SCIENTIFIC SECTION

An *ex vivo* investigation to compare orthodontic bonding using a 4-META-based adhesive or a composite adhesive to acid-etched and sandblasted enamel

S. A. Clark, P. H. Gordon and J. F. McCabe
Newcastle Dental School, UK

Abstract

Objective: This study investigates the shear-peel orthodontic bond strengths of brackets bonded with an unfilled acrylic resin containing 4-META (MCP Bond®) or a no-mix composite adhesive (Right On®) to acid-etched or sandblasted enamel.

Design: *Ex vivo*.

Materials and methods: Eighty human pre-molar teeth were separated into four equal groups, according to the adhesive used and method of enamel pre-treatment. Group I—Right On® with enamel etched using phosphoric acid for 30 seconds. Group II—Right On® with enamel sandblasted using 50-μm alumina particles at 80 psi for 3 seconds. Group III—MCP Bond® with enamel etched using phosphoric acid for 30 seconds. Group IV—MCP Bond® with enamel sandblasted using 50-μm alumina particles at 80 psi for 3 seconds. Subsequently, the specimens were stored in distilled water for 24 hours prior to bond strength testing using an Instron® universal testing machine. Each debonded tooth was scored using the adhesive remnant index (ARI) to determine the site of bond failure.

Results: The mean bond strength (1 SD) were Group I: 10.7 (2.7) MPa, Group II: 5.3 (1.3) MPa, Group III: 15.9 (3.4) MPa, Group IV: 15.0 (2.2) MPa. Statistical analysis using one-way analysis of variance and Tukey test found no statistical difference between Group III and Group IV ($P > 0.05$), but the other groups were statistically different from each other ($P < 0.05$). The data were found to fit the Weibull distribution and Weibull analysis showed stress required for a 5 per cent probability of failure was: Group I: 5.77 MPa; Group II: 3.32 MPa; Group III: 10.31 MPa; Group IV: 10.58 MPa. Chi-square test showed a statistically significant difference existed between the ARI scores ($P < 0.001$), principally through less adhesive remnants being observed on the sandblasted specimens.

Conclusion: The adhesive containing 4-META achieved significantly higher bond strengths than the composite adhesive, particularly in the case of sandblasted enamel.

Index words: 4-META, acid-etching, acrylic, orthodontic bonding, sandblasting.

Received 10 January 2002; accepted 21 March 2002

Introduction

Orthodontic brackets are routinely bonded to enamel using the acid-etch technique in conjunction with a composite-type orthodontic adhesive. Several alternative approaches to bonding, including the use of different enamel preparations[1–3] and adhesive systems[4–8] have been investigated, with the exception of resin modified glass ionomer cements,[5] they have not yet achieved the success required in order to justify their routine clinical use. Both the procedure used for enamel surface preparation[1–3,] and the nature of the adhesive[6–10] can affect the reliability of the bond, the ease of de-bond and the ease of enamel 'clean-up' procedures at the end of treatment.

Address for correspondence: Professor J. F. McCabe, Dental Materials Science Unit, Newcastle Dental School, Framlington Place, Newcastle Upon Tyne NE2 4BW, UK. Email: j.f.mccabe@ncl.ac.uk

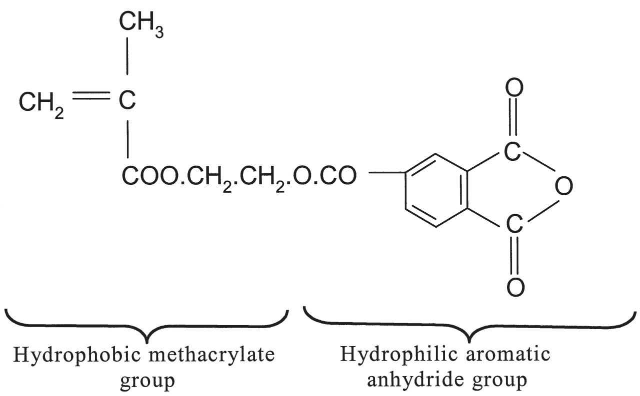

Fig. 1 Molecular structure of the 4-META monomer.

