Effect of H_3PO_4 concentration on bond strength

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Most suppliers of H_3PO_4 products recommend a concentration range of 37% to 50% H_3PO_4 solution or gel for etching in operative dentistry,[1-5] preventive dentistry[6,7] and orthodontics.[8-17] However, enamel detachment or calcium loss often occurs following etching with 37% to 50% H_3PO_4.[16-26] Numerous investigations have been carried out to define the appropriate concentration of H_3PO_4 for use as an enamel etchant. Concentrations of 5% to 50% with various composites or etching times have been recommended in previous studies, with varying results of bond strength, surface roughness, debonding interface and enamel loss.[14,16,17,22-28] The results are confusing to clinicians. The aims of the present investigation were: 1. To determine the bond strength produced by various concentrations of etchant; 2. To determine the percentage of debonding interface at various concentrations of etchant; 3. To examine enamel detachment; and 4. To determine the appropriate concentration of H_3PO_4 solution for adequate bond strength and minimal enamel detachment.

Materials and methods

One hundred premolars (first or second, maxillary or mandibular) were extracted for orthodontic purposes from 9- to 16-year old patients. The crowns of the extracted teeth were intact without enamel cracks; none had

Abstract

Prior to bonding, the enamel surface of the tooth is normally etched using a solution of 37%-50% phosphoric acid (H_3PO_4) for 60 seconds. The purpose of this study was to evaluate the tensile bond strength, debonding interface distribution and enamel surface detachment of various concentrations of H_3PO_4 solution, from 2% to 80%, applied for 15 seconds. Statistically significant differences in bond strength were found among the various concentrations tested: concentrations in the 10% to 60% range produced greater bond strengths than both the weaker and stronger concentrations. The weaker the bond strength, the greater the debonding interface between resin and enamel. The greater the bond strength, the greater the debonding interface between the bracket and resin. Enamel detachment occurred as the H_3PO_4 concentration rose above 30%. To obtain greater bond strength and less enamel detachment, 10%- 30% concentrations of phosphoric acid for 15 seconds etching are suggested for clinical bonding.

Key words

Phosphoric acid • Debonding interface • Etchant concentration • Bond strength

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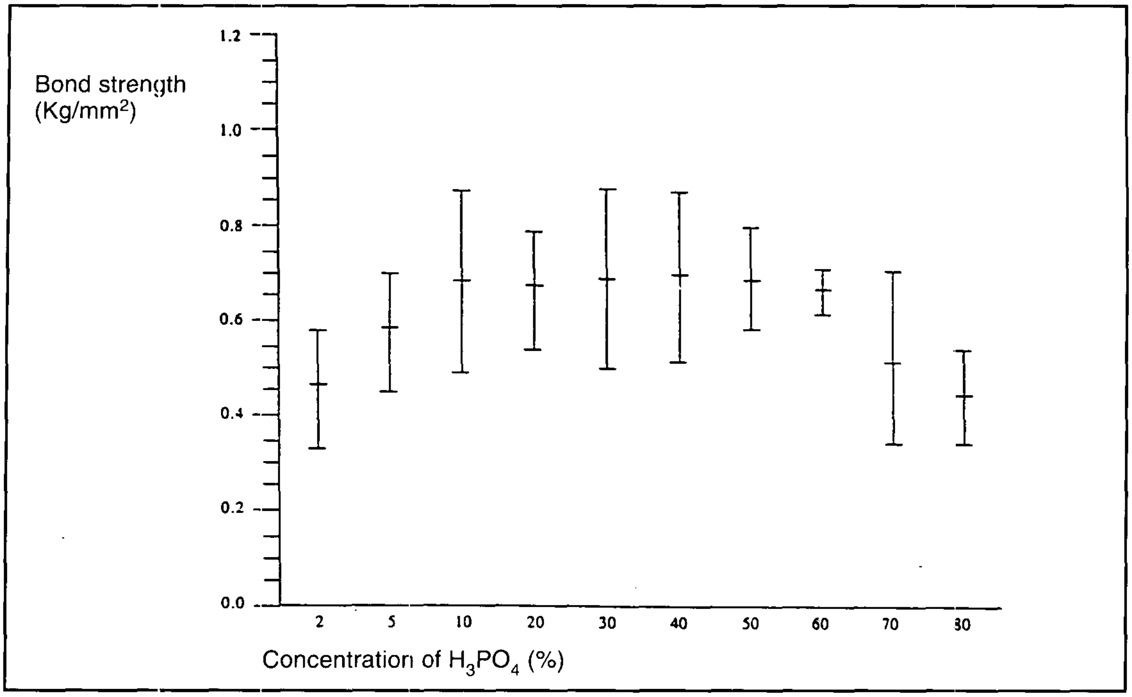
Figure 1
Mean and standard deviations of bond strength with 10 various H_3PO_4 concentrations.



