



Original Article

บทความวิชาการ

Shear-peel bond strength of metal bracket to porcelain surface treated with 1.23% acidulated phosphate fluoride gel

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Abstracts

Objective The purpose of this study was to compare the shear-peel bond strength of metal bracket to the prepared porcelain surface with two different surface preparations; silane and 1.23% acidulated phosphate fluoride (APF) gel.

Materials and methods The samples were comprised of 60 porcelain disks (diameter 10 mm, thickness 4 mm). All were unglazed with green stones and then were randomly assigned into 2 groups (30 specimens each). Group 1 (control), the porcelain surface was prepared with silane. Group 2, the porcelain surface was etched with 1.23% APF gel for 10 minutes. A central incisor metal bracket was bonded to the prepared porcelain surface with light-cured adhesive resin and stored in distilled water at 37°C for 24 hours. The shear-peel bond strength was measured by the Instron Universal Testing Machine with 250 Newton (N) load cell at 0.5 mm/min crosshead speed. Difference between the shear-peel bond strength of the two surface preparation procedures was analyzed with independent sample T-test at $p = 0.05$. Survival probability of bracket was also analyzed by Weibull analysis.

Results No significant difference was found between the shear-peel bond strength of the APF gel group (9.42 ± 1.93 megapascals, MPa) and that of the silane group (9.68 ± 1.91 MPa) ($p = 0.604$).

Conclusion The 1.23% APF gel can probably be an alternative of surface preparation when bonding metal bracket to porcelain surface after unglazing with a green stone bur.

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Key words: acidulated phosphate fluoride gel; metal bracket; porcelain surface treatment; shear-peel bond strength; Weibull analysis

Introduction

Dental porcelain is a popular restorative material, especially for adult patients, where it is used for restorations such as veneer, crown, and bridge. As the demand for adult orthodontic treatment increases, orthodontists are more likely to deal with the problem of placing brackets on teeth restored with porcelain.

Conventional acid-etch technique is not effective in preparation of non-enamel surface for mechanical retention of orthodontic attachment.¹ Ghassemi-Tary² found that increasing surface roughness of porcelain enhanced the shear bond strength of brackets bonded to the porcelain surface. Numerous conditioning methods have been suggested for pretreating porcelain surfaces.³ Organosilane coupling agents were suggested to enhance bonding of brackets to porcelain.^{4,5} Silane was used as a coupling agent to increase the bond strength to either glazed or roughened porcelains in many studies,^{5,6-8} but there is a tendency for cohesive failure of porcelain during the debonding process.⁹⁻¹² Additionally, the limited shelf-life time of silane causes a problem for orthodontists when finding it expired without other spare bottles. Mechanical roughening with the fine or coarse diamond burs and sandblasting were reported to provoke crack initiation and propagation within the porcelain.^{11,13} Since the restorations usually remain in the mouth after debonding the brackets, porcelain damage due to extreme roughening of the surfaces during pretreatment or debonding must be avoided.^{4,7}

Hydrofluoric (HF) acid and acidulated phosphate fluoride gel (APF gel) were reported to facilitate micromechanical retention.^{4,14-16} Both acids can etch glass or porcelain and thus create a mechanically retentive surface.¹⁷ Nelson and Barghi¹⁸ found that an APF gel etch produced bond strength comparable to an HF acid etched control. In their study, a 10-minute etch produced the highest bond strength for APF gel whereas the control was etched 1-minute with 10%

HF acid. Despite its effectiveness, the hazard of HF acid are well recognized. Mucosal contact with HF can cause erythema and burning associated with loss of tissue, along with intense pain for several days.¹⁹⁻²¹ APF gel (1.23% fluoride) is a topical fluoride gel commonly used in dental offices and dental divisions in rural hospitals. It is safe for oral tissue and therefore can probably be substituted for a harmful HF acid to etch porcelain before bonding to bracket.

