Influence of the curing time for the adhesive on the oxygen-inhibited layer thickness and the shear bond strength to dentin

Yong-Hoon Choi[1], Ji-Hyun Bae[1], Ho-Hyun Son[1], In-Bog Lee[1], Chung-Moon Um[1],
Seung-Ho Baek[1], Oh-Young Kim[2], Chang-Keun Kim[3], Byeong-Hoon Cho[1]*
[1]*Department of Conservative Dentistry & Dental Research Institute, College of Dentistry, Seoul National University,*
[2]*Department of Polymer Science and Engineering, Dankook University,*
[3]*Department of Chemical Engineering, Chung-ang University*

ABSTRACT

Objectives : This study investigated the hypothesis that increasing light-curing time would leave the oxygen-inhibited layer (OIL) of the adhesive thinner, and in turn, result in lower shear bond strength (SBS) than those obtained by the routine curing procedures.

Methods : 120 human extracted posterior teeth were randomly divided into three groups for bonding with three adhesives: All Bond 2®, One Step®, and Adper Prompt®. They were subsequently divided into four subgourps with different light-curing time (10, 20, 30 and 60 s). The assigned adhesives were applied on superficial occlusal dentin according to the manufacturer's instructions and cured with one of the four curing times. Composite resin cylinder, 2.35 mm in diameter, were built on the cured adhesive and light-cured for 40 s. SBS were measured after 24 h from the bonding using a universal testing machine (crosshead speed 1.0 mm/min). The relative thickness of the OIL and the degree of conversion (DC) were determined from the adhesive on a slide glass using FT-NIR in an absorbance mode. Data were analysed with One-way ANOVA and Duncan's multiple test ($p < 0.05$).

Results : With increasing cure time, although there were no significant difference in th SBS of One-step and Adper Prompt ($p > 0.05$), those of All Bond 2 decreased significantly ($p < 0.05$). The relative thicknesses of the OIL on each adhesive were not affected by the cure time ($p > 0.05$). Although the DC of All-Bond 2 were statistically not different with increasing cure time ($p > 0.05$), those of One-Step and Adper Prompt showed an increasing trends with increasing cure time ($p < 0.05$).

Conclusions : Increasing light-curing time did not affect on the relative thickness of the OIL of the adhesives, and in turn, on the SBS to dentin. [J Kor Acad Cons Dent 29(2):177-184, 2004]

Key words : Oxygen inhibited layer, Shear bond strength, FT-NIR, Light curing time

Ⅰ. INTRODUCTION

Adhesion to dentin has imposed some inherent challenges to clinicians because dentin is a more heterogeneous substrate with much higher organic and water content than enamel. Adhesive systems for dentin adhesion has been greatly improved during the latest decade. However, there is still controversy concerning the source of the bond strength, including the role of the hybrid layer[1-3] and the thickness of the adhesive layer produced by dentin bonding systems[4-7]. Although there seems to be no relationship between the thickness

*Corresponding author: **Byeong-Hoon Cho***
Department of Conservative Dentistry,
College of Dentistry, Seoul National University
28-2 Yeongun-dong, Chongro-gu, Seoul, Korea 110-749
Tel : 82-2-760-3514, 2651 Fax : 82-2-764-3514
E-mail : chobh@plaza.snu.ac.kr

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of the hybrid layer and the tensile bond strength[8,9], there is limited information correlating the bond strength and the physical or mechanical properties of the adhesive resin layer.

Generally, prolonged light-curing time produced higher degree of conversion (DC) in adhesives[10], and the mechanical properties of photo-polymerizing dental materials increased with increasing DC[11,12]. As a physical factor of the adhesive layer, the thickness of it may relieve the stress from the polymerizing composite resin[13-15] and the occlusal load[16], and attribute to increase bond strength. However, the material dependency of the relationship between the adhesive layer thickness and the bond strength was also reported. That is, for the bonding agent containing no solvent, bond strength increased with increasing adhesive layer thickness. But the bond strength of the bonding agent containing solvent decreased when the thickness of the adhesive layer increased[7]. It was also reported that no linear relationship between the degree of conversion and the microhardness[17].

