European Journal of Orthodontics 29 (2007) 559–563
doi:10.1093/ejo/cjm052
Advance Access publication 14 September 2007

An *in vitro* comparative assessment of different enamel contaminants during bracket bonding

Andreas Faltermeier*, Michael Behr**, Martin Rosentritt**, Claudia Reicheneder* and Dieter Müßig*
Departments of *Orthodontics and **Prosthetic Dentistry, University Clinics, Regensburg, Germany

SUMMARY In orthodontics, adhesive failures can occur because of saliva contamination during bonding. However, most *in vitro* studies concerning bond strength of saliva-contaminated enamel disregard the influence of temperature changes in a wet environment. The aim of the present study was to compare the influence of saliva, blood and etching gel remnant contamination on shear bond strength (SBS) after thermocycling.

After etching of extracted human third molars (n = 80), a conventional primer (Transbond XT) and a moisture-insensitive primer (Transbond MIP) were evaluated using the adhesive, Transbond XT, under dry conditions and after contamination with saliva, blood and etching gel remnants. To simulate temperature changes and the moisture of saliva in the oral cavity, all samples were thermocycled (6000× 5°C/55°C) in a mastication device before SBS testing. A Mann–Whitney U test was used to determine statistical differences.

Under dry conditions Transbond XT and Transbond MIP showed no significant difference in SBS. However, clinically unacceptable (P = 0.005) bond strength was observed using Transbond XT after saliva and blood contamination. In wet conditions only Transbond MIP showed sufficient bond strength.

If contamination during bonding is expected, a hydrophilic primer should be used. Under dry conditions hydrophilic or hydrophobic primers could be applied. Blood contamination seems to be a more serious problem for bond strength than saliva or etching gel contamination.

Introduction

The investigations of Buonocore (1955) on the direct bonding technique revolutionized the appearance of fixed orthodontic appliances. Over the years a great deal of attention has been paid to improve the acid-etching technique, primers and adhesives. Nonetheless, adhesive failures still exist because of contamination during bonding. As contaminants, saliva, blood and etching gel remnants are described (Schaneveldt and Foley, 2002; Reddy *et al.*, 2003). Some researchers have reported a decline in bracket bond strength as a result of saliva and moisture exposure during bonding (Webster *et al.*, 2001; Rajagopal *et al.*, 2004; Campoy *et al.*, 2005). However, most *in vitro* studies on bond strength after saliva contamination did not use an artificial ageing procedure before testing, despite the fact that thermocycling of the specimens has been recommended to consider the durability of the bond (Buonocore, 1981; Schaneveldt and Foley, 2002).

Saliva contact with the etched tooth results in plugging of porosities caused by acid etching and in a reduction of surface energy (Rajagopal *et al.*, 2004). Early resins were manufactured of hydrophobic monomers, which performed well only in dry environments (Hormati *et al.*, 1980; Grandhi *et al.*, 2001; Rajagopal *et al.*, 2004). Nowadays hydrophilic components, such as hydroxyethyl methacrylate (HEMA) which is well known in restorative dentistry for dentine bonding, are available in adhesives for enamel bracket bonding. These moisture-insensitive primers (MIP) perform adequately even in the presence of moisture (Rajagopal *et al.*, 2004). However, there are controversial reports on whether MIPs fulfil the requirements for bond strength in a dry environment (Littlewood *et al.*, 2000; Kula *et al.*, 2003).

A common procedure for surgical exposure and orthodontic-assisted eruption of impacted teeth is direct bonding of orthodontic buttons or brackets (Reddy *et al.*, 2003). The presence of blood makes it difficult to place a button on the unerupted tooth. Therefore, buttons or brackets often have to be rebonded which is an unpleasant procedure for patients.

Another problem of direct bonding might be the removal of the etching gel. According to the manufacturer's protocol, the tooth should be rinsed thoroughly with oil-free water after etching. If this process is performed inadequately, etching gel remnants stay on the enamel and could obstruct bonding.