To our knowledge, there is no published study available in the searched data base which directly investigates the bond strength of metal bracket to porcelain surface prepared by 1.23% APF gel as compared to silane coupling agent. The purpose of this study was to compare the shear-peel bond strength of metal bracket to the prepared porcelain surface with 1.23% APF gel and silane.

Material and methods

Seventy-two cylindrical porcelain disks (diameter: 10 mm; thickness: 4 mm) were made from a conventional feldspathic porcelain, Vita VMK95 (VITA Zahnfabrik, Bad Sackingen, Germany). Twelve specimens were fired each time according to the manufacturer's recommendations which resulted in 6 different production groups. All porcelain disk samples were unglazed with green stone burs under water coolant to create a flat surface. Disks were viewed at 20x magnification with a stereomicroscope (EMZ-TR, Meji Techno Co.,Ltd., Japan) to ensure that all flatten surfaces were free from defects such as cracked line, pit and fissure.

Two disks were randomly selected from each group. A total of 12 specimens were subjected to Vicker's microhardness test on a microhardness testing machine (Microhardness tester, FM-700e, Ser. No. FMX 0074, Future-Tech Corp., Japan). Three indentations were made on the top surface of each specimen using 1000 g load and a dwell time of 15 seconds. The mean

value was calculated as Vicker's Hardness Numbers (VHN). For statistical analysis, Kruskal-Wallis test was used. The test was performed at the 95% level of confidence. This procedure was made to confirm that six groups of porcelain disks had the same mechanical property although these specimens were baked in different production time.

The sixty disks were randomly divided into 2 groups (30 samples each); Group 1: the control group; Group 2: the experimental group.

Group 1: Specimens were ultrasonically cleaned in distilled water for 10 minutes and dried with oil-free compressed air. Silane solution (Porcelain Primer, Ormco Corp., USA) was applied over the porcelain specimen for 1 minute according to the manufacturer's instructions. The specimens were then rinsed with water spray and dried with oil-free compressed air.

Group 2: Disks were etched with 1.23% APF gel (Topical Fluoride Gel, Pascal Corp., USA) for 10 minutes, rinsed with water spray and dried with oil-free compressed air. Each sample was then ultrasonically cleaned as previously described.

A total of 60 metal brackets for maxillary central incisor with bracket base area of 15.25 mm² (DynaLock™, 3M Unitek, USA) were bonded to both conditioned porcelain surface with a light-curing adhesive resin (Transbond XT Light Cure Adhesive Paste, 3M Unitek, USA). The bracket was seated and positioned under manual pressure at the center of the disk with an orthodontic bracket applicator. The excess of resin around the bracket was removed using an amalgam carver. The adhesive resin was polymerized for 10 seconds each on the mesial and the distal aspects of the bracket with a Spectrum800 visible light-curing unit (Dentsply, USA). The output of the light-curing unit was measured after using with each

specimen by a curing radiometer to ensure a minimum irradiance of at least 550 mW/cm². The same operator performed all bracket bonding procedures to minimize variation.

Each specimen was embedded into a self-curing acrylic resin prefabricated mount which retained in a PVC ring with 48 mm. in diameter and 38 mm. in height for testing. The specimens were stored in distilled water at 37°C for 24 hours. At the end of the storage period, the brackets were debonded with a shear-peel load in an Instron Universal Testing Machine (Instron ID 5566H1612, Instron Corp., USA) with a crosshead speed of 0.5 mm/min. A static load cell with 250 N was used to measure the maximum force required to dislodge the bracket. Shear-peel bond strength in MPa was determined by dividing the shear-peel force value (N) with the nominal bracket base area (mm²). Shear-peel bond strength in MPa was determined for each specimen.

After debonding, the porcelain surfaces were examined with a stereomicroscope at a magnification of 10 to determine the amount of composite resin remain according to the adhesive remnant index (ARI).²² The ARI scale has a range from 0 to 4, with 0 indicating no resin left on the porcelain; 1 = less than half of the resin left; 2 = more than half of the resin left; and 3 = all resin retained with bracket imprint. The index was modified by including a score of 4 for samples with a damaged porcelain surface.²³

Statistical analysis of the data included the calculation of the mean shear-peel bond strength and standard deviation for each group. Data were analyzed by independent sample T-test for mean bond strength and Weibull test for examining the probability of failure. Chi-square test was used to evaluate differences in the ARI scores between groups. Statistical significance were tested at $p = 0.05$.

