ORIGINAL RESEARCH

Effect of Simulated Pulpal Pressure on Knoop Hardness of Two Self-etch Adhesives with Different Aggressiveness

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Abstract

Aim: Simulated pulpal pressure (PP) has been shown to affect the bond strength and nanoleakage of different adhesives at dentin interfaces but the effect of simulated PP on polymerization of adhesives has not been studied yet. Furthermore, it has been proposed that strong and mild self-etch adhesives have different polymerization behaviors. This study aimed to evaluate the effect of simulated PP on polymerization of two self-etch adhesives, Adper Prompt L-Pop (APLP) and Adper Easy Bond (AEB), by means of the Knoop hardness test.

Materials and methods: Sixty caries-free human molars were used to prepare deep dentin specimens with a mean remaining dentin thickness of 0.9 mm. The specimens were bonded in the absence or presence of PP. The specimens were assigned to four equal groups ($n = 15$) as follows: AEB/-PP, APLP/-PP, AEB/+PP, and APLP/+PP. Bonding procedures were completed; then the specimens' hardness was measured with the Knoop test. Data were analyzed with two-way ANOVA and the *t* test.

Results: In the absence of PP, the hardness of AEB was significantly higher than that of APLP ($p < 0.001$). In contrast, when PP was simulated, the hardness of APLP was higher than that of AEB ($p = 0.002$). The hardness of AEB was not influenced by the presence of PP ($p = 0.153$). Simulation of PP resulted in a significant improvement in the hardness of APLP ($p < 0.001$).

Conclusion: The polymerization degree of strong self-etch adhesive was lower compared to mild self-etch adhesive. In the presence of hydrostatic PP, the polymerization degree of strong self-etch adhesive was higher than mild self-etch adhesive.

Clinical significance: Dentin moisture caused by positive PP might improve polymerization of strong self-etch adhesives.

Keywords: Knoop hardness, Polymerization, Pulpal pressure, Self-etch adhesives.

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Introduction

Dentin bonding has not been a reliable procedure so far. High organic content, outward flow of fluids under continuous pulpal pressure (PP), and presence of the smear layer are responsible for this problem. Outward movement of tubular fluid might be induced by any change in the osmotic gradient. Removal of the smear layer by acid conditioning induces fluid flow, especially in deep dentin areas.[1] Some of the all-in-one adhesives have a comparatively mild etching effect that might modify and partially remove the smear layer. This effect could lead to a decrease in outward fluid flow during resin application. However, hypertonicity of the adhesive solution or solvent evaporation by air blasting might provoke fluid flow.[2] Etch-and-rinse adhesives are more sensitive to humidity of the dentin but self-etch adhesives are more compatible with the hydrophilic nature of the dentin substrate because of their hydrophilicity.[3]

Apart from dentin internal wetness originating from tubular fluid, external sources of water are also available, which can affect the bonding procedure. Water is contained in the adhesive mixture as a solvent. In self-etch adhesives, water is an essential component as an ionization medium for functional monomers. Phosphoric acid ester (PAE) such as bis[2-(methacryloxy)ethyl] phosphate (2-MP) has been widely used in the formulation of commercial self-etch adhesives. This can ionize in the presence of water and react with tooth hydroxyapatite (HAP) to form complex structures.[4,5] Contemporary self-etch adhesives contain high amounts of hydrophilic monomers. This property improves their bonding to the water-wet dentin. As the dentinal substrate gets closer to the pulp and dentin humidity increases, the role of these monomers becomes crucial. Volatile solvents (i.e., ethanol and acetone) and air blasting help in the displacement of water from the adhesive layer.[6] However, high amounts of hydrophilic monomers and solvents contribute to high permeability and phase separation. A high content of hydrophilic monomers results in increased water uptake from the underlying wet dentin, leading to adhesive plasticization. Residual water in the adhesive layer would interfere with adhesive polymerization. Areas of incomplete monomer conversion behave as permeable membranes and adsorb water, leading to nanoleakage.[7,8] Optimal monomer conversion in the adhesive layer provides higher bond strength, adhesive stability, and good mechanical properties, whereas increased water uptake, uncured monomer elution, high

