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SEM and EDS comparative studies about the homogeneity of some cements used for luting of posts

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ABSTRACT

Objectives. The aim of the study was to evaluate comparatively by scanning electron microscope (SEM) and energy dispersive spectrophotometry (EDS) the physical and chemical homogeneity of some luting materials used for cementation of three types of posts.

Materials and method. We selected 12 irretrievable monoradicular teeth extracted in our dental offices. After extraction, all teeth were prepared for cementation with 4 different cements (NANOCORE DUAL, CEMBEST, PANAVIA SA and CEMENT-ONE) of three types of posts: prefabricated titanium post, FibreKleer 4x fiberglass post (Pentron) and post cast from NiCrMo alloy. Afterwards, the samples were embedded in resin and prepared metallographically for examination with the Phenom ProX scanning electron microscope equipped with an energy dispersive spectrophotometer with X Rays.

Results. SEM studies provide valuable ultrastructural information regarding the degree of filling with luting materials. The four studied cements do not present an inhomogeneous composition in the EDS analysis, not having significantly increased values between the 2 analyzed points for each of the 12 samples taken in the study. Conclusions. Among the four luting materials studied comparatively, PANAVIA SA seems to best fill the existing spaces between posts and the limits of dental preparations, being closely followed by CEMENT-One and CEMBEST. In last place is NANOCORE DUAL, which presents discontinuities in the mass of material and at the border with the preparations.

Keywords: SEM, EDS, fiberglass post, titanium post, casting post, luting materials

INTRODUCTION

Selecting the optimal way to restore endodontically treated teeth is still a controversial topic, which is intensely debated in the specialized literature. Unlike vital teeth, endodontically treated teeth have substantially different mechanical properties [1]. These changes are due to the quantitative loss of hard substance both through the expansion of the carious processes themselves and through the preparation of the access cavities necessary for endodontic therapy [2,3]. Endodontically treated teeth present a higher risk of fracture compared to vital teeth [4] and therefore preserving as much of the hard dental structure as possible is essential to establish and maintain a balance between biological, mechanical, adhesive, functional and aesthetic parameters [5]. In recent years, the restorative approach to endodontically treated teeth has changed. The development of dental adhesive techniques has expanded the therapeutic options of clinicians. The

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posts casted from dental alloys or prefabricated posts have increasingly been replaced by fiberglass posts due to the superior esthetic result. In parallel, new materials for luting these devices in the root canals appeared, with superior properties. In vitro evaluation of the quality of these abutment cementation materials can be done by scanning electron microscopy (SEM) and energy dispersive spectrophotometry (EDS). It is important to note that interferences between spectral series of different chemical elements having the same energy may occur in EDS examinations. This fact can influence the results obtained, and they must be interpreted accordingly. The aim of the study was to evaluate comparatively by scanning electron microscope (SEM) and energy dispersive spectrophotometry (EDS) the physical and chemical homogeneity of some luting materials used for cementation of three types of posts (post cast from NiCrMo alloy, prefabricated titanium post and fiberglass post), used to restore dental abutments.

MATERIALS AND METHODS

The study was carried out with the consent of the Ethics Committee of the Faculty of Dental Medicine – "Titu Maiorescu" University of Bucharest (No. 2/15.01.2018), respecting human rights and without harming the health of patients or the environment. We selected 12 irretrievable monoradicular teeth extracted in our dental offices. After extraction, all teeth were prepared for cementation with 4 different cements (NANOCORE DUAL, CEMBEST, PANAVIA SA and CEMENT-ONE) of three types of posts, respectively: prefabricated titanium post, FibreKleer 4x fiberglass post (Pentron) and post casted from NiCrMo alloy. The coding of the 12 samples is shown in table 1.

TABLE 1. The coding of samples

Device / Luting agent	Prefabricated titanium post	Fiberglass Post	Cast post
NANOCORE DUAL	1.1	1.2	1.3
CEMBEST	2.1	2.2	2.3
PANAVIA SA	3.1	3.2	3.3
CEMENT_ONE	4.1	4.2	4.3

Protocol for preparation and obturation of root canals

We created the access cavity with a long neck diamond burr for the turbine (FG-314, D 801L G, Stoddard). We widened the pulp chamber with the Endo Access Z152 inactive tip bur. We identified the root canals with the author of a DG 16 endodontic probe. We performed the permeabilization of the teeth, along the entire length of the canals, with Kerr-file NiTi ISO 10 manual needles. We determined the working length by inserting a Kerr file ISO 15 needle into the root canal until the tip of the needle was visible at the level of the apical foramen. We measured the distance between the incisal landmark and the apex, from which I subtracted 1 mm. We performed the mechanical root canal treatment using rotary instruments, the crown-down technique. For the preparation of the root canals, we used the X-Smart endodontic motor (Dentsply Sirona), in the reciprocating movement, using a single instrument Reciprocal Blue System (VDW GmbH), needle R25. During the preparation, we performed endodontic lavages with sodium hypochlorite (Cloraxid 5.25% Cerkamed) and ethylenediaminetetraacetic acid (EDTA 17%, Meta-Biomed). After drying the root canals with Reciproc Blue 25 paper cones, we obturated them using specially designed, calibrated Reciproc Blue R25 gutta-percha cones and a resin-based sealer (Adseal, Meta-Biomed), respecting the determined working length. We sectioned and removed the excess with a gutta-percha cutter. The teeth were coronally obturated with a provisional cement (MD-Temp, Meta-Biomed) and were kept in saline at room temperature for 6 days.

