Shear bond strength of orthodontic brackets to aged resin composite surfaces: effect of surface conditioning

Mehmet Bayram*, Cemal Yeşilyurt**, Adem Kuşgöz***, Mustafa Ülker**** and Metin Nur*

Departments of, *Orthodontics, **Operative Dentistry, ***Pediatric Dentistry, Karadeniz Technical University, Trabzon and ****Department of Operative Dentistry, Erciyes University, Kayseri, Turkey

Correspondence to: Dr Mehmet Bayram, Aydınlıkevler Mahallesi, 615 Nolu Ustaoğlu Sokak, Borankent Sitesi B Blok No: 4/13, TR-61040 Trabzon, Turkey. E-mail: dtmehmetbayram@yahoo.com

SUMMARY The aim of this study was to investigate the effects of surface conditioning protocols on the shear bond strength (SBS) of metal brackets to aged composite resin surfaces *in vitro*.

Ninety composite resin discs, 6 mm in diameter and 2 mm in height, were prepared and treated with an ageing procedure. After ageing, the specimens were randomly assigned to one of the following groups: (1) control with no surface treatment, (2) 38 per cent phosphoric acid gel, (3) 9.6 per cent hydrofluoric acid gel, (4) airborne aluminium trioxide particle abrasion, (5) sodium bicarbonate particle abrasion, and (6) diamond bur. The metal brackets were bonded to composite surfaces by means of an orthodontic adhesive (Transbond XT). All specimens were stored in water for 1 week at 37°C and then thermocycled (1000 cycles, 5–55°C) prior to SBS testing. SBS values and residual adhesive on the composite surface were evaluated.

Analysis of variance showed a significant difference ($P = 0.000$) between the groups. Group 6 had the highest mean SBS (10.61 MPa), followed by group 4 (10.29 MPa).

The results of this study suggest that a clinically acceptable bond strength can be achieved by surface conditioning of aged resin composite via the application of hydrofluoric acid, aluminium trioxide particle abrasion, sodium bicarbonate particle abrasion, or a diamond bur.

Introduction

The demand for orthodontic treatment has been gradually increasing among the adult population. This increase in the number of orthodontic patients presents new problems to the orthodontists. As many patients have restored teeth with various restorative materials, such as composite resin, amalgam, and porcelain, orthodontists are more likely to face the difficulty of bonding orthodontic attachments to these materials.

Particularly in adolescent orthodontic patients, composite resin restorations are often present on the labial surfaces of maxillary incisors and occasionally on the buccal surfaces of posterior teeth. The frequency of composite resin restorations in posterior teeth has increased with the improvement in the properties of aesthetic filling materials (Brunthaler *et al.*, 2003).

There have been many reports on bonding brackets to amalgam (Zachrisson *et al.*, 1995; Buyukyilmaz and Zachrisson, 1998) and porcelain (Huang and Kao, 2001; Kocadereli *et al.*, 2001; Kitayama *et al.*, 2003; Ozcan *et al.*, 2004) using different surface conditioning methods and adhesives, but minimal research (Kao *et al.*, 1995; Jost-Brinkmann *et al.*, 1996; Chunhacheevachaloke and Tyas, 1997; Lai *et al.*, 1999) has been carried out to quantify the bonding of brackets to the restorative composite resin surface. In previous studies, brackets were bonded to fresh composite in patients referred for orthodontic treatment whereas composite restorations have been ageing for a long

time in a humid oral environment. Since clinical failure of brackets bonded to composite resin restorations using conventional bonding procedures has frequently been encountered, the purpose of this *in vitro* study was to investigate the shear bond strength (SBS) of orthodontic brackets bonded to aged restorative composite resin surfaces treated with various surface roughening methods.

Materials and methods

Composite discs

Ninety restorative composite resin discs, 6 mm in diameter and 2 mm thick, were prepared from a nano-filled resin composite (Filtek Supreme XT; 3M ESPE, St Paul, Minnesota, USA) by conventional condensation methods using a teflon mould. To create a smooth flat surface after light curing, the composite was compressed with a glass slide and excess material extruded. The composite was light polymerized with a light-emitting diode device (LED; Elipar; 3M ESPE) at an intensity of 950 mW/cm² for 20 seconds through the glass plate at a 90 degree angle to the top of the surface.

