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ORIGINAL STUDY

BRACKET BONDING TO ENAMEL AND DENTIN - ESEM STUDIES -

Delia Ioana Ciocan[1], Florin Miculescu[2], Lucian Toma Ciocan[3], Dragos Stanciu[1]

[1]Orthodontics and Dento-Facial Orthopedics Department, Faculty of Dental Medicine, "Carol Davila" University of Medicine and Pharmacy, Bucharest, Romania
[2]Material Science and Engineering Faculty, Polytechnic University, Bucharest, Romania
[3]Prosthodontics Technology and Dental Materials Department, Faculty of Dental Medicine, "Carol Davila" University of Medicine and Pharmacy, Bucharest, Romania

delia_ciocanaru@yahoo.com

ABSTRACT

The researches proposed to investigate, using scanning eletrono-microscopy methods, the structure and surface morphology of the coronal hard dental tissues, the enamel and the dentin, although the interfaces between these and adhesive system and orthodontic brackets. There have been taken into the study 30 human extracted wisdom teeth, which have been maintained into cloramine T 10% solution for 48 hours and then transferred in a physiologic saline solution. To 15 from them there have been attached using specific adhesive systems orthodontic brackets. The surface of the other 15 was analyzed also direct, without other preparations, and also after demineralization for 30 and 60 seconds by 37% orthophosphoric acid. All of the samples have been subjected to imagistic and spectral analysis using a electron-microscope Phillips-30-XL. Maintaining a long time of etchant on enamel surface (about 60 seconds) in addition to producing an irreversibly superficial destruction compromising its structure affects also adhesion by increasing the residual dentine debris. Bracket and complementarity between the surface morphology and the surface of the enamel makes that the strength of the link assembly to depend predominantly to cohesive fracture resistance of the adhesive composite material.

KEYWORDS*: brackets, adhesion, enamel, dentin, etching time, ESEM.*

Introduction

Modern dentistry has been significantly marked by the evolution of the adhesion materials. Due to this in nowadays orthodontics it is possible to use very thin fixation elements as brackets are and still succeed in obtaining a favorable clinical outcome. Despite this clinical success that usually is obtained, the reasons that sometimes the adhesion is hard to be obtained or maintained are barely known. For this reason we developed the current study. For being able to make objective evaluations [1] of the imagistic analysis we made an ESEM (Environment Scanning Electrono Microscopy) study on hard dental tissues implied in adhesive fixation of fixed orthodontics appliances.

The researches made in this article were possible due to the interdiciplinarity good collaboration between Orthodontics and Dento-Facial Orthopedics Department, Faculty of Dental Medicine,

"Carol Davila" University of Medicine and Pharmacy, Bucharest and Electrono-microcopy Laboratory from Biomaterials Department of Materials Science and Engineering Faculty, Polytechnic University, Bucharest, Romania.

Aim of the study

The researches proposed to investigate, using scanning eletrono-microscopy methods, the structure and surface morphology of the corronal hard dental tissues, the enamel and the dentin, although the interfaces between these and adhesive system and orthodontic brackets.

Materials And Methods

There have been taken into the study 30 human extracted wisdom teeth, which have been maintained into cloramine T 10% solution for 48 hours and then transferred in a physiologic saline solution [2]. To 15 from them there have been attached using specific adhesive systems [3] orthodontic brackets. The surface of the other 15 was analyzed also direct, without other preparations, and also after demineralization for 30 and 60 seconds by orthophosphoric acid 37% [4], see figure 1.

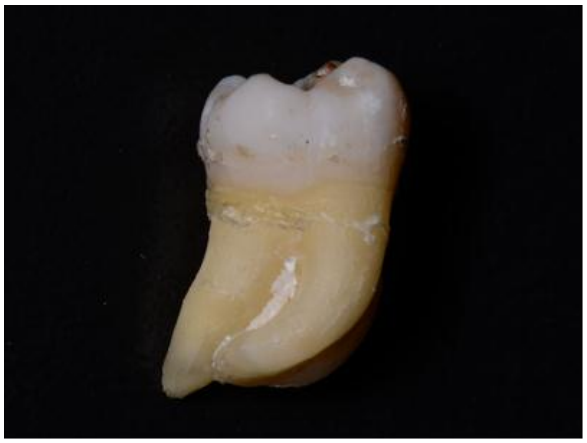


Figure 1. *Extracted wisdom tooth utilized as biologic testing sample*

The teeth to which have been attached orthodontic elements have been prepared for specific electron-microscopy investigation by being embeded in epoxidic resin afterwards being sectioned upon longitudinal and transversal direction. In this way was possible to make analysis at the interface level, see figure 2.

