Influence of additional etching on shear bond strength of self-etching adhesive system to enamel

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ABSTRACT

Recently, self-etching adhesive system has been introduced to simplify the clinical bonding procedures. It is less acidic compared to the phosphoric acid, thus there is doubt whether this system has enough bond strength to enamel. The purpose of this study was to investigate the influence of additional etching on the adhesion of resin composite to enamel.

Ninety extracted bovine permanent anterior teeth were used. The labial surfaces of the crown were ground with 600-grit abrasive paper under wet condition. The teeth were randomly divided into six groups of 15 teeth each. Clearfil SE Bond®, Adper™ Prompt L-Pop and Tyrian SPE™ were used as self-etching primers. Each self-etching primers were applied in both enamel specimens with and without additional etching. For additional etching groups, enamel surface was pretreated with 32% phosphoric acid (UNI-ETCH, Bisco, Inc., Schaumburg, IL, USA). Hybrid resin composite Clearfil AP-X, (Kuraray Co., Ltd., Osaka, Japan) was packed into the mold and light-cured for 40 seconds. Twenty-four hours after storage, the specimens were tested in shear bond strength. The data for each group were subjected to independent t - test at $p < 0.01$ to make comparisons among the groups.

In Clearfil SE Bond®, shear bond strength of additional etching group was higher than no additional etching group ($p < 0.01$). In Adper™ Prompt L-Pop and Tyrian SPE, there were no significant difference between additional etching and non-etching groups ($p > 0.01$).

In conclusion, self-etching adhesive system with weak acid seems to have higher bond strength to enamel with additional etching, while self-etching adhesive system with strong acid seems not. [J Kor Acad Cons Dent 31(4):263-268, 2006]

Key words : Self-etching adhesive, Phosphoric acid etching, Shear bond strength, Enamel

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Ⅰ. INTRODUCTION

Since the acid-etch technique was first introduced in dentistry by Buonocore[1] in 1955, phosphoric acid has been routinely used to etch the enamel surface in order to create micromechanical retention with resin composites. Formation of tag-like resin extensions into the enamel microporosi-

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ties were considered the predominant bonding mechanism of resin to etched enamel[2,3].

Recently, in an effort to simplify the dentin/enamel bonding systems, a single procedure using self-etching adhesive system has been proposed. These acidic primers include a phosphorated resin molecule that performs two functions simultaneously that are conditioning and priming of tooth.

Several self-etching adhesive systems are on the market today, and the acidity of self-etching systems allows mineralized tissue to be etched in a single treatment step. However, there are some controversies on the etching effect of the acid of the self etching adhesives. Some studies have reported that weaker acids etch enamel as effectively as 30% to 40% phosphoric acid[4]; but data from other studies indicate that weaker acids provide significantly lower shear bond strengths when the manufacturers' recommended application times are used to etch enamel[5]. Self-etching primer may exhibit insufficient decalcification of enamel if it is a weak acid[6] and the priming time may influence the morphologies of the tooth-resin interfaces or bond strength[7,8].

Up to now, many self-etching adhesive systems vary in their acidity by differences in the composition and concentration of polymerizable acids and/or acidic resin-monomers in these systems[9,10]. Therefore the purpose of this study was to evaluate the influence of additional etching on shear bond strength of self-etching adhesive systems to enamel.

Ⅱ. MATERIALS AND METHODS

Ninety extracted sound bovine permanent anterior teeth were chosen. They were sectioned at cemento-enamel junction and embedded in autopolymerizing acrylic resin (Orthodontic Resin, Dentsply/Detrey, Konstanz, Germany) molds so that the prepared enamel surface was 2 ㎜ above acrylic resin cylinders. Specimens were placed in tap water to reduce the temperature rise from the exothermic polymerization reaction.

After the resin have completely polymerized, the labial surfaces of the teeth were ground with model trimming wheel and 600-grit abrasive paper under wet conditions to expose enamel surfaces. After ultrasonic cleaning with distilled water for 3 minutes to remove any surface debris, these surfaces were washed and dried with oil-free compressed air (Hotman, Dentro, Tokyo, Japan). The teeth were randomly divided into six groups of 15 teeth each and treated in the manner of Table 1. Composition of each system is shown in Table 2.

Each etching group was pretreated with 32% phosphoric acid with benzalkonium chloride (UNI-ETCH, Bisco, Inc., Schaumburg, IL, USA) for 15 seconds while non-etching groups received no pretreatment. After the specimens were fully rinsed with running water for 10 seconds and dried in Clearfil groups, Clearfil SE Bond® (Kuraray Co., Ltd., Osaka, Japan) primer was applied to the tooth surface with Microbrush (Int'l, Co., Ltd., Dungarvan, Waterford, Ireland). In Adper groups, after the two reservoirs were sequentially squeezed to mix the respective components, Adper™ Prompt L-Pop (3M ESPE AG., Germany) was applied and scrubbed on the bonding site of enamel for 15 seconds and then gently dried for 3 to 5 seconds with compress air. In Tyrian groups, Tyrian SPE™ (BISCO, Inc., Schaumburg., IL USA) primer were applied to the tooth surface with Microbrush.

