In vitro Study About Resistance of Adhesive Cements

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Adhesive cements (that chemically adhere to both the tooth and the restoration material) are considered to be a highly specialised group of composite cements. They involve a three-step procedure (acid etching, bonding agents or silane and composite resin), like cements using acid etching technique and have similar physical properties as the latest. The purpose of this study was to statistically analyse the values of the adhesive bond between dental tissue and ceramic, using three types of cements (both commercial products and a product from Romania) and one type of ceramic. The separate combinations were studied in order to identify the weakest link of the interface of dental-tissue-ceramic in terms of shear bond strength. The adhesion values recorded at the interface of cement-enamel for Dualcim and Nexus cements were significantly different from those recorded for Variolink cement. The highest resistance to adhesion was recorded to the interface cement Variolink – dentin, the average being 16.19 MPa.

Keywords: adhesion, enamel, dentin, ceramic

Adhesive cements are unique due to the achievement of adhesive bonding for both interfaces: dental tissues and restoration material. They have been modified chemically to have a high shear bond strength and simultaneously adhere to etched dental tissues and metal restorations, noble and non-noble, electrolytic etched or sandblasted. The adhesive technique for dental tissues is very sensitive and for the metal it varies depending on the alloy and involves the use of a special silane (metal primer) [1].

Some authors have investigated the use of adhesive cements for ceramic restorations on zirconium or alumina structure for dental abutments with reduced coronary height or high angulation. Treatment of surfaces of these restorations through micro-sandblasting, followed by application of an adhesive containing phosphate monomer (silane) has shown some promises, but there is a risk of lowering the structure strength of the zirconium infrastructure [2]. In a recent study (2014), Chen et al. [3] have shown that the use of adhesive cement together with specific silane determined the adhesion to zirconium.

There is a process of learning and adaptation for the use of these materials and the manufacturer's instructions should always be followed strictly to get the best result possible. Because they are relatively expensive, they have a sensitive working technique and generally the excess is difficult to remove, the use of adhesive cements would typically be indicated for cementing fully ceramic and metallic restorations, PMF restorations or restorations on zirconium infrastructure only in cases of dental abutments with low retentivity [4].

To take full advantage of adhesive cements, the abutments must be thoroughly cleaned by professional brushing, to remove residual temporary cement and any remaining debris before the lutting procedure, and then cleaned with chlorhexidine gluconate 0.12% [5]. Also, must be avoided using a temporary cement based on eugenol because residual eugenol may decrease adhesion of composite cement to hard dental tissues.

Experimental part

The materials used for the mechanical evaluation of adhesive bond between dental tissue and ceramic were:

- 60 recently extracted teeth, with integral dental crown, of which 55 maxillary and mandibular third molars, 3 premolars and 2 incisors, which were kept in artificial saliva after their extraction.
- Dental cements: Variolink II (Ivoclar Vivadent AG), Nexus NX3 (Kerr) and Dualcim (Chemistry Research Institute Raluca Ripan, Cluj Napoca, Romania).
- Orthophosphoric acid 37%, the product Total Etch™ (Ivoclar Vivadent AG), the product Acidenta (Chemistry Research Institute Raluca Ripan, Cluj Napoca, Romania).
- Dentin adhesives (primer and bonding): Syntac (Ivoclar Vivadent AG), Heliobond (Ivoclar Vivadent), Optibond Solo Plus (Kerr), Dentadez Foto (Chemistry Research Institute) Raluca Ripan, Cluj Napoca, Romania).
- Silane for ceramic mass: Monobond Plus (Ivoclar Vivadent AG).
- Hydrofluoric acid 4,9%: IPS Ceramic Etching Gel (Ivoclar Vivadent AG).
- 50 ceramic rods made of lithium disilicate glass ceramics IPS e.max Press (Ivoclar Vivadent AG).

Each of these cements was used according to the manufacturer's indications described in the instructions of each cement. The adhesive protocol used for each cement, both to dentin and enamel, was the acid etching technique.

