European Journal of Orthodontics 31 (2009) 402–406
doi:10.1093/ejo/cjn118
Advance Access publication 1 April 2009

Effects of different silanes and acid concentrations on bond strength of brackets to porcelain surfaces

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SUMMARY The aim of this study was to determine the optimum silane-coupling agent and the optimum concentration of acid agent when bonding to porcelain surfaces.

Eighty deglazed feldspathic porcelain discs with a diameter of 10 mm and a thickness of 2 mm mounted in acrylic resin blocks were randomly divided into four groups. In groups 1 and 2, the porcelain surfaces were etched with 9.6 per cent hydrofluoric (HF) acid and in groups 3 and 4 with 5 per cent HF acid. In groups 1 and 3, the Dynalock maxillary central incisor brackets were bonded with Pulpdent silane and Unite bonding adhesive and in groups 2 and 4 with Reliance silane and Unite. Shear forces were applied to the samples using an Instron universal test machine. The non-parametric Kruskal–Wallis test was used to determine significant differences in bond strengths between the four groups and Dunn's multiple comparison test to compare subgroups.

The mean bond strengths and standard deviations of groups 1 to 4 were 5.51 $\pm$ 1.19, 6.54 $\pm$ 0.002, 4.55 $\pm$ 1.93, and 6.39 $\pm$ 0.45 MPa, respectively. Specimens bonded with Reliance showed a statistically significantly higher *in vitro* bond strength than those bonded with Pulpdent. The concentration of etching gels did not result in any statistically significant difference on the *in vitro* bond strength when evaluated separately.

Introduction

With the increased demand for adult orthodontic treatment and the increased popularity of aesthetic dentistry, clinicians are often faced with the problem of bonding fixed orthodontic brackets to teeth that have different types of restorations, including fixed porcelain prostheses and veneer laminates. However, the bond strength of composite resins to ceramic restorations has often been reported to be inadequate (Zachrisson and Büyükyilmaz, 1993; Cochran *et al.*, 1997; Gillis and Redlich, 1998).

Roughening of the porcelain surface is generally regarded as compulsory for reliable bond strength (Zachrisson and Büyükyilmaz, 1993; Cochran *et al.*, 1997; Jost-Brinkmann and Böhme, 1999). Various surface roughening methods have been reported in the literature (Kocadereli *et al.*, 2001; Schmage *et al.*, 2003; Özcan *et al.*, 2004; Ajlouni *et al.*, 2005). Mechanical roughening with fine or coarse diamond stone (Barbosa *et al.*, 1995; Cochran *et al.*, 1997) burs, sandblasting (Zachrisson *et al.*, 1996; Cochran *et al.*, 1997; Andreasen and Stieg, 1998; Kocadereli *et al.*, 2001), or sandpaper discs (Barbosa *et al.*, 1995; Cochran *et al.*, 1997) are reported to be insufficient in order to establish the bond strength of composite resins to porcelain surfaces (Smith *et al.*, 1988; Shahverdi *et al.*, 1998). The purpose of mechanical alteration of the porcelain surface is to remove the glaze and roughen the surface to provide sufficient mechanical retention to bond orthodontic attachments.

