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The Effect of Subject Age on the Microtensile Bond Strengths of a Resin and a Resin-modified Glass Ionomer Adhesive to Tooth Structure

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Clinical Relevance

For the materials tested, there was no difference in adhesion to young and aged tooth structure.

SUMMARY

In this study, the microtensile bond strengths of an etch-and-rinse resin adhesive to dentin and enamel and a resin-modified glass ionomer adhesive to dentin were determined on teeth known to have originated from subjects over 60 years of age. The same tests were repeated on teeth originating from young subjects. The resin adhesive was Prime & Bond NT (Caulk/Dentsply), while the resin-modified glass ionomer adhesive was Fuji Bond LC (GC America). Both were paired with the same hybrid resin composite, TPH[3] (Caulk/Dentsply). Testing was performed after 48 hours using a "non-trimming" microtensile test at a crosshead speed of 0.6 mm/minute. No significant differences were observed between the young and aged teeth for any comparison (*p*>0.05). SEM evaluation of the etched dentinal surfaces demonstrated less depth of decalcification in the intertubular areas of aged dentin, but there was no observable difference within the tubules of young and aged dentin.

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INTRODUCTION

For many years, the application of resin to enamel that has been etched with phosphoric acid has produced a strong, well-sealed interface, while more recently, the adhesion of resin to dentin has become feasible with the advent of hydrophilic resins capable of infiltrating into and polymerizing within a moist decalcified dentinal surface to form a hybrid layer.[1]

Whether these adhesive techniques are equally effective in young and older patients has not been established. It has been repeatedly demonstrated *in vitro* that most of the mechanical properties of dentin decline with the increased age of subjects.[2-4] It has been stated that aged dentin may be considered sclerotic, due to a narrowing of the dentinal tubules,[3-4] but it has not been established whether this narrowing is equivalent to sclerotic root surface dentin, which has a hypermineralized surface due to exposure to oral conditions.[5] The laboratory bond strengths of resin adhesives to sclerotic root surface dentin are lower than that observed with normal dentin,[6-7] although the clinical performance of both resin and resin-modified glass ionomer adhesives in restorations of non-carious Class V lesions is not adversely affected by sclerosis.[8-11] Although it has been established that resin/dentin hybrid layers are thinner in teeth derived from aged subjects versus those of young subjects,[12] only one study to date has addressed the effect of the subject's age on the bond strength of adhesives to tooth structure. In 1993, Tagami and others[13] found no difference in bond strength to young or aged dentin of four dentin-adhesive resins of that time period.

This study determined the effect of subject age greater than 60 years on the microtensile bond strengths of a current etch-and-rinse resin adhesive to dentin and enamel and a resin-modified glass ionomer adhesive to dentin.

METHODS AND MATERIALS

Extracted teeth were collected in accordance with the human subjects' regulations at the Medical College of Georgia. Clinicians who harvested the teeth were asked to segregate those extracted from patients age 60 or more and, over approximately one year, collected 15 caries-free molars that had been extracted for periodontal reasons. These teeth were matched to an equal number of molars collected over the same period by the same clinicians and which were judged by appearance to have been erupted and to have originated from young adult patients. All of the teeth were stored in water saturated with thymol.

For dentin adhesion testing, the occlusal enamel of each tooth was removed using a slow-speed water-cooled saw (Isomet, Buehler, Ltd, Lake Bluff, IL, USA) equipped with a diamond-impregnated disk, followed by hand finishing for 30 seconds on wet 320-grit silicon carbide paper, to create a realistic smear layer on the surface of the occlusal mid-coronal dentin. For enamel adhesion testing, flat surfaces were prepared on the facial and lingual surface enamel above the height of contour using 320-grit silicon carbide paper under running water at 60 rpm on a polishing machine (Ecomet 3, Buehler, Ltd). These flat areas were prepared in a plane estimated to be parallel to the underlying dentino-enamel junction.