Ageing procedure

The composite specimens were aged in an accelerated ageing chamber. The specimens were mounted on a panel attached to the frame of an accelerated weathering tester (QUV; The

Q-Panel Co., Cleveland, Ohio, USA) and were kept there for 300 hours. In the weathering tester, the specimens were exposed to continuous ultraviolet (UV) and visible light, at a temperature of 43.3°C and with a programmed cycle of 18 minutes of distilled water spray within each 2 hour period (Dootz *et al.*, 1993; Anil *et al.*, 1999, 2000; Ramoglu *et al.*, 2008).

After completion of the ageing procedure, all specimens were stored in distilled water for 24 hours at room temperature. Thereafter, the specimens were embedded in acrylic resin blocks, leaving the smooth surfaces of the composite discs exposed for bonding.

Adhesive and brackets

The light-cured orthodontic adhesive used in the present investigation for bonding the brackets to the composite discs was Transbond™ XT (3M Unitek, Monrovia, California, USA). Upper right stainless steel central incisor brackets (Mini Master Series; American Orthodontics, Sheboygan, Wisconsin, USA) were used for bonding to the composite surfaces. According to the manufacturer, the mean area of each bracket base was 10.88 mm^2.

Composite surface treatment

The specimens were randomly divided into six groups of 15 specimens according to the following surface treatment methods:

Group 1. Control with no surface treatment.
Group 2. Coating with a thin layer of 38 per cent phosphoric acid (Pulpdent, Watertown, Massachusetts, USA) for 60 seconds, then rinsed with water for 60 seconds, and dried with compressed oil-free air.
Group 3. Coating with a thin layer of 9.6 per cent hydrofluoric acid (Pulpdent) for 60 seconds, then rinsed with water for 2 minutes, and dried with compressed oil-free air.
Group 4. Sandblasted with sodium bicarbonate particles for 15 seconds, using an air-abrasive unit (Air-Flow Handy; EMS, Nyon, Switzerland) at 2.2 atmospheric pressure and a distance of 10 mm.
Group 5. Sandblasted with 50 μm AI$_2$O$_3$ particles for 10 seconds using a microetcher (Danville Engineering Inc., Danville, California, USA) at a distance of 10 mm.
Group 6. Roughened at high speed with a diamond bur (150 μm; 856/018, Diatech Diamant AG, Heerbrugg, Switzerland) under water cooling.

The bracket bonding procedures were carried out by the one author (MB). For the bonding process, a thin layer of Transbond primer was applied on the treated composite surface, and the brackets were bonded with the adhesive composite resin at the centre of composite disc with a light force. Excess adhesive was removed using a small scaler. Polymerization for a total of 20 seconds from two directions using the LED was performed.

All specimens were stored in distilled water for 1 week at 37°C and then thermocycled (1000 cycles, 5–55°C) prior to SBS testing. During thermocycling, the dwell time for the specimens in each well was 30 seconds and the transfer time between the wells 4 seconds.

SBS test

SBS testing was performed with using a universal testing device (Lloyd Instruments Plc, Fareham, Hampshire, UK). A schematic illustration of the SBS test set-up is shown in Figure 1. For shear testing, the specimens were stressed in an occluso-gingival direction at a crosshead speed of 1 mm/minute. The maximum load necessary to debond was recorded in Newtons and converted to megapascals (MPa) as a ratio of Newtons to surface area of the bracket base.

Determination of fracture sites

After debonding, the fracture sites were examined with a stereomicroscope (Stemi 2000-C; Carl Zeiss, Göttingen, Germany) at ×10 magnification. Any adhesive remaining on the aged composite surface was evaluated with the adhesive remnant index (ARI; Årtun and Bergland, 1984) and scored with respect to the amount of resin material adhering to the composite surface. The ARI scale has a range between 0 and 3, with 0 indicating that no adhesive remains on the composite surface; 1, less than 50 per cent of adhesive remains on the composite surface; 2, more than 50 per cent of the adhesive remains; and 3, the entire adhesive remains on the tooth, along with the impression of the bracket base.

