standardize the different results obtained by researchers under different conditions, the behavior and performance of dental cements must now meet the requirements of the standard *ISO 4049 Dentistry-Polymer based restorative materials* [9-11].

The following adhesive cements were used in the study:Maxcem Elite, Kerr; RelyX Ultimate Clicker, 3M Espe and Variolink Esthetic LC/DC, Ivoclar Vivadent.

Adhesive resin cement - RelyX™ Ultimate Clicker, 3M ESPE has a very good adhesion strength, high marginal integrity and wear and tear resistance. It can be used both in association with the total-adhesive technique and with the self-adhesive one. It can be used to cement all restorations (inlays, onlays, crowns, dental bridges, implant abutments, endodontic posts) made of alumina or zirconium oxide, all-ceramic restorations such as glass ceramic, noble alloy or titanium [12].

Maxcem Elite, Kerr is a dual cure resin cement, self-etching and self-adhesive that adheres to all types of substrates: dentin, enamel, all-ceramic, metal and metal-ceramic restorations. It is indicated for luting ceramic, resin and metal restorations, respectively inlays, onlays, crowns, endodontic posts, bridges, veneers (in which case it is still necessary to use an adhesive system) and restorations on implants [13].

Variolink Esthetic LC/DC, Ivoclar Vivadent is a composite luting material, light-curable and with dual setting, for the final cementation of ceramic and composite restorations. It was chosen in the study due to the good adhesion to the dental structure and the dual cure [14].

The purpose of this study is to analyze with the help of the scanning electron microscope, the adhesion to the dental structure of the self-etch (Maxcem Elite, Kerr), universal (RelyX Ultimate Clicker, 3M Espe) and dual cure (Variolink Esthetic LC Ivoclar Vivadent) cements which were used for luting the following types of ceramic crowns IPS E.max CAD-On, Ivoclar Vivadent, IPS E.max Press, Ivoclar Vivadent and zirconia-based ceramics, Novodent GS. Moreover, the attachment of *Lactobacillus acidophilus* on the obtained cemented dental structures was assesed in order establish their ability to allow adherence and biofilm formation of microorganisms with cariogenic potential.

2. Material and Method

The study was performed on 45 teeth, which after extraction were subjected to a short cycle of autoclave sterilization at 134 ° for 35 minutes and then rehydrated for 48 hours in distilled water.

The teeth were fixed in acrylic arches and prepared for restoration with all-ceramic crowns; 2-step impression was made (impression material is found in Table 1); in the laboratory, microprostheses were made, which were cemented with adhesive cements after testing. Depending on the ceramic material and the adhesive cement used, the batch was divided into 3 equal groups (Table 2).

Group I – teeth were prepared for full ceramic crowns (IPS E.max CAD-On Ceramics, Ivoclar Vivadent); cementation with Maxcem Elite, Kerr.

Group II- teeth were prepared for full ceramic crowns (IPS E.max Press Ceramics, Ivoclar Vivadent); the crowns were cemented with RelyX Ultimate Clicker, 3M Espe

Group III - teeth were prepared for zirconia crowns, Novodent GS; the crowns were cemented with Variolink Esthetic LC Ivoclar Vivadent.

Tables 3 and 4 show the chemical composition of the ceramic materials and adhesive cements used in the study. The rest of the materials used in this study are presented in Table 1.

Table 1

Dental materials used in the study /*Materiale dentare utilizate în studio*

Material / *Material*	
Adper Single Bond 2, 3M Espe	2-step ER adhesive system; BisGMA, HEMA, dimethacrylates, ethanol, water, a new photoinitiator system and a functional methacrylate copolymer of polyacrylic and polyacetonic acids, spherical silica particles with nanometer diameter / *Sistem adeziv ER în 2 trepte BisGMA, HEMA, dimetacrilați, etanol, apă, un nou sistem fotoinițiator și un copolimer funcțional de metacrilat de acizi poliacrilici și poliacetonici, particule sferice de silice cu diametru nanometric*
Hydrofluoric acid / *Acid fluorhidric* 10%, Angelus	Conditioner for ceramics / *Condiționer pentru ceramică*
Silano, Angelus	Adhesive agent for ceramics ethanol-based solvent / *Agent adeziv pentru ceramică solvent pe bază de etanol*
Speedex Putty Kit, Coltene	Condensing silicone impression material with physical properties similar to addition silicones, used in the double impression technique / *Material de amprentă din silicon condensat cu proprietăți fizice similare cu siliconii de adăugare, utilizat în tehnica dublei amprentări*
Zeta Plus, Oranwash, Indurent gel, Zhermack kit	Condensing silicone impression material, used in the double impression technique / *Material de amprentă siliconică condensată, utilizat în tehnica dublei amprentei.*

Table 2

Distribution by groups / *Distribuția pe grupe*

	Group 1 / *Grupul 1*	**Group 2 / *Grupul 2***	**Group 3 / *Grupul 3***
MATERIAL / *MATERIAL*	IPS E.max CAD-On, Ivoclar Vivadent	IPS E.max Press, Ivoclar Vivadent	Zirconia, Novodent GS
ADHESIVE CEMENT/ *CIMENT ADEZIV*	Maxcem Elite, Kerr	RelyX Ultimate Clicker, 3M Espe	Variolink Esthetic, Ivoclar Vivadent

