SHEAR BOND STRENGTH AND FAILURE MODE OF CERAMIC BRACKETS TO GLAZED FELDSPATHIC PORCELAIN USING DIFFERENT BONDING PROTOCOLS (IN VITRO STUDY)
Mohamed M. El-Ramly*, Mohamed I. Mowafy, PhD; Sherief H. Abdelhafiez, PhD

ABSTRACT

OBJECTIVE: To evaluate the shear bond strength (SBS) of ceramic brackets bonded to feldspathic porcelain using different bonding protocols.

MATERIAL AND METHODS: 30 porcelain fused to metal (PFM) disks were fabricated and randomly divided into three equal groups of 10 each. Each group had a different surface treatment as follows: - Group A: air abrasion, etching with hydrofluoric acid (HF), silane and Reliance Assure Plus (RA plus). Group B: air abrasion, silane and RA Plus (manufacturer's recommendation). Group C: air abrasion and RA Plus. Ceramic brackets were bonded to PFM discs using light cure-composite resin. Following thermocycling, SBS was measured using a universal testing machine. After bracket debonding, Failure mode was measured under stereomicroscope.

RESULTS: Group A and group B showed the highest SBS value with a mean of 8.86 ± 1.99 and 10.32 ± 4.39 MPa respectively with no statistically significant difference among them. However, group C showed statistically significant lower SBS with a mean value of 1.84 ± 0.74 Mpa. Failure modes of groups A and B were predominantly at the adhesive / bracket interface. Meanwhile, group C showed mixed adhesive / porcelain interface and cohesive resin failure.

CONCLUSION: Air abrasion and silane resulted in clinically acceptable bond strength among the groups regardless the use of HF. Silane appears to be integral in achieving adequate bond strength. The universal primer used in the current study can not be used as a substitute to silane.

KEYWORDS: Feldspathic porcelain, Ceramic Brackets, Shear Bond Strength, Reliance Assure Plus.

INTRODUCTION

The increased esthetics of newer orthodontic appliances contributed, among other reasons, to the increase of the number of adult patients seeking orthodontic treatment. Providing orthodontic treatment for adult patients poses many problems to the orthodontist; one of which is the possibility of having to bond attachments to restored teeth. Ceramic crowns are considered one of the methods of restoring damaged teeth.

Various types of ceramics are currently used. They could be classified into silica based like feldspathic, leucite, and lithium disilicate, and non-silica based like zirconia. Porcelain fused to metal (PFM) crowns are well-established means of restoration of badly destructed teeth. Despite the emergence of more esthetic alternatives, it is still widely used for its acceptable esthetics and reasonable price. In addition, owing to the high esthetic value and great ability to mimic natural tooth shades, feldspathic porcelain is used in the fabrication of anterior veneers as well as being used as a veneering material over zirconia cores in all ceramic crowns and bridges.

Ceramic brackets were introduced in the 1980s; they quickly gained popularity for their unmatched esthetics and high efficiency. They were able to overcome the shortcomings of their less esthetic predecessors (polycarbonate brackets), which suffered from plastic deformation and color instability. Ceramic brackets have higher bond strength than stainless steel brackets with lower clinical failure rate. The downside to this is the probability of fracture of the substrate (enamel or porcelain) especially while debonding the brackets.

Bonding to porcelain is considered challenging due to the inert nature of the material.
The porcelain surface does not respond to etching with phosphoric acid; however, it requires conditioning to alter the surface characteristics. The surface treatment aims to create micro roughness that increases the surface area thus enhancing the bond strength. This could be achieved either chemically, mechanically or by both methods. The main method to chemically roughen the ceramic surface includes etching with hydrofluoric acid (HF) to create micro-porosities that aid in the micromechanical retention of the adhesive. HF dissolves the silica in the ceramic forming a porous surface that increases the surface area and enhances bond strength. Two recent systematic reviews considered etching with 9.6% HF for one minute followed by silane application as the gold standard of ceramic bonding. However, it should be used with caution for its hazardous effects on soft tissue. Trakyali et al. conducted a study testing lower concentrations of HF and concluded that there was no statistically significant difference between 5% and 10% HF.

