The influence of moisture control on bond strength of composite resin treated with self-etching adhesive system

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국문초록

Self-etching adhesive system에서 수분 조절이 레진의 치질접착강도에 미치는 영향

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최근에 많이 사용되어지고 있는 치과용 접착제는 산 부식 후 수분이 있는 상태에서 적용하는 wet-bonding 술식을 많이 추천하고 있다. 하지만, self-etching primer의 경우 산부식과 priming 과정이 동시에 시행되고, 제조자들은 건조된 치아표면에 적용할 것을 추천하고 있다. 그러나 건조된 정도에 대하여서는 별다른 추천사항이 없으며, 수분이 self-etching primer에 어떤 영향을 미치는지에 대하여서는 별다른 연구가 이루어져 있지 않은 상태이다. 이에 본 연구에서는 치질 삭제 후 남아있는 수분이 self-etching primer의 레진 접착 강도에 어떤 영향을 미치는 지를 알아보고자 하였다.

발거한 대구치 96개를 이용하여 물기가 있는 상태에서 #600 사포로 표면을 연마하고, 법랑질 면을 노출시킨 군과 상아질을 노출시킨 군으로 분류 후, 30분 공기 중 방치 군 (1군), 5초 공기 건조 군 (2군), 1초 공기 건조 군 (3군), 솜으로 약간의 물기를 제거한 군(blot dry)(4군) 등 총 8개의 군으로 나누었다. Self-etching adhesive system인 Clearfil SE Bond primer를 20초간 적용하고, bonding제 도포 후 10초간 광중합 시행하였다. 접착제 처리한 치아면에 몰드를 고정한 후 Clearfil AP-X 복합레진을 2mm 충전하고, 40초간 광중합을 시행하였다. 24시간 후 전단 응력 결합강도를 측정하였으며, 그 결과는 다음과 같이 나타났다.

법랑질과 상아질 모두에서, 30분 건조군과 5초 공기건조군이 1초와 blot drying 군보다 높은 결합강도를 보였으며, 통계학적으로 유의한 차이를 보였다($p<0.05$).

본 실험 결과에 의하면 self-etching adhesive system을 사용함에 있어서 법랑질과 상아질군 공히 건조된 상태에서 사용하여야 하며 수분의 존재시 치아와의 결합력이 감소하는 것으로 나타났다. 따라서 임상에서 접착제의 적용시 수분의 조절에 주의하여야 할 것으로 사료된다.

Key words : Dentin bonding system, Self-etching primer, Surface wetness, Clearfil SE

Ⅰ. Introduction

A goal of restorative dentistry is to develope adhesive restorative materials that are durable and provide an effective seal at the restoration/tooth interface. To achieve this purpose, many enamel-dentin bonding systems were introduced and improved in a new formula. These new adhesive materials not only attempt to improve the quality of the bond, but also simplifying the clinical procedures.

Most recent innovative changes of bonding concept are the "total-etch" and "wet-bonding" techniques, and these methods have been used for many years. However, when using the wet bonding technique it is difficult to maintain the optimal level of moisture.

In order to simplify the clinical procedures and improve the quality of the bonding, a self-etching adhesive system was developed. It was developed to eliminate the conditioning, rinsing, and drying steps that may prove to be critical and difficult to standardize in operative conditions[1]. Also it has been reported that the self-etching primer system produce high bond strengths to dentin, independent of regional differences-deep vs superficial dentin, crown vs root dentin[2].

Theoretically, the new acidic part of the primer dissolves the smear layer, incorporating it into the mixture, as it demineralize the dentin and encapsulates the collagen fibers and hydroxyapatite crystals. The acidity of the primer is less than that of 32% or 37% phosphoric acid gels, but is sufficient to etch through smear layers into the underlying enamel or dentin[3,4]. Barkmeier et al[5] examined the effect of this system, an acidic primer, on enamel and dentin through shear bond strength testing and evaluation of marginal microleakage. SEM examination revealed resin penetration into both enamel and dentin surfaces, indicating that adequate conditioning was achieved and produced high bond strengths to both enamel and dentin. Since the etching and priming processes run parallel to each other, depth of demineralization and penetration depth of the bonding agent are identical, and it should minimize voids, as all the entire region would be completely encapsulated by the resin primer. As a result, light curing of these penetrated monomers and copolymerization of the overlying resin bonding agent and composite resin form a continuous bond with the tooth surface which is capable of resisting the effect of microleakage[6].

