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Original article

The Influence of Bracket Type and Etching Time on Shear Bond Strength to Enamel: An *In Vitro* Study

Vladimir Mitić[1,2], Ana Todorović[1,2], Aleksandar Mitić[1,3], Kosta Todorović[1,4], Milan Miljković[5]

[1]*University of Niš, Faculty of Medicine, Niš, Serbia*
[2]*Clinic of Dental Medicine, Department of Orthopedics of Jaws and Teeth, Niš, Serbia*
[3]*Clinic of Dental Medicine, Department of Dental Diseases and Endodontics, Niš, Serbia*
[4]*Clinic of Dental Medicine, Department of Oral Surgery, Niš, Serbia*
[5]*University of Niš, Faculty of Medicine, Research Centre for Biomedicine, Niš, Serbia*

SUMMARY

Introduction/Aim. The length of tooth enamel conditioning time and the size of bracket bases are some the factors influencing the bond strength between the two adherents. The aim of this study was to compare the shear bond strength (SBS) between two different types of bonded metal brackets and acid-etched enamel surface in two different times.

Material and methods. Forty extracted human premolars were randomly divided into four groups. In groups 1 and 2, metal brackets Topic (Dentaurum, Germany) were bonded after etching with 37% phosphoric acid (Gel, Reliance, USA) for 15 s and 30 s, while in groups 3 and 4, metal brackets Equilibrium mini (Dentaurum, Germany) were bonded after etching for 15 s and 30 s. Brackets were bonded using flowable composite Heliosit Orthodontic (Ivoclar Vivadent, Liechtenstein) and light-cured using a LED lamp. The SBS was measured by an electronic dynamometer at a cross-head speed of 5 mm/min. Results. Statistical analysis revealed that SBS is affected by both bracket type ($t = 10.62$; $p < 0.01$) and etching time ($t = 2.81$; $p = 0.008$). The Equilibrium mini brackets with previous enamel etching for 30 s showed the highest SBS values of 10.8 ± 0.75 MPa, while the Topic brackets with previous enamel etching for 15 s showed the lowest values of 4.85 ± 0.53 MPa.

Conclusion. Increasing the etching time of tooth enamel leads to higher values in bond strength, especially when using a bracket with a smaller base.

Keywords: **adhesion, dental materials, demineralization, separation force**

Corresponding author:
Vladimir Mitić
e-mail: mident@mts.rs

INTRODUCTION

The basic principle of adhesion lies in the application of an adequate acid in an optimal time action, where such an ultrastructure of the hardest human tissue will be formed for the penetration of primer with low viscosity that will lead to an adequate connection between two adherents. In this way, they should create such a connection that could withstand both masticatory forces and the forces that occur during therapy with fixed orthodontic appliances over a long period, as well as biodegradation of material that is closely related to saliva and bacteria in the oral environment (1).

For these purposes, it is almost unthinkable to conduct research on any material *in vitro* so that the same results of a laboratory study can later be reproduced in a clinical setting. Clinical and laboratory studies are equally important in evaluating the quality of the adhesion between teeth and orthodontic brackets. The main problem of laboratory research is the inability to simulate clinical conditions, which can significantly affect the reliability of the data obtained such as applied methods used in the preparation, storage and preparation of samples, applied force, location of applied force and conditions of applied tests (2 - 5). However, the bonding strength of *in vitro* studies is of appropriate value, as it may correlate with “acceptable” bonding strength for clinical use. The acceptable range of bond strength should be large enough so that careless debonding should not occur during therapy but also optimal enough that, when debonded for its purpose, it can be easily removed without damaging the enamel.

The specific structure of the enamel requires surface pretreatment with the appropriate concentration of acid in the optimal time of acid-etching on the previously planned tooth surface, which should be slightly larger than the selected sizes of bracket bases. The acid applied in adhesive dentistry leads to changes in the ultrastructural characteristics of the surface part of the enamel (6) which will be prepared for the next phase, i.e. penetration of the appropriate low viscosity adhesive primer. This would lead to the manifestation of optimal physical and chemical characteristics of adhesive materials in order to obtain adequate bond strength between the two adherents. This is possible owing to the aggressive application of acid on the tooth surface, which leads to the dissolution of hydroxyapatite in the form of cracks, depressions and canals, which further creates favorable conditions for the next phase of direct placement of brackets. In this way, there is a process of demineralization and dissolution of the structure of hydroxyapatite. The degree of decomposition will depend on several factors: tooth type or orientation of enamel prisms in relation to the surface plane of the tooth (which will depend on the localization of acid, cervical, middle or incisal third of the tooth) (7), the concentration of the applied acid and the time of exposure of the enamel to the action of the acid (8, 9).

