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# Evaluation of Etching Time and Concentration on Shear Bond Strength of Metallic Brackets Using a 10-MDP Containing Adhesive

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## Abstract

**Background:** Bonding is the most commonly used technique to attach brackets to tooth surfaces.
**Objectives:** The aim of this study was to determine the effects of time and concentration of etching with phosphoric acid on shear bond strength (SBS) of orthodontic brackets after using 10-MDP containing adhesive.
**Methods:** Fifty intact premolars were randomly divided into 5 groups (n = 10). They were mounted in resin blocks. In control group a gel without phosphoric acid and in-group 2, 3, 4, and 5, phosphoric acid 37% for 5 or 15 seconds and phosphoric acid 15% for 5 or 15 seconds was used respectively. Clearfil SE Bond was used as an adhesive resin for all samples. After bonding of brackets, they were put under 300 thermocycling aging regime and after that the bracket/tooth SBS was measured by universal testing machine. The adhesive remnant index (ARI) was evaluated under a stereomicroscope. Data were statistically analyzed using two-way ANOVA and Kruskal-Wallis test.
**Results:** The highest mean SBS belonged to the 15% phosphoric acid etching in 15 seconds while the lowest value was seen in 37% phosphoric acid etching in 5 seconds. The effect of etching time on SBS was not significant ($P = 0.31$) but the effect of concentration in SBS has been significant ($P < 0.001$). In terms of ARI, in application of 15% phosphoric acid, more than 50% of the resin remained on tooth surface. In application of 37% phosphoric acid, all the resin remained on tooth surface.
**Conclusions:** In using of Clearfil SE bond, containing 10-MDP, preparing enamel by 15% phosphoric acid had created strong enough SBS.

*Keywords:* Orthodontics, Etching, Shear Strength, 10-MDP

## 1. Background

Bonding is the most commonly used technique to attach brackets to tooth surfaces. The basis for the adhesion of brackets to enamel has been enamel etching with phosphoric acid, as first proposed by Buonocore in 1955 (1, 2). Unfortunately, in spite of the fact that bonding technique has many advantages such as high esthetic and simple procedure, still have some drawbacks such as failure of resin bond due to excessive force. This problem lead to increase in cost and length of treatment. An ideal bond should be able to resistant forces applied during the course of treatment while maintaining the enamel surface unharmed after debonding (3-8). Recently introduced dental adhesives and composite resins are highly reliable, with higher bond strength and less microleakage, which explain their frequent use in orthodontics (9-11).

Advances in adhesive technology have led to the introduction of self-etch primers to orthodontic treatment. The basic composition of self-etch primers and self-etch adhesive systems is an aqueous solution of acidic functional monomers, with a pH relatively higher than that of phosphoric acid etchants. Bi- or multi-functional monomers are added to provide strength to the cross-linking formed from monomeric matrix (12) because self-etch adhesive systems do not require a separate acid conditioning step and moist post-rinse control, they are considered simplified adhesive materials. Some advantages are offered over conventional etch-and- rinse systems, such as reduction of postoperative sensibility and less sensitive technique. Another advantage is that infiltration of adhesive resin tends to occur simultaneously with the self-etch process, although there are some controversies (13-16). The current self-etch adhesive systems are classified based on the number of clinical application steps: one-step or two-step adhesives (16). Two-step self-etch adhesive systems include the use of a hydrophilic etching primer, which combines acidic monomers that simultaneously etch and prime tooth substrate (17, 18) and after solvent evaporation, a layer of hydrophobic and bonding agent seal the dentin (19). One-step self-etch adhesive systems are all-in-one adhesives, which combine the etching, priming and bonding (20). The bonding mechanism of self-etch adhesive systems has been intensely investigated and two-fold bonding mechanisms; micro-mechanical interlocking and chemical bonding. The functional acidic monomers are able to chemically interact with hydroxyapatite and are composed by specific carboxylic, phosphonic or phosphate groups, such as: The dihydrogenphosphate group from 10-MDPmonomer is responsible for etching and chemical bonding, while its long carbonyl chain provides the hydrophobic properties and hydrolytic stability to this acidic monomer. 10-MDP forms a strong ionic bond with calcium from hydroxyapatite of enamel or dentin, also resulting in Ca- salt (21).