Scanning electron microscope analysis

One sample from each test group was examined under a scanning electron microscope (SEM) to evaluate the effects of the different surface preparation methods on the aged composite surfaces. The specimens were stored for 2 days
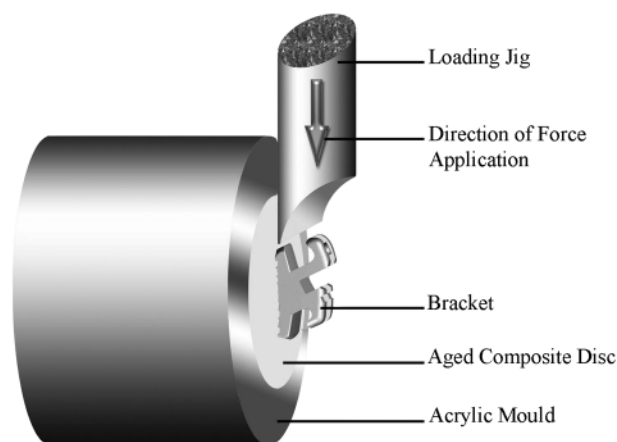
Figure 1   Schematic illustration of the shear bond strength test set-up.

in absolute alcohol, air dried for 2 hours, mounted on SEM stubs, and sputter coated with 10 nm gold particles using a Polaron Sc500 sputter-coating device (VG Microtech Inc., Uckfield, Sussex, UK) and observed under a SEM (JSM-5600; Jeol Ltd, Tokyo, Japan) at an operating voltage of 10 kV. The specimens were observed at a 90 degree angle and a 23 mm working distance. Photomicrographs were taken at ×500 magnification for visual inspection.

Statistical analysis

One-way analysis of variance (ANOVA) was performed to examine the effects of surface treatment on SBS. The Tamhane test was used for *post hoc* comparisons of the groups. The results were evaluated with a 95 per cent confidence interval. The significance level was set at $P < 0.05$.

Results

SBS comparisons

The descriptive statistics and the results of the Tamhane *post hoc* test for the SBS values of are shown in Table 1. Figure 2 shows the mean SBS values of the various surface preparation methods. It can be seen that group 6 had the highest mean SBS value (10.61 MPa), followed by group 5 (10.29 MPa). The lowest mean SBS value was observed in group 1 (2.77 MPa).

Using SBS (MPa) as the dependent variable, ANOVA showed a significant difference ($F = 22.63$, $P = 0.000$) between the groups. The results of the Tamhane *post hoc* test indicated that there was no significant difference between groups 1 and 2. Additionally, there were no statistically significant differences between groups 3, 4, 5, and 6.

Adhesive remnant index

Table 2 shows the distribution of ARI scores (failure modes) expressed as a frequency of occurrence for all groups tested. Groups 1, 2, and 3 showed an ARI score of 0, whereas group 6 had an ARI score of 3, i.e. groups 1, 2, and 3 failed at the composite–adhesive interface, whereas the failure site was at the bracket–adhesive interface for group 6.

SEM analysis

Figure 3 shows SEM photomicrographs of the composite surfaces treated with the various surface preparation techniques. Group 6 showed a rougher surface and a larger area for micromechanical retention compared with the other treatments. Both groups 1 and 2 had relatively smooth surfaces. The particle abrasion-treated groups (groups 4 and 5) exhibited rougher surfaces than the acid-treated groups (groups 2 and 3).

Table 1   Descriptive statistics [mean, standard deviation (SD) and standard error (SE)] of the shear bond strength values and results of the multiple comparison test.

Groups	Mean (MPa)	SD (MPa)	SE (MPa)	Range (MPa)	Tamhane*
Control	2.77	0.34	0.11	2.08–3.25	A
38% phosphoric acid	3.71	1.22	0.36	2.32–5.77	A
9.6% hydrofluoric acid	7.21	1.89	0.87	4.07–11.19	B
Sodium bicarbonate particle abrasion	8.26	2.16	0.65	5.62–15.77	B
Aluminium trioxide particle abrasion	10.29	1.92	1.03	5.81–12.76	B
Diamond bur	10.61	2.28	0.68	7.19–15.12	B

