BASIC SCIENCE RESEARCH

An In Vitro Investigation of a Comparison of Bond Strengths of Composite to Etched and Air-Abraded Human Enamel Surfaces

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Purpose: The purposes of the study were to measure the tensile bond strength of composite resin to human enamel specimens that had been either etched or air-abraded, and to compare the quality of the marginal seal, through the assessment of microleakage, of composite resin to human enamel specimens that had been either etched or air-abraded.

Materials and Methods: Thirty mandibular molar teeth were decoronated and sectioned mesio-distally to produce six groups, each containing ten specimens that were embedded in acrylic resin using a jig. In each of the four treatment groups, the specimen surfaces were treated by either abrasion with 27 or 50 μm alumina at 4 mm or 20 mm distance, and a composite resin was bonded to the treated surfaces in a standardized manner. In the two control groups the specimens were treated with 15 seconds exposure to 36% phosphoric acid gel and then similarly treated before being stored in sterile water for 1 week. All specimens were then subjected to tensile bond strength testing at either 1 or 5 mm/min crosshead speed. For the microleakage study, the degree of dye penetration was measured 32 times for each treatment group, using a neutral methylene blue dye at the interface between composite and either 27 or 50 μm air-abraded tooth structure or etched enamel surfaces.

Results: The mean bond strength values recorded for Group 1 (phosphoric acid etch, 5 mm/min crosshead speed) was 25.4 MPa; Group 2 (phosphoric acid etch, 1 mm/min), 22.2 MPa; Group 3 (27 μm alumina at 4 mm distance), 16.8 MPa; Group 4 (50 μm alumina at 4 mm distance), 16.9 MPa; Group 5 (27 μm alumina at 20 mm distance), 4.2 MPa; and for Group 6 (50 μm alumina at 20 mm distance) 3.4 MPa. An analysis of variance (ANOVA) demonstrated significant differences among the groups, and a multiple comparison test (Tukey) demonstrated that conventionally etched specimens had a greater bond strength than air-abraded specimen groups. No significant difference in dye penetration could be demonstrated among the groups ($p = 0.58$).

Conclusions: Composite resin applied to enamel surfaces prepared using an acid etch procedure exhibited higher bond strengths than those prepared with air abrasion technology. The abrasion particle size did not affect the bond strength produced, but the latter was adversely affected by the distance of the air abrasion nozzle from the enamel surface. The crosshead speed of the bond testing apparatus had no effect on the bond strengths recorded. The marginal seal of composite to prepared enamel was unaffected by the method of enamel preparation.

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DURING THE past few decades scientific developments in cariology, dental materials, and diagnostic systems have changed the approach to the diagnosis and management of dental

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caries.[1] The use of air abrasion as an alternative technique for the removal of enamel and dentine has been discussed.[2-4] Advantages of this technique have been reported to include reduced pressure, noise, and vibration compared with the use of rotary instruments, and the technique has been reported to be well tolerated by patients.[5] However, this technique is not without its limitations in that for those cavities to be restored using amalgam or indirect cast restorations, there is a need for additional finishing of the cavities with rotary instruments.

Air abrasion has been described to involve the use of aluminum oxide powder, which is carried in a fine stream of compressed air. As the particles collide with a solid target, in this case enamel or dentine, the kinetic energy of the particles is released, resulting in fracture of microscopic fragments of the target. The target substance must be hard and brittle enough to cause rapid deceleration of the particles so that their kinetic energy can cause a destructive collision. Soft targets are resilient to air abrasion. Therefore, enamel and dentine is cut readily while soft tissues remain unaffected.