The lithium disilicate glass ceramic rods were manufactured in the dental laboratory as follows: there were used prefabricated wax rods with 3 mm diameter, invested into a packed mass PressVest (Ivoclar Vivadent AG) for getting a pattern. In this pattern, the lithium disilicate glass ceramic (IPS e.max Press, Ivoclar Vivadent AG) was hot-pressed in a pressing furnace Programat EP3000 (Ivoclar Vivadent AG) according to the manufacturer's indications. Later, ceramic rods were unpacked by cutting the pattern followed by sandblasting, then they were finished and cut to a length of 10 mm. This way we obtained the same diameter for each rod used in the experiment i.e. 3 mm, whereas the pressing technique has an accuracy of 1:1 (fig. 1).

Obtaining the test samples was performed by fixing teeth in acrylic resin. In order to adequately adapting the samples to the table testing machine, a silicone key was

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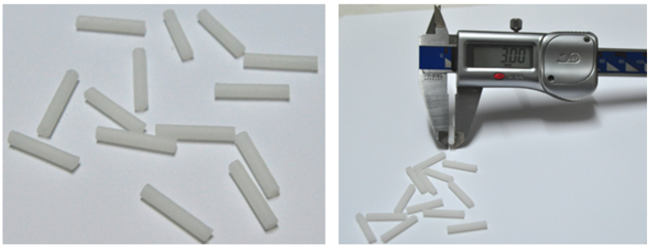

Fig. 1. Ceramic rods and measuring instrumeny - calliper

made for imprinting in which the acrylic resin was moulded. Before acrylic resin setts, two randomly chosen teeth of those taken for the experiment were introduced in acrylic resin and thus being secured. To obtain adhesions to enamel, upright teeth were fixed in acrylic resin and for the adhesion to dentin, the teeth were partially sectioned (fig. 2).

Fig. 2. Teeth fixed acrylic resin and prepared for fixing the ceramic rods on enamel and dentin

After obtaining these samples, they were divided into two groups, one for the enamel and one for dentin, in order to cement the ceramic rods to enamel and dentin with the cements taken into study.

Group I is used to evaluate the adhesive cementing to enamel and comprises 15 samples (30 teeth), for cementing 10 ceramic rods with each of the three cements studied. Two rods on each teeth will be cemented, each rod on one axial surface of the tooth, randomly chosen and prepared in advance for adhesive cementation.

Preparing the enamel for adhesive cementation was achieved by drilling the axial surface until a flat surface was obtained using cylindrical diamond burs: there were used successively two granulations of diamond burs: medium (blue ring) and fine (yellow ring) at a speed of 40000 rot/min (fig. 3).

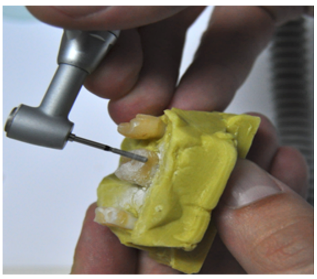

Fig. 3. Surface preparation by drilling

Subsequently the samples were washed under running water for 15 s and then dried with air for 15-20 s. The surfaces thus prepared were acid etched with 37% ortophosphoric acid: the product Total Etch ™ (Ivoclar Vivadent AG) was used for the cemented samples with Variolink II (Ivoclar Vivadent AG) and Nexus NX3 (Kerr) and Acidenta product (Chemistry Research Institute Raluca Ripan, Cluj Napoca, Romania) for the samples cemented with Dualcim (Chemistry Research Institute Raluca Ripan, Cluj Napoca, Romania).

After etching, depending on the cement used, the adhesives recommended by the cement manufacturer were applied to enamel as follows:

- for Dualcim adhesive Dentadez Foto;
- for Nexus NX3 the adhesive Optibond Solo Plus;
- for Variolink II adhesive Syntac, followed by Heliobond.

The cementing surface of ceramic rods was initially cleaned with garnet paper Smirdex® 270 Silicon Carbide P600, for one minute, in wet environment and is presented in figure 4.

