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ORIGINAL RESEARCH

Comparative Evaluation of Self-etching Primers and Phosphoric Acid Effectiveness on Composite to Enamel Bond: An *in vitro* Study

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ABSTRACT

Aim: The aim of the present study was to investigate the effectiveness of the one total-etch self-priming adhesive, one two-step self-etching primer adhesive, and one 'all-in-one' self-etching adhesive system on the adhesion of a resin composite to enamel.

Materials and methods: Thirty-six freshly extracted human mandibular molars were selected for this study. A flat area about 5 mm in diameter was created on the exposed mesial surface of enamel of each tooth by moist grinding with 320, 420 and 600 grit silicon carbide paper. Twelve teeth were randomly assigned into three groups. In group 1, Adper Easy One (3M ESPE), a one step self-etching primer adhesive was applied and light curing unit for 10 seconds. In group 2, Adper SE Plus, a two-step self-etching primer with bottle A containing the aqueous primer and bottle B containing the acidic adhesive was applied and light cured for 10 seconds. Group 3 (control)—etchant 37% phosphoric acid is applied to the surface for 15 seconds and rinsed with water and air dried and adhesive (single bond 2) is applied to the surface and tube is placed and light cured for 20 seconds. Composite material (Z350) was placed in the tube and light cured for 40 seconds in all the groups.

Bond strength testing was done using universal testing machine at the enamel-composite interface. The debonded enamel surface was evaluated in stereomicroscope to assess the cohesive, adhesive or mixed fracture. Data was statistically analyzed by one way analysis of variance (ANOVA).

Results: Group 1 performed least among all groups with a mean score of 19.46 MPa. Group 2 had a mean score of 25.67 MPa. Group 3 had a mean score of 27.16 MPa.

Conclusion: Under the conditions of this *in vitro* study, the bond strength values of the two-step self-etching primer systems tested were similar to the total-etch. And, one step self-etching primers have lower bond strength compared to the total-etch.

Keywords: Self-etching primers, Shear bond strength, Fracture.

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INTRODUCTION

Restoration means to restore an object back to its normal. The purpose of restorative dentistry is to restore a damaged tooth back to its normal form, function and esthetics and to enhance the general health of the patient. In recent times, esthetics is gaining prime importance while planning a dental restoration.[1]

Buonocore (1955) has been called father of adhesive dentistry, as he has pioneered the art of acid etching the enamel with phosphoric acid causing preferential dissolution of interprismatic enamel, allowing micromechanical retention of adhesive resins. This bond strength depends on the resin tags, and technique is called total-etch technique.[2]

Total-etch technique is still the most effective approach to achieving efficient and stable bonding to enamel. Selective dissolution of hydroxyapatite crystals through etching is allowed by *in situ* polymerization of resin that is reabsorbed by capillary attraction within the created etch-pits. Therefore, enveloping individually exposed hydroxyapatite crystals. Two types of resin tags interlock within the etch-pits 'macro'-tags filling space surrounding the enamel prisms. While numerous 'micro'-tags result from resin infiltration/polymerization within the tiny etch-pits at the cores of the etched enamel prisms. The latter are especially thought to contribute the most with regard to retention to enamel.[1,2]

The recent advances in the adhesive restoration and the resin materials are aimed at further simplifying the restorative procedures with better physical properties. Advances in adhesion have given rise to scope for research into the bonding, bond strength, durability and microleakage.[3]

Various new bonding systems are in the market. Two new systems have been developed over the conventional etching and bonding technique (total-etch technique). They are: (1) two-step technique in which etchant and primer in one bottle and bonding agent in another bottle. (2) One step technique in that etchant, primer and bonding agent all is in one bottle, both these systems are called as self-etching primers.[2]

These self-etching primers do not require a separate acid conditioning step. They eliminate the factors, like overetching, overdrying, and overwetting.[4] They are composed of aqueous mixtures of acidic functional monomers, generally phosphoric acid esters, with a PH relatively higher than that of phosphoric acid etchants. Self-etching primers are less technique sensitive than total-etch technique.[5]

Adper Easy One (one bottle system) and Adper SE Plus (two-bottle system) are two recently introduced self-etching primer systems in which as per the available indexed literature the bond strength to enamel has not been established.

Hence, in this study, the bond strength of Adper Easy One and Adper SE Plus to ground enamel has been compared with conventional total-etch technique [i.e. 37% phosphoric acid used as etchant and single bond two (3M ESPE) used as adhesive].

The aim of the present study was to investigate the effectiveness of the one total-etch self-priming adhesive, one two-step self-etching primer adhesive, and one 'all-in-one' self-etching adhesive system on the adhesion of a resin composite to enamel.

