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Nanostructure of Orthodontic Adhesives

Vladan D. Mirjanić

University of Banja Luka, Faculty of Medicine, Department of Stomatology, Republic of Srpska, B&H,
vladan.mirjanic@med.unibl.org

Đorđe D. Mirjanić

Community Health Center, Banja Luka, Republic of Srpska, B&H, djordjemirjanic@gmail.com

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Abstract

Nanostructure of the orthodontic adhesives for bonding brackets to teeth most frequently used nowadays in clinical procedure is analyzed by way of Atomic Force Microscopy (AFM). Study was done on 5 orthodontic adhesives. After determining the properties of adhesive, a correlation was determined between the nanostructure of tested adhesive and the strength of tooth bracket interface. Based on AFM images of analyzed adhesives, and by way of correlations of arithmetic means of debonding strength (I) and the average adhesive roughness (Ra, Rq, Rz), it has been concluded that with an increase of average adhesive roughness's, increases the debonding strength. (I). It was observed that with all the roughness parameters (Ra, Rz, and Rq), the strongest bond and the weakest bond was determined Higher roughness of Resilience Orthodontic bonding solutions at the nano level is probably enabled by a bigger number of thorns that penetrate into micro concaves formed under the influence of acids. Higher roughness is a consequence of chemical structure itself of the composite material.

Key words: Orthodontic adhesives, nanostructure, Atomic Force Microscopy (AFM)

1. INTRODUCTION

Tooth enamel is made of a billion crystals of carbonized hydroxyapatite [1-3] that are packed in individual prisms winding from enamel-dentin border toward the tooth surface. When the enamel prisms are observed on cross-section by way of electronic microscopes, they do not have an appearance of a prism (small stick) but are seen as structures in the form of a key hole, with 6-8-micron diameter. With such an appearance, a larger part is differentiated, designated as „head“ while the narrower is designated as „tail“. Each head fits between two tails. Crystals are in the area of the head lined along the longitudinal axis, designated as a „C axis“, while on the periphery („tail“) they are ordered at an angle of 30° [4-6].

The mineral phase with mature enamel takes up about 87% of total volume of enamel mass, and makes over 95% of weight mass, of which only 5% belongs to organic matters and water (other biological mineralized tissues contain about 20%). 3-5% of voluminous mass are made of porosities formed from the network of channels. Through it, throughout the whole enamel cover, diffuse the fluids, ions and small molecules. This area is located between the prisms, but also between the crystals. This network is joined by morphological structures richer in proteins such as the above-mentioned striae of Retzius, enamel tufts and spindles. Canicular system is considered to have a protective role

because 1) it enables physiological remineralization of enamel prisms throughout life and 2) space, liquids and proteins partly participate in the amortization of big pressures that are released during chewing and prevent forming of fractures. At the same time, this canicular system enables penetration of acids, and even of bacteria and helps the development of caries and erosions. [3, 4, 5, 7, 8].

Enamel surface is not flat. It has a wavy structure because at places where Retzius' striae end such striae overlap in form of the steps, with the appearance of shallow grooves referred to as *perikymata*. At certain places, especially with deciduous teeth, there are a number of microns of enamel on the surface without prismatic organization – *aprismatic enamel*. [3, 4].

Although enamel has pronounced hardness, it is also especially fragile at the same time and glass-like, and as such it would be prone to braking. Despite that, enamel can withstand loads higher than 1000 N several times during the day. The overall enamel microstructure is formed in such a way to adjust to such loads.

This is also contributed by the support of elastic dentin and the structures such as enamel tufts at the dentin-enamel junction [9-11].

Enamel is in constant dynamic communication with oral cavity ecosystem. Demineralization and remineralization processes are always present and their balance ensures enamel integrity.

If external aggressive factors direct balance toward demineralization activities, the integrity of the crystal grid weakens, hardness and resistance of enamel reduces, which, after crossing a certain limit of mechanical resistance of enamel, leads to its cracking and formation of cavities, as a beginning of irreversible damage. [7, 8].

In the paper will be use currently the most modern technology that is based on Atomic Force Microscopy (AFM) for testing nanostructure of orthodontic adhesives for bonding brackets to teeth. [12, 13].

The topography of adhesive nanostructure will be used for statistical analysis.

