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Research Article

Influence of Varied Acid Concentration and Varied Etching Time on Shear Bond Strength of Dental Resin-Composites

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Abstract

Purpose: To evaluate the influence of varied acid concentration as well as varied etching time on the shear bond strength of dental resin-composites bonded to the dentin of human molar teeth.

Materials and Methods: Eighty extracted human molars were used. The teeth crowns were horizontally embedded in a self-curing polymethyl methacrylate (PMMA) so that about 3 mm of the buccal surface was projecting over the surface of the set PMMA. Exposure of the dentin was carried out using a high-speed diamond bur and flattened on wet SiC paper, grits 180-320 under running tap water. Four aqueous phosphoric acid solutions; 10%, 25%, 37% and 50% (weight %) were made up with distilled water and liquid orthophosphoric acid. The eighty specimens were randomly allocated to four groups, 20 specimens each. Specimens of Group I were etched with 10%, Group II with 25%, Group III with 37% and Group IV with 50% phosphoric acid solution. Specimens of each of these groups were divided into four subgroups, each of which was etched for one of the following time periods; 10 s, 20 s, 30 s and 40 s. A 3-step etch-and-rinse adhesive (Scotchbond Multi-Purpose, 3M ESPE, St. Paul, MN, USA) was used. After etching and rinsing, the Scotchbond primer and adhesive were applied according to the manufacturer instructions. A resin-composite (Filtek™ Z350 XT, 3M ESPE, St. Paul, MN, USA) disc specimen (4 mm × 2 mm) was built to each dentin surface using a metallic mold and light-activated for 40 s. Debonding of the disc specimens from the dentin surfaces was achieved using a universal testing machine (model 3365, Instron, High Wycombe, UK).

Results: Dependent on the acid concentration used and the etching time applied, the mean value of shear bond strength ranged from 6.49 MPa to 23.06 MPa. At a given etching time (10 s), there was a positive strong (r^2 = 0.982) correlation between bond strength and acid concentration. Also, at a given acid concentration (10%), linear regression analysis revealed a positive strong (r^2 = 0. 943) correlation between bond strength and etching time.

Conclusion: There was, to some extent, compensation between the concentration of the etching acid and the length of the etching time.

Keywords: Shear bond strength, Acid concentration, Etching time, Dentin surface

Introduction

One of the most crucial goals of modern dental materials science research is to develop a truly adhesive bond between a restorative material and the natural tooth structure [1,2]. Establishing such a bond has the potential to enhance conservation of tooth structure during restoration of teeth as well as to increase the clinical durability of restorations [2].

Bonding to enamel and dentin has become so critical to the extent that it is no longer acceptable that a restorative material is just required to replace the defective dental structure, but it is also mandatory to adhere, seal, provide a durable, long-lasting surface in a very harsh environment, and yet biocompatible [2,3].

The development of adhesive systems has caused profound changes in the dental practice [4]. The primary aim of dental adhesives is to provide retention to resin-composite fillings or cements. A good adhesive should be able to prevent leakage along the restoration margins as well as withstanding mechanical forces, particularly shrinkage stress from the lining composite. Clinically, failure of restorations occurs more often due to inadequate sealing, with subsequent discoloration of the cavity margins, than due to loss of retention [5,6].

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In 1955, Buonocore established the basis for modern restorative dentistry when he described a technique for bonding acrylic materials to enamel using phosphoric acid [1]. The strong adhesive joints formed by bonding resins to etched enamel resulted in the widespread use of "acid-etching techniques" rather than pins for the retention of resin-composite restorations [7,8]. At the beginning, Buonocore thought that "acid-etching" simply increased the microscopic surface area available for resin retention. Later on, John Gwinnett looked at the interface more closely and reported that adhesive resins could penetrate into acid-etched enamel prisms where they could actually envelop apatite crystallites rendering them acid-resistant [9]. With regard to dentin bonding, Nakabayashi et al. [10] were the first to demonstrate true hybrid layer formation in acid-etched dentin. This was best observed by transmission electron microscopy and later demonstrated by scanning electron microscopy following argon ion beam etching [11]. Nakabayashi's group was the first to demonstrate that resins could infiltrate into acid-etched dentin to form a new structure composed of a resin-matrix reinforced by collagen fibrils. He named this new biocomposite the "hybrid layer" [9,10].