Opinions are divided when it comes to the optimal time of tooth enamel etching when bonding brackets. Some authors (10, 11) suggest etching of tooth enamel in the time interval of 15 s per tooth. However, the existence of variations in enamel solubility not only in one patient but also in the same tooth depending on the location of the applied acid leads to microdetails in finding the best solution to such a complex challenge. According to other authors (12, 13), the enamel etching time of 30 s, which is the gold standard, is adequate for most of our patients.

Apart from the adhesives used, the size and design of the bracket bases have a significant role in the bonding of brackets to the teeth. The evolution of brackets has included modifications of bracket-base design to achieve satisfactory bond strength, with mechanical base adhesive and adhesive enamel retention, while facilitating debonding without damage to the enamel surface (14). The area of bracket bases is only one of the factors responsible for obtaining optimal bond strength, the selection of which should be done before the initial bonding. This will decide the size of the future demineralized zone, formed by the application of the appropriate acid on the surface of the tooth. The demineralized zone should be slightly larger than the base of the brackets. With controlled and careful acid-etching of the enamel, it is possible to achieve the appropriate size of the demineralized zone.

Successful bonding should be the objective of highest priority, while unexpected debonding is expensive and additional time is necessary to place the same or new brackets. That is the reason why the research related to the bracket bond strength, more adequate adhesive materials, and simplified procedures is a never-ending process (15, 16).

In this *in vitro* study, the shear bond strength of two different types of metal orthodontic brackets with prior enamel etching with 37% orthophosphoric acid in two different times during their bonding for buccal surfaces was compared. Finding the most favorable combination of the size of the bracket-bases and the time of the acid-etched enamel could significantly improve the bond strength between these two adherents.

MATERIAL AND METHODS

Sample preparation

This study was approved by the Ethics Committee of the Clinic for Dental Medicine in Niš, Serbia (14/7-2019-4 EO). The study was performed on 40 human premolars extracted for orthodontic reasons from patients aged 10 - 14 years. The criteria for the selection of teeth were: intact enamel surface which was not previously exposed to chemical agents, without caries and cracks due to the pressure of the pliers during extraction. All other teeth were excluded from the study. The preparation of biomaterials was performed by collecting and storing the teeth in 0.1% thymol solution for 6 months and rinsing them with a sterile physiological solution. The teeth were cleaned using pumice and dental polishers for 10 s.

Bonding technique

The process of bonding and applying orthodontic brackets to the enamel surface included conventional methods of bonding brackets for teeth, as follows: the buccal surface of each tooth was washed with saline, dried, tooth enamel was acid-etched with a 37% phosphoric acid gel (Gel Etching Agent, Reliance Orthodontic Products, Itasca, USA), the area where the brackets were to be bonded was washed, dried and flowable composite applications were applied (Heliosit Orthodontic, Vivadent Ivoclar, Liechteinstein). The study used forty metal orthodontic brackets for premolars, twenty Equilibrium mini and twenty Topic type with inch slot 0.022 (Dentaurum, GmbH&Co. KG, Inspringen, Germany). Equilibrium mini bracket with a quadrangular base shape is the main characteristic of these brackets which represent a smaller and more concave atomic base shape with an 8.7 mm^2 bracket base size for premolars with laser-structured bracket base. The Topic bracket is with laser-structured base and super-rounded edges with a bracket base size for premolars of 12.7 mm^2. The tested samples were divided into four groups of 10 samples proposed as a standard that requires a minimum in previous *in vitro* studies (17, 18), investigating shear bond strength as follows: in the first and second group of dental specimens (n = 10), metal orthodontic brackets of the Topic type were bonded, while in the third and fourth group, fitted Equilibrium mini brackets were placed. The enamel acid-etching of buccal tooth surfaces was performed using 37% phosphoric acid in the liquid for 15 s in the first and third group and 30 s in the second and fourth group. After washing and drying, the appropriate amount of Heliosit was applied to the base of brackets and placed on the previously acid-etched surfaces of the teeth. Six-point positioning and pressure were performed to squeeze out excess adhesive under the bracket bases. Any excess around the brackets was removed with a sharp probe. After placing the brackets, their polymerization was performed with a LED lamp Woodpecker Dental Curing Light (LED B. Curing Light, Guangxi, China) with light intensity of 1200 - 1400 mW/cm^2, optical wavelength 420 - 480 nm; voltage of 3.7 V; 1500 mAh, each tooth for 40 s; 10 s on the occlusal, gingival, mesial and distal sides. All brackets were bonded by one operator (VM). Storage of dental material was performed at a standardized time of 24 h in an airtight chamber and humid environment (GFL, model Pt 100, England) to prevent dehydration (100% humidity at 37 ± 1^0 C).

