

# IMPACT OF DIFFERENT ACID CONCENTRATIONS ON BOND STRENGTH OF METALLIC BRACKETS AND ENAMEL DAMAGE - an in vitro study

## ПРОЦЕНА НА ВЛИЈАНИЕТО НА РАЗЛИЧНИ КОНЦЕНТРАЦИИ НА СРЕДСТВО ЗА НАГРИЗУВАЊЕ НА ЕМАЈЛОТ ВРЗ ЈАЧИНАТА НА ВРСКАТА ПОМЕЃУ ЕМАЈЛОТ И МЕТАЛНИТЕ БРЕКЕТИ И ОШТЕТУВАЊЕ НА ЕМАЈЛОТ - ин витро студија

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### Abstract

**Introduction:** Enamel etching is an important and necessary phase of bonding protocol. Different etching duration and different acid concentration can be used to prepare the tooth surface for bonding. Generally, the longer the etching duration and the higher the acid concentration, the stronger the bond strength between bracket and tooth surface. However recent studies are focused on lowering the etch concentration during bonding of brackets. **Aim of the study:** To evaluate the enamel damage caused by different acid concentrations during enamel etching; to determine the impact of acid concentration during enamel etching on the bond strength of orthodontic brackets; to determine the etch concentration which will achieve the optimal bond strength while minimizing enamel damage. **Material and methods:** The material for our In Vitro study consisted of 30 intact premolar teeth, extracted for orthodontic reasons. The teeth were categorized in three groups. Each group of teeth was etched with different acid concentration (3.7%, 18.5% and 37%). Subsequently, shear bond strength of orthodontic brackets was tested in Universal Testing Machine. **Results and discussion:** The lowest mean bond strength (7.59 MPa) was recorded in the teeth etched with 3.7% phosphoric acid. While the highest mean SBS was found in the group of teeth etched with 37% phosphoric acid (10.35). The group of teeth etched with 18.5% phosphoric acid represented intermediate SBS value, which was 9.16 MPa. Teeth etched with 37% phosphoric acid showed higher adhesion at the enamel/resin interface, while teeth etched with 3.7% phosphoric acid had higher adhesion at the resin/bracket interface. Findings for the group of teeth etched with 18.5% phosphoric acid were in between. **Conclusions:** We consider that lower concentrations of acid should be used during bracket bonding.

### Апстракт

**Вовед:** Нагризувањето на емајлот на забот е важна и неопходна фаза во протоколот на поставување на фиксниот ортодонтски апарат. За подготовка на емајловата површина и врзување на брекетот за забот може да се користи различното време на нагризување и различната концентрација на киселина. Општо земено, колку е подолго времето на нагризување и поголема концентрацијата на киселина, толку е појака врска меѓу брекетот и емајлот на забот. Сепак, неодамнешните студии се фокусираат на намалување на концентрацијата на киселината за нагризување. **Цел на студијата:** да се процени штетата предизвикана од различни концентрации на киселина на емајлот, да се утврди влијанието на концентрацијата на киселината врз јачината на врска меѓу забот и брекетот, да се одреди концентрацијата на киселината со која би се постигнала оптимална јачина на врска со минимално оштетување на емајлот. **Материјал и метод:** Материјалот за оваа Ин Витро студија го сочинуваат 30 здрави премолари (екстрахирани од ортодонтски причини, во цел на екстракциона ортодонска терапија), поделени во 3 групи. Кај секоја група беше извршено нагризување со три различни концентрации на киселина (3,7%, 18,5% и 37%). Последователно, јачината на врзување беше тестирана во машината за универзално испитување (Universal Testing Machine). **Резултати и дискусија:** Најниска средна јачина на врска (7,59 МПа) регистриравме кај забите нагризувани со 3,7% фосфорна киселина, додека највисока (најака) просечна јачина на врска е регистрирана кај групата заби кои беа нагризувани со 37% фосфорна киселина (10,35 МПа). Групата заби нагризувани со 18,5% фосфорна киселина покажа средни вредности на јачина на врска (9,16 МПа). **Заклучоци:** Од добиените резултати изведовме заклучок дека е поцелисходно да се користат пониски концентрации на киселина за нагризување при поставувањето на фиксниот ортодонтски апарат во секојдневната пракса.