Topography represents the surface of the nanostructure of adhesive and tooth enamel which is obtained by calculating Ra, Rq, and Rz roughness. Roughness is defined as a complex set of irregularities or bulging and prongs which give appearance to the surface and make influence on wetting, quality of adhesion and lightness. Although it is underlined that micromechanical roughness is a basis of a good junction between the etched enamel and resin, precise characteristics of enamel necessary to realize such a bond are not known. [14].

The influence that roughness has on the bond strength not completely understood either. [15]. Higher roughness is assumed to provide a bigger contact area through which contact with resin is realized, and thereby a stronger bond too. [16].

Something that has not been investigated so far in detail is a surface roughness at microscopic level [17] where nano characterization of surface roughness could provide biophysical mechanisms on enamel surface [18]. AFM with high lateral and vertical resolution enables testing the roughness at micro and nano levels without higher interference of macroscopic components such as the wavy surface [19]. AFM microprobe does not require preparation of a sample and thus jeopardizing the original surface is avoided. Thereby it represents a direct way to experimentally detect and quantify the surface roughness.

Each sample will have 256 lines. For statistical analysis the dimensions of each of observed nanostructures are calculated.

2. MATERIAL AND METHODS

Nanotechnological device JSPM-5200 which is located in NanoLab module for biomedical engineering at the Faculty of Mechanical Engineering of Belgrade University [20-22] was used to test the nanostructure of adhesive. This is an integrated nanosystem with a number of operating modes with which it is possible to realize the following functions: STM, AFM, MFM, ECSPM etc.

JSPM-5200 consists of AFM base, the anti-vibration table, AFM amplifier, SPM controller, computer and optional components such as the microscopic system with CCD camera, vacuum system, etc. [23]. Adhesive samples are fixed to AFM microscope holder.

Testing the surface was done in „contact mode“ function, which means that the physical contact between AFM probe and tooth surface is a constant force.

The scan was analyzed by using the program WinSPM (Processing). This program package enables the user to perform different processing functions in order to improve the quality of the image obtained by the scanning program. These functions include: image levelling, adjustment of the light and contrast, application of different filters, etc.

The analysis of the profile on the image of the scanned surface may be done in a number of ways: Single, Multi, Extra and Multiple Images. With Single analysis, one production line may be placed in whichever direction within the image, while the distances between two points and the height difference between up to three marker pairs are measured. With Multi Analysis up to five arbitrary lines in whichever direction within the image may be placed.

With Extra analysis the roughness of scanned area is measured within the placed rectangular area, while with Multiple Images Analysis up to three images may be placed, while the profile is analyzed on the same line. Here we used the Multi analysis of the profile.

This program, WinSPM (Processing) also enables generation of three-dimensional images of scanned area (bird-eye-view). The parameters that may be adjusted are the following: Position (direction of view), Zoom (height per Z-axis) and Centering (centering the surface with regards to the screen).

We finally use the function of making reports which is used to display images, profiles and 3D images in the form of reports for printing that are presented in research results. It is implied that the format of the page is A4, in vertical layout. The data on measuring for the selected 2D image may be presented.

The dimensions of each of observed nanostructures are calculated for statistical analysis, while the height of certain nano-structures, i.e. the basic parameter is represented by the difference between the „highest hill“ and the „deepest valley“ along the Z-axis.

3. RESULTS

Due to the limitation of the space we will not here present the AFM images of adhesive samples but will be present the results of regression analysis of analyzed adhesives with regression parameters for each adhesive as well as the comparison of samples by average roughness for each adhesive: Sample 1- ConTecLC – Dentaurum, Sample 2- GC Fuji Ortho LC, Sample 3- Heliosit, Orthodontic (Ivoclar, Vivadent) and Sample 4-Resilience Orthodontic bonding solutions, Ortho Technology Inc. Florida. Then will be present the correlations of arithmetic means of debonding strength (I) and of average Ra, Rq, and Rz roughness for all adhesive Table 1 shows the distribution of arithmetic means of average Ra roughness for all

four analyzed samples Total data (with arithmetic means of measuring images).

The distribution of arithmetic means of average roughness Rq for all four analyzed samples is presented in Table 2.