Protocol for the preparation of radicular lodges

To prepare the root canals, in order to receive a post, we used a pivot calibration bur (FibreKleer, Petron), with 1.375 diameter, purple color code, mounted on the contra-angle piece, at low speed. The canals were prepared on 2/3 of the length of the root, remaining obturated at least 4 mm up to the level of the apex, having a cylindrical-conical shape. Depending on the type of post received, the teeth are prepared for cementation or for impression.

Protocol for obtaining cast posts

The imprinting of the root canals was done in 2 steps with condensation silicones (Zetaplus, Zermack). Cast posts were made in the dental laboratory. The protocol for cementing the posts was carried out specifically, following the indications of the manufacturers of the cementing materials.

Afterwards, the samples were sent to the BIOMAT Research Center of the POLITEHNICA University of Bucharest, where they were embedded in resin and prepared metallographically (successive sanding with abrasive paper with a grain size of 300-1000 μm and then sanding with a 1 μm diamond powder suspension) (Figure 1) for examination with the Phenom ProX scanning electron microscope equipped with EDS. EDS analysis was done in 2 points to see possible inhomogeneities of the luting cements. The interface between the dental preparation and the cemented post was also examined by SEM to assess the degree of filling of the space with luting material.

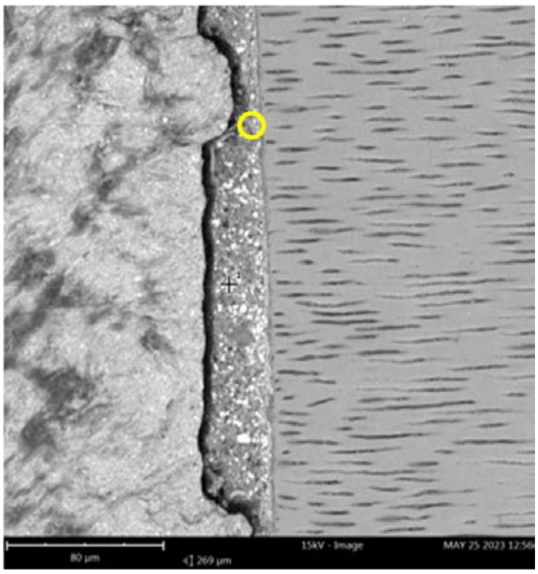
FIGURE 1. Metallographically prepared samples

RESULTS

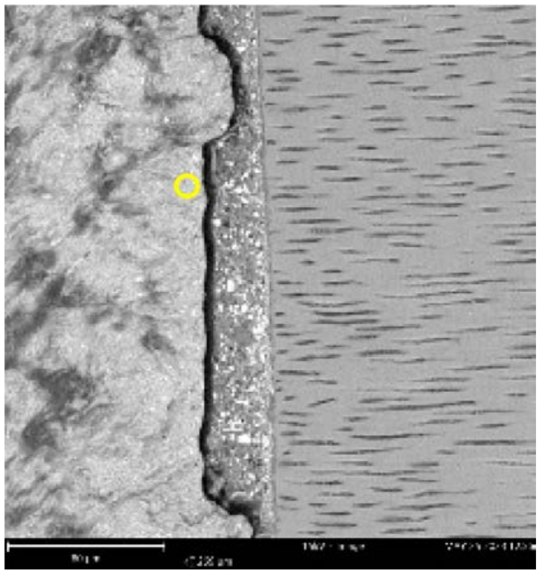
Figure 2 show the SEM examinations and EDS analyzes performed on NANOCORE DUAL used for cementation of the posts. In the case of sample 1.1, the luting materials adhered intimately to the dentin, but at the interface with the titanium post there is a uniform space, of approximately 4 µm, between the post and the cementing material. This filling defect is probably due to screw insertion of the post. In sample 1.2 we noticed there are larger filling defects, there are void in the cementing material, with a maximum length of 100 µm and a width of 40 µm. In the case of sample 1.3, the cementing material adhered intimately to the cast post, but at the interface with the dental preparation we detect a space that varies between 15-70 µm. Figure 3 present the SEM examinations and EDS analyzes performed on CEMBEST used for cementation of the posts. In the case of sample 2.1, there are gaps of material both at the interface with the tooth (between 1 and 20 µm) and with the titanium post (between 10-20 µm), we observe fracture lines in the material (25 µm) and voids with a diameter of approximately 20 µm. In sample 2.2 there is a relatively uniform space at the interface with the fiberglass post (approximately 10 µm), fracture lines (5 µm) and voids in the cement mass (with diameters reaching up to 60 µm). In the case of sample 2.3, the cement adheres intimately to the tooth, but there is a gap of approximately 4 µm at the interface with the cast post. Fracture lines are visible in the luting material and in the tooth. In figure 4 it is visible the SEM examinations and EDS analyzes performed on PANAVIA SA used for cementation of the posts. Examining sample 3.1, we found that the cement adhered intimately to the titanium post and the tooth, but in its mass there are fracture lines of maximum 10 µm. In sample 3.2 it is visible that the cement adheres to the tooth and to the fiberglass pivot, but in some places there are spaces of 10-20 µm. The material is arranged evenly, without gaps or fracture lines. In the case of sample 3.3, the cement adhered intimately to the tooth and the cast post, with submicronic spaces in some places. Figure 5 show the SEM examinations and EDS analyzes performed on CEMENT-ONE used for cementation of the posts. In our study, on sample 4.1 the luting cement does not present voids, it adheres intimately to the titanium post, but there are wider spaces (15-20 µm) at the interface with the tooth. On sample 4.2, the cement adheres intimately to the fiberglass post, but presents spaces at the interface with the tooth (20 µm). On sample 4.3, the cement adheres intimately to the tooth and presents microspaces (approximately 5 µm) at the interface with the cast post. In this case, the presence of small material voids is highlighted.