Recently, sandblasting has been introduced into dentistry as a means of surface preparation for bonding[11–13]. Furthermore, adhesion promoters such as 4-META (4-methacryloxyethyl trimelliticanhydride; Figure 1) used in conjunction with an unfilled resin offer the potential of adequate bonding combined with easy debond and clean-up at the end of treatment.

The aim of this investigation was to compare orthodontic bonding to enamel, which has been pretreated, either by acid-etching or sandblasting, and using a composite resin or an unfilled acrylic resin containing the adhesion promoter 4-META. The following parameters were investigated: shear-peel bond strength and site of bond failure as indicated by the Adhesive Remnant Index (ARI).[1]

Materials and methods

Tooth sample, grouping and mounting procedure

Eighty non-carious premolar teeth, extracted for orthodontic purposes from patients under the age of 18 years, were collected from three sources in the Northumbria region (Sunderland, Newcastle, and Gateshead). The teeth were stored separately, according to their source and extraction date, in 0.5 per cent (w/v) chloramine-T solution for 1 month at room temperature before being transferred into distilled water and refrigerated at 4°C before use. All teeth were stored for at least 1 month prior to bond strength testing and teeth not used within 6 months of their extraction date were discarded.[14] All teeth were examined under ×4 magnification to assess whether damage had occurred to the enamel surface during the extraction process. Any damaged teeth were discarded.

To minimize potential variability in the shear bond strength analysis resulting from tooth type[15], each experimental group was allocated 20 premolars in a stratified manner according to premolar nomenclature and tooth source, so each group had the same quantity of premolar types from each source.

In order to aid retention of a tooth in its mounting, each root was grooved using a high-speed bur. The vertically positioned tooth was then mounted in a polyester resin to a level 1 mm below the cemento-enamel junction. Possible dehydration of the tooth during the curing of the polyester resin was minimized by storing the ‘mounted tooth’ in a humidity chamber at room temperature for 4 hours, by which time the resin had reached its initial set. The ‘mounted tooth’ was then immersed in distilled water at room temperature, where the resin underwent a complete cure. The polyester resin mounting blocks were later trimmed so that the specimens could be mounted in a vice during bond strength testing. Particular attention was paid to ensure that the vertical axis of the tooth and bucco-lingual axis of the crown were parallel to the trimmed (clamping) sides of the resin-mounting block. Once these had been trimmed, the tooth/resin-mounting block was returned to distilled water and refrigerated to await bonding.

Adhesives used

Two types of orthodontic adhesives were evaluated in the current study and used according to the manufacturers’ instructions:

- Right On® (TP Orthodontics): a ‘no-mix’ autopolymerizing composite resin.
- MCP Bond® (Sun Medical Company): an autopolymerizing unfilled polymethylmetacrylate resin, containing 5 per cent of the adhesion promoter 4-META.

Enamel pretreatment

Two different methods of enamel pre-treatment were used:

1. *Acid-etched:* The buccal enamel surface was polished for 10 seconds with a rubber cup and oil/fluoride free pumice, followed by rinsing with distilled water for 10 seconds and drying with oil-free compressed air for 10 seconds. The tooth’s surface was etched for 30 seconds using the etchant supplied by the manufacturer. The tooth was finally rinsed with distilled water for 30 seconds and dried with oil-free compressed air for 30 seconds.

2. *Sandblasting:* The buccal enamel surface was evenly sandblasted in the area, where the bracket was to be bonded, using 50-μm aluminium oxide particles at 80 psi for 3 seconds at a distance of 10 mm from the tooth surface. The tooth was finally rinsed with distilled water for 30 seconds and dried with oil-free compressed air for 30 seconds.