Table 3

Composition of ceramic materials / *Compoziția materialelor ceramice*

IPS E. max CAD-On, Ivoclar Vivadent	**IPS E. max Press, Ivoclar Vivadent**	**Zirconia, Novodent GS**
SiO2 57.0 - 80.0 Li2O 11.0 - 19.0 K2O 0.0 - 13.0 P2O5 0.0 - 11.0 ZrO2 0.0 - 8.0 ZnO 0.0 - 8.0 Al2O3 0.0 - 5.0 MgO 0.0 - 5.0 Coloring oxides0.0 - 8.0	SiO2 57.0 - 80.0 Li2O 11.0 - 19.0 K2O 0.0 - 13.0 P2O5 0.0 - 11.0 ZrO2 0.0 - 8.0 ZnO 0.0 - 8.0 MgO 0.0 - 10.0 Coloring oxides0.0 - 8.0	ZrO2

Table 4

The composition of adhesive cements/*Compozția cimenturilor adezive*

Maxcem Elite, Kerr	**RelyX Ultimate Clicker,** 3M Espe	**Variolink Esthetic,** Ivoclar Vivadent
- Barium aluminosilicate30-60% / *Aluminosilicat de Bariu 30-60%* - Ytterbium fluoride10-30% / *Fluorură de Ytterbiu 10-30%* -1.6-hexanediol bis methacrylate5-10% / *1,6-hexanedil bis-metacrilat 5-10%* - 2-Hydroxy-1,3-propanediyl bis-methacrylate 5-10% / *Bis-metacrilat de 2-hidroxi-1,3-propanedil 5-10%* - 7,7,9 (or 7,9,9) -Trimethyl-4,13-dioxo-3,14-dioxa-5,12-diazahexadecan-1,16-diylbismethacrylat 1-5% / *7,7,9 (sau 7,9,9) -trimetil-4,13-dioxo-3,14-dioxa-5,12-diazahexadecan-1,16-diism bismetacrilat 1-5%* - 3 Trimethoxylylpro Propyl methacrylate 1-5% / *Metacrilat de 3-trimetoxililpropropil 1-5%* - Fumarate silica 1-5% / *Silice fumata 1-5%*	- Bisphenol a diglycidyl ether dimethacrylate (BisGMA) / *Dimetacrilat de bisfenol A-diglicidileter (BisGMA)* -Triethylene glycol dimethacrylate(TEGDMA) / *Dimetacrilat de trietilen glycol (TEGDMA)* -Fillings / *Umpluturi* -Pigments / *Pigmenți* -Zirconia/silica photoinitiators / *Fotoinițiatori de zirconiu/silice* -Fumarate silica / *Siliciu fumurat* Basis / Bază: - Methacrylate monomers / *monomeri metacrilat*; - Radio opacifiants / *radioopacifianți*; - Filling / *umplutură*; -Initiators and stabilizers / *inițiatori și stabilizatori;* -Rheological additives / *aditivi reologici*. Catalyst*/ Catalizator*: -Methacrylate monomers / *monomeri de metacrilat*; - Alkaline radio opacifiants / *radioopacifianți alcalini*; -Initiators and stabilizers / *inițiatori și stabilizatori*; - Pigments / *pigmenți*; - Dyes / *coloranți* - Fluorescents / *fluorescenti*; -Rheological additives*/ aditivi reologivi*	-Urethane dimethacrylate (UDMA) / *Dimetacrilat de uretan (UDMA) - Monomeri suplimentari de metacrilat,* -Additional methacrylate monomers / *Trifluorura de iterbiu* -Ytterbium trifluoride / *Trifluorură de iterbiu*-Mixed spheroid oxide - Initiators and stabilizers / *Inițiator și stabilizatori* -Pigments / *Pigmenți* Matrix / *Matrice*: - Urethane dimethacrylate / *dimetacrilat de uretan* - 1,10-Decanediol dimethacrylate 3- <10% / *dimetacrilat de 1,10-decanediol 3- <10%* Fillings / *Umpluturi*: - Ytterbium trifluoride 10- <25%particles size: 0,04 up to 0.2 μm Average: 0.1 μm / *trifluorură de iterbiu 10- <25% dimensiunea particulelor: 0,04 până la 0,2 μm Media: 0,1 μm*

After luting the crowns, the teeth were embedded in acrylic resin and sectioned, following SEM (scanning electron microscopy) analysis.

2.1. Therapeutic protocol

a. Preparation of dental abutment. Each tooth was prepared in the form of a non-retentive cylindrical-conical abutment, by removing a variable layer of 1.5-2 mm from the dental tissue on all surfaces; also, a straight rebate with a width of 1mm was performed.
b. By means of condensing silicones a 2-step impression was made (table 1).
c. In the laboratory the ceramic crowns were performed
d. Adhesive cementation of the crown on the dental abutment

2.1.1.Adhesive cementing protocol

2.1.1.1.Adhesive cementing protocol with Maxcem Elite, Kerr

The inner surface of the restoration was prepared for adhesion in the laboratory by sandblasting with 50μm diameter aluminum particles at a pressure of 30 psi (0.2 MPa). The cement was inserted in the crown, it was applied and maintained on the abutment with moderate pressure until its setting. The excess cement was removed in its gel phase, a phase that was obtained after a short light curing of 2-3 seconds; the final light curing was performed with the Elipar ™ Deep Cure LED lamp, 20 sec/each surface.