### Introduction

Enamel etching is an important and necessary phase of bonding protocol. Different time of etching and dif-

ferent acid concentration can be used to prepare the tooth surface for bonding. Generally, the longer is the etching time and the higher is acid concentration, the stronger is the bond strength between bracket and tooth surface. But

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not necessarily. For example, some studies demonstrate poorer adhesion when enamel is over etched. Especially, etching time beyond 90 seconds resulted in significantly lower bond strengths than all other etching times<sup>1</sup>.

The idea of etching enamel surfaces with orthophosphoric acid was first introduced by Buonocore in 1955. He used 85% orthophosphoric acid for 30 seconds and discovered that the bond strength of acrylic restorative resins was significantly increased by etching of the enamel surface<sup>2</sup>.

However, at that time it was not intended to be used for bonding orthodontic brackets. It was Dr. George Newman who introduced the idea of bonding different plastic attachments to the tooth surface, hence initiating a very important transition in orthodontic fixed treatment, transition from banding of the teeth to direct bonding of brackets.

Bracket failure is a common problem during fixed orthodontic treatment. It is annoying for an orthodontist and can prolong and complicate the treatment plan. Therefore, creating a good bond strength is necessary for a successful fixed orthodontic treatment.

We also have to mention that many studies report that there is a significant difference of shear bond strength of brackets bonded to enamel comparing to brackets bonded to teeth with cavities that have been filled with different composites<sup>3</sup>.

However, enamel etching can be harmful for enamel surface. Different acid concentration can have a different impact on enamel surface. The best choice would be to achieve the best bond strength with lowest acid concentration.

Iatrogenic effects of etching were listed as below<sup>4</sup>:

- Fracture and cracking of enamel upon debonding
- Increased surface porosity – possible staining
- Loss of acquired fluoride in outer of 10mm of enamel surface
- Loss of enamel during etching
- Resin tags retained in enamel – causing discoloration of resin
- Rougher surface if over etched

Considering all those risk factors, some studies were conducted in order to evaluate the bond strength of orthodontic brackets without enamel etching. The conclusions of those studies were astonishing suggesting that the application of filled adhesive without acid etch not only provides sufficient bond strength for bracket bonding, but also results in minimum resin remnants<sup>5</sup>. However, it was remarked by the researchers of the study itself that the same has limitations and that those results may not yet be applicable in general.

Another reason to reduce the acid concentrations used during orthodontic etching is the problems that acid

can cause on oral mucosa. It was reported that inadequate rinsing of dental acid etchants or remaining dental acid etchants can cause problems, including chemical burning, irritation and inflammation, intra and extra orally<sup>6</sup>.

Etching enamel surfaces with phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) is an accepted and widely applied technique to improve bonding of dental resins to enamel in restorative dentistry, in preventive dentistry and for direct bonding of orthodontic attachments<sup>7</sup>.

The depth of etch and the amount of enamel surface loss depends on many factors, such as: the type of acid used, the concentration of acid, etching duration and the chemical composition of the enamel<sup>8</sup>. According to Silverstone et al.<sup>9</sup>, three types of etching patterns can be revealed when examining with scanning electron microscopy after acid etching:

Type 1: Generalized roughening of the enamel surface, with preferential dissolution of the prism centers, resulting in a “honeycomb” appearance.

Type 2: preferential dissolution of prism peripheries resulting in a “cobblestone” appearance.

Type 3: Combination of type 1 and type 2 patterns (some regions resembling hollowed prism centers adjacent to areas where the prism peripheries appeared to be removed)

Galil and Wright<sup>10</sup> described two more types of etch patterns located in the cervical third of the buccal surfaces of the teeth:

Type 4: pitted enamel surfaces, as well as structures, which look like unfinished puzzles, maps or network.

Type 5: flat, smooth surface

A review of the literature concerning the relationship between the type of etching pattern and bond strength seems to indicate that regular and distinct type 1 and type 2 patterns provide maximum adhesion<sup>11</sup>.