Results

The mean surface hardness values from six groups of fired porcelain are shown in Table 1. There was no statistically significant difference among the six groups ($p = 0.540$).

The mean shear-peel bond strength, standard deviation (SD), minimum, and maximum values for each tested group are depicted in Table 2. Average strengths with SD were 9.42 ± 1.93 MPa and 9.68 ± 1.91 MPa for APF gel group and silane group, respectively. No statistically significant difference in mean shear-peel

bond strength was observed between APF gel group and silane group ($p = 0.604$).

Weibull analysis was used to examine the probability of failure and the results are shown in Figure 1. The curves consist of the cumulative probability of bond failure plotted against applied load. The probability of bond failure at 9.68 MPa (147.63 N) was calculated for each group as this was the mean debonding force required to debond the control group. The probabilities of bond failure at 9.68 MPa were 47.2 percent for silane group and 52.8 percent for APF gel group.

Table 1 Mean surface hardness value from six groups of fired porcelain

Firing groups (12 disks/cycle)	Specimens (n)	Total indentations	VHN Mean \pm SD
1	2	6	499.18 \pm 8.88
2	2	6	506.35 \pm 19.77
3	2	6	510.38 \pm 17.77
4	2	6	502.86 \pm 20.95
5	2	6	504.32 \pm 15.72
6	2	6	488.21 \pm 25.03

Kruskal-Wallis test, $p = 0.540$

VHN = Vicker's hardness number

Table 2 Shear-peel bond strength, in MPa

Treatment	Shear-peel bond strength (MPa)		
	Mean \pm SD	Max.	Min.
APF gel	9.42 \pm 1.93	14.212	6.144
Silane	9.68 \pm 1.91	14.225	6.980

T-test = -0.521 , $p = 0.604$

APF gel = acidulated phosphate fluoride gel

MPa = megapascal

Max. = maximum

Min. = minimum

The ARI scores for the two tested groups are presented in Figure 2. The results of chi-square comparisons for the ARI indicated that there was significant difference ($p \leq 0.001$) between the groups treated with silane and APF gel. With the use of silane,

there were higher frequencies of ARI scores of 3 and 4, which indicated that more composite resin was remained on the porcelain surfaces and higher percentages of damaged porcelain surfaces.