Chemical preparation of the porcelain surface with hydrofluoric (HF) acid significantly increases the bond strength of orthodontic attachments (Hayakawa *et al.*, 1992; Major *et al.*, 1995; Zachrisson *et al.*, 1996; Cochran *et al.*, 1997; Bourke and Rock, 1999; Kocadereli *et al.*, 2001) but traditional phosphoric acid etching is not sufficient in the preparation of porcelain surfaces for the mechanical retention of orthodontic attachments. Previous studies have shown that chemical conditioning using a coupling agent such as silane increases the adhesion of the composite resin bond to the porcelain surface (Tylka and Stewart, 1994; Aida *et al.*, 1995; Nebbe and Stein, 1996; Zachrisson *et al.*, 1996; Cochran *et al.*, 1997; Wood *et al.*, 1997; Gillis and Redlich, 1998; Chung *et al.*, 1999). Silanes, such as gamma-methacryloxypropyl-trimethoxylane, are coupling agents developed for bonding glass fillers within polymer structures (Bowen and Rodriguez, 1962). The silane agent is a bifunctional molecule, with one end connected to the hydroxyl groups in the silica molecule of the porcelain facets and the other creating double bonds with the monomers in the adhesive. When silanating agents are used, orthodontic attachments have been shown to bond to porcelain teeth, cast crowns, and porcelain-fused-to-metal crowns (Newman, 1983; Bishara *et al.*, 2005). To improve bond strengths of composite resins to porcelain surfaces, combinations of different mechanical and chemical surface conditioning methods are recommended (Zachrisson and Büyükyilmaz, 1993; Thurmond *et al.*, 1994; Barbosa *et al.*, 1995; Kupiec *et al.*, 1996; Chung and Hwang, 1997; Zachrisson, 2000; Kocadereli *et al.*, 2001). A combination of mechanical surface preparation and etching the porcelain surface with HF acid followed by the application of primer and bonding agent is recommended (Gwinnett, 1988; Barbosa *et al.*, 1995).

The objective of this study was to determine the effect of four different surface conditioning methods on the shear bond strength (SBS) of metal brackets bonded with resin composite bonding cement to feldspathic porcelain surfaces.

Material and methods

Eighty flat-surface feldspathic porcelain discs with a diameter of 10 mm and a thickness of 2 mm were fabricated, according to the manufacturer's recommendations, with glazed surfaces. The discs were fabricated from IPS Classic porcelain (Ivoclar Vivadent AG) by the sintering technique and baked under vacuum at 920°C. To stimulate the moisture and temperature changes in the oral environment, all discs were stored in deionized water at 37°C for 30 days and then thermal cycled in deionized water at 5 ± 2°C and 55 ± 2°C for 5000 cycles. The total period of exposure to both was 10 seconds, with a dwell time of 5 seconds in each bath. The discs were than kept in distilled water at 37°C for 6 weeks before surface-conditioning procedures. The water was changed weekly. The specimens were embedded in acrylic moulds, so that only the glazed surface of the disc remained uncovered. Table 1 shows the surface conditioning and bonding methods for the specimens that were randomly divided into four groups of 20. This is the minimum number recommended for laboratory bond strength testing (Fox *et al.*, 1994).

In all groups, porcelain surface roughening was performed by sandblasting with an air abrasion device (Microetcher II Intraoral Sandblaster, Danville Engineering) filled with 50 μm aluminium oxide (Danville Engineering), from a distance of approximately 5 mm for 5 seconds. In groups 1 and 2, the porcelain surfaces were etched with 9.6 per cent HF acid gel (Porc-Etch) for 120 seconds and rinsed. The specimens in groups 3 and 4 were etched with 5 per cent HF acid gel (IPS Ceramic Etchant Gel) for 120 seconds. All specimens were washed and rinsed thoroughly to remove the residual acid and then air-dried. In groups 1 and 3, Pulpdent silane-coupling agent was used for chemical conditioning of the porcelain surface and in groups 2 and 4, Reliance silane-coupling agent. Silane was applied for 60 seconds in all groups.

Upper central incisor metal brackets with a horizontal retention groove base with a surface area of 7.6 mm^2 (Dynalock, 3M Unitek) were bonded to the prepared porcelain surfaces with a chemical cure composite resin (Unite, 3M Unitek) in accordance with manufacturer's recommendation. The excess resin was carefully removed from the porcelain surface with a dental probe. All specimens were stored in 37°C distilled water for 1 week and subjected to thermocycling before SBS testing.