Table 1. Distribution of arithmetic means of average adhesive roughness Ra

Place of measurement	Ra [nm]			
	Sample 1	Sample 2	Sample 3	Sample 4
1	0.605	52.400	0.911	36.800
2	1.100	94.500	6.070	21.100
3	1.150	66.400	1.650	19.500
4	2.260	18.400	3.070	44.300
5	1.380	131.500	1.090	19.200
6	0.851	23.050	4.100	9.290
7	1.110	80.700	2.680	41.200
8	1.310	15.600	5.580	8.050
9	1.130	44.800	0.783	21.100
10	1.730	45.900	5.810	48.500
11	1.420	72.900	5.400	19.000
12	1.870	84.500	4.000	15.400
13	1.040	68.400	3.690	4.900
14	1.490	21.500	6.320	23.400
15	2.360	18.400	3.090	23.700
Xsr	1.387	55.930	3.616	23.696

Table 2. Distribution of arithmetic means of average roughness of adhesive Rq

Place of measurement	Rq [nm]			
	Sample 1	Sample 2	Sample 3	Sample 4
1	0.964	59.900	1.040	40.500
2	1.440	102.200	7.930	26.800
3	1.290	79.900	2.120	25.000
4	2.900	22.100	3.710	50.800
5	2.440	146.800	1.290	23.900
6	1.150	28.600	5.050	10.100
7	1.310	88.600	6.400	51.000
8	1.670	18.300	7.640	10.800
9	1.340	51.000	1.000	24.400
10	2.060	55.400	7.010	50.800
11	1.770	80.300	6.400	22.900
12	2.540	113.800	4.960	17.500
13	1.230	74.100	4.190	5.710
14	2.070	25.100	7.170	25.200
15	2.650	26.600	3.870	25.500
Xsr	1.788	64.847	4.652	27.394

The regression diagram of arithmetic means of average Ra roughness for all four analyzed samples is presented in Figure 1.

The regression diagram of arithmetic means of average roughness Rq for all four analyzed samples is presented in Figure 2.

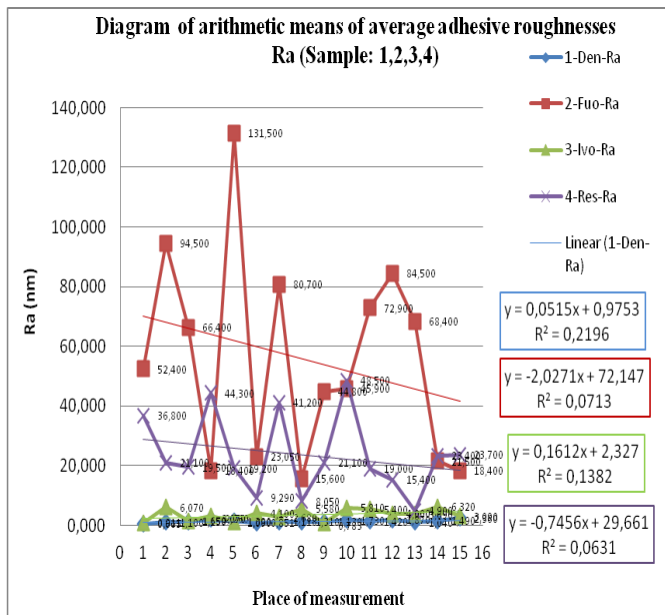


Figure 1. Regression diagram of arithmetic means of average roughness of adhesive Ra

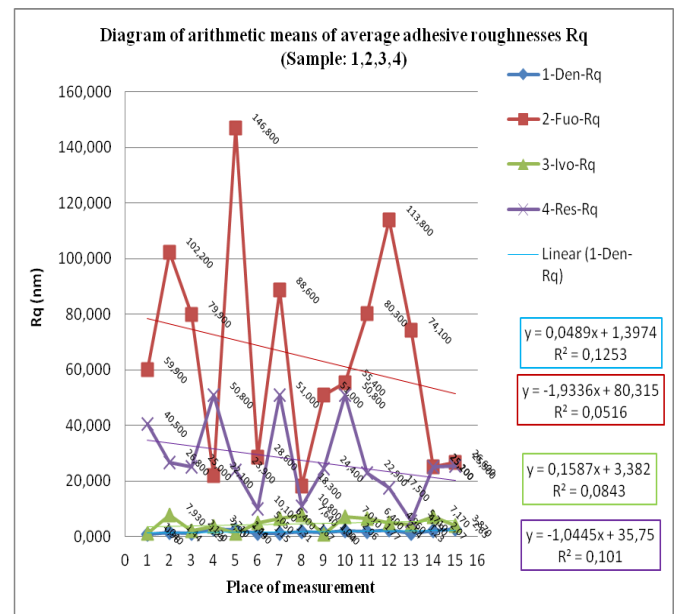


Figure 2. Regression diagram of arithmetic means of average adhesive roughness Rq

The distribution of arithmetic means of average roughness Rz for all four analyzed samples is presented in Table 3.