*Groups shown with different letters were significantly different at the $P = 0.05$ level according to the Tamhane test.
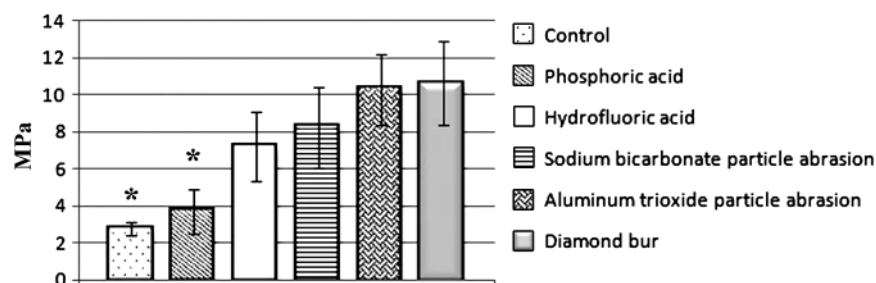
Figure 2   Bar chart showing the mean shear bond strength and standard deviation values of orthodontic attachments bonded to the aged composite surfaces treated with various surface preparation methods. The vertical bar indicates standard deviation. Significant differences among groups are showed by asterisks ($P = 0.05$).
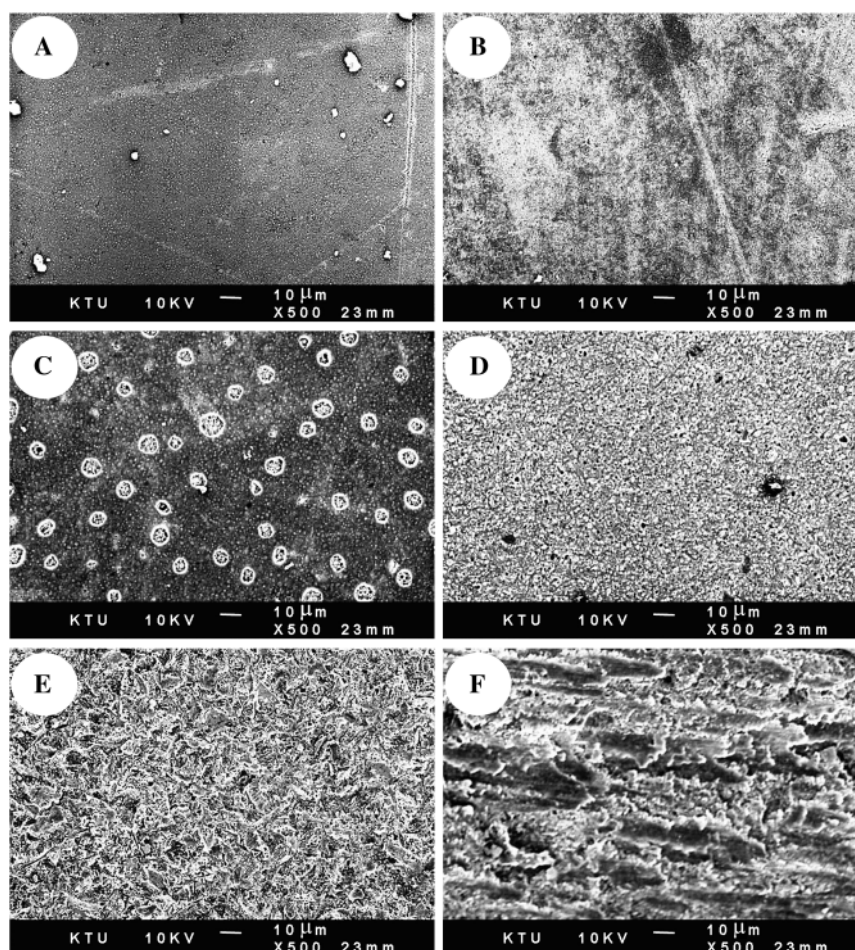
Discussion

When an orthodontic attachment is bonded to a composite restoration in the oral cavity, it is likely that the restoration has been ageing for a long time in a humid environment. This means that water saturation of composite resin has been reached and free radical activity has ended. Absorbed

Table 2 Frequency and percentage occurrence of the adhesive remnant index (ARI) scores.