The etch-and-rinse resin adhesive chosen for the study was Prime & Bond NT (Dentsply/Caulk, Milford, DE, USA), while the resin-modified glass ionomer adhesive chosen was Fuji Bond LC (GC America, Alsip, IL, USA). Both adhesives have been effective in the clinical trials of non-retentive Class V restorations,[8,14-15] and both were paired with the hybrid resin composite TPH[3] (Dentsply/Caulk). Each adhesive was applied according to the manufacturer's instructions, which are listed in Table 1. Following application of the adhesives, approximately 6 mm thick cores of the resin composite were built incrementally on the flattened dentin or enamel surfaces of each tooth. Increment thickness was limited to 2 mm, and initial curing was accomplished with the light directed perpendicular to the flattened area of the tooth for 40 seconds per increment using a fast halogen light source (VIP, BISCO, Inc, Schaumburg, IL, USA). Final curing was accomplished with the light directed perpendicular to the initial cure, with an additional 45 seconds curing at each of four locations around the circumference of the core build-up, for a total of five minutes of curing per specimen. Light output was verified throughout the study as 600 mW/cm^2, using the unit's built-in radiometer.

Table 1: *Manufacturer, Resin Composite, Instructions/Technique for Adhesives*

	Technique
Prime & Bond NT Lot 051211	Etch 15 seconds, rinse, blot dry Apply, keeping surface wet, for 20 seconds Air dry, light cure for 10 seconds
Caulk 34% Tooth Conditioner Gel Lot 0507252 (Caulk/Dentsply, Milford, DE USA)	(phosphoric acid etchant)
Fuji Bond LC Lot 0501111	Condition 10 seconds, rinse, lightly air dry Mix two scoops powder, 1 drop liquid Apply, light cure for 20 seconds
Cavity Conditioner Lot 0412281 (GC America, Alsip, IL, USA)	(polyacrylic acid 20-25% + aluminum chloride hexahydrate 1-5% conditioner)
TPH3 Lot 0301281 (Caulk/Dentsply, Milford, DE, USA)	**(Resin Composite for Both Adhesives)**

After storage in distilled water at 37°C for 24 hours, the restored teeth were sectioned facio-lingually into serial slabs approximately 0.8 mm thick using a slow-speed water-cooled diamond saw (Isomet, Buehler, Ltd). Each slab was then sectioned perpendicular to the flat-ground enamel or dentin into resin composite and tooth structure beams approximately 0.8 x 0.8 mm in cross section, according to the "non-trimming" version of the microtensile test.[16] Each tooth yielded 10 to 12 beams. After an additional 24 hours of storage in distilled water at 37°C, seven beams per tooth were selected at random and the dimensions of each beam were measured with a digital caliper (Absolute Digimatic Model CD 6" CS, Mitutoyo Corp, Kanagawa, Japan) accurate to ± 5 µm. The beams were then affixed to a Ciucchi device (Kuraray Co, Ltd, Osaka, Japan), using cyanoacrylate glue (Zapit, Dental Ventures of America, Corona, CA, USA) and tested to failure under tension in a universal testing machine (Vitrodyne V1000, Chatillon, Largo, FL, USA) at a crosshead speed of 0.6 mm/minute. The type of failure was observed at 2.5x magnification and was categorized as adhesive, cohesive or mixed.

Table 2: *Mean Fuji Bond LC/Dentin Microtensile Bond Strengths*

MPa (SD)			
Substrate	**n**	**Bond Strengths**	
Young Teeth	5	22.3 (6.2)	a
Aged Teeth	5	25.6 (7.8)	a

Groups with same letters not significantly different, two-sample t-*test,* p=*0.48.*

Table 3: *Mean Prime & Bond NT/Dentin Microtensile Bond Strengths*

MPa (SD)			
Substrate	**n**	**Bond Strengths**	
Young Teeth	5	55.4 (7.0)	a
Aged Teeth	5	60.2 (4.5)	a

Groups with same letters not significantly different, two-sample t-*test,* p=*0.24.*

Table 4: *Mean Prime & Bond NT/Enamel Microtensile Bond Strengths*

MPa (SD)						
Substrate	**n**	**Bond Strengths**		**failure type (n=35 beams)**		
				a	**m-e**	**c-d**
Young Teeth	5	149.6 (5.9)	a	13	0	22
Aged Teeth	5	47.3 (3.6)	a	14	0	21

Groups with same letters not significantly different, two-sample t-*test,* p=*0.24.*

In addition to the microtensile testing, dentin from a young and from an aged tooth were etched for 15 seconds with the same phosphoric acid etchant used for the microtensile specimens. This dentin was then dehydrated in alcohol and sputter-coated. The surfaces were examined in a SEM (Model XL30 FEG, Philips Electronics, Eindhoven, The Netherlands), operated at 5kV.