Bracket debonding

The strength required to separate the brackets from the tooth surfaces was measured by fixing the specimens using the upper and lower pair of terminals in an electronic dynamometer HBM (Wagezelle Load Cell, Hottinger BaldwinMesstechnik, Germany). The dental specimens were covered with a lower pair of clamps coated with rubber up to the enamel-cement junction. The traction load was achieved by 0.9 mm thick wire in the area of the lower pair of wings on the brackets, at a constant speed of 5 mm/min. The direction of the force was gingival-occlusal. The device automatically recorded the force with an accuracy of 0.1 N. The individual value of the obtained force was divided by the total area of the bracket base (expressed in mm^2), which represents the size of the contact surfaces. In this way, all values are expressed in N/mm^2, i.e. in megapascals (MPa).

Statistical analysis

The difference in the shear bond strength (SBS) in the two types of brackets was tested by independent samples t-test at the level of $p < 0.05$. The same test was used to assess differences in the strength of the debonding at different etching times. The total combined effect of both factors was analyzed by regression analysis with dummy variables. IBM SPSS Statistics v.22 statistical software was used.

RESULTS

The total average bond strength for all observed cases (regardless of etching time and bracket type) is 7.5 MPa. This bond strength varies considerably (SD = 2.37) depending on the bracket type and etching time.

The SBS required to debond the tooth brackets in relation to the type of brackets used is shown in Table 1. The SBS required to debond tooth bracket is higher with Equilibrium mini brackets than with Topic brackets (Figure 1). The mean difference in SBS of 4.04 MPa between the Equilibrium mini and Topic brackets is statistically significant ($t = 10.62$; $p < 0{,}001$) at the level of $p < 0.05$ (95% confidence interval ranges from 3.26 to 4.82). The SBS is also affected by the enamel etching time. Higher SBS was obtained after enamel etching for 30 s compared to 15 s (Figure 2). The mean difference in SBS of 1.94 MPa between the 30 s and 15 s etching time is statistically significant ($t = 2.81$; $p = 0.008$) at the level of $p < 0.05$ (95% confidence interval ranges from 0.53 to 3.34).

Table 1. *Descriptive statistics of SBS (MPa) for two bracket types in two etching times*

Bracket type	Etching time (s)	Mean±SD (MPa)	N*
Equilibrium mini	15	8.2027±0.49269	10
	30	10.8300±0.75137	10
	Total	9.5164±1.48286	20
Topic	15	4.8515±0.53427	10
	30	6.0988±0.56978	10
	Total	5.4751±0.83573	20
Total	15	6.5271±1.79045	20
	30	8.4644±2.51231	20
	Total	7.4958	40