The clinical success of Clearfil SE bond might be a result of its chemical composition, specifically the monomer 10-MDP. This monomer bonds chemically to hydroxyapatite by forming stable calcium-phosphate salt without causing strong decalcification.

Although in vitro studies have indicated the selective enamel etching for bonding of self-etch adhesive systems (22-25), which tends to increase the bond strength, diffusion of such acidic monomers beyond the classic hybrid layer (interfacial zone) and their ion- exchange interactions with the available hydroxyapatite could result in formation of stable organic –inorganic complexes. On the other hand, presence of adequate hydroxyapatite is necessary to obtain a chemical bond between this monomer and enamel. Over solution of mineral structure because of etching can produce weak interface in the area of resin infiltration in etched enamel. Therefore, providing the optimum parameters for etching enamel before application of self-etch adhesive system is essential (16, 26).

## 2. Objectives

The following research was aimed to determine the effects of time and concentration of etching with phosphoric acid on shear bond strength of metallic orthodontic brackets on tooth surface after using 10-MDP.

## 3. Methods

Fifty extracted intact premolars with no visible cracks or caries were gathered from a dental clinic in Tehran. They were disinfected in 0.5% Chloramine-T solution (4°C) for one week. The teeth were then washed with water For 15 seconds. They were mounted horizontally in polymethyle methacrylate resin blocks (Acroparse, Iran) so that only labial surface of teeth was exposed. The samples were randomly divided into 5 groups (n = 10).

In control group (10 samples) a gel without phosphoric acid was used and Clearfil SE Bond (Kuraray Dental, Osaka, Japan) which is a self-adhesive 2-step system was used which required primer and subsequently adhesive (15 seconds application of primer and adhesive for 10 seconds). Air syringe was used with light pressure on bonding surface. The samples were cured (LED, 1000 mw/Cm$^3$) for 10 seconds, 5 seconds on mesial and 5 seconds on distal surface. The composite resin was added to the backing of the brackets and 300gr of force was applied on the tooth surface to gain an equal thickness of composite for all samples. The samples were cured for 10 seconds (5 seconds from mesial and 5 seconds from distal).

In group 2 and 3, 10 teeth were prepared using phosphoric acid 37% for 5 or15 seconds respectively. Then samples were washed by air/water syringe for 15 seconds and dried using air syringe. All the remaining steps were done similar to other group.

In group 4 and 5, teeth were etched by phosphoric acid 15% for 5 or 15 seconds respectively and the remaining steps were similar to other groups.

Different concentrations of phosphoric acid etchant gel (15 and 37%wt) were prepared using 85% orthophosphoric acid (Merk, Germany) and ethyl cellulose as thickening agent.

All samples in the five groups were put under 300 thermocycling aging regime between 5°C and 55°C and duration of 20 seconds. The samples were tested for shear bond strength (SBS) by Roell-7060 universal testing machine (Zwick/Roell, Germany). The obtained value (N) was divided by the bracket surface area (mm$^2$) to calculate the SBS in mega Pascals (MPa). After debonding remaining resin on the buccal surface of enamel was evaluated under a stereomicroscope (Nikon, SMZ800, Japan) at 10X magnification to score the amount of remaining adhesive using the ARI. Remaining resin on the tooth surface was graded according to the following: (0)- no residual resin remaining on enamel (1) -less than 50% of surface is covered by resin (2) - more than 50% of surface is covered by resin (3) -The entire surface is covered by resin.

Results were analyzed using two-way ANOVA followed by Dunnet multiple comparision. The Kruskal-Wallis test was also applied to analyze the ARI results.

## 4. Results

Descriptive results showed mean and SD plus maximum and minimum of SBS of groups after etching with 15 and 37% phosphoric acid in 5 and 15 seconds. The highest mean SBS belonged to the 15% phosphoric acid etching in 15 seconds group while the lowest value was seen in 37% phosphoric acid etching in 5 seconds group (Table 1).