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susceptibility to degradation, lower mechanical properties, and continuous etching are associated with low degree of conversion.[9] One study showed that enamel bond strength was not influenced by polymerization of the adhesive but dentin bond strength decreased significantly in the case of deficient polymerization.[10]

One study tested the bond strength of self-etch adhesives under simulated pulpal pressure (SPP) and recommended the use of etch-and-rinse three-step adhesives instead of these simplified adhesives in deep dentin areas.[11] However, another study reported less nanoleakage in the deep dentin for the self-etch approach compared to the etch-and-rinse technique.[12] Simulated pulpal pressure (SPP) has been shown to affect the bond strength and nanoleakage of different adhesives at dentin interfaces.[11] Water seepage through the smear layer on to the dentin surface is increased with hydrostatic PP during adhesive application. As mentioned above, increased moisture plasticizes the polymer chains and reduces monomer conversion. This can cause bond degradation and reduced bond durability.[13] The effect of hydrostatic PP is so remarkable that many studies have applied tubule occluding agents to optimize bonding.[14]

To our knowledge, the effect of SPP on polymerization of adhesives has not been studied yet. Due to the importance of polymerization of an adhesive in its performance, we investigated the polymerization behaviors of two self-etch adhesives [Adper Easy Bond (AEB) and Adper Prompt L-Pop (APLP)] with different strengths on the superficial and deep dentin in a parallel study. That study focused on the availability of adequate HAP to react with and neutralize the adhesives in different dentin substrates. The present study aimed to investigate the effect of SPP on polymerization of the same adhesives by means of the Knoop hardness test. The hypotheses to be tested were that polymerization of the two adhesives would not be different, and the presence of SPP would not affect polymerization of the adhesives.

Materials and Methods

Sixty human third molars (from subjects 18–30 years of age) were used in this study. The teeth were sound, caries-free, and without any restorations. The study protocol was approved by the University Ethics Committee. The teeth were stored in 0.5% chloramine solution at 4°C and used within 1 month of extraction.

Specimen Preparation

Two cuts paralleled with the occlusal surface of the teeth were made with a diamond saw (Isomet; Buehler, Lake Bluff, IL, USA). The first cut removed the root 2 mm below the cementoenamel junction (CEJ) and the second removed the crowns 2 mm above the CEJ. Then the occlusal surface of the specimens was wet-ground with 400-grit silicon carbide papers to achieve a remaining dentin thickness of 0.9 ± 0.1 mm. The thickness of the remaining dentin was measured manually with a dental caliper (Stainless Steel, Iwanson Caliper 0–10 mm, Neuhausen, Germany) in areas corresponding to the highest pulp horn. After flattening all the specimens, their surfaces were further abraded with 600-grit silicon carbide paper to create a uniform smear layer.[13] In the last stage when the appropriate depth of the dentin was achieved, the pulpal tissue was removed with an excavator, avoiding contact with the pulp chamber walls.

Experimental Groups

The specimens were randomly divided into four equal groups ($n = 15$) as follows:

APLP/−PP: Adper Prompt L-Pop was applied, without pulp pressure simulation.

APLP/+PP: Adper Prompt L-Pop was applied, under simulated pulp pressure.

APLP/−PP: Adper Easy Bond was applied, without pulp pressure simulation.

AEB/+PP: Adper Easy Bond was applied, under simulated pulp pressure.