2.1.1.2Adhesive cementing protocol with RelyX Ultimate Clicker, 3M Espe

The inner surface of the restoration was prepared for adhesion by applying 10% hydrofluoric acid for 60 sec., followed by washing and application of silane. It was slightly dried.

The tooth surface was prepared for adhesion by applying phosphoric acid for 15 sec, followed by washing and light drying; then the adhesive was applied and light-cured for 10 sec with the Elipar ™ Deep Cure LED lamp.

The cement was introduced into the ceramic crown, and then the crown was applied and maintained on the abutment, with moderate pressure until the cement setting; the light curing was done with the Elipar ™ Deep Cure LED lamp, 20 sec/each surface.

2.1.1.3Adhesive cementing protocol with Variolink Esthetic LC, Ivoclar Vivadent

The inner surface of the zirconia restoration did not benefit from prior preparations. Zirconium-based ceramics, with polycrystalline microstructure without glass, do not benefit from improved adhesion by treatment with hydrofluoric acid and silanization [15-18].

At the level of the dental abutment, after isolation, etching was performed with phosphoric acid for 15 sec, followed by washing and drying, after which the adhesive was applied and lightcured for 10 sec with the Elipar ™ Deep Cure LED lamp; the cement was applied to the inner surface of the restoration; the restoration was correctly positioned and fixed on the abutment by pressure. Excess cement was removed after setting (cement with dual cure) with a scalpel blade # 12.

2.2 SEM Analysis

In order to perform the SEM analysis, the samples wereembedded in resin, and then sectioned horizontally, in a single area (middle) and fixed on an aluminum support of STAB type.

The new formed system was introduced in the Quorum type cover and covered with a 9nm gold layer to perform the conductivity of the investigated sample at SEM for 60 sec. The investigation of the samples was performed using the QUANTA INSPECT F scanning electron microscope equipped with a 1.2 nm resolution field-emission gun (FEG) and energy-dispersive X-ray spectrometer (EDS) with the resolution the MnK of 133eV.

2.3. FTIR Microscopy

IR microscopy was performed by using a Thermo FTIR Nicolet iN10 MX microscope; the spectra were recorded in reflection mode over the wave number range of 675–4000 cm^{-1}, with a resolution of 4 cm^{-1}. The spectra were further corrected using Kramers-Kronig. The spectra were recorded using an imaging detector (MCT detector) in reflection mode, the collection time being 3 s.

2.4. Microbial attachment analysis

Adherence and monospecific biofilm development was assessed at 8h exposurein Lactobacilli MRS (DE MAN, ROGOSA and SHARPE) brothusing sterile 6 well plates (Nunc). One sterile dental material was added in a sterile plate well and 50uL ofMRS broth inoculated with 10^5 CFU (colony forming units) /mL*L.acidophilus* ATCC11975 were added on the section of each cemented enamel materials. The samples were allowed to incubate at 37 ^oC in 5% CO_2 atmosphere for 8h to assess the ability to attach and start biofilm formation on the tested materials. After incubation, materials were carefully washed with sterile saline buffer to remove any unattached microbial cells and then immersed in 1mL sterile saline buffer in sterile tubes to perform biofilm detachment by vigouros vortexing and sonication (10 seconds). The resulting biofilm - detached cell suspensions were further diluted and 10 µL of each serial dilution were plated in triplicate on MRS agar.After 24h of incubation at 37 ^oC, viable count was performed and the CFU/mL values for each group were obtained.

3.Results and Discussion

GROUP I – FULL CERAMIC CROWN IPS E.max CAD-on + Maxcem Elite / *GRUP I- COROANA TOTAL CERAMICA IPS E.max CAD-on + Maxcem Elite*

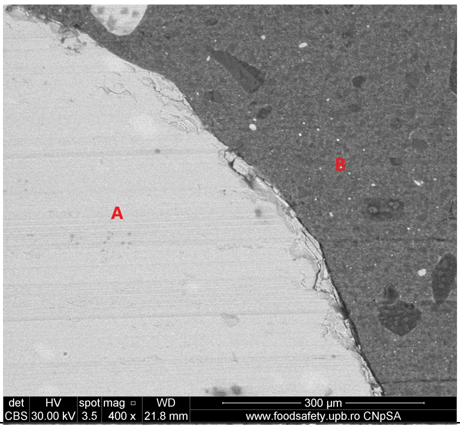

Fig. 1 - SEM image of the IPS Emax crown interface area (A) - adhesive cement (B) / *Imagine SEM 400x a zonei de interfață a coroanei IPS Emax (A) – ciment adeziv (B)*

At the crown-adhesive cement interface there are areas in which the cement adheres very well to the ceramics and limited areas with detached microfragments from the ceramics, but there is no net separation of the 2 materials. There are no gaps, the mass of the adhesive is homogeneous, evenly arranged and intimate adherent to the crown. / *La interfața ciment-adeziv ciment există zone în care cimentul aderă foarte bine la ceramică și zone limitate cu microfragmente detașate de ceramică, dar nu există o separare netă a celor 2 materiale. Nu există goluri, masa adezivului este omogenă, dispusă uniform și aderentă intimă la coroană.*

