UNIVERSITY OF MEDICINE AND PHARMACY
„CAROL DAVILA”, BUCHAREST

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DENTISTRY
DOCTORAL STUDIES

PHD THESIS

PhD supervisor:
ACAD. PROF. UNIV. DR. ANDREI ILIESCU

PhD Student:
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STUDIES ON THE ASSESSMENT OF AESTHETIC RESTORATION MADE OF COMPOSITE MATERIALS

PHD THESIS SUMMARY

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Cuprins:

Introduction

The current desire for the quality of the composite restoration is that the restoration material retains its integrity, shape, color, surface characteristics and correct marginal adaptation over time. In these studies, the quality of composite resin restorations, applied to teeth with endodontic treatment completed by root filling, is analyzed. In this context, it is extremely important that the composite restoration protects the quality of the sealing of the endodontic system, being performed in the same treatment session, as much as possible, to prevent micro infiltration from the coronal side.

The elimination of germs is imperative for the success of endodontic treatment, the most widespread cause of its failure being coronary percolation, which provides not only microorganisms and bacterial toxins, but also nutrients, coming from saliva.

The PhD thesis is structured in two parts, the general part which includes two chapters, respectively the current state of knowledge regarding restorative materials made of composite materials, composite resins, being made of micro- and nano-particles of glass and ceramics, embedded in organic polymer. They are electrical insulators and have a complex composition.

The following subchapter describes the structure and properties of composite materials, characterized by atomic and nuclear methods. It includes the elemental analysis of dental compositions by PIXE, ERDA and XRF, their microscopic structure, respectively the inorganic particles, micro-spectroscopic studies with m-PIXE, the organic polymer, changes of the composites in the oral environment.

Theoretical aspects are presented regarding polymerization shrinkage, Ca^{2+} and F^- release and ion diffusion, correlations of enamel demineralization with Ca concentration in composites, measured by PIXE, heterogeneous Ca distribution measured by μ-PIXE and electrochemical processes, PIXE- studies Microbeam PIGE of the dental hard-composite interface.

The notions of microbiology specific to dentistry, respectively in restorative dentistry and endodontics, are the subject of the 2nd chapter.

The 3rd and 4th chapters present the personal contributions, the working hypothesis and the general research methodology.

The personal research includes a number of three chapters. The 5th chapter includes a clinical, radiological, statistical and microbiological study, presenting the consequences of defective coronal restoration on the endodontic system, in teeth with prior endodontic

treatment, completed by root filling. A separate sub-chapter is reserved for the microscopic assessment, with the dental microscope, of the integrity of the composite resin restorations.

In the case of infiltration of the root filling, in the case of defective coronal restorations, microorganisms organized in the form of a biofilm penetrate the pulp chamber and the root canal, at the interface with the root canal wall and the root filling material, in the apical ramifications, compromising the sealing quality the endodontic space.

The 6th chapter of the personal research is represented by a microscopic experimental study on freshly extracted teeth, regarding the quality of the coronal restoration.

Chapter 7 includes personal research on the potential of the *in-air* PIXE (particle-induced x-ray emission) method for elemental analysis of dental composites as well as the characterization of composite resins by Compton/Rayleigh scattering ratio, measured with portable XRF spectrometers.

The processes involved in composite-tooth interactions are extremely complex, in which atomic and nuclear methods play an increasingly important role. It is a field with many possibilities, which is developing both in terms of the emergence of new methods and in that of fundamental and applied dental research.

Due to the complexity and diversity of the elemental composition, dental materials with different compositions may show similar or close C/R ratios, and materials with the same qualitative composition but with different concentrations show different values of the C/R ratio.

This elemental analysis aims to detect the differences in BULK vs. CLASSIC composites in mineral phase compositions.

The results and conclusions of the studies will contribute to increasing the performance of composite resin restorations, allowing that, following the resumption of endodontic treatment, in case of infiltration of the canal filling, the application of the new composite material restoration takes place as soon as possible, preferably in the same session treatment.

The conclusions of the conducted studies were the basis for the formulation of the general conclusions of this PhD thesis and the research results allowed the development of recommendations with clinical applicability regarding the quality of the composite restoration and the consequences of a deficient composite filling.

Coronal restoration made with composite resins must be applied in such a way as to prevent micro infiltration, preventing microbial invasion as well as bacterial endotoxins, which cause and maintain periapical inflammation.

The crown restoration applied following the endodontic treatment, completed with root filling, is as important as the quality of the endodontic treatment itself.

Personal contributions

In this work I proposed to study ways to improve the physical and chemical properties of composite materials, the marginal sealing of this type of restoration, to increase the clinical performance of these biomaterials.

With the help of the first study, we tried to prove the fact that the composite resin restoration made inadequately, can influence the quality of the root filling, with the infiltration of the sealing of the endodontic system, potentially jeopardizing the long-term evolution of the success of the endodontic treatment. We also carried out a microscopic assessment in a clinical study on the integrity of the coronary restoration using composite resin, at the same time we studied from a radiological point of view the deficient coronary restoration and its consequences on the quality of the root filling.

In the experimental microscopic study on freshly extracted teeth, we analyzed the quality of the composite resin crown restoration using transmitted light microscopy and stereomicroscopy.

The following experimental study on the structure and properties of composite materials by atomic and nuclear methods of analysis, refers to the potential of the *in-air* PIXE method for elemental analysis of dental composites as well as to the characterization of composite resins by the Compton-Rayleigh scattering ratio, measured with portable spectrophotometers.

1. Working hypothesis and general objectives

The general objective of this research is to analyze the quality of composite restorations, both in a clinical, radiological context and materialized in a microbiological and statistical study, with regard to the consequences of root filling infiltration, which starts from the coronary level, mainly due to inadequate composite restorations.

The purpose of the experimental study is to evaluate the extent to which the microscopically analyzed coronal restorations present deficiencies, as well as their consequences on the quality of the root fillings.

To improve the structure and physical and chemical properties of the composite material as well as to increase its clinical performance, we used atomic and nuclear methods of elemental analysis as well as characterization of composite resins by spectrometry.

2. General research methodology

We studied a number of 258 teeth with prior endodontic treatments, being coronally restored with composite material placed in class I and II cavities, in upper and lower premolars and molars. Study protocols are in accordance with The Scientific Research Ethics Committee of the UMF "Carol Davila" from Bucharest, approval no. 33192/02.11.2023. A number of 28 teeth were excluded from the study for the following reasons: complete and incomplete cracks of dentin and cementum (13 teeth), vertical fracture of crown and root (4 teeth), severe periodontal pathology (11 teeth).

A number of 230 teeth were included in the research (202 molars and 28 premolars) of which: premolar 1 - 10 teeth; premolar 2 - 28 teeth; molar 1 - 136 teeth; molar 2 - 89 teeth and molar 3 - 5 teeth. From the total number of 230 teeth studied, a number of 49 teeth were extracted, a number of 19 teeth due to endodontic treatment complications and 30 teeth due to severe periodontal pathology, on the latter the experimental study was carried out.

From the total number of teeth studied clinically and radiologically (181 teeth), a number of 110 teeth were selected with deficient coronal restorations and infiltrated root fillings, and in the remaining 69 teeth, the coronal restoration and root filling were considered correct.