Fig. 4. Ceramic rods preparing. Cleaning on a sandpaper Smirdex® 270 Silicon Carbide P600

These rods were then cleaned for 5 min in an ultrasonic bath with distilled water, dried with air jet for 20 s and afterwards etched with hydrofluoric acid 4.9 % (product IPS Ceramic Etching Gel, Ivoclar Vivadent) for 20 s , in accordance with the indications of the producer. Subsequently they were washed with running water for 15 s and reintroduced in the ultrasonic bath with distilled water for 5 min. Afterwards they were dried with air jet for 60 s and then divided randomly in three groups of 10 rods which are to be cemented, to each of the three cements studied.

For Variolink II, after acid etching of ceramic mass, the producer indicates the silanization of ceramic mass with Monobond Plus and then Heliobond. For other cements studied, the manufacturers do not indicate the use of a silane after the acid etching at the ceramic mass. All adhesive cements studied have dual setting mechanism and preparation of each cement was done according to the manufacturer's indications. A minimum amount of cement was applied on the surface of cementing the ceramic rod, and then the rod has been fixed to the enamel.

The excess of cement was removed after its pre-photo-polymerization for 1-3 s. After excess removing, the final photo-polymerization of 20 s continued from four directions (superior, inferior, lateral-left and lateral-right); antipodal directions of polymerization from the axis of ceramic rod were used. The lamp of polymerization used both for composite cement and for adhesives was a halogen lamp Optilux 501 (Kerr), whose bulb has been replaced with a new one at the beginning of this experiment (OptiBulb-Kerr). The samples prepared to test the adhesion of the enamel are shown in figure 5.

Fig. 5. Samples prepared for testing the adhesion to enamel

Group II. This group is used for testing the adhesion to dentin and comprises 15 samples (30 teeth), for cementing 10 ceramic rods with each of the three cements studied. Each tooth from this group was sectioned in the long axis (crown-root) to expose a large surface of dentin. The sectioning was done with a diamond disc at conventional speed of 40000 rpm, distilled water-cooled. After sectioning, each dentin surface was finished with a diamond bur of fine granulation (yellow ring).

For all the cements studied, the adhesive technique used to both enamel and dentin was acid etching. The dentin was acid etched with 37% ortophosphoric acid as follows: product Total Etch ™ (Ivoclar Vivadent AG) was used for samples cemented with Variolink II (Ivoclar Vivadent AG) and Nexus NX3 (Kerr) and the product Acidenta (Chemistry Research Institute Raluca Ripan, Cluj Napoca, Romania), for the samples cemented with Dualcim (Chemistry Research Institute Raluca Ripan, Cluj Napoca, Romania). The etching time was 10 s for all samples.

After etching, depending on the cement used, the adhesives recommended by the cement production company were applied to enamel as follows:

- for Dualcim the adhesive Dentadez Foto;
- for Nexus NX3 the adhesive Optibond Solo Plus;
- for Variolink II the adhesive Syntac, followed by Heliobond.

Preparing the ceramic rods was performed similar to Group I.

The samples prepared to test the adhesion to enamel are presented in figure 6.

After samples achivement, three from each group, for each cement were tested at 10 min after manufacturing in order to obtain the initial values. The remaining samples were immersed in artificial saliva (Artisial®/ Biocodex, France) and kept at room temperature for a period of 6 months when a test was performed, after ageing the samples, in laboratory conditions.

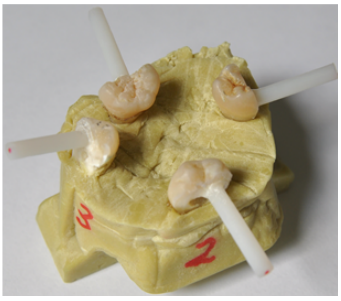

Fig. 6. Samples prepared for testing the adhesion to dentin

Adhesion to dentin and to enamel was performed by recording the resistance to shear bond strength. The values were recorded on a device for mechanical testing Instron Universal-Loyd type (fig. 7). The lowering speed of the measuring rod was set at 0.75 mm/min.