Table 3. The distribution of arithmetic means of average adhesive roughness

Place of measurement	Rz [nm]			
	Sample 1	Sample 2	Sample 3	Sample 4
1	5.580	183.600	3.970	110.100
2	7.680	286.800	33.100	109.700
3	4.840	301.600	8.720	99.900
4	14.600	78.900	17.600	183.300
5	11.300	423.600	5.670	103.600
6	7.630	97.800	20.400	30.200
7	5.070	255.900	17.400	192.800
8	7.670	61.600	26.500	61.000
9	6.690	187.700	5.480	76.700
10	9.220	211.500	27.600	133.000
11	8.330	220.900	27.100	81.300
12	15.000	441.900	20.400	59.700
13	5.800	201.100	16.500	19.400
14	12.900	82.700	23.400	72.700
15	8.510	150.900	14.300	67.900
Xsr	8.721	212.433	17.876	93.420

Regression diagram of arithmetic means of average Rz roughness for all four analyzed samples is presented in Figure 3.

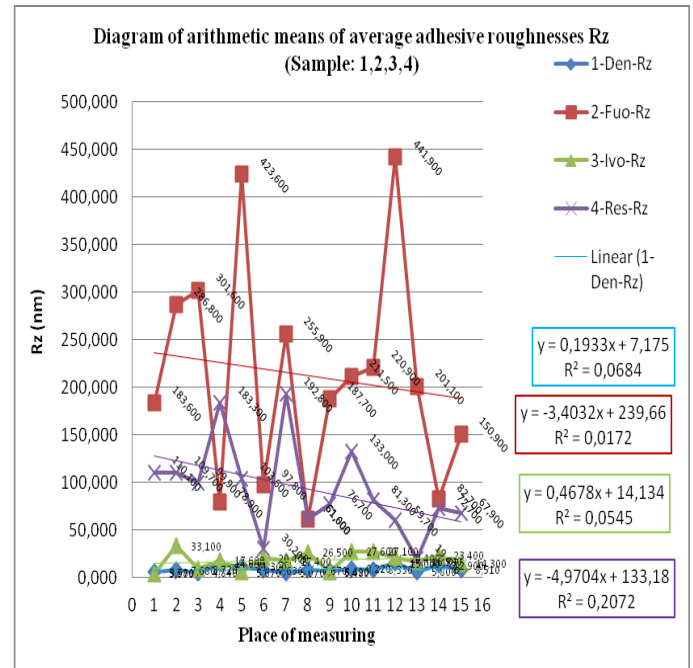


Figure 3. Regression diagram of arithmetic means of average adhesive roughness Rz

The regression parameters of the samples show functional dependence of average adhesive roughness with regards to the place of measurement. Regression parameters of all analyzed samples by adhesive roughness (Ra, Rz, Rq) are presented in Table 4., Table 5, Table 6. and Table 7.

Table 4. Regression parameters of 1st sample

Parameter designation	Regression equation (y=ax+b)	a	b	Determination coefficient (R ²)	Correlation coefficient (r)
1-Den-Ra	y = 0.0515x + 0.9753	0.0515	0.9753	0.2196	0.4686
1-Den-Rz	y = 0.1933x + 7.175	0.1933	7.175	0.0684	0.2615
1-Den-Rq	y = 0.0489x + 1.3974	0.0489	1.3974	0.1253	0.3540

Table 5. Regression parameters of 2nd sample

Parameter designation	Regression equation (y=ax+b)	a	b	Determination coefficient (R ²)	Correlation coefficient (r)
2-Fuo-Ra	y = -2.0271x + 72.147	-2.0271	72.147	0.0713	0.2670
2-Fuo-Rz	y = -3.4032x + 239.66	-3.4032	239.66	0.0172	0.1311
2-Fuo-Rq	y = -1.9336x + 80.315	-1.9336	80.315	0.0516	0.2272

