In vitro shear bond strength of orthodontic bondings without liquid resin

Alexander T. H. Tang, Lars Björkman, Ewa Adamczak, Anna Andlin-Sobocki and Jan Ekstrand

Unit of Basic Oral Sciences, Institution of Odontology, Karolinska Institutet, Huddinge, Sweden

Tang ATH, Björkman L, Adamczak E, Andlin-Sobocki A, Ekstrand J. In vitro shear bond strength of orthodontic bondings without liquid resin. Acta Odontol Scand 2000;58:44–48. Oslo. ISSN 0001-6357.

This study aimed at evaluating the early shear bond strength of enamel-composite-bracket adhesion accomplished without the use of liquid resin. Orthodontic brackets were bonded to the buccal surfaces of healthy extracted premolars in the test group by Transbond XT ($n = 8$) and Phase II ($n = 8$) composites but not the enclosed liquid resins in these products. Brackets bonded with the same materials ($n = 8$ for each) along with their corresponding liquid resin served as controls. The specimens were tested for shear bond strength after 24-h storage in water at 37°C. The fractured surfaces were graded with Adhesive Remnant Index (ARI) under a 2×-dissection microscope. Enamel of the randomly selected test and control specimens was dissolved by 20% formic acid. Afterwards, the enamel side of the bonding materials in both groups ($n = 4$) was examined under the scanning electron microscope. ANOVA was used for statistical analyses. Our laboratory data suggest that the enamel adhesion produced by these two commercial materials without the use of liquid resin does not differ significantly in their early in vitro shear bond strength. □ *Enamel adhesion; orthodontic bondings; shear bond strength*

Jan Ekstrand, Unit of Basic Oral Sciences, Institution of Odontology, Karolinska Institutet, Box 4064, SE-14104, Huddinge, Sweden. Tel. +46 8 58587967, fax. +46 8 7745935, e-mail. Jan.A.Ekstrand@ofa.ki.se

The use of acid etching and bis-GMA-based liquid resin to promote the bond strength of orthodontic attachments to enamel has survived with great clinical success for more than 3 decades (1, 2). Its clinical applications in orthodontic treatment and subsequent retention are well documented (3–5).

It has been postulated that enamel adhesion is achieved by mechanical interlocking between the etched enamel prisms and resin tags (6, 7). This necessitates the use of a fluid unfilled liquid resin to thoroughly wet the porous enamel surface for adhesion (8, 9). Opposite opinions argue that the use of unfilled liquid resin does not promote the bond strength of enamel adhesion (10, 11). If enamel adhesion does not entirely depend on mechanical interlocking between the etched enamel prisms and resin tags, orthodontic attachments may be bonded without the use of liquid resin.

Previous works carried out in a calibrated in vitro model suggest that chemically cured liquid-paste orthodontic bonding materials are more cytotoxic than light cured and chemically cured 2-paste materials (12, 13). The results of these studies suggest that the source of cytotoxicity stems from the liquid component of the materials. Although the implication of in vitro data is uncertain in an in vivo environment, the search for an orthodontic bonding technique on enamel which obviates the use of liquid resin is of interest in, at least, a biological perspective. The aim of this study was to evaluate the early shear bond strength of enamel-composite-bracket adhesion without the use of liquid resin.

Materials and methods

Teeth

Thirty-nine healthy first and second premolars, extracted for orthodontic reasons, were collected from dental clinics in the County Council of Uppsala, Sweden. The average age of patients was 14.4 years, with a range from 13.0 to 16.8 years. The teeth were cleaned using tap water and afterwards stored in physiological saline at 4°C. The storage period did not exceed 3 months. The extracted teeth were examined under a 2×-dissection microscope with adequate lighting to exclude teeth with major enamel defects ($n = 3$). Nine and 27 of the remaining teeth ($n = 36$) were collected from male and female patients, respectively. All of them were mounted in Vel-mix stone (Kerr Italia, Torino, Italy) and a brass ring with the cemento-enamel junction embedded in the dental stone (Fig. 1). The teeth were aligned so that the tangent of the mid-buccal surface of the tooth was perpendicular to the bottom of the brass ring.