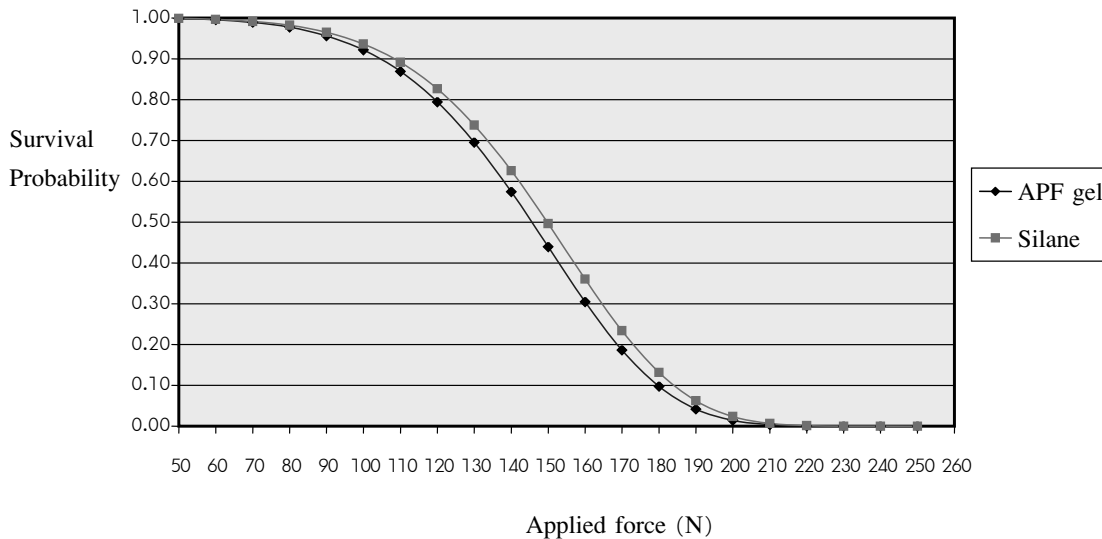
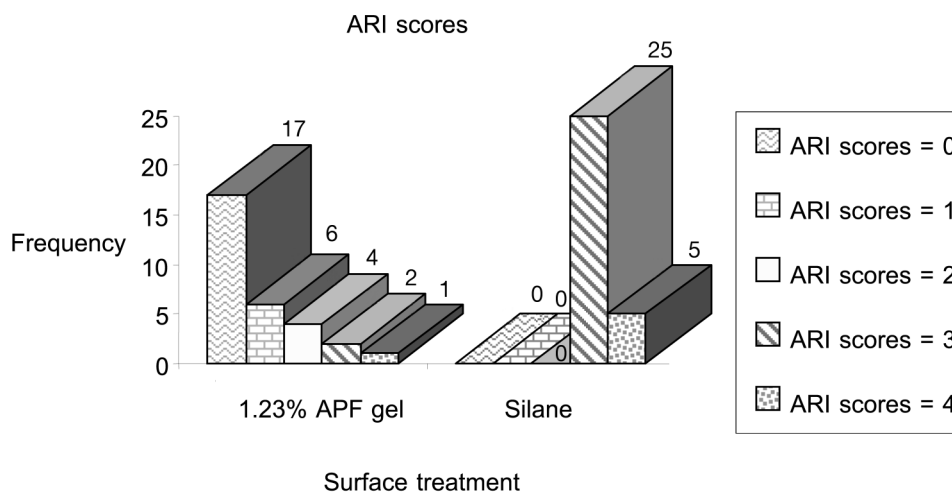


Figure 1 Weibull curves for brackets bonded on the surface treated with APF gel ($\alpha = 155.23, \beta = 5.70$) and silane ($\alpha = 159.44, \beta = 5.83$).

APF gel = acidulated phosphate fluoride gel



Chi-square = 32.167, df = 4, $p \leq 0.001$

Figure 2 Frequency distribution of the ARI scores.

ARI = adhesive remnant index

APF gel = acidulated phosphate fluoride gel

Discussion

All porcelain disks had the consistent mechanical property as seen from the surface hardness values. From literature, it is rarely to find studies that calibrated porcelain samples by their mechanical properties. Most studies had shown merely geometric characteristics such as size, shape, dimension or color.^{24,25} Describing the porcelain samples by their geometric characteristics may not be sufficient for calibration of the porcelain samples which could be much varied in every production step.

The mean bond strength of APF gel group is about 9.42 MPa. Preparation with APF gel produced lower bond strength than using silane although there was no significant difference of the shear-peel bond strength between the two groups. It is likely that the bond strength of APF gel group could be derived from micromechanical retention whereas the other group's came from chemical retention. Many studies reported that a scanning electron microscopy of the porcelain etched with APF gel revealed a relatively smooth homogenous surface compared with the HF acid etched^{17,26-28} because a low concentration of HF acid produced by APF gel created a micromechanical retentive surface only on the superficial layer of the porcelain.²⁹ However, acid precipitates still deposited on the surface after acid treatment.^{26,29} Ultrasonication on the surface will be effective in removing these precipitates, whereas rinsing with distilled water only will result in the fluorine containing precipitates remaining on the surface.²⁶ According to this reason, we used ultrasonic cleaning after APF gel treatment. However, during intra-oral bonding procedure, ultrasonic cleaning is impossible, so the precipitates remain on the surface. On the other hand, we cleaned porcelain specimens by ultrasonic wave before silane was applied in order to create a fresh bonding surface for hydrolysis and adsorption of silane on ceramic surface. Reynolds³⁰ reported that a bond strength of 6-8 MPa was adequate for routine clinical use. In