*number of brackets

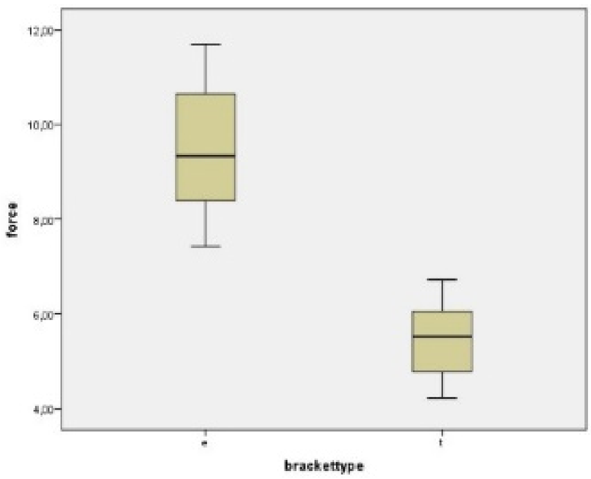

Figure 1. *SBS in relation to bracket type*

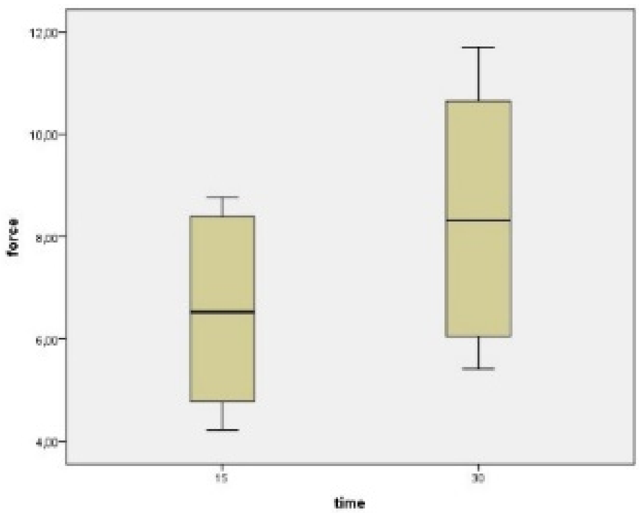

Figure 2. *SBS in relation to etching time*

In addition to the main effects, a small but statistically significant interaction effect of bracket type and etching time was registered. An increase in bond strength when debonding tooth brackets with the enamel etching time extending from 15 s to 30 s is greater with an Equilibrium mini bracket than with a Topic bracket (Figure 3).

The regression model in which the dependent variable is the SBS and the predictor variables are the bracket type and acid-etching time (variables with values of the bracket type topic = 1, otherwise 0, and the etching 15 s = 1, otherwise 0) explains almost 94% of the variance of the dependent variable (R2 = 0.941). All coefficients of the regression equation are statistically significant at $p < 0.5$ (Table 2).

Of the total sum of squares 218.364, the model explains 205.607 or 94.16%. F statistic is statistically significant at the level of $p < 0.5$ (Table 3).

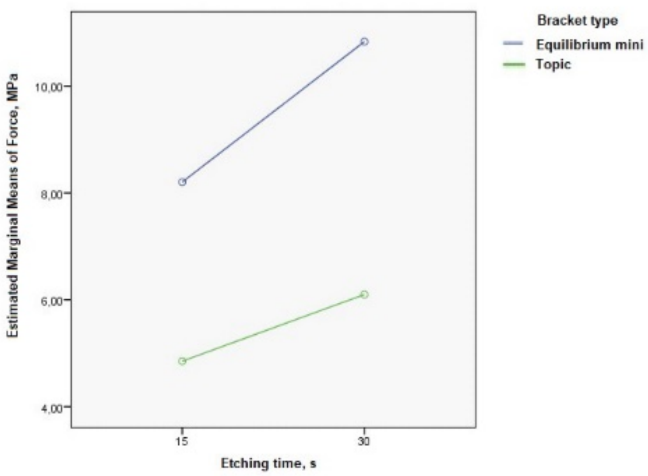

Figure 3. *Interaction effect of bracket type and etching time*

Table 2. *Regression analysis coefficients*

Source	Type III Sum of Squares	df*	Mean Square	F	Sig	Partial Eta Squared
Corrected model	205.607[a]	3	68.536	193.413	0.000	0.942
Intercept	2247.453	1	2247.453	6342.472	0.000	0.994
Bracket type	163.315	1	163.315	460.886	0.000	0.928
time	37.532	1	37.532	105.919	0.000	0.746
Bracket type* Etching time	4.760	1	4.760	13.434	0.001	0.272
Error	12.757	36	0.354			
Total	2465.816	40				
Corrected total	218.364	39				

*degree of freedom

Table 3. *ANOVA test results*

Model	Sum of squares	df*	Mean of squares	F statistic	Sig
Regression	205.607	3	68.536	193.413	0.000
Residual	12.757	36	0.354		
Total	218.364	39			

*degree of freedom

DISCUSSION

The most adequate preparation of the tooth surface for orthodontics is considered to be the use of 35%- 37% phosphoric acid, which creates an appropriate microstructural surface of tooth enamel for later optimal penetration of adhesives to obtain the best possible connection between the two adherents.