The purpose of this study was to compare the effect of saliva, blood and etching gel remnants as contaminants on the enamel surface during the bracket-bonding process. A conventional primer (Transbond XT, 3M Unitek, Monrovia, California, USA) and an MIP (Transbond MIP, 3M Unitek) were evaluated using the adhesive Transbond XT (3M Unitek). The results were compared with a control group bonded under dry conditions. To simulate temperature changes and the moisture in the oral cavity, all samples were

exposed to thermocycling (6000× 5°C/55°C) in a mastication device before testing.

Materials and methods

A total of 80 recently extracted third molars were collected and stored in 0.5 per cent chloramines-T. The roots were removed and the crowns were embedded in auto-polymerization acrylic resin so that the facial surface of each tooth was parallel to the base of the polymer. The teeth were cleaned with a non-fluoridated pumice paste and rubber cups. The enamel surface of each tooth was etched with 20 per cent phosphoric acid (Gluma Etch 20 Gel, Heraeus Kulzer, Hanau, Germany) for 30 seconds. A frosted appearance indicated a successful etch. Eight groups (10 teeth per group) were formed:

Group 1: Transbond XT in dry conditions (control).
Group 2: Transbond MIP in dry conditions (control).
Group 3: Transbond XT after saliva application.
Group 4: Transbond MIP after saliva application.
Group 5: Transbond XT after blood application.
Group 6: Transbond MIP after blood application.
Group 7: Transbond XT after insufficient removal of etching gel with a rinse of oil-free water for only 2 seconds.
Group 8: Transbond MIP after insufficient removal of etching gel with a rinse of oil-free water for only 2 seconds.

The applications of the used primers were as follows: the conventional primer Transbond XT was applied, gently thinned with air and light-cured for 20 seconds (Ortholux, 3M Unitek). Transbond MIP was thinned with air after 10 seconds of application and light-cured for 20 seconds (Ortholux, 3M Unitek). For contamination tests, sufficient saliva or blood (both human) was applied for 15 seconds on the enamel to permit full hydration of the surface.

Metal brackets (Ormesh, Ormco Corporation, Glendora, California, USA) were bonded to the teeth using Transbond XT adhesive. All brackets were placed centrally on the flat buccal surfaces of the teeth. The excess resin was carefully removed from the tooth using a dental probe. The samples were then light-cured with a light-emitting diode curing device (Ortholux) for 20 seconds. All brackets were bonded by the same operator.

To simulate the moisture and temperature changes in the oral environment, all samples were exposed to thermocycling 24 hours after preparation. All groups were alternately flooded every 2 minutes with warm (55°C) and cold (5°C) distilled water for 6000 cycles in a mastication device (Rosentritt *et al.*, 1997).

Shear bond strength (SBS) testing was performed using the universal testing machine Zwick 1446 (Figure 1, Zwick, Ulm, Germany) at a crosshead speed of 1 mm/minute. The embedded tooth and the adhesively fixed bracket were positioned in the testing apparatus so that the bracket slot was placed horizontally. A knife-edge shearing rod was used to deliver the shear force at the bracket base–enamel interface. All brackets were shear tested to failure. SBS was determined using the formula $\sigma_{shear} = F\max/A_{bracket\ base\ surface}$ (MPa). The surface area of the bracket bases was determined by measuring the length and width and computing the mean area.

To determine statistical differences, a Mann–Whitney *U* test was performed. The means and standard deviations were calculated. The level of significance was set at $\alpha = 0.05$.