OBJECTIVES

The objective of this study is to investigate the effectiveness of one step self-etching primers, two-step etching primers and conventional phosphoric acid etching and bonding on the adhesion of composite to enamel by measuring the shear bond strength.

MATERIALS AND METHODS

Thirty-six freshly extracted human mandibular molars were selected for this study (Fig. 1). Teeth with pre-existing caries, restoration, fractures or cracks were eliminated. Teeth were cleaned and stored frozen until testing. Before procedure, teeth were cleaned with water, autoclaved and maintained in saline (0.9% w/v) solution.

A flat area about 5 mm in diameter was created on the exposed mesial surface of enamel of each tooth by moist grinding with 320, 420 and 600 grit silicon carbide paper in the order (Fig. 2). Twelve teeth were randomly assigned into three groups.

Fig. 1: Mounted samples in acrylic molds

Fig. 2: Preparation of samples using silicon disks

In group 1 (experimental): Adper Easy one (3M ESPE) is a one step self-etching primer. The adhesive was applied with the disposable applicator for 20 seconds to the mesial enamel surfaces of the teeth and, subsequently, air thinned for approximately 5 seconds until the film no longer moves, indicating complete vaporization of the solvent. Silicon tube (3×2 mm^2) was attached to the surface and the adhesive cured with a light curing unit for 10 seconds and the tube was filled with the composite material (Z350) and light cured for 40 seconds and the tube is removed.

In group 2 (experimental): Adper SE Plus a two-step self-etching primer with bottle A containing the aqueous primer and bottle B containing the acidic adhesive was applied and lightly dried. Thin adhesive layer was air dried to adjust film thickness and place the tube to the surface and light cured for 10 seconds. The tube is filled with the composite material and light cured for 40 seconds and removed.

Group 3 (control): Etchant 37% phosphoric acid is applied to the surface for 15 seconds and rinsed with water and air dried and adhesive (single bond 2) is applied to the surface, and tube is placed and light cured for 20 seconds. Composite

material is placed in the tube, and light cured for 40 seconds and tube is removed (Fig. 3).

All teeth were stored for 24 hours in tap water at 37°C. Bond strength testing was done using a blunt edge shearing chisel in a universal testing machine (Instron model 5500) with a crosshead speed of 1 mm/min with the shearing load at the enamel-composite interface (Fig. 4). Shear bond strength (MPa) was calculated by dividing the failure load (Newton) by cross-sectional area of the bonded composite.

The debonded enamel surface was evaluated in stereomicroscope to assess the composite-enamel pattern, i.e. cohesive, adhesive or mixed fracture (Figs 5 and 6). Data was statistically analyzed by-one way analysis of variance (ANOVA).

RESULTS

The present *in vitro* study was conducted to investigate the effectiveness of one step self-etching primers, two-step self-etching primers, conventional phosphoric acid etching and bonding on the adhesion of composite to enamel by measuring the shear bond strength.