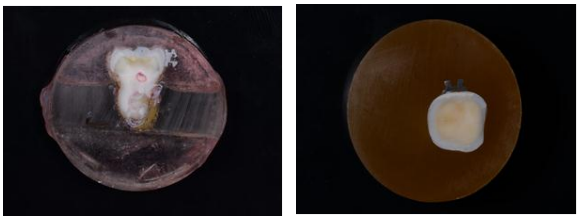


Figure 2. *Longitudinal and transversal sections through the bracket-adhesive-tooth samples after being embeded in epoxidic resin for being subjected to ESEM investigations*

All of the samples have been subjected to imagistic and spectral analysis using a electron-microscope Phillips-30-XL, this device having the advantage that can achieve images with a resolution as high as 2000x without implying other preparations or altering the investigated samples, see figure 3.

Figure 3. *Phillips XL-30-ESEM electrono-microscope device, used for sample analysis.*

Results and Discussion

In figure 4 it can be noticed the macrostructural difference between the surface of the normal coronal enamel (left part of the image) and the orthophosphoric 37% acid demineralized one (right part of the same image). Even with a 25x

magnitude it can be identified a rough enamel surface prepared for adhesive attachment of an orthodontic fixation element.

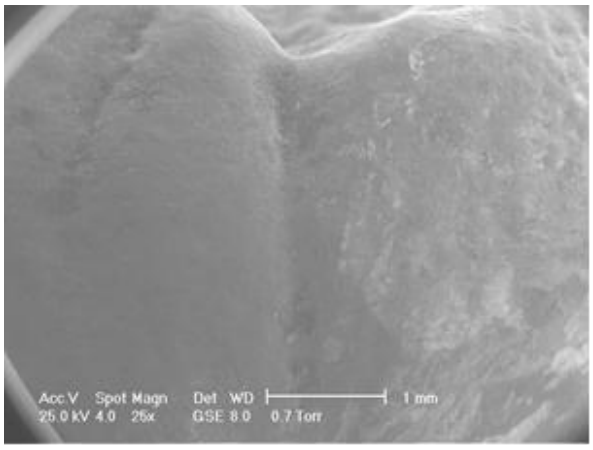
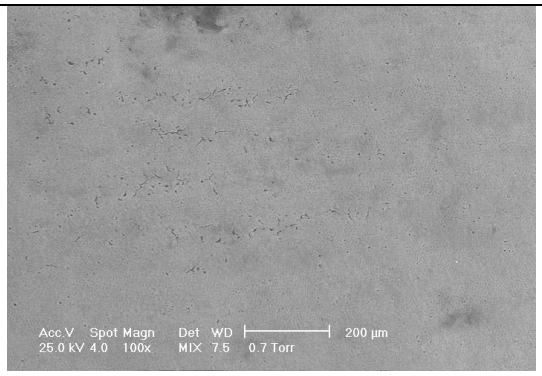
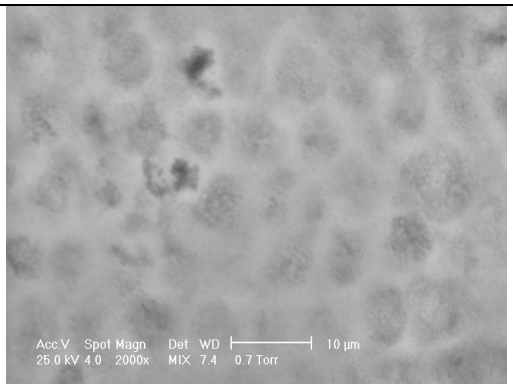


Figure 4. *ESEM morphologic image of coronal enamel, natural-left part and orthophosphoric 37% acid demineralized one-right part*