Though it was repeatedly demonstrated that etching pattern largely depends on the acidity of the conditioner, there is a controversy between authors regarding the acid concentration that should be applied to get the best etching results [12,13]. There has not been general agreement between practitioners about the use of mild self-etch adhesives together with reports about low bonding effectiveness of these adhesives [14]. On the contrary, Chan et al. [15], showed that high bond strength results can be achieved in dentin using etching acids which are more dilute than those commercially available.

In addition, it was reported that application of high acid concentration in the etching procedures may produce aggressive decalcification of the dentin which, in turn, may result in the production of a deep demineralized layer, with collapse of the unsupported surface collagen, which is inaccessible to complete resin infiltration. This results in a weak uninfiltrated band of collagen between the hybrid layer and unetched dentin which may be susceptible to long-term degradation due to slow hydrolysis of exposed, unprotected collagen [14,16,17].

Moreover, there has been little information about the optimal time of acid application to get the optimal bonding between a restorative material and the tooth structure. Hashimoto et al. [18], reported that prolonged acid-conditioning times (longer than recommended by the manufacturer) created a demineralized dentin zone within the bond structure that resulted in lower bond strength.

Therefore, this study was conducted to investigate the effect of varied acid concentration and varied etching time on the bond strength. The two null hypotheses investigated were; (i) varied acid concentration will not affect bond strength and (ii) varied time periods of acid-conditioning will not affect bond strength.

Materials and Methods

Tooth preparation

Shear bond strength (SBS) was determined on sound dentin surfaces of eighty extracted human molars (first and second lower molars) with unaffected buccal surfaces. The teeth crowns were horizontally embedded in a self-curing polymethyl methacrylate (PMMA; Esschem Co., PA, USA). PMMA was mixed according to the instructions of the manufacturer and poured into a cylindrical mould measuring 4 cm in diameter and 3 cm in depth. The embedding process was done carefully so that the buccal surface of each tooth crown projected 3 mm above the surface of the set PMMA.

Dentin surface preparation

Buccal dentin was exposed using a high-speed diamond bur (8-3 Kiyohara Industrial Park, Utsunomiya, Tochigi, Japan) and flattened on wet SiC paper, grits 180-320 under running tap water. Care was taken to ensure that all specimens were prepared nearly similarly. The prepared surfaces were dried for 3-5 s before etching.

Surface etching

Four aqueous phosphoric acid solutions: 10%, 25%, 37% and 50% (weight %) were made up with distilled water and liquid orthophosphoric acid. The eighty specimens were randomly allocated to four groups, 20 specimens each. Specimens of Group I were etched with 10%, Group II with 25%, Group III with 37% and Group IV with 50% phosphoric acid solution. Specimens of each of these groups were divided into four subgroups, each of which was etched for one of the following time periods: 10 s, 20 s, 30 s and 40 s.

Application of adhesive and resin-composite

A 3-step etch-and-rinse adhesive (Scotchbond Multi-Purpose, 3M ESPE, St. Paul, MN, USA) was used to bond the resin-composite to the dentin surface. After etching and rinsing the dentin surface of all specimens, the Scotchbond primer and adhesive were applied according to the manufacturer instructions. A resin-composite (Filtek™ Z350 XT, 3M ESPE, St. Paul, MN, USA) disc specimen (4 mm × 2 mm) was built to each dentin surface using a metallic mold and light-activated for 40 s using the tungsten-halogen curing unit Translux Energy (850 mW/cm^2, Heraeus Kulzer, Hanau, Germany).

Mechanical testing (debonding)

Shear bond strength (SBS) was measured at room temperature using a universal testing machine (model 3365, Instron, High Wycombe, UK). The test assembly was done so that the long axis of the molar crown was set vertically and the shearing blade (stainless steel chisel with a pointed end measuring 2 mm in thickness) contacted the upper part of the resin-composite disc at the dentin-resin interface. Loading was continued at a crosshead speed of 1 mm/min until debonding of the disc specimen from the dentin surface. Only specimens with adhesive failure were considered. Values of shear bond strength for all test groups were recorded and compared. Data acquisition was done using the software (Bluehill LE testing software, Instron Ltd).