Air abrasion and grinding with diamond burs are among the mechanical methods to alter the porcelain surface. Removal of the surface glaze with air abrasion not diamond burs have been advocated by a few authors as diamond burs may produce micro cracks; in addition, air abrasion removes only a small amount of the surface and is more uniform. Air abrasion involves pressurized blasting of aluminum oxide particles on the ceramic surface to increase the surface area and create a micro retentive surface, which is required for mechanical retention of the adhesive.

In addition to roughening, one of the chemical agents used is silane which has a bifunctional group that couples the inorganic silica in the porcelain to the organic groups of the bonding resin. Moreover, it increases the wettability of the porcelain surface. Contradictory reports are present regarding the effectiveness of using silane alone on the bond strength. Karan et al. recommended the use of air abrasion and HF without silane as a method of surface preparation. On the contrary, most studies recommended the application of silane after roughening of the porcelain surface.

Universal primers are now available that can bond to different substrates. One of the monomers used is 10-Methacyloxyalkyd dihydrogen phosphate (MDP), which is a bifunctional phosphate monomer that allows for the formation of a chemical bond between the resin of the adhesive and the ceramic. Reliance assure plus (RA plus) is one of these MDP containing primers. The efficiency of RA Plus in bonding orthodontic metallic brackets to feldspathic porcelain was tested by Mehta et al. Feldspathic porcelain samples were first sandblasted and silane and RA plus were applied. Tensile bond strength testing showed that RA plus was able to achieve bond strengths comparable to other conventional bonding protocols.

Naseh et al. investigated the effect using RA plus without silane on bonding metal brackets to feldspathic porcelain and compared it to Transbond primer. The first group represented the conventional protocol; the samples were sandblasted and etched with 9.6% HF for 2 minutes followed by the application of silane and Transbond primer. In the second group, the same was repeated without the application of silane and RA plus was applied instead of Transbond. The samples were stored in 37°C water for 24 hours and thermocycled for 2000 cycles. The results showed that the mean shear bond strength (SBS) for both groups was 15.61 and 15.71 MPa respectively for feldspathic porcelain. The authors concluded that RA plus yielded high bond strengths without the need for silane application.

Studies that compare the SBS of ceramic brackets to feldspathic porcelain using RA plus universal primer are scarce. The purpose of this study is to evaluate the SBS of ceramic brackets to glazed feldspathic porcelain using RA plus with different bonding protocols. In addition, the failure mode was measured.

The null hypothesis is that there is no difference between the different bonding protocols in the SBS of ceramic brackets bonded to glazed feldspathic porcelain.

MATERIALS AND METHODS
The sample size was calculated using G Power computer software version 3.1.9.2 (Universität Kiel, Germany). Based on a study done by Mehta et al. that aimed to investigate different bonding procedures for metal orthodontic attachments to feldspathic porcelain and zirconia surfaces with a mean of 4.657 MPa and standard deviation of 0.6020 MPa. At a level of significance 5% (α=0.05) and a power of 90% was adopted. The minimal required sample size was found to be 10 discs per group (number of groups =3), (total sample size = 30 discs).

Thirty feldspathic porcelain disc (G-ceram, Atlas-Enta, Izmir-Turkey) with dimensions of 10 mm in diameter and 2 mm in thickness (1.5 mm feldspathic porcelain and 0.5 mm cobalt-chromium metal frame work) were fabricated to mimic the surface of an upper central incisor. A glaze layer (G-ceram, Atlas-Enta, Izmir-Turkey) was applied according to manufacturer’s recommendations on the surfaces of the discs, which were then fired in a ceramic furnace.