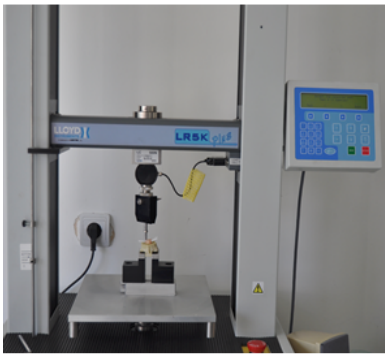

Fig. 7. Mechanical testing machine Lloyd Instruments LR5K plus

The samples were specifically designed to match the table of the testing device used. The testing table was set for each sample so the lowering axis so that the device rod was parallel to the ceramic-tooth interface. The mechanical testing of samples is shown in figure 8 and the graphic obtained in figure 9.

Results and discussions

The mean values obtained as a result of the test of adhesion to enamel and dentin is shown in table 1, as the ratio of the measured force and the corresponding areas were previously calculated.

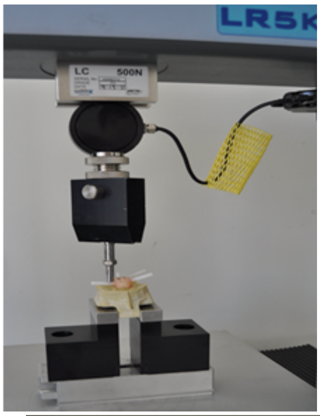

Fig. 8. Mechanical testing of the samples

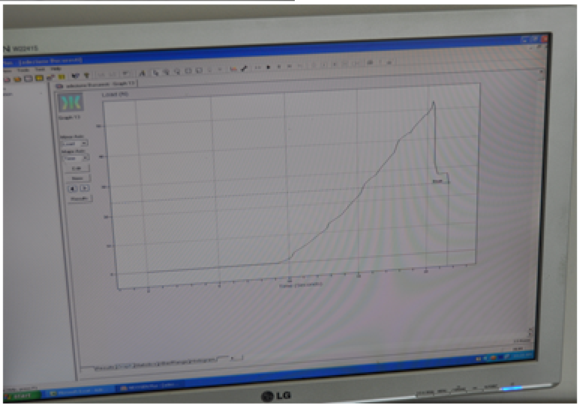

Fig. 9. Graphic representation of the obtained values. Graphic recorded during mechanical testing

For this type of test were used bars of pressed ceramic shown in figure 2. The recordings were made after 10 minutes from the cementing the ceramic rods to enamel and dentin and respectively after 6 months.

In table 2 is presented the statistical analysis of the mean values of adhesion to enamel depending on the cementing material used in this study. Analysis of variation showed that there were statistically significant differences for Nexus cementing material compared with Dualcim material ($p<0.003$), Variolink ($p<0.008$), in terms of measured adhesion.

In figure 10 is presented the mean value of the adhesion resistance to enamel for the three types of cement. It is noted that the lowest value was obtained for Dualcim cement, and the highest for Variolink cement.

In table 3 is presented the statistical analysis of the mean values of adhesion to dentin for the three types of cement used in the study. Analysis of the variation in this case reveals that there are statistically significant differences for Variolink cementing material ($p<0.001$) compared with Nexus cementing material ($p<0.0051$).

We also have statistically significant differences for Dualcim cementing material compared to Variolink cement. The mean value of adhesion resistance to dentin obtained indicates Variolink cement with the best values and it is shown graphically in figure 11.

The statistical comparison between the dentin and enamel for the three cementing materials used is presented in table 4. The results obtained do not show statistically significant differences between the dentin and enamel for the three cementing materials.

The statistical comparison depending on the cementing material to enamel and dentin (fig. 12) shows that there are no statistically significant differences in terms of measure mean values of adhesion. The results for adhesion are better for Variolink cement than for the Nexus and Dualcim cements.