From the group of teeth that was studied clinically and radiologically, teeth that showed deficiencies of the coronal restoration, microscopic investigations were made with the dental microscope on a number of 41 teeth.

Microbiological investigations were also carried out on a number of 34 teeth.

The experimental study was carried out on a number of 49 teeth. Of these, only stereomicroscopy was performed on 8 teeth and thin sections were obtained from 33 teeth that were investigated with a laboratory microscope to highlight marginal sealing. A number of 8 teeth were removed from the experimental study due to errors made during their sectioning.

Deficiencies of the marginal closure of the restorations were identified in 41 teeth. Of these cases, 31 had fillings with moderate marginal surplus and 9 with fillings with marginal deficit. This means a percentage of 39.24% and respectively 11.39% of the initial set.

In-air PIXE method was used at the Tandetron HH-NIPNE (INFIN-HH) using 3 MeV protons and an SDD detector for qualitative elemental analysis of dental composites. Up to 15 elements including trace elements were detected in these biomaterials.

Regarding the FRX spectra of a selection of seven dental composites, they were recorded with portable spectrometers. For the first time, the ratio of Compton/Rayleigh scattering line intensities (C/R) was monitored for the purpose of characterizing dental materials. The C/R ratio proved to be a relevant characteristic of biomaterials, covering a wide range of values (between 0.5 and 8).

Method of evaluation

For the data analysis, the studied parameters were described in relation to the registration modality and the groups were compared, in this sense the Fischer test was used. Data analysis was performed with SPSS Statistics software, and the significance threshold used was $p<0.05$.

Regarding the personal research, the microbiological clinical statistical study aiming at the quality of the coronary restoration, I found the following:

The study was conducted on 230 teeth, most of them being 1st molars (n=90; 42.6%) or 2nd molars (n=89; 38.7%). Almost half of teeth studied had incorrect fillings (n=161; 70%), periapical pathology was observed in almost half of the number of teeth (n=110; 47.8%), 18 teeth (7.8%) were extracted due to endodontic treatment complications. The odds of having a periapical radiolucency are about 3 times higher in teeth with incorrect coronal restorations than in those with correct coronal restorations, OR [95% CI] = 3.16 [2.52; 3.96]; $p<0.001$. The existence of an incorrect obturation does not significantly increase the risk of tooth extraction for reasons related to complications of endodontic treatment, with or without periapical radiolucency, related to root filling infiltration, OR (95% CI) = 1.16 [1.08; 1.25]; $p<0.001$.

3. Personal research, clinical, radiological, statistical and microbiological study regarding the quality of the composite resin coronal restoration in teeth with endodontic treatment, completed with root filling

Marginal adaptation of restorations involves two aspects: the perfect sealing of the interface between the filling material and the surface of the cavity, so that there are no infiltrations of fluids and bacteria from the oral cavity. The placement of the filling material must be done in such a way that it extends to the limit between the prepared surface (cavity walls) and the external coronal surface.

3.1 Clinical study on the deficient coronary restoration and its consequences on root filling:

The quality of composite resin restorations together with the correct endodontic treatment is the long-term guarantee of a filling that ensures the absence of marginal infiltration, which can be the causative factor of compromising the quality of the root filling. In the present study, the importance of properly performed composite crown restoration after the resumption of endodontic treatment, in the case of marginal infiltration of the root filling, is illustrated. It must be taken into account that before accessing the endodontic system in order to resume the endodontic treatment, the dentin affected by the carious process must be completely removed.

Consequently, the root filling must be followed in the shortest possible time by a correct filling of the endodontic access cavity and, if necessary, of other deep cavities of the tooth, taking into account possible enamel cracks, marginal infiltrations, secondary caries, marginal discoloration, cracks or fractures of the restoration margin and some errors during the application of the restorative material, regarding the correctness of the insertion, shaping and finishing steps.

3.1.1. Clinical aspects of the quality of composite resin crown restorations – microscopic evaluation:

3.1.1.1 Introduction

The present study addresses the issue of the marginal adaptation of restorations, from the point of view of their correct extension [106, 107], respectively of the restorative material

in relation to the boundary prepared surface - unprepared surface (taking into account the landmark considered, namely the tip of the angle cavosurface).

3.1.1.2 Material and method

The selection criteria of the teeth that presented occlusal and approximal fillings of some class I and II cavities were the following: tooth type (premolar or molar, maxillary and mandibular), composite coronal restoration. Six types of restorative materials were used: Ariston, Tetric Ceram, Tetric Bulk (Ivoclar-Vivadent) and Valux, Filtek, and Filtek Bolk (3M-ESPE). The age of the obturation was not taken into account, in most cases clinical records were available and other information was obtained, such as the occlusal context and the evolution of periodontal pathology in the medical history.

From the group of teeth that was clinically and radiologically studied, which presented deficiencies of the coronal restoration, microscopic investigations were carried out with the dental microscope on a number of 41 teeth, which were photographed with the digital camera attached to the dental microscope, and then the image was be transferred to a PC. Surface measurements of restorations with deficit or moderate surplus were determined, finding that those with moderate surplus were more common than those with deficit.

The marginal defects of the restorations were identified with a ZEISS dental microscope with a built-in digital camera, controlled by an attached PC.

3.1.1.3 Results and discussions

Although with this type of microscope it was not possible to evaluate the magnitude of the filling deficiencies in terms of their correct extension in relation to the prepared surface - unprepared surface boundary (with the apex of the cavosurface angle), numerous such deficiencies were identified, which are responsible, in most situations, by the infiltration of the endodontic space seal.

Next, we present clinical situations represented by lateral teeth with coronal fillings deficiencies:

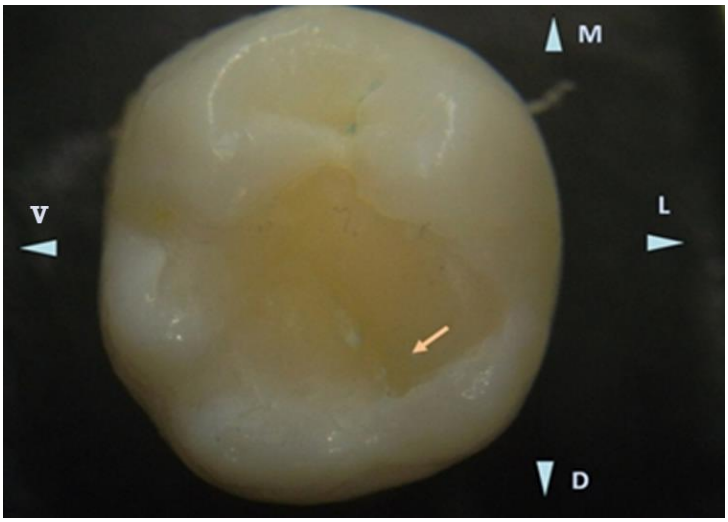


Fig. 5.4 Extended occlusal restoration at 3.8.