Experimental groups

The four groups used were as follows:

1. Right On®/etched (control group). Right On® adhesive with acid-etched (manufacturer's etchant—37 per cent phosphoric acid) enamel pretreatment.
2. Right On®/sandblasted. Right On® adhesive with sandblasted enamel pretreatment.
3. MCP Bond®/etched. MCP Bond® adhesive with acid-etched (manufacturer's etchant—65 per cent phosphoric acid) enamel pretreatment.
4. MCP Bond®/sandblasted. MCP Bond® system with sandblasted enamel pretreatment.

Bracket placement technique

Premolar brackets (Ovation®, GAC) with a foil mesh base and an average base area of 14.93 ± 0.21 mm^2 and 15.26 ± 0.15 mm^2 for upper and lower brackets, respectively, were used. Bracket placement was performed at room temperature, i.e. 21 ± 2°C. Each bracket was positioned on the mid coronal point of the tooth and to avoid the entrapment of air within the adhesive, the bracket was initially placed along its gingival edge before being fully seated with a 'rolling' action. Firm pressure was applied to minimize the adhesive thickness and careful attention paid to ensure that adhesive extruded from the entire bracket base perimeter, thereby confirming the entire bonding surface was covered with adhesive. Any excess adhesive visible to the naked eye was removed immediately with a sharp straight probe. After bonding, all specimens were immersed in distilled water maintained at 37°C for 24 hours to await shear-peel bond strength testing.

Bond strength testing

The shear-peel bond strength was determined using an Instron® universal-testing machine. The specimens were removed from the distilled water, dried, and immediately mounted on the testing machine. The resin mounting block was positioned in the vice clamps so that the bracket base was vertical in all planes and directly below the load cell. A 0.8-mm diameter round stainless steel wire loop was attached via a universal joint to the load cell at one end and placed under the gingival tie wings adjacent to the bracket base at the other end. This ensured that the force vector was as near as possible parallel to the bracket base. Using a crosshead speed of 1 mm per minute, the shear force required to dislodge the bracket was measured.

Analysis of the site of bond failure

Debonded teeth and brackets were examined under ×30 magnification to evaluate and categorize the site of bond failure. Any excess adhesive retained around the bracket base periphery was ignored when considering the quantity of retained adhesive.[16] A modified version of the ARI[1] was used to assess the amount of resin retained on the enamel surfaces after debonding. The ARI categories used were:

- no adhesive retained on the tooth;
- less than or equal to 10 per cent of the adhesive retained on the tooth;
- greater than 10 per cent, but less than 100 per cent of the adhesive retained on the tooth;
- all adhesive retained on the tooth, with a distinct imprint of the bracket base.

Any enamel fractures that occurred during debonding were also recorded.

Statistical analysis

Descriptive statistics, including mean, standard deviation, and minimum and maximum stress values were calculated for each of the experimental groups. One-way analysis of variance (ANOVA) was used to determine whether significant differences existed between the 'means' of the various experimental groups. To determine if the 'means' were significantly different from each other, a Tukey test was employed at the chosen level of probability ($P < 0.05$). Weibull analysis was used to calculate the Weibull modulus, characteristic strength and the required stress for 1 and 5 per cent probabilities of failure.

Chi-square test was used to determine any significant differences between experimental groups' ARI scores.

Scanning electron micrograph specimen preparation

For each adhesive system tested, a bonded specimen was placed in 10 per cent hydrochloric acid to dissolve the tooth. The 'resin impression' (negative) of the enamel's pretreated surface was examined by a scanning electron microscope to demonstrate the quality of micro-mechanical retention achieved by each adhesive system.

Results

The mean shear bond stress, standard deviations, minimum and maximum stress values are tabulated for each experimental group in Table 1. One-way analysis of variance revealed a statistically significant difference between the mean values ($P < 0.001$) and Tukey test showed that there was a statistically significant difference ($P < 0.05$) between all experimental groups except MCP Bond®/etched and MCP Bond®/sandblasted.

