Bond strength of younger and older permanent teeth with various etching times

Der Horng. Sheen BDS; Wei Nan. Wang BDS; and Tien Hsiang. Tarng BDS, MS

An acid etching technique is generally used to increase the retention of acrylic restorative material[1-7] and direct bonded orthodontic brackets.[8-12] Silverstone[13] found that phosphoric acid solution used for etching in concentrations between 20% and 50%, applied to the enamel for 60 seconds, created the most retentive condition. Most manufacturers of orthodontic composite resin suggest an etching time of 60 seconds. Hence, one minute conditioning has become standard practice in the direct bonding of orthodontic brackets. Several recent reports[14-22] have indicated that a traditional acid etching time of 60 seconds could be reduced to 15 or even 5 seconds without altering the enamel surface or decreasing shear bond strength.

Younger permanent teeth have a prismless layer in the enamel surface.[23-26] Older permanent teeth may have a different morphology and composition[27-30] which may influence the results of acid etching or bonding of resin. The purposes of the present investigation were to evaluate the bond strength of younger and older permanent teeth etched with 37% phosphoric acid solution for 15 and 60 seconds and analyze the location of fracture at the time of debonding.

Materials and methods

Forty premolars were extracted for orthodontic treatment or severe periodontal disease. Twenty of the teeth were from patients ages 9 to 16 years old and were categorized as younger permanent teeth. The remaining 20 were from patients 48 to 69 years of age and were categorized as older permanent teeth (these had cervical abrasion, calculus deposition, tobacco stain, occlusal or contact facet formation). The teeth were intact without enamel cracks by forceps, free of caries, and had

Abstract

The purpose of this study was to analyze the tensile bond strength and debonding failure mode in younger and older permanent teeth after etching for 15 and 60 seconds. Bond strength did not change significantly when etching time was increased from 15 to 60 seconds in younger or older permanent teeth. However, regardless of etching time, the bond strength of the older permanent teeth was greater than that of the younger teeth with statistically significant differences. Four types of debonding interface were found; bracket base-resin, within the resin, resin-enamel and enamel detachment. There were no statistically significant differences noted among younger and older permanent teeth with 15 or 60 second etching fracture mode incidence at debondings. Enamel detachment was found only at etching times of 60 seconds. To reduce enamel destruction and save chair time, 15 second etching on either younger or older permanent teeth is suggested.

Key Words

Bond strength • Failure mode • Younger permanent teeth • Older permanent teeth

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Table 1
Bond strength of older and younger permanent teeth

	Etching time	Mean (kg/mm²)	S.D. (kg/mm²)
Younger permanent teeth	15 sec	0.71	0.17
	60 sec	0.68	0.21
Older permanent teeth	15 sec	0.82	0.10
	60 sec	0.82	0.14

Sample size in each group = 10

$F_{(1,38)} = 6.41$, $p=0.0156<0.05$

Scheffe's test, p=0.05. Bond strength of older permanent teeth with 15 sec or 60 sec etching was stronger than that of younger permanent teeth with 15 or 60 sec etching.

not been treated with fluoride, alcohol, H_2O_2, or formalin etc. The teeth were extracted and washed with water then kept in a sealed container with a normal saline solution for 1-3 months until testing.

The younger and older groups were randomly subdivided into two groups each, resulting in four groups of 10 teeth. The buccal surface of each crown was polished with a fine pumice powder-water paste (Moyco Industries Inc. Philadelphia, Penn) which contained no fluoride or oil for 10 seconds, then washed with a water spray and air dried. The buccal surfaces of all the teeth in one younger and one older group were etched with 37% phosphoric acid for 15 seconds; teeth in the two other groups were etched for 60 seconds. The surfaces were washed with an abundant water spray and dried with an air spray. The crowns appeared chalky white in color.