Samples names	
Dualcim_enamel_ceramic	7.65
Dualcim_enamel_ceramic	8.06
Dualcim_enamel_ceramic	7.58
Dualcim_enamel_ceramic	8.25
Dualcim_enamel_ceramic_after 10 minutes	6.99
Dualcim_dentin_ceramic	6.51
Dualcim_ dentin _ceramic	7.23
Dualcim_ dentin_ceramic	7.68
Dualcim_ dentin _ceramic	7.05
Dualcim_ dentin _ceramic_after 10 minutes	7.89
Variolink II dentin_ ceramic	16.37
Variolink II dentin_ ceramic	16.18
Variolink II dentin_ ceramic	15.23
Variolink II dentin_ ceramic	16.98
Variolink II dentin_ceramic_after_10 minutes	17.15
Variolink II enamel_ ceramic	16.88
Variolink II enamel_ ceramic	15.37
Variolink II enamel_ ceramic	15.12
Variolink II enamel_ ceramic	15.67
Variolink II enamel_ceramic_after_10 minutes	18.54
Nexus 3 dentin_ ceramic	12.02
Nexus 3 dentin_ ceramic	12.11
Nexus 3 dentin_ ceramic	11.76
Nexus 3 dentin_ ceramic	11.98
Nexus 3 dentin_ ceramic_after_10 minutes	15.78
Nexus_enamel_ ceramic	12.65
Nexus_enamel_ ceramic	13.34
Nexus_enamel_ ceramic	11.98
Nexus_enamel_ ceramic	11.35
Nexus_enamel_ ceramic_after_10 minutes	16.22

Table 1
MEASURED VALUES AT MECHANICAL TESTING OF THE ADHESION FOR ALL THE TESTED SAMPLES

Sample type	Mean (MPa)	SD	N	SEM	p (t test)			ANOVA	
					vs. Dualcim	vs. Variolink	vs. Nexus	p	F
Dualcim	7.885	0.323	7	0.161	1.000	**0.000**	**0.003**	**0.000**	129.3
Variolink	15.76	0.780	7	0.390	**0.000**	1.000	**0.008**		
Nexus	12.33	0.857	7	0.429	**0.003**	**0.008**	1.000		

Table 2
STATISTICAL ANALYSIS OF THE MEAN VALUES OF ADHESION TO ENAMEL DEPENDING ON THE CEMENTING MATERIAL

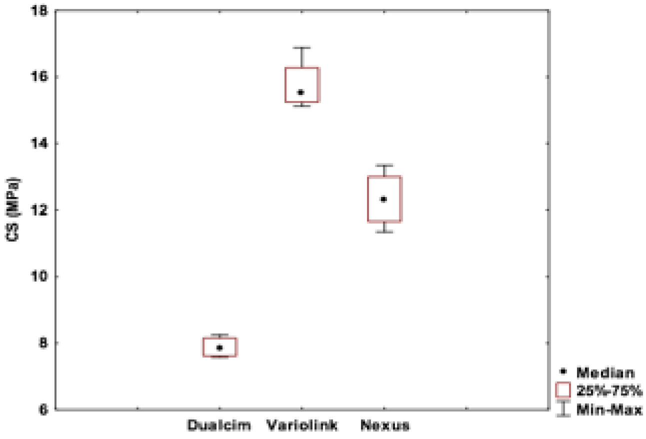

Fig. 10. Mean value of the adhesion strength to enamel for all three tested materials.

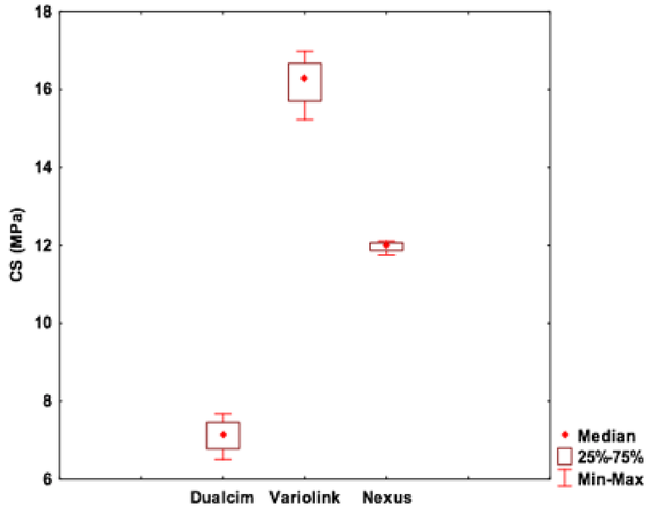

Fig. 11. Mean value of adhesion strength to dentin for all three tested materials