In the laboratory, Wang et al. (19) presented results related to the bond strength using six orthodontic brackets of different sizes with values of 4.32 - 9.32 MPa and concluded that higher bond strength values were more present when bonding brackets with larger bases, which is contrary to the results of our study. According to Griffith's defect theory (20), when uniform materials are tested, the strength of the material decreases with increasing the surface area, which is confirmed by the results of Sano (21), who discovered that the value of bonded materials is inversely proportional to bonded joints. The results of the study by Sano are in keeping with the results of our study. This suggests that although the bracket base area may influence bond strength, the size of bracket base design may have an important influence over adhesion to the enamel. We believe that an important factor that plays a role in obtaining the best possible connection between the two adherents is the size of adaptive surface of the concave bases of the brackets for premolars, which means that the smaller concave base of the brackets achieves better adaptability to the convex buccal surface of the teeth of posterior region. This detail was also pointed out by the results of Molina (22), Pham (23) and De Mela (24) who concluded that the concave base of the brackets as well as their different geometries are important in more adequate adhesion of the adhesive material and in obtaining optimal bond strength. The higher values obtained in this study unequivocally indicate that when placing brackets in the posterior zone, brackets with a smaller base area are of greater importance, which achieves a more optimal and closer relationship with the convex buccal surface of posterior teeth.

Silva et al. (25) in their study concluded that there was no increase in strength adhesion with prolonged tooth enamel etching. Our study showed higher values of bond strength using phosphoric acid over a longer time interval of 30 s, noting that in group 3 the highest bond strength was obtained with prior enamel acid-etching for 15 s using smaller brackets bases, while the lowest bond strength was obtained in group 1.

In their study of Heliosit Orthodontic, Yousry and Abdel-Haffiez (26) reached a value of 10.4 ± 2.8 MPa 24 h after bracket placement, using Gemini-type metal brackets with an average premolar base size of 9.82 mm^2. The values of our study correlated with the previously mentioned study using the same time of acid-etching enamel and a smaller bracket base, while the values in the remaining three groups showed lower values.

The results of *in vitro* studies should be applied in clinical conditions and shorter acid-etching time of the enamel reduces the clinical time of bonding (27). If the acid-etching time of tooth enamel had been shortened, it would have positive effects on lower tooth demineralization but not by reducing the bond strength of the two adherents. Obtaining a quality ultrastructural morphology of human teeth enamel will give better opportunities for penetration of adhesive materials (28, 29) because the strength of the shear bond between enamel and bracket bases is only one of the components that affects the final outcome in clinical conditions. In addition, the bonding area size (contact of the brackets' bases with the demineralized zone) is also a significant factor in obtaining an optimal bond strength between the two adherents. If the difference in the size of these two factors is greater, it will represent a predilection site for the penetration of microorganisms in clinical conditions but also a possible factor in the reduced strength of the bond. In addition to the concentration, form and optimal time of acid-etching, attention should be paid to its precise application to the tooth surface, which will affect the controlled size of the bonding area.

The use of a primer is an essential part of the bonding process of adhesives which allows good wetting and penetration into the demineralized part of the acid-etched enamel. Using a primer in combination with more consistent adhesives will create certain thickness between the two adherents. The surface created in this way will resist any pressure. A smaller thickness of the adhesive between the adherents will give a stronger bond due to: better penetration of the adhesive into the base of the brackets, intimate contact between the two adherents, less possibility of creating voids and cracks, less polymerization shrinkage in smaller areas of the adhesive and quality polymerization, which according to Mohamadi (30) depends on the consistency and viscosity of dental materials. Based on this information, we decided to use Heliosit Orthodontic flowable composite, whose properties according to the manufacturer's recommendation are based on application without a prior use of a primer. Specific composition of this dental material makes it significantly more viscous compared to other adhesives with higher degree of consistency and enables injectability and non-stickiness. If the bond strength using these materials shows clinical acceptability, then they could gain an advantage over orthodontic adhesives due to the reduction in bonding time.