Figure 2 shows a plot of the cumulative probability of failure against applied shear stress for each of the experimental groups. The plot illustrates that for probabilities of failure less than 30 per cent, the level of stress required to dislodge the brackets increases in the order: Right On®/sandblasted, Right On® etched, MCP Bond®/etched, and MCP Bond®/sandblasted. However, at higher levels of probability of failure, MCP Bond®/etched requires higher levels of stress than MCP Bond®/sandblasted for bond failure to occur.

Results from the Weibull analysis are displayed in Table 2. The Weibull modulus ranged from 4.20 for Right On®/etched to 7.20 for MCP Bond®/sandblasted, while the characteristic stress ranges from 6.03 MPa for Right On®/sandblasted to 17.25 MPa for MCP Bond®/etched. The stress levels required for 1 and 5 per cent probabilities of failure are shown for each group and their associated 95 per cent confidence intervals. Also shown are the groups that were not significantly differ-

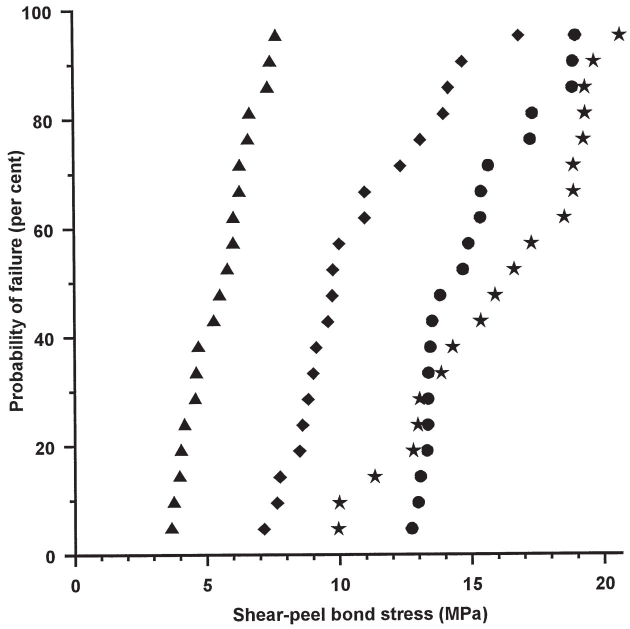

Fig. 2 Probability of failure versus shear bond stress for the adhesive systems. ▲, Right On® sandblasted; ◆ Right On® etched; ★ MCP Bond® etched; ● MCP Bond® sandblasted

Table 1 Shear bond stress for the adhesive systems

Adhesive system	Mean stress (MPa)	SD (MPa)	Minimum value (MPa)	Maximum value (MPa)
Right On® etched	10.65	2.70	7.15	16.86
Right On® sandblasted	5.32	1.27	3.67	7.68
MCP Bond® etched	15.91*	3.44	9.95	20.67
MCP Bond® sandblasted	15.02*	2.15	12.73	19.00

*Denotes no statistical significant difference between adhesive systems with Tukey test ($P > 0.05$).

Table 2 Weibull analysis of shear bond stress

Adhesive system	Weibull Modulus	Characteristic stress (MPa)	Stress for 1% probability of failure (MPa; 95% CI)	Stress for 5% probability of failure (MPa; 95% CI)
Right On® etched	4.20	11.70	$3.92^{A,B}$ (2.61–5.87)	5.77 (4.34–7.68)
Right On® sandblasted	4.98	6.03	2.40^{A} (1.67–3.44)	3.32 (2.58–4.28)
MCP Bond® etched	5.77	17.25	$7.77^{B,C}$ (5.62–10.74)	10.31^{D} (8.23–12.91)
MCP Bond® sandblasted	7.20	15.98	8.43^{C} (6.65–10.69)	10.58^{D} (8.95–12.50)

Matching superscript letters denotes no statistically significant difference between adhesive systems in the same column, at the 95% confidence interval (CI).

ent from one another at the 95 per cent confidence interval level. In fact, MCP Bond®/etched and MCP Bond®/sandblasted were not significantly different for all probabilities of failure at the 95 per cent confidence interval level.