DISCUSSIONS

Although in vitro studies have reported an increased physical and mechanical strength of the posts, there are also clinical studies that have reported therapeutic failure [6] after the use of various posts on endodontically treated teeth. Some of the causes of therapeutic failure are represented by the incorrect setting of the material for luting the posts or the insufficient filling of the existing microspaces between the root preparation and the cemented device. For dual cements, there is still a lack of consensus regarding the photopolymerization efficiency of the luting materials. Hashimoto et al. [7] state that the maximum set cannot be achieved by the self-polymerization mechanism alone, be-

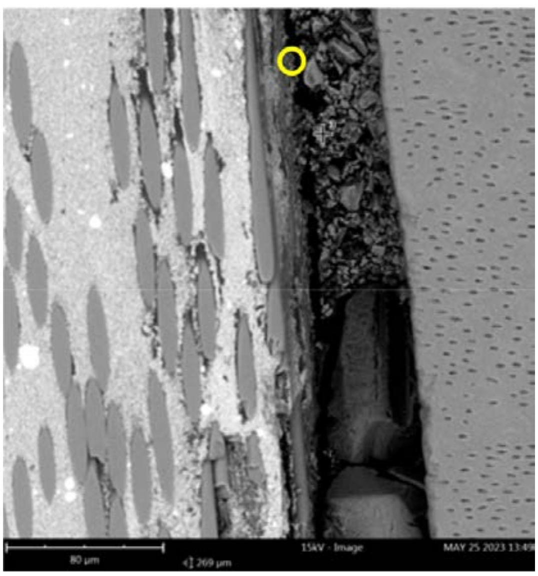


Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	54.70	36.70
40	Zr	Zirconium	5.96	30.38
8	O	Oxygen	22.02	19.67
5	B	Boron	15.52	9.37
20	Ca	Calcium	0.94	2.09
39	Y	Yttrium	0.18	0.90
11	Na	Sodium	0.63	0.81
14	Si	Silicon	0.05	0.08

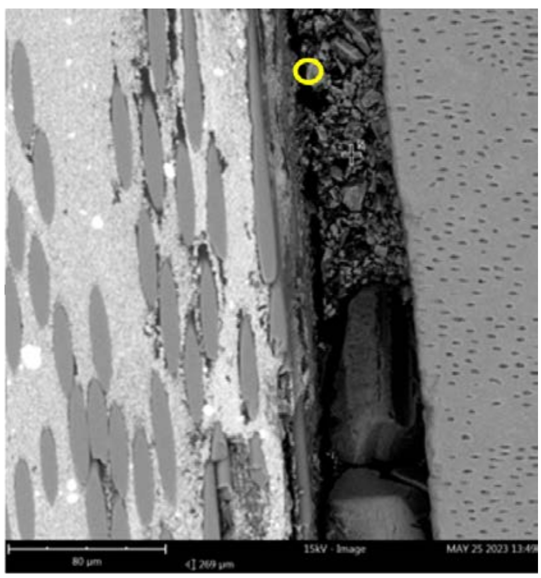


Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	53.30	34.20
8	O	Oxygen	35.22	30.11
40	Zr	Zirconium	3.82	18.63
20	Ca	Calcium	6.76	14.47
30	Zn	Zinc	0.58	2.03
22	Ti	Titanium	0.12	0.31
12	Mg	Magnesium	0.20	0.26

a) sample 1.1

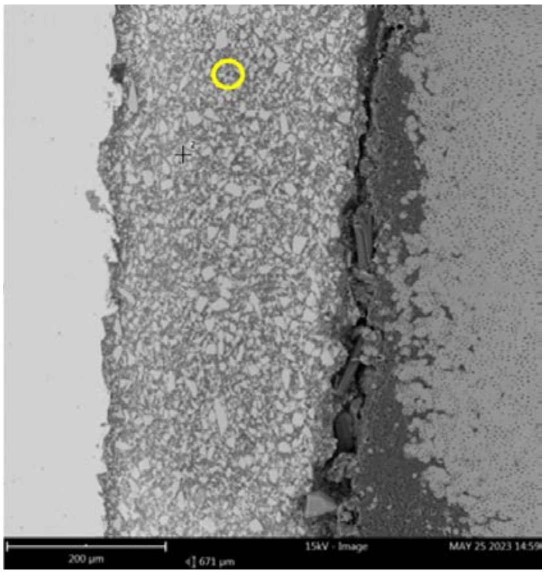


Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	47.09	44.06
6	C	Carbon	41.02	28.82
20	Ca	Calcium	3.61	8.47
14	Si	Silicon	3.04	5.00
56	Ba	Barium	0.55	4.38
15	P	Phosphorus	2.07	3.76
13	Al	Aluminium	1.76	2.78
40	Zr	Zirconium	0.39	2.08
12	Mg	Magnesium	0.46	0.66
39	Y	Yttrium	0.00	0.00