caries; none had been treated with H_2O_2, formalin, alcohol or other chemical agents after extraction. The extracted teeth were washed and stored with physiologic saline in a sealed box for 1 to 3 months until testing. The teeth were randomly divided into 10 groups of 10 teeth each. The buccal surface of each crown was polished with a fine pumice powder (Moyco Industries Inc., Philadelphia, Penn) for 10 seconds, then the enamel surface sprayed with water for 10 seconds and dried with air spray. In preparation for bonding, the buccal enamel of the teeth in each group was etched with a concentration of phosphoric acid solution (Wakyo Pure Chemical Industry LTD., Tokyo, Japan), 2%, 5%, 10%, 20%, 30%, 40%, 50%, 60%, 70% or 80% by weight, for 15 seconds.[18] The surface was again sprayed with water for 10 seconds and dried with air spray.

A grooved retention base premolar bracket (Unitek Corp., Monrovia, Calif) with contoured curvature of the premolar buccal surface was chosen for bonding. In order to standardize the bonding area of the resin, the bracket base was outlined in the center of the etched surface by marking with a pencil, and the enamel outside the encircled area was coated with red nail polish before bonding. Concise (3M Corp., St. Paul, Minn) sealant and Concise composite resin were thoroughly mixed and immediately applied to the demarcated etched enamel and bracket base with a placement scaler. Excess composite resin was removed with a dental probe. The bracketed tooth was left to air-dry for 10 minutes until it was set completely. Ten group specimens were incubated in a 37°C water bath for 24 hours.

Table 1
The results of bond strength with 10 different phosphoric acid concentrations.

Phosphoric acid Concentration (% wt/wt)	Mean (Kg/mm²)	S.D. (Kg/mm²)
2%	0.46	0.12
5%	0.58	0.12
10%	0.68	0.19
20%	0.67	0.12
30%	0.69	0.09
40%	0.70	0.17
50%	0.69	0.11
60%	0.67	0.05
70%	0.53	0.18
80%	0.45	0.10

S.D.: Standard deviation
Sample Size: 10 in each group

Bond strength was measured on an Instron machine (Instron Corp., Model 1000, Boston, Mass) and debonding interfaces were examined using scanning electron microscopy and mapping of energy dispersive x-ray spectrometry (Phillips Corp., E.D.A.X. SW 9100,

Table 2
The mean and standard deviation of percentages of debonding interface distribution, the significance of simple main effect and the post hoc treatment with Scheffe's test of various debonding interfaces of 10 various H_3PO_4 concentrations.

	A		B		C		D		P	S
	Mean	SD	Mean	SD	Mean	SD	Mean	SD		
2%	4.6	3.44	9.0	6.34	86.2	6.3	-		0.0001	C>A,B
5%	16.5	16.5	30.5	13.6	53.0	25.4	-		0.0042	C>A,B
10%	28.5	15.8	35.0	10.3	36.5	19.6	-		0.490	
20%	37.5	8.25	36.5	5.80	26.0	12.0	-		0.390	
30%	43.5	5.80	39.5	8.31	17.0	10.1	-		0.0001	A,B>C
40%	46.0	4.60	35.5	6.85	17.5	7.55	1.00	2.11	0.0001	A>B>C,D
50%	39.5	9.56	35.0	7.82	21.8	12.4	3.70	4.85	0.0001	A,B>C,D
60%	29.5	14.2	33.0	18.1	32.7	8.41	4.80	5.09	0.014	B,C>A>D
70%	18.0	12.5	30.0	11.1	41.2	7.22	10.8	3.29	0.0001	B,C>A,D
80%	16.5	10.3	27.5	7.55	44.8	8.65	11.2	2.57	0.0001	C>B>A,D