**Table 1.** Shear Bond Strength of Metallic Brackets to Tooth Surface in Different Protocols of Etching With Phosphoric Acid

| Group | Etching Time, s | Mean ± SD | Min | Max |
|---|---|---|---|---|
| **Control** | - | 7.71 ± 2.37 | 3.74 | 12.19 |
| **15% phosphoric acid** | 5 | 7.05 ± 1.73 | 3.2 | 9.37 |
| **15% phosphoric acid** | 15 | 7.88 ± 1.83 | 5.33 | 10.51 |
| **37% phosphoric acid** | 5 | 3.77 ± 1.09 | 2.69 | 5.62 |
| **37% phosphoric acid** | 15 | 3.95 ± 1.51 | 2.76 | 7.78 |

Two-way ANOVA showed that the effect of etching time on shear bond strength was not significant (P = 0.31) but the effect of phosphoric acid concentration in bond strength have been significant (P < 0.001). In other words more or less equal amounts of bond strength have been seen in 5 and 15 seconds of etching time groups. By increasing the concentration of acid from 15 to 37% the bond strength values have been significantly reduced. The results of Dunnet multiple comparisons showed that there was significant difference in shear bond strength between control groups and etching protocols of 5 second (P < 0.0001) and 15 second (P < 0.0001) with application of 37% phosphoric acid. However; the difference in control groups and etching protocols of 5 seconds (P = 0.81) and 15 seconds (P = 0.99) with application of 15 % phosphoric acid was not significant.

In terms of ARI, Kruskal-Wallis test showed that in application of 15% phosphoric acid, more than 50% of the resin remained on tooth surface. In application of 37% phosphoric acid, all the resin remained on tooth surface but the cases of bracket separation was the highest. In control group bond failure often occurs at the tooth-composite interface.

## 5. Discussion

Failure of resin bond due to excessive force is a major drawback of fixed orthodontic appliances requiring composite for bonding of brackets. Application of a composite resin with optimal mechanical properties and sufficiently high bond strength would be beneficial for this purpose. Several laboratory tests are commonly used to evaluate the bonding performance of adhesives, such as tensile and shear bond strength tests. In the present study, shear bond strength was tested which is a very popular method with acceptable reproducibility. This method has high similarity with clinical situations and it seems that mastication forces probably insert higher force on brackets (27, 28). The oral cavity cannot be simulated completely using laboratory and experimental methods due to its multifactorial character. Some researchers have shown that after inserting thermocycling, the strength of adhesive dental materials is negatively influenced (29, 30). In the present study, all the samples were exposed to 300 thermocycles between 5°C and 55oc for 20 seconds in order to simulate the clinical conditions.

In recent study, the degree of SBS on different groups has been evaluated to be between 3.77 to 7.88 MPa. The highest mean SBS belonged to the 15% phosphoric acid etching in 15 seconds group, however the difference in control groups and etching protocols of 5 seconds (P = 0.81) and 15 seconds (P = 0.99) with application of 15 % phosphoric acid was not significant. The bond strength decreased as the concentration of phosphoric acid increased and the 37% phosphoric acid showed the lowest SBS. So the concentration of phosphoric acid in the latter group is not recommended for use in clinical setting. The result of this study showed that the etching time does not have great effects on shear bond strength of brackets to tooth surface but the concentration of phosphoric acid has significant effects in this regard.

However; Mardaga and Shannon reported that gradual increase in the amounts of the tensile bond strength was by increasing the etching time with 37% phosphoric acid for 15, 20, 30 and 60 seconds (31). In contrast with the findings of the previous study, Beech and Jalaly showed that the amounts of bond strength of orthodontic brackets to etched enamel using 50% phosphoric acid for 5, 15 and 60 seconds did not show significant differences (32). Similar results were reported in a study done by Barkmeier et al. which suggested that the shear bond strength of one type of composite resin to etched enamel surface with phosphoric acid for 15 and 60 seconds were the same (2). In another study, it was also reported that tensile strength of composite resin to etched enamel surface with 10 to 60% phosphoric acid for 60 seconds did not show significant difference but the degree of bond strength had decreased significantly with increased acid concentrations above 60% (33).