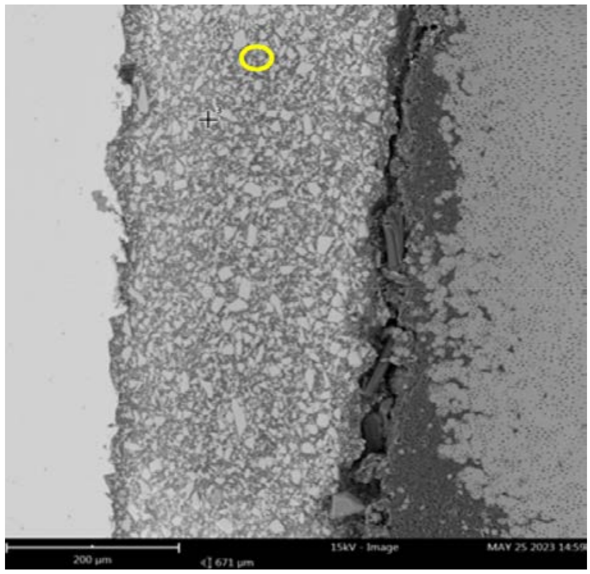


Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	45.25	43.07
6	C	Carbon	42.14	30.12
20	Ca	Calcium	3.37	8.05
35	Br	Bromine	1.24	5.90
15	P	Phosphorus	2.55	4.69
14	Si	Silicon	2.67	4.47
9	F	Fluorine	1.86	2.10
12	Mg	Magnesium	0.72	1.04
22	Ti	Titanium	0.20	0.56

b) sample 1.2



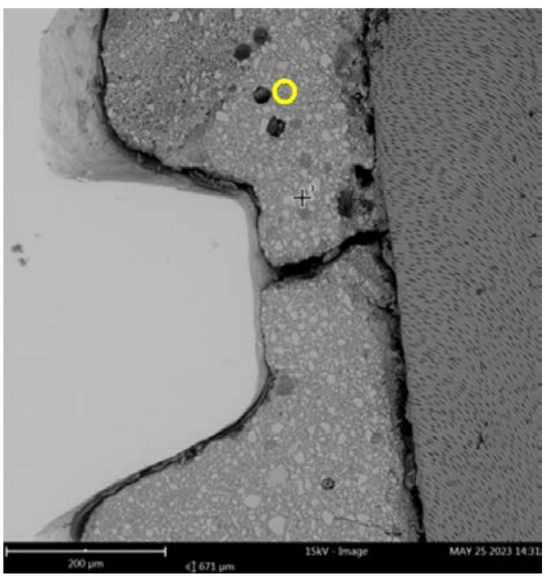
Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	55.80	51.60
6	C	Carbon	32.60	22.63
14	Si	Silicon	7.68	12.46
58	Ce	Cerium	0.91	7.38
13	Al	Aluminium	1.98	3.09
22	Ti	Titanium	1.03	2.84



Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	67.91	57.00
14	Si	Silicon	11.76	17.33
6	C	Carbon	14.78	9.31
58	Ce	Cerium	1.16	8.56
13	Al	Aluminium	2.94	4.16
22	Ti	Titanium	1.45	3.65

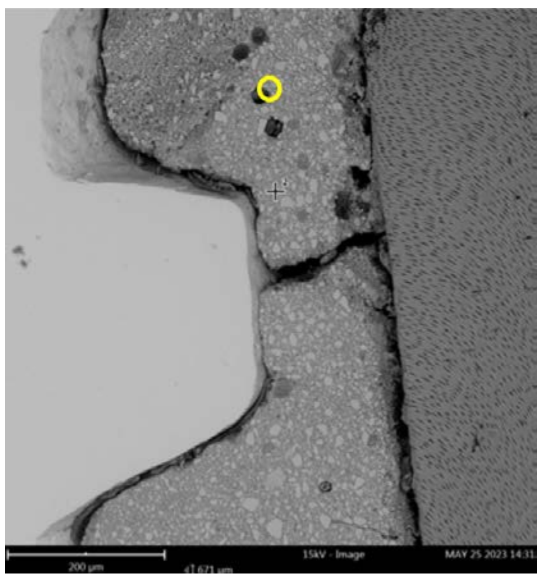
c) sample 1.3

FIGURE 2. SEM examinations and EDX analyzes performed on NANOCORE DUAL used for cementation of the posts

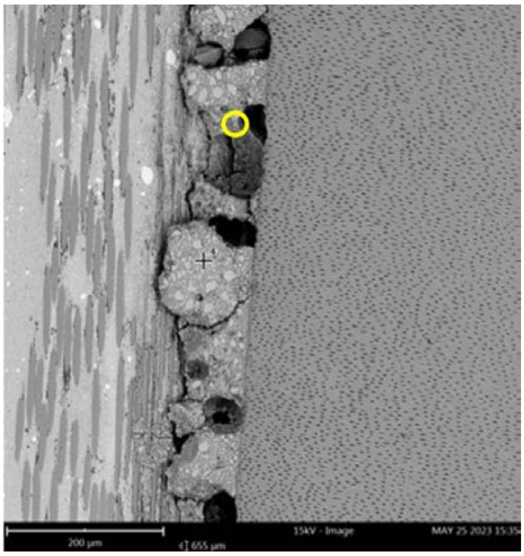


Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	50.83	32.61
57	La	Lanthanum	3.15	17.54
13	Al	Aluminium	11.10	12.01
9	F	Fluorine	15.52	11.82
14	Si	Silicon	8.39	9.45
20	Ca	Calcium	5.50	8.84
22	Ti	Titanium	2.08	4.00
15	P	Phosphorus	1.71	2.13
11	Na	Sodium	1.15	1.06
12	Mg	Magnesium	0.57	0.55