The ARI was used as a means of defining the site of bond failure between the enamel, the adhesive and the bracket base. The distributions of scores are listed in Table 3. Statistical analysis using chi-square (χ^2) test was invalid as a number of the observed frequencies were less than 1 and more than 20 per cent of expected frequencies were less than 5. Pooling of ARI scores 1 and 2 and comparing these with ARI score 3 alone showed that there was a significant difference among the four groups ($P < 0.001$). The data distribution here indicated that bond failures for experimental groups with etched enamel were observed more frequently at the bracket-adhesive interface, as compared to the experimental groups with sandblasted enamel.

Table 3 also shows that six test specimens in the MCP Bond®/etched group experienced enamel fractures during debonding, an example of which can be seen in Figure 3.

Figures 4–7 show scanning electron micrographs of the 'resin impressions' achieved by the adhesive system for each experimental group.

Discussion

Reynolds[17] has suggested that for an adhesive system to have acceptable clinical performance *in vitro* bond strengths between 6 and 8 MPa are required. Using these values for this study, Right On®/etched would have a clinical failure rate of around 5 per cent, which can be

Table 3 The adhesive remnant index scores and enamel fractures for adhesive systems tested

Adhesive system	Adhesive Remnant Index				Number of teeth with enamel fractures
	1	2	3	4	
Right On® etched	1	8	11	0	0
Right On® sandblasted	4	13	3	0	0
MCP Bond® etched	0	3	17	0	6
MCP Bond® sandblasted	1	16	3	0	0

Pooling of ARI scores 1 and 2 and comparing with ARI score 3 alone, chi-square test shows a statistically significant difference between the adhesive systems ($\chi^2 = 28.44$, DF = 3, $P < 0.001$).
ARI categories: 1—no adhesive retained on the tooth; 2—less than or equal to 10 per cent of the adhesive retained on the tooth; 3—greater than 10 per cent, but less than 100 per cent of the adhesive retained on the tooth; 4—all adhesive retained on the tooth, with a distinct imprint of the bracket base.

Fig. 3 Scanning electron micrograph of a debonded MCP Bond®/etched specimen showing fractured enamel surface.

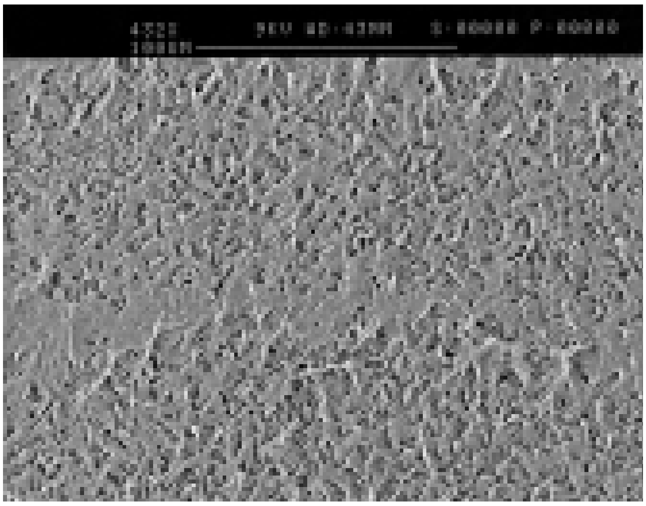

Fig. 4 Scanning electron micrograph of a Right On® 'resin impression' resulting from an etched enamel surface. Demonstrating a honeycomb etch pattern.