Statistical analysis

A two-way analysis of variance was conducted to compare the factors: acid concentration (at 4 levels) and etching time (at 4 levels). As the interaction was significant ($p = 0.001$), the comparison between groups was conducted at each etching time using a one-way analysis of variance. The significance level was established at ($p \leq 0.05$). Levene's test for homogeneity of variance was carried out for the data of each etching time (p ≤

0.05). As equal variances were confirmed (p = 0.802) for data at all etching time groups, the Bonferroni post hoc test was used to determine the differences in shear bond strength between groups. Linear regression analysis was performed to investigate relationship between bond strength and acid concentration and between bond strength and etching time of all groups.

Results

Upon fitting the two-way ANOVA model to the data for the outcome measurement "shear bond strength", there was a statistically significant interaction (P = 0.001) between the two factors "concentration" and "time". This meant that the data for each concentration or time had to be considered separately. Data of bond strength (mean and standard deviation) at different acid concentrations and different etching times are presented in Table 1 and shown in Figure 1.

Table 1: Mean data and standard deviations of shear bond strength for all groups (at different acid concentration and etching time). Each bond strength value represents the mean of five measurements.

Group	Acid concentration	Etching time	Mean bond strength (MPa)	St. deviation
I	10%	10 s	7.05	0.89
		20 s	10.29	0.79
		30 s	11.89	1.42
		40 s	13.04	1.26
II	25%	10 s	10.49	1.15
		20 s	19.77	1.30
		30 s	21.29	1.61
		40 s	11.70	1.46
III	37%	10 s	13.33	1.21
		20 s	23.06	1.24
		30 s	21.41	1.59
		40 s	15.26	1.16
IV	50%	10 s	18.56	1.28
		20 s	20.19	0.91
		30 s	11.40	2.18
		40 s	6.49	1.03

Statistically significant differences were found between the investigated acid concentrations at the four etching times selected: at 10 s (p = 0.006), at 20 s (p = 0.001), at 30 s (p = 0.001) and at 40 s (p = 0.003).

The strongest bond strength was recorded for 37% acid concentration at 20 s etching time (23.06 MPa) followed by 37% at 30 s (21.41 MPa) and 25% at 30 s (21.29 MPa). The weakest bond strength was recorded for 50% at 40 s (6.49 MPa) followed by 10% at 10 s (7.05 MPa) and 10% at 20 s (10.29 MPa).

At 10% acid concentration, increasing the etching time (10-40 s) led to an increase in the bond strength (7.05-13.04 MPa) systematically. At 25%, extending the etching time from 10 s to 30 s resulted in an increase in the bond strength from 10.49 MPa to 21.29 MPa. However, longer etching time (40 s) at the same concentration (25%) recorded lower bond strength (11.70 MPa) than that recorded at 20 s and 30 s. For both 37% and 50% acid concentrations, the highest bond strength was recorded at 20 s (23.06 MPa and 20.19 MPa), respectively. Extending the etching beyond 20 s at both concentrations resulted in lower bond strength. For relatively high acid concentrations (37% and 50%), the highest bond strength was recorded at 20 s.

From the data recorded, it was clear that there was compensation, to some extent, between the concentration of the etching acid and the etching time. This means that a relatively weaker acid concentration was compensated for by a relatively longer etching time, and a shorter etching time was compensated for by a stronger acid solution to give comparable bond strength. Examples of this compensation were obvious in case of 25% and 30 s (21.29 MPa), 37% and 20 s (23.06 MPa), and 50% and 10 s (18.56 MPa). However, this compensation was missing with some other data, where nearly equal bond strength was obtained with different acid concentrations and same etching time. This was clear in case of 25% at 30 s (21.29 MPa) and 37% at 30 s (21.41 MPa).