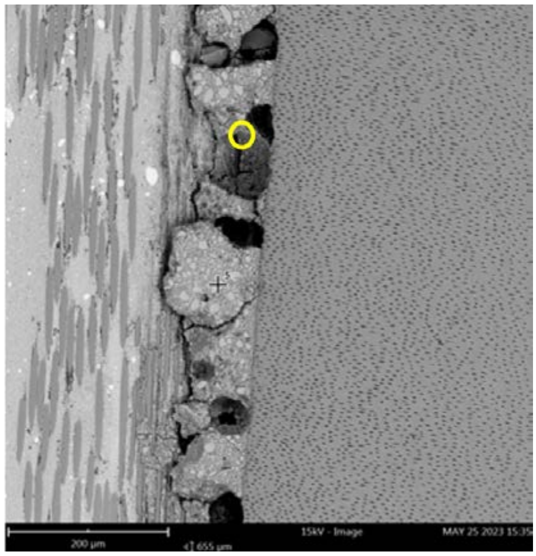
a) sample 2.1



Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	46.16	35.04
57	La	Lanthanum	2.06	13.57
6	C	Carbon	20.26	11.54
9	F	Fluorine	12.19	10.99
13	Al	Aluminium	6.56	8.40
14	Si	Silicon	5.11	6.81
20	Ca	Calcium	3.36	6.38
22	Ti	Titanium	1.84	4.17
15	P	Phosphorus	1.00	1.47
11	Na	Sodium	1.03	1.13
12	Mg	Magnesium	0.43	0.50

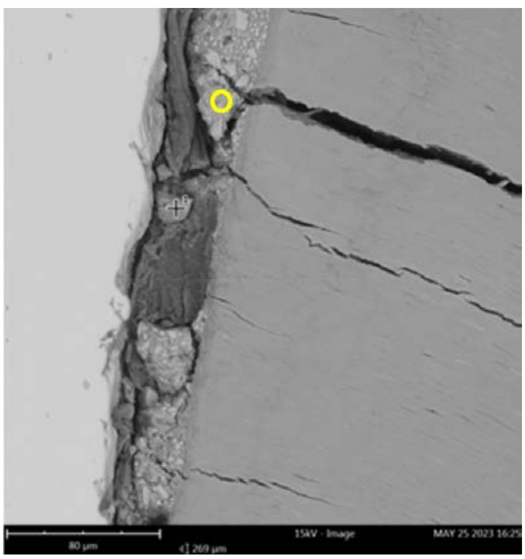


Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	31.88	22.95
6	C	Carbon	39.40	21.29
35	Br	Bromine	4.89	17.56
57	La	Lanthanum	2.08	12.99
13	Al	Aluminium	6.33	7.68
9	F	Fluorine	6.96	5.95
20	Ca	Calcium	3.25	5.85
11	Na	Sodium	4.15	4.29
15	P	Phosphorus	0.86	1.20
12	Mg	Magnesium	0.21	0.23



Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	33.90	25.72
6	C	Carbon	30.58	17.41
57	La	Lanthanum	2.46	16.24
13	Al	Aluminium	7.64	9.77
9	F	Fluorine	8.50	7.66
14	Si	Silicon	5.73	7.63
11	Na	Sodium	6.58	7.18
20	Ca	Calcium	3.76	7.15
15	P	Phosphorus	0.84	1.24

b) sample 2.2

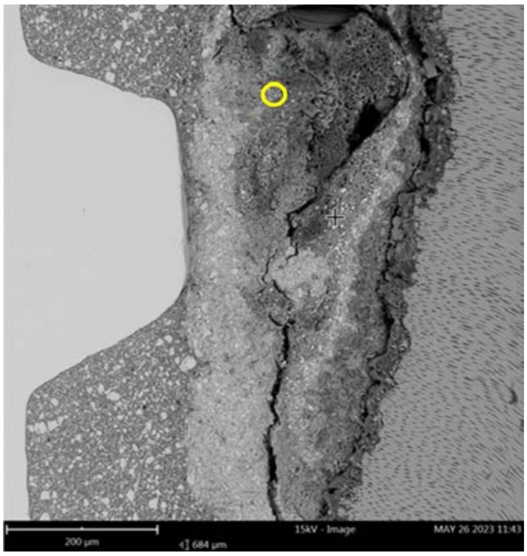


Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
14	Si	Silicon	28.83	41.77
8	O	Oxygen	33.35	27.53
6	C	Carbon	33.87	20.99
28	Ni	Nickel	1.62	4.91
24	Cr	Chromium	0.71	1.91
20	Ca	Calcium	0.81	1.68
15	P	Phosphorus	0.45	0.73
13	Al	Aluminium	0.35	0.48

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	59.46	46.59
8	O	Oxygen	34.49	36.00
28	Ni	Nickel	1.80	6.89
20	Ca	Calcium	1.50	3.92
24	Cr	Chromium	0.85	2.90
15	P	Phosphorus	0.94	1.90
14	Si	Silicon	0.66	1.20
16	S	Sulfur	0.19	0.40
13	Al	Aluminium	0.11	0.20

c) sample 2.3

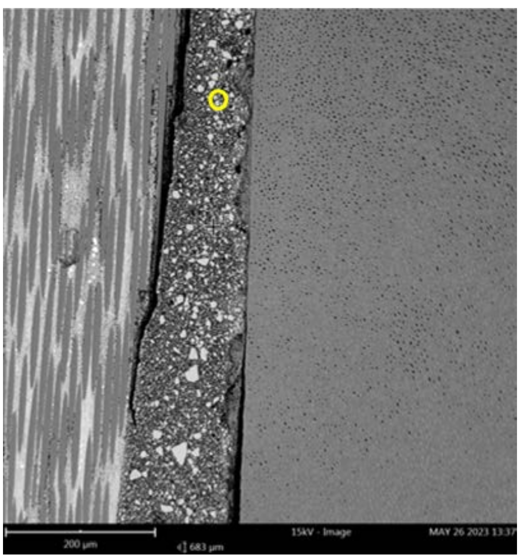
FIGURE 3. SEM examinations and EDX analyzes performed on CEMBEST used for cementation of the posts