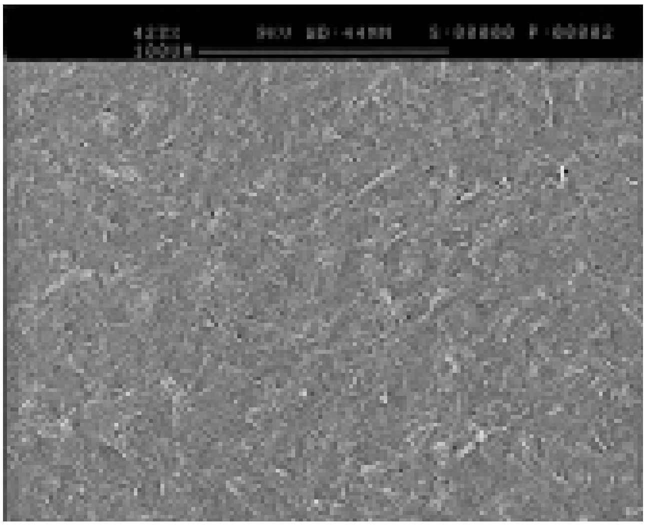

Fig. 5 Scanning electron micrograph of a Right On® 'resin impression' resulting from a sandblasted enamel surface.

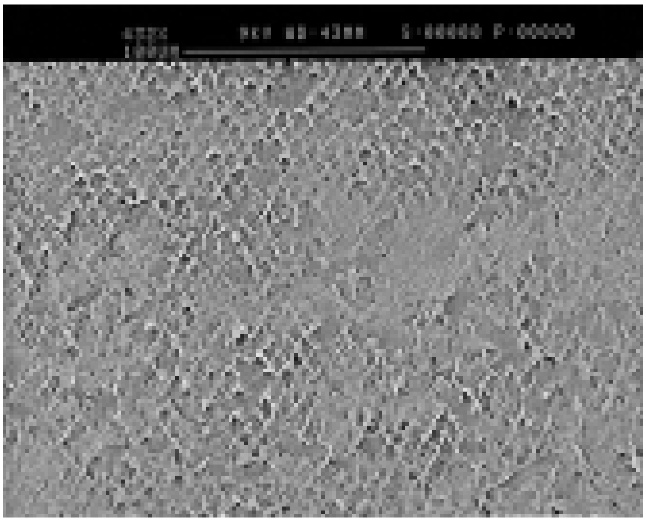

Fig. 6 Scanning electron micrograph of a MCP Bond® 'resin impression' resulting from an etched enamel surface. Demonstrating a cobblestone etch pattern.

considered acceptable. Right On®/sandblasted adhesive system is unlikely to be acceptable clinically, as its bond strengths were significantly lower than Right On®/etched and this concurs with previous research[3,18–21]. However, both MCP Bond®/etched and MCP Bond®/sandblasted adhesive systems showed potentially favourable bond strengths and warrant further discussion.

Analysis of the surface topography of sandblasted enamel[3,20] has shown it has a uniform roughness (Figures 5 and 7) that is comparable to acid-etched enamel. Furthermore, the etch patterns achieved using

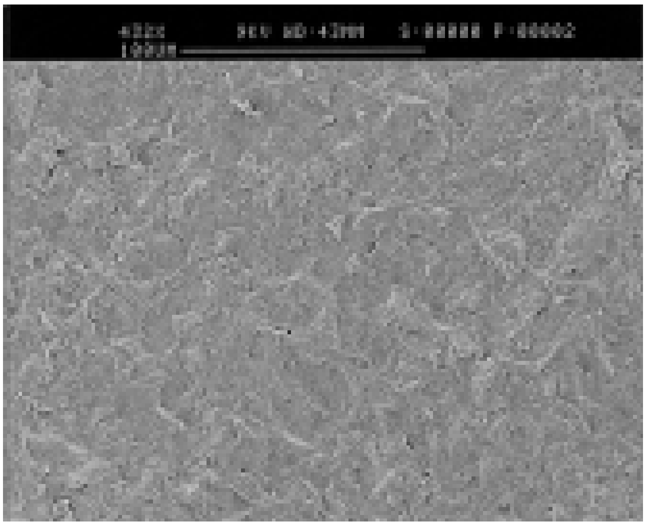

Fig. 7 Scanning electron micrograph of a MCP Bond® 'resin impression' resulting from a sandblasted enamel surface.

acid-etching have been shown to be somewhat unreliable[22] (Figures 4 and 6). A greater consistency in the surface roughness achieved by sandblasting compared with etching may explain the greater consistency (lower variance) of bond strengths seen in this study for sandblasted enamel compared with etched enamel.