But, in clinical usage of self-etching primer system, there are some problems that must be solved. First, the enamel etching, using the self-etching primer system, is controversial. The acidic part of the primer is neutralized by calcium and phosphate ions released during demineralization. When the water is evaporated during air drying, the concentrations of solubilized calcium and phosphate within the primer may exceed the solubility product constants for a number of calcium and phosphate. Thus it is conceivable that residues of the primer or possibly precipitates of calcium phosphates could remain on the tooth surface and thereby masked the etching pattern. Therefore, demineralization is self-limiting, in that the high concentration of these ions tends to inhibit further dissolution of hydroxyapatite[7].

Second, manufacturer recommended dry bonding technique, to avoid diluting the acidic component of the primer with excess surface water. It has already been proven that proper application of the adhesive primer plays an important role in achieving good bond strength[8]. However, clinicians have been using the wet bonding technique for many years, so they are still becoming adept at using this techniques. Sometimes, adequate moisture control is difficult or not possible in some clinical situations. In these condition, the tooth surface is moist and the bonding quality can be affected. But little research has been conducted regarding the effects of moisture when using the self-etching adhesive system.

The purpose of this study is to determine the influence of moisture control on bond strength of the self-etching adhesive system by measuring shear bond strengths of composite resin to enamel and dentin treated with self-etching adhesive system.

Ⅱ. Mateirals and Methods

Ninety-six extracted, noncarious human molars stored at 4℃ in isotonic saline were used in this study. The ages of the patients were not known and the reasons for extraction ranged from periodontally compromised teeth to impacted teeth. During the last 24 hours before beginning the experiment, they were kept in distilled water.

The teeth were embedded in auto-polymerizing acrylic resin (Orthodontic Resin, Dentsply/Detray, Konstanz, Germany) molds so that the prepared enamel and dentin surfaces were 2 mm above the acrylic resin cylinders, and placed in tap water to reduce the temperature rise from the exothermic polymerization reaction.

After the resin had completely polymerized, the occlusal surfaces of the teeth were ground perpendicular to the long axis of the tooth on a water-cooled, model trimming wheel to create flat enamel and dentin surfaces. Then the enamel and dentin surfaces were hand finished with using wet 600-grit silicon carbide abrasive papers using twenty 15 cm long strokes. After ultrasonic cleaning with distilled water for 3 minutes to remove the excess debris, these surfaces were washed and dried with oil-free compressed air (Hotman, Dentro, Tokyo, Japan). The teeth were randomly divided into eight groups of 12 teeth each and treated in the manner (Table 1). Then the Clearfil SE Bond primer (Kuraray Co., Ltd., Osaka, Japan)(Table 2) was applied to the tooth surface with Microbrush (Int'l, Co., Ltd., Dungarvan, Waterford, Ireland) and allowed to sit for 20 seconds according to the manufacturer's recommendations.

After primed tooth surface was dried with oil-free compressed air for 5 seconds from a distance of 10 cm, the bonding agent was applied to the surface and irradiated with curing unit (Spectrum 800, Dentsply/Detray, Konstanz, Germany) for 10 seconds, with the intensity set at 400 mW/cm^2.

A mount jig (Ultradent Product Inc., South Jordan, Utah, U.S.A.) with an internal ring of 2.3798 mm in diameter and height of 2.0 mm was placed against the tooth surface and stabilized with an alignment tube. A 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 placed in 37℃ distilled water. Twenty-four hours after storage, the specimens in each group were tested in shear mode using a chisel-shaped rod in an Instron testing machine (Type 4411, Instron Corp., Canton, Massachusettes, U.S.A) at a crosshead speed of 1 mm/minute.