Groups	N	0 (%)	1 (%)	2 (%)	3 (%)
Control	15	15 (100)	—	—	—
38 per cent phosphoric acid	15	15 (100)	—	—	—
9.6 per cent hydrofluoric acid	15	15 (100)	—	—	—
Sodium bicarbonate particle abrasion	15	14 (93.3)	1 (6.7)	—	—
Aluminium trioxide particle abrasion	15	—	—	1 (6.7)	14 (93.3)
Diamond bur	15	—	—	—	15 (100)

water causes softening of the matrix, microcrack formation, resin degradation, and debonding of the filler–matrix interfaces (Ferracane and Marker, 1992). A tendency for bond strength between new and old composite to decrease after ageing and storage of the old material in saliva has been reported (Boyer *et al.*, 1984; Chiba *et al.*, 1989).

In vitro studies simulating the ageing process of composites have used methods such as thermocycling and storage in aqueous media or citric acid (Yap *et al.*, 1999; Ozcan *et al.*, 2007). Additionally, with accelerated ageing (Dootz *et al.*, 1993; Anil *et al.*, 1999, 2000; Ramoglu *et al.*, 2008), composite samples are exposed to continuous UV and visible light and distilled water, which may change the physical properties of composites. According to the manufacturer of the weathering instrument used in the current study, 300 hours of ageing is equivalent to 1 year of clinical service (Powers *et al.*, 1978; Anil *et al.*, 1999). In the present study, an accelerated ageing process was used to provide a more realistic simulation for gaining older composite specimens. However, there is no consensus as to

Figure 3 Scanning electron photomicrographs of the composite surfaces after application of various surface preparation methods: (A) Control (no surface treatment), (B) 38 per cent phosphoric acid, (C) 9.6 per cent hydrofluoric acid, (D) sodium bicarbonate particle abrasion, (E) aluminium trioxide particle abrasion, and (F) diamond bur.

which ageing regimen best simulates oral conditions (Tezvergil *et al.*, 2003).

The three possible mechanisms during bracket bonding with the use of intermediate adhesive are chemical bond formation to the matrix, chemical bonds to the exposed filler particles, and micromechanical retention caused by penetration of the monomer components to microcracks in the matrix (Brosh *et al.*, 1997). Clinically, the bonding between two composite layers is achieved in the presence of an oxygen-inhibited layer of unpolymerized resin (Li, 1997). However, aged restorations do not contain an unpolymerized surface layer (Kupiec and Barkmeier, 1996). The unreactive methacrylate groups, which allow for adhesion of intermediate adhesive agents, are reduced with time, thereby reducing adhesion relative to that of a fresh composite (Ozcan *et al.*, 2007). Thus, the age of the restoration is an important factor in bracket bonding.

Several techniques have been suggested to improve the composite–composite bond. One technique is roughening the surface (Kupiec and Barkmeier, 1996) and the others are based on attempts to improve adhesion of new resin to cross-linked polymer matrix or filler particles of the composite (Brosh *et al.*, 1997; Kallio *et al.*, 2001). Improving the bond strength between new and old composite usually requires increased surface roughness to promote mechanical interlocking and coating of old composite with unfilled resin bonding agents to advance surface wetting and chemical bonding (Pounders *et al.*, 1987).

The bond in orthodontics must be semi-permanent and bond strength should be sufficiently high to resist accidental debonding during treatment but low enough so that excessive force does need not to be applied during debonding. It has been suggested that a clinically adequate bond strength for a metal orthodontic bracket to enamel should be 6–8 MPa (Reynolds, 1975). The mean SBS values of all surface conditioning groups, except in groups 1 and 2, were within this range or exceeded these limits and therefore could be considered sufficient for clinical applications.

Among the surface conditioning methods, conventional acid etching (group 2) produced the lowest SBS values. It has previously been shown that phosphoric acid is relatively ineffective in providing micromechanical retention on resin composites (Martin *et al.*, 2001). The ARI scores showed that bond failure occurred at the composite–adhesive interface in group 2. This demonstrates a weak connection between the resin composite and adhesive.