Metallic molds of 30 mm length and 20 mm internal diameter were fabricated. The inner surface of the ring was lightly coated with petroleum jelly to facilitate easy removal of the
acrylic molds. The molds were then filled with auto polymerizing acrylic resin and the disks were then embedded into the center of the unset self-cure acrylic resin. The acrylic resin was allowed to set before removal from the metal rings (Fig. 1).

All the samples underwent prophylaxis using a low-speed rotary instrument with a rubber cup and oil-free pumice. This was followed by rinsing and drying with oil free air. Micro etcher intraoral sandblaster (Danville Materials, San Ramon, CA, USA) was used to air abrade all the samples with 50μm Al₂O₃ at 40 psi for 5 seconds at a distance of 5mm. following air abrasion the samples were rinsed and dried with oil free air, then randomly divided into three groups according to the surface preparation.

Group A: 4% HF (Porc etch, Reliance Orthodontics Products, IL, USA) was applied for 2 minutes, rinsed for 15 seconds, followed by drying with oil-free air. Following that, two layers of Silane (Porcelain Conditioner, Reliance Orthodontics Products, IL, USA) were applied then left to dry for 90 seconds. Finally, a single layer of RA Plus (Reliance Orthodontics Products, IL, USA) was applied with a micro-brush, compressed with air, and then light cured using a light-emitting-diode light curing unit (Radii plus light curing unit, SDI Limited, Bayswater Victoria AU) for 20 seconds.

Group B: only silane was applied without HF followed by RA plus as previously mentioned.

Group C: a single layer of Assure Plus was applied without HF or silane.

Following surface preparation, 30 upper central incisor monocrystalline ceramic brackets (Perfect clear sapphire, Hubit Products Co. Ltd., Dongan-gu, Republic of Korea) with bracket base dimension of 3.2 X 3.8mm were bonded using Transbond XT adhesive (3M Unitek, Monrovia, CA, USA). A force of 200 g was applied to the bracket using a pressure gauge. Any excess material was removed using the tip of a sharp examination probe. Then cured using LED curing light for 20 seconds on the mesial and distal sides of the bracket.

All specimens were stored at 37°C in Distilled water for 24 hours. The samples were then subjected to thermocycling for 2500 cycles in water between 5°C and 55°C(26) the exposure to each bath was 20 seconds and the dwell time was 10 seconds between the two baths.

SBS was then measured using an Instron Universal Testing Machine (Model 3345; Instron Co., Norwood, MA, USA) at a 1mm/min crosshead speed. The specimens were oriented so that the stainless steel blade was perpendicular to the bracket (Fig. 2). The load at which failure occurred was recorded for each specimen in Newton (N). The SBS was calculated according to the following equation: $SBS (MPa) = \frac{\text{Force (N)}}{\text{Area (mm}^2\text{)}}$

Following shear bond testing, all samples were examined using an optical microscope (Stereomicroscope) at 10x magnification for failure mode measurement.

Mode of failure was measured at the site of bond failure and categorized based on the method of Naseh et al. (23) as follows:

Cohesive in the porcelain (CP): Fracture or crack within the porcelain

Adhesive at the porcelain interface (AP): No fracture occurred in the porcelain. No adhesive remained on the porcelain surface; the entire adhesive remained on the bracket base.

Cohesive in resin (CR): Some resin remained on the porcelain surface and some on the bracket base.

Adhesive at the bracket interface (AB): The entire resin remained on the porcelain surface.

The statistical analysis was performed using Statistical Package for Social Sciences software, version 25(SPSS Inc., Illinois, Chicago, USA ). Normality of the SBS was detected using descriptive statistics (histograms and box plots), and Shapiro Wilk test. One Way ANOVA followed by Tukey’s test with Bonferroni correction was applied to compare between the groups regarding SBS. Mode of failure was compared by Monte Carlo modification of Chi Square test. Followed by post-hoc. Significance level was set $p$ value of 0.05.