The data for each group were subjected to one-

Table 1. Experimental groups following drying condition of enamel and dentin surfaces

	Enamel / Dentin drying condition
Group 1	dried for 30 min. at room temperature dry no visible moisture was seen
Group 2	dried for 5s with oil-free air dry from a distance of 10 cm, no visible moisture, excessived desiccation avoided
Group 3	dried for 1s with oil-free air dry from a distance of 10 cm, removed the excessive moisture
Group 4	blot dry with cotton pellet, whole surface was visibly moist

Table 2. Composition of Clearfil SE Bond

Clearfil SE Bond	
PRIMER	BOND
10-Methacryloyloxydecyl dihydrogen phosphate (MDP)	10-Methacryloyloxydecyl dihydrogen phosphate (MDP)
2-Hydroxyethyl methacrylate (HEMA)	Bis-phenol A diglycidylmethacrylate (Bis-GMA)
Hydrophilic dimethacrylate	2-Hydroxyethyl methacrylate (HEMA)
dl-Camphorquinone	dl-Camphorquinone
N,N-diethanol-p-toluidine	Hydrophobic dimethacrylate
Water	N,N-diethanol-p-toluidine
	silanated colloidal silica

Table 3. Shear bond strength of resin composite to enamel treated with Clearfil SE Bond (MPa)

(Mean S.D.)

Group	n	Shear bond strength
E1 (30min, dry)	12	27.5±5.5
E2 (5s, air dry)	12	26.2±6.3
E3 (1s, air dry)	12	18.6±4.1*
E4 (blot dry)	12	17.3±4.3*

*statistically significant difference by One-way ANOVA ($p<0.05$).

Table 4. Shear bond strength of resin composite to dentin treated with Clearfil SE Bond (MPa)

(Mean S.D.)

Group	n	Shear bond strength
D1 (30min, dry)	12	24.8±4.6
D2 (5s, air dry)	12	25.7±3.2
D3 (1s, air dry)	12	17.1±3.1*
D4 (blot dry)	12	16.8±2.4*

*statistically significant difference by One-way ANOVA ($p<0.05$).

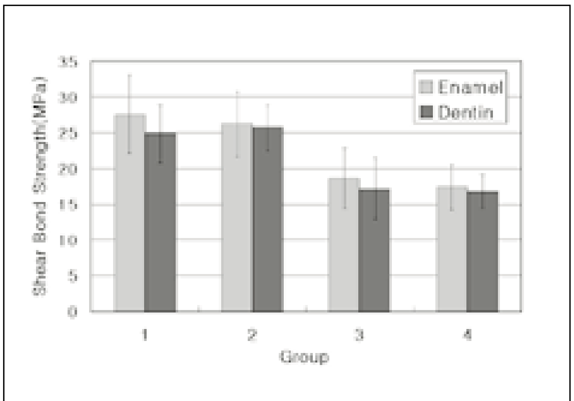

Fig. 1. Shear bond strength of resin composite to enamel and dentin treated with Clearfil SE Bond.

way ANOVAs followed by the Duncan's multiple range test at $p<0.05$ to make comparisons among the groups.

Ⅲ. Results

The results of the shear bond strength tests to enamel are shown in table 3, and those of dentin in Table 4.

In both the enamel and dentin experimental groups, there were statistically significant differences ($p<0.05$) between these groups which were dried for 30 minutes at room temperature (E1, D1)or for 5 seconds with oil-free air (E2, D2) and those which were dried for 1 second with oil-free air (E3, D3) or blotted dry (E4, D4)($p<0.05$).

Ⅳ. Discussion

Recently developed adhesive systems are characterized by their simple application procedure. Among simplified adhesives the self-etching approach currently appears most promising in avoiding inadequate hybridization due to collagen collapse in over-dry conditions or due to residual solvent in over-wet conditions. In self-etching adhesive system, simplification of the clinical application procedure is obtained not only by reduction of application steps, but by omission of postconditioning rinsing phase. As a rinse phase is not required, the risk of substrate contamination with saliva or other dentin-porosity blocking agents is virtually eliminated. As a supplementary advantage, the controversy of post-conditioning drying or keeping the dentin moist, as in wet-bonding technique is avoided. The actual rationale behind these systems is to superficially demineralize dentin and to simultaneously penetrate it to the depth of demineralization with monomers that can be polymerized in situ. However, in clinical situation, the complete dry of cavity is difficult.

Based on their interaction with dentin, there are two primary types of self-etching adhesive system distinguished[9].