Because multiple beams derived from the same tooth may not be independent samples, bond strengths were calculated for each beam, then the mean of the beams for each tooth were used as the five samples per group. Data for each of the three adhesive/substrate combinations were analyzed statistically using a two-sample *t*-test, at a significance level of 5% to compare young versus aged teeth. Since there were only two groups in each comparison and since the number of beams was the same for each tooth, this approach was equivalent to exact statistical methods for comparing two groups that incorporate the intra-class correlation among the beams within each age group.[17] At a significance level of 5%, the sample size of five teeth per group of this study, which was dictated by a lack of sufficiently aged teeth, provided 80% power for detecting a 20% difference in mean bond strength, assuming a coefficient of variation of no more than 10%.

RESULTS

Data for each subgroup were found to be normally distributed. No significant differences were found between bond strengths to dentin from young versus aged teeth for either the resin (p=0.24) or the glass ionomer (p=0.48) adhesive, and none were found between enamel bond strengths for the resin adhesive (p=0.49). There were no pretest failures in any group. Failures between both adhesives and dentin were entirely adhesive, while failures along the dentino-enamel junction occurred in approximately 60% of the specimens in both groups of enamel bond strength specimens. Complete results are presented in Tables 2 through 4.

Dentin derived from teeth of aged subjects was uniformly darker in color and more translucent than that derived from young teeth. The SEM analysis of etched dentin from the aged teeth showed tubule patency and density very similar to young dentin. Etching topography was largely the same as for young dentin, except for the presence of some calcified plate-like structures, which could not be identified (Figure 1).

DISCUSSION

The authors regret that more aged teeth were not available to provide an increased sample size for this study, but they felt fortunate to locate 15 intact molars from older subjects over the course of a year. This gradual collection of teeth over a one-year period produced a range of storage times within the subject teeth,

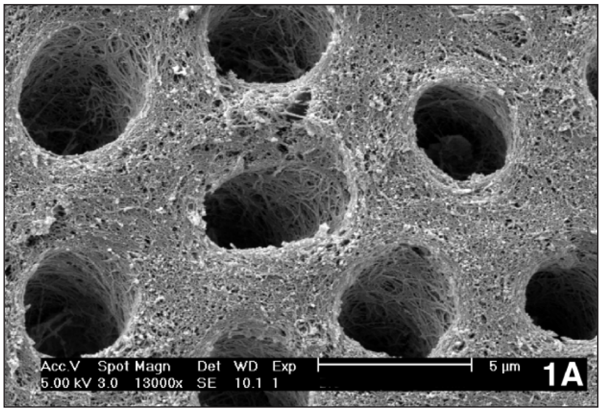

Figure 1A. *Etching pattern for young dentin, with decalcified collagen matrix evident between and within tubules. Collagen mesh of intertubular etched dentin is partially obscured by residual silica viscosity modifiers from the etchant used. (Original magnification = 13000x).*

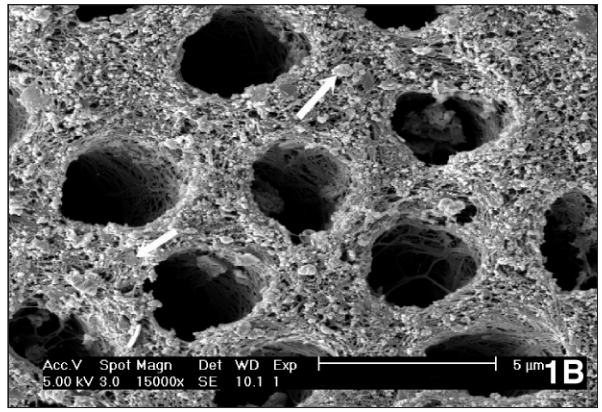

Figure 1B. *Etching pattern for older dentin showing decalcification pattern similar to young dentin, except for the presence of plate-like calcified structures of intertubular dentin (arrows). (Original magnification = 15000x).*

although this was the same in both the experimental and control groups. Since obtaining a larger sample of aged teeth would further lengthen the storage intervals, the authors determined that a sufficient number of teeth were available for a small study and proceeded.