Results

The hydrophobic Transbond XT primer revealed a distinctive and significant ($P = 0.005$) decrease in SBS after saliva and blood contamination (Table 1, Figure 2a). Following etching gel contamination, no significant change of SBS was observed. Saliva and etching gel contamination did not significantly influence the SBS of brackets bonded with the hydrophilic Transbond MIP (Figure 2b). A significant ($P = 0.005$) decrease in SBS could only be detected after blood contamination using Transbond MIP. A comparison of SBS values for Transbond XT and MIP bonded brackets showed no significant changes under dry conditions or after etching gel contamination. In wet conditions (after saliva and blood contamination) a significant decrease in SBS for Transbond XT bonded brackets was found in comparison with Transbond MIP ($P = 0.005$). Transbond MIP primer revealed the highest SBS mean values (9.29 ± 1.16 MPa) under dry conditions (Table 1).

Figure 1 Overall view of the Zwick universal testing machine.

Table 1 Means and standard deviations for shear bond strength.

	Shear bond strength (MPa)
Transbond XT primer under dry conditions (control group)	8.71 ± 1.37
Transbond MIP under dry conditions (control group)	9.29 ± 1.16
Transbond XT primer after saliva contamination	3.42 ± 0.78*
Transbond MIP after saliva contamination	8.82 ± 1.21^{NS}
Transbond XT primer after blood contamination	2.37 ± 1.13*
Transbond MIP after blood contamination	7.08 ± 0.78*
Transbond XT primer after etching gel contamination	8.47 ± 0.78^{NS}
Transbond MIP after etching gel contamination	9.16 ± 0.95^{NS}

MIP, moisture insensitive primer.
*P = 0.005; NS, not significant.

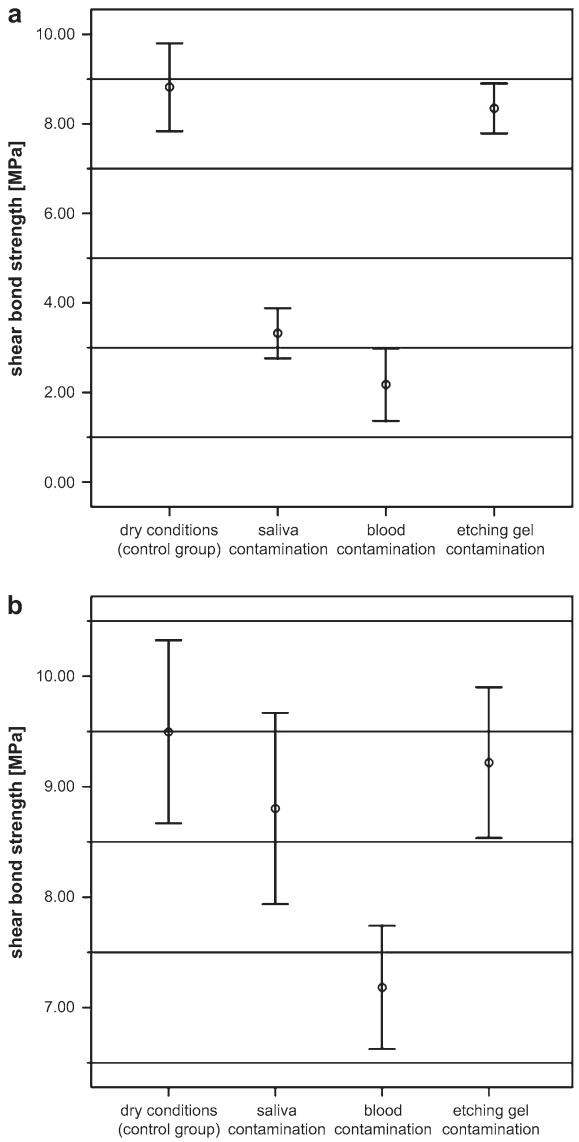

Figure 2 Shear bond strength (MPa) of brackets bonded with Transbond XT primer (a) and Transbond moisture-insensitive primer (b) after enamel surface contamination.