Table 6. Regression parameters of 3rd sample

Parameter designation	Regression equation (y=ax+b)	a	b	Determination coefficient (R ²)	Correlation coefficient (r)
3-Ivo-Ra	y = 0.1612x + 2.327	0.1612	2.327	0.1382	0.3718
3-Ivo-Rz	y = 0.4678x + 14.134	0.4678	14.134	0.0545	0.2335
3-Ivo-Rq	y = 0.1588x + 3.382	0.1588	3.382	0.0843	0.2903

Table 7. Regression parameters of 4th sample

Parameter designation	Regression equation (y=ax+b)	a	b	Determination coefficient (R ²)	Correlation coefficient (r)
4-Res-Ra	y = -0.745x + 29.661	-0.7456	29.661	0.0631	0.2512
4-Res-Rz	y = -4.9704x + 133.18	-4.9704	133.18	0.2072	0.4552
4-Res-Rq	y = -1.0445x + 35.75	-1.0445	35.75	0.101	0.3178

The correlation analysis of the debonding strength (I) and average roughness is here reported only for Sample 1-ConTec LC – Dentaurum for which measuring of the debonding strength was performed (I), while for the other samples (Sample 2- GC Fuji Ortho LC, Sample 3- Heliosit, Orthodontic (Ivoclar, Vivadent) and Sample 4-Resilience Orthodontic bonding

solutions, Ortho Technology inc. Florida.) the analysis was carried out on the basis of experimental research done by other authors, and it will be presented through the discussion in this paper.

Debonding strengths (I) for the places of measurement with the average adhesive roughness Ra, Rq and Rz are presented in Table 8.

Table 8. Correlation of arithmetic means of debonding strengths (I) and average adhesive Ra, Rq and Rz roughness (Sample 1- ConTec LC – Dentaurum)

Place of measurement	Ra [nm]	I [MPa]	Rq [nm]	I [MPa]	Rz [nm]	I [MPa]
1	0.605	5.400	0.964	5.400	4.840	7.550
2	0.851	7.200	1.150	7.200	5.070	6.030
3	1.040	6.940	1.230	6.940	5.580	5.400
4	1.100	5.820	1.290	7.550	5.800	6.940
5	1.110	6.030	1.310	6.030	6.690	7.220
6	1.130	7.220	1.340	7.220	7.630	7.200
7	1.150	7.550	1.440	5.820	7.670	6.800
8	1.310	6.800	1.670	6.800	7.680	5.820
9	1.380	6.510	1.770	5.550	8.330	5.550
10	1.420	5.550	2.060	6.250	8.510	7.640
11	1.490	5.900	2.070	5.900	9.220	6.250
12	1.730	6.250	2.440	6.510	11.300	6.510
13	1.870	8.000	2.540	8.000	12.900	5.900
14	2.260	8.030	2.650	7.640	14.600	8.030
15	2.360	7.640	2.900	8.030	15.000	8.000
Xsr-den	1.387	6.723	1.788	6.723	8.721	6.723

The correlation of arithmetic means of debonding force (I) and average adhesive Ra roughness

(Sample 1- ConTec LC – Dentaurum) is presented in Figure 4.