In the 1980s, the use of this alternative technique, in particular one which did not require adjunctive local anaesthesia, was generally well received. This, however, gradually diminished, partly due to concerns regarding the potential toxicity of inhaled aluminum oxide particles coupled with the frequent need to use conventional rotary instruments to finish cavity preparation. It has, however, been suggested that air abrasion is re-emerging as a clinical technique for removing hard tooth substance. The main reasons for this are improved suction technology together with reassurance that inhaled aluminum oxide particles do not pose a health hazard. The further development of adhesive restorative materials has also allowed conservative tooth preparation without the need for cavity finishing with rotary instruments. The use of air abrasion as a treatment modality for phobic patients and its use as a diagnostic aid in assessing pit and fissure caries are now well documented.[4-6]

It has been suggested that air abrasion at high pressure can roughen the tooth surface, making it receptive to composite resin without the need for acid-etching, but some reports show disappointing bond strengths when air abrasion is employed.[7-9] It has also been suggested that materials that have a poor bond strength to tooth substance are at risk of microleakage with its potential complications. Studies on microleakage of adhesive materials to tooth substance are well documented.[2,11,12] The effects of microleakage include an increase in the risk of secondary caries, the production of marginal stain, an increase in the risk of hypersensitivity, an increase in the risk of insult to the pulp, and an acceleration of the breakdown of the restorative material.

Considering the developments in the technology for air abrasion, coupled with the improvements in the adhesive restorative materials, the hypotheses for this study were:

1. The tensile bond strength of composite resin to air-abraded enamel and acid-etched enamel would be similar.
2. If the bond strengths for composite resin bonded to air-abraded and acid-etched enamel were similar, they should be equally susceptible to microleakage.

The aims of this study were to

1. measure the tensile bond strength of composite resin to human enamel specimens that had been either etched or air-abraded, and
2. compare the quality of the marginal seal of composite resin to human enamel specimens that had been either etched or air-abraded.

Materials and Methods

The materials used in this study were selected as representative of materials currently in use in clinical practice. The composite resin used in this study was Spectrum (Dentsply DeTrey, Germany), a light cured hybrid composite consisting of polymerizable bisGMA monomers, TEGDMA diluent, photoinitiators, stabilizers, sub-micron filler particles of bariumaluminum-borosilicate, and highly dispersed silicon oxide. The total inorganic filling was 77% by weight. The bonding agent used was Prime&Bond NT (Dentsply DeTrey), a universal self-priming dental adhesive designed for enamel and dentine. It consisted of di- and trimethacrylate resins, amorphous silica, PENTA, photoinitiators, stabilizers, and cetylamine hydrofluoride in an acetone solvent.

Specimen Preparation

Tooth preparation

Recently extracted, non-carious human third molar teeth, were used for the preparation of the enamel specimens. The extracted teeth were cleaned with a hand scaler to remove tissue remnants and stored in thymol, containing water. A water-cooled Microslice 2 sectioning machine (Metals Research Ltd, Cambridge, UK) was used to remove the roots from each tooth. The roots were discarded and the crowns hemisected in the mesio-distal plane. Each crown was carefully examined, and the flattest of the smooth enamel tooth surfaces was selected for use in the study. After sectioning, pulpal remnants were removed using an excavator.

Mechanical retention was created by cutting two or three shallow grooves into the inner dentine surface of the specimens. Each specimen was placed with the enamel surface contacting a small piece of adhesive material (Blutak, Bostik-Findley, Staffordshire, UK) in the central area of the base of the flask (Fig 1). The flask was filled with auto-polymerizing poly (methyl methacrylate) acrylic resin (Simplex Rapid, KemDent, Wiltshire, UK) and left in warm water for 30 minutes to allow polymerization to proceed to completion. Once polymerized, the block of acrylic resin was removed from the flask to reveal an area of exposed enamel held securely in a cylindrical block of acrylic resin.

The surface of the enamel specimens was flattened using a lapping machine together with wet 1200 grade glass paper. The surface of the enamel was checked regularly during the lapping procedure with a notched aluminum guide to demarcate the area required for bonding. If flat enamel did not fill the bonding area (circular area of 2.5 mm diameter, 4.91 mm^2) or if dentine became exposed, the specimen was discarded.