We would also like to share the observation that the placement of dental specimens in acrylic blocks leads to a rigid connection, which we avoided in this study, because it would take us away from the simulated clinical condition that exists in the oral environment where the teeth are not in a rigid connection with the alveolar bone but are connected in the upper and lower jaw via periodontal fibers - a kind of natural shock absorbers. This experimental study encourages us to take the obtained results with a certain amount of caution.

CONCLUSION

Based on the obtained results, the following conclusions were drawn in this study:

- Acid-etching enamel for 30 s gives greater bond strengths in both groups of the tested metal orthodontic brackets;
- The highest bond strength was obtained by bonding smaller brackets (Equilibrium mini) with acid-etching the enamel for 30 s;
- Optimal bond strength can be achieved by applying a shorter enamel etching time with the use of a smaller bracket base.

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Uticaj tipa bravica i vremena kondicioniranja na jačinu veze sa zubnom gleđi: *in vitro* studija

Vladimir Mitić[1,2], Ana Todorović[1,2], Aleksandar Mitić[1,3], Kosta Todorović[1,4], Milan Miljković[5]

[1]*Univerzitet u Nišu, Medicinski fakultet, Niš, Srbija*
[2]*Klinika za dentalnu medicinu, Služba za ortopediju vilica, Niš, Srbija*
[3]*Klinika za dentalnu medicinu, Služba za bolesti zuba i endodonciju, Niš, Srbija*
[4]*Klinika za dentalnu medicinu, Služba za oralnu hirurgiju, Niš, Srbija*
[5]*Univerzitet u Nišu, Medicinski fakultet, Naučnoistraživački centar za biomedicinu, Niš, Srbija*

SAŽETAK

Uvod/Cilj. Dužina vremena kondicioniranja gleđi zuba i veličina baza bravica faktori su koji utiču na jačinu veze između dva adherenta. Cilj ovog istraživanja bio je da se uporedi jačina veze između dva različita tipa metalnih bravica vezanih za površinu gleđi, koja je bila nagrizana kiselinom u dva različita vremenska intervala.
Materijali i metode. Četrdeset ekstrahovanih ljudskih pretkutnjaka nasumično je podeljeno u četiri grupe. U grupi 1 i grupi 2 metalne bravice *Topic* (Dentaurum, Nemačka) aplikovane su nakon nagrizanja 37% fosfornom kiselinom (Gel, Reliance, USA) koje je trajalo 15 s i 30 s. U grupi 3 i grupi 4 metalne bravice *Equilibrium mini* (Dentaurum, Nemačka) aplikovane su nakon nagrizanja istog trajanja (15 s i 30 s). Bravice su zalepljene korišćenjem tečnog kompozita *Heliosit Orthodontic* (Ivoclar Vivadent, Lihtenštajn) i svetlosno polimerizovane korišćenjem LED lampe. Jačina adhezivne veze merena je elektronskim dinamometrom pri brzini poprečne glave koja je iznosila 5 mm/min.
Rezultati. Statistička analiza pokazala je da na jačinu adhezivne veze utiču i tip bravice (t = 10,62; p < 0,01) i vreme nagrizanja (t = 2,81; p = 0,008). *Equilibrium mini* bravice, sa prethodnim nagrizanjem gleđi u trajanju od 30 s, pokazale su najveće vrednosti (10,8 ± 0,75 MPa), a Topic bravice, sa prethodnim nagrizanjem gleđi od 15 s, najniže vrednosti (4,85 ± 0,53 MPa).
Zaključak. Produženo vreme nagrizanja zubne gleđi rezultira većom jačinom adhezivne veze, posebno kada se koristi bravica sa manjom bazom.

Ključne reči: adhezija, dentalni materijali, demineralizacija, sila razdvajanja