Tested materials

Two bis-GMA based bonding materials, Transbond XT (3M Unitek, California, USA) and Phase II (Reliance, Illinois, USA) were used in this study. Corresponding Victory metal brackets (3M Unitek, California, USA) were bonded onto the buccal surfaces of all the extracted teeth by the same operator. A sharp scaler was used to remove excessive bonding materials before they set hard. In the control group ($n_{\text{Transbond XT}} = 9$, $n_{\text{Phase II}} = 9$), the bonding materials were handled in accordance with the manufacturer's instruction. The same procedures were carried out in the test group ($n_{\text{Transbond XT}} = 9$, $n_{\text{Phase II}} = 9$), but the application of the liquid resin on the etched enamel surface prior to the placement of brackets was spared. For Transbond XT, a Demetron VCL 400 light curing unit was used to cure the bonding material. Its output was

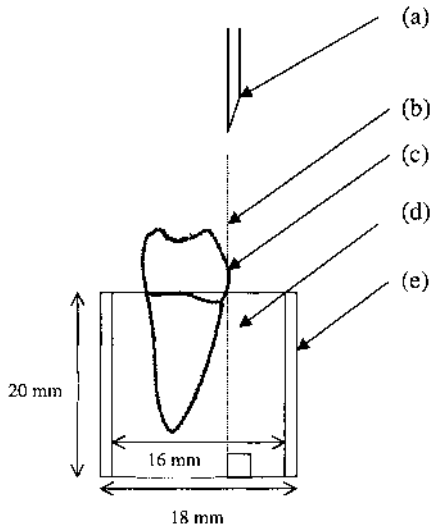

Fig. 1. Schematic diagram of the experimental design, (a) knife-edge metal rod applying a shear force perpendicular to the base of the brass ring, (b) tangent to the mid-buccal surface of the mounted tooth, (c) mid-buccal surface aligned perpendicular to the base of the brass ring and where the bracket was bonded, (d) dental stone, and (e) brass ring.

monitored by a Demetron radiometer each time before and after use to secure proper curing of the materials.

Experimental design

All the specimens in both groups were kept for 24–27 h in water at 37°C before testing. Shear bond strengths of specimens in each ($n = 8$) of the four groups were assessed using the Alwetron TCT 50 (Lorentzen & Wettre AB, Kista, Sweden). Shear stress, at 1.0 mm/min, was applied to the edge of the bracket base in a direction perpendicular to the bottom of the brass ring using a knife-edge metal rod. The shear bond strength at which the bracket dislodged from the enamel surface was recorded by a computer.

The fractured surfaces of the bondings were examined carefully under a 2×-dissection microscope (Olympus, Tokyo, Japan) with adequate lighting. The patterns of the fracture surfaces were recorded with the Adhesive Remnant Index (ARI) (14).

Specimens ($n = 4$) from test and control groups bonded with Transbond XT and Phase II were randomly chosen and decalcified in 20% formic acid solution. After decalcification, the bracket-resin specimens were immersed in de-ionized water. They were subsequently oven dried at 37°C and sputtered to give a 20 nm platinum coating (Polaron, Wafford, England). The enamel surfaces of the resins were examined under scanning electron microscope (JEOL 820, Tokyo, Japan). The observations were computerized by KS400 v2.0 (Kontron Elektronik, Munich, Germany) and documented by photographic printouts.

Statistical analysis

Shear bond strength in MegaPascal (MPa) was obtained by dividing the shear bond force (N) by the surface area of the bracket base (approximately 12.5 mm^2). Data obtained from the shear stress experiments were analyzed by ANOVA. The dependent variable was the shear stress (MPa). The independent factors were the materials (Transbond XT and Phase II) and bonding techniques (test and control groups). All statistical procedures were carried out in SPSS 9.0.

Results

Shear stress test

The shear bond strengths of the test and control groups are shown as means, standard deviations, and 95% confidence intervals in Table 1. For Transbond XT, the test group had its mean bond strength approximately 2.5 MPa higher than that of the control. The mean bond strength of the test group of Phase II, however, was roughly 2.5 MPa lower than the control. Nonetheless, their 95% confidence intervals covered similar ranges of shear bond strength, implying that true difference might not exist between the test and control groups. Statistical analyses by ANOVA indicated that there was neither significant difference in shear bond strength between the test and control groups ($P = 0.762$, df = 1, F = 0.094) nor significant difference in shear bond strength between the two commercial materials Phase II and Transbond XT ($P = 0.100$, df = 1, F = 2.887).