The color difference between the enamel surface and that of the composite resin makes it possible to detect the obturation limits by microscopy. The disto-lingual cusp is incorrectly restored - a negative relief is observed. (10x)

V – vestibular; L – lingual; M – mesial; D-distal

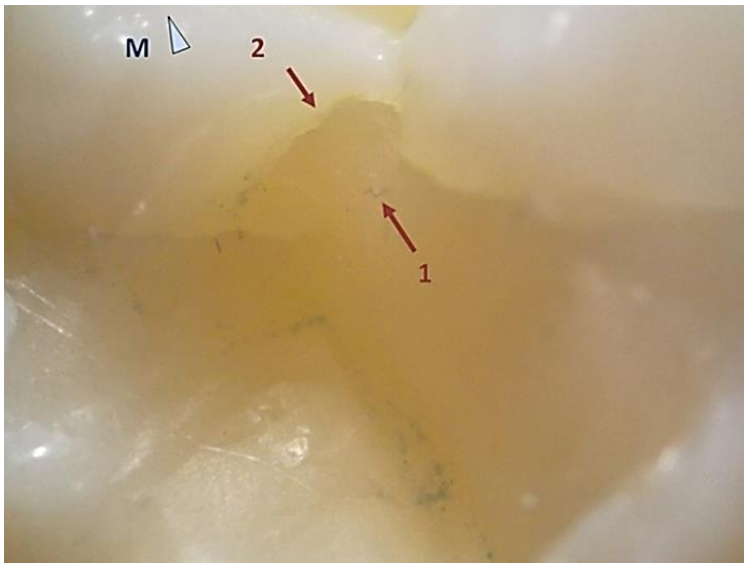


Fig. 5.5 Detail image: the mesial limit of the occlusal obturation at 2.8.

Marginal adaptation is relatively good in this area (arrow 2). The finishing of the edge is not appropriate: the fractured edge (arrow 1) and marginal color can be observed through transparency, as well as the presence of a dehiscent area, which implies micro infiltration (20x)

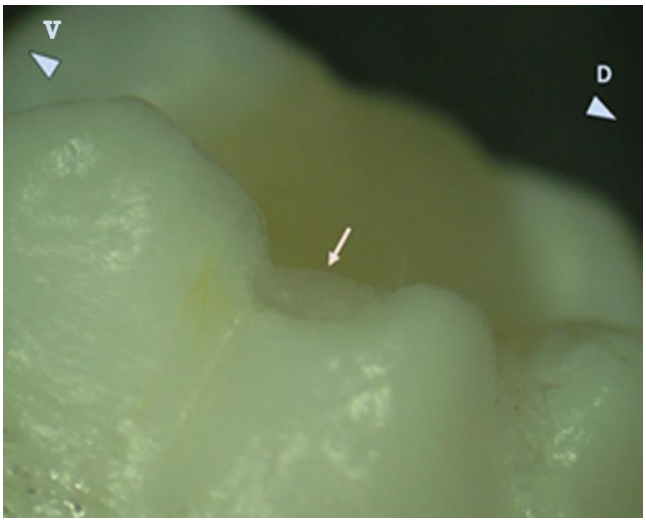


Fig. 5.6 Vestibular detail of the occlusal restoration at 3.8. A portion of moderately excess material can be seen covering the DV cusp slopes reflected light stereomicroscopy (12.5x)
V – vestibular D-distal

3.2 Radiological study on the quality of coronary restoration:

3.2.1 Results and discussions:

We have selected, for example, a number of 12 clinical cases, in which we identified the composite restoration with deficiencies, which resulted in the infiltration of the root filling, necessitating the resumption of endodontic treatment, followed by coronary restoration, in the same session, using composite resin.

Depending on the predetermined radiological criteria, the technical quality of the root filling is interpreted aside, as well as possibly the presence of elements that betray a non-conforming composite resin restoration, which would allow micro infiltration.

Correct restorations combined with dense three-dimensional root filling had the highest success rate [126-127].

The quality of good endodontic treatment (Rx) was more important than the technical quality of the coronal restoration when evaluating the periapical status of the endodontically treated tooth. [116-125, 221]

Clinical Case No. 1

Tooth 4.6, with multiple incorrect composite resin crown restorations; after the removal of the coronal obturations, it is found on palpation with a probe, at the level of the opening holes of the mesiobuccal and mesiolingual canals as well as on the approach, with the endodontic instruments, of the infiltrated sealer, which justifies the therapeutic attitude of resuming the endodontic treatment.

Fig. 5.9 Tooth 4.6 with multiple deficient coronal restorations, with appearance of virtual spaces and voids, infiltrated root filling

Fig. 5.10 Tooth 4.6 after the resumption of endodontic treatment

Clinical Case No. 2

Tooth 2.7 has a composite resin coronal restoration, with a deficit in the distal area due to the fracture of a fragment of the restorative material. Following the removal of the remaining coronal obturation, infiltrated sealer was identified, necessitating the resumption of endodontic treatment.

Fig 5.13 Tooth 2.7 dehiscent coronary obturation

Fig. 5.14 Tooth 2.7 after the resumption of endodontic treatment. The composite resin coronary restoration was performed in the same session

Clinical Case No. 3

Tooth 4.6 with an occlusal crown filling made of composite resin incorrectly adapted to the cavity walls. The radiological image betrays a misfit of the restorative material at the level of the vertical cavity, which determined the infiltration of the root filling, compromising its quality. On probe inspection, the fragility of the distal wall of the tooth structure could be observed on the coronal surface. Tooth 46 has a cover crown indication to avoid crown fracture. Resumption of endodontic treatment is recommended due to compromised root filling.

Fig. 5.25 Tooth 4.6: with improperly adapted coronal obturation compromising the quality of the root filling

Fig. 5.26 Tooth 4.6: it is decided to resume the treatment and the coronal obturation in the same treatment session.

3.3 Statistical data analysis

From the 258 teeth analyzed, a number of 28 teeth were excluded from the study for the following reasons: complete and incomplete dentine and cementum cracks (13 teeth), vertical crown and radicular fracture (4 teeth), severe periodontal pathology (11 teeth). Thus, a number of 230 teeth were included in the study.

Endodontic treatments were carried out in a similar proportion to maxillary and mandibular teeth, being generally more frequently carried out on molars than on premolars, and of these the teeth on which endodontic treatments were most frequently carried out were molars 1 and 2 (Table 5.1.).

Table 5.1. Teeth analyzed

		Tooth					
		Pm1	Pm2	M1	M2	M3	Total
Maxillary	Nr	1	11	51	47	2	112
	%	0.9%	9.8%	45.5%	42.0%	1.8%	100.0%
Mandibular	Nr	9	17	47	42	3	118
	%	7.6%	14.4%	39.8%	35.6%	2.5%	100.0%
Total	Nr	10	28	98	89	5	230
	%	4.3%	12.2%	42.6%	38.7%	2.2%	100.0%

Analyzing the relationship between **the correctness of the composite obturation and the tooth extraction in relation to infiltrated, compromised root filling, with and without periapical radiolucency** for 198 teeth (teeth extracted for other reasons were excluded from the analysis), it was found that although a statistically significant difference, the value of OR close to 1 suggests that the existence of an incorrect obturation does not increase the risk of tooth extraction for reasons related to endodontic treatment (Table 5.5)

Table 5.5 The relationship between the correctness of the composite restoration and the tooth extraction

			Tooth extraction was performed in relation to the infiltration of the root filling		
			NO	YES	Total
Composite restoration	Correct	Nr	69	0	69
		%	100.0%	0.0%	100.0%
	Incorrect	Nr	111	18	129
		%	86.0%	14.0%	100.0%
Total		Nr	120	180	18
		%	52.2%	90.9%	9.1%
OR (95% CI) = 1.16 [1.08; 1.25]; p<0.001					

3.4 Microbiological investigation of the microbial load of the endodontic system in case of deficient coronal restorations:

3.4.1 Personal research

In order to highlight the infiltration of the endodontic system, with the compromise of the endodontic treatment completed by root filling, in the situation of incorrectly adapted restorations and identified by us as having deficiencies, we resorted to highlighting a possible bacterial load in the endodontic system.