Discussion

In this study, the SBS of brackets on contaminated enamel was tested after thermocycling. To consider the durability of the bond, Buonocore (1981) and Schaneveldt and Foley (2002) recommended thermocycling of specimens. According to Pfeiffer and Marx (1989), the temperature of food ranges between −8°C and 81°C. It was concluded that in the oral cavity the temperature on interfaces between different material groups could be 5–52°C (Pfeiffer and Marx, 1989). It has been reported that the bond strength of different adhesive resins is reduced after thermocycling (Bishara *et al.*, 1975; Klockowski *et al.*, 1989; Komori and Ishikawa, 1997; Daub *et al.*, 2006). Two reasons for this phenomenon could be water uptake and the effect of different coefficients of thermal expansion. Increased water sorption is likely to be the main factor, which affects bond strength since water is able to penetrate into the polymer. As a result, the secondary chemical-bonding forces (van der Waals forces) between the polymer chains are reduced and the mechanical properties of the resin decreased (Rantala *et al.*, 2003). This plasticizing affects mainly the properties of the adhesive. Daub *et al.* (2006) stated that the amount of water absorbed by the polymer and the rate of absorption are diffusion controlled and depend mostly on material factors. Previous investigations on bond strength of saliva-contaminated enamel have disregarded the influence of temperature changes in a wet environment (Webster *et al.*, 2001; Schaneveldt and Foley, 2002; Rajagopal *et al.*, 2004; Campoy *et al.*, 2005). Often samples were immersed in distilled water only for a short period of time (Schaneveldt and Foley, 2002; Kula *et al.*, 2003). Yap and Wee (2002) suggested that if thermocycling is added to water storage, water absorption of composites is accelerated and resins absorb even more water.

Another reason for the decline in bond strength of thermocycled samples could be the differences in the coefficient of thermal expansion between the bracket, the adhesive and the enamel (Anusavice, 2003; Arici and Arici, 2003; Daub *et al.*, 2006). Consequently, in this investigation, thermocycling (6000× 5°C/55°C) in a mastication device before SBS testing was used to simulate cyclic stress at two different temperature extremes and to reproduce water absorption expected in the oral environment.

The difficulty of orthodontic bracket bonding is its semi-permanent nature. The bond strength should be sufficiently high to resist accidental debonding during treatment, but low enough to remove the bracket from the tooth without generating excessive force which might damage the periodontium (Özcan *et al.*, 2004). It is a common belief that the clinically adequate SBS for a stainless steel bracket to enamel should be 6–8 MPa (Gillis and Redlich, 1998; Bourke and Rock, 1999; Özcan *et al.*, 2004). In the present study no significant differences

between conventional primer (XT) and MIP could be observed under dry conditions. These results are in contrast to the findings of Littlewood *et al.* (2000). They reported a significant reduction of SBS under dry conditions using a hydrophilic primer. The results of the present study indicate that after saliva and blood contamination MIP revealed a higher SBS compared with the conventional primer XT. These findings are in agreement with previous reports (Webster *et al.*, 2001; Rajagopal *et al.*, 2004; Campoy *et al.*, 2005). Webster *et al.* (2001) concluded that uncontaminated (without saliva contact) enamel surfaces resulted in the highest bond strengths for hydrophilic and hydrophobic adhesives. Rajagopal *et al.* (2004) investigated the effect of conventional, moisture-insensitive and self-etching primers after contamination with natural saliva *in vitro*. They found a superior bond strength for MIP and self-etching primers in cases of moisture contamination.

The introduction of MIP in orthodontics derives from efforts in restorative dentistry to improve bond strength to dentine (Kanca, 1996; Frankenberger *et al.*, 2000). According to the investigation of Newman *et al.* (2001), adhesion promoters containing pyromellitic glycerol dimethycrylate and HEMA and other acrylates improve bond strength and promote bonding under slightly moist conditions. Especially the hydrophilic monomer, HEMA, which allows a lower contact angle and an extension of the molecule (Rajagopal *et al.*, 2004), is efficient even in the presence of saliva. Conventional primers consisting of a glycidal methycrylate (BisGMA) reveal a hydrophobic characteristic and are not able to penetrate the saliva on the etched enamel. In addition, minerals and saliva proteins compromise the setting of the adhesive on the contaminated tooth surface (Itoh *et al.*, 1999).