The increased bond strengths achieved with MCP Bond® were most likely a result of it being an unfilled acrylic material containing 5 per cent 4-META monomer.[23] 4-META is a difunctional monomer exhibiting a hydrophobic methacrylate group and a hydrophilic aromatic anhydride group. Functionally, the hydrophobic methacrylate group is able to combine with resins in composite/acrylic adhesives, whilst the hydrophilic aromatic anhydride group is able to promote adhesion to the tooth surface. It is thought that increased bond strengths are achieved through an ability of 4-META to enhance diffusion into the tooth surface.[23,24] Such a mechanism would explain why bond strengths with MCP Bond® are higher than with the relatively hydrophobic Right On® adhesive. When comparing resin impressions of both Right On® and MCP Bond® to sandblasted enamel (Figures 5 and 7) no apparent difference is visible, however, further detailed investigation is required before this can be fully determined.

In this study the ARI was used to characterize the site of bond failure. It was observed that acid-etching of enamel resulted in larger amounts of adhesive residue on the enamel surface after de-bonding compared with sandblasted enamel, confirming previous findings.[3,18,19]

However, in the current work some adhesive remnants were also present in the Right On®/sandblasted group.

The bond strengths achieved by MCP Bond®/etched and MCP Bond®/sandblasted adhesive systems were greater than those of the control and in the MCP Bond®/etched group this sometimes led to enamel damage (Figure 3). While MCP Bond®/sandblasted results in bond failure principally along the enamel/adhesive interface, the stresses created at this interface did not result in any enamel fractures. Clinical debonding forces, whether accidental or deliberate, do differ from the *in vitro* forces used in this study. However, the performance of the MCP Bond®/sandblasted adhesive system, with its relatively soft acrylic resin, offers an easy and 'safe' debond and clean-up procedure, and these attributes would warrant further investigation.

Two principal concerns arise over the use of intra-oral sandblasting, namely the amount of enamel lost during enamel preparation, and the health and safety issues arising as a result of the potential inhalation of alumina particles. There is relatively little evidence available to indicate the enamel damage which may result from sandblasting, but where it exists, it would suggests that it is potentially no worse than acid-etching.[21]

As rubber dam isolation is not practical in orthodontics, the inhalation of alumina particles is potentially hazardous unless other adequate precautions are taken. Further work is now required to develop a sandblasting protocol, which can be used safely and effectively in orthodontics.

Conclusion

The results from this investigation indicate that:

1. MCP Bond® (containing 5 per cent 4-META) was shown to increase the shear-peel bond strength to acid-etched and sandblasted enamel surfaces compared to the composite resin Right On®.
2. MCP Bond®/etched and MCP Bond®/sandblasted adhesive systems showed comparable mean shear bond strengths and probabilities of failures for all given shear-peel bond stresses.
3. Acid-etched and sandblasted enamel pre-treatments showed mixed sites of bond failure with etched teeth showing higher levels of retained resin on the enamel surface than for sandblasted teeth.
4. High bond strengths achieved with MCP Bond®/etched may jeopardize enamel surfaces, potentially making this adhesive system clinically unacceptable.
5. MCP Bond®/sandblasted has shown favourable *in vitro* shear bond testing results and makes this adhesive system well suited to proceed to clinical trial.

Acknowledgements

The authors would like to acknowledge Mr T. E. Carrick and Mrs S. Rusby for their invaluable assistance during specimen testing and Dr T. Booth for his assistance with scanning electron microscopy.