Consequently, in order to highlight and compare the microbial load, we performed collections from the endodontic system, from the pulp chamber and from the surface of the coronal restoration in order to compare the identified microbial load.

Taking into account all these data, we resorted to our own research, in this sense, samples from the root canal, pulp chamber and occlusal surfaces were collected in conditions of isolation with the dam.

Taking into account all these data, we resorted to our own research, in this sense, samples from the root canal, pulp chamber and occlusal surfaces were collected in conditions of isolation with rubber dam.

Sterile dental kits, sterile paper cones, glass slides for making smears, physiological serum, gram stain kit, cedar oil, optical microscope were needed to carry out this procedure.

Smear examination is performed by scanning several consecutive fields both horizontally and vertically, then the most representative image is photographed.

Fig 5.33 Graphic representation of the types of bacteria found in the root canal.

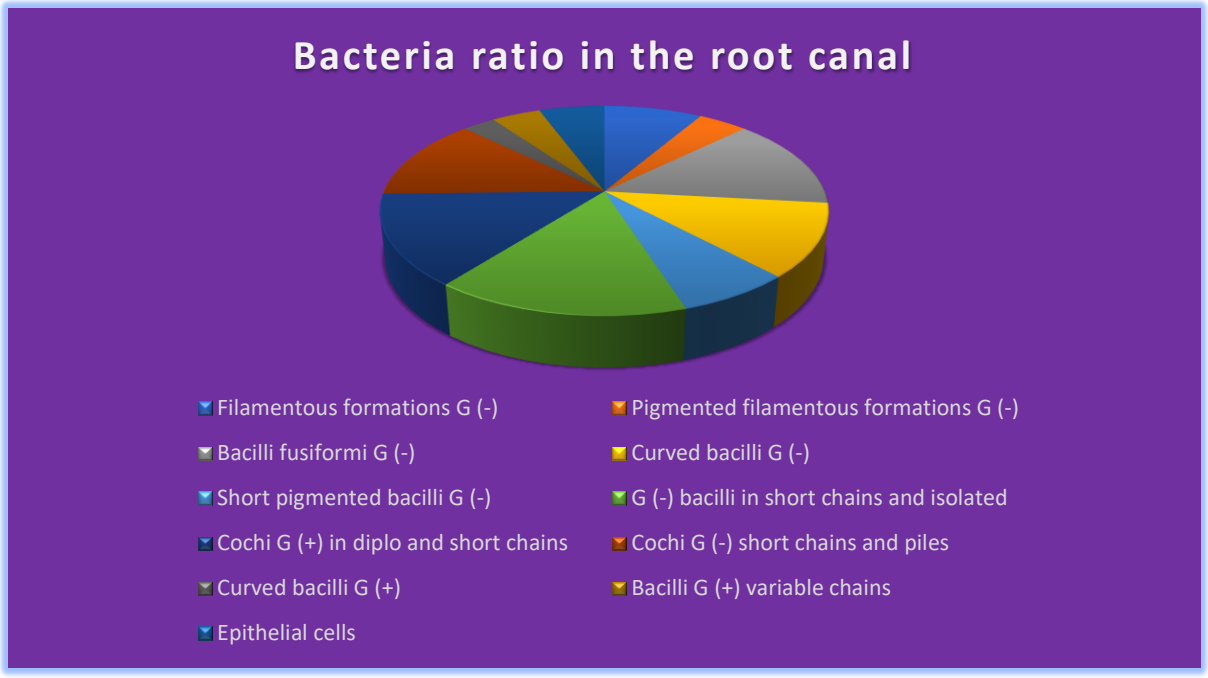
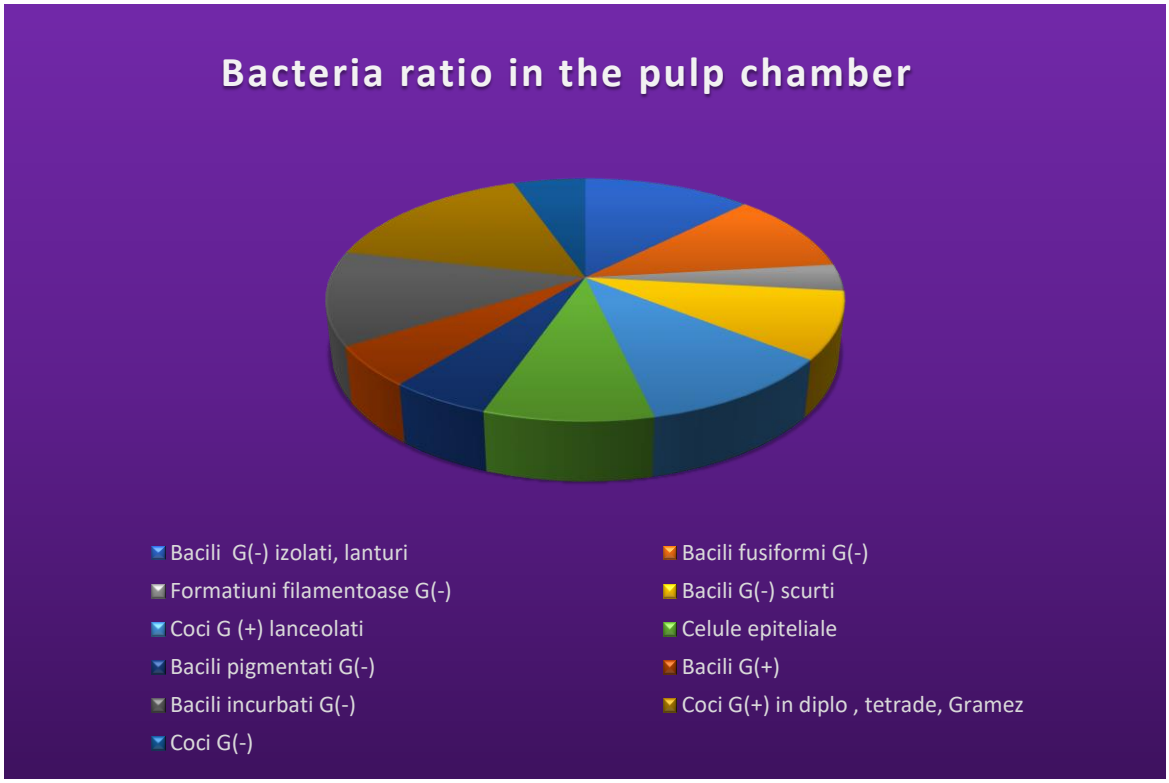


Fig. 5.34 Graphic representation of the types of bacteria found in the pulp chamber



3.4.2 Discussions

Bacterial adhesion and biofilm formation are influenced by the surface characteristics of restorative materials such as: chemical composition, surface finish and polymerization shrinkage.