Buonocore used phosphoric acid to obtain as effective an adhesion on enamel surface as on metal surfaces. The concentration of the first phosphoric acid solution used by Buonocore was 85%, and it was applied for 30 seconds<sup>2</sup>. Buonocore adhered acrylic materials on the non-etched teeth surfaces and etched surfaces. Although acrylic materials adhered on the etched surfaces were bonded with enough strength that they needed debonding procedures, failures were observed on non-etched surfaces<sup>12</sup>. This technique was an important advance in directly bonding orthodontic attachments to the tooth surface by means of micro-retention. However, honeycomb structures were not obtained in enamel prisms after etching with 85% phosphoric acid, and successful results in terms of retention were not achieved.

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Many researchers reported that a 35-38% concentration of phosphoric acid is effective in terms of optimum bond strength; however, 5-10% concentration changes did not have negative effects on bond strength<sup>13,14,15,16,17</sup>.

Today, 35-38% orthophosphoric acid is effectively used to change enamel surface characteristics and to provide micro mechanic bond strength.

Etching procedures with phosphoric acid differ in terms of microtopographic etching patterns over enamel surfaces<sup>18,19</sup>. The intended etching pattern was only observed in 1/20 of enamel etched with phosphoric acid. This was attributed to the presence of aprismatic enamel and partial contact between phosphoric acid and the enamel surface<sup>20</sup>. Microtopographic evaluation, depending on the etching procedure, revealed a non-uniform depth. It was reported that a depth of 3-15 µm or more is necessary to provide optimum shear bond strength and penetration<sup>21</sup>. However, in literature, surface depths between 10 µm and 175 µm were presented. The difference in the depth is thought to be caused by aprismatic enamel and remineralization of Ca-P to enamel surface<sup>21,22</sup>.

The concentrations of phosphoric acid recommended for clinical use in dentistry range from 30% to 60%<sup>7</sup>. However, many studies report that the higher the concentration - the bigger the enamel surface loss. Consequently, concerning the acid concentrations, it is preferable to use low concentrations causing minimal loss of enamel while securing an adequate bond.

The dilemma of the high bond strength of brackets, which is achieved after etching an enamel with 37% acid was also reported, since debonding these brackets often leads to enamel cracks and fractures<sup>23</sup>.

Lately, safer enamel etching and bracket debonding is being promoted in order to enhance enamel resistance to demineralization during orthodontic treatment.

## Aim of the study

Enamel loss and bracket failure are one of the essential complications during the fixed orthodontic treatment.

Hence, the aims of our study were:

1. To evaluate the enamel damage caused by different acid concentrations during enamel etching
2. To determine the impact of acid concentration during enamel etching on the bond strength of orthodontic brackets
3. To determine the etch concentration which will achieve the optimal bond strength while minimizing enamel damage

## Material and methods

The material for our In Vitro study consisted of 30 intact premolar teeth, extracted for orthodontic purposes. The criteria for tooth selection were as follows: no caries, no enamel cracks or fractures, or any kind of enamel defect. The extracted teeth were collected at the University Clinical Dental Center of Kosova, Department of Oral Surgery and Dental Office "Donident" in Prishtina.

Until the beginning of the research, the teeth were stored in 0.9% NaCl solution.

The teeth were mounted in self-cured acrylic resin and were randomly categorized/divided in 3 groups, each group containing 10 teeth.

First group of teeth was etched with 3.7 percent phosphoric acid.

Second group of teeth was etched with 18.5 percent phosphoric acid.

Third group of teeth was etched with 37 percent phosphoric acid.

Etching duration for all groups was 30 seconds. Dentaurem ConTec Go 37% phosphoric acid etching gel was used for etching the third group of teeth, while for the first and second group of teeth, 37% acid was diluted with distilled water.

All etched teeth were washed by air water spray for 15 seconds, and dried with oil free air syringe.

Brackets were bonded on the labial surface of the teeth according to general rules of bracket placement. One component "no mix" bracket adhesive in syringes will be used. Orthodontic bonding system Dentaurem ConTec Go adhesive was used. The type of brackets used for this study was Dentaurem "discovery" brackets, System Roth 22.