References

1. Årtun J, Bergland S. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pre-treatment. *Am J Orthod* 1984; **85**: 333–340.
2. Fraunhofer JA, Allen DJ, Orbell GM. Laser etching of enamel for direct bonding. *Angle Orthod* 1993; **63**: 73–76.
3. Sargison AE, McCabe JF, Millett DT. A laboratory investigation to compare enamel preparation by sand blasting and acid etching prior to bracket bonding. *Br J Orthod* 1999; **26**: 141–146.
4. Norevall LI, Marcusson A, Persson M. A clinical evaluation of a glass ionomer cement as an orthodontic bonding adhesive compared with an acrylic resin. *Eur J Orthod* 1996; **18**: 373–384.
5. Fricker JP. A 12 month clinical evaluation of a light activated glass ionomer cement for the direct bonding of orthodontic brackets. *Am J Orthod Dentofacial Orthop* 1994; **105**: 502–505.
6. Moin K, Dogon IL. An evaluation of shear strength, measurements of unfilled and filled combinations. *Am J Orthod* 1978; **74**: 531–535.
7. Kusai Al-Munajed M, Gordon PH, McCabe JF. The use of a cyanoacrylate adhesive for bonding orthodontic brackets: an *ex vivo* study. *J Orthod* 2000; **27**: 255–260.
8. Ireland AJ, Sherriff M. Use of an adhesive resin for bonding orthodontic brackets. *Eur J Orthod* 1994; **16**: 27–34.
9. Cook PA, Youngson CC. An *in vitro* study of the bond strength of glass ionomer cement in the direct bonding of orthodontic brackets. *Br J Orthod* 1988; **15**: 247–253.
10. Pus MD, Way DC. Enamel loss due to orthodontic bonding with filled and unfilled resins using various clean-up techniques. *Am J Orthod* 1980; **80**: 269–283.
11. Zachrisson BU, Büyükyilmaz T. Recent advances in bonding to gold, amalgam and porcelain. *J Clin Orthod* 1993; **27**: 661–675.
12. Millet DT, McCabe JF, Gordon PH. The role of sandblasting on the retention of metallic brackets applied with glass ionomer cement. *Br J Orthod* 1993; **20**:117–122.
13. Wood DP, Paleczny GJ, Johnson LN. The effect of sandblasting on the retention of orthodontic bands. *Angle Orthod* 1996: **66**: 207–214.

14. ISO/TR 11405. *Dental Materials Guidance on Testing of Adhesion to Tooth Structure*. Technical report 1994(E).
15. Hobson RS, McCabe JF, Hogg SD. Bond strength to surface enamel for different tooth types. *Dent Mat* 2001; **17**: 184–189.
16. O'Brien KD, Watts DC, Read MJF. Residual debris and bond strength—is there a relationship. *Am J Orthod Dentofac Orthop* 1988; **94**: 222–230.
17. Reynolds IR. A review of direct orthodontic bonding. *Br J Orthod* 1975; **2**: 171–178.
18. Olsen ME, Bishara SE, Damon P, Jakobsen JR. Comparison of shear bond strength and surface structure between conventional acid etching and air abrasion of human enamel. *Am J Orthod Dentofac Orthop* 1997; **112**: 502–506.
19. Canay Ş, Kocadereli I, Akça E. The effect of enamel air abrasion on the retention of bonded metallic orthodontic brackets. *Am J Orthod Dentofac Orthop* 2000; **117**: 15–19.
20. Reisner KR, Levitt HL, Mante F. Enamel preparation for orthodontic bonding: a comparison between the use of a sandblaster and current techniques. *Am J Orthod Dentofac Orthop* 1997; **111**: 366–373.
21. Waveren Hogervorst WL, Feilzer AL, Prahl-Andersen B. The air-abrasion technique versus the conventional acid-etching technique: a quantification of the surface enamel loss and comparison of shear bond strength. *Am J Orthod Dentofac Orthop* 2000; **117**: 20–26.
22. Mattick CR, Hobson RS. A comparative micro-topographical study of the buccal enamel of different tooth types. *J Orthod* 2000; **27**: 143–148.
23. Hotta K, Mogi M, Mura F, Nakabayashi N. Effect of 4-MET on bond strength and penetration of monomers into enamel. *Dent Mat* 1992; **8**: 173–175.
24. Nakabayashi N. Biocompatibility and promotion of adhesion to tooth substrates. *CRC Critic Rev Biocompat* 1984; **1**: 25–52.