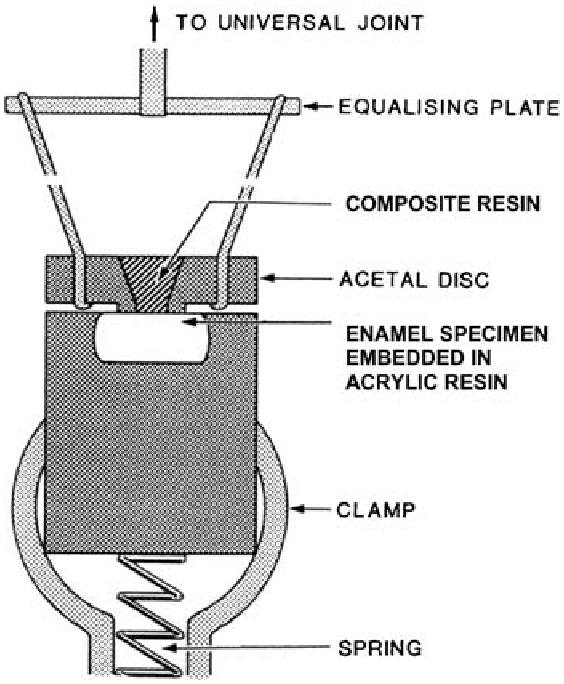

Figure 1. A diagrammatic representation of the jig used to hold the enamel specimens in acrylic resin for testing.

Surface preparation

Tooth specimens were divided into six groups, each containing ten specimens, according to the surface treatment used.

Group 1	Surfaces were etched using 36% buffered phosphoric acid etchant and tested at 5 mm/s crosshead speed.
Group 2	Surfaces were etched using 36% buffered phosphoric acid etchant and tested at 1 mm/s cross-head speed.
Group 3	Surfaces were air-abraded at 4 mm distance using 27 μm aluminum oxide particles.
Group 4	Surfaces were air-abraded at 4 mm distance using 50 μm aluminum oxide particles.
Group 5	Surfaces were air-abraded at 20 mm distance using 27 μm aluminum oxide particles.
Group 6	Surfaces were air-abraded at 20 mm distance using 50 μm aluminum oxide particles.

Etching

The exposed enamel surface of the specimens in Groups 1 and 2 was acid-etched for 15 seconds using 36% phosphoric acid gel and washed for 15 seconds. The specimen was then lightly dried with air using a dental triple syringe. One layer of Prime&Bond NT was applied to the whole surface of enamel using the applicator provided, left undisturbed for 20 seconds, then gently blown with air to remove solvent. The adhesive was immediately light cured for 10 seconds, and the specimen inserted into the aluminum tube of the bonding jig. The jig contained a spring that ensured the specimen was compressed against an acetal disc that would contain the composite resin in a centrally placed countersunk well. The acetal discs were lightly coated with PTFE aerosol lubricant to prevent composite adhering directly to them. The specimen was placed in the jig to allow a bonding area of 2.5 mm in diameter and 7 mm in thickness. Composite resin was added and cured for 40 seconds.

The acrylic resin block with attached composite sample was removed from the jig and placed in a thermocycling machine. Specimens were cycled for 24 hours with a dwell time of 20 seconds in 37°C distilled water, followed by 20 seconds in 5°C distilled water before being returned to the jig for subsequent testing with the Lloyd's machine (Model L2000R, Lloyd Instruments, Southampton, UK). For testing, the acetal ring was connected rigidly to the upper member of the Lloyd's bond strength testing machine.

Air abrasion

Specimens in Groups 3 to 6 were prepared by subjecting the enamel surface to air abrasion with either 27 or

50 μm aluminum oxide particles using an air abrasion machine with a 90° handpiece and 0.018″ aperture (Air Touch Tower Unit, Dentsply, UK). The air pressure was regulated to 80 psi (5.18 bar) for a time exposure of 5 seconds using a 0.018 nozzle.