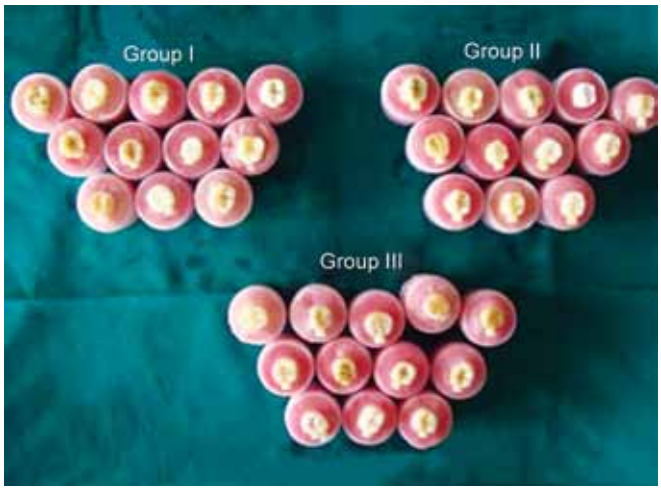

Fig. 3: Restored samples of all the 3 groups

Fig. 4: Sample loaded in Instron machine

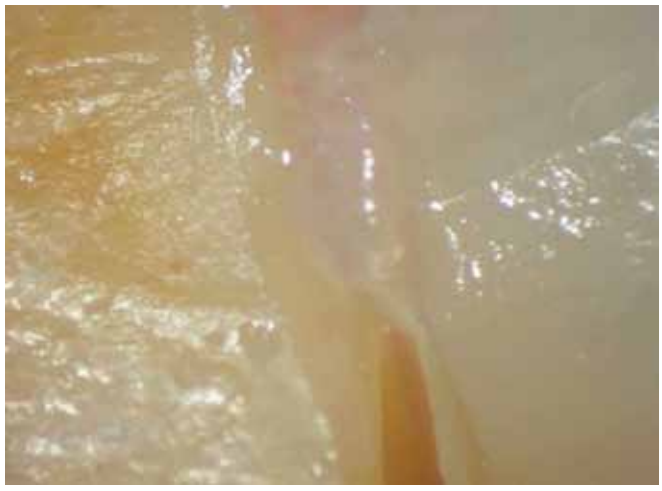

Fig. 5: Adhesive fracture

Fig. 6: Mixed fracture

Descriptive data are presented as numbers and percentages with corresponding bond strength scores for each group. The analysis was done using ANOVA test. As statistical significance was detected in all the groups, they were further analyzed among the groups using the Kruskal-Wallis test.

A p-value of 0.001 or less was considered statistically significant.

In group I, the highest bond strength was 24.673 MPa and least bond strength was 14.804 MPa. In group II, the highest bond strength was 30.610 MPa and least bond strength was 21.970 MPa and, in group III, the highest bond strength was 33.644 MPa and least bond strength was 24.419 MPa (Table 1).

Compilation of the scores of three groups with the mean values of each group with comparison of the groups were statistical significant.

- Group 3 as a mean score of 27.16 MPa.
- Group 2 as a mean score of 25.67 MPa.
- Group 1 performed least among all groups with a mean score of 19.46 MPa (Table 2).

Table 1: Bond strength scores (MPa) of the samples

Sr. no	*Group 1*	*Group 2*	*Group 3*
1.	19.902	29.223	24.419
2.	24.673	25.659	28.601
3.	21.667	23.847	25.847
4.	23.180	30.610	25.981
5.	14.804	28.685	28.462
6.	17.789	24.016	33.644
7.	19.681	23.434	26.540
8.	16.105	26.972	25.759
9.	21.082	24.707	28.326
10.	17.877	20.334	29.671
11.	20.692	24.458	27.150
12.	18.978	21.970	26.635

As the values were statistical significant (p-value = 0.000). The values of the groups were further analyzed using Kruskal-Wallis test.

Intergroup comparison was done using the Kruskal-Wallis test. Comparison between groups 1 and 2 showed statistical significant. Comparison between groups 2 and 3 showed statistical significant. Comparison between groups 1 and 3 was not statistical significant (Table 3).

Present failure mode of all the samples in individual group (Table 4).

Distribution of failure mode of the samples is recorded in percentage. In all the groups, most failure are in adhesive in nature. In group 1, 50% samples are adhesive failure, 25% samples are cohesive failure and 25% samples are mixed failure. In group II, 58.3% samples are adhesive failure, 16.7% are cohesive failure, 25.0% samples are mixed failure. In group III, 52.8% samples are adhesive failure, 19.4% samples are cohesive failure and 27.8% samples are mixed failure (Table 5).

The chi-square test was done. The results were not statistically insignificant (Table 6).

Table 3: Statistical analysis of intergroup comparison

Group comparison	*Mean difference*	*Std. error*	*Significance*
Group 1 *vs* 2	–5.625000	1.066627	0.000
Group 1 *vs* 3	7.048750	1.066627	0.000
Group 2 *vs* 3	1.423750	1.066627	0.420

Table 4: Stereomicroscopic observation of area of fracture of all the samples

Sr. no	*Group 1*	*Group 2*	*Group 3*
1.	3	2	2
2.	2	2	2
3.	2	3	3
4.	3	1	2
5.	2	2	2
6.	2	2	1
7.	2	3	3
8.	1	2	1
9.	2	1	2
10.	1	2	2
11.	3	2	3
12.	1	3	3

Note: 1—cohesive failure; 2—adhesive failure; 3—mixed failure

Table 5: Distribution of fracture pattern in all the samples

	Fracture pattern			
Groups	*1*	*2*	*3*	*Total*
Group 1	3	6	3	12
	25.0%	50.0%	25.0%	100.0%
Group 2	2	7	3	12
	16.7%	58.3%	25.0%	100.0%
Group 3	2	6	4	12
	16.7%	50.0%	33.3%	100.0%
Total	7	19	10	36
	19.4%	52.8%	27.8%	100.0%

Table 6: Statistical analysis

Chi-square value	*df*	*p-value*
0.591	4	0.964

DISCUSSION

Enamel bonding has traditionally been dependent upon the infiltration of resin into surface porosity created by acid conditioning agents. In the past, phosphoric acid has been the principal etchant used for enamel conditioning. More recently, newer systems have been introduced, which use a single treatment step to condition or etch both the enamel and dentin surfaces with agents other the phosphoric acid. These systems are generally referred to as self-etch adhesives.[6] The self-etching materials were introduced to dentistry at a time when less technique-sensitive adhesive materials were desired, although these qualities can be very appealing to the clinicians. It needs to be evaluated, as to how these new materials interact with the enamel surface, since the current self-etching materials have higher PH values.[7]