Barkmeier et al. evaluated the effects of changes in acid concentrations (5% and 37%) and time of etching (15 and 60 seconds) on the amounts of shear bond strength and it was shown that no significant difference was present comparing different conditions (34). In other research done by Ohsawa, the strength of sealants bond to etched enamel surface with increasing concentrations of phosphoric acid was evaluated. They concluded that by increasing the acid concentration, the degree of bond strength had increased which is against the findings of this study (35).

The concentration of phosphoric acid, which is commonly used in clinical situations, is higher than 37%, which is based on the results of Chow and Brown research in 1973. They showed that higher than 27% concentrations of acid can lead to monocalcium phosphate monohydrate production but less concentrated solutions can produce decalcium phosphate dehydrate. The first substance is more soluble and is readily removed from enamel surface after washing. The increase in bond strength after using acid etching on enamel surface is probably because of surface roughness, which is considerable and as a result, resin can easily flow on these types of surfaces (36). On the other hand providing the optimum parameters for etching enamel before application of self-etch adhesive system is due to presence of adequate hydroxyapatite to obtain a chemical bond between this monomer and enamel. Over solution of mineral structure because of etching can produce weak interface in the area of resin infiltration in etched enamel. The result of this study confirm that the over solution of etching by increasing the concentration of the phosphoric acid before application of self-etch adhesive system decreased the amount of SBS.

Adhesive should have enough bond strength and resistance against orthodontic and masticatory forces but its removal should be easy after treatment and should cause no damage to enamel. The amount of remaining adhesive is evaluated by ARI index in different investigations. The result of this study showed that in application of 15% phosphoric acid, more than 50% of the resin remained on tooth surface. In application of 37% phosphoric acid, all the resin remained on tooth surface. According to the most researches, in order to prevent break or crack on enamel surface it is better that resin remnant be remained on tooth surface after debonding.

### *5.1. Conclusions*

According to these findings, in using etch adhesive of Clearfil SE bond, containing 10-MDP, preparing enamel by 15% phosphoric acid in both 5 and 15 seconds time, had created strong enough bond between brackets and tooth surface. However when applying 37% phosphoric acid not enough bond strength was obtained between brackets and tooth surface.

## Footnote

**Authors' Contribution:** Study concept and design: Esfandiar Akhavan Niaki; acquisition of data: Masomeh Esmaily; analysis and interpretation of data: Masomeh Esmaily; drafting of the manuscript: Yasamin Farajzadeh Jalali; critical revision of the manuscript for important intellectual content: Yasamin Farajzadeh Jalali; statistical analysis: Masomeh Esmaily; administrative, technical, and material support: Esfandiar Akhavan Niaki; study supervision: Esfandiar Akhavan Niaki.