Forty premolar brackets (Dyna-lok, batch no. 018-503, Unitek Corp., Monrovia, Calif) were selected. The bracket base consisted of a mesh-shaped arc with a surface area about 3.1 x 3.4 mm (10.54 mm²) that easily fit the curvature of the tooth's buccal surface. The contour of the premolar bracket base was demarcated in pencil on the etched enamel surface and area outside the pencil contour was coated with red nail polish in order to standardize the bonding size. The etched enamel and the bracket base were coated with a sealant (Concise, 3M Corp., St. Paul, Minn), and the composite resin was thoroughly mixed and immediately applied to the bracket base. The bracket was then accurately pressed onto the demarcated etched buccal enamel with a placement scaler. Once the bracket was in the correct position, the scaler was removed. The excess resin was carefully removed from the margin of the bracket base with a dental probe until the resin was set.

All specimens were placed in a 37°C water bath for 24 hours. Then the tensile bond strength was tested with an Instron machine (Instron Corp., Model 1000, Boston, Mass) at 2 mm/min in crosshead speed. The failure mode percentages of enamel surfaces and bracket bases in each respective group were examined and calculated with a scanning electron microscope (Canscan Corp., Serial 4, Cambridge, England) and energy dispersive x-ray spectrometry (Philips Corporation, EDAX, SW 9100, Hillegon, Holland). The bond strength and debonding interface distribution data were recorded and analyzed. The mean and standard deviations were determined. Data were analyzed by one-way and dependent two-way analyses of variance at 95% level of confidence.[31] Details of the procedures were described in previous studies.[21,32]

Results

The mean bond strengths of younger permanent teeth with 15- and 60- second acid etching times were 0.71 kg/mm² and 0.68 kg/mm² respectively. The mean bond strengths of older permanent teeth with 15- and 60-second etching times were the same at 0.82 kg/mm². The statistical analysis with one-way ANOVA showed the F value was 6.41, and was statistically different ($p=0.0156<0.05$). Scheffe's test was chosen for further analysis and comparison and showed a statistically significant difference ($p=0.05$). The older permanent teeth had a stronger bond strength than did the younger permanent teeth with either 15- or 60-second etching. However, there was no statistical difference between older and younger groups, as shown in Table 1.

The four types of failure mode found are shown in Table 2. These were: 1) cohesive failure between resin and bracket base; 2) failure within the resin itself; 3) failure between resin and enamel; and 4) enamel detachment. A comparison of the percentage of failure mode among the various groups by two-way ANOVA is shown in Table 3.

The statistical results showed the F value of comparison between the four groups and the failure mode interaction was 0.34, and was not statistically different ($p=0.9598>0.05$). The F value of comparison of the four groups was 0 and was not statistically different ($p=1.00>0.05$). The F value of comparison of failure mode was 145.1 and was

Table 2
Failure modes (%)

Etch time	Metal-Resin Mean	S.D.	Range	C.V.	Within Resin Mean	S.D.	Range	C.V.	Resin-Enamel Mean	S.D.	Range	C.V.	Enamel Detachment Mean	S.D.	Range	C.V.
Younger perm. teeth 15"	43.0	5.7	32-50	13.2	22.0	8.0	10-30	36.6	35.0	8.5	20-45	24.2	-	-	-	-
60"	45.0	10.0	30-65	22.2	23.0	10.6	10-45	46.1	30.0	11.3	15-40	37.7	2.0	4.2	0-10	210.8
Older perm. teeth 15"	44.0	10.3	30-60	23.5	24.0	12.0	10-50	49.8	32.0	12.0	10-55	37.7	-	-	-	-
60"	45.0	13.5	25-60	30.1	20.0	7.5	10-45	37.3	33.0	17.5	10-60	53.1	2.0	4.2	0-10	210.8

S.D.= Standard Deviation
C.V.= Coefficient Variation

statistically different ($p=0.0001<0.05$), shown in Table 3.