Scanning electron micrographs

In the control group, both bonding materials showed well-formed resin tags on the enamel side of the composite (Fig. 2). Penetrations of resin tags into the peripheral sheaths and cores of the etched enamel prisms were observed. In the test group, Transbond XT and Phase II composites only showed solid and amorphous surfaces, although the textures of the two materials were different (Fig. 3).

Table 1. Shear bond strength of the test and control groups of Transbond XT and Phase II. Shown are the number of teeth tested (*n*), mean, standard deviation (SD), and 95% confidence interval (95% CI) in MegaPascal (MPa) of the specimens tested

Groups	*n*	Mean	SD	95% CI
Transbond XT				
Test	8	20.6	3.0	18.5–22.6
Control	8	18.0	4.3	15.0–21.0
Phase II				
Test	8	15.0	6.4	10.5–19.4
Control	8	18.5	1. 9	17.2–19.8

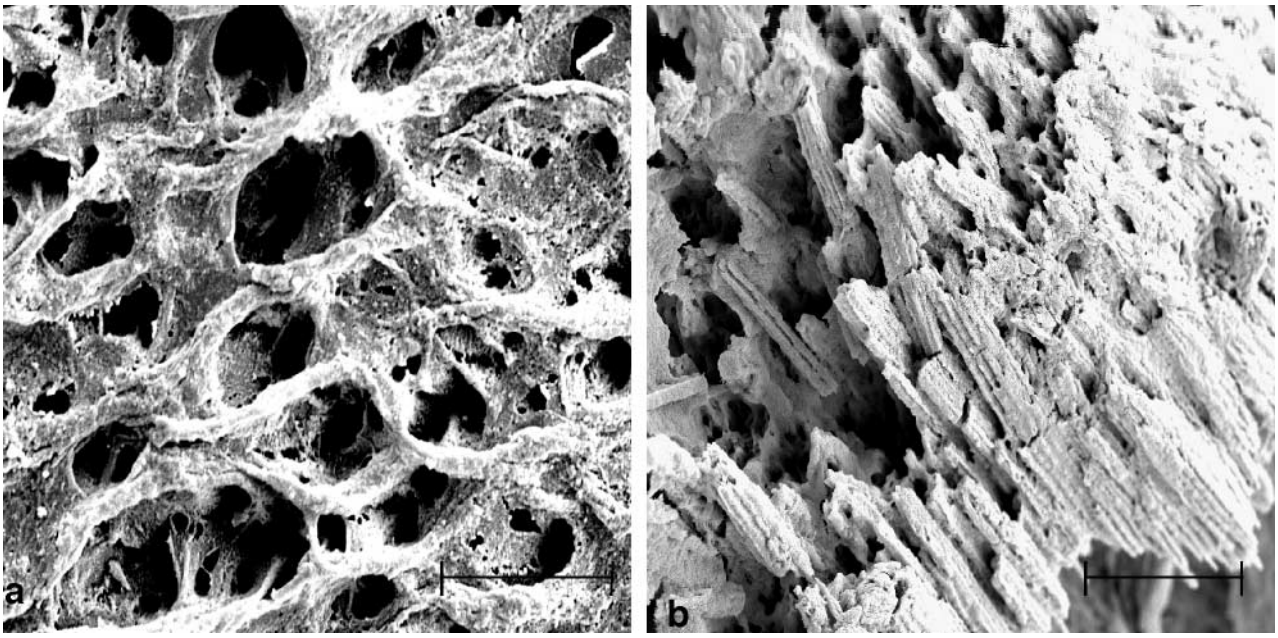

Fig. 2. Scanning electron micrographs of the enamel side of the bonding materials (a) Transbond XT (×1000, WD = 15 mm, 15 kV, scale bar = 25 μm) and (b) Phase II (×150, WD = 15 mm, 15 kV, scale bar = 100 μm) of the control group. Both bonding materials displayed well-defined resin penetration into the etched enamel.

Observations under dissection microscope

The observations under the dissection microscope are summarized in Table 2.