Pulpal Pressure Simulation

The crown segments were fixed, using cyanoacrylate glue, to a Plexiglas plate. The glass was perforated with an 18-gauge stainless steel tube. The tube was connected to a water column through a thin plastic tube that permitted communication between the pulp chamber and the hydraulic pressure device. The hydraulic pressure device was filled with water to reproduce a pressure of 15 cm H_2O at the bonded dentin surface. The specimens were connected to this device during the application and curing of adhesives.[15]

Bonding Procedures

The compositions of the adhesives and application methods are summarized in Table 1. The surfaces were checked to ensure uniform distribution of the adhesives and a glass cover slip was placed on the top of the adhesive layer to create a flat surface and avoid contact with the atmospheric oxygen during light activation. The output intensity of LED curing light (Blue LEX 1200W, MONITEX, San-Chong City, Taipei, Taiwan) was measured with a radiometer before curing the specimens and rechecked every 10 specimens. The light intensity was 1000 mW/cm^2. After the bonding procedures were completed, the specimens were kept in a dry dark container at 37°C and immediately subjected to hardness testing.

Hardness Measurement

The hardness of the specimens was measured with a hardness tester (Shimadzu Corporation, Kyoto, Japan), equipped with

Table 1: The chemical compositions of the adhesives used in this study

Adhesive	*Composition*	*Lot number*	*Application mode*
Adper prompt L-Pop(3M ESPE, Neuss, Germany)	Methacrylated phosphoric esters, bis-GMA, 2-hydroxy-ethyl methaKrylate (HEMA), polyalkenoic acid, camphorqui-none, stabilizers, water	602032	Rubbing for 15 s Gentle air-drying Applying second coat and gentle air-drying Light-curing for 10 seconds
Adper easy bond (3M ESPE, Neuss, Germany)	Methacrylated phosphoric esters, bis-GMA, 2-hydroxy-ethyl methacrylate (HEMA), polyal-kenoic acid, 1,6 hexanedioldimeth-acrylate, silica filler, camphorquinone, stabilizers, water, ethanol	590946	Rubbing for 20 seconds Gentle air-drying Light-curing for 10 seconds

Knoop indenter at 0.245 N load and 10 seconds of dwell time. Six indentations were made on the top of the adhesive-bonded dentin surfaces on sites near the pulp horns. The Knoop hardness was determined by examining the surface under a light microscope at ×40 and expressed as the Knoop hardness number (KHN) (Fig. 1).

The Knoop hardness data were statistically analyzed with two-way ANOVA. Pulpal pressure and adhesive were the main factors. The *t* test was used for two-by-two comparisons of the groups. Statistical significance was set at $\alpha = 0.05$.

Results

Means and standard deviations are presented in Table 2. Two-way ANOVA revealed significant interaction between PP and adhesive type ($p < 0.001$). The effect of PP on Knoop hardness was significant ($p < 0.001$). However, the effect of adhesive type on hardness was not significant ($p = 0.96$). The results of the *t* test were as follows:

- The hardness of AEB was not influenced by the presence of PP ($p = 0.153$).
- Simulation of PP resulted in a significant improvement in the hardness of APLP ($p < 0.001$).
- In the absence of PP, the hardness of AEB was significantly higher than that of APLP ($p < 0.001$).
- When PP was simulated, the hardness of APLP was higher than that of AEB ($p = 0.002$).

Discussion

Simplified self-etch adhesives containing high concentrations of 2-hydroxyethyl methacrylate (HEMA) and other hydrophilic monomers exhibited some deficiencies in polymerization. Formation of an HEMA-rich oxygen-inhibited layer might induce water movement as a result of osmotic gradient between this layer and the underlying hydrated dentin.[16] The interaction of mild self-etch adhesive with dentin is limited to only few microns and dentin tubules might remain plugged in.[11] Therefore during adhesive application, fluid movement might be reduced. However, composition of the adhesive might provoke fluid movement.[17] Strong self-etch adhesives with vigorous interaction patterns are more like etch-and-rinse adhesives.[18] This can induce more fluid movement. The first hypothesis was rejected because in the absence of PP the hardness of AEB was higher than that of APLP, which can be translated into higher DC of AEB. In contrast, when PP was simulated the hardness of APLP was higher than that of AEB. Although both adhesives consist of similar resins, the contribution of each component in the adhesive formula is different. Both adhesives are mainly composed of 2-MP, HEMA, and bisphenol A-Bis glycrolyl methacrylate (Bis-GAM).[19] Differences in the monomer ratio would alter adhesive reactivity and initial viscosity, which in turn would alter its polymerization behavior.[20] Considering the pH of the adhesives, 2-MP content of APLP might be higher than that of AEB.[21] The higher 2-MP content might result in increased initial viscosity.[21] Adper Easy Bond contains higher amount of Bis-GMA. Bis-GMA is a viscous molecule with high cross-linking affinity, which can enhance degree of polymerization and mechanical properties.[22] Another compositional difference of AEB and APLP is the presence of silica fillers in AEB. Filled adhesives create strong and thick layers.[4]