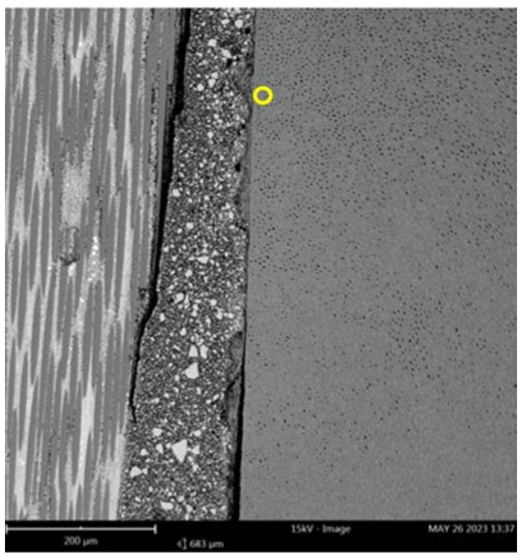
Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	71.86	46.14
40	Zr	Zirconium	5.80	28.30
8	O	Oxygen	18.23	15.59
20	Ca	Calcium	2.90	6.21
39	Y	Yttrium	0.35	1.67
22	Ti	Titanium	0.42	1.09
35	Br	Bromine	0.13	0.55

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	47.74	35.72
8	O	Oxygen	28.07	27.98
7	N	Nitrogen	16.89	14.74
20	Ca	Calcium	3.76	9.38
40	Zr	Zirconium	1.19	6.75
22	Ti	Titanium	0.93	2.78
15	P	Phosphorus	1.02	1.97
14	Si	Silicon	0.40	0.69

a) sample 3.1

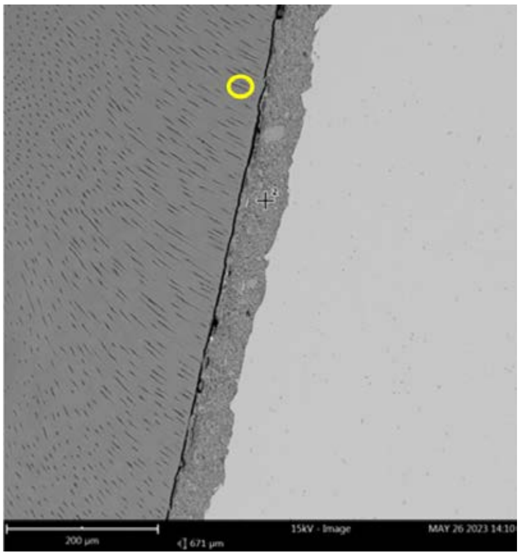


Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	38.51	31.18
6	C	Carbon	44.03	26.76
56	Ba	Barium	2.80	19.48
14	Si	Silicon	8.79	12.48
13	Al	Aluminium	3.27	4.46
38	Sr	Strontium	0.52	2.32
15	P	Phosphorus	1.04	1.63
20	Ca	Calcium	0.56	1.14
11	Na	Sodium	0.47	0.55

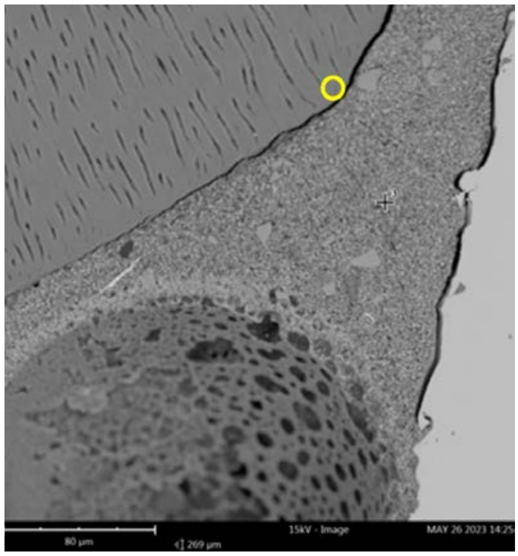


Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	38.01	31.08
6	C	Carbon	44.53	26.86
56	Ba	Barium	2.80	19.48
14	Si	Silicon	8.79	12.48
13	Al	Aluminium	3.27	4.46
38	Sr	Strontium	0.52	2.32
15	P	Phosphorus	1.04	1.63
20	Ca	Calcium	0.56	1.14
11	Na	Sodium	0.47	0.55