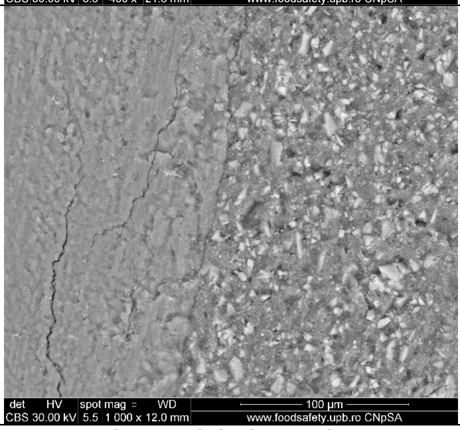

Fig. 2 - SEM image of the adhesive cement interface area / *Imagine SEM 1000x, a zonei de interfață a cimentului adeziv*

There is a very good adhesion of the cement to the dental structure, without cracks, gaps and/or fracture lines. / *Există o aderență foarte bună a cimentului la structura dentară, fără fisuri, goluri și / sau linii de fractură.*

GROUP II – FULL CERAMIC CROWN IPS E.max Press+ RelyX Ultimate Click / *GRUP II – COROANA TOTAL CERAMICA IPS E.max Press+ RelyX Ultimate Click*

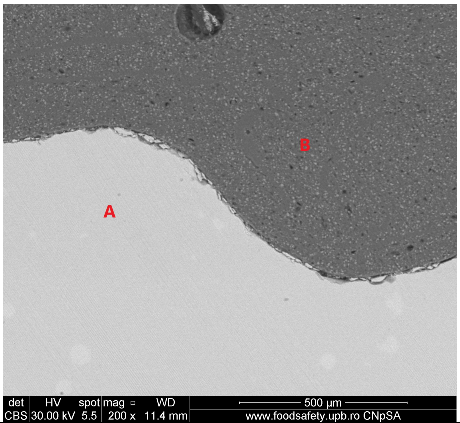

Fig. 3 - SEM image of the crown interface area IPS E.max Press, (A) - adhesive cement (B) / *Imagine SEM 200x a zonei de interfață coroana IPS E.max Press, (A)- ciment adeziv (B)*

At the border between the two materials, detached microfragments from the ceramic can be seen, probably due to the stresses triggered by the cement polymerization shrinkage. There is no clear demarcation limit of the materials. / *La limita dintre cele două materiale se observă microfragmente detașate din ceramică, apărute probabil din cauza tensiunilor declanșate de contracția de polimerizare a cimentului. Nu se remarcă o limită clară de demarcație a materialelor, ceea ce sugerează faptul că cimentul aderă foarte bine la ceramică.*

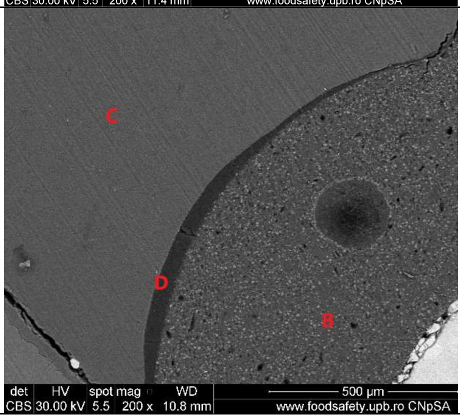

Fig. 4 - SEM image of the tooth interface area (C) - adhesive system (D) - adhesive cement (B), 200x / *Imagine SEM a zonei de interfață dinte (C) - sistem adeziv (D) - ciment adeziv (B), 200x*

There is very good adhesion between the 3 substrates, tooth, adhesive and cement, the interfaces do not show cracks, gaps or detached fragments. At the tooth-adhesive boundary, the hybridized layer is slightly highlighted, which suggests a minimal chemical adhesion. / *Se remarcă adeziunea foarte bună între cele 3 substraturi, dinte, adeziv și ciment;interfețele nu prezintă fisuri, goluri sau fragmente detașate. La limita dinte-adeziv, stratul hibridizat este puțin evidențiat, ceea ce ne sugerează o adeziune chimică minimă.*

GROUP III – ZIRCONIA CROWN Novodent GS + Variolink Estethic LC / *GRUP III – COROANA ZIRCONIA Novodent GS + Variolink Estethic LC*

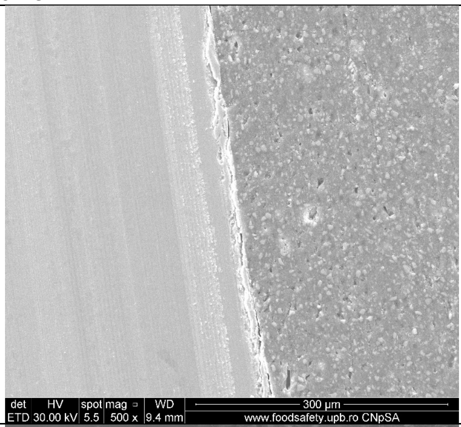

Fig. 5 - SEM image of the crown area - adhesive cement. / *Imagine SEM 500 x a zonei coroană - ciment adeziv.*

Presence of microcracks can be observed at the boundary of the crown with the cement, appeared as a result of the tensions triggered by the polymerization shrinkage of the cement. / *Se observă prezența microfisurilor la limita coroanei cu cimentul, apărute ca urmare a tensiunilor declanșate de contracția de polimerizare a cimentului.*