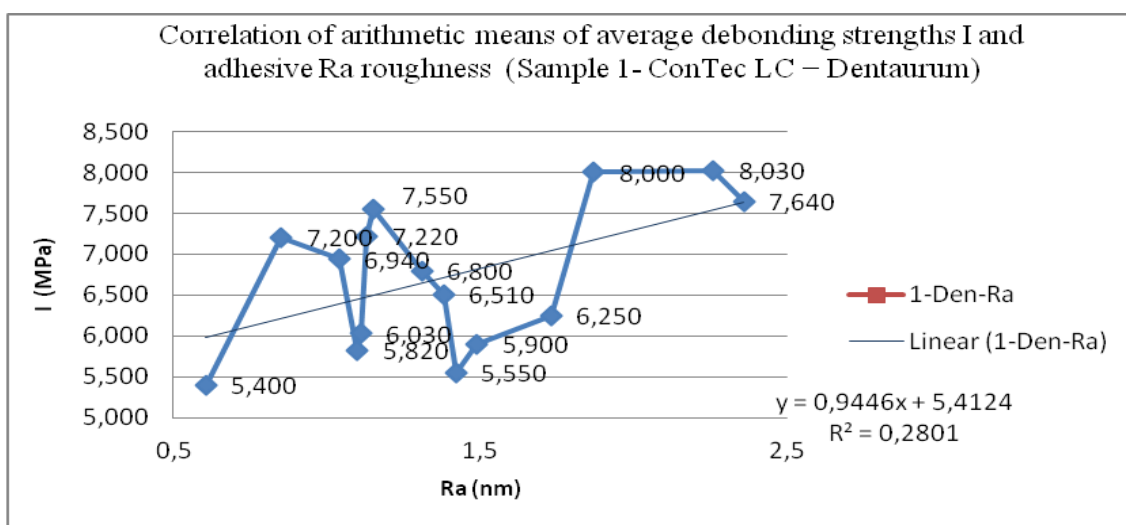


Figure 4. Correlation of arithmetic means of debonding strength (I) and average adhesive Ra roughness (Sample 1- ConTec LC – Dentaurum)

Correlation of arithmetic means of debonding strengths (I) and average adhesive roughness Rq

(Sample 1- ConTec LC – Dentaurum) is presented in Figure 5.

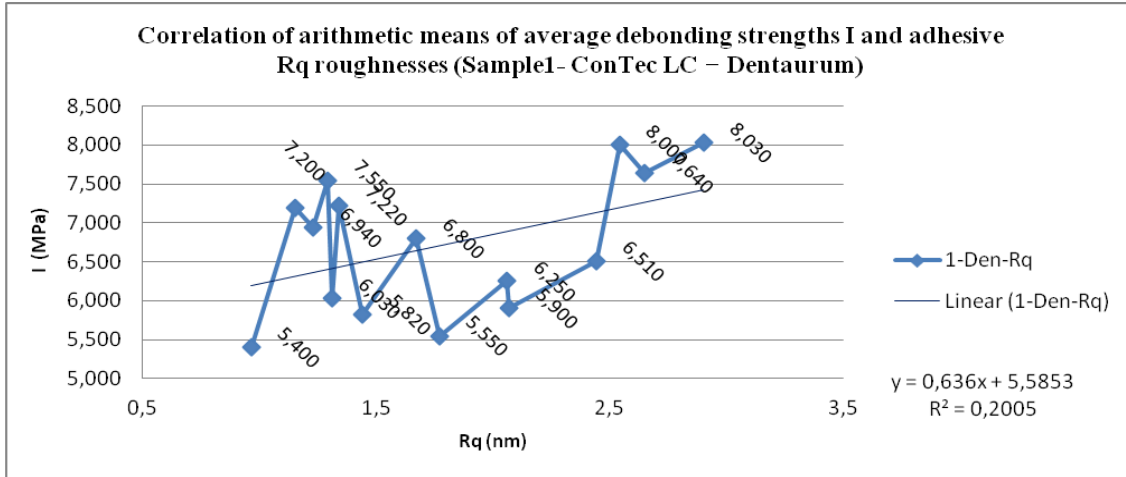


Figure 5. Correlation of arithmetic means of debonding strengths (I) and average roughness of adhesive Rq, (Sample 1 –ConTec LC – Dentaurum)

The correlation of arithmetic means of debonding strengths (I) and average adhesive roughness Rz

(Sample 1- ConTec LC – Dentaurum) is presented in Figure 12.