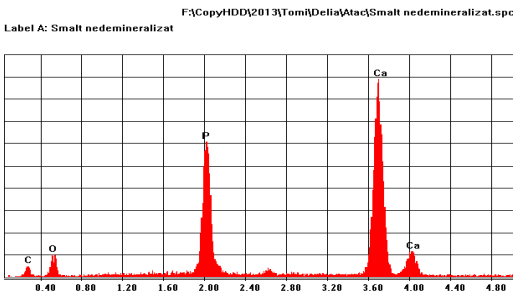
At the analysis of the intact enamel surface, figure 5, it could be noticed also to 100x and 2000x that this surface is not patterned, the enamel prisms having a variable diameter between 8 and 10 microns, the interprismatic substance following a protein surface cover which justified the high percentage of carbon on the surface 22,13% weight respectively 36,5% atomic. Very interesting was the identifying on the enamel surface of different types of defect, figure 5.b, with variable diameter that can reach up to 9 microns. These mineralizing defects are located at the border of the enamel prisms and are areas rich in organic substance [5, 6]. Although the defects have been identified only on the cusps surface, where usually is not a common place for a dental decay to be appear, these defects can explain etiopathogenic mechanism for destruction of hard dental tissues by a dental decay process.



a) surface morphology 100x



b) surface morphology 2000x



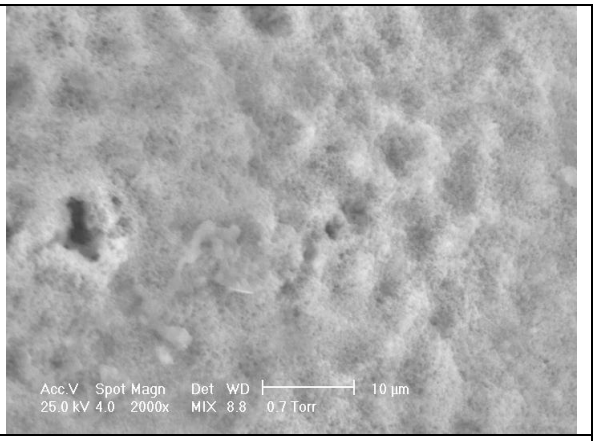
c) elemental chemical distribution on the surface

Element	Wt %	At %
C	22.13	6.95
O	28.69	35.96
P	1 .92	10.96
Ca	32.25	1 .13
T tal	100	100

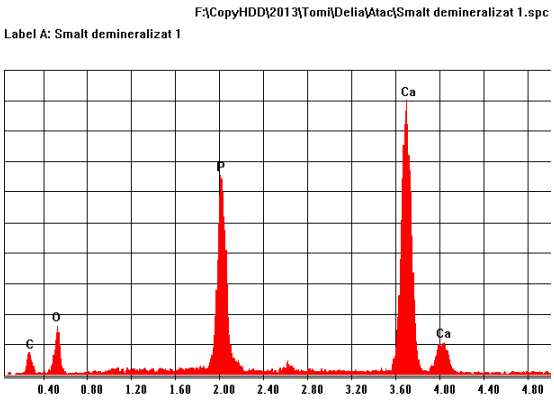
d) values for elemental chemical distribution on the surface

Figure 5. *Morphological (qualitative) and EDAX (quantitative) ESEM analysis of natural enamel surface*

a) surface morphology 100x



b) surface morphology 2000x



c) elemental chemical distribution on the surface

Element	Wt	At %
	25.38	40.03
O	31. 2	37.68
P	14.75	9.02
Ca	2 .06	13.27
Total	100	100

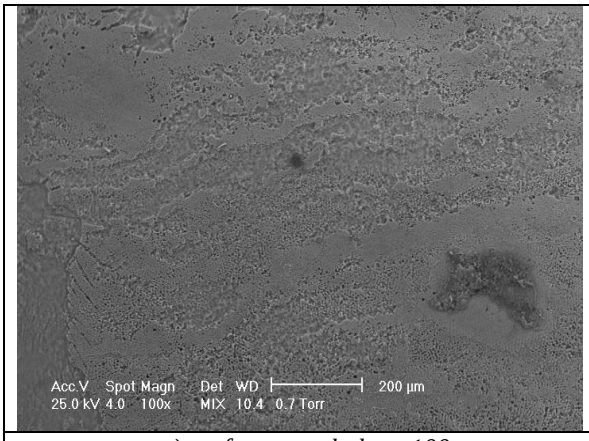
d) values for elemental chemical distribution on the surface