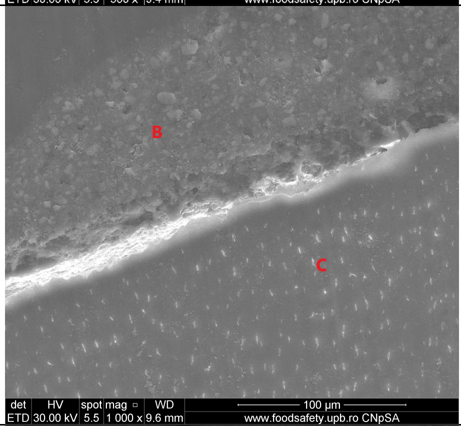

Fig. 6 - SEM image of the interface adhesive cement (B) - adhesive system (hybrid layer) - tooth (dentin) (C) / *Imagine SEM 1000 x a zonei de interfață ciment adeziv (B)- sistem adeziv (strat hibrid)- dinte (dentină) (C)*

There is very good adhesion of the structures involved, without cracks or gaps.
The hybridized layer is very well highlighted, with a uniform, homogeneous thickness, the micro-retentions being present on the whole adhesion area. / *Se remarcă adeziunea foarte bună a structurilor implicate, fără fisuri sau goluri.*
Stratul hibridizat este foarte bine evidențiat, cu o grosime uniformă, omogen, microretențiile fiind prezente pe toată zona de aderență.

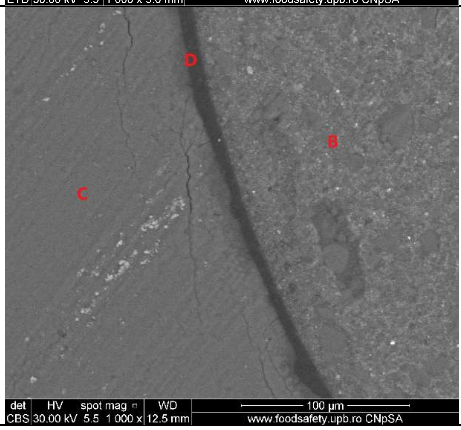

Fig.7 - SEM image of the interface area tooth(enamel) (C) - adhesive system (D) - cement (B) / *Imagine SEM 1000x a zonei de interfață dinte (smalt) (C)- sistem adeziv (D)- ciment (B)*

No cracks or gaps are observed on the SEM image of the tooth-adhesive-cement interface; the adhesive layer has a relatively constant thickness.
The hybridized layer is less pronounced as opposed to dentin adhesion, but it is present. / *Pe imaginea SEM a interfeței dinte-adeziv-ciment nu se observă fisuri sau goluri; stratul de adeziv are grosime relativ constantă.*
Stratul hibridizat este mai slab evidențiat spre deosebire de adeziunea la dentină, dar este prezent.

An important aspect that ensures the long-term success of a direct or indirect restoration is the quality of the adhesion between the materials used and the dental structure.
In this study three types of ceramics were analyzed (IPS E.max CAD-on-Ivoclar Vivadent, IPS E.max Press-Ivoclar Vivadent, Zirconia Novodent GS) and three types of adhesive cements: self-adhesive, universal and dual cure cement, MaxCem Elite Kerr, RelyX Ultimate Clicker-3M Espe, Variolink Esthetic LC/DC, Ivoclar Vivadent.

Analyzing with the help of SEM the tooth-cement adhesive interface, it was observed that the adhesion is made without gaps, cracks, fractures at the level of the structures involved for all 3 cements (Fig. 2, 4, 6, 7). This supports the long-term success of the adhesion. The hybridized, homogeneous and uniform layer, present especially in Figures 6 and 7, suggests a good chemical retention of the dentin adhesive, respectively enamel.

Instead, the crown-adhesive cement adhesive interface presented, on all analyzed models, discontinuities, micro-fragments and micro-cracks. These may be due to the stresses triggered by the polymerization shrinkage of the cements, the fracture resistance that is different for each material but also to an insufficient preparation of the internal surfaces of the crowns.

Currently, all available cements based on resin show polymerization shrinkage. To this fact it also adds their application at the level of preparations that have a high C factor. These factors can generate enough stress to lead to the debonding of the cementing material [19]. However, there are insufficient studies in the literature on the stress generated by adhesive cements and also studies to make a correlation between the stress generated by the polymerization shrinkage and the fracture strength of the adhesive cemented material.

At the SEM analysis of the ceramic crown-adhesive cement interface MaxCem Elite (Kerr) (Fig. 1) it was observed an alternation of areas in which the cement adheres very well to ceramics and areas with detached micro-fragments from ceramics, there is no net separation of the 2 materials. The appearance of these micro-fragments can be determined by the polymerization shrinkage of the cement, but also the fracture resistance of this material should be taken into account.

There are studies showing that MaxCem Ellite (Kerr) cement, compared to other types of self-adhesive cements, has a more pronounced mechanical stress and a higher polymerization shrinkage [20].

Also in the case of RelyX Ultimate Clicker cement, at the adhesive-crown cement interface the presence of micro-cracks is observed (fig. 3) which could be due on the one hand to the cement polymerization shrinkage, which FRASSETTO A et al. [20], following the tests performed, found with the highest contraction coefficient, and on the other hand, when the micro-cracks appear, the fracture resistance of the ceramic must be taken into account, which is different in the case of the two IPS E-max materials. Although both materials are ceramics based on lithium disilicate glass, the different sizes of lithium-disilicate crystals that form in the structure of the material differentiate them. This leads to different mechanical properties, fracture resistance being one of them. Fracture resistance is responsible for the initiation and propagation of cracks and the clinical performance of restorations. From this point of view, IPS e.max Press is superior to IPS e.max CAD ceramics, as claimed by Alkadi et al. [21].