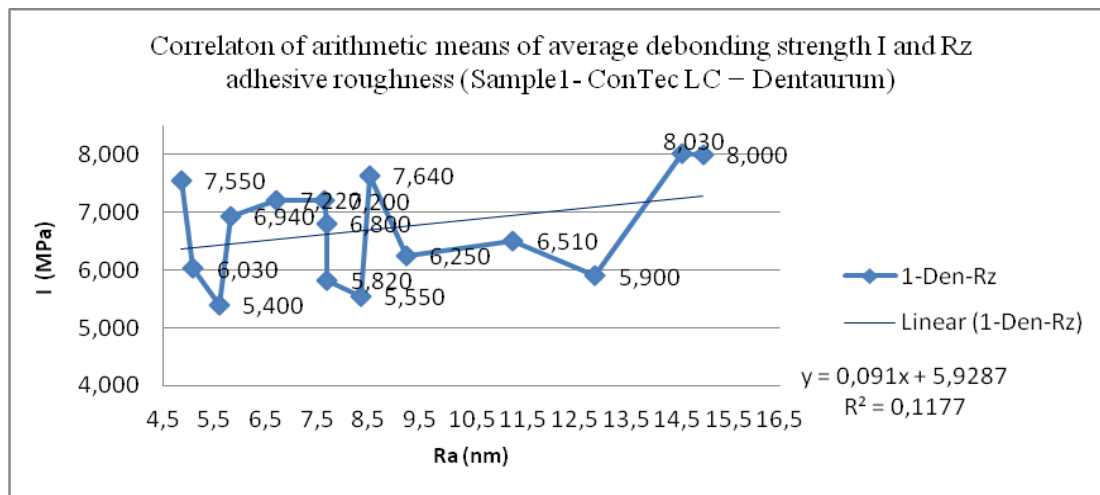


Figure 6. Correlation of arithmetic means of debonding strength (I) and average adhesive roughness Rz (Sample 1 –ConTec LC – Dentaurum)

Regression parameters of the analyzed (Sample 1 roughness (Ra, Rz, Rq) are presented in Table 9. –ConTec LC – Dentaurum) sample by adhesive

Table 9. Correlation parameters Sample 1 –ConTec LC – Dentaurum

Parameter designation	Regression equation (y=ax+b)	a	b	Determination coefficient (R ²)	Correlation coefficient (r)
1-Den-Ra	y = 0.9446x + 5.4124	0.9446	5.4124	0.2801	0.529245
1-Den-Rz	y = 0.091x + 5.9287	0.091	5.9287	0.1177	0.343074
1-Den-Rq	y = 0.636x + 5.5853	0.636	5.5853	0.2005	0.447772

4. DISCUSSION

Based on a detailed overview of large literature we determined that most authors agree that the strongest bond of considered adhesives: Sample 1-ConTec LC – Dentaurum, Sample 2 - GC Fuji Ortho LC, Sample 3 - Heliosit, Orthodontic (Ivoclar, Vivadent) and Sample 4 - Resilience Orthodontic bonding solutions, Ortho

Technology inc. Florida.) is realized by the adhesive Heliosit, Orthodontic (Ivoclar,Vivadent) [24-29,30-35]. After this obtained data we believed that the most purposeful-fastest way to get to a new adhesive that would fulfill the most important requirements sought from it, i.e. expected in practice, is to analyze its structure and all other physical-chemical characteristics in more detail, so that, based on that, focusing of further

research on improvement of that adhesive type can be recommended, while keeping its main elements of structure and chemical composition.

With the analysis of the results of obtained roughness by way of AFM, the most frequently are used, as follows: mean roughness (Ra), roughness by the least square method (Rq), and roughness that are determined as the biggest difference of heights (Rz). Roughness are expressed in nanometers (nm).

Based on Figures 9 through 12, by way of correlation of arithmetic means of debonding strengths (I) and average adhesive roughness (Ra, Rz and Rq) for sample 1 – ConTec LC – Dentaaurum, we can conclude that with an increase in the average adhesive roughness increases the debonding strength (I). The analysis that was conducted for bond strengths of other adhesives [37-41, 42-46] (Sample 2 – GC Fuji Ortho LC, Sample 3 – Heliosit, Orthodontic (Ivoclar, Vivadent) and Sample 4 – Resilience Orthodontic bonding solutions, Ortho Technology Inc. Florida.) also show the same dependence of debonding strength on roughness, i.e. with an increase in roughness increases the bond strength.

If we compared the obtained roughness results for individual materials with the obtained values in literature for bond strengths [25-29, 32-36] we could see that with all the roughness parameters (Ra, Rz and Rq) the

strongest bond was made with Resilience Orthodontic bonding solutions, followed by Heliosit, Orthodontic (Ivoclar, Vivadent), GC Fuji Ortho LC, and the weakest with ConTec LC – Dentaaurum. The differences between the last three adhesives are not so noticeable. On the other hand, the biggest material roughness was reported with GC Fuji Ortho LC, followed by Resilience Orthodontic bonding solutions, while with the other two, it is by far lower. If we take into account that GC Fuji Ortho LC is a material on the basis of glass ionomer and that it does not require etching the enamel with acid, we assume that its big roughness increases the total contact surface through which the chemical bond between the hydroxyl groups of polyacrylic acid with calcium ions in hydroxyapatite is realized. [47, 48].