Table 3
STATISTICAL ANALYSIS OF THE ADHESION TO DENTIN ACCORDING TO LUTING MATERIAL

Sample type	Mean (MPa)	SD	N	SEM	p (t test)			ANOVA	
					vs. Dualcim	vs. Variolink	vs. Nexus	p	F
Dualcim	7.118	0.484	7	0.242	1.000	0.000	0.000	0.000	316.1
Variolink	16.19	0.725	7	0.363	0.000	1.000	0.001		
Nexus	11.97	0.149	7	0.074	0.000	0.001	1.000		

SD – standard deviation, N – measurements number, SEM – standard error of the mean, $p < 0.05$ (statistically significant).

Table 4
STATISTICAL COMPARISON BETWEEN DENTIN AND ENAMEL FOR ALL MATERIALS

Sample type	p (t test)			p (t test)			ANOVA	
	Enamel			Dentin				
	Dualcim	Variolink	Nexus	Dualcim	Variolink	Nexus	p	F
Enamel	1.000	1.000	1.000	0.0835	0.3595	0.4217	0.878	0.024
Dentin	0.0835	0.3595	0.4217	1.000	1.000	1.000		

D – standard deviation, N – measurements number, SEM – standard error of the mean, $p < 0.05$ (statistically significant).

The results of the ANOVA test regarding the comparison of adhesion values to enamel and dentin in terms of the cementation materials used are given in table 4. In all cases the differences are not statistically significant ($p<0.05$) but for the bigger values of F (the Fischer coefficient) obtained in dentin, we have increased statistically significant differences.

The shear bond strength for adhesion is the most commonly used laboratory parameter to evaluate the strength to pulling away the restoration materials from the dental structures. A major disadvantage of these tests is that they do not take under consideration the three-dimensional geometry of dental abutments and constant variations of polymerisation shrinkage values. Therefore, data from adhesion tests should be evaluated together with the clinical results [6]. However, this test is an excellent tool for screening new materials and comparison of different systems for cementing.

The use of composite cements for lutting dental restorations, particularly those fully ceramic, is quite

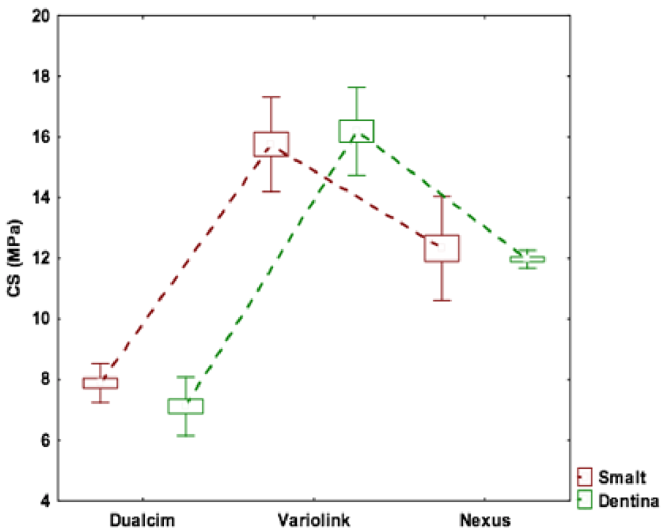

Fig. 12. Statistical comparison between enamel and dentin according to the luting material

common. Suitable polymerisation of composite cement will lead to higher adhesion forces between the tooth and the restoration. Light intensity is one of the most important factors affecting the polymerisation to light-curing cements. Recent studies have shown a positive correlation between the light intensity and the degree of conversion of restorative materials [7]. Since 1994, Rueggebert et al. [8] suggested that the appropriate intensity for a unit of photo-polymerisation is 400 mW/ cm^2, in order to initiate the material's polymerisation, but it becomes inadequate when the lamp of photo-polymerisation produces a light intensity below 233 mW/ cm^2.

A better bonding to denting can increase the resistance of remaining dental structures and reduces the marginal micro-infiltration between the tooth and the restoration, features which increase the survival rate of the tooth-restoration complex. To support the use of these materials, we tested the hypothesis of research which indicates the values of resistance to adhesion for different materials of cementing interposed between ceramic and enamel or dentin.