Regarding the bacteriology of endodontic infection, studies have been conducted in an attempt to define the microbial component of infected root canals. [130, 131] The harvesting areas are divided according to the place of the sample, from the level of the pulp chamber and the root canal.

Several recent studies have used sampled and cultured samples or genetic detection methods, such as the PCR technique, which allow for the detection of more susceptible species and the enumeration of species initially present. Table 5.9 shows the types of bacteria that were most frequently isolated by culture. Although a number of studies that have been published have sought to find relationships between species and specific clinically defined conditions, no acceptable consensus has emerged.

Indeed, examination of Table 5.9 indicates a close relationship of the endodontic microbiota with other ecological niches in the oral cavity and underlines the conclusion that endodontic infections are truly endogenous in nature [132, 133, 134].

Tabel 5.9. Bacteria most commonly found in endodontic infections (taken, adapted and processed from Richard et al, 2006)

Bacterial genus or group	The frequency of isolation in %	Bacterial genus or group	The frequency of isolation in %
Bacteroides	**70**	Capnocitophaga	**17**
Prevotella	**60**	Actinomices	**16**
Lactobacillus	**51**	Leuconostoc	**13**
Streptococi oral	**41**	Porphiromonas	**10**
Clostridium	**36**	Candida	**10**
Fusobacterium	**33**	Veillonella	**9**
Propionibacterium	**29**	Gamella	**8**
Peptostreptococcus	**25**	Staphylociccus	**7**
Corynebacterium		Aerococus	**5**
Bifidobacterium	**21**	Saccaromyces	**3**
Eubacteria	**20**	Enterococcus	**3**

The results of recent studies suggest that E. faecalis can survive in the dentinal tubules due to its ability to bind to collagen and thus act as an epicenter of infection, even after the canal is obturated. This organism is often isolated after failed root canal treatments. Also, E. faecalis is frequently found, especially associated with chronic apical infections, it is a challenge for a successful treatment.

4. Personal research - Experimental, microscopic study on freshly extracted teeth on the quality of composite resin crown restoration

In a simplistic way, we can appreciate the fillings as correct when their edges are placed exactly on the border between the prepared surface (the surfaces exposed by making the cavity, after removing the tissues affected by the carious lesion) and the external enamel surface. The enamel edge of the cavity may or may not be beveled. If beveling has been done, the resulting surface must be covered with composite.

In contrast to this ideal situation (Fig. 6.1a), we can encounter fillings with a moderate surplus or deficit of restorative material during the clinical examination. We consider as follows:

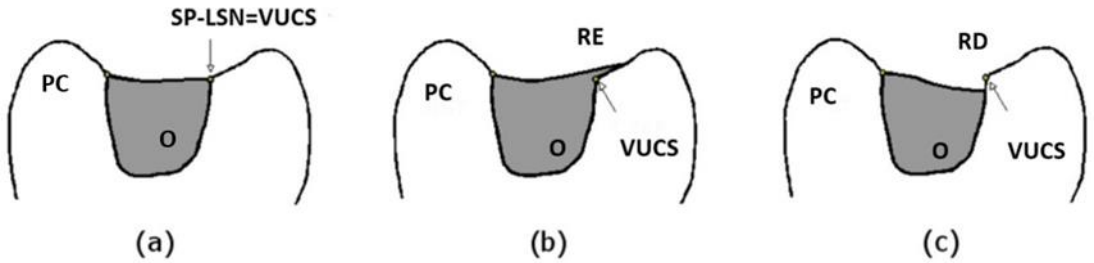


Fig 6. 1 Sketches of fillings made in Class I cavities, on a molar, per section

O= obturation, PC = cavity wall

SP-LSN (prepared surface – unprepared surface limit)

a. Correct restoration (good marginal fit)

b. Moderate Surplus Restoration (RE), (VUCS is exceeded)

c. Restoration with deficit (DF) (restoration did not reach VUCS)

4.1 Results:

- assessment of marginal adaptation inaccuracies through micrometric techniques;

In the image in figure 6.2, a moderate surplus obturation can be seen at the level of the internal cuspid slope at 28. The reduced thickness of the material determined its fracturing.

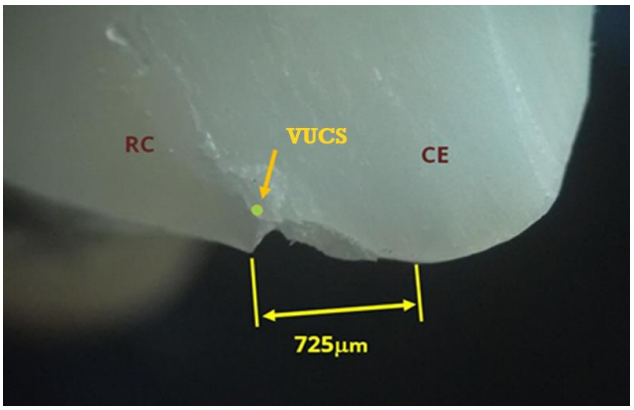


Fig. 6.2 Excess crown restoration at level of 2.8 (RLM, 40x)

The extension of the surplus area, partially detached by fracturing and detaching a fragment of the restorative material, evaluated by micrometry is ≈ 725µm

RC – composite resin/CS – enamel cusp/VUCS tip of the cavosurface angle

In figure 6.3, it can be observed the marginal aspect of an obturation made in a Class II cavity on 2.6. Near the vestibular cusp, the restorative material was not extended to the edge of the preparation, i.e. to the point corresponding to the tip the cavosurface angle. The material-deficient area represents a retentive area that favors the occurrence of marginal secondary caries and marginal infiltration.

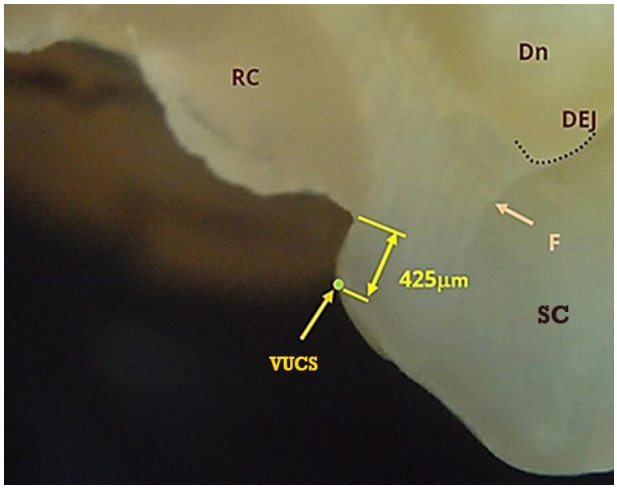


Fig. 6.3 Class II cavity restored with composite at 2.6, near the vestibular cusp. The material deficit evaluated by micrometry is ≈ 425 µm.

In the enamel, there is a crack (F) with an approximately vertical direction that extends to the dentin-enamel junction (DEJ), which can generate a niche for the attachment of germs and the propagation of micro infiltration.