The results of SBS testing after blood contamination indicate that human blood seems to be a greater barrier for the adhesives to penetrate. This might be of concern when bonding orthodontic buttons or brackets during surgical exposure of impacted teeth. Often glass ionomer cements (GICs) are used for bonding brackets to the surface of unerupted teeth, because of their enhanced curing in a wet environment (Reddy *et al.*, 2003). However, those authors found that the beneficial wetting phenomenon of GICs is not achieved after blood contamination during curing. They stated that, without contamination, composite resins have a greater bond strength than resin-reinforced GICs. After blood contamination, both materials showed a significant decrease in bond strength (Reddy *et al.*, 2003). In the present study, blood seems to be a physical barrier that impedes the mechanical retention of the adhesive to the etched tooth. It was assumed that because of the compound of blood, the physical barrier was greater than that of saliva. Hence, bond strength is reduced in comparison with saliva contamination.

Etching gel remnants on the enamel do not seem to significantly influence SBS. Despite this, etching gel should be removed according to the manufacturer's instruction to avoid enamel damage.

In the present investigation an attempt was made to simulate clinical conditions using thermocycling. Therefore, the results of the present investigation are difficult to compare with other studies on contamination during bonding, because often SBS was determined under dry conditions or after water exposure for a short period of time, and the influence of water penetration into the bracket, resin and tooth interface was disregarded. Nevertheles, it should be remembered that this was an *in vitro* study, and care should be taken in interpreting the results to those that might be obtained *in vivo*.

Conclusions

If contamination during bonding is anticipated, a hydrophilic primer should be used. Under dry conditions hydrophilic or hydrophobic primers could be applied. Blood contamination seems to be a more serious problem for bond strength than saliva or etching gel contamination.

Address for correspondence

Dr Andreas Faltermeier
Department of Orthodontics
University Medical Centre
Franz-Josef-Strauss-Allee 11
D-93042 Regensburg
Germany
E-mail: Andreas.Faltermeier@klinik.uni-regensburg.de

References

Anusavice K J 2003 Phillips' science of dental materials, 11th edn. Saunders, St Louis p. 55

Arici S, Arici N 2003 Effects of thermocycling on the bond strength of a resin-modified glass ionomer cement: an *in vitro* comparative study. Angle Orthodontist 73: 692–696

Bishara S E, Khowassah A M, Oesterle L J 1975 Effect of humidity and temperature changes on orthodontic direct-bonding adhesive systems. Journal of Dental Research 54: 751–757

Bourke B M, Rock W P 1999 Factors affecting the shear bond strength of orthodontic brackets to porcelain. British Journal of Orthodontics 26: 285–290

Buonocore M G 1955 A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. Journal of Dental Research 34: 849–853

Buonocore M G 1981 Retrospectives on bonding. Dental Clinics of North America 25: 241–255

Campoy D, Vicente A, Bravo L A 2005 Effect of saliva contamination on the shear bond strength of orthodontic brackets bonded with a self-etching primer. Angle Orthodontist 75: 865–869

Daub J, Berzins D W, Linn B J, Bradley T G 2006 Bond strength of direct and indirect bonded brackets after thermocycling. Angle Orthodontist 76: 295–300

Frankenberger R, Kramer N, Obershachtsiek H, Petschelt A 2000 Dentin bond strength and marginal adaption after NaOCL treatment. Operative Dentistry 25: 40–45

Gillis I, Redlich M 1998 The effect of different porcelain conditioning techniques on shear bond strength of stainless steel brackets. American Journal of Orthodontics and Dentofacial Orthopedics 114: 387–392