## References


1. Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. *J Dent Res.* 1955;**34**(6):849–53. [PubMed: 13271655].
2. Barkmeier WW, Shaffer SE, Gwinnett AJ. Effects of 15 vs 60 second enamel acid conditioning on adhesion and morphology. *Oper Dent.* 1986;**11**(3):111–6. [PubMed: 3532043].
3. Isman E, Karaarslan ES, Oksayan R, Tuncdemir AR, Usumez S, Adanir N, et al. Inadequate shear bond strengths of self-etch, self-adhesive systems for secure orthodontic bonding. *Dent Mater J.* 2012;**31**(6):947–53. [PubMed: 23207199].
4. van Waveren Hogervorst WL, Feilzer AJ, Prahl-Andersen B. The air-abrasion technique versus the conventional acid-etching technique: A quantification of surface enamel loss and a comparison of shear bond strength. *Am J Orthod Dentofacial Orthop.* 2000;**117**:20–6.
5. Chen CS, Hsu ML, Chang KD, Kuang SH, Chen PT, Gung YW. Failure analysis: enamel fracture after debonding orthodontic brackets. *Angle Orthod.* 2008;**78**(6):1071–7. doi: 10.2319/091907-449.1. [PubMed: 18947273].
6. Sudjalim TR, Woods MG, Manton DJ. Prevention of white spot lesions in orthodontic practice: a contemporary review. *Aust Dent J.* 2006;**51**(4):284–9. [PubMed: 17256301] quiz 347.
7. Stecksen-Blicks C, Renfors G, Oscarson ND, Bergstrand F, Twetman S. Caries-preventive effectiveness of a fluoride varnish: a randomized controlled trial in adolescents with fixed orthodontic appliances. *Caries Res.* 2007;**41**(6):455–9. doi: 10.1159/000107932. [PubMed: 17827963].
8. Gorton J, Featherstone JD. In vivo inhibition of demineralization around orthodontic brackets. *Am J Orthod Dentofacial Orthop.* 2003;**123**(1):10–4. doi: 10.1067/mod.2003.47. [PubMed: 12532056].
9. Uysal T, Yagci A, Uysal B, Akdogan G. Are nano-composites and nano-ionomers suitable for orthodontic bracket bonding?. *Eur J Orthod.* 2010;**32**(1):78–82. doi: 10.1093/ejo/cjp012. [PubMed: 19401356].
10. Powers JM, Kim HB, Turner DS. Orthodontic adhesives and bond strength testing. *Semin Orthod.* 1997;**3**(3):147–56. [PubMed: 9573876].
11. Knoll M, Gwinnett AJ, Wolff MS. Shear strength of brackets bonded to anterior and posterior teeth. *Am J Orthod.* 1986;**89**(6):476–9. [PubMed: 3521300].
12. Van Landuyt KL, Snauwaert J, De Munck J, Peumans M, Yoshida Y, Poitevin A, et al. Systematic review of the chemical composition of contemporary dental adhesives. *Biomaterials.* 2007;**28**(26):3757–85. doi: 10.1016/j.biomaterials.2007.04.044. [PubMed: 17543382].
13. Tay FR, King NM, Chan KM, Pashley DH. How can nanoleakage occur in self-etching adhesive systems that demineralize and infiltrate simultaneously?. *J Adhes Dent.* 2002;**4**(4):255–69. [PubMed: 12666745].
14. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, et al. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. *Oper Dent.* 2003;**28**(3):215–35. [PubMed: 12760693].