addition, Thrumond et al.³¹ reported that the visual examination of the debonded specimens generally showed cohesive failures in porcelain for the treatment groups with a mean bond strength above 13 MPa. Therefore, the bond strength determined in this study is probably acceptable for clinical usage. This result disagrees with previous studies that bond strengths of APF gel treated surface for 2, 4 and 5 minutes, either glazed or unglazed porcelain, were inadequate to routine orthodontic therapy.^{9,16,23}

Whether or not, deglazing the porcelain before orthodontic bonding remains controversial. Several studies advocated removal of the glaze in order to create mechanical retention for the adhesive agent.^{3,9,10,17} Zachrisson et al.⁹ explained that the felspathic porcelains may sometimes have an alumina overglaze, which cannot be distinguished. In this case, silane will be ineffective because it will not enhance bonding to porcelains which contain only a small amount of silica. Careful removal of the glaze in the area of the bonding will increase the surface area available for chemical or mechanical retention. Thus, for this reason we removed the glaze. On the contrary, some studies have shown that a satisfactory bond strength to glazed ceramic can be achieved.^{1,7,32}

Thermocycling of at least 500 cycles is required to test the bond strength of brackets to ceramic because of artificial aging and also the different thermal expansion coefficients among ceramic, resin, and metal.³ The temperature change can also contribute to water contamination at the bond interface and weaken the resin over a long time. Thermocycling usually has a significant effect on the bond strength, the bond values decrease when thermocycling is applied.^{3,9,13,14} Further researches are suggested to investigate this factor.

Fox et al.³³ suggested that all bond strength testing should include some form of survival analysis. This gives the clinician a better idea of how the material or bracket is likely to perform in the clinical situation.

Weibull analysis was used to calculate the probability of failure at given values of applied force. A higher Weibull shape parameter ($\beta = 5.83$) was recorded with the silane group, indicating greater bond reliability with this surface treatment compared with the APF gel treatment ($\beta = 5.70$). The Weibull curves indicate that for a given survival probability, less force would be required to dislodge a bracket bonded to porcelain with APF gel treatment compared with one treated with silane. However, similarity in shape of two curves was observed. This result indicates that the two treatment methods may behave similarly in the clinical situation with respect to bond reliability and bond failure.

The factor that primarily affects ARI scores is probably the type of surface preparation. A significant frequency of ARI score in APF gel group was the incidence of no resin left on the porcelain, meaning that APF gel group had lower bond strength between brackets and porcelain surface. This finding exhibited adhesive failure between porcelain and adhesive resin that were observed mainly in the APF gel group. Adhesive failure is preferred to avoid ceramic fracture during debonding.³ From a debond perspective, this would be an advantage as less clinical time would have to be spent removing residual resin, thereby the clinician can save chairtime and easily clean without damage to the porcelain surface. From our study, porcelain surface damage at debonding was found in one specimen, therefore, patients should be warned about the risk of the damage to porcelain surface and the need for possible repair or replacement after orthodontic treatment.

In the present study, we only tested the 10 minutes of 1.23% APF gel application to the porcelain surface. This treating time is quite long for orthodontists and patients, but the clinicians can prepare archwire or other works in the mean time. We suggest that this method can be used when bonding for few brackets is required. Future studies to evaluate the most effective treating

time which may range from 6 to 9 minutes should be done and clinical trials are necessary for testing its efficacy.

Conclusions

Within the limitations of this in vitro study, no significant difference in shear-peel bond strength was observed between APF gel and silane treatment. The application of 1.23% APF gel for 10 minutes can probably be an alternative of surface treatment for bonding metal bracket to porcelain surface after unglazed with a green stone bur.