Grandhi R K, Conbe E C, Spiedel T M 2001 Shear bond strength of stainless steel orthodontic brackets with a moisture-insensitive primer. American Journal of Orthodontics and Dentofacial Orthopedics 119: 251–257

Hormati A A, Fuller J L, Denehy G E 1980 Effect of contamination and mechanical disturbance on the quality of acid etched level. Journal of the American Dental Association 100: 34–38

Itoh T, Matsuto N, Fukushima T, Inoue Y, Oniki Y, Matsumoto M 1999 Effect of contamination and etching on enamel bond strength of new light-cured glass ionomer cements. Angle Orthodontist 69: 450–456

Kanca J 1996 Wet bonding: effect of drying time and distance. American Journal of Dentistry 9: 273–276

Klockowski R, Davis E L, Joynt R B, Wieczkowski Jr G, MacDonald A 1989 Bond strength and durability of glass ionomer cements used as bonding agents in the placement of orthodontic brackets. American Journal of Orthodontics and Dentofacial Orthopedics 96: 60–64

Komori A, Ishikawa H 1997 Evaluation of resin-modified glass ionomer cement for use as an orthodontic agent. Angle Orthodontist 67: 183–196

Kula K S, Nash T D, Purk J H 2003 Shear-peel bond strength of orthodontic primers in wet conditions. Orthodontics and Craniofacial Research 6: 96–100

Littlewood S, Mitchell L, Greenwood D, Bubb N, Wood D 2000 Investigation of a hydrophilic primer for orthodontic bonding: an *in vitro* study. Journal of Orthodontics 27: 181–186

Newman R A, Newman G V, Sengupta A 2001 *In vitro* bond strengths of resin modified glass ionomer cements and composite resin self-cure adhesives: introduction of an adhesive system with increased bond strength and inhibition of calcification. Angle Orthodontist 71: 312–317

Özcan M, Vallittu P K, Peltomäki T, Huysmans M C, Kalk W 2004 Bonding polycarbonate brackets to ceramic: effects of substrate treatment on bond strength. American Journal of Orthodontics and Dentofacial Orthopedics 126: 220–227

Pfeiffer P, Marx R 1989 Temperaturbelastung von Adhäsivbrücken und ihre Auswirkung auf die Verbundfestigkeit von Klebebrücken. Schweizer Monatsschrift der Zahnmedizin 99: 782

Rajagopal R, Padmanabhan S, Gnanamani J 2004 A comparison of shear bond strength and debonding characteristics of conventional, moisture-insensitive, and self-etching primers *in vitro*. Angle Orthodontist 74: 264–268

Rantala L I, Lastumäki T M, Peltomäki T, Vallittu P K 2003 Fatigue resistance of removable orthodontic appliance reinforced with glass fibre weave. Journal of Oral Rehabilitation 30: 501–506

Reddy L, Marker V A, Ellis E 2003 Bond strength for orthodontic brackets contaminated by blood: composite versus resin-modified glass ionomer cements. Journal of Oral and Maxillofacial Surgery 61: 206–213

Rosentritt M, Leibrock A, Lang R, Behr M, Scharnagl P, Handel G 1997 Apparatur zur Simulation des Kauorgans (Regensburger Kausimulator). Materialprüfung 39: 77–80

Schaneveldt S, Foley T F 2002 Bond strength comparison of moisture-insensitive primers. American Journal of Orthodontics and Dentofacial Orthopedics 122: 267–273

Webster M J, Nanda R S, Duncanson M G, Khajotia S S, Sinha P K 2001 The effect of saliva on shear bond strengths of hydrophilic bonding systems. American Journal of Orthodontics and Dentofacial Orthopedics 119: 54–58

Yap A U, Wee K E 2002 Effects of cyclic temperature changes on water sorption and solubility of composite restoratives. Operative Dentistry 27: 147–153