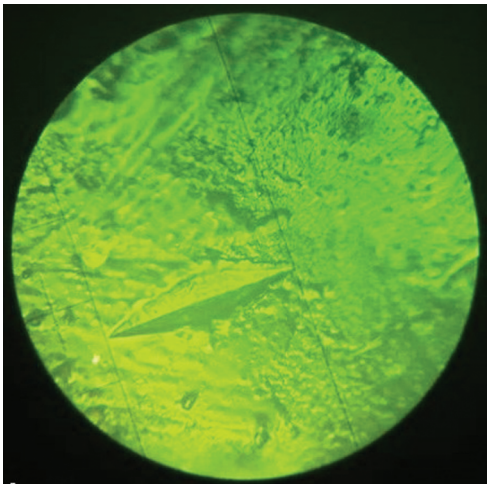

Fig. 1: Demonstrates the indentation marks with 80× magnification created by the Knoop hardness test on the adhesive layer. The dimensions of these indentations were used to calculate the Knoop hardness number

Table 2: Mean Knoop hardness numbers ± standard deviations (min/max) of the study groups and statistical significance

Dentin bonding agent	*PP*		*p value*	*E.F*
	−	+		
AEB	12.5 ± 1.2(10.1/14.3)$^{A, a}$	11.3 ± 2.9(8.2/17.5)$^{A, c}$	0.153	0.054
APLP	9.3 ± 1.1 (8/10.8)$^{B, b}$	14.5 ± 2.2 (11.1/18.5)$^{C, d}$	<0.001	3.06
p value	<0.001	<0.002	–	
E.F	2.78	1.26	–	–

*AEB, Adper easy bond; APLP, Adper prompt L-Pop; −PP, without pulpal pressure; +PP, with pulpal pressure; E.F, Cohen's d effect size

Same uppercase letters represent no statistically significant difference in the row. Same lowercase letters represent no statistically significant difference in the column

Simulated pulpal pressure caused higher polymerization degree for APLP, but it did not exert any effect on polymerization of AEB. Therefore, the second hypothesis was accepted in part. Adper Easy Bond with a pH of ~2.0 might not be strong enough to totally remove the smear layer. This could explain why a positive PP had no effect on the polymerization of this adhesive. In partial accordance with this finding, PP did not affect the bond strength of mild self-etch adhesives but reduced the bond strength of strong self-etch adhesives.[23] Polymerization of AEB has been shown to be independent of the type of substrate and mode of application.[24] Adper Easy Bond-bonded specimens showed high bond strength and stability when stored under SPP[25] but in those studies PP was not simulated during application of the adhesive.