Oxygen was known to inhibit the polymerization of the surface layer of the composite resin. This is because of the greater ability of oxygen compared with that of a monomer molecule, to react with growing radicals[18]. Thus, it was assumed that oxygen from the ambient atmosphere might prevent polymerization of thin outer-most layer of the adhesive. It supplies sufficient double methyl methacrylate bonds for copolymerization of the adhesive resin with the restorative resin, so that the shear bond strength can increase[19-21]. Boyer, et al[19] reported that the oxygen inhibited layer induces brittleness due to "inadequate" links. Other researchers have found that the degree of conversion, independent of cure method, has a critical effect on the final mechanical properties and wear rate of composite materials[22]. The thickness of the inhibited layer has been found to depend on the viscosity, the composition, and the initiating system of the composite resin[18].

Generally, manufacturers recommend light-curing of their commercial adhesives for 10 or 20 seconds. In the clinical situations, increasing light-curing time may produce higher degree of conversion, so that the thickness of the well-polymerized adhesive layer may increase and, in turn, the adhesive layer may supply better stress-absorbing effect and increase the bond strength. However, it may also decrease the thickness of the oxygen-inhibited layer, which result in decrease of bond strength. In this study, the hypothesis was evaluated that increasing the light-curing time for the adhesive would result in a thinner oxygen-inhibited layer, and consequently lower shear bond strength.

Ⅱ. MATERIALS AND METHODS

Measuring the shear bond strength

One hundred and twenty extracted, non-carious human molars, which were stored at 4℃ in distilled water, were used in this study. The teeth were embedded in acrylic molds using self-curing acrylic resin (Orthodontic Resin, Dentsply/Detray, Konstanz, Germany) so that the prepared dentin surfaces were 2 mm above the acrylic mold, 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 cut away with the Isomet low speed saw (Isomet; Buehler Ltd, Lake Bluff, IL, U.S.A.) in order to expose the middle dentin, and create flat surfaces perpendicular to their long axis. The exposed section was examined to ensure no pulpal horn was exposed.

The exposed surfaces were abraded with 500-grit silicon carbide paper under a stream of water to produce a uniform smear layer, and then rinsed with water. The teeth were randomly divided into three groups; the teeth in each group were treated with one of the following three dentin bonding agent; 1) All Bond 2® (Bisco, Inc., Schaumburg, IL, USA.), 2) One Step® (Bisco, Inc., Schaumburg, IL, USA), 3) Adper prompt® (3M ESPE. St. Paul. MN, USA). After bonding with the assigned bonding agents following the manufacturer's instructions, each group was divided into four subgroups; 10, 20, 30 and 60 seconds light-curing subgroups, and the bonding agents were cured for the assigned curing time in order to evaluate the influence of the curing time on the shear bond strength to dentin. A mounting jig (Ultradent Product Inc, South Jordan, UT, USA) with an internal ring of 2.38 mm in diameter and 2.0 mm in height was place against the tooth surface and stabilized with an alignment tube. Denfil® composite resin (Vericom Co., Anyang, Korea) was packed into the mold and light-cured for 40 seconds using ULTRA PLUS light curing unit (light intensity: 600 mW/cm^2; Hilux. Benlioglu Dental Inc. Ankara, Turkey). After polymerization, the alignment tube and mold were removed and the specimens were placed in distilled water at room temperature for 24 hours. The specimens were tested in a shear mode using a chisel-on-composite cylinder method[23] in an Instron testing machine (Type 4466, Instron Corp., Canton, MA, USA) at a crosshead speed of 1 mm/minute.

Measuring the oxygen-inhibited layer thickness and the degree of conversion

The near-infrared (NIR) spectra from 8 specimens in each subgroups (cured for 10, 20, 30, or 60 seconds) for three adhesives (All Bond 2®, One Step®, Adper Prompt®) were recorded in absorbance mode using FT-IR (NEXUS, Nicolet Instrument Corp. Madison, WI, USA). Three spectra from a specimen were collected at three stages of specimen preparation, that is, before curing, after curing, and after removing the uncured layer from the cured specimen in order to calculate the degree of conversion and the relative thickness of the oxygen-inhibited layer. Adhesive was gently painted on a slide glass with a disposable brush, and a NIR spectrum of the uncured adhesive was collected from the adhesive on the slide glass mounted on the specimen holder in the FT-IR chamber. The area of the methacrylate =C-H peak at 6164 cm^{-1} in the absorbance mode was recorded using baseline technique. The specimen was cured immediately after collecting the spectrum of the uncured adhesive. The second NIR spectrum of the cured specimen was collected in the same way. After collecting the second spectrum of the cured adhesive, the specimen was wiped off with 75% ethyl alcohol sponge to remove the uncured oxygen inhibited layer. The third spectrum of the specimen without oxygen-inhibited layer was then collected. The relative thickness of the oxygen-inhibited layer was obtained by subtracting the height of the metahcrylate peak at 6164 cm^{-1} of the third spectral measurement after removing the uncured layer from the second one after curing. The degree of conversion was also obtained from the height of the methacrylate peak at 6164 cm^{-1} of the first and the second spectra.