After primed tooth surface was dried with oil-free compressed air for 5 seconds, the bonding agent was applied following the manufacturer's instruction, and air-thinned, and irradiated using a curing light (Optilux 501) for 10 seconds with the intensity of 400 mW/cm^2.

A mounting jig (Ultradent Product Inc., South Jordan, Utah, USA) with an internal ring of 2.38 ㎜ in diameter and height of 2.0 ㎜ was placed against the tooth surface and stabilized with an alignment tube. Hybrid resin composite (Clearfil AP-X, Kuraray Co., Ltd., Osaka, Japan) was packed into the mold and light-cured for 40 seconds. After polymerization, the alignment tube and mold were removed and the specimens were

Table 1. Experimental groups following surface treatment methods

Groups	Pretreatment of Enamel	Self-Etching Adhesive
Clearfil - non etching	Not etched	Clearfil SE Bond® (Kuraray Co., Ltd., Osaka, Japan)
Clearfil - etching	Acid Etched	
Adper - non etching	Not etched	Adper™ Prompt L-Pop (3M ESPE AG., Germany)
Adper - etching	Acid Etched	
Tyrian - non etching	Not etched	Tyrian SPE™ (BISCO, Inc., Schaumburg., IL USA)
Tyrian - etching	Acid Etched	

Table 2. Composition of the self-etching adhesive systems tested

Adhesive System		Composition*
Clearfil SE Bond®	PRIMER	MDP, HEMA, Hydrophilic dimethacrylate dl-Camphorquinone, aromatic tert-amine, Water
	BOND	MDP, Bis-GMA, HEMA, dl-Camphorquinone Hydrophobic dimethacrylate N,N-diethanol-p-toluidine, Silanated colloidal silica
Adper™ Prompt L-Pop	Liquid 1 (red)	Methacrylated phosphoric esters, Bis-GMA Initiators based on camphorquinone, Stabilizers
	Liquid 2 (yellow)	water, HEMA, Polyalkenoic acid, Stabilizers
Tyrian SPE™	Part A	Ethanol
	Part B	2-Acrylamido-2-methyl Propanesuflonic acid Bis(2-(methacryloyloxy)ethyl) phosphate, Ethanol
	One-Setp Plus	Biphenyl Dimethacrylate, Hydroxyethyl Methacrylate, Methacrylate, Acetone

MDP : 10-methacryloyloxy methacrylate; HEMA : 2-hydroyxylethyl methacrylate;
Bis-GMA : bisphenol-A glycidylmethacrylate.
* According to manufacturer's information.

placed in 37°C, 100% humidity. Twenty-four hours after storage, the specimens were tested in shear mode using a chisel-shaped rod in an Instron testing machine (Type 4202, Instron Corp., Canton, Massachusettes, USA) at crosshead speed of 1 mm/minute.

The data for each group were subjected to independent *t*-test at $p < 0.01$ to make comparisons between the groups.

Ⅲ. RESULTS

The results of the shear bond strength tests to enamel were shown in Table 3.

In Clearfil SE Bond®, additional phosphoric acid etching group showed higher shear bond strength than priming only group ($p < 0.01$). However, there were no significant differences between additional etching group and non-etching groups in both Adper™ Prompt L-Pop and Tyrian SPE™ ($p > 0.01$).

Table 3. Shear bond strength of resin composite to enamel with self-etching adhesive (MPa)

Groups		n	Mean ± SD
Clearfil	No etching	15	24.04 ± 4.6 *
	Etching	15	34.14 ± 5.1 *
Adper	No etching	15	23.36 ± 5.3
	Etching	15	23.25 ± 4.9
Tyrian	No etching	15	22.95 ± 5.2
	Etching	15	22.15 ± 4.8

* Significantly different at $p < 0.01$.

Ⅳ. DISCUSSION

Currently, clinical use of self-etching adhesive systems are increased because it is convenient and less technique sensitive. However, enamel bonding of self-etching adhesive systems is still a point of discussion. In the present study, additional etching procedure improved the bond strength of Clearfil SE Bond® and did not improve in Adper™ Prompt L-Pop and Tyrian SPE™.

Several studies have shown that self-etching adhesive with a resin composite did not provide as good bonding to the enamel surface as phosphoric acid etching system[11,12]. However, several studies reported good bonding performances of self-etching adhesive system on enamel[12]. Etching of enamel with 30% to 40% phosphoric acid gives bond strength about 20 MPa[13,14], and the results of this study showed good enamel bond strength comparable to these previous results.