In contrast to the present study, Moll et al. reported decreased bond strength for APLP in the presence of PP.[26] Another study reported better dentin sealing with APLP compared to an etch-and-rinse adhesive under PP.[27] Adper Prompt L-Pop was applied in two layers, which might have reduced adhesive permeability compared to a single layer of AEB. Bilayer application of the adhesive improves resin infiltration into the hybrid layer and polymerization by sealing the oxygen-inhibited layer.[28]

Several studies have confirmed that acid-base reaction of acidic functional monomers of strong self-etch adhesives with amines of CQ-based photoinitiator systems could compromise

the polymerization of the adhesive. Acidic monomers of APLP have extensive capacity to be neutralized. Chemical reaction with HAP of the tooth substrate buffers these monomers; thus, more amines survive to accelerate photopolymerization. Another component required for this interaction is water. This aqueous medium can be supplied from two sources. First, the intrinsic water from adhesive content; second, water provided by dentin moisture in the presence of PP. Dentinal fluid was considered as a factor interfering in polymerization of the adhesive but in the case of active application dentin moisture might intermix homogeneously with the hydrophilic adhesive APLP. In our study, additional supply of water from tubular fluid might have provided an extra medium for acidic monomers to interact with HAP and their neutralization. Consumption of these monomers might prohibit the possibility of interference with polymerization reaction.

The AEB and APLP contain 10% water. It was shown that more water incorporation up to 20% in 2-MP-based self-etch adhesives in the presence of HAP more negatively influenced polymerization of the mixture with lower 2-MP content and higher pH.[21] A previous study showed the adverse effect of the water content, when it was more than 10%, on polymerization of a model adhesive applied on a glass.[29] However, this effect decreased when the adhesive was applied on dentin. Wang et al.[30] reported that increasing the water content from 20 to 60% caused a considerable reduction in polymerization of APLP. They only used components of one of the reservoirs of APLP, which consisted of methacrylate phosphate, initiators, and stabilizers. Also they applied the mixture on a glass slide.

Another study reported higher shear bond strength for APLP when applied on the dry dentin compared to the wet dentin surface. It was concluded that dentin surface moisture diluted the adhesive and interfered with its etching capacity.[1] It has been claimed that viscosity is one of the factors affecting adhesive polymerization.[31] According to the gel effect or the Trommsdrof-Norrish phenomenon, polymerization rate increases at high viscosity. Therefore, excess water can reduce the rate of polymerization.[31] However, the internal dentin moisture caused by PP is limited within dentinal tubules. While the dentin surface is covered with the smear layer, as APLP etches through the smear layer, it penetrates into the intertubular and peritubular dentin[29] and the tubular fluid comes into contact with the adhesive additional supply of water for the acid-base reaction. Therefore, the internal and external dentin moisture might have distinctive effects on adhesive properties.

One study reported penetration of acidic monomers of APLP deep into dentinal tubules. Although the resin near the adhesive/dentin interface was polymerized to about 93% as a result of interaction with dentin minerals, this was not the case in water-filled dentinal tubules. Unpolymerized acidic monomers continued to demineralize the peritubular dentin.[30] Positive PP that creates continuous outward movement of the tubular fluid was not taken into account in that study. It might limit the penetration of monomers and attenuate the undesirable continuous etching.

The influence of hydrostatic PP on dentin bonding and adhesive layer is so remarkable that several studies have attempted to simulate PP. Water seepage under PP contributes to adhesive plasticization, polymer degradation, and difficulties in dentin sealing.[11,13,15,23] Therefore, by simulating PP an *in vitro* study more readily approximates the clinical situation.

The samples were tested for hardness within 15 minutes after light-curing and during this period they were kept in a dark container. Therefore, only the light-activated polymerization was taken into account in this study. While preparation configuration causes attenuation of light irradiance in deep dentin areas, the role of HAP-triggered chemically activated polymerization is more significant and is recommended to be studied in future. The curing time was 10 minutes based on manufacturer's instructions. Feitosa et al. showed that the detrimental effect of PP on AEB could be eliminated by extending the curing time.[32]

Appropriate selection of the photoinitiator system affects the adhesive strength and stability. Diphenyl (2,4,6-trimethyl benzoyl) phosphine oxide can be used instead of CQ-amine because of compatibility with water and acidic nature of self-etch adhesives.[33]

Despite its simple nature, the hardness test can detect small changes in polymer cross-linking[34] but for more precise information about double bond conversion Raman spectroscopy is recommended for future investigations. Different macromolecules