VUCS – apex of the cavosurface angle /RC – composite resin

SC – enamel cusp/Dn – dentin/JDS (DEJ) dentin-enamel junction

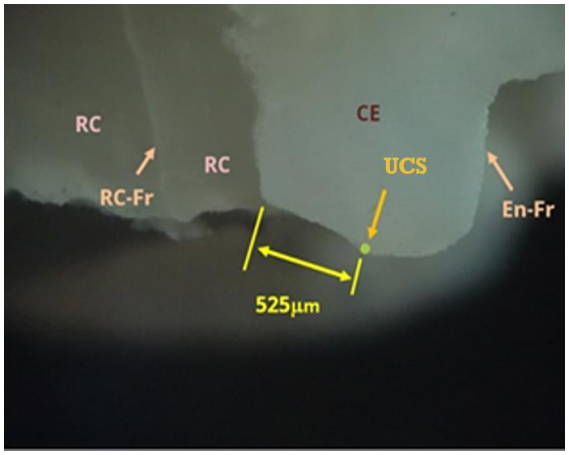


Fig. 6.6 Deficient obturation in the area of the palatal cusp of a maxillary second premolar with a very abraded occlusal surface (40x)

The relief of the tooth at the time of placement of the obturation was already significantly modified by pathological wear. It appears that the cavity preparation was performed with marginal beveling, but the beveling area was not adequately covered with composite resin (RC). Thus, I assessed that the obturation is deficient.

The composite resin filling also has a fracture (RC-Fr) with an approximately mesiodistal direction

The obturation deficit evaluated by micrometry ≈ 525 µm

RC – composite resin

CS – enamel cusp

s- Fr – enamel fracture

RC – Fr – composite filling fracture

VUCS- apex of the cavosurface angle (40x)

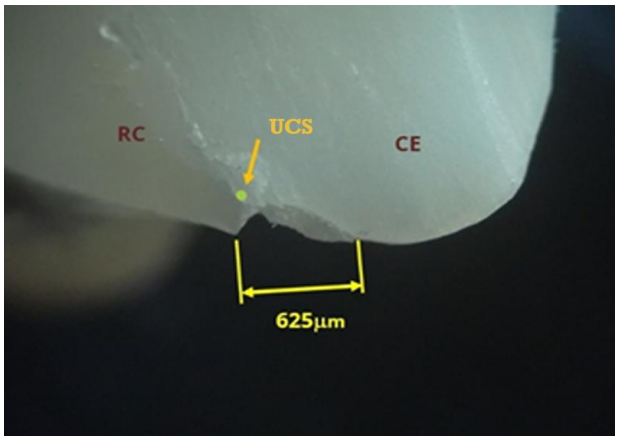


Fig. 6.7. Obturation of a Class I cavity with moderate excess on a maxillary second molar (40x)

Due to the reduced thickness of the overtopping zone, it fractured in the area of the internal cuspid slope.

I classified this case of obturation as one surplus.

Although the excess fractured and detached, the obturation cannot be considered optimal currently, considering the step left immediately coronal to the corresponding point

the cavosurface angle

RC – composite resin

CS– enamel cusp

VUCS – apex of the cavosurface angle

Statistical processing: frequency analysis

As previously shown for the sections obtained from 33 teeth, used for this purpose, values regarding the degree of marginal misfit were obtained by micrometry (in microns, operating with steps of ±25μm). The largest misfit found in all sections for each tooth was taken into account. In the few cases where no adaptation misfit was found, a section without predilection was considered.

5. EXPERIMENTAL STUDY ON THE STRUCTURE AND PROPERTIES OF COMPOSITE MATERIALS, CARRIED OUT THROUGH ATOMIC AND NUCLEAR METHODS OF ANALYSIS

The applications of ion beam analysis (IBA), X-ray spectrometry (XRS), and other atomic and nuclear methods of analysis in the study of dental composites and other dental biomaterials have provided valuable results in recent decades. In this regard, more than twenty years ago a group of researchers first used particle-induced X-ray emission (PIXE) and X-ray fluorescence (XRF) for the analysis of dental composites. Prior to these studies, PIXE applications to dental materials were mostly directed to dental alloys and metal implants [169-173]. Notable exceptions in the field of biomaterials in the nineties include investigations into the solubility and permeability of dental composites [173] and their interactions with dentin, but these studies were carried out by other methods.

5.1 Results and discussions:

IMPROVED RESOLUTION AND SENSITIVITY OF THE IN-AIR PIXE METHOD:

In the following we will present a segment of the inorganic phase because it can be studied by accessible physical methods (XRF, PIXE, m-PIXE, PIGE, m-PIGE, ERDA, RBS).

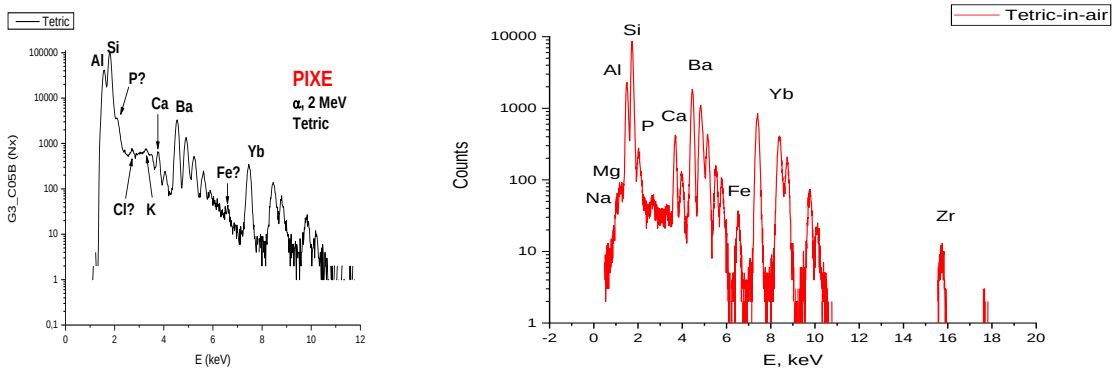


Fig. 7.2. PIXE spectra of Tetric Ceram composite recorded by excitation with 2 MeV a-particles in vacuum (*left*) and with 3 MeV protons in-air (*right*). It is noted that the Proton Spectrum in-air has better resolution.

Fig. 7.2 shows a PIXE spectrum of the Tetric Ceram composite obtained by bombardment with a-particles of 2 MeV in vacuum, which highlights the presence of Al, Si, P, Cl, K, Ca, Ba, Fe and Yb (Fig. 7.2 left).

PIXE and ERDA studies determined that the main mineral elements in Tetric Ceram are Ca (0.29 ± 0.07%), Zr (0.61±0.27%), Ba (4.7±1.6%) and Yb (5.7±1.9%)