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การศึกษากำลังแรงยึดแบบเขื่อน-ปอกของ แบรกเกตโลหะต่อพอร์ซเลนซึ่งผ่านการ ปรับสภาพผิวด้วยวุ้นแอสิดูเลตฟอสเฟต ฟลูออไรด์ความเข้มข้นร้อยละ 1.23

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บทคัดย่อ

วัตถุประสงค์ เพื่อเปรียบเทียบค่ากำลังแรงยึดแบบเขื่อน-ปอกของแบรกเกตโลหะต่อพอร์ซเลนซึ่งผ่านการปรับสภาพผิวสองวิธี ระหว่างไซเลนและวุ้นแอสิดูเลตฟอสเฟตฟลูออไรด์ (เอฟไอเอฟ) ความเข้มข้นร้อยละ 1.23

วัสดุและวิธีการ นำแผ่นพอร์ซเลน จำนวน 60 แผ่น (ขนาดเส้นผ่าศูนย์กลาง 10 มม. หนา 4 มม.) มาขจัดผิวเคลือบด้วยหัวกรอหินสีเขียวก่อนแบ่งเป็นกลุ่มอย่างสุ่ม 2 กลุ่ม (กลุ่มละ 30 ชิ้น) กลุ่มที่ 1 (กลุ่มควบคุม) ปรับสภาพผิวพอร์ซเลนด้วยไซเลน ส่วนกลุ่มที่ 2 ปรับสภาพผิวพอร์ซเลนด้วยวุ้นแอสิดูเลตฟอสเฟตฟลูออไรด์ความเข้มข้นร้อยละ 1.23 นาน 10 นาที หลังจากนั้นนำชิ้นตัวอย่างทั้งหมดมาติดแบรกเกตโลหะที่ฟันตัดซี่กลางบนยึดด้วยวัสดุเรซินชนิดบ่มด้วยแสงและนำไปแช่ในน้ำกลั่นอุณหภูมิ 37 องศาเซลเซียส นาน 24 ชั่วโมง ก่อนการทดสอบแรงยึดด้วยเครื่องทดสอบแรงทั่วไปอินสตรอน กำหนดตุ้มน้ำหนัก 250 นิวตัน ที่อัตราเร็ว 0.5 มม. ต่อนาที วิเคราะห์ความแตกต่างระหว่างค่าเฉลี่ยกำลังแรงยึดแบบเขื่อน-ปอกของทั้งสองกลุ่มโดยใช้สถิติอินดีเพนธ์เด็นท์ แซมเปิล ที-เทสที่ระดับนัยสำคัญ $p = 0.05$ และวิเคราะห์ความน่าจะเป็นของการอยู่รอดของแบรกเกตด้วยการวิเคราะห์ไวนบูลล์

ผลการศึกษา ไม่พบความแตกต่างอย่างมีนัยสำคัญทางสถิติระหว่างกำลังแรงยึดแบบเขื่อน-ปอกของกลุ่มที่ปรับสภาพผิวพอร์ซเลนด้วยวุ้นเอฟไอเอฟ (9.42 ± 1.93 เมกะปาสคาล) และกลุ่มที่ปรับสภาพผิวด้วยไซเลน (9.68 ± 1.91 เมกะปาสคาล) ($p = 0.604$)

สรุป วุ้นเอฟไอเอฟความเข้มข้นร้อยละ 1.23 สามารถนำมาใช้เป็นทางเลือกในการปรับสภาพผิวพอร์ซเลนก่อนการติดแบรกเกตโลหะ หลังผ่านการขจัดผิวเคลือบของพอร์ซเลนด้วยหัวกรอหินสีเขียวแล้ว

(จ.ทันต. จุฬาฯ 2553;33:109-18)

คำสำคัญ: การปรับสภาพผิวพอร์ซเลน; การวิเคราะห์ไวนบูลล์; กำลังแรงยึดแบบเขื่อน-ปอก; แบรกเกตโลหะ; วุ้นแอสิดูเลตฟอสเฟตฟลูออไรด์