Figure 6. *Morphological (qualitative) and EDAX (quantitative) ESEM analysis of 37% phosphoric acid etched enamel surface for 30 seconds*

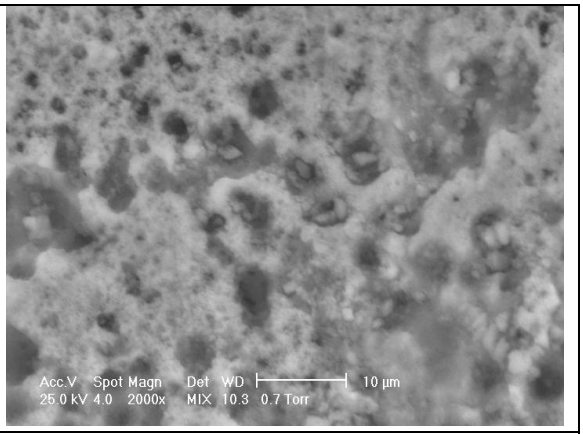
Demineralized enamel with 37% phosphoric acid for 30 seconds, see figure 6, shows, as expected, a rough surface with surface roughness equal in size to the diameter of the enamel prisms. This suggest that preferential acid attack was made on the outskirts of prisms, figure 6.a) and b). At 2000x magnification, Figure 4.6.b), it was observed that after etching defects normally present on the surface enamel increase their diameter compared to figure 4.5.b). Qualitative by etching is obtaining a higher adhesion surface area in brackets used in orthodontics. Quantitatively, the percentage of organic matter after demineralization of the enamel surface increases to 25.38% and 40.03% atomic weight, favoring chemical bonds with the latest polymer adhesives used in determining orthodontic anchorage.

Demineralized enamel with 37% phosphoric acid for 60 seconds, figure 7 also shows a rougher surface than normal enamel with irregular surface roughness more than demineralized a shorter time [7]. Here and there are large areas of demineralization observed in surface structures could no longer be identified normal and prism-specific organization of SMAT interprismatică substance normally figures 7.a) and b). Type EDAX results of quantitative analyzes performed on demineralized enamel surface for 60 seconds shows a very low level of carbon (15.62% or 28.44% At Wt) and lower values than normal enamel (22.13 At that 36.5% Wt%), which indicates that the prolongation of demineralization

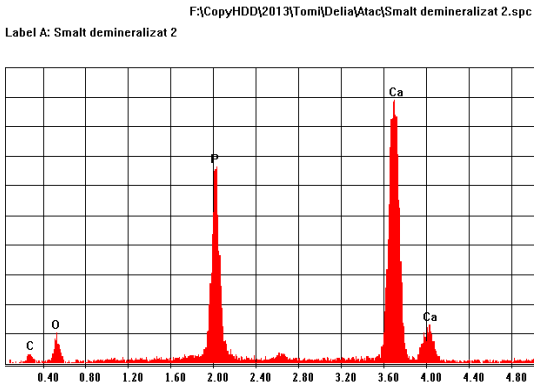
time can compromise the adhesion of adhesives. The adhesion is compromised and large deposits of debris remaining on the surface of demineralized dentin, figure 7.b). This observation is confirmed by various studies in the literature [8].



a) surface morphology 100x



b) surface morphology 2000x



c) elemental chemical distribution on the surface

Element	Wt %	At %
C	15.62	28.44
O	27.5	37.59
P	18.2	12.9
Ca	38.62	21.07
Total	100	100

d) values for elemental chemical distribution on the surface

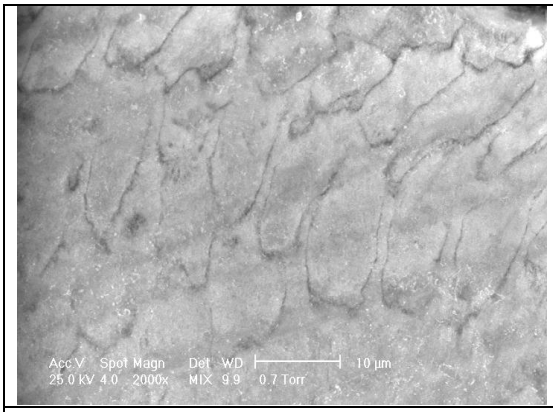
Figure 7. *Morphological (qualitative) and EDAX (quantitative) ESEM analysis of 37% phosphoric acid etched enamel surface for 60 seconds*

The analysis section of demineralized enamel, figure 8, shows the elliptical enamel prisms, section direction never being perpendicular to the longitudinal trajectory prisms, their diameter ranging up to 12-15 microns.