Sheffe's test was chosen for the post hoc treatment of broken mode. The α value with 0.05 was chosen, and the rank of broken mode was metal-resin > enamel-resin > within resin > enamel detachment (Table 4) The major failure modes (65-68%) were associated with the metal and resin, and included the metal-resin interface and within the resin itself. Enamel detachment was found when etching time was 60 seconds. (2%)

Table 3
Analysis of percentages of failure mode with two-way ANOVA

Source	Sum of squares	DF	Mean square	F value	PR>F
Group of etched teeth	0	3		0	1.00
Debonding mode	40415	3		145.1	0.0001
Group*Type	285	9		0.34	0.9598
Error	13374	144	92.9		
Corrected total	54074	159			

Table 4
The post hoc treatment with Sheffe's test of four failure modes

metal-resin > resin-enamel > within resin > enamel detachment

Note: $\alpha = 0.05$

Discussion

The present study showed that the difference in bond strength between 15- and 60-second etching times in younger and older permanent teeth was not statistically significant. However, regardless of etching time, the bond strength of the older permanent teeth was stronger than that of the younger teeth, with a statistically significant difference. The results showed that orthodontic bonding with a short etching time (15 seconds) was suggested in younger or adult orthodontic patients and the older the teeth, the greater the bond strength.

For younger permanent teeth, prolonged etching time or mechanically removing the prismless layer will help to increase retention.[25,33,34] However, the bond strength of a short etching time (15 seconds) showed the same results as the longer etching time in this test. The prismless layer on the enamel surface of younger permanent teeth may not influence retention. This confirmed previous

studies[14-22] that a 15-second acid etch is enough to create sufficient retention on the enamel surface for bracket bonding in teenagers.

In older permanent teeth, there were no significant differences in bond strength between 15- and 60-second acid etching. The results were different from an earlier study[15] which indicated that the enamel surface of older teeth etched for 60 seconds had a stronger bond strength than that etched for 15 seconds. The difference may be explained by the fact that earlier researchers used surface impression to detect surface irregularity with scanning electron microscope to indicate the quality of mechanical retention. In this study, the debonding technique was used which detected the bond strength directly.

Bond strength in the older permanent teeth was greater than in the younger teeth, regardless of etching time. Bhaskar[29] found that the enamel surfaces of recently erupted teeth are completely covered with pronounced perikymata and rod-ends. With age, the perikymata and rod-ends may wear away. Bhaskar also found localized increases of certain elements such as nitrogen and fluoride in the superficial enamel layer of older teeth. This suggested a continuous uptake, probably from the oral environment, during aging. As a result of age changes in the organic portion of enamel, presumably near the surface, teeth may become harder and their resistance to decay increased. Older teeth also become much less permeable to fluids. Thus, the enamel may become harder with age and thereby reinforce the bond strength.

There were no obvious differences in the percentages of failure mode distribution between brackets and teeth among the four groups. Enamel detachments were found only in the 60-second acid etching groups which indicated that prolonged etching time (60 seconds) may result in enamel surface destruction but does not increase bond strength. For saving time in clinical practice, and decreasing enamel destruction, it seems that 15 seconds is the optimal etching time for younger or older permanent teeth.

In their in vivo study, Årtun and Bergland[35] showed that the mode of loosening was mainly

between the bracket base and adhesive with 37% phosphoric acid etching. This failure mode may include failure at the metal-resin interface or within the resin itself. Our in vitro study showed that the failure mode which included metal-resin and within the resin was from 65% to 68% under SEM or EDAX observation and detection. Hence, both the in vivo and the in vitro studies had similar results: the major failure mode occurs between the metal and the adhesive.

Conclusions

1. A 15-second etch with 37% phosphoric acid solution is preferable to a 60-second etch for older or younger permanent teeth.
2. The bond strength of older permanent teeth was stronger than that of younger teeth, with either 15- or 60-second etching time.
3. Enamel detachment was found on teeth etched for 60 seconds.

Author Address

Der Horng. Sheen B.D.S.
Orthodontic and Pedodontic Section,
Department of Dentistry
Tri-Service General Hospital
8, Section 3, Ding-Chow Road
Taipei, Taiwan
Republic of China, 100.

D.H. Sheen is an Assistant in the School of Dentistry, National Defense Medical Center in Taipei, Taiwan.

W.N. Wang is Head of the Orthodontic and Pedodontic Section, Department of Dentistry, Tri-Service General Hospital, Taipei, Taiwan and an Associate Professor in the School of Dentistry, National Defense Medical Center.

T.H. Tarng is Chief Resident, Orthodontic and Pedodontic Section, Department of Dentistry, Tri-Service General Hospital and Instructor, School of Dentistry, National Defense Medical Center.

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