A NOVEL MICROSHEAR TEST TO MEASURE DENTAL ADHESION

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INTRODUCTION

A key factor in determining the strength and durability of polymeric dental restorative composites, sealants and adhesives is the quality of their interfaces/interphases. Bulk mechanical strength tests such as tensile, transverse and shear tests are commonly used in studying the interfacial properties of dental composites. For evaluating the adhesion between dental composites and tooth structure, uniaxial tensile or shear bond tests are routinely applied to relatively large areas of enamel and/or dentin. Human enamel consists mainly of apatitic calcium phosphate arranged in a highly ordered prismatic array [1, 2]. This prismatic structure causes enamel to behave anisotropically. Similarly, dentin has an anisotropic character because of its ordered tubular structure [3, 4]. Thus, depending on the position and area of the bonding site, both enamel and dentin can present a complex, variable substrate for dental adhesion studies. In an effort to address these potential regional and size effects on bonding to tooth structure, several microtensile bond test methods recently have been developed [4, 5]. Although these micro-bond tests have a number of advantages (e.g., more precise correlation of adhesion with substrate loci) over both conventional macrotensile and macroshear bond tests, they are highly labor-intensive with regard to specimen preparation. The primary purpose of this study was to develop a microshear bond test that has many of the advantages of the microtensile methods but without their highly labor-intensive nature.

An additional purpose was to study how regional tooth structure variation and the orientation of enamel prisms and dentinal tubules, due to tooth sectioning, affects the bonding ability of a self-etching primer system and a conventional adhesive system that uses a separate acidic conditioner (mass fraction, 40 % phosphoric acid, H_3PO_4). Some of the opportunities that the microshear bond strength test offers in enhancing our understanding of dental adhesive systems also will become evident.

MATERIALS AND METHODS

The most important elements of the microshear test come with the design of the test. As seen in Figure 1, cylinders of cured composite resin are bonded onto a prepared substrate and tested in shear by activating the microvise. Tooth slices were taken from extracted human molars with the selected enamel region sectioned transversely (horizontal), obliquely, and parallel (axial and horizontal) to the enamel prism (Figure 2). Each slice was approximately 1.0 mm thick. The enamel or dentin slices were bonded to aluminum tabs by first air abrading the aluminum with 50 µm aluminum oxide powder followed by application of a cyanoacrylate adhesive. The exposed tooth surface was then resurfaced under water with 320 grit SiC paper. The tooth was treated with either a conventional adhesive system (Clearfil Photobond) or a self-etching primer system (Clearfil Liner Bond 2V). The protocol for the conventional adhesive system included: etching the enamel with the H₃PO₄ gel (K-etchant gel, Kuraray) for 30 s, rinsing the enamel with H₂O and drying (but not desiccating); then Photo-Bond adhesive resin was applied. The self-etching primer protocol included: conditioning with the self-etch primer for 30 s and then removing the excess with air. The adhesive resin was then applied and light-cured. Subsequently, an iris was cut from a micro-bore Tygon tube that had an internal diameter of approximately 0.7 mm and affixed to the enamel or dentin surface; the resin composite was injected into the iris. The resin was photo-irradiated with visible light (maximum absorbance peak 470 nm) for 60 s. After curing, the iris was removed and the specimens were stored in distilled water at 37 °C for 24 h. The cylindrical specimens were tested in shear at a rate of 0.5 mm/min. The change in load as a function of time was recorded and the shear strength was calculated from the equation $\tau =$ $F/(\pi r^2)$, where τ is the estimated interfacial shear strength, F is the load at failure, and r is the radius of the resin cylinder.

RESULTS AND DISCUSSION

In the case of enamel bonding, the bond strengths of the conventional adhesive system were significantly influenced by the anisotropic structure of enamel; high at the surface perpendicular to the enamel prisms (26 MPa to 31 MPa) and low at the surface parallel to the enamel prisms (13 MPa to 14 MPa; one way ANOVA, P < 0.05, Student's t-test, P < 0.05). The lower bond strengths obtained with the H₃PO₄ based adhesive, when the bonding surface was parallel to the enamel prisms, probably resulted from a weakened apatitic interface created by the over-etching with H₃PO₄. By contrast, the effect of the self-etching primer was less influenced by the orientation of the prismatic structure of enamel; 20 MPa

to 22 MPa was obtained from all surfaces (one way ANOVA, P > 0.05; Student's t-test, P > 0.05). With both bonding systems, no significant differences in enamel bonding between cuspal and middle coronal enamel were observed (Student's t-test, P > 0.05). However, significant differences were observed in the case of the horizontal sections with the conventional system (Student's t-test, P < 0.05) for axially and obliquely sectioned enamel.