Statistical analyses and all pairwise multiple comparisons of the shear bond strength, the degree of conversion, and the oxygen-inhibited layer thickness were performed with One-way ANOVA and the Duncan's multiple range test at $p = 0.05$. Statistical calculations were performed using SigmaStat (Version 2.03; Jandel Scientific, Chicago, IL, USA).

Ⅲ. RESULTS

Shear bond strength

The mean shear bond strength of three experimental groups were shown in Table 1. The shear bond strengths of All Bond 2® cured for 10 s or 20 s were significantly higher than those cured for 30 s or 60 s ($p < 0.05$). The shear bond strength of One-Step® were statically not different, even though the cure time was increased ($p > 0.05$). Those of Adper Prompt® were significantly lower after being cured for 10 s than for the longer cure times ($p < 0.05$).

Oxygen inhibited layer thickness and Degree of conversion

The difference between the % absorbance of the second and the third NIR spectra measured using FT-IR spectroscopy was represented the relative thickness of the oxygen-inhibited layer. The val-

Table 1. Mean shear bond strength of three dentin adhesive systems with increasing cure time

(Mean ± S.D., n = 10, unit: MPa).

	10 sec	20 sec	30 sec	60 sec
All bond 2	26.44 ± 7.82[a]	30.73 ± 5.52[a]	17.81 ± 6.34[b]	17.63 ± 5.02[b]
One Step	20.46 ± 5.68[a]	17.06 ± 6.13[a]	15.26 ± 3.36[a]	18.39 ± 5.25[a]
Adper Prompt	15.96 ± 7.42[a]	21.04 ± 4.78[b]	22.62 ± 3.01[b]	18.85 ± 3.03[b]

* The same superscripts mean that they are not significantly different (One-way ANOVA, $p < 0.05$)

Table 2. The difference between the % absorbance of the second spectra obtained after curing and that of the third spectra obtained after removing overlying uncured layer with alcohol sponge (% Absorbance; Mean ± S.D., n = 8)

	10 sec	20 sec	30 sec	60 sec
All bond 2	0.0023 ± 0.00010[a]	0.0022 ± 0.0003[a]	0.0023 ± 0.0008[a]	0.0022 ± 0.0006[a]
One Step	0.0013 ± 0.0004[a]	0.0011 ± 0.0002[a]	0.0011 ± 0.0001[a]	0.001 ± 0.0001[a]
Adper Prompt	0.0017 ± 0.0004[a]	0.0015 ± 0.0004[a]	0.0018 ± 0.0002[a]	0.0016 ± 0.0003[a]

* The same superscripts mean that they are not significantly different (One-way ANOVA, $p < 0.05$)

Table 3. The degree of conversion of the three adhesives after curing with increasing cure time

(Mean ± S.D., n = 8, Unit: %)

	10 sec	20 sec	30 sec	60 sec
All bond 2	40.05 ± 14.28[a]	42.78 ± 7.37[a]	47.28 ± 14.94[a]	42.63 ± 16.87[a]
One Step	57.42 ± 2.71[a]	65.54 ± 6.59[b]	63.18 ± 4.83[b]	67.08 ± 3.47[b]
Adper Prompt	19.69 ± 10.4[a]	26.81 ± 10.46[a]	32.23 ± 9.7[a]	40.65 ± 14.95[b]

* The same superscripts mean that they are not significantly different (One-way ANOVA, $p < 0.05$)

ues for each adhesive were statically not different even with the increasing cure time (Table 2, $p > 0.05$). All-Bond 2®, which had a separate and relatively viscous bonding agent, showed higher relative thickness of the oxygen-inhibited layer than the others having lower viscosity. The relative thickness of the oxygen-inhibited layer was not affected by the cure time.