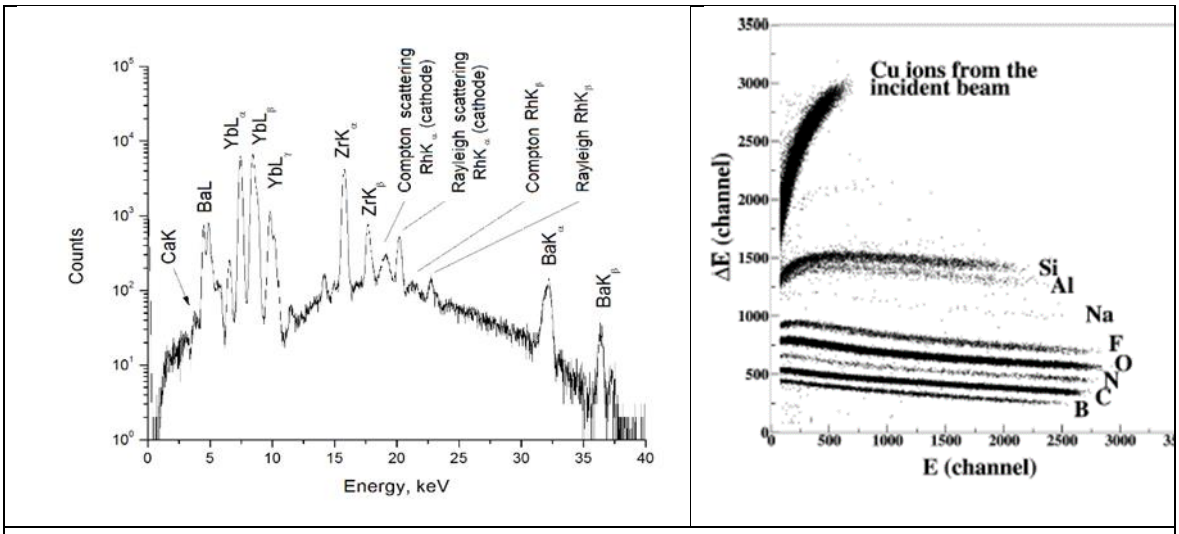


Fig. 7.3 XRF (left) and ERDA (right) spectra of Tetric Ceram composite.

Table 7.2

In-air PIXE qualitative analysis of Ariston, Tetric CLASS and Tetric BULK* dental composites produced by Ivoclar-Vivadent, showing limited differences between BULK and CLASS

Dental composite resin	Na	Mg	Al	Si	P	S	Cl	K	Ca	Sc	Ba	Fe	Yb	Sr	Zr
Ariston	++	+	++	+++	+	++	+	0	+++	0	++	+	+++	0	0
Tetric CLASS	0	+	+++	+++	++	0	+	0	++	0	+++	+	+++	0	++
Tetric BULK	+	+	+++	+++	+++	0	+	+	+	+	+++	+	+++	++	++
* Symbols: 0, not detected; +, trace element; ++, minor element; +++, major element.															

Therefore, the results of the in-air PIXE analysis revealed significant differences between the older Ariston and Tetric CLASS, but only small differences between Tetric BULK and Tetric CLASS. The differences in the composition of the mineral phase between Tetric BULK on the one hand, and Tetric CLASS on the other, are rather quantitative and not qualitative.

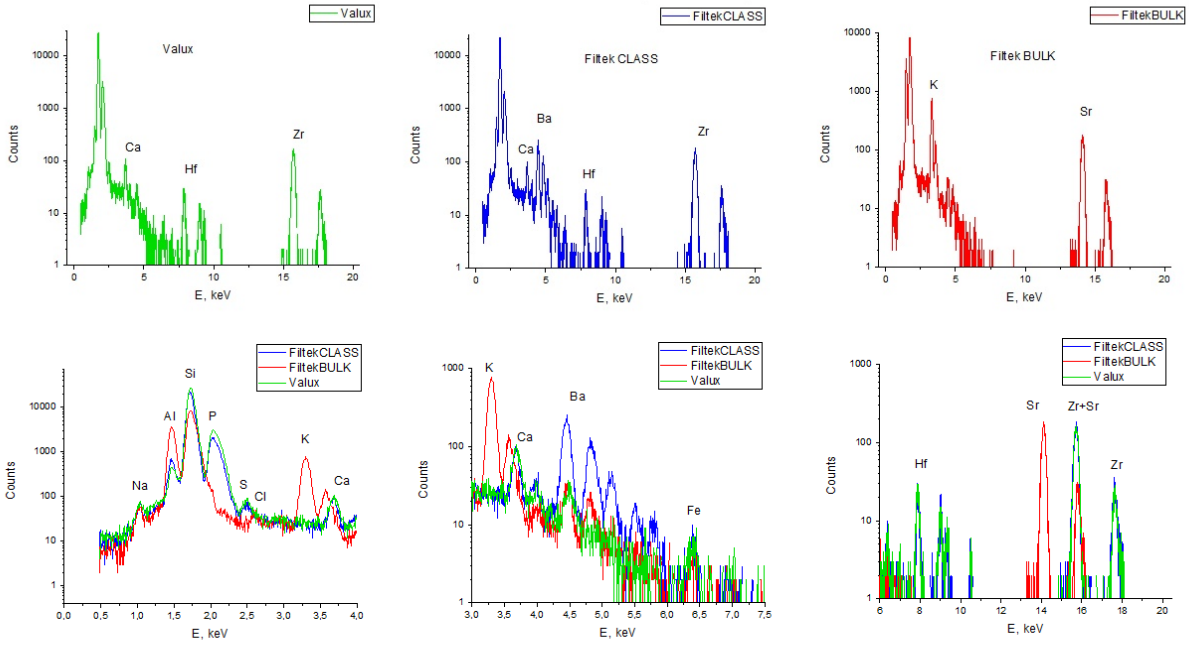


Fig. 7.5. – In-air PIXE spectra of the older Valux Plus and Filtek CLASS and the newer Filtek BULK (3M-ESPE). Separate spectra of each composite (top) and superimposed spectra in the low, intermediate, and high energy domains (bottom). Valux and Tetric CLASS showed relatively close elemental compositions, while Filtek BULK showed significant differences compared to Filtek CLASS and Valux.

6. Conclusions and personal contributions

6.1 Personal contributions:

The studies carried out in the research that I carried out contribute to a multi-disciplinary approach to the studied topic, corroborating notions of restorative odontotherapy with those of endodontics, radiology, microbiology as well as data on the structure and properties of composite materials made by nuclear and atomic physical methods of analysis.

The studies I have carried out in this thesis are original interdisciplinary studies, thanks to the following aspects:

-The moment of applying the composite coronal restoration can be crucial, avoiding the recontamination of the endodontic system, which can have an impact on the success of the endodontic treatment in the long term. (Chapter 5.1, 5.2.)

- The adhesion of biofilms to the surface of various composite restorations, the pulp chamber and the root canals is a certainty. (Chapter 5.4)

- Regarding the microscopic experimental study on freshly extracted teeth regarding the quality of the coronal restoration made of composite resins, the existence of a moderate surplus or a deficit of restorative material can influence the marginal adaptation and can influence the longevity of the restoration. (Chapter 6.3; 6.4)

-According to the studies carried out in the thesis, composite materials from more recent generations seem to contain higher concentrations of light elements and/or lower

- the study on the structure and properties of the composites studied by atomic and nuclear methods of analysis, respectively PIXE and the C/R ratio, is intended to improve the physical-chemical properties and the clinical performance of the composite materials. (Chapter 7.2)

The present study highlights a remarkable potential of the PIXE method in air for the qualitative elemental analysis of dental composites with the present assembly and with its subsequent improvements, with a view to applications in biomaterials research. (Chapter 7.1)

A complementary and relevant synthetic parameter of elemental composition could be the ratio of Compton/Rayleigh scattering intensities in XRF spectra, measured with a portable spectrometer. (Chap7.2)

The use of magnification devices is difficult in the posterior area of the dental arches, especially when the coronal destruction involves the distal surface of the tooth.