Cementing materials tested in this study has shown no significant differences between enamel and dentin. Hikita et al. [9] have tested the adhesion to dentin for Variolink and Nexus cements obtaining similar values. Other studies [10] have shown that Variolink II presented a higher bonding resistance than Nexus II, which we also obtained in this study. Regarding the treatment of surfaces, few systems of composite cement seem to be able to produce an adequate degree of conversion and high adhesion resistance with dentin [11], while adhesion to enamel is no longer a problem for contemporary cements [12].

Dentin bonding was the target of numerous studies [13, 14, 15], in an attempt to achieve adhesive systems able to interact effectively with this delicate substrate. Compared to enamel, dentin bonding is more difficult to achieve, due to its morphological characteristics, to its high organic content and tubular structures partially filled with odontoblastic process [16]. Formation of remaining dentinal detritus which leads to the closure of tubules through detritus plugs and thus reduces the dentin permeability is another reason why the interaction between the adhesive system and dentin is difficult [17].

The comparison between enamel and dentin in terms of mean values (table 4) obtained for adhesion, highlights that the Variolink cement does not have notable differences.

In Dualcim cement we have obtained similar values to both enamel and dentin, the best value being registered for enamel (7.885 MPa). For Nexus cementing material were obtained better values for adhesion to enamel (12.33 MPa) compared with the mean values of adhesion to dentin (11.97 MPa).

The values of adhesion registered on the interface of cement-enamel for Dualcim and Nexus cements were significantly different from those recorded for Variolink cement. The highest resistance to adhesion was registered on interface Variolink cement-dentin, the average being of 16.19 MPa.

Studies to determine the performance of different adhesive systems and techniques are important especially for their relative values obtained, but the numerical comparisons are not always possible. When similar studies are consulted, a big variability can be observed between the results of shear bond strength tests, depending on the method used. Therefore, MPa numbers obtained by different authors are not compared, but it is rather preferred to explore trends in the behaviour of the materials studied.

All crowns, fully ceramic inlays or onlays benefit, also, of adhesive cementation.

In vitro and in vivo studies published in the literature regarding the resistance to fracture and sealing the interface of cementation argue that adhesive cementation provides a uniform distribution of mechanical stress and stops the propagation of micro cracks at the internal face of fully ceramic restorations [18].

Acid etching of fully ceramic reconstructions should be done carefully, by respecting the etching time and the concentration of hydrofluoric acid indicated by the manufacturer, because there is the danger of over-etching, which lowers composite cement adhesion [19]. In addition, the various methods of cleaning the acid etched ceramic surfaces do not influence the adhesion of composite cement to ceramic [20]. When we cement a ceramic restoration (for example a fully ceramic crown) it is indicated to use a self-etching adhesive system with a light-curing cement or a dual cure cement, depending on the degree of translucency of the crown.

The pattern of cements fracture included in this study showed that the main trap of adhesion to dentin is an incomplete adherence to instrumented dentin. The remanence of dentinal detritus may be responsible for the resistance to low shear bond strenght, since it has been shown clearly that, to achieve a positive link to dentin, the dentinal detritus must be removed and the collagen fibers must be exposed to allow adhesive materials to enter this network [21].

The shear bond strength test, which is frequently used to determine adhesion, often produces cohesive fractures (detachment of interface substrate). This method of fracturing provides only limited information about the true bond strength. On the other hand, low levels of adhesion to dentin and enamel, match the smaller sizes of interface, and the connection area of tested specimens (ceramic rods with 3mm diameter) greatly reduces the number of defects that may form and leads to a more uniform distribution of pressures applied.

Conclusions

Although adhesive cementation involves a complex mechanism and a longer working time, it is recommended its use to get the best possible connections between micro-prostheses and dental tissues.

In our study, the adhesion of Variolink cement to dentin was significantly higher than to the other two composite cements Nexus and Dualcim, but there wasn't any significant difference between the enamel and dentin for the three materials.

The glass ionomer cements influenced by the storage media were studied in [22].

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