The Air Touch handpiece was held securely in a retort stand with the nozzle tip positioned at one of the two specified distances from the enamel surface. It was oriented carefully so the stream of abrasion particles was perpendicular to the enamel surface. The specimen was held upright in a lapping guide so it could be moved under the stream of particles. The air abrasion was performed over a laboratory extraction tray with good suction to prevent excessive dust contamination of the environment. Following air abrasion, the specimen was washed and dried before the application of the Prime&Bond NT. The specimens were then assembled in the jig as previously described.

Bond Strength Testing

After thermocycling, the specimens were tested to failure under a tensile load on the Lloyd's machine. The acetal ring containing the composite resin was held in the lower member of the jig and connected rigidly to the load cell by way of a self-centering metal connector. The crosshead speed of the load cell was set at either 5 mm/min or 1 mm/min. The force at failure was recorded and the tensile bond strength was calculated in MPa.

After bond strength testing, all enamel and composite fracture surfaces were examined by one operator under the stereo-microscope, to record their mode of failure. Failure was recorded as either

1. *Adhesive failure* if the fracture was seen at the interface between the composite and enamel, or
2. *Cohesive failure within the composite* if the fracture was localized within the composite adjacent to the bond, or
3. *Cohesive failure within the tooth* if the fracture was contained within the tooth substance adjacent to the bond.

Where a combination of failures occurred, the enamel surface was examined, and the fraction of bonding area with composite still attached was estimated to evaluate the predominant mode of failure.

Statistical Analysis

The results were subjected to statistical analysis using a one-way analysis of variance (ANOVA) and, where appropriate, Tukey test.

Investigation of microleakage

The materials used for this investigation were the same as those described for the bond strength study. Enamel surfaces of non-carious human third molar teeth were used in this study. The teeth were not sectioned, but cleaned using a slurry of pumice and a rubber polishing cup operating at 1500 rpm. Specimens were then washed and dried with air from a triple dental syringe.

Six teeth were used and divided into three groups. Teeth 1 and 2 acted as controls and were acid-etched with 36% phosphoric acid. Teeth 3 and 4 were prepared with the "Air Touch" set at 80 psi and using 27 μm alumina particles. Teeth 5 and 6 were prepared as above at the same 4 mm distance from the tooth but with 50 μm alumina. In all cases, a foil template with a 2 mm × 10 mm rectangular window, was firmly held over the buccal and lingual/palatal surfaces of the tooth and the exposed area of enamel etched for 15 seconds or air-abraded for 5 seconds. The settings and nozzle size used with the "Air Touch" machine were the same as for those used during the tensile bond strength testing.

The composite resin was applied to the exposed surface of the tooth using the matrix and light cured for 40 seconds. After the light curing, two coats of an acid resistant nail varnish were painted over the whole crown to within 1 mm of the bonded composite specimens. The teeth were immersed in small glass vials containing neutral methylene blue dye for 1 week. The teeth were removed from the dye and the roots resected. The crowns were sectioned parallel to their approximal surfaces using the microslice machine. In total, the cut surfaces provided 16 sites per tooth where the interface between the composite and enamel could be examined under 10× magnification to assess the degree of dye penetration.

The 16 measurements made for each tooth were categorized into four depths of dye penetration. The results of the three groups were evaluated statistically using a chi-square test with a predetermined confidence interval of 95%.

Results

Tensile Bond Tests

The results of the tensile bond tests are given in Table 1. A one-way ANOVA found significant difference among the groups tested ($F = 25.92$; $p < 0.01$). Pairwise multiple comparisons tests (Tukey) demonstrated that the control groups (Groups 1 and 2) etched with phosphoric acid did not have a significantly greater bond strength to enamel than the specimens that had been

Table 1. Tensile Bond Strengths Achieved Between Composite and Etched or Air-Abraded Human Enamel Together with Modes of Failure