At a given etching time (10 s), there was a positive strong (r^2 = 0.982) correlation between bond strength and acid concentration as shown in Figure 2. This was not applied to the

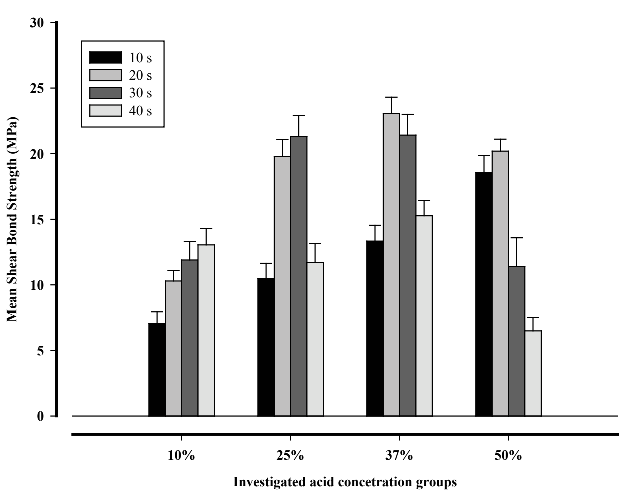

Figure 1: Error bar showing mean shear bond strength at different acid concentration and different etching time. Each bond strength value represents the mean of five measurements.

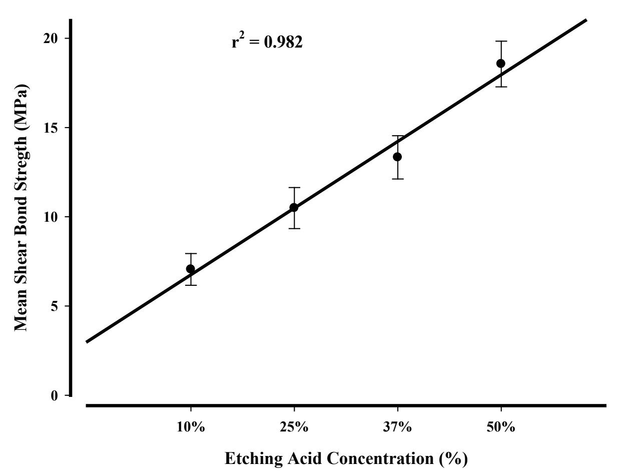

Figure 2: At a given etching time (10 s), linear regression analysis revealed a strong positive correlation between shear bond strength (MPa) and concentration of the etching acid within the selected range (10-50%).

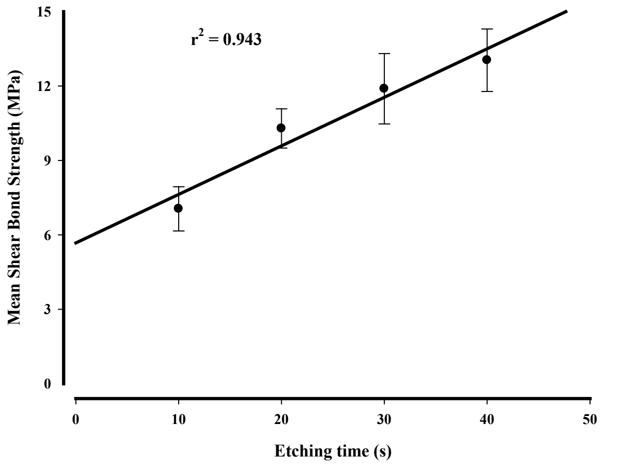

Figure 3: At a given acid concentration (10%), linear regression analysis revealed a strong positive correlation between shear bond strength (MPa) and etching time within the selected range (10-40 s).

other three etching times: 20 s, 30 s and 40 s. Also, at a given acid concentration (10%), linear regression analysis revealed a positive strong (r^2 = 0. 943) correlation between bond strength and etching time as shown in Figure 3. Again, this was not the case for the other three concentrations: 25%, 37% and 50%.

Discussion

An effective resin adhesion enhances the clinical behavior of restorative materials by blocking, or at least reducing, interfacial micro leakage together with rendering it easier to have conservative cavity preparations without traditional mechanical retention [19,20]. Creating adhesion for retaining and sealing a restoration involves uniting two dissimilar surfaces: a mineralized tooth structure and a restorative material [21]. When applied to surfaces, adhesives join materials together to resist separation and transmit loads across the bonds. Adhesion is not an inherent property of an adhesive. Rather, it is the response of an assembly to deformation loads. In the dental literature, this is often referred to as "bond strength" [22,23].