Fig. (1): Feldspathic porcelain disc embedded in self-cure acrylic resin cylinder.
RESULTS

Shear bond strength

Group A (air abrasion + HF + Silane + Assure Plus) and group B (air abrasion + silane + Assure Plus) showed the highest mean SBS, which was 8.86 ± 1.99 MPa, and 10.32 ± 4.39 MPa respectively. On the other hand, group C (air abrasion + Assure Plus) showed the lowest mean SBS 1.84 ± 0.74 (Table 1). One-way ANOVA showed statistically significant difference in the SBS between the groups (F=37.025, P<0.0001). Tukey’s test with Bonferroni correction showed that, when compared to each other, groups A and B had no statistically significant difference in SBS (P =0.364). On the contrary, group C showed statistically significantly lower SBS when compared to both groups (P< 0.0001).

Failure mode

None of the specimens showed bracket fracture. Stereo micrographs of the predominant failure modes in each group are shown in (Fig. 3). Groups A and B showed adhesive failure that is at the bracket interface, with only a single specimen in group A showing cohesive porcelain failure. Furthermore, in group C 60% of the samples showed failure that is adhesive at porcelain interface (AP) and the remaining 40% showed cohesive resin failure (CP) (Fig. 4). Monte Carlo modification of Chi Square test showed a statistically significant difference between the groups (X²=16.696, P=0.001). Pair wise comparisons showed no statistically significant difference in the mode of failure when group A was compared to group B (P=1). On the contrary, when groups A and B were compared to group C there was a statistically significant difference in the failure mode (P=0.027 and P=0.009 respectively).

DISCUSSION

Bracket debonding is a major reason for lengthening orthodontic treatment. The real dilemma is when orthodontists are required to bond brackets on surfaces other than enamel, which occurs more frequently nowadays due to the rise in the numbers of adult patients demanding orthodontic treatment. Bonding requirements in orthodontics differs from that of restorative and prosthodontic dentistry, the bond is not required to be permanent; too high bond strengths are avoided since they increase the risk of restoration damage while debonding.

In this study, we attempted to answer two questions. First, is HF acid necessary for achieving adequate bond strength between resin and feldspathic porcelain? Many orthodontists are hesitant to use HF for its deleterious effects on soft
tissue.\(^{(13, 16)}\) Trakyali et al.\(^{(17)}\) conducted a study testing lower concentrations of HF and concluded that there was no statistically significant difference between 5% and 10% HF. In addition, the use of lower concentration is recommended to reduce the risk of soft tissue injury; this is why 4% HF was used in our study.

The second question is, can RA plus universal primer be a substitute to silane? Zachrisson et al.\(^{(16)}\) pointed out the importance of storage and thermocycling as an accelerated aging process and considered the results of bond strength studies that did not perform storage in 37°C distilled water and thermocycling to be invalid. Storage in water is important to detect materials that cannot resist the wet conditions of the oral environment. Additionally, thermocycling mimics the oral temperature fluctuations, which negatively affects the bond strength.\(^{(29)}\) There is a lot of debate regarding the number of cycles that should be performed, which ranged from 100 to 20,000 cycles.\(^{(30, 31)}\) The International Organization for Standardization\(^{(32)}\) recommended a minimum of 500 cycles in water between 5 and 55°C. Many authors suggested that higher number of cycles are needed to truly mimic the oral environment.\(^{(16, 33)}\) Fox et al.\(^{(34)}\) shed the light on the lack of standardization in bond strength studies. Which explains why it is difficult to compare the results of different studies as many variables could affect the bond strength.