Test Group	*Tensile Bond Strength (MPa) Mean ± SD*	*% Mode of Failure*		
		Adhesive	*Cohesive*	*Tensile*
Group 1; AE, NT, and Comp.	25.44 ± 8.47	20	70	10
Group 2; AE, NT, and Comp. (low crosshead speed)	22.25 ± 2.93	20	40	40
Group 3; no AE, AB 27 μm, 4 mm, NT, and Comp.	16.82 ± 6.20	40	60	0
Group 4; no AE, AB 50 μm, 4 mm, NT, and Comp.	16.90 ± 5.44	40	60	0
Group 5; no AE, AB 27 μm, 20 mm, NT, and Comp.	4.18 ± 2.24	100	0	0
Group 6; no AE, AB 50 μm, 20 mm, NT, and Comp.	3.42 ± 2.30	100	0	0

Values connected by a vertical bar are not significantly different.

air-abraded at 4 mm from their surface (Groups 3 and 4).

The specimens that had been air abraded at 20 mm distance from the enamel surfaces had a significantly lower tensile bond strength to enamel than all other groups.

Mode of Failure

An analysis of the specimens demonstrated that adhesive failures were associated with the lowest bond strengths while predominantly cohesive failures were associated with greater bond strengths. Cohesive failures within tooth were associated only with the highest bond strengths.

Microleakage

The results are presented in Table 2. Using a dye penetration test, no significant difference in microleakage could be demonstrated between composite bonded to acid-etched enamel and composite bonded to enamel air-abraded with 27 μm diameter particles of aluminum oxide or enamel air-abraded with 50 μm diameter particles of aluminum oxide.

Discussion

This study was undertaken to investigate whether the use of air abrasion for the preparation of enamel surfaces and for bonding to composite resin, was as effective as conventional acid etching, in terms of the bond strength of enamel to composite resin produced. The results of the tensile bond strengths achieved in the current study are in agreement with those from other reported studies. There is a wide range of bond strengths reported in the current literature ranging from 12 MPa to 35 MPa.[7,9,13] The recommended bond strength required to resist polymerization shrinkage is reported to be 17 MPa on average. For those specimens subjected to air abrasion at 4 mm distance (Groups 3 and 4), the values were 16.8 MPa and 16.9 MPa for 27 μm and 50 μm, respectively. Those specimens subjected to air abrasion at 20 mm distance (Groups 5 and 6) had low values of 4.18 MPa and 3.42 MPa for 27 μm and 50 μm, respectively. However, those specimens subjected to acid etching (Groups 1 and 2) had high values of 25.4 MPa and 22.2 MPa for 5 mm/s and 1 mm/s crosshead speeds. It is, however, difficult to make direct comparisons with the results of other

Table 2. Microleakage Between Composite and Etched or Air-Abraded Human Enamel Was Measured Using a Dye Penetration Method with Methylene Blue

	Number of Sites with Microleakage of 0 mm	*Number of Sites with Microleakage of <0.5 mm*	*Number of Sites with Microleakage of 0.5 to 0.99 mm*	*Number of Sites with Microleakage of >1.0 mm*
Test group				
Tooth 1 and 2 (acid etch)	26	4	2	0
Tooth 3 and 4 (air abrasion with 27 μm)	25	5	2	0
Tooth 4 and 5 (air abrasion with 50 μm)	27	3	2	0

Sixteen measurements were made for each of the two teeth in each group. The amount of leakage was categorized into four depths of penetration. No significant differences could be demonstrated among the groups $\chi^2 = 0.577$, DF = 4, $p > 0.05$.

studies due to the differences in methodology used.

It is appreciated that the tensile bond strength testing apparatus relies on the acetal disc being located parallel to the enamel surface so that the column of composite is perpendicular to that surface when the bonding takes place. This avoids shearing forces on the composite during the testing procedure that could reduce the tensile bond strength. Every attempt was made to ensure this. An additional potential source of error could also have occurred during the packing of the composite resin into the central well of the acetal disc of the testing jig. If the composite resin was not well adapted to the enamel surface, a reduced area for the bonding interface that could adversely affect the results would be present; however, those specimens that were poorly packed were clearly visible on removal from the jig and were discarded. Poorly packed specimens were easily seen after the bond strength testing was complete and these specimens were discarded.