The samples were tested for shear bond strength (SBS) with universal testing machine. Shear bond strength values were expressed in MPa. The de-bonded teeth were examined under 10x magnification microscope for the amount of remnant left and were scored according to Adhesive Remnant Index (ARI). The tests were performed at the Faculty of Mechanical Engineering – University of Prishtina.

## Results and discussion

Table 1 contains calculated bond strength for each tooth, ARI score of each tooth, the mean bond strength for specific group of teeth and the ranges for each group. The lowest mean bond strength (7.59 MPa) was recorded among teeth etched with 3.7% phosphoric acid. While the highest mean SBS was found in the group of teeth etched

with 37% phosphoric acid (10.35). The group of teeth etched with 18.5% phosphoric acid showed intermediate SBS value, which was 9.16 MPa.

Based on the results of some studies, the clinically acceptable range of shear bond strength for bonding of orthodontic brackets is 5.9-7.8 MPa<sup>24</sup>. According to these results, two groups of teeth in our study, etched with 18.5 and 37% phosphoric acid showed sufficient SBS. Regarding the group of teeth etched with 3.7% phosphoric acid, the mean SBS was satisfying, however few teeth among that group had lower SBS than the one considered as clinically acceptable value in the literature.

Similar with our study, it is the goal of many researchers to study the bond strength of orthodontic attachments when using lower concentration of acid in order to minimize enamel damage. Thus, it was reported that a 25% of phosphoric acid concentration with 60 sec etching duration marked sufficient bond strength<sup>25</sup>.

Furthermore, some studies suggest that even 15% of phosphoric acid, in both 5 and 15 second duration, create strong enough bond between brackets and tooth surface<sup>26</sup>.

The effect of variations in acid concentration (5% and 37% H3PO4) and duration of etching (15 and 60 seconds)

on the shear bond strength of an orthodontic bonding system to etched enamel was studied and it was reported that the shear bond strength was not significantly different<sup>27</sup>.

Many studies suggest that the concentration of acid can be reduced clinically without having an adverse effect on the retention of bonded brackets<sup>28</sup>. A certain study found out that reducing the phosphoric acid concentration from 37% to 15% and applying it for 60 seconds had no significant increase in the failure of bonded attachments<sup>16,29</sup>.

However, whenever we discuss acid concentration regarding a shear bond strength, we have to take into consideration that, lately, orthodontic treatments have been more prevalent among adults. This has been emphasised because in such situations brackets and different fixed orthodontic attachments often are to be bonded to different materials, like metals, ceramics or composites. Many studies report that there is a significant difference of shear bond strength of brackets bonded to enamel compared with brackets bonded to teeth with cavities that have been filled with different composites<sup>3</sup>.

The ARI scores of teeth included in our study were in correlation with the acid concentration used during etch-

**Table 1.** Shear bond strength between bracket base and enamel in groups with different acid concentration (3.7%, 18.5%, 37%) and ARI sores for each tooth

Tooth	3.7%		18.5%		37%	
	MPa	ARI	MPa	ARI	MPa	ARI
1	8.0	1	9.8	2	10.2	2
2	6.7	0	8.8	1	9.4	1
3	7.3	0	8.9	1	9.7	1
4	9.0	1	10.4	3	9.9	1
5	5.2	0	10.1	2	11.0	3
6	6.4	0	9.9	2	11.7	3
7	7.7	0	8.4	0	11.2	2
8	8.7	1	8.7	1	9.9	2
9	9.0	1	8.6	1	9.7	2
10	7.9	0	9.0	2	10.8	2
X	7.59		9.16		10.35	
Range	5.2-9.0		8.4-10.4		9.4-11.7	

ing. Hence, the highest amount of adhesive left on the teeth was recorded in the group etched with 37% phosphoric acid. While decreasing the etch percentage resulted with lower ARI scores, these results correspond with literature findings<sup>20</sup>.