On the other hand, higher roughness of Resilience Orthodontic bonding solutions at a nano level probably enables higher number of thorns that penetrate into microrecesses formed under the action of acids. Higher roughness is a consequence of the chemical structure itself of the composite material. [49, 50].

Correlations of arithmetic means of debonding strengths (I) and average roughness Ra for ConTec LC – Dentaaurum, GC Fuji Ortho LC, Heliosit, Orthodontic (Ivoclar, Vivadent) and Resilience Orthodontic bonding solutions are presented in Table 10.

Table 10. Correlation of arithmetic means of debonding strengths (I) and average adhesive Ra, Rq and Rz roughness for ConTec LC – Dentaaurum, GC Fuji Ortho LC, Heliosit, Orthodontic (Ivoclar, Vivadent) and Resilience Orthodontic bonding solutions

Type of adhesive	Ra [nm]	I [MPa]	Rq [nm]	I [mPa]	Rz[nm]	I [MPa]
ConTec LC – Dentaaurum	1.387	6.723	1.788	6.723	8.721	6.723
GC Fuji Ortho LC	55.930	7.035	64.847	7.035	212.433	7.035
Heliosit, Orthodontic (Ivoclar, Vivadent)	3.616	8.807	4.652	8.807	17.876	8.807
Resilience Orthodontic bonding solutions	23.696	10.600	27.394	10.600	93.420	10.600

More recent researches have shown that the adhesives based on glass-ionomer structure show the property of releasing the Fluor ions into deeper parts of tooth prisms and as such encourage its remineralization. This was not noted with clean composite adhesives. This fact may be of relevance in possible preventive action against the caries development too [51].

It is well known that the treatment with fixed dentures increases the risk of development of carious process which jeopardizes the treatment itself and discourages the patient. The risk related to the patient is crucial in that, while the additional factors such as the materials being applied can contribute even more. The placing of the fixed brackets disrupts the ecosystem in oral cavity which was proved to lead toward an increase in the number of cariogenic bacteria and the development of white spots [52].

5. CONCLUSION

Based on the obtained results of research, their statistical processing and detailed analysis, the following conclusions may be drawn:

- That with an increase in average roughness Ra, Rq, and Rz of adhesives, increases the debonding strength I.
- Higher roughness of Resilience Orthodontic bonding solutions at a nano level is probably enabled by a bigger number of thorns penetrating into micro cavities formed under the action of acids.
- Glass-ionomer adhesives have a satisfactory adhesive power and exert less aggression on enamel surface; they even have certain protective properties toward the bacteria, so that they can have an advantage in application, especially with the caries of risk patients or with hypo mineralized enamel.

– Adhesive power of glass-ionomer adhesives is based on a big contact surface (roughness) which provides a bigger number of chemical relations of COO groups of polyacrylic acid with calcium cations.

– After debonding the orthodontic brackets fixed with composite material by way of enamel etching, a long and complex treatment of enamel remineralization is necessary.

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Nanostruktura ortodontskih adheziva

Vladan D. Mirjanić, Đorđe D. Mirjanić

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Apstrakt

Nanostruktura ortodontskih adheziva za pričvršćivanje zubnih podupirača koji se najčešće koriste u današnjoj kliničkoj proceduri analizirana je pomoću mikroskopije atomskih sila (MAS). Studija je izvedena za 5 ortodontskih adheziva. Nakon određivanja osobina adheziva, utvrđena je korelacija između nanostrukture testiranog adheziva i čvrstoće interfejsa zubnog podupirača. Na osnovu MAS slika analiziranih adheziva i korelacijama aritmetičkih sredina čvrstoće vezivanja (I) i prosečne hrapavosti adheziva (Ra, Rk, Rz), zaključeno je da uz povećanje prosečne hrapavosti adheziva povećava čvrstoća vezivanja (I). Uočeno je da je sa svim parametrima hrapavosti (Ra, Rz, Rk) utvrđena najjača i najslabija veza. Veća hrapavost otpornosti ortodontskih veza na nano nivou verovatno omogućuje veći broj trnja koji prodiru u mikro konkave nastale pod uticajem kiselina. Viša hrapavost je posledica same hemijske strukture kompozitnog materijala.

Ključne reči: *Ortodontski adhezivi, nano-struktura, mikroskopija atomskih sila (MAS)*