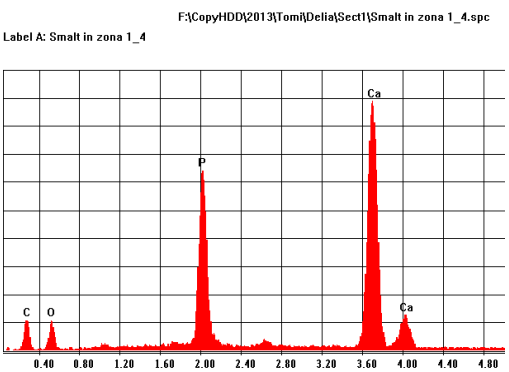
The demineralized enamel interprismatic spaces are wider and narrows progressively to a maximum depth of demineralization of 30 microns, figure 8.a). From the chemical point of view, in section, the ratio and composition of organic and inorganic structures is slightly affected by surface etching procedure.

Because sometimes there is a need to conduct a clinical adhesion to dentin, we image and spectral investigated this layer also. In figure 9 a), b) and d) can be seen the channeling structure of coronal dentin, dentinal tubules being equal, evenly, about 2-3 microns in diameter and tough in between channels substance about 10 microns.

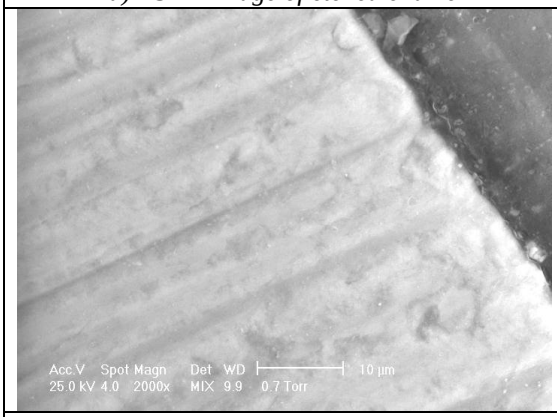
In the same area the proportion of mineral substance is only 1-2% richer than the enamel dentine, figure 9 c) and e), but the channeling structure and increased percentage of organic matter justify the deficiency to achieve and maintain a favorable adhesion of the modern adhesive systems [9, 10, 11,12].