Table 2 represents adhesive remnant index among groups of teeth etched with different acid concentrations. According to the results of our study, the highest ARI scores were found among teeth etched with higher concentration of acid. This means that teeth etched with 37% phosphoric acid showed higher adhesion at the enamel/resin interface, while teeth etched with 3.7% phosphoric acid had higher adhesion at the resin/bracket interface. Findings for the group of teeth etched with 18.5% phosphoric acid were in-between. Similar conclusions regarding the correlation between the etch concentration and ARI index were reported in literature. Hence, Niaki<sup>30</sup> reports that when applying 15% phosphoric acid, more than 50% of the resin remained on the tooth surface, while all the resin remained on the tooth surface when applying 37% phosphoric acid.

**Table 2.** Adhesive remnant index (ARI) scores in groups according to different acid concentration (3.7%, 18.5%, 37%)

ARI	3.7%	18.5%	37%	Total
0	6	1	0	7
1	4	4	3	11
2	0	4	5	9
3	0	1	2	3

Another finding that has been reported when comparing phosphoric acid concentrations was that etching with 37% acid resulted in higher amounts of adhesive left on the teeth than when etching with 2% acid (according to ARI scores)<sup>16</sup>. These findings are also in line with the results of our study regarding the ARI index, and they draw the conclusion that when using 37% acid, the bond strength between the enamel and the resin is often higher than the one between the resin and the bracket. Alternatively, after etching with 2% acid the adhesion between the enamel and the resin appears to be lower than the adhesion between the resin and the bracket. This is particularly important when using the ceramic bracket, since it has been reported that the bond strength of the first ceramic brackets was very high compared to metal brackets<sup>31</sup>.

## Conclusion

- Lower concentrations of acid during etching provided sufficient shear bond strength of orthodontic brackets
- The highest ARI scores were found in teeth etched with higher concentration of acid

We believe that further clinical studies should be conducted regarding this issue.

## Reference

1. Peterson B.E., Callegari B.J., Mihalik C.A., Marsh C.M., Dunn W.J. (2016): Journal of the World Federation of Orthodontists, Vol. 5, 122-25
2. Buonocore M.G. (1955): A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces, in: J Dent Res, Vol. 38, 849-53
3. Seyhan Cezairli N., Kuçukekenci A.S., Basoglu H. (2019): Evaluation of Shear Bond Strength Between Orthodontic Brackets and Three Aged Bulk Fill Composites, in: ODOVTOS-Int J Dental Sc, Vol 21, No. 3, 89-99
4. Kanniyappan P., Kishore Kumar S., Manjula W.S. (2015): Enamel Pretreatment Before Bonding Brackets – A literature Review, in: Biomed & Pharmacol J, Vol. 8(Spl. Edn.), 631-40
5. Boruziniat A., Khazaei Y., Motaghi S., Moghaddas M. (2015): Evaluation of bond strength of orthodontic brackets without enamel etching, in: J Clin Exp Dent, Vol 7, No. 4, 519-23
6. Do-kyeong K., Jae-won K., Ryeong-mi J., Da-som J., Da-young Y., Na-yeon O., Ji-hye J. (2019): Effects of dental acid etchants in oral epithelial cells, in: Oral Biol Res, Vol. 43, No. 4, 299-305
7. Gottlieb E.W., Retief D.H., Jamison H.C. (1982): An optimal concentration of phosphoric acid as an etching agent. Part I: Tensile bond strength studies, in: J Prosthet Dent, Vol. 48, No. 1, 48-51
8. Retief D.H., Harris B.E., Bradley E.L., Denys F.R. (1985): Pyruvic acid as an etching agent in clinical dentistry, in: J Biomed Mater Res, Vol. 19, 335-48
9. Silverstone L.M., Saxton C.A., Dogon I.L., Fejerskov O. (1975): Variation in the pattern of acid etching of human dental enamel examined by scanning electron microscopy, in: Caries Res, Vol. 9, 373-87
10. Galil K.A., Wright G.Z. (1979): Acid etching patterns on buccal surfaces of permanent teeth, in: Pediatric dent, Vol. 1, 230-34
11. Carstensen W. (1992): The effects of different phosphoric acid concentrations on surface enamel, in: Angle Orthod, Vol. 62, No. 1, 51-58
12. Rossouw P.E. (2010): A Historical Overview of the Development of the Acid-Etch Bonding System in Orthodontics, in: Seminars in Orthodontist, Vol. 16, 2-23
13. Øgaard B., Fjeld M. (2010): The enamel surface and bonding in orthodontics, in: Seminars in Orthodontist Vol:16 pp.37-48
14. Zachrisson B.U., Büyükyılmaz T. (2005): Bonding in Orthodontics. In: Orthodontics: current principles and techniques, Graber TM, Vanarsdall RL, Vig KW of editors: 579-659, Elsevier Health Sciences, St Louis
15. Üşümez S., Erverdi N (2008): Adhesive and bonding in orthodontics, 45-67, Elsevier.
16. Carstensen W. (1995): Effect of reduction of phosphoric acid concentration on the shear bond strength of brackets, in: Am J Orthod Dentofac, Vol. 108, 274-7
17. Bhad W.A., Hazarey P.V. (1995): Scanning electron microscopic