The degree of conversion of All-Bond 2® adhesive composed of hydrophilic monomers showed no statistical difference with increasing cure time ($p > 0.05$). The degree of conversion of One-Step®, which has more acetone than the fourth generation precursor, was significantly lower after curing for 10 s than after curing for 20 s, 30 s, or 60 s ($p < 0.05$). Adper Prompt® having acidic mon-omers showed significantly lower degree of conversion than those of the other adhesives after curing for the assigned cure times except 60 s (Table 3).

Ⅳ. DISCUSSION

The correlations between two of the three variables tested in this study, that is, the cure time, the relative thickness of the oxygen-inhibited layer, and the shear bond strength, were evaluated. Firstly, although increasing light-curing time increased the shear bond strength of Adper Prompt® with curing for more than 20 s or decreased that of All-Bond 2® after 30 or 60 s cure, the correlations between the cure time and the shear bond strength of three adhesives were very low. Although the decrease of the shear bond strength in the case of All-Bond 2® seemed to be met well with the hypothesis, the minor increase in the case of Adper Prompt® could not be made sense, but a possibility might be the role of the acidic monomer inhibiting the acceleration of the polymerization by basic amines and resulting slow increase of the degree of conversion of Adper prompt®. The shear bond strength of All bond 2® was highest between 3 adhesives, especially at 20 s curing time that recommended by manufacture. Secondly, the relative thickness of the oxygen-inhibited layer of three adhesives were not affected by increasing light-curing time. Instead, it was independent to the curing time and consistent for a certain adhesive material even with the increase of cure time ($p < 0.05$). This result met with the assumption that the diffusion coefficient of the oxygen into an adhesive would be the determinant of the thickness of the oxygen-inhibited layer and might be affected by the viscosity of the adhesive[20]. Thirdly, all the adhesives did not show any correlation between the oxygen-inhibited layer thickness and the shear bond strength. Therefore, it seemed that the thickness of the oxygen-inhibited layer was determined by the diffusibility of the atmospheric oxygen and the viscosity of the adhesive[18,24].

In this study, the relative thickness of the oxygen-inhibited layer and the degree of conversion were measured using NIR spectroscopic technique, in order to measure them simultaneously. Currently, IR spectroscopy using the wavelengths of mid-IR or near-IR regions, Raman spectroscopy and DSC are the methods used widely to measure the double bond conversion of resin systems[10,25]. Among them, Mid-IR spectroscopy is used the most commonly. Using the wavelengths of near-IR region, high transmittance of the light in the near-IR range can pass through relatively thick specimens. As the conversion values obtained by near-IR and mid-IR techniques did not differ significantly, near-IR spectroscopy can be used to measure the conversion of dental resins up to 4 mm thick[26].

According to this study, oxygen-inhibited layer were not significantly different within a certain adhesive material, regardless of the increase in the light-curing time. The depth of the oxygen-inhibited layer remaining on the surface of the adhesive was reported to be affected more by the atmospheric oxygen pressure, the temperature, and the viscosity of the adhesive itself[18,24]. In this study, as the thickness of the adhesive layer coated on a glass with a brush was so thin, we could not measure it. Thus, the correction of the % absorbance with the thickness of the specimen, which is used routinely in NIR technique, was not performed. Therefore, the thickness reported in this study is not the absolute thickness of the oxygenn-inhibited layer. From the data, regardless of the total thickness of the adhesive layer and the cure time, the reduction in % absorbance after removing the overlying uncured layer from the underlying cured adhesive layer was not different within a material. Such independency to the total thickness of the adhesive and the cure time resulted in almost the same values within a material. This dependency on a material might be supported by the suggestions that the thickness of the oxygen-inhibited layer would mainly be determined by the viscosity of the adhesive and the diffusion of the atmospheric oxygen[18,24].

Degree of conversion were not significantly different with increasing cure time in All Bond 2® group ($p > 0.05$). Those in One Step® group increased significantly after curing for 20, 30 or 60 s comparing to those for 10 s ($p < 0.05$), but among the subgroups after curing for 20, 30 and 60 s, the degree of conversion were not significantly different ($p > 0.05$). In the case of Adper Prompt®, the degree of conversion increased progressively along with increasing cure time ($p < 0.05$). The low degree of conversion after short cure time may result from the inhibitory effect of the acidic monomer in the acceleration stage of the polymerization by amines. However, there is little correlation between the degree of conversion and the shear bond strength (All Bond 2®; $r = 0.168$, One Step®; $r = 0.5$, Adper Prompt®; $r =$