Comparing the cementation of ceramic crowns based on lithium disilicate glass cemented with adhesive cements with those conventionally cemented, Mobilio et al. [22] show that the adhesive cemented ones have higher failure rates and also in their case the failure is most often determined by the fracture, while the conventionally cemented crowns with glass-ionomer cements are lost by debonding.

Good adhesion to dentin of Maxcem and RelyX cements is also presented in the study of Vieira-Filho et al. who observed that the dentinal area with which the adhesive cements come into contact is important, the cements showing a superior adhesion to the deep dentin compared to the superficial dentin[23].

In the case of zirconium-based ceramic reconstitutions, it is recommended to use resin-based cements because they showed a strong adhesion to both dentin and zirconia compared to conventional cements [24].

In group III of materials, when examining the SEM images it was observed on the one hand that the cement used, Variolink Esthetic LC/DC formed a continuous connection, without gaps and cracks with the tooth, but at the interface with the zirconia crown, microcracks appeared probably as a result of the stresses triggered by the cement polymerization shrinkage (Fig. 5).

The good adhesion between Variolink cement and dentin is also presented by Wang et al. [25], and Patroi et al. [26] argue that there is no significant difference between the adhesion to enamel and dentin of this cement.

Although, due to its composition, zirconia-based ceramics do not benefit from hydrofluoric acid treatment and silanization before adhesive cementation, clinical results have shown that additional preparation of this material is needed in order to improve the adhesion quality.

Opinions on the preparation of the zirconia surface for adhesion are divided and there are many studies that verify the possibilities for its improvement. Thus, Martins et al. [27] claim that by treating the zirconia surface with glass particles and silane, the adhesion quality is greatly improved.

Conrad claims that the treatment of the zirconia ceramic surface by different processes (tribochemical silica coating, abrasion with aluminum oxide particles 250-μm or 50-μm, combination of abrasion with aluminum oxide particles 50-μm with the use of hydrofluoric acid or abrasion with diamond rotating tools) has a limited influence on the adhesion to zirconia-based ceramics. Conrad also shows that many adhesive cements are able to adhere to crowns based on zirconium oxide. [28].

DS Russo claims that there is no universal adhesion protocol, although in the literature the most verified pretreatment methods are tribochemical silica coating and abrasion with aluminum oxide particles. An improved adhesion would be expected after the physico-chemical conditioning of zirconia. Of course, contamination of surfaces has a negative effect on adhesion [29].

Conrad [28] concludes that, while the mechanical properties of adhesive cements are important when cementing glass-based ceramic restorations, zirconia-based ceramic crowns can be conventionally cemented due to their high fracture strength. These restorations do not require an adhesive retention interface.

In group I, the cement-crown interface is highlighted by FTIR microscopy (Figure 8). Based on the video image it can clearly see that the interface is less than 10um but thickness in the FTIR images is considerable larger which means that strong interactions appears between the two phases, and this can be seen at both maps highlighted in Figure 8 (corresponding to phosphate - 1065 as well as to carbonyl region – 1658cm^{-1}). Also, a characteristic FTIR spectrum is appended.