All specimens were mounted in the jig of a universal test machine (Instron 3345, High Wycombe, Bucks, UK) and shear force was applied to the adhesive interface until fracture occurred. The specimens were loaded at a crosshead speed of 1 mm/minute. The force required to shear the bracket was recorded, and the bond strengths were calculated in megapascals (MPa). The sheared surfaces were further observed visually. Digital photographs of sheared surfaces were projected on a wall at ×10 magnification to assess the amount of adhesive remaining on the tooth surface by eye inspection. The adhesive remnant index (ARI) as described by Årtun and Bergland (1984) was used to score the adhesive remaining on the surface after bracket removal. ARI scores range from 3 to 0.

Table 1 Characteristics of four surface conditioning methods to porcelain.

Conditioning method		Manufacturer
Group 1		
Sandblasting	50 μm aluminium oxide, 5 seconds	Microetcher II Intraoral Sandblaster, Danville Engineering, San Ramon, California, USA
Hydrofluoric acid	9.6%, 120 seconds	Porc-Etch, Reliance Orthodontic Products Inc, Itasca, Illinois, USA
Silane-coupling agent	60 seconds	Silane Bond Enhancer, Pulpdent, Watertown, Massachusetts, USA
Bonding agent		Unite, 3M Unitek, Monrovia, California, USA
Group 2		
Sandblasting	50 μm aluminium oxide, 5 seconds	Microetcher II Intraoral Sandblaster, Danville Engineering
Hydrofluoric acid	9.6% 120 seconds	Porc-Etch, Reliance Orthodontic Products Inc.
Silane-coupling agent	60 seconds	Reliance Porcelain Conditioner, Reliance Orthodontic Products Inc.
Bonding agent		Unite, 3M Unitek
Group 3		
Sandblasting	50 μm aluminium oxide, 5 seconds	Microetcher II Intraoral Sandblaster, Danville Engineering
Hydrofluoric acid	5%, 120 seconds	IPS Ceramic Etchant Gel, Ivoclar Vivadent AG, Schaan, Lichtenstein
Silane-coupling agent	60 seconds	Silane Bond Enhancer, Pulpdent
Bonding agent		Unite, 3M Unitek
Group 4		
Sandblasting	50 μm aluminium oxide, 5 seconds	Microetcher II Intraoral Sandblaster, Danville Engineering
Hydrofluoric acid	5%, 120 seconds	IPS Ceramic Etchant Gel, Ivoclar Vivadent AG
Silane-coupling agent	60 seconds	Reliance Porcelain Conditioner, Reliance Orthodontic Products Inc.
Bonding agent		Unite, 3M Unitek

Statistical analysis was performed using GraphPad Prism Software Version 3.0 for Windows (San Diego, California, USA). Descriptive statistics, including the mean, standard deviation (SD), and minimum and maximum values were calculated for each of the four groups. The non-parametric Kruskal–Wallis test was used to determine whether significant differences were present in bond strength between the four groups and Dunn's multiple comparison test to compare subgroups. The frequency distribution of the ARI scores between the four bonding procedures tested was compared using the chi-square test. A Spearman correlation test was used to evaluate the relationship between ARI scores and bond strength measurements. Significance for all statistical tests was predetermined at $P < 0.05$.

Results

The mean SBS and SD for each group are presented in Table 2.

The lowest SBS was with 5 per cent HF acid and Pulpdent silane (4.55 MPa) and the highest with 9.6 per cent HF acid and Reliance silane combination (6.54 MPa). The results of the non-parametric Kruskal–Wallis test showed a statistically significant difference in bond strength among the four groups ($P < 0.001$).

A statistically significant differences was observed between groups 1 and 2, ($P < 0.05$), groups 1 and 4 ($P < 0.05$), groups 2 and 3, ($P < 0.01$), and groups 3 and 4 ($P < 0.001$; Table 3).