Transbond XT. Both test and control groups exhibited the same pattern of bond failure under shear stress. A quarter of the teeth in the test and control groups had no composite left on the tooth surfaces upon debonding. Half of the teeth in both groups had approximately 10% to

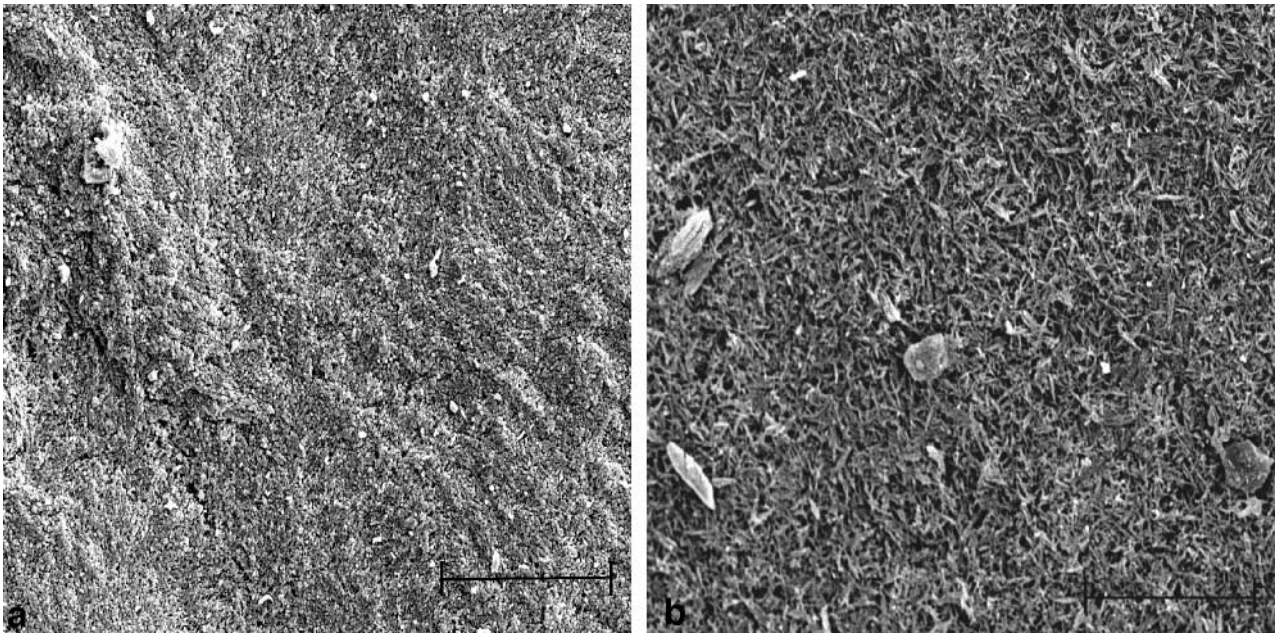

Fig. 3. Scanning electron micrographs of the enamel side of the bonding materials (a) Transbond XT (×1000, WD = 15 mm, 15 kV, scale bar = 25 μm) and (b) Phase II (×1000, WD = 15 mm, 15 kV, scale bar = 25 μm) of the test group. Both bonding materials displayed amorphous surfaces.

Table 2. Patterns of fractured surfaces indicated by Adhesive Remnant Index (ARI). Shown are the number of teeth tested (*n*) and the percentage of teeth in the test and control groups of Transbond XT and Phase II under scores 0–3 of ARI

Materials	*n*	0	1	2	3
Transbond XT					
Test	8	25.0	50.0	12.5	12.5
Control	8	25.0	50.0	12.5	12.5
Phase II					
Test	8	62.5	37.5	0	0
Control	8	12.5	50.0	0	37.5

ARI: 0–3.
0 = No adhesive remained on the tooth.
1 = <50% adhesive remained on the tooth.
2 = >50% adhesive remained on the tooth.
3 = 100% adhesive remained on the tooth, with distinct impression of the bracket mesh.

20% of the composites left on the tooth surfaces. Another quarter of the teeth in both groups had 60% to 100% of the composites left on the teeth. In other words, the weakest points of both the test and control groups were located between the tooth surface and the composite.