A: Interface between bracket and resin
B: Interface within the resin itself
C: Interface between resin and enamel
D: Tooth fragment
P: Significance of simple main effect
S: Scheffe's test (α=0.05)
S.D.: Standard deviation
Sample Size: 10 in each group
- : No finding

Hillegon, Holland). The distributive percentages of debonding interface were calculated. Data on bond strengths and debonding interface percentages were recorded and means and standard deviations were determined with SAS software (SAS Institute Inc., Cary, NC) by one-way and two-way ANOVA. Scheffe's test was then used to identify statistically significant differences between the level of the bond strength and debonding interface under investigation.[29] Detailed procedures of bond strength and debonding interface analyses have been described in a previous study.[18]

Results

Tensile bond strength: The mean bond strengths were 0.46, 0.58, 0.68, 0.67, 0.69, 0.70, 0.69, 0.67, 0.53 and 0.45Kg/mm^2 for H_3PO_4 concentrations of 2%, 5%, 10%, 20%, 30%, 40%, 50%, 60%, 70% and 80%, respectively, shown in Figure 1 and Table 1. The statistical analysis of bond strength with one-way ANOVA gave an F value of 3.9, i.e., a statistically significant difference (P=0.0003). The least significant difference was chosen (α= 0.05) for further analysis and comparison. It was a statistically significant difference (P=0.01). The results of the least significant difference test showed that the bond strengths could be divided into three groups. The strongest bond strengths were found from 10% to 60% H_3PO_4, the second strongest bond strengths were 5% and 70% H_3PO_4, and the weakest bond strengths were in the 2% and 80% H_3PO_4 groups.

Table 3
The statistical results of 10 various H_3PO_4 concentrations and percentages of debonding interfaces with two-way ANOVA.

	F Value	PR > F
Group of various concentrations of H_3PO_4	1.19	0.304
Type of debonding interface	44.40	0.000
Group and type interaction	21.00	0.000

P<0.05

Debonding interface: The debonding interfaces between the bracket base and tooth surface were located as follows: (1) between bracket base and resin, (2) within the resin itself, (3) between resin and enamel. In some cases, enamel detachment occurred. The percentages of mean and standard deviation of debonding interface distribution in each concentration of H_3PO_4 are shown in Table 2. No enamel detachment was found with concentrations of H_3PO_4 below 30%. A comparison of the percentages of debonding interface distribution was performed among 10 various concentrations of H_3PO_4 by two-way ANOVA. The statistical results are shown in Table 3. The F value of comparison between 10 various concentrations of H_3PO_4 and percentages of debonding interface distribution interaction was 21.0, which was statistically significant (P=0.000). The F value of comparison among 10 various concentrations of H_3PO_4 was 1.19, which was not statistically significant (P=0.304). The F value of comparison among four types of the percentage of debonding interface distribution was 44.4, and was statistically significant (P=0.000). The significances of simple main effect of debonding interface of the 10 concentrations of H_3PO_4 were 0.0001, 0.0042, 0.490, 0.390, 0.0001, 0.0001, 0.0001, 0.014, 0.0001 and 0.0001 respectively. Debonding interfaces of the concentrations of 2%, 5%, 30%, 40%, 50%, 60%, 70%, and 80% H_3PO_4 were statistically significant (P< 0.01). The Scheffe's test was chosen for the post hoc treatment, the α value with 0.05 was chosen and the ranks of various debonding interfaces of each concentration are shown in Table 2. The major debonding interfaces of 60% and 70% H_3PO_4 concentrations were between resin and bracket. At 30% to 50% H_3PO_4 concentrations, the major debonding interfaces were between bracket and resin or within the resin itself. However, the major debonding interfaces of the concentrations of 2%, 5% and 80% H_3PO_4 were between resin and enamel.

Discussion

Etching enamel with H_3PO_4 results in the loss of superficial enamel and preferential dissolution of the underlying enamel.[24] The depth of etch (subsurface) or the amount of superficial enamel removed during etching depends on the concentration of H_3PO_4 and the duration of its application.[24] From a biological viewpoint, a low concentration is preferable because it causes minimal enamel detachment or calcium loss[20-27] while securing an adequate bond.