It has been reported that the "restoration-tooth" bond strength depends upon the tooth structure, the type of etching acid used and restorative material applied [24]. Moreover, it is widely recognized that the characteristics of the bonding substrate greatly affect the bond strength. Such clinically relevant substrates include caries-affected, caries-infected, sclerotic, deep, and bur cut dentin [25].

Numerous dental investigations can be conducted both in vivo as well as in vitro. However, in case of investigating bond strength between resin-composite restorations and human dental structure, laboratory screening tests may have some advantages over clinical trials provided that these laboratory investigations closely simulate the clinical conditions. Some of these advantages include patient-independence, saving money and time, ability to evaluate the effect of a single variable if all other variables are kept constant and ease of achievements [26].

In addition to removal of smear layer, acid-etching increases the permeability of resins to enamel [27] and dentin [28]. In dentin, acid-etching with 37% (weight %) phosphoric acid completely demineralizes the surface of the intertubular dentin matrix to create pores at the nano scale within the underlying collagen fibrillar matrix. This permits infiltration of dental adhesives into and around collagen fibrils to gain retention for resin-composite restorations [9,29].

Influence of Varied Acid Concentration

Since its introduction to the dental profession, there has been a controversy regarding the type and concentration of etchant, length of etching, and rinsing time. In the past, some author's recommended 50% (w/w) orthophosphoric acid buffered with 7% zinc oxide [30]. Later study [31] reported that phosphoric acid concentrations of 30 - 40% with a 60 s etching time produced a highly retentive enamel surface topography. Turner et. al. [32], found that the best bonding can be obtained by 37% phosphoric acid with an application time of 60 s, followed by copious rinsing with water for 15 - 60 s. Other studies [33,34] used phosphoric acid with varied concentrations (5 - 37) % with different application times (15 - 60) s and reported that the acid concentration can be reduced markedly without a significant increase in the failure of bonded attachments.

In this study, the application of lower and higher concentrations-than the concentration mostly used (37%)-of phosphoric acid for etching purposes was to investigate the possibility of using other concentrations in our daily practice, maintaining the same degree of success, as well as to study the effect of varying the acid concentration on the bond strength.

Besides the most commonly applied acid concentration (37%), other three acid concentrations were chosen; two lower (10% and 25%) and one higher (50%). All of these concentrations were applied for the same etching time periods pre-determined to dentin surfaces prepared very similarly. Upon evaluating the bond strength results, it was clear that the concentration of 37% gave- in most etching time periods- the greatest bond strength compared to the other concentrations. For weaker acid concentrations (10% and 25%), good bond strength was only achieved with relatively longer etching time periods. Poor bond strength recorded for these weaker concentrations at a relatively short etching time may be explained on the basis that there was no enough time for the weaker acid to demineralize the dentin surface to create a favorable environment for bonding. At higher acid concentration, however, a good bonding was only recorded at relatively short etching time periods. This may be interpreted by ability of strong acid concentrations to demineralize the dentin surface in short time. Weak bonding for high concentrations applied for a relatively longer time periods may be reasoned by what has been reported [14-17] that if a strong acid was applied to the dentin surface for a long time this may produce an aggressive decalcification of the dentin and create a deep layer of demineralized dentin inaccessible to resin infiltration which negatively affects the bonding characteristics.

As varied concentration of the etching acid resulted in varied bond strength, the first null hypothesis was rejected. A strong positive correlation (r^2 = 0.982) was found between the bond strength and the acid concentration when the etching time is relatively short (10 s) (Figure 2). Keeping the etching time short, increasing the acid concentration gradually produces a gradual improvement in the dentin surface preparation for bonding.

Influence of Varied Etching Time

Once again, there has been a controversy between researchers about the best application time for producing the greatest bonding results. Some investigations showed that the 15 s duration for phosphoric acid concentration 37% was better than other durations and any increase in the time could lead to an adverse result [35]. Other studies [36,37] demonstrated that the optimal etching time for applying phosphoric acid 37% to the orthodontic bonding area of mandibular premolars was 30s.

In the current study, a reasonable range of etching time periods (10 - 40s) was examined to investigate which time is best to be allowed to etch a dentin surface. Clinically, etching for a period of time less than 10 s will be insufficient to properly prepare the dentin surface for good bonding. On the other side, extending the etching time more than 40 s, especially in a multi-layered restoration, may significantly increase the time consumed for completing a resin-composite restoration. With the exception of 10% acid concentration, the etching time of 20 s nearly produced the best bonding results. At relatively high acid concentrations (37% and 50%), extending the etching time to 30 - 40 s resulted in lower bonding strength than those recorded at 20s. Etching for 10s exhibited an acceptable bonding only when the acid concentration was relatively high (50%).