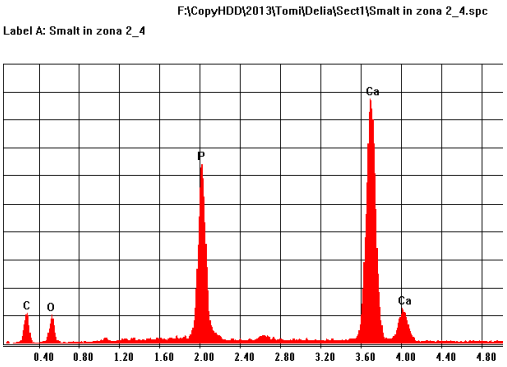
a) ESEM image of etched enamel



b) EDAX analysis of etched enamel



c) ESEM image of natural enamel

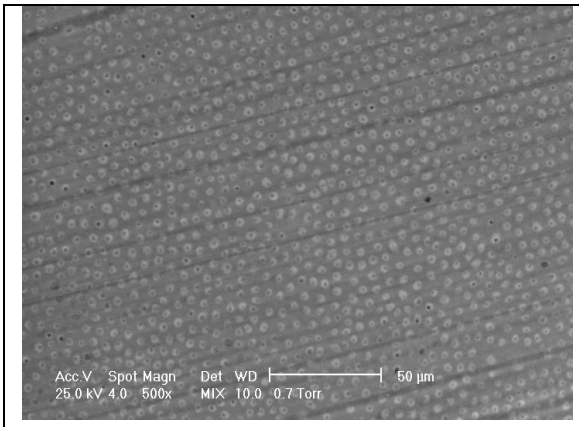


d) EDAX analysis of natural enamel

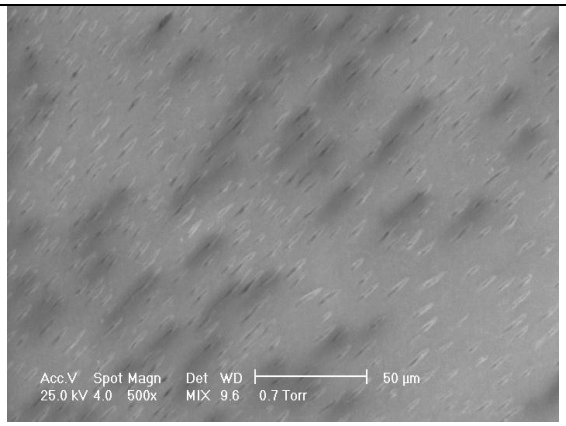
Element	Wt %	At %
C	36.29	54.12
O	23.24	26.02
P	13.4	7.7
Ca	27. 6	12.
Total	100	100

e) chemical composition distribution deep in the enamel

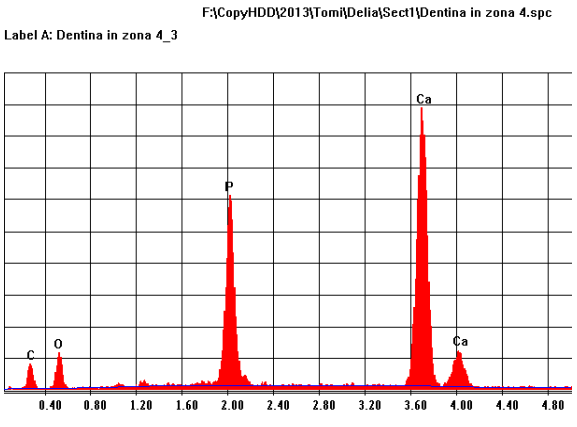
Figure 8. *Enamel cross section ESEM analysis*



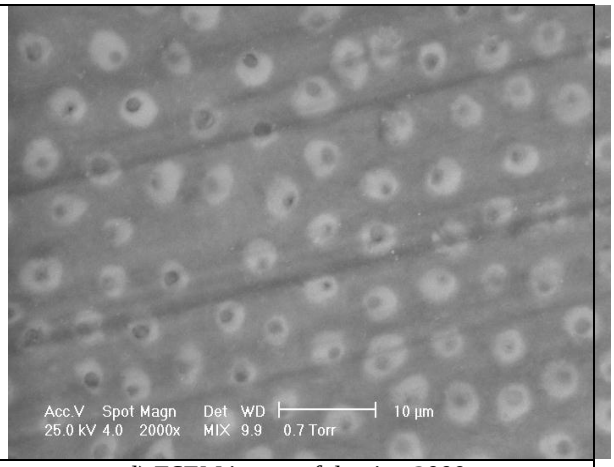
a) ESEM image of cross sectioned dentin x500



b) ESEM image of longitudinal sectioned dentin x500



c) EDAX analysis of dentin

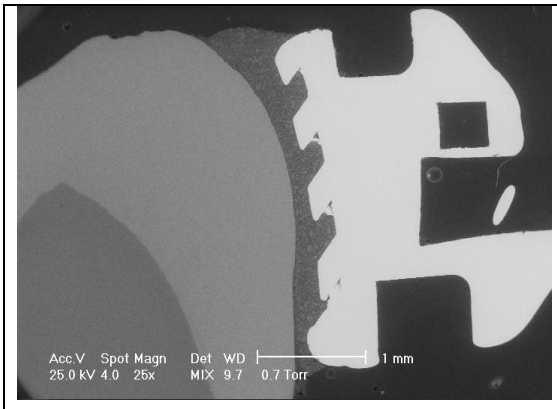


d) ESEM image of dentin x2000

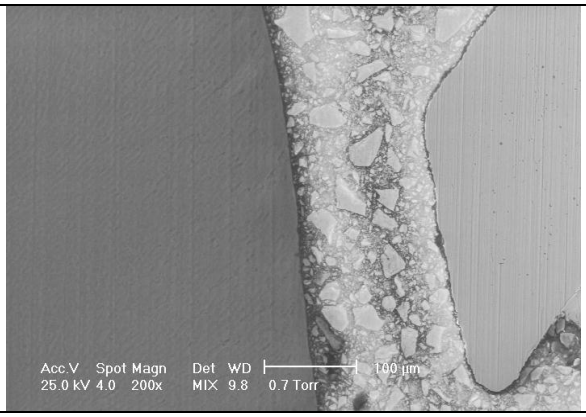
Element	*Wt %*	*At %*
C	*29. 3*	*45.38*
O	*28.3*	*32.*
P	*13.74*	*8.27*
Ca	*2 .73*	*13 37*
Total	*100*	*100*