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- study and shear bond strength measurement with 5% and 37% phosphoric acid, in: *Am J Orthod Dentofac*, Vol. 108, 410-4
18. Mattick C.R., Hobson R.S. (2000): A comparative micro-topographic study of the buccal enamel of different tooth types, in: *J Orthod*, Vol. 27, 143-8
  19. Hosein I., Sherriff M., Ireland A.J. (2004): Enamel loss during bonding, debonding and cleanup with use of self etching primer, in: *Am J Orthod Dentofac*, Vol. 126, 17-24
  20. Diedrich P. (1981): Enamel alterations from bracket bonding and debonding a study with the scanning electron microscope, in: *Am J Ortho*, Vol. 79, 500-22
  21. Hobson R.S., McCabe J.F. (2002): Relationship between enamel etch characteristic and resin-enamel bond strength, in: *Br Dent J*, Vol. 192, 163-8
  22. Daronch M., DeGoes M.F., Garrilho M.R., Chan D.C., Darochi M., Sinhoreti M.A. (2003): Antibacterial and conventional self etching primer system: morphological evaluation of intact primary enamel, in: *Journal of Clinical Pediatric Dentistry*, Vol. 27, 251-6
  23. Bishara S. E., Ostby A. W., Laffoon, J., Warren J. J. (2008): Enamel cracks and ceramic bracket failure during debonding in vitro, in: *Angle Orthod*, Vol. 78, 1078-83
  24. Graber TM, Eliades T, Athanasiou A. (2005): Risk management in orthodontics: experts' guide to malpractice, in: *Br Dent J*.114-5
  25. Al-Suleiman M., Baba F., Sawan M.N., Suliman A. (2014): Mechanical Evaluation of the Effect of Reducing Phosphoric Acid Concentrations and Etching Duration on the Bond Strength of Orthodontic Brackets, in: *J Dent Oral Disord Ther* 2(2); 1-5
  26. Akhavan Niaki E., Esmaily M., Jalali Y.F. (2017) Evaluation of Etching Time and Concentration on Shear Bond Strength of Metallic Brackets Using a 10-MDP Containing Adhesive, in: *Iran J Ortho*;12(1):e6362
  27. Barkmeier W.W., Gwinnett A.J., Shaffer S.E. (1987): Effects of reduced acid concentration and etching time on bond strength and enamel morphology, in: *J Clin Orthod*, Vol. 21, 395-8.
  28. Legler L.R., Retief H. (1989): Effects of phosphoric acid concentration and etching duration on shear strength of bonding resin to enamel, in: *Am J Orthod Dentofac*, Vol. 96, No. 6, 485-92
  29. Sadowsky P.L., Retief D.H., Cox P.R., Hernández-Orsini R., Rape W.G, Bradley E.L. (1990): Effects of etchant concentration and duration on the retention of the orthodontic brackets: An in vivo study, in: *Am J Orthod Dentofac*, Vol. 98, No. 5, 417-21
  30. Akhavan Niaki E., Esmaily M., Farajzadeh Jalali Y. (2017): Devaluation of etching time and concentration on shear bond strength of metallic brackets using a 10-MDP containing adhesive, in: *Iran J Ortho*, 12(1): e6362
  31. Odegaard J., Segner D. (1988): Shear bond strength of metal brackets compared with a new ceramic bracket, in: *Am J Orthod Dentofac*, Vol. 94, 201-6