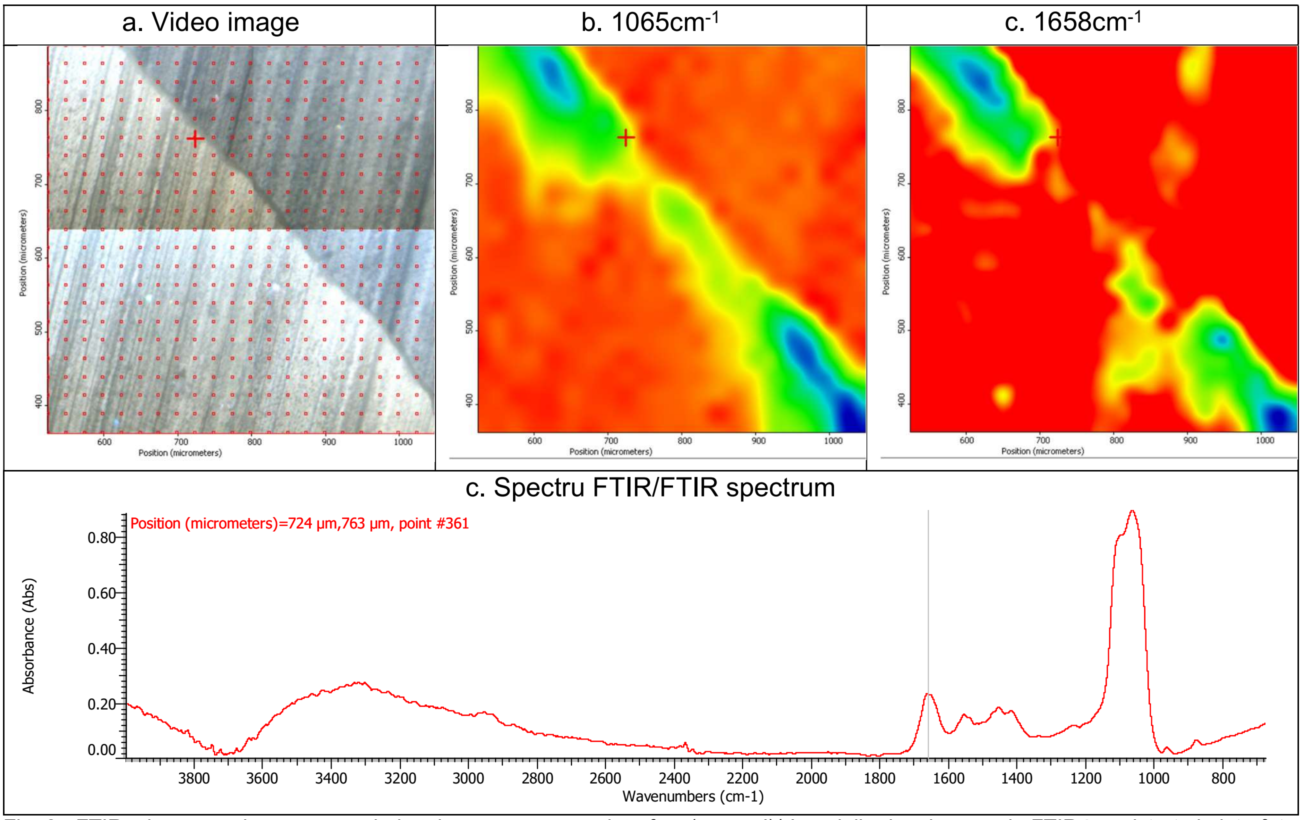

Fig. 8- FTIR microscopy images recorded at the cement-crown interface(group I)/ *Imaginile de microscopie FTIR înregistrate la interfața ciment dentar-coroană (grupul I)*

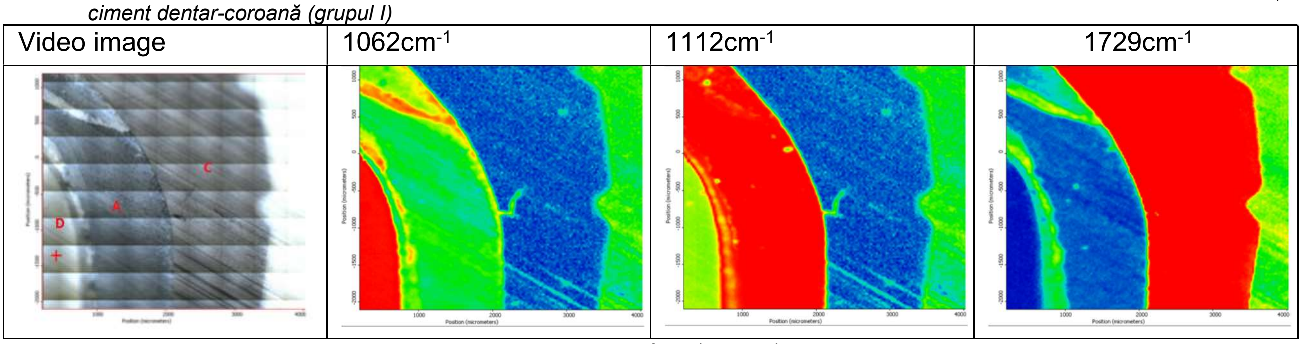

Fig. 9 - FTIR microscopy images recorded at the cement-crown interface (group 2) / *Imaginile de microscopie FTIR înregistrate la interfața ciment dentar-coroană (grupul II).*

In group II, an overview of the section is highlighted by FTIR microscopy (Figure 9). Based on the video image it can clearly see the specific regions, from the dentin (D) to the crown (C) and the intermediate layer namely dental cement (Cem) and even the adhesive layer (A). Based on the maps recorded at the specific wavelengths corresponding to HA (1062cm^{-1}), cement (1112cm^{-1}) and crown (1729cm^{-1}). It is important to mention that even if some cracks appear (indicated in the circle) at the crown level, cement is well filling these cracks. Also, at the dentin-cement interface, due to the pretreatment, a specific area can be highlighted in all the FTIR maps.

In group III, an overview of the section is highlighted by FTIR microscopy (Figure 10). Based on the video image, it can clearly see the specific regions, from the dentin to the crown and the intermediate layer namely dental cement as presented previously. Based on the maps recorded at the specific wavelengths corresponding to HA (1058cm^{-1}), cement (675cm^{-1} – minimum wavelength compatible with the MCT A detector) and crown (1729cm^{-1}). It is important to mention that both interfaces are smooth, with no visible cracks/defects. In this case, no dentin surface treatment was requested and thus no specific areas between dentin and cement are visible. In general, it can sees a very good adhesion between the used cement and the both surfaces.