Chi-square test revealed significant differences in ARI scores between groups 1 and 2, groups 1 and 4, groups 2 and 3, and groups 3 and 4 (all $P < 0.001$). There was no statistically significant difference between groups 1 and 3 or groups 3 and 4 ($P > 0.05$; Table 4).

There was a positive correlation between bond strength values and ARI scores for all four groups ($r = 0.471$, $P < 0.001$).

Table 2 Mean shear bond strengths in megapascals (MPa) for each group calculated using the non-parametric Kruskal–Wallis (KW) test.

Groups*	MPa (mean ± standard deviation)
Group 1	5.515 ± 1.191
Group 2	6.549 ± 0.002
Group 3	4.551 ± 1.937
Group 4	6.39 ± 0.455
KW	22.46
P	***

Table 3 Correlation between groups was calculated in megapascals (MPa) using the Dunn's multiple comparison test.

Groups*	MPa
Group 1/group 2	*
Group 1/group 3	ns
Group 1/group 4	*
Group 2/group 3	***
Group 2/group 4	ns
Group 3/group 4	**

Table 4 Comparison of adhesive remnant index (ARI) scores between groups using the chi-square test.

Groups*	ARI
Group 1/group 2	***
Group 1/group 3	ns
Group 1/group 4	***
Group 2/group 3	***
Group 2/group 4	*
Group 3/group 4	***

*For details of the groups refer to Table 1.
ns, not significant; *$P < 0.05$, **$P < 0.01$; ***$P < 0.001$.

Discussion

The aim of this study was to evaluate different surface preparation combinations on the SBS of metal brackets to porcelain surfaces.

Previous research has shown that the optimal bracket bonding force is 6–10 MPa (Cochran *et al.*, 1997; Bourke and Rock, 1999). However, the direct transfer of this value to clinical situations is not universally accepted because the bracket–porcelain bond is influenced by many environmental factors (Zachrisson *et al.*, 1996; Zachrisson 2000). In the present study, in groups 2 and 4, the SBS was above 6 MPa, but less than 13 MPa which could clinically cause cohesive fractures (Thurmond *et al.*, 1994), whereas in groups 1 and 3, they were found to be below those needed for clinically minimal orthodontic loading. The present study was performed under *in vitro* conditions. The findings may therefore indicate that a combination of Pulpdent silane with 5 or 9.6 per cent HF acid is inadequate for porcelain surface preparation in order to bond metal brackets; clinically, however, it is not always possible to make extrapolations from *in vitro* studies to clinical situations.

Previous research has indicated that thermocycling weakened bond strength from a mean of 18.69–9.53 MPa (Bourke and Rock, 1999). In another study, it was found that the effect of thermal cycling was not significant (Smith *et al.*, 1988). In the present investigation, the specimens were subjected to thermal cycling as a means of artificially ageing or weakening bonds prior to testing, as described by Zachrisson *et al.* (1996).

It has been recommended that methods that provide a sufficient bond with less roughening should be used (Eustaquio *et al.*, 1988; Kao *et al.*, 1988; Zelos *et al.*, 1994).

In this study, deglazing was performed with a sandblaster and not with a green stone, in order to avoid microcracks (Eustaquio *et al.*, 1988; Zachrisson *et al.*, 1996). Although Kocadereli *et al.* (2001) concluded that roughening the porcelain surface with a sandblaster did not increase the resistance to debonding forces, many authors have recommended using an intraoral sandblaster for surface roughening (Eustaquio *et al.*, 1988; Smith *et al.*, 1988; Wolf *et al.*, 1993; Zachrisson *et al.*, 1996; Chung and Hwang, 1997; Shahverdi *et al.*, 1998; Jost-Brinkmann and Böhme, 1999).

Although sandblasting and HF acid etching were both used in order to prepare the porcelain surfaces, the results of this study demonstrated low bond strengths in all groups. Considering ARI scores, this result could be explained as a bond failure between the adhesive resin and porcelain surface. This could be due to the characteristics of the base of the brackets used. In previous studies, lower SBS were recorded during debonding Dynalock brackets (Wang *et al.*, 2004). Further research using different brackets with different base structures should be performed.