The product examined in this study offered both as enamel and dentin bonding agents; thus, it is conceivable that a combined application of self-etching primers on enamel

Table 2: Comparison of mean bond strengths (MPa) value in all the groups using ANOVA (mean average bond strength)

	N	*Mean bond strength*	*Std. deviation*	*Minimum*	*Maximum*	*F-value*	*p-value*
Group 1	12	19.70250	2.829016	14.804	24.673	24.422	0.00
Group 2	12	25.32750	3.047056	20.334	30.610		
Group 3	12	26.75125	1.786226	23.644	29.671		

and dentin surfaces could take place without separate or selective acid etching.

For this study, the selected pair of adhesives and resin composites, from the same manufacturer for each group of specimens, is used. This is because this would be the most likely way that resins would be purchased by the dentists.

This study has compared the enamel bond strength and nature of bond failure involving self-etching primers and total-etch adhesive systems.

It has been postulated that minimum bond strength of 17 to 20 MPa to enamel and dentin is needed to resist contraction forces of resin composite materials. Etching of enamel with 30 to 40% phosphoric acid gives bond strength of about 20 MPa and clinical experiences also confirm that this bond strength is sufficient for successful retention of resin restorations. All the adhesive systems used in this study achieved the optimal bond strength values to the enamel.[4] However, in this study, one step self-etching primers (group 1) achieved lower bond strength when compared with two-step self-etching primers and total-etch adhesive system. The results were statistically significant. Two-step self-etching primers (group 2) and total-etch adhesives (group 3) have similar bond strength. The result between these two groups was statistically insignificant.

Other bond strength studies, however, reported similar bond strength to our study. In the similar studies, one step self-etch adhesives usually obtained lower bond strength than two-step self-etch and three-step total-etch adhesive systems.[8] In other studies, results have shown that one step self-etch adhesives can produce shear bond strengths to ground enamel that are even higher than those of total-etch adhesive systems.[9,10]

In this study, the self-etching systems showed lower bond strengths to enamel compared to a total-etch system. This could be due to less demineralization of enamel by the priming agent compared. Because demineralization and resin infiltration occur simultaneously when using self-etch adhesives, dissolved hydroxyapatite crystals and smear layer remnants get incorporated into the polymerized resin layer.[6,8,11]

Mild self-etch Adper SE Plus showed the highest bond strength values, while the acidic Adper Easy One showed the lowest values. This finding is consistent with previous literature which states that mild two-step self-etching primers usually performs better than acidic one step self-etching primers.[12]

Other studies reported that the etching pattern of self-etching adhesives was not as well defined as that of total-etch adhesives both on ground and ungrounded enamel. However, other studies linked the lack of a defined etching pattern with low enamel bond strengths with self-etching primers.[3]

Until recently, it has been advocated that bonding procedures on enamel provide reliable and relatively stable bonds, based mainly on the homogeneous characteristics of the substrate. However, it is important to consider that, over the last four decades, the clinical stability of bonds to enamel has been almost exclusively related to the use of hydrophobic resins.[13]

The failure mode of self-etching primers was primarily adhesive in all groups. When phosphoric acid was applied for total-etch systems, an increase in adhesive and mixed fractures occurred; this could be due to the increased bond strength of total etch systems with phosphoric acid etching.

New techniques should not be viewed as alternatives to well established restorative methods unless they offer advantages from a scientific and clinical views point.[14]

Even when the current effectiveness of total-etch dental adhesives is acknowledged, the concept of self-etching primers is promising and deserves increasing attention in the future.[10] The time saving and efficiency of the self-etching primers approach combined is certainly a significant advantage when compared to the many multiple step adhesive systems. Nevertheless, it is questionable whether enamel adhesion tests *in vitro* are representative of *in vivo* performance of bonding materials which were believed to be less effective under clinical conditions rather than under laboratory ones.[10] More research is needed to know the mechanism of performance of these self-etching primers, especially on the enamel surface.

CONCLUSION

Under the conditions of this *in vitro* study, the bond strength values of the two-step self-etching primer systems tested were similar to the total-etch. And, one step self-etching primers have lower bond strength compared to the total-etch. The failure mode was similar among groups. The majority of the samples failed at the interface, between adhesive and enamel.

Future efforts should be directed toward understanding the ultrastructural components at the adhesive interface of these self-etching primer systems and their relationship to bond strength. Long-term clinical success will confirm the importance of simultaneous demineralization and infiltration of the dental substrate with resin, and its effect in avoiding hydrolysis of the interfacial components, which may be a primary advantage of these adhesive systems.

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