Although many previous studies of this type have been based on the assumption that microtensile specimens are independent, that approach does not take into account the possible correlation among the samples within each tooth (the intra-class correlation), which can result in variance estimates that are too small, thereby inflating the probability of falsely rejecting the null hypothesis that there was no difference between comparison groups. The statistical approach used in this article, while appearing to be simplistic, is mathematically equivalent to exact methods that incorporate the intra-class correlation directly into the comparison of two groups, as long as there are equal numbers of specimens with complete data for each tooth in each group.[17]

The relatively high incidence of failures through tooth structure in the enamel bond strength specimens observed in this study for both ages of teeth is probably attributable to the long interval between extraction and testing, an effect observed previously by the authors with teeth that have been stored for longer intervals. The dentin evaluated for bond strength in this study was relatively superficial and would be expected to produce thin resin/dentin hybrid layers, according to previous work by Prati and others,[12] especially in older teeth. The observation by those authors, that thinner hybrid layers do not necessarily translate into reduced bond strength of resin to dentin, and also the finding of no difference in bond strengths between young and aged dentin by Tagami and others,[13] appears to be supported by this study.

Although it is well known that the permeability of aged dentin is much lower than that of young dentin,[18] microscopic examination did not show occluded tubules in the level of dentin evaluated in this study. It also seemed to confirm a slightly thicker collar of calcification of intertubular dentin of older teeth, although the overall appearance of the etched dentin was relatively similar in both young and older teeth. A recent study has reported that total-etch adhesive systems bonded to hydrated dentin exhibit large reductions in polymerization contraction stresses due to water movement from dentin into the bonded interface.[19] Although this study seems to support the fact that this also occurs in older dentin, it should be confirmed in future studies.

The authors would not expect a different result had this study been conducted with an alcohol-based resin adhesive, rather than the acetone-based adhesive chosen, as acetone-based adhesives are probably more technique sensitive in terms of requiring a hydrated etched dentin surface for adequate infiltration and formation of hybrid layers. Also, the authors do not agree with the manufacturer's instructions for use of Fuji Bond LC as the adhesive for resin composite restorative materials along enamel margins. Although effective in the previously cited clinical trials of Class V restorations, the use of a glass ionomer as an enamel adhesive prevents the formation of a very strong micromechanical bond of resin to etched enamel. It seems preferable to finish away any glass ionomer material from enamel margins and to proceed with conventional enamel etching with phosphoric acid, especially in restorations that will receive significant occlusal stresses. For this reason, along with the lack of available teeth, the bond strength of the glass ionomer adhesive to enamel was not tested in this study.

Many clinicians have based their choice of glass ionomer adhesives or restorative materials for older patients on the expectation that the increased calcification of dentinal tubules would be less disruptive to the adhesion of these materials than to resin-based adhesives. While this is likely true for sclerotic root surface dentin, it may not be true on all dentin in aged teeth, if the results of this study are representative. The results of this study also raise questions about the validity of equating aged dentin with the sclerotic dentin of non-carious cervical lesions in terms of affinity for adhesive resins.

Despite the lack of statistical power, given the small differences in the observed means within this study, the authors are confident that the findings of no significant differences are clinically relevant for the conditions evaluated in this study.

CONCLUSIONS

Within the limitations of this study, no significant differences in the adhesion of resin to enamel or dentin, or the adhesion of glass ionomer to dentin, were found for aged versus young teeth.

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For further information, contact Dr Mark Belcher, Chair, Search Committee, at mbelche@siue.edu.

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ERRATUM

In *Operative Dentistry* **33(3)**, the study "The Effect of Subject Age on the Microtensile Bond Strengths of a Resin and a Resin-modified Glass Ionomer Adhesive to Tooth Structure," pgs 282-286, has a typographical error in Table 4, which appears on page 284. The microtensile bond strength of Prime & Bond NT to enamel in young teeth should be listed as 49.5 MPa instead of 149.5 MPa.