0.18). There were controversies about the relationship between the degree of conversion and the mechanical property of the polymeric dental materials. Peutzfeldt et al.[27] and Palin et al.[28] reported positive relations between the degree of conversion and the diametral tensile strength, the flexural strength, and the flexural modulus. However, Tassery et al.[17] reported that there is no linear relationship between the degree of conversion and the microhardness of composite resins. But these studies were conducted with composite resins, and tested with longer light curing time more than 1 hour using indirect technique. In this study, as the rate of polymerization reaction was so fast in the adhesives using photoinitiation system that the polymerization of the adhesive was completed before the recommended cure time, the correlations between the degree of conversion and the bond strength of the adhesive layer were very low. As the relative thickness of the oxygen-inhibited layer were the same with increasing cure time, our hypothesis that increasing cure time would decrease the thickness of the oxygen-inhibited layer and, as a result, would decrease the bond strength was rejected. Clinically, the recommended cure time by the manufacturers will be sufficient to get the optimal properties of the assigned adhesive and increase of cure time without appropriate scientific background will loose the most valuable time of dentists. Additionally, within the limitation of this study, as the degree of conversion of the adhesive systems containing acidic monomer increased with increasing cure time, the appropriate cure time for those self-etching adhesives should be evaluated with further study.

Ⅴ. CONCLUSION

This study investigated the hypothesis that increasing light-curing time would leave the oxygen-inhibited layer of the adhesive thinner, and in turn, result in lower shear bond strength than those obtained by the routine curing procedures. The effect of increased light curing time on the thickness of oxygen-inhibited layers and degree of conversion were also investigated.

According to this study, the results were as follows:

1. The shear bond strength of All Bond 2® cured for 10 s or 20 s were significantly higher than those cured for 30 s or 60 s ($p < 0.05$). However, there were no significant difference in the shear bond strength of One-step® and Adper Prompt® with increasing cure time ($p > 0.05$).
2. The relative thicknesses of the oxygen-inhibited layer in each adhesive were not affected by the cure time ($p > 0.05$).
3. Although the degree of conversion of All-Bond 2® were statistically not different with increasing cure time ($p > 0.05$), those of One-Step® and Adper Prompt® showed an increasing trends with increasing cure time ($p < 0.05$).

It was shown that increasing light-curing time did not affect on the relative thickness of the oxygen-inhibited layer of the adhesives, and in turn, on the shear bond strength to dentin.

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국문초록

광조사 시간이 접착제의 표면 미중합층의 두께와 전단접착강도에 미치는 영향에 관한 연구

최용훈[1] · 배지현[1] · 손호현[1] · 이인복[1] · 백승호[1] · 엄정문[1] · 김오영[2] · 김창근[3] · 조병훈[1*]
[1]서울대학교 치과대학 치과보존학교실, 서울대학교 치학연구소, [2]단국대학교 고분자공학과, [3]중앙대학교 화학공학과

본 연구는 광중합형 상아질 접착제에서 광조사 시간의 증가가 접착제 표면의 미중합층에 대한 영향과 그에 따른 전단접착강도에 대해 연구하고자 120개의 치아를 아크릴 몰드에 식립한 후 상아질이 노출되도록 연마하였다. 3 종류의 접착제 〔All Bond2 (AB2), One-Step (OS) and Adper Prompt (AP)〕를 40개 치아에 제조사의 지시대로 도포한 후 각각 다른 광조사 시간 (10, 20, 30 and 60 sec)동안 광조사 하고 복합레진을 접착한 24시간 후 전단접착강도를 측정하였다. 미중합층의 두께와 중합률은 슬라이드 글라스와 FT-NIR을 이용하여 FT-NIR spectrum에서의 peak height를 비교 측정하여 다음과 같은 결론을 얻었다.

1. 전단접착강도에서 AB2는 20초 이후 감소하고, AP는 30초까지 증가하였으며, OS는 차이를 보이지 않았다.
2. 미중합층 두께는 3가지 접착제 모두 유의성 있는 차이를 보이지 않았다.
3. 중합률에서 OS는 10초와 나머지군 사이에 유의성 있는 차이를, AP는 60초에서 유의성 있게 증가되고, AB2의 경우 차이를 나타내지 않았다. 〔J Kor Acad Cons Dent 29(2):177-184, 2004〕

주요어 : 표면 미중합층, 전단접착강도, FT-NIR, 광조사 시간