Regarding the ability of *L. acidophilus* to attach on the tested dental materials, our results showed they present different microbial attachment abilities, depending on the utilized ceramics and cement. Differences among group I and II materials were not significant, in terms of Lactobacillus attachment (CFU/mL values ranging 3000-3500), but group III (zirconia ceramics cemented section) showed a significant 1 log attachment inhibition of *L. acidophilus* as compared with the other 2

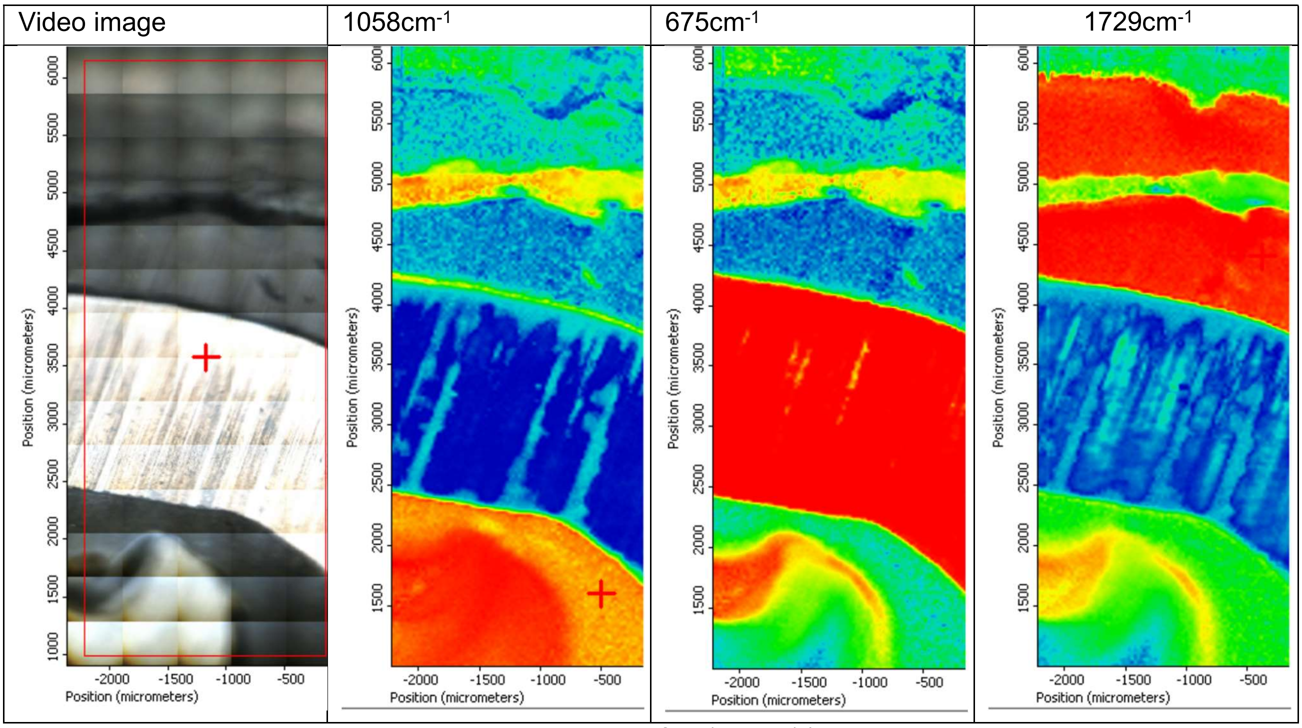

Fig. 10 - FTIR microscopy images recorded at the cement-crown interface (group III) / *Imaginile de microscopie FTIR înregistrate la interfața ciment dentar-coroană (grupul III).*

materials (Figure 11). This result can be explained by the recent literature studies which report an increased antimicrobial and biofilm development inhibition of Zirconia ceramics against dental pathogens [30,31]. Also, differences in microbial attachment can be explained by different surface porosity and cracks which were observed by SEM analysis on the evaluated dental sections. It is well known that microbial attachment is influenced by surface roughness and also hydrophobicity [32,33].

4. Conclusions

Analyzing the images obtained with SEM, we observed the good adhesion to the dental structure of all cements used, the tooth-adhesive cement interface or tooth-adhesive system-adhesive cement interface without gaps or cracks, on some SEM images the hybridization being obvious. FTIR microscopy can be also used to evaluate the interfaces and even the ability of the cement to fill the available cracks.

Regarding the adhesive cement-crown interface, the appearance of detached microfragments from the three types of materials used, can be related to several factors: the polymerization shrinkage of the adhesive cements used, the method of processing the inner surfaces of the crowns and the fracture resistance of materials, more studies being needed to determine whether there is a correlation between these factors. The evaluated cements showed slightly different ability to allow the adherence of *L. acidophilus*, the best attachment inhibition potential being observed in the case of group III ceramics. Adherence modulation of microorganisms with cariogenic

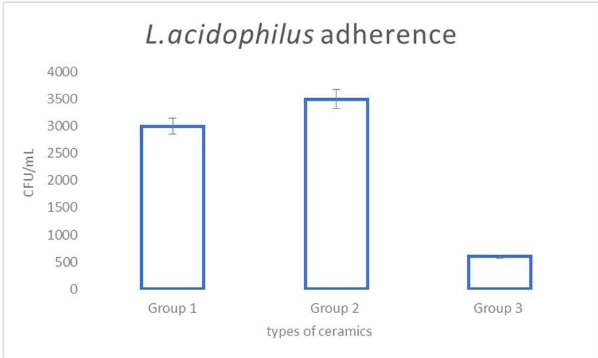

Fig. 11 - Graphic representation of the *L. acidophilus* attachment after 8h of incubation on the evaluated dental ceramics cemented sections. / *Reprezentarea grafică a atașamentului L. acidophilus după 8 ore de incubație pe secțiunile cimentate din ceramica dentară evaluate*

potential represent an efficient strategy to limit oral biofilm formation and thus degradation of enamel and prosthetic oral materials, such as ceramics and cements.

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