Variation of etching time confirmed a great variation of the bond strength; therefore the second null hypothesis was rejected. A strong positive correlation ($r^2 = 0.943$) was found between the bond strength and the etching time when the acid concentration was kept low (10%) (Figure 3). Extending the etching form 10 to 40 s gave a chance to the weak acid to attack the dentin surface to prepare it for bonding. The bond strength results obtained from varied etching acid concentration and varied etching time confirmed that there was, to some point, compensation between the two test parameters (concentration and time), where a weak acid gave an acceptable bonding at a relatively long etching time and a short etching time achieved an acceptable bonding with a relatively strong acid.

Conclusions

- A concentration of 37% of phosphoric acid produced the greatest bonding results with majority of etching times examined.
- At short etching time (10 s), there was a strong correlation between the bond strength and the acid concentration.
- Similarly, at weak acid concentration (10%), there was a strong correlation between the bond strength and the etching time.
- There was, to some extent, a compensation between the concentration of the etching acid and the length of the etching time.

References

1. Stangel I, Ellis TH, Sacher E. Adhesion to tooth structure mediated by contemporary bonding systems. *Dent Clin North Am.* 2007;51(3):677-694.
2. Rueggeberg FA. Substrate for adhesion testing to tooth structure-review of the literature. *Dent Mater.* 1991;7(1):2-10.
3. Negm MM, Combe EC, Chem C, Grant AA. Factors affecting the adhesion of polycarboxylate cement to enamel and dentin. *J Prosthet Dent*. 1981;45(4):405-410.
4. Sano H, Shono T, Sonoda H, et al. Relationship between surface area for adhesion and tensile bond strength-evaluation of a micro-tensile bond test. *Dent Mater*. 1994;10(4):236-240.
5. Gaengler P, Hoyer I, Montag R, Gaebler P. Micromorphological evaluation of posterior composite restorations-a 10-year report. *J Oral Rehab.* 2004;31(10):991-1000.
6. Opdam NJ, Loomans BA, Roeters FJ, Bronkhorst EM. Five-year clinical performance of posterior resin composite restorations placed by dental students. *J Dentistry.* 2004;32(5):379-383.
7. Gilpatrick RQ, Ross JA, Simonsen RJ. Resin-to-enamel bond strength with various etching times. *Quint Inter*. 1991;22(1):47-49.
8. Gwinnett AJ, Kanca J. Micromorphology of the bonded dentin interface and its relationship to bond strength. *American J Dent.* 1992;5(2):73-77.
9. Pashley DH, Tay FR, Breschi L, et al. State of the art etch-and-rinse adhesives. *Dent Mater*. 2011;27(1):1-16.
10. Nakabayashi N, Kojima K, Masuhara E. The promotion of adhesion by the infiltration of monomers into tooth substrates. *J Biomed Mater Sci*. 1982;16(3):265-273.
11. Inokoshi S, Hosoda H, Harnirattisai C, Shimida Y, Tatsumi T. A study on the resin-impregnated layer of dentin Part I. A comparative study on the decalcified and undecalcified sections and the application of argon ion beam etching to disclose the resin-impregnated layer of dentin. *Jap Conserv Dent*. 1990;33:427-442.
12. Pashley DH, Tay FR. Aggressiveness of contemporary self-etching adhesives. Part II: etching effects on unground enamel. *Dent Mater.* 2001;17(5):430-444.
13. Perdigao J, Lopes L, Lambrechts P, Leitao J, Van Meerbeek B, Vanherle G. Effects of a self-etching primer on enamel shear bond strengths and SEM morphology. *Am Dent*. 1997;10(3):141-146.
14. Tay FR, Pashley DH, King NM, et al. Aggressiveness of self-etch adhesives on unground enamel. *Oper Dent*. 2004;29(3):309-316.
15. Chan AR, Titley KC, Cherneky R, Smith DC. A short- and long-term shear bond strength study using acids of varying dilutions on bovine dentine. *J Dent*. 1997;25(2):145-152.
16. Hashimoto M, Ohno H, Yoshida E, et al. Resin-enamel bonds made with self-etching primers on ground enamel. *Eur Oral Sci*. 2003;111(5):447-453.
17. Yoshiyama M, Matsuo T, Ebisu S, Pashley D. Regional bond strengths of self-etching/self-priming adhesive systems. *J Dent*. 1998;26(7):609-616.
18. Hashimoto M, Ohno H, Endo K, Kaga M, Sano H, Oguchi H. The effect of hybrid layer thickness on bond strength: demineralized dentin zone of the hybrid layer. *Dent Mater*. 2000;16(6):406-411.
19. Wiecskowski G, Yu XY, Davis EL, et al. Microleakage in various dentin bonding agent/composite resin systems. *Oper Dent*. 1992;(Suppl 5):62–67.
20. Eden E, Cogulu D, Attin T. The effect of finishing and polishing systems on surface roughness, microhardness and microleakage of a nanohybrid composite. *J Int Dent Med Res.* 2012;5(3):155-160
21. Latta MA, Barkmeier WW. Dental adhesives in contemporary restorative dentistry. *Dent Clin North Am*. 1998;42(4):567-577.
22. Swift EJ. Bonding systems for restorative materials-a comprehensive review. *Pediat Dent*. 1998;20(2):80-84.
23. Pardo NP, Araya PL, Pardo MP. Effect of different surface treatments on the bonds strength of a resin cement in zirconia frameworks. *J Int Dent Med Res*. 2016;9(1):1-5.
24. Lee HL, Orlowski JA, Scheidt GC, Lee JR. Effects of acid etchants on dentin. *J Dent Res*. 1973;52:1228-1232.
25. Perdigao J. Dentin bonding-variables related to the clinical situation and the substrate treatment. *Dent Mater*. 2010;26(2):24-37.
26. Van Meerbeek B, De Munck J, Yoshida Y, et al. Adhesion to enamel and dentin: current status and future challenges. *Oper Dent*. 2003;28(3):215-235.
27. Gwinnett AJ, Matsui A. A study of enamel adhesives. The physical relationship between enamel and adhesives. *Arch Oral Bio*. 1967;12(12):1615-1619.