The mean SBS of group A (air abrasion + HF + Silane + RA plus) was 8.86 ± 1.99 MPa. Many authors recommended the same protocol for conditioning feldspathic porcelain. In agreement with our study, Türkkahraman et al.\(^{(35)}\) reported SBS of 10.45 MPa with the same regimen; they air abraded the samples with aluminum oxide and etched them with 9.6% HF for 2 minutes finally, silane was applied. Following surface preparation, ceramic brackets were bonded using light-cured composite adhesive. Utilizing the same surface preparation, Elekdag et al.\(^{(36)}\) reported a slightly higher SBS of 15.9 MPa. This higher SBS could be attributed to the silane coated ceramic brackets used in this study, which makes these brackets chemically retained compared to the mechanically retained brackets used in our study. In addition, the samples were thermocycled for only 500 cycles compared to 2500 cycles in our study. On the contrary, Cevik et al.\(^{(26)}\) reported a low mean SBS of 2.71 MPa with the same protocol. The authors explained that the base characteristics of the brackets used in the study could be the reason of the low SBS, this explanation could be also confirmed by the low ARI scores reported in the study, which indicates the lack of adequate adhesion between the adhesive and the bracket.

In Group B (air abrasion + silane + RA plus) the same surface preparation was made as in group A without the application of HF. The mean SBS was 10.32 ± 4.39 MPa. Many authors reported high bond strengths with the same protocol, Cevik et al.\(^{(26)}\) reported SBS of 8.58 MPa with a similar specimen preparation. The samples were sandblasted with aluminum oxide, silane was applied and ceramic brackets were bonded using light cure composite resin. Storage in 37°C water and thermocycling for 2500 cycles was performed. The previous results were also confirmed by Wang et al.\(^{(37)}\) who reported a SBS of 10.98 MPa with a similar protocol, the thermocycling in this study was 500 cycles. Jamal et al.\(^{(38)}\) evaluated the effectiveness of RA plus in bonding ceramic brackets to feldspathic porcelain. The samples were roughened by a diamond bur, silane was applied followed by RA plus. The authors reported a high SBS of 17.29 MPa and conclude that RA plus yielded clinically acceptable bind strength. The samples in this study were stored in water for a week with no thermocycling that could explain the higher SBS compared to our study. On the opposite end of the spectrum, Türkkahraman et al.\(^{(35)}\) reported low SBS of 5.46 MPa with the same protocol. The authors concluded that air abrasion followed by silane application resulted in below than acceptable SBS and is not recommended for clinical use.

In group C (air abrasion + RA plus) it was tested if RA plus could serve the same function as silane. This group showed a mean bond strength of 1.84 ± 0.74 MPa. Which was significantly lower compared to the other groups. In line with our findings, Köcedereli et al.\(^{(39)}\) reported low bond strength with the same protocol and did not recommend its use clinically. Tahmasbi et al.\(^{(40)}\) evaluated the SBS of metal brackets to feldspathic porcelain bonded with a universal primer (Scotchbond™ Universal adhesive). The samples were first roughened with bur and etched with 9.6% HF for 90 seconds. Silane was applied to half the samples followed by the application of the universal primer. The specimens were stored in distilled water for 24 hours and thermocycled for 500 cycles. The results showed that the mean SBS was 12.7 MPa when silane was used and 4.4 MPa in the group where the universal primer was used alone. The authors concluded that the bond strength is highly dependent on the presence or absence of silane. In addition, the universal primer alone did not yield acceptable bond strength. On the other hand, a high bond strength was reported by Isolan et al.\(^{(41)}\) who tested the bond strength of resin to feldspathic porcelain using a universal primer (Scotchbond Universal). The porcelain was etched with 10% HF and the universal primer was applied. The samples were stored in distilled water for 24 hours. The mean SBS was 29.0 MPa. Similarly, Aboushady et al.\(^{(41)}\) evaluated the SBS of ceramic brackets to Lithium disilicate using RA plus similar...
to the protocol used in our study. The authors reported a high mean SBS of 10.27 MPa. Direct comparison of our results with the results of the previous studies would be difficult owing to the different methodology used in our study and these studies.

Reynolds(62) reported that a bond strength of a minimum of 6 to 8 MPa is required to be able to withstand orthodontic forces. In our study, the mean SBS of groups A and B lied in the acceptable range. Meanwhile the SBS of group C was lower than the acceptable bond strength.