e) chemical composition distribution deep in the dentin

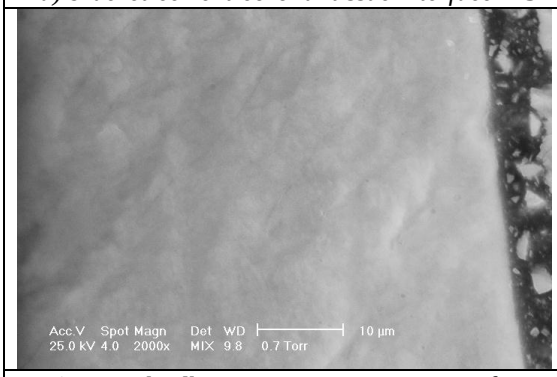
Figure 9. *Dentin cross section ESEM analysis*



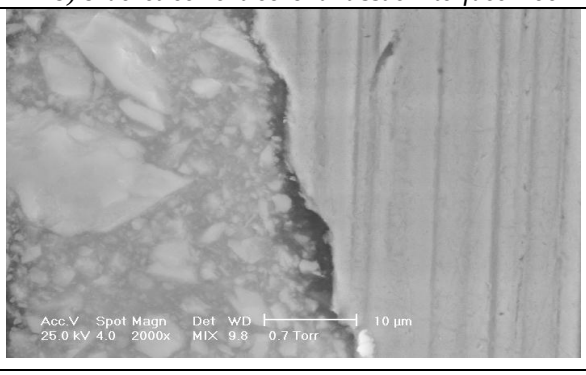
a) bracket-cement-coronal tissue interface x25



b) bracket-cement-coronal tissue interfacex200



c) enamel-adhesive-composite resin interface



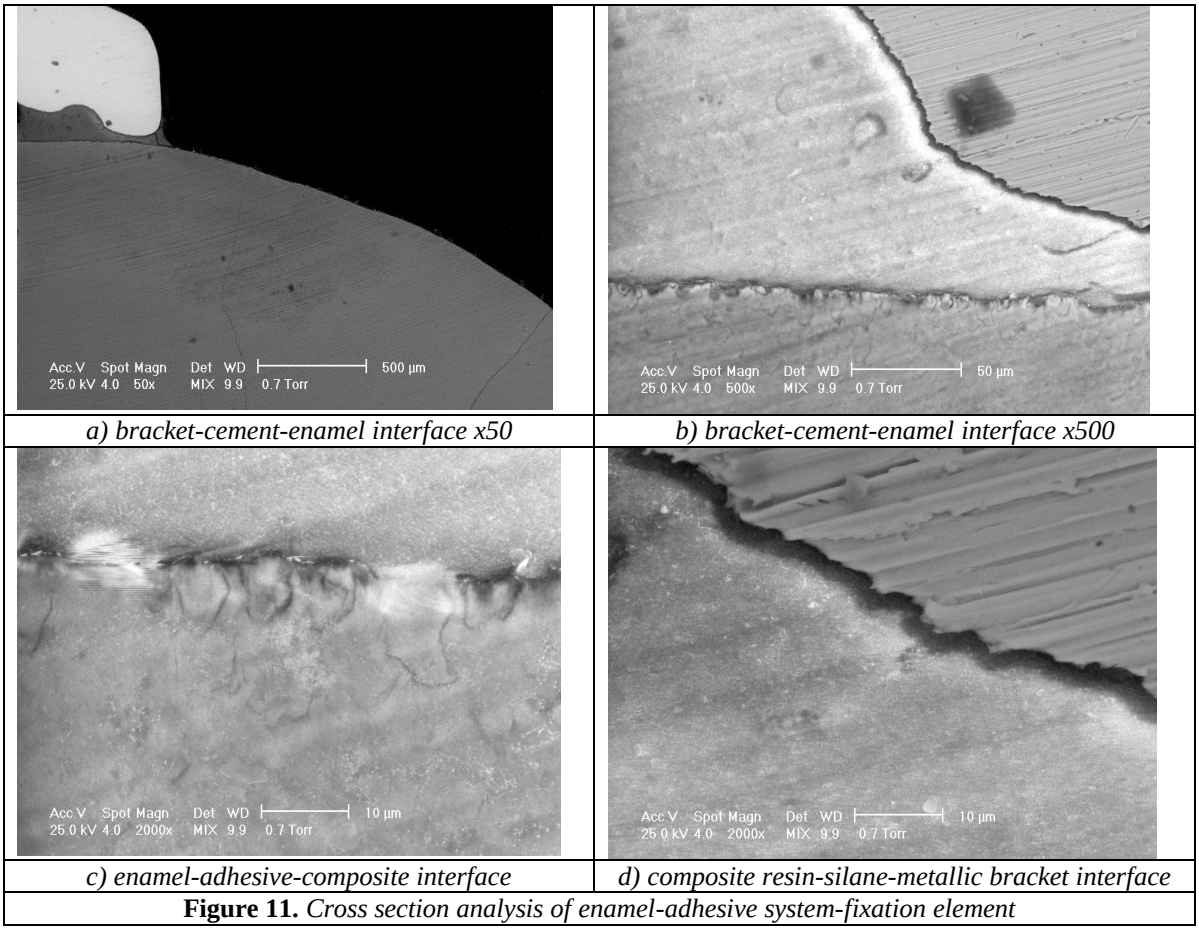
d) composite resin-silane-metallic bracket interface