Hydrofluoric acid is well recognized to be hazardous *in vivo*; it is a harmful and irritating compound for soft tissues. Hydrofluoric acid acts by dissolving the glass particles of the filling, leaving gaps or pores that allow micromechanical retention (Swift *et al.*, 1992). In this study, the SBS values were increased by hydrofluoric acid application creating the area for micromechanical retention.

Sodium bicarbonate and Al_2O_3 particle abrasion are surface treatments that cause more microretentive areas for micromechanical interlocking than acid application. However, according to SEM photomicrographs, the application of a diamond bur creates macro- and microretentive areas. These applications remove the resin and expose the filling particles, thus damaging the surface characteristics of the restoration. Thus, an increase in SBS values was expected from the particle abrasion and diamond bur groups.

In previous studies (Kao *et al.*, 1995; Jost-Brinkmann *et al.*, 1996; Chunhacheevachaloke and Tyas, 1997; Lai *et al.*, 1999) that investigated the SBS of orthodontic brackets to restorative composite resin surface, the composite specimens used were not exposed to an ageing procedure simulating the oral condition before bonding of the brackets. Most of these investigations evaluated the bond strength of non-metal orthodontic brackets and various adhesives to resin composite surfaces. The SBS values found in those studies were higher than those in the current research. This may be due to their use of fresh composite specimens. Kao *et al.* (1995) in their study investigated the torsional bond strength of ceramic brackets to composite resin laminate veneers but did not use an ageing procedure for their specimens before bonding and found that torsional bond strength was higher than SBS.

According to the results of the current study, clinically adequate bond strengths were achieved in groups 3, 4, 5, and 6. It is suggested that the use of sandblasting with Al_2O_3 and sodium bicarbonate particles is more appropriate for roughening the composite surface to increase SBS, as producing a limited roughening area on the composite surface for bracket bonding may be easier and more controllable with these applications. Although clinically adequate SBS values were observed in groups 3 and 6, these methods are contraindicated due to the hazardous effect of hydrofluoric acid on the soft tissues and the uncontrollable abrasive effect of the diamond bur.

When any one of surface treatment protocols tested in this study is used during bonding, scar formation on composite surface created during surface treatment must be also taken into consideration after debonding. Remnant adhesives on the composite surface have to be carefully removed and the surface must be polished using composite finishing discs to regain a smooth and glossy composite surface. Thus, plaque accumulation and discolouration of composite restorations are prevented.

Conclusions

Orthodontic bonding to aged restorative resin composite was evaluated *in vitro* with five different surface conditioning methods. Within the limitations of this study, the conclusions are as follows:

1. Surface roughening is effective in bonding an orthodontic attachment to aged resin composite surfaces.

2. Clinically, adequate SBS values can be obtained with the application of hydrofluoric acid, sodium bicarbonate particle abrasion, AI_2O_3 particle abrasion, or a diamond bur.

3. The failure patterns of brackets were at the bracket–adhesive interface for sandblasting with Al_2O_3 particles and diamond bur application and at the composite–adhesive interface for phosphoric and hydrofluoric acids.

References

Anil N, Hekimoglu C, Buyukbas N, Ercan M T 2000 Microleakage study of various soft denture liners by autoradiography: effect of accelerated aging. Journal of Prosthetic Dentistry 84: 394–399

Anil N, Hekimoglu C, Sahin S 1999 Color stability of heat-polymerized and autopolymerized soft denture liners. Journal of Prosthetic Dentistry 81: 481–484

Årtun J, Bergland S 1984 Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. American Journal of Orthodontics 85: 333–340

Boyer D B, Chan K C, Reinhardt J W 1984 Build-up and repair of light-cured composites: bond strength. Journal of Dental Research 63: 1241–1244

Brosh T, Pilo R, Bichacho N, Blutstein R 1997 Effect of combinations of surface treatments and bonding agents on the bond strength of repaired composites. Journal of Prosthetic Dentistry 77: 122–126

Brunthaler A, König F, Lucas T, Sperr W, Schedle A 2003 Longevity of direct resin composite restorations in posterior teeth. Clinical Oral Investigations 7: 63–70