The failure mode is an important adjunct to bond strength testing, since it enables us to better comprehend the quality of the bond strength. Zachrisson et al.(40) pointed out that it is only possible to measure the true bond strength of any adhesive if the failure is in the adhesive layer. After finishing treatment, it is of utmost importance to maintain a sound restoration and to be able to restore the surface to its original luster. There are pros and cons to having the failure at the bracket base or at the porcelain surface. Failure at the bracket base will leave most of the adhesive at the surface of the restoration, which will ensure that the surface remains intact; the downside is that considerable chair time will be needed to remove the residual adhesive. Meanwhile, failure at the porcelain surface will leave less amount of adhesive that will be easier to remove, however it increases the possibility of porcelain fracture.(43)

The failure mode of group A was predominantly adhesive at the bracket interface only one sample showed cohesive porcelain fracture. Thurmond et al.(46) stated that bond strengths higher than 13 MPa could induce cohesive porcelain fracture. Although none of the samples in this group had SBS higher than the 13 MPa threshold. Porcelain fracture may be attributed to microcracks present in this specific specimen and cannot be generalized on the whole study. Failure at the adhesive bracket interface indicates that the bond strength of the adhesive to ceramic exceeded the mechanical interlocking provided by the bracket. Utilizing the same protocol but with metal brackets, Trakyali et al.(17) reported similar failure mode with most of the samples remaining on the porcelain surface. Elekdag et al.(36) reported a different debonding pattern where most the failure was at the adhesive porcelain interface. In line with the previous study, Cevik et al.(26) found that failure was mainly at the adhesive porcelain interface. As shown from the results of the fore mentioned studies there is no agreement between the authors regarding Failure modes, which could be attributed to the great variety of brackets used in these studies with different base designs.

Similarly, all the samples of group B showed failure at the adhesive bracket interface. Using the same surface preparation with polycarbonate and metal brackets Ozcan et al.(46) and Zhang et al.(47) found that most of the adhesive remained on the porcelain surface after debonding. The previous findings were contradicted by Cevik et al.(26) who reported failure mostly at the adhesive porcelain interface. However, Wang et al.(8) reported mixed adhesive cohesive failure, lower incisor brackets were used in this study which could be the reason for this different results.

Different failure modes were seen in group C were most of the samples failed at the adhesive porcelain interface which indicates low adhesion between the adhesive and the ceramic surface. Similar findings were reported by Karan et al.(9) in a study done with metal brackets. Aboushady et al.(41) and Tahmasbi et al.(39) reported different results with most the samples showing failure that was mostly at the bracket adhesive interface. However, Isolan et al.(40) reported that most the samples had cohesive porcelain failure. differences in the methodology used makes it difficult to do direct comparison between the previous studies and our study regarding failure mode.

The oral environment presents a number of parameters that are difficult to reproduce in the lab. For instance, extreme PH fluctuations, humidity, oral microflora that were shown to be able to degrade the orthodontic adhesive.(48) Temperature variations, which is not well reproduced even with thermocycling as individuals are not pound to strict thermal alterations throughout the day. Clinically, brackets face stresses in all directions, which is not the case in invitro studies as the force is in a single direction. Failure is induced by gradually increasing the load, which is not how the brackets fail in the patient's mouth. Despite the previous, in vitro bond strength studies remain a useful tool to measure the exact magnitude of the bond strength which cannot be done in vivo. In addition, it enables us to test the quality of bond strength for further validation by in vivo studies.

**CONCLUSIONS**

Within the limitations of this *in vitro* study, the following conclusions were drawn:

Air abrasion and silane resulted in clinically acceptable bond strength among the groups. The use HF did not increase the SBS. In this study the most important factor that affected the SBS was silane. In addition, the universal primer used in the current study can not be used as a substitute to silane.

**REFERENCES**

2. Christensen L, Luther F. Adults seeking orthodontic treatment: expectations, periodontal
31. Newman SM, Dressler KB, Grenadier MR. Direct bonding of orthodontic brackets to