In the case of occlusal cavities, beveling the enamel edges is not indicated, in order to avoid the appearance of thin edges of the restorative material, which may fracture under the action of occlusal forces. Beveling at the cavo surface angle increases the restored surface and expands the composite surface that will be subject to a wear phenomenon.

Future studies will consider increasing the number of clinical cases and teeth studied, as well as aiming at the experimental research to expand the research on a larger batch of extracted teeth.

The expansion of the research area related to the addressed field could include the following aspects:

the choice and characterization of other composite materials with improved properties, such as some of their antibacterial effects

- expanding the microbiological studies to decrease the bacterial load in the endodontic system, the microbiological implications in the apical pulp-hair infectious process.

- the results of the experimental study on extracted teeth must be viewed from the perspective of the limits associated with ""in vitro"" studies.

Considering the fact that the incidence of secondary caries, the main cause for the failure of this type of restoration is strictly related to the longevity of the composite resin restoration, in these conditions the susceptibility to microbial infiltration plays an important role. Bacterial adhesion and biofilm formation are dependent on the surface characteristics of the composite restorative material.

In-air PIXE demonstrates high experimental potential for elemental analysis of dental composites, suitable for a wide variety of applications, including in vivo PIXE, dental research, among others. Thus, better spectroscopic resolution, implicitly better analytical sensitivity, simpler and faster operation, and the possibility of measurements on wet samples (equaled only by XRF) represent important advantages.

For quantitative analysis, it is necessary to refine the present PIXE in-air experimental setup with special accessories for measuring the collected charge.

The differences are highlighted by the higher C/R ratio values for the newer composites. Of course, future studies guided by this rule are needed for validation.

Thus, the C/R ratio together with known values of density and radio-opacity would allow to develop calibration relations useful for further estimation of the two macroscopic characteristics of dental composites from the scatter lines of FRX spectra.

Further studies are needed for a better understanding of the relationship between the Compton/Rayleigh scattering ratio and the elemental composition of dental materials for the characterization of dental biomaterials. Thus, the C/R ratio together with known values of

density and radio-opacity would allow to develop useful calibration relationships for further estimation of the two macroscopic characteristics of dental composites.

Based on the results, in-air PIXE analysis of dental composites is suitable for a wide variety of applications in biomaterials research, including:

- the identification of unknown composites, for medico-legal purposes. For purely medical purposes, this can be done both in "in vivo" conditions, with the patient positioned at the accelerator, and "in vitro", on tiny amounts of biomaterial sampled with a milling cutter.

- in vivo analysis of dental composite fillings in patients who show adverse reactions and who claim to replace the fillings with other biomaterials.

- classification, similarities and differences between dental composites in order to optimize restorative therapy.

- correlations between the elemental composition and the properties of dental composites (mechanical, chemical, radio-opacity, marginal fit, stability over time).

- the study of the changes undergone by dental composites during in vivo use, in order to develop new materials with improved qualities, the design of "in vitro" experiments.

- the study of the interactions between dental composites and hard dental tissues

Conclusions:

The general conclusions are summarized in highlighting some important aspects in relation to the clinical-radiological, statistical, microbiological and experimental studies carried out in the thesis as follows:

The composite coronal restoration should be performed in the same treatment session as the root canal obturation, in order to prevent re-infection of the endodontic system. Corono-apical infiltration in conditions of defective restoration is the major cause of recurrent endodontic pathology

The implementation of composite obturation materials is indisputably the most used in the field of restorative therapy, the benefits that this bio-material offers when it is applied correctly are recognized. The purpose of my study is to highlight the possible deficiencies observed in teeth with composite restorations, with pre-existing endodontic treatment. In order to improve the results obtained after classic procedures, to demonstrate the fact that the coronal restoration can influence the long-term prognosis of the endodontic treatment, I carried out within the clinical research a study that addresses the problem of marginal adaptation, from the point of view of the correct expansion of the fillings as well as the

identification of other deficiencies of this type of restorations.

Thus, from the group of teeth studied from a clinical-radiological point of view that presented different deficiencies of the coronal restoration, microscopic investigations were carried out with the dental microscope on a number of teeth filmed with the digital camera of the dental microscope, the image being transferred to a PC. This allowed measurements at the level of restoration with deficit or surplus, highlighting that the higher frequency was observed in the case of fillings with a moderate surplus of composite material.

In this context, the clinical examination and the radiological evaluation must be carried out rigorously to establish the integrity and correctness of the composite resin coronary restoration, the quality of the canal obturation, as well as the periapical pathology, if it exists. Also, the need to institute the resumption of treatment in certain situations is appreciated endodontic treatment completed with root canal obturation followed in the same session by coronary restoration. Considering the fact that the incidence of secondary caries, the main cause for the failure of this type of restoration is strictly related to the longevity of the composite resin restoration, in these conditions the susceptibility to microbial infiltration plays an important role.

Bacterial adhesion and biofilm formation are dependent on the surface characteristics of the composite restorative material. Thus, the success rate of endodontic treatment correlates with permanent coronal restorations, to exemplify the consequences of crown-apical infiltration of deficient composite restorations, we analyzed series of radiographs from selected patients following the quality of the root canal obturation, corroborating these data with the integrity of the restoration coronary, instituting the required treatment, namely the resumption of endodontic treatment and application in the same session of the appropriate composite restoration. In order to demonstrate the fact that in certain situations, infiltration in the corono-apical sense is a reality, in the case of the clinical situations that we have analyzed, we highlighted this through a microbiological examination, collecting samples from the surface of the coronary restoration materials from the room level pulp as well as from the root canal. As I mentioned, the accumulation of biofilm on the surface of the restoration and in the case of the creation of a niche, at the interface of the restoration material, the prepared cavity can lead to the failure of the restoration procedure. This film formed at the interface or under the composite restoration can lead to the degradation of the restoration material but also to the infiltration of the canal fillings which can lead to its compromise, in other words the longevity of the restoration depends on the susceptibility to bacterial colonization.

According to what is described in the studies presented by laboratory stereomicroscopy, but also in vivo with endodontic microscopes, it is possible to appreciate, in detail, the quality of restoration of dental morphology and to evaluate, in a more limited way, the quality of marginal adaptations; through laboratory microscopy, in transmitted light, in reflected light, it is possible to appreciate more precisely how correct the marginal adaptation is, and on the radiological image, the quality of the sealing of the endodontic space. By PIXE in air we observed significant differences between Ariston composite and Tetric Ceram, but relatively small differences between the more recent Tetric BULK and Tetric CERAM. (VIVADENT IVOCLAR).

The new Filtek BULK product has a significantly different mineral composition compared to Filtek and Valux, (3M-ESPE) while these last two biomaterials have relatively close elemental compositions. At low energies, all the composites selected for this study showed high levels of Al and Si, probably in grains of an aluminosilicate glass or their respective oxides. In the range of intermediate and high energies, Ariston, Tetric and Tetric BULK (Ivoclar-Vivadent) composites showed high levels of Yb, while Valux, Filtek and Filtek BULK (3M-ESPE) showed high concentrations of Sr and/or Zr .

PIXE in the air with SDD detector brings together significant advantages. On the one hand, the method allowed the detection of elements lighter than Ca (Na, Mg, Al, Si, P, K), some trace elements (Ti, Fe, Cu, Zn) and some heavier major elements (Ba , Yb, Sr, Zr).