The results of this study indicate that the strongest bond strengths resulted from concentrations of H_3PO_4 in the range of 10% to 60%. However, enamel detachment did occur as the concentration increased above 30%. The suggested application of 37%-50% H_3PO_4 for 60 seconds may damage the enamel. The lowest concentrations of H_3PO_4 tested (2% and 5%) resulted in weaker bond strengths. Ten percent to 30% concentrations of H_3PO_4 applied for 15 seconds should result in less enamel detachment or calcium loss while producing adequate bond strength for clinical usage.

The major debonding interface distributions of lower concentrations of H_3PO_4 (2% and 5%) were between the resin and enamel (53.0%-86.2%). There was no enamel detachment, as shown in Table 2. The reason for this may be related to insufficient etching time on the enamel surface to achieve retention.[23-25]

The higher concentrations of H_3PO_4 (70% and 80%) also resulted in weaker bond strengths. The major debonding interface distributions were between enamel and resin (41.2% to 44.8%) and enamel detachments were found (10.8% to 11.2%) as shown in Table 2. The enamel surface structure may have been destroyed by overetching with greater concentrations of H_3PO_4 causing the retention to fail.

The total amount of calcium dissolved increased with an increase in H_3PO_4 concentration with 60 seconds etching and reached a maximum with 20% to 50% H_3PO_4, and a further increase in acid concentration resulted in a decrease in the total calcium dissolved.[24] The amounts of subsurface calcium dissolved or depths of etch with phosphoric acid concentrations ranging from 10% to 60% for 60 seconds etching did not differ significantly. Further increase in acid concentration resulted in a decrease in the depth of etch or total calcium dissolved.[17,23] Bond strengths with H_3PO_4 concentrations of 10% to 60% were not significantly different, and decreased with higher concentrations.[17,23,24] The findings of the present study are in agreement with those earlier reports. However, even with the use of a 15-second etch, enamel detachment was found as the concentration of H_3PO_4 increased above 30%; the greater the concentration of H_3PO_4 over 30%, the greater the enamel detachment.

In their in vivo study, Sadowsky et al.[28] found that reducing the etching concentration from 37% to 15% of H_3PO_4 for 60 seconds etching did not significantly affect the retention of bonded orthodontic attachments. Hence, they suggested that the reduction of etchant concentration and duration of etching should be considered. The results of their in vivo study correspond with those of our in vitro study. Zidan and Hill[24] tested bond strengths with 0.5%, 2%, 5% and 35% H_3PO_4 and found that they were not significantly different except with 0.5% H_3PO_4. Their sample size was small (five specimens in each concentration group) and they may have used a different method of bond strength testing.

Retief,[17] Bryant et al.[22] as well as Zidan and Hill,[24] analyzed the debonding interface with scanning electron microscopy, an excellent tool for examining tooth surfaces qualitatively but not quantitatively.[30] In our serial studies, a scanning electron microscope with energy dispersive x-ray spectrometry was used to analyze the debonding interface qualitatively as well as quantitatively.

Concentrations of H_3PO_4 greater than 27% produced monocalcium phosphate monohydrate as the main reactant, which was readily soluble and would be completely washed away after etching. However, less than 27% H_3PO_4 produced dicalcium phosphate dihydrate, which was less soluble. The products, if not completely removed after the etching procedure, may interfere with the bonding of composite resin to the etched enamel surface.[27] Hence, in this test, the surface after etching was carefully washed with water spray for 10 seconds before bonding.

There was no statistical difference in bond strength between 15 and 60 seconds of H_3PO_4 etching. However, there was no enamel detachment as a 15-second etch was used.[18] Hence, to minimize enamel detachment or calcium loss, a 15-second etch was chosen in this test. This is different from previous studies which used etching times over 15 seconds.[21-25]

From the statistical analysis of debonding interfaces of all specimens, the greater the bond strength, the greater the debonding interface between the bracket and resin. Choosing the appropriate bracket base with greater bond strength will be analyzed in future studies.

Conclusions

1. Ten percent to 30% concentrations of H_3PO_4 solution applied for 15 seconds etching resulted in bond strength adequate for clinical bonding with minimal enamel detachment.

2. Lower (2% to 5%) and higher (70% to 80%) concentrations of H_3PO_4 solution resulted in lower bond strengths with debonding interfaces between the resin and enamel. The other concentrations tested—10% to 50%—produced greater bond strengths, with debonding interfaces located between the bracket and resin.

3. Enamel detachment was found as the concentrations of H_3PO_4 solution increased above 30%.

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