b) sample 3.2



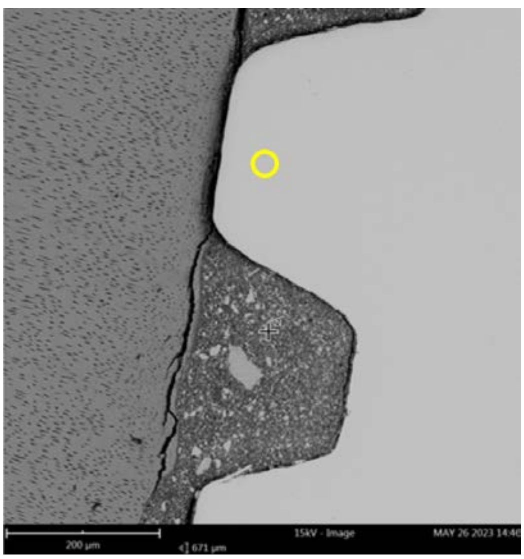
Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	41.99	34.40
6	C	Carbon	40.98	25.20
56	Ba	Barium	2.63	18.50
14	Si	Silicon	8.83	12.70
13	Al	Aluminium	2.28	3.15
28	Ni	Nickel	0.65	1.94
11	Na	Sodium	1.56	1.84
24	Cr	Chromium	0.36	0.96
20	Ca	Calcium	0.39	0.79
15	P	Phosphorus	0.33	0.52



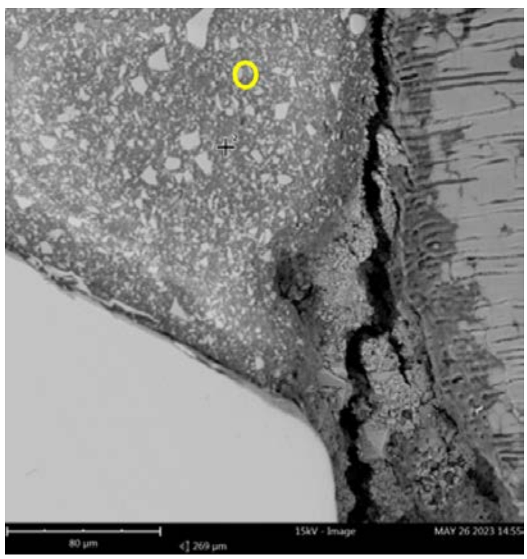
Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	47.15	43.26
6	C	Carbon	34.51	23.77
56	Ba	Barium	1.12	8.79
14	Si	Silicon	5.09	8.20
9	F	Fluorine	5.80	6.32
13	Al	Aluminium	2.65	4.10
11	Na	Sodium	2.77	3.65
20	Ca	Calcium	0.58	1.34
15	P	Phosphorus	0.33	0.58

c) sample 3.3

FIGURE 4. SEM examinations and EDX analyzes performed on PANAVIA SA used for cementation of the posts

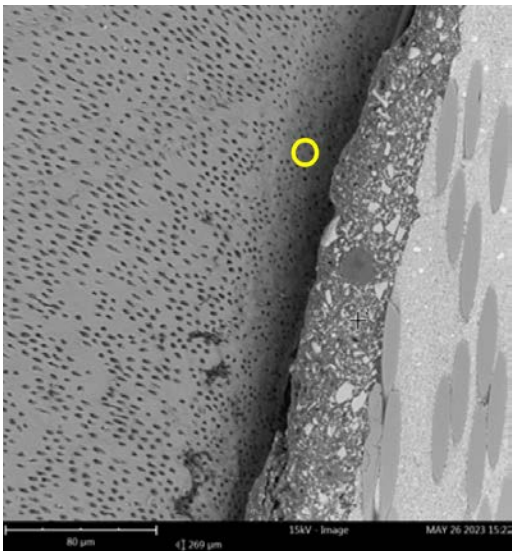


Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	59.88	50.23
8	O	Oxygen	36.04	40.27
14	Si	Silicon	2.26	4.43
22	Ti	Titanium	1.04	3.48
13	Al	Aluminium	0.57	1.07
20	Ca	Calcium	0.10	0.29
15	P	Phosphorus	0.11	0.23

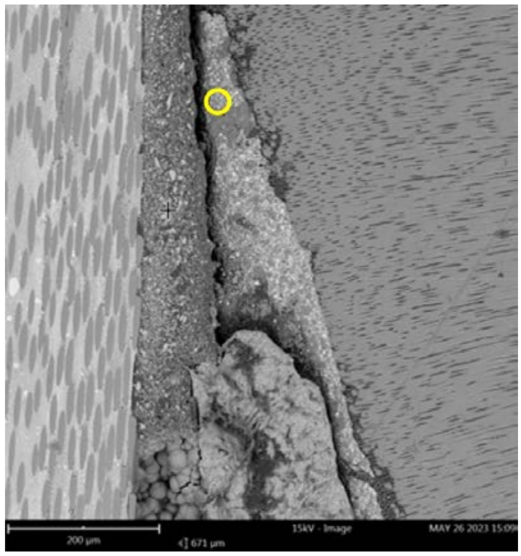


Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	61.16	50.04
8	O	Oxygen	31.79	34.65
14	Si	Silicon	4.12	7.89
22	Ti	Titanium	1.23	4.03
13	Al	Aluminium	1.18	2.16
15	P	Phosphorus	0.30	0.62
20	Ca	Calcium	0.23	0.62