28. Pashley DH, Ciucchi B, Sano H. Permeability of dentin to adhesive agents. *Quint Inter*. 1993;24(9):618-631.

29. Swift Jr EJ, Perdigao J, Heymann HO. Bonding to enamel and dentin: a brief history and state of the art. *Quint Inter*. 1995;26(2):95-110.

30. Williams B, von Fraunhofer JA. The influence of the time of etching and washing on the bond strength of fissure sealants applied to enamel. *J Oral Rehab*. 1977;4(2):139-143.

31. Horn HR: Practical considerations for composite resins and acid etching. *Dent Clin North Am.* 1981;25:365-376.

32. Turner C, Courts FJ, Gombola GG. The removal of phosphoric acid and calcium phosphate precipitates: an analysis of rinse time. *Pediat Dent*. 1987;9(3):208-211.

33. Legler LR, Retief H, Bradley EL, Denys FR, Sadowsky PL. Effects of phosphoric acid concentration and etching duration on shear strength of bonding resin to enamel. *Am J Orthod and Dentofac Orthop.* 1989; 96(6):485-492.

34. Carstensen W. Effects of reduced phosphoric acid concentration on shear bond strength. *Am J Orthod Dentofacial Orthop.* 1995;108(2):274-277.

35. Al-Suleiman M, Baba F, Sawan MN, Suliman A. Mechanical evaluation of the effect of reducing phosphoric acid concentrations and etching duration on the bond strength of orthodontic brackets. *J Dent, Oral Disord & Therapy*. 2014;2(2):1-5.

36. Gardner A, Hobson R. Variations in acid-etch patterns with different acids and etch times. *Am J Ortho and Dentofac Orthop*. 2001;120(1):64-67.

37. Johnston CD, Hussey DL, Burden DJ. Effect of etch duration on the microstructure of molar enamel, an *in vitro* study. *Am J Orthod and Dentofac Orthop*. 1996;109(5):531-534.

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