The use of strong acids to etch porcelain as suggested by Calamia (1983) may produce increased bond strengths since the action of an acid such as 9.6 per cent HF is to create a series of surface pits by preferential dissolution of the glass phase from the ceramic matrix (Al Edris *et al.*, 1990). However, HF acid must be used with great care, as it is extremely corrosive, and is capable of causing severe trauma to soft tissues and tooth substance (Hayakawa *et al.*, 1992; Shahverdi *et al.*, 1998). It has been concluded by some authors that 5 per cent HF acid used for 120 seconds on porcelain surfaces gives the highest bond strength (Chen *et al.*, 1998), while others have suggested 9.6 per cent HF acid in gel form applied for 2–4 minutes (Zachrisson and Büyükyilmaz, 1993). The advice of the manufacturer is to use 9.6 per cent HF acid for 2 minutes on porcelain surfaces. In this study, two different concentrations of acid gel were used for 120 seconds. Since the slight difference between the 5 and 9.6 per cent HF groups was statistically insignificant and also considering the harmful effects of a higher concentration of HF acid, it could be suggested that there is no need to use 9.6 per cent HF acid in order to achieve higher bond strength.

Kocadereli *et al.* (2001) reported that silane application after surface roughening of porcelain surfaces, which provides a chemical link between porcelain and composite resin (Lu *et al.*, 1992), increases the bond strength of orthodontic attachments. In this study, two different silanes combined with two different HF acid concentrations were used in order to compare different acid and silane combinations. The statistical difference between the Pulpdent and Reliance groups would suggest that higher bond strengths will be achieved when bonding brackets to porcelain surfaces with Reliance silane. It should be emphasized that the differences between *in vitro* and *in vivo* bond strengths need to be considered carefully, especially when bonding to other restorative materials.

In the present study, the groups bonded with Reliance showed lower bond strength between the bracket surface and composite resin, leaving more adhesive on the porcelain surface than the groups bonded with Pulpdent. This result was similar to the failure site of composite brackets bonded to porcelain (Huang and Kao, 2001). Reliance and Pulpdent are widely used in orthodontic practice for direct bonding of brackets to glazed porcelain surfaces. Both silanes are used for bond enhancement of composites, resin cements, and other organic resins to porcelain surfaces by building a chemical bond between the silicon (in the structure of porcelain material) and bracket adhesive. One of the objectives of this *in vitro* study was to compare these two different silanes. As the manufacturers do not disclose the exact chemical compositions, it is not possible to discuss the findings of this study on the basis of chemical composition.

Examination of the debonded surfaces showed no damage to the surfaces in any group. This observation is important because damage to the porcelain surface could affect aesthetics and the strength of the porcelain restoration.

Thirty-five per cent of the samples in group 2 (Reliance silane + 9.6 per cent HF acid) and 20 per cent of the samples in group 4 (Reliance silane + 5 per cent HF acid) required further treatment to remove adhesive traces from the porcelain surface, a procedure that could cause additional damage to the porcelain restoration surfaces. In group 1 (Pulpdent silane + 9.6 per cent HF acid) and group 3 (Pulpdent silane + 5 per cent HF acid), no adhesive remnant was observed on any samples after debonding.

Conclusion

Orthodontic bonding forces to porcelain were evaluated *in vitro* with four different combinations and the following conclusions were reached:

1. Although the use of 9.6 per cent HF acid increased bond strength, there was no statistically significant difference between the groups etched with 5 and 9 per cent HF acid. Therefore, 5 per cent HF acid etch could be recommended for intraoral applications in order to prevent tissue irritation without a loss in bond strength.
2. Silanization with Reliance resulted in higher bond strengths than Pulpdent but allowed removal without damage to porcelain surfaces.

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