Buyukyilmaz T, Zachrisson B U 1998 Improved orthodontic bonding to silver amalgam. Part 2. Lathe-cut, admixed, and spherical amalgams with different intermediate resins. Angle Orthodontist 68: 337–344

Chiba K, Hosoda H, Fusayama T 1989 The addition of an adhesive composite resin to the same material: bond strength and clinical techniques. Journal of Prosthetic Dentistry 61: 669–675

Chunhacheevachaloke E, Tyas M J 1997 Shear bond strength of ceramic brackets to resin-composite surfaces. Australian Orthodontic Journal 15: 10–15

Dootz E R, Koran A, Craig R G 1993 Physical property comparison of 11 soft denture lining materials as a function of accelerated aging. Journal of Prosthetic Dentistry 69: 114–119

Ferracane J L, Marker V A 1992 Solvent degradation and reduced fracture toughness in aged composites. Journal of Dental Research 71: 13–19

Huang T H, Kao C T 2001 The shear bond strength of composite brackets on porcelain teeth. European Journal of Orthodontics 23: 433–439

Jost-Brinkmann P G, Can S, Drost C 1996 In-vitro study of the adhesive strength of brackets on metals, ceramic and composite. Part 2: bonding to porcelain and composite resin. Journal of Orofacial Orthopedics 57: 132–141

Kallio T T, Lastumäki T M, Vallittu P K 2001 Bonding of restorative and veneering composite resin to some polymeric composites. Dental Materials 17: 80–86

Kao E C, Eliades T, Rezvan E, Johnston W M 1995 Torsional bond strength and failure pattern of ceramic brackets bonded to composite resin laminate veneers. European Journal of Orthodontics 17: 533–540

Kitayama Y, Komori A, Nakahara R 2003 Tensile and shear bond strength of resin-reinforced glass ionomer cement to glazed porcelain. Angle Orthodontist 73: 451–456

Kocadereli I, Canay Ş, Akça K 2001 Tensile bond strength of ceramic orthodontic brackets bonded to porcelain surfaces. American Journal of Orthodontics and Dentofacial Orthopedics 119: 617–620

Kupiec K A, Barkmeier W W 1996 Laboratory evaluation of surface treatments for composite repair. Operative Dentistry 21: 59–62

Lai P Y, Woods M G, Tyas M J 1999 Bond strengths of orthodontic brackets to restorative resin composite surfaces. Australian Orthodontic Journal 15: 235–245

Li J 1997 Effects of surface properties on bond strength between layers of newly cured dental composites. Journal of Oral Rehabilitation 24: 358–360

Lucena-Martín C, González-López S, Navajas-Rodríguez de Mondelo JM 2001 The effect of various surface treatments and bonding agents on the repaired strength of heat-treated composites. Journal of Prosthetic Dentistry 86: 481–488

Ozcan M, Barbosa S H, Melo R M, Galhano G A, Bottino M A 2007 Effect of surface conditioning methods on the microtensile bond strength of resin composite to composite after aging conditions. Dental Materials 23: 1276–1282

Ozcan 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

Pounders B, Gregory W A, Powers J M 1987 Bond strengths of repaired composite resins. Operative Dentistry 12: 127–131

Powers J M, Dennison J B, Koran A 1978 Color stability of restorative resins under accelerated aging. Journal of Dental Research 57: 964–970

Ramoglu S I, Usumez S, Buyukyilmaz T 2008 Accelerated aging effects on surface hardness and roughness of lingual retainer adhesives. Angle Orthodontist 78: 140–144

Reynolds I R 1975 A review of direct orthodontic bonding. British Journal of Orthodontics 2: 171–178

Swift E J, Le Valley B D, Boyer D B 1992 Evaluation of new methods for composite repair. Dental Materials 8: 362–365

Tezvergil A, Lassila L V, Vallittu P K 2003 Composite-composite repair bond strength: effect of different adhesion primers. Journal of Dentistry 31: 521–525

Yap A U, Sau C W, Lye K W 1999 Effects of aging on repair bond strengths of a polyacid-modified composite resin. Operative Dentistry 24: 371–376

Zachrisson B U, Buyukyilmaz T, Zachrisson Y Ø 1995 Improving orthodontic bonding to silver amalgam. Angle Orthodontist 65: 35–42