15. Reis AF, Giannini M, Pereira PN. Long-term TEM analysis of the nanoleakage patterns in resin-dentin interfaces produced by different bonding strategies. *Dent Mater.* 2007;**23**(9):1164–72. doi: 10.1016/j.dental.2006.10.006. [PubMed: 17125829].
16. Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, Van Landuyt KL. State of the art of self-etch adhesives. *Dent Mater.* 2011;**27**(1):17–28. doi: 10.1016/j.dental.2010.10.023. [PubMed: 21109301].
17. Chigira H, Yukitani W, Hasegawa T, Manabe A, Itoh K, Hayakawa T, et al. Self-etching dentin primers containing phenyl-P. *J Dent Res.* 1994;**73**(5):1088–95. [PubMed: 8006236].
18. Watanabe I, Nakabayashi N, Pashley DH. Bonding to ground dentin by a phenyl-P self-etching primer. *J Dent Res.* 1994;**73**(6):1212–20. [PubMed: 8046111].
19. Tay FR, Pashley DH. Have dentin adhesives become too hydrophilic?. *J Can Dent Assoc.* 2003;**69**(11):726–31. [PubMed: 14653938].
20. De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts P, Braem M, et al. A critical review of the durability of adhesion to tooth tissue: methods and results. *J Dent Res.* 2005;**84**(2):118–32. [PubMed: 15668328].
21. Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, et al. Comparative study on adhesive performance of functional monomers. *J Dent Res.* 2004;**83**(6):454–8. [PubMed: 15153451].
22. Shinohara MS, de Oliveira MT, Di Hipolito V, Giannini M, de Goes MF. SEM analysis of the acid-etched enamel patterns promoted by acidic monomers and phosphoric acids. *J Appl Oral Sci.* 2006;**14**(6):427–35. [PubMed: 19089243].
23. Luhrs AK, Guhr S, Schilke R, Borchers L, Geurtsen W, Gunay H. Shear bond strength of self-etch adhesives to enamel with additional phosphoric acid etching. *Oper Dent.* 2008;**33**(2):155–62. doi: 10.2341/07-63. [PubMed: 18435189].
24. Erickson RL, Barkmeier WW, Kimmes NS. Bond strength of self-etch adhesives to pre-etched enamel. *Dent Mater.* 2009;**25**(10):1187–94. doi: 10.1016/j.dental.2009.04.004. [PubMed: 19473695].
25. Devarasa GM, Subba Reddy VV, Chaitra NL, Swarna YM. Self-etching adhesive on intact enamel, with and without pre-etching. *Microsc Res Tech.* 2012;**75**(5):650–4. doi: 10.1002/jemt.21106. [PubMed: 22065398].
26. Yoshihara K, Yoshida Y, Hayakawa S, Nagaoka N, Irie M, Ogawa T, et al. Nanolayering of phosphoric acid ester monomer on enamel and dentin. *Acta Biomater.* 2011;**7**(8):3187–95. doi: 10.1016/j.actbio.2011.04.026. [PubMed: 21575747].
27. Katona TR. A comparison of the stresses developed in tension, shear peel, and torsion strength testing of direct bonded orthodontic brackets. *Am J Orthod Dentofacial Orthop.* 1997;**112**(3):244–51. doi: 10.1016/S0889-5406(97)70251-8. [PubMed: 9294351].
28. Katona TR, Long RW. Effect of loading mode on bond strength of orthodontic brackets bonded with 2 systems. *Am J Orthod Dentofacial Orthop.* 2006;**129**(1):60–4. doi: 10.1016/j.ajodo.2004.09.020. [PubMed: 16443480].
29. Bishara SE, Ajlouni R, Laffoon JF. Effect of thermocycling on the shear bond strength of a cyanoacrylate orthodontic adhesive. *Am J Orthod Dentofacial Orthop.* 2003;**123**(1):21–4. doi: 10.1067/mod.2003.1. [PubMed: 12532058].
30. Bishara SE, Ostby AW, Laffoon JF, Warren J. Shear bond strength comparison of two adhesive systems following thermocycling. A new self-etch primer and a resin-modified glass ionomer. *Angle Orthod.* 2007;**77**(2):337–41. doi: 10.2319/0003-3219(2007)077[0337:SBSCOT]2.0.CO;2. [PubMed: 17319771].
31. Mardaga WJ, Shannon IL. Decreasing the depth of etch for direct bonding in orthodontics. *J Clin Orthod.* 1982;**16**(2):130–2. [PubMed: 7047564].
32. Beech DR, Jalaly T. Bonding of polymers to enamel: influence of deposits formed during etching, etching time and period of water immersion. *J Dent Res.* 1980;**59**(7):1156–62. [PubMed: 6991561].
33. Gottlieb EW, Retief DH, Jamison HC. An optimal concentration of phosphoric acid as an etching agent. Part I: Tensile bond strength studies. *J Prosthet Dent.* 1982;**48**(1):48–51. [PubMed: 7050352].
34. Barkmeier WW, Gwinnett AJ, Shaffer SE. Effects of reduced acid concentration and etching time on bond strength and enamel morphology. *J Clin Orthod.* 1987;**21**(6):395–8. [PubMed: 3298319].
35. Osawa T. Studies on solubility and adhesion of the enamel in pre-treatment for caries preventive sealing. *Bull Tokyo Dent Coll.* 1972;**13**(1):65–82. [PubMed: 4501648].
36. Chow LC, Brown WE. Phosphoric acid conditioning of teeth for pit and fissure sealants. *J Dent Res.* 1973;**52**(5):1158. [PubMed: 4517762].