The first group of moderate self-etching systems is constituted by Clearfil SE Bond[10], Clearfil Liner Bond 2V[11], F2000 primer/adhesive systems (3M dental products Inc., St. Paul, MN, U.S.A), and Unifil Bond (GC, Tokyo, Japan), all of which have a pH of about 2. The latest self-etching primer system, Clearfil SE Bond (Kuraray, Osaka, Japan, known in Japan as Clearfil Mega Bond) combines the two separate primer solutions of Clearfil Liner Bond 2V into a single bottle[2]. This reduces the etching time 30s to 20s[12].

The second group consists of adhesive systems such as Prompt L-Pop (ESPE, Seefeld, Germany)[13], a self-etching adhesive, and Prime & Bond NT (Dentsply/DeTrey, Konstanz, Germany) in which a nonrinse conditioner (NRC)[14] is used as a self-etching primer treatment. These adhesives have a pH of 1 or less and interact more profoundly with dentin. Despite the fact that these "strong" self-etching primers are not rinsed off, their interfacial ultramorphologic features closely resemble those of total-etch systems that use phosphoric acid that is rinsed off[15]. Based on this fact, it might be assumed that water dilution of acidic composition in Clearfil SE Bond would influence the etching ability. But there has not been enough research conducted in water dilution and etching ability to determine this fact.

This study evaluated the shear bond strength of self-etching adhesive system, Clearfil SE Bond, in the presence of varying amount of water that are to be expected during clinical bonding procedures.

According to the manufacturer's recommendations, dry bonding is an effective technique to use with the Clearfil SE Bond system. But, the degree of dryness is not stipulated in the manufacturer's recommendation. Following the results of this experiment, both enamel and dentin, 5 seconds and 30 minutes dry groups showed higher bond strength than 1 second and blot dry groups. 1 second air drying and blot drying with a cotton pellet are both representative of procedures which are used in the wet bonding technique. And it was found that 5 seconds of air drying was sufficient time for the extra moisture to evaporate from the tooth surface. As expected, moisture had a detrimental effect on the bond strength of the self-etching primer.

The low bond strengths of the blot drying and 1s air drying groups might have been caused by the resulting dilution and incomplete diffusion of primer components. Such dilution and incomplete diffusion could preclude the production of high-quality polymers, thus compromising bond strength.

The high moisture condition can adverse effect on Clearfil SE system. Camphoroquinone (CQ) is widely used as a photoinitiator for bonding systems and it is also used in Clearfil system. It is impossible for CQ to initiate the polymerization of water-soluble monomers in the water. Furthermore it is doubtful that CQ can sufficiently initiate the polymerization of diffused monomer, inside the dentin due to its water content and the fact that it has a lower partition coefficient than resin. Assuming that water may be retained in the HEMA, and that the diffusion of the light-activating components may be delayed by interference with the water, it seems reasonable to suspect that large variations in cure may exist within the hybrid layer. To solve this problem, 2-hydroxy-3-(3,4-dimethyl-9-oxo-9H-thioxanthen-2-yloxy)-N,N,N-Trimethy l-1-1-propanaminium chloride (QTX) was introduced as a alternative to CQ. It is supposed that QTX could initiate sufficient polymerization of diffused monomers inside the dentin despite the presence of water and can improve the polymerization degree of the diffused monomers, result in improve bond strengths to dentin[16]. Of course, there is not sufficient research in this area to come to a definite conclusion.

Clearfil SE Bond system contains water as a primer component. When water is used as a solvent, the HEMA[17] molecules do not saturate the collagen mesh as quickly as it would if an acetone-HEMA solvent were used[12]. Instead, the water and HEMA molecules compete for space within the collagen mesh. The presence of water inside the collagen mesh may cause a lower degree of polymerization than occurs with the HEMA/Bis-GMA bonding resin. This results in a lower molecular weight of the poly-HEMA and a weaker interpenetrating network[18]. If excessive mositure was present on the primed tooth surface, Bis-GMA component of bonding mixes poorly with water. Therefore, the Bis-GMA monomer forms an emulsion with the water, and during curing process, a Bis-GMA “sponge” or Bis-GMA droplets cure depending on the geometrical shape of the Bis-GMA phase. Thus, an increase in water will increase the pore size of the “sponge” or increase the distance between the droplets. Therefore, the reduced conversion caused by water contamination also reduces bond strength[17].