Phase II. In the control group, 12.5% of the teeth had no composite left on the tooth surface. Half of the teeth in this group had 10% or less material left on the tooth surfaces. In contrast to the test group, 37.5% of the failure occurred at the composite bracket interfaces, which left all the materials on the tooth surfaces; 62.5% of the teeth in the test group had no composite left on the tooth surfaces upon debonding; 37.5% of the teeth in this group had approximately 10% of the composites left on the teeth. In other words, the weakest point of the test group was located between the tooth surface and the composite.

Discussion

This study aimed at assessing the feasibility of achieving reliable enamel adhesion using an orthodontic bonding technique free of liquid resin. The results indicate that in the absence of liquid resin and hence the resin tags for mechanical interlocking, the shear bond strengths of enamel adhesion in the test and control groups are similar. The mean, standard deviations, and confidence limits of the shear bond strengths in both test and control groups are higher than the recommended 5.9 to 7.8 MPa (15). Additionally, observations from the electron micrographs in the present study confirm that in the absence of the liquid resin, no interlocking tags are formed. This observation is contrary to previous findings (16, 17). It seemed to us that the early strength of enamel adhesion might well be obtained by bonding without the use of a wetting liquid resin to achieve mechanical interlocking between the etched enamel prisms and the resin tags. A previous in vitro study of the tensile bond strength of Phase II without the use of liquid resin seemed not to lower the bond strength to any significant extent (18). Our results supplemented these findings in a shear direction. Unfortunately, the data of the present study cannot verify which mechanism is most important in securing enamel adhesion.

Surface tension might play a role in securing enamel adhesion. By acid etching, the surface tension of enamel could be raised from 28 to 72 dynes/cm (19). Close adaptation of composites on enamel surfaces might lower and hence stabilize the high surface tension of the etched enamel. This may explain the success of the liquid resin-free technique in vitro. The pressure during bracket placement and the roughness of the etched enamel surface probably enable the close adaptation (wetting) of composites on enamel surface (20). The importance of surface tension in enamel adhesion, however, was uncertain. The data in this study seem to favor surface tension as a major binding force in enamel adhesion. However, epoxy resin was initially developed as industrial glue for hard surfaces (21). Since enamel is a highly mineralized hard surface, perhaps a clean and dry surface produced by acid etching is all that is needed for the 'glue' to work.

A shift in the adhesion fracture pattern was recorded by ARI for Phase II but not for Transbond XT between the test groups and the control groups. Phase II was relatively more viscous in consistency. The use of liquid resin might dilute Phase II composite and might bring about a weak point in the composite at the composite–bracket interface. On the other hand, Transbond XT is less viscous in consistency. The use of liquid resin might not give much additional dilution effect in a less than 0.3 mm thick material (22).

An in vitro study has shown that only the topical application of aminfluoride, among aminfluoride, sodium fluoride, and acidulated phosphate fluoride, can reduce the polar component of free surface energy of enamel (23). All the extracted teeth were collected from Uppsala, Sweden, where the natural fluoride level in drinking water is roughly 1 ppm. It did not seem to compromise the shear bond strength of enamel adhesion to any significant extent in the experimental environment of this study compared with previously reported data (24–27).

ISO 11405 recommends 3 methods of specimen storage before mechanical testing (28). Short-term storage, i.e. in a 37°C water bath for 24 h, was chosen for this study before subjecting the specimens to shear stress. Long-term and thermocycling storage methods do not seem to simulate the in vivo environment in all aspects. The in vivo performance of the fixed appliances bonded with liquid resin-free technique will be assessed in a future clinical trial. Nonetheless, the early in vitro shear bond strength seems promising.

In conclusion, our data suggest that Transbond XT and Phase II might enable enough shear bond strength between sound enamel surface and metal brackets without the use of liquid resin. This might imply that mechanical interlocking between the etched enamel prisms and resin tags is probably not the only important mechanism by

which to secure enamel adhesion. The clinical application of orthodontic bonding without liquid resin has yet to be supported by clinical trials.

Acknowledgement.—This study was supported by a grant from the Swedish Medical Research Council (09439-05X).