For the seven cuspal specimens tested with rods that were sectioned transversely (horizontally); the mean shear strength and standard deviation was 31.1 ± 3.8 MPa. The result for five specimens that were sectioned obliquely was 27.8 ± 2.2 MPa. The result for eight specimens that were cut parallel to or longitudinally along the prisms was 13.9 ± 3.1 MPa. The results of this particular study showed that the surface parallel to the enamel prism rods was significantly weaker than were the surfaces cut transversely or obliquely. (One-way analysis of variance, F = 60.8; df = 5, 34; p < 0.05).

In the case of dentin bonding, the self-etching primer system gave significantly higher bond strengths (22 MPa to 26 MPa) than the conventional (12 MPa to 16 MPa; Student's t-test, P < 0.05). With regard to sectioning orientation and region, no significant differences were observed with either of the systems (one-way ANOVA, P > 0.05), but the mean shear bond strengths tended to be slightly higher toward the cusp, and lower close to the root dentin. SEM microphotography showed that the selfetching primer effectively modified the smear layer without being excessively destructive of either enamel or dentin surfaces. The moderate surface treatment of dentin by the self-etching primer may account for the high bond strengths obtained with this adhesive system. By contrast, the H₃PO₄ based adhesive gave lower bond strengths to dentin because the hybrid layer formed does not provide good mechanical interlocking with the infused, polymerized resin due to acid-induced collapse of demineralized collagen.

CONCLUSIONS

A microshear test has been developed that allows one to study adhesion of very small areas of tooth structure $(0.4 \text{ mm}^2 \text{ to } 1.0 \text{ mm}^2)$ that are not accessible to conventional macroshear testing. Thus, the researcher can now horizontally and vertically map interfacial adhesion of different areas and depths of tooth structure, respectively. Furthermore, this method requires significantly fewer extracted teeth for adhesion studies compared to the typical macro adhesion test methods.

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Figure 2. Tooth showing sectioning orientation

Tooth Region	Cut Direction	Bonding Agent	
		H_3PO_4 etchant	Self-etching primer
Enamel		K-etchant +	Clearfil Liner Bond
		Clearfil Photobond	2 V
Cusp	Horizontal	$31.1 \pm 3.8 (n=7)^{a}$	$21.9 \pm 3.3 (n=7)^{e, t, g}$
	Axial	$13.9 \pm 3.1 (n=8)^{h, 1}$	$21.3 \pm 2.3 (n=7)^{t,g}$
	Oblique	$27.8 \pm 2.2 (n=5)^{a, b}$	$22.2 \pm 2.4 (n=6)^{t,g}$
Mid-coronal	Horizontal	$14.2 \pm 2.5 (n=7)^{h, i}$	$20.1 \pm 2.9 (n=8)^{g}$
	Axial	$13.3 \pm 2.7 (n=7)^{h, 1}$	20.3 ± 2.4 (n=6) ^g
	Oblique	$26.5 \pm 1.8 (n=6)^{b, c}$	$21.2 \pm 3.3 (n=6)^{t,g}$

Table 1. Micro-shear bond strength of Enamel (MPa \pm SD)

Table 2. Micro-shear bond strength of Dentin (MPa \pm SD)

Tooth Region	Cut Direction	Bonding Agent	
		H_3PO_4 etchant	Self-etching primer
Dentin		K-etchant +	Clearfil Liner Bond
		Clearfil Photobond	2 V
Cusp	Horizontal	$16.1 \pm 3.2 (n=10)^{h}$	$26.2 \pm 3.7 (n=6)^{b, c, d}$
	Axial	$16.1 \pm 3.2 (n=8)^{h, 1}$	$23.4 \pm 3.2 (n=6)^{d, e, t, g}$
	Oblique	$15.0 \pm 2.4 (n=5)^{h, 1}$	$23.8 \pm 2.8 (n=7)^{c, d, e, t}$
DEJ	Horizontal	$15.1 \pm 2.0 (n=7)^{h,1}$	$24.9 \pm 1.6 (n=6)^{c, d, e}$
	Axial	$15.0 \pm 2.7 (n=9)^{h, 1}$	$22.2 \pm 2.6 (n=7)^{e, t, g}$
	Oblique	$14.5 \pm 2.3 (n=5)^{h, 1}$	$22.8 \pm 2.2 (n=10)^{e, t, g}$
Root	Horizontal	$12.2 \pm 2.9 (n=5)^{i}$	$22.5 \pm 2.1 (n=5)^{e, f, g}$
	Axial	$13.3 \pm 2.3 (n=8)^{h,i}$	$21.9 \pm 3.8 (n=8)^{d, e, f, g}$
	Oblique	$13.0 \pm 2.8 (n=6)^{h, i}$	$22.0 \pm 2.6 (n=6)^{e, f, g}$

DEJ is the dentinoenamel junction

Numbers in parentheses represent the numbers of samples tested.

SD is the standard deviation of the measurement.

Sample inner diameter; 0.673 mm \pm 0.0269 mm

Groups identified by different superscript letters were significantly different (P < 0.05).

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