Figure 10. *Cross section analysis of enamel-adhesive system-fixation element*

In figures 10 and 11 are presented the results of imaging obtained after investigating the two samples (both metal bracket-adhesive-tooth samples) prepared in a different manner. In the

second test we used additional 37% phosphoric acid conditioning of enamel surface before applying adhesive agent and the metal fixing bracket.

In figure 10 is observed the adhesion of the adhesive system accuracy to the tooth surface as well as to the metal. Not etched enamel surface makes enamel-resin adhesive interface linear, figure 10.c), prisms appeared smooth compared to image of demineralized enamel interface, figure 11.c) where the surface area over which adhesion is achieved is much higher [13, 14]. The volume of the composite resin in the interface depends on the correlation between the morphology of the surface of the enamel and of the bracket [15]. If the premises are made of high adhesion, bond strength values are dependent on the material resistance to cohesive fracture. The adhesion to the material from which the bracket it is made, as shown in shown in Figure 10.d) and 11.d) is proportional to the contact area dependent on the method of manufacturing, the surface roughness and the presence or absence of its compliance with silane substances that encourage a chemical bond to the polymer matrix of the composite cement.



a) bracket-cement-enamel interface x50 | *b) bracket-cement-enamel interface x500*

c) enamel-adhesive-composite interface | *d) composite resin-silane-metallic bracket interface*

Figure 11. *Cross section analysis of enamel-adhesive system-fixation element*

Conclusions

After ESEM studying on dental hard tissues and adhesion were observed on several important issues.

The normal enamel surface is irregular, enamel prisms having a variable diameter from 8 to

10 microns, the in between prismatic substance continues with a protein coating surface. At the surface the enamel is damaged. These defects are located at the periphery of demineralization of enamel

prisms and are areas rich in organic matter, thus may jeopardize achieving proper adhesion to orthodontic brackets.

Demineralized enamel surface with 37% phosphoric acid for 30 seconds is favorable both quantitative as well as qualitative in terms of the concentration of the constituent elements to increase the surface area for attachment of orthodontic brackets.

Maintaining a long time of etchant on enamel surface (about 60 seconds) in addition to producing a irreversibly superficial destruction compromising its structure affects also adhesion by increasing the residual dentine debris. The depths of etching pits in enamel are on a depth of 30 microns, seen by increasing the interprismatic spaces.

The structure of dentin is improper to obtain a proper adhesion to orthodontic elements.

Bracket and complementarity between the surface morphology and the surface of the enamel makes that the strength of the link assembly to depend predominantly to cohesive fracture resistance of the adhesive composite material. The surface roughness of the bracket and the use of an organosilane coating can significantly increase the relative area and its connection to the fixing agent.

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