References

1. Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. J Dent Res 1955;34: 849–53.
2. Newman GV. Epoxy adhesives for orthodontic attachments: progress report. Am J Orthod 1965;51:701.
3. Gwinnett AJ. State of the art and science of bonding in orthodontic treatment. Council on Dental Materials, Instruments and Equipment. J Am Dent Assoc 1982;105:844–50.
4. Zachrisson BU. A post-treatment evaluation of direct bonding in orthodontics. Am J Orthod 1977;71:173–89.
5. Zachrisson BU. Bonding in orthodontics. In: Graber TM, Vanarsdall RL, editors. Orthodontics: Current principles and techniques. 2nd ed. St. Louis: Mosby; 1994. p. 542–626.
6. Gwinnett AJ, Buonocore MG. Adhesives and caries prevention. Br Dent J 1965;123:77–80.
7. McCabe JF, Walls AWG. Adhesive restorative materials: bonding of resin-based materials. In: McCabe JF, Walls AWG, editors. Applied dental materials. 8th ed. Oxford: Blackwell Science; 1998. p. 189–201.
8. Matasa CG. Adhesion and its ten commandments. Am J Orthod Dentofac Orthop 1989;95:355–6.
9. Galan D, Lynch E. Principles of enamel etching. J Ir Dent Assoc 1993;39:104–11.
10. Raadal M. Bond strength of composites applied to acid-etched enamel. Scand J Dent Res 1978;86:157–62.
11. Retief DH, Woods E. Is a low viscosity bonding resin necessary? J Oral Rehab 1981;8:255–66.
12. Tang ATH, LI J, Ekstrand J, Liu Y. Cytotoxicity tests of *in situ* polymerized resins: methodological comparisons and introduction of tissue culture insert as a testing device. J Biomed Mater Res 1999;45:214–22.
13. Tang ATH, Liu Y, Björkman L, Ekstrand J. *In vitro* cytotoxicity of orthodontic bonding resins on human oral fibroblasts. Am J Orthod Dentofac Orthop 1999;166:132–8.
14. Årtun J, Bergland S. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. Am J Orthod 1984;85:333–40.
15. Reynolds IR. A review of direct orthodontic bonding. Br J Orthod 1979;2:171–8.
16. Asmussen E. Penetration of restorative resins into acid etched enamel. I. Acta Odontol Scand 1977;35:175–82.
17. Jorgensen KD, Shimokobe H. Adaptation of resinous restorative materials to acid etched enamel surfaces. Scand J Dent Res 1975;83:31–6.
18. Wang WN, Tarng TH. Evaluation of sealant in orthodontic bonding. Am J Orthod Dentofac Orthop 1991;100:209–11.
19. Jendresen MD, Glantz PO. Clinical adhesiveness of selected dental materials. An *in vivo* study. Acta Odontol Scand 1981;39:39–45.
20. Jendresen MD, Glantz PO, Baier RE, Eick JD. Microtopography and clinical adhesiveness of an acid etched tooth surface. An *in vivo* study. Acta Odontol Scand 1981;39:47–53.
21. White IR. Adhesives. In: Adams RM, editor. Occupational skin diseases. 2nd ed. Philadelphia: W. B. Saunders Company; 1990. p. 395–407.
22. Ceen RF, Gwinnett AJ. Microscopic evaluation of the thickness of sealants used in orthodontic bonding. Am J Orthod 1980; 78:623–9.
23. de Jong HP, van Pelt AWJ, Busscher HJ, Arends J. The effect of topical fluoride applications on the surface free energy of human enamel—an *in vitro* study. J Dent Res 1984;63:635–41.
24. Legler LR, Retief DH, Bradley EL, Denys FR, Sadowsky PL. Effects of phosphoric acid concentration and etch duration on the shear bond strength of an orthodontic bonding resin to enamel. An *in vitro* study. Am J Orthod Dentofac Orthop 1989;96:485–92.
25. Ødegaard J, Segner D. The use of visible light-curing composites in bonding ceramic brackets. Am J Orthod Dentofac Orthop 1990;97:188–93.
26. Jacobs G, Kuftinec MM, Showfety KJ, von Fraunhofer JA. Bonding characteristics of impacted versus erupted permanent teeth. Am J Orthod 1986;89:242–5.
27. Komori A, Ishikawa H. Evaluation of a resin reinforced glass ionomer cement for the use as an orthodontic bonding agent. Angle Orthod 1997;67:189–96.
28. Guidance on testing of adhesion to tooth structure. International Organization of Standardization, Geneve, ISO 11405 TR; 1994.

Received for publication 28 June 1999
Accepted 5 October 1999