a) sample 4.1

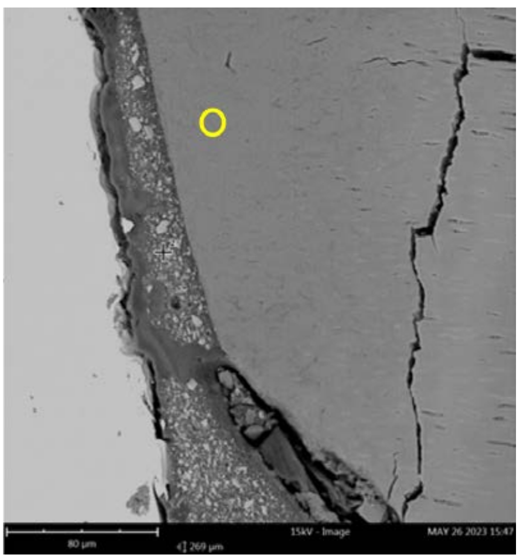


Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	57.06	36.10
8	O	Oxygen	27.79	23.42
56	Ba	Barium	2.23	16.15
14	Si	Silicon	10.39	15.37
35	Br	Bromine	1.81	7.62
20	Ca	Calcium	0.37	0.78
15	P	Phosphorus	0.34	0.55

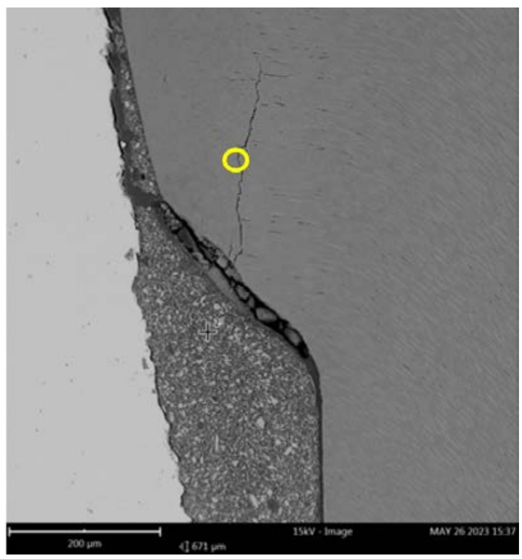


Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	39.44	34.59
6	C	Carbon	46.70	30.75
56	Ba	Barium	1.34	10.06
14	Si	Silicon	6.21	9.56
20	Ca	Calcium	2.66	5.84
35	Br	Bromine	1.21	5.31
15	P	Phosphorus	1.79	3.04
11	Na	Sodium	0.41	0.52
12	Mg	Magnesium	0.25	0.33

b) sample 4.2



Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	66.23	56.81
14	Si	Silicon	9.53	14.35
6	C	Carbon	18.26	11.76
58	Ce	Cerium	0.93	7.00
13	Al	Aluminium	2.56	3.70
22	Ti	Titanium	1.15	2.96
28	Ni	Nickel	0.69	2.17
20	Ca	Calcium	0.39	0.85
15	P	Phosphorus	0.24	0.41



Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	48.65	49.02
6	C	Carbon	43.76	33.10
14	Si	Silicon	4.68	8.28
58	Ce	Cerium	0.42	3.67
13	Al	Aluminium	1.45	2.46
22	Ti	Titanium	0.49	1.47
38	Sr	Strontium	0.23	1.25
20	Ca	Calcium	0.15	0.38
15	P	Phosphorus	0.18	0.36

c) sample 4.3

FIGURE 5. SEM examinations and EDX analyzes performed on CEMENT ONE used for cementation of the posts

cause the chemical activator of the self-polymerization reaction may have limited effectiveness on some dual cements [8]. Of course, as in the case of coronal fillings, the setting of cements based on composite resins is also inhibited by the local presence of eugenol-based materials [9]. Because of the limited access for both light-curing beam propagation and post application, it can be difficult to insert composite resin cements into the pulp chamber and especially into the root canals. This fact favors the appearance of material voids or even the incomplete setting of the composite resin if it is desired to use a minimally invasive technique for the preparation of roots [10]. Studies [11,12] have shown that teeth with small access cavities have an increased predisposition to the appearance of voids in the layers of composite materials. At the same time, the use of glassionomer cements as luting agents for posts in endodontically treated teeth showed encouraging results regarding the resistance of adhesion to the hard dental structure and to the device to be cemented [13,14]. NANOCORE DUAL manufactured by Dentalica, Italy, is a fluoride-releasing composite resin used for abutment restorations and abutment cementation. It is applied after acid attack and bonding. CEMBEST produced by BMSDental is a glassionomer cement, used for fixing crowns, dental bridges, inlays, onlays, posts or orthodontic devices. PANAVIA SA produced by Kuraray Noritake Dental is a precursor material to self-adhesive resin cements that contains a monomer that adheres to any material without requiring a special primer. It is used as a luting agent for crowns, dental bridges, inlays, onlays, posts or adhesive bridges. It has a dual self- and light-curing set: after the initial chemical/self-curing set, it is completed with a marginal light-curing set. CEMENT ONE is a self-adhesive, dual, self-demineralizing and polymerizable resin cement. No etching, primer or bonding required. It is used for cementing dental crowns and bridges, inlays, onlays, posts or Maryland bridges.

CONCLUSIONS

1. SEM and EDS studies provide valuable ultrastructural information regarding the degree of filling with luting material of the existing microspaces between the pivots and dental preparations and the degree of homogeneity of the luting material. 2. The four studied luting materials do not present an inhomogeneous composition at the EDX analysis, not having significantly increased values between the two analyzed points for each of the 12 samples taken in the study. 3. Among the four luting materials studied comparatively, PANAVIA SA seems to best fill the existing spaces between posts and the limits of dental preparations, being closely followed by CEMENT-One and CEMBEST. In last place is NANOCORE DUAL, which presents discontinuities in the mass of material and at the border with the preparations.

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