Jacobson et al[17] tested the effects of water on the polymerization of a mixture of adhesive monomers by measuring the conversion rate of double bonds by FT-IR. In the presence of no water, the conversion rate was 53.5%. Although addition of 5% water only reduced the conversion rate to 52.6%, 10% water reduced it to 31.5%, and 20% water further reduced it to 22.7%. Thus, it appears that excess residual water may adversely affect the polymerization of the resin, thereby lowering its final mechanical properties.

According to Pashley et al[10], the rate of evaporation of water from water-HEMA mixtures is inversely related to the relative humidity of the environment and to the vapor pressure of water. Relative high humidity lowers the net flux of water vapor, so residual water might interfere with polymerization of adhesive monomers, thereby lowering the quality of the hybrid layer. The degree of environmental humidity is high and comparable to that of the oral cavity when no rubber dam is used, whereas with rubber dam use the environmental humidity will be similar to that of the ambient air in the operatory[19]. Thus, rubber dam application while using the bonding system is a very important procedure.

MDP is incorporated in both primer and bonding agents of the Clearfil SE Bond system (Table 2). A similar phenomenon has been reported as “MDP balls” when an aqueous mixture of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) and 30% HEMA was applied to smear layer retained dentin[20] and the same such micelles resulted. Such interface deficiencies undoubtedly weaken the resin-dentin bond and result in incompletely sealed tubules.

Excessive water that was not adequately removed during priming appeared to cause deterioration of the bonded assembly along the hybrid layer-resin interface[21], as well as phase separation of the hydrophobic and hydrophilic monomer components, which caused blistering and the formation of the globules at the resin-dentin interface[9].

In treating more complex cavities like cervical abrasion or gingival box-like cavities, moisture control of operative site is difficult to perform. A simple, flat bonding site used in this study does not exist in the clinical situations. Therefore, using an air syringe to remove excess surface water from complex cavities could produce areas of ideal surface moisture, but could also result in over-wet or over-dry line angles[11]. Therefore, nonuniform surface condition might produced, leading to lower bond strengths. So, when air drying before the application of self-etching system, warm and oil-free air is preferred, careful usage, sufficient drying time, and visual examination are recommended.

The self-etching primer systems efficiently bonded to dry tooth surfaces. Thus before any bonding procedure is begun, adequate isolation and moisture control of the substrate to be bonded to must be achieved. Following the result of this experiment, using an oil-free compressed air for 5s sufficiently dry the tooth surface without causing excessive drying the tooth structure. In practical view, clinicians should be cautious not to overwetting the dentin surface, such as puddling around the line angles of cavity. More research is needed to examine the varying drying methods and times for better bonding strengths when using the self-etching adhesive system.

Ⅴ. Conclusion

Recently, self-etching adhesive systems have been developed and bonding procedures simplified into two steps, which are simultaneously applied to both enamel and dentin. These systems are easy to use and have the potential for good clinical success. It has been reported that the self-etching adhesive systems produce high bond strength to enamel and dentin.

The purpose of this study was to study the influence of moisture control on bond strength of self-etching primer by measuring enamel and dentin shear bond strengths.

96 human molars were divided into 8 groups. Group E1 (Enamel, 30min room temperature dry), Group E2 (Enamel, 5s air dry), Group E3 (Enamel, 1s air dry), Group E4 (Enamel, blot dry), Group D1 (Dentin, 30min room temperature dry), Group D2 (Dentin, 5s air dry), Group D3 (Dentin, 1s air dry), Group D4 (Dentin, blot dry). Clearfil SE Bond primer was applied to the enamel and dentin surfaces and after 20s surfaces were air dried. Then the bonding agent was applied and light cured for 10 seconds. A 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 a shear bond test.

Both in enamel and dentin, 30 min drying and 5 seconds air drying groups showed significantly higher bond strengths than 1s air drying and blot drying groups ($p<0.05$). Using the self-etching adhesive system, this study concluded that the complete dry bonding technique is effective on both enamel and dentin. When the excess moisture is existing on the bonding surface, shear bond strength decreased rapidly. So in clinical application of self-etching adhesive system, the cautious moisture control is needed.

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