In the present study, the shear bond strength of etching group was significantly higher than priming only group in Clearfil SE Bond®. This can be explained by the acidity of each self-etching bonding systems. Clearfil SE Bond® had a pH value of 2.6, while Adper™ Prompt L-Pop and Tyrian SPE™ had a pH value of 1.3 and 1.0 each[15]. According to Barkmeier *et al.*[16], primer with a pH around 1.4 presumably allows mineralized tissues to be conditioned and primed in one treatment step.

In the self-etching primer adhesive system, the acidic part of the primer may be neutralized partly by calcium and phosphate ions released during demineralization. Demineralization is therefore self-limiting, in that the high concentration of these ions tends to limit further dissolution of apatite. Thus the acidity of self-etching system is very important part of demineralizaiton of enamel, and it is coincident with the result of this experiment.

Clearfil SE Bond® contains a functional monomer, 10-methacryloyloxydecyl dihydrogen phosphate (MDP) and it may also bind to calcium. Recently, Yoshida *et al.*[17] reported that MDP tightly adheres to hydroxyapatite and its calcium salt hardly dissolves in water. MDP is considered a more favorable adhesive monomer since it has two hydroxyl groups that chelate to calcium ions of enamel[18]. Acidic monomers in primer, such as 4-acryloxyethyl trimet (4-AET) and 2-hydroxyethyl methacrylate (HEMA), may promote the penetration of bonding resin into dentin and enamel and/or may increase the wettability of bonding agent on tooth surface, resulting in an effective seal of flowable composite[19]. The difference in bonding capacity to tooth substrates among MDP (Clearfil SE Bond®), Bis-GMA (Adper™ Prompt L-Pop) and Bis(2-(methacryloyloxy)ethyl) phosphate (Tyrian SPE™) is unclear, but it is very interesting that these three adhesives show similar bonding performance for enamel without pretreatment of 32% phosphoric aicd. If the etching effect of Clearfil SE Bond® is less effective than other groups, this result may indicate that other factors - e.g., filled bonding layer - of the bonding system can affect the final bond strength.

In conclusion, additional phosphoric acid etching on the enamel surface can improve shear bond strength of mild acidic self-etching system. However, it does not affect on the strong acidic self-etching system.

Further investigation is needed to evaluate the clinical significance of additional etching on the other self-etching systems which have different pH acidic primer.

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국문초록

부가적인 산부식이 자가산부식 접착제의 법랑질에 대한 전단결합강도에 미치는 영향

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최근 많이 소개되고 있는 자가산부식 접착제는 상아질에 대한 접착은 우수한 것으로 알려져 있지만 법랑질에 대한 접착은 인산에 비해 낮은 산도로 인해 충분한 부식이 일어나는지에 대한 논란이 되고 있다.

본 연구에서는 서로 다른 산도를 가진 1단계 혹은 2단계의 자가산부식 접착제에서 부가적인 산부식이 법랑질과 복합 레진 사이의 전단결합강도에 미치는 영향을 평가하고자 하였다.

90개의 발거된 우치의 순면을 물기가 있는 상태에서 #600 사포로 표면을 연마하여 평활한 법랑질을 노출시킨 후, 임의로 6군으로 분류했다. 사용재료는 Clearfil SE Bond®, Adper™ Prompt L-Pop, 그리고 Tyrian SPE™를 사용하였으며, 각 재료에 추가적인 산부식을 시행한 군과 그렇지 않은 군으로 나누어 표면 처리한 후, 제조사의 지시대로 접착과정을 시행하였다. 각 시편에 복합레진을 2 ㎜ 두께로 충전하고, 40초간 광중합을 하였다. 그 후 시편을 37℃, 100% 상대습도에서 24시간 보관 후 전단응력 접착강도를 측정하였다. 결과치는 independent t - test를 이용하여 통계분석 하였으며 그 결과는 다음과 같이 나타났다.

Clearfil SE Bond®에서 부가적인 산부식을 시행한 군이 그렇지 않은 군보다 유의하게 높은 접착강도를 나타내었다 ($p < 0.01$).

Adper™ Prompt L-Pop과 Tyrian SPE™에서는 부가적인 산 부식이 결합강도에 통계적인 유의차를 나타내지 않았다 ($p > 0.01$).

본 연구의 결과로 보아, 강산을 함유하고 있는 접착제를 사용할 경우에는 부가적인 부식 처치가 접착력을 증가시키지 않으나, 약산을 함유하고 있는 접착제의 경우에는 부가적인 산부식이 접착력을 증진시키는 것으로 사료된다.

주요어: 자가산부식 접착제, 인산 산부식, 전단결합강도, 법랑질