Previous studies have used a variety of crosshead speeds in their methodology, and this can make comparison between results from different studies difficult. Therefore, in this study the effect of the crosshead speed was also investigated. In the current study, 5 mm/min was used as the standard and is faster than recommended in other studies.[14,15] The tensile bond strengths achieved when this was lowered to 1 mm/min were not significantly different. This would therefore suggest this is not a significant part of the methodology for bond strength testing.

In this study, the bond strength achieved for those specimens which were subjected to air abrasion was significantly lower than those subjected to acid etching; however, there are some clinical situations when it may be preferable to have a lower bond strength, such as for the cementation of a resin bonded porcelain veneer, whereby if debonding was to occur, failure would occur at the bonding interface rather than within the tooth structure. Similarly, the bonding of fissure sealants to enamel do not require a high tensile bond strength and there is evidence to support that air-abraded surfaces do not require etching before fissure sealants are placed;[16] however, it should be noted that it has been documented that etching after air abrasion ensures the highest bond strength.[8,9]

The results from the current study showed no significant difference in mean tensile bond strength between specimen groups bonded onto enamel air-abraded with 27 μm and 50 μm diameter particles of aluminum oxide. The mode of failure was similarly unaffected by the particle size. It may have been expected that the smaller particles would have created a more retentive microscopic pattern, or that the greater kinetic energy of the larger particles would have resulted in a deeper, more aggressive pattern. This is an area that requires further investigation using scanning electron microscopy of the abraded surfaces and measuring the surface roughness using profilometry.

There was a significant reduction in mean tensile bond strength when the nozzle of the air abrasion apparatus is moved away from enamel surfaces. This result is not surprising, as the 20 mm distance tested is much greater than that recommended by the manufacturer. It is suspected that over this distance the aluminum oxide particles lost too much kinetic energy to be able to alter the enamel surface sufficiently to make it receptive to bonding. The results of this study would support the manufacturer's recommendations in terms of distance from the tooth surface when using the air abrasion system.

With regards to the microleakage study, the results indicated that there was no significant difference in microleakage between composite resin bonded to enamel specimens that had been acid-etched and those air-abraded with 27 μm or 50 μm diameter particles of aluminum oxide. In this study, composite bonded to air-abraded enamel produced as good a seal as composite bonded to acid-etched enamel; however, as the bond strength for air-abraded specimen groups is lower and close to the force of polymerization shrinkage, there would be a greater risk of this bond failing due to polymerization shrinkage and cuspal flexure. Therefore, there could be an increased risk of subsequent microleakage in the long term.

The results of this study would suggest that there is adhesion of composite resin to enamel that has been air-abraded; however, the bond is of a lower strength than that achieved when bonding to enamel which has been acid-etched. The results do not suggest that air abrasion should replace acid etching as a method for rendering enamel receptive to adhesion, but it may be acceptable in some clinical procedures where a lower strength bond is required.

Conclusions

Within the limitations of the methodology of the study, the main conclusions which can be drawn are as follows.

The control groups (Groups 1 and 2) that had been etched with phosphoric acid had a non-significantly greater tensile bond strength to enamel than the specimens that had been air-abraded at 4 mm from their surface (Groups 3 and 4).

The specimens that had been air-abraded at 20 mm distance from the enamel surfaces had a significantly lower tensile bond strength to enamel than all other groups.

The abrasion particle size did not affect the bond strength and the bond strength was adversely affected by increasing the distance of the air abrasion nozzle from the enamel.

Decreasing the crosshead speed of the tensile bond strength testing machine did not significantly affect the bond strength.

No significant difference in microleakage could be demonstrated using a dye penetration test between composite bonded to acid-etched enamel and composite bonded to enamel air-abraded with 27 μm diameter particles of aluminum oxide or enamel air-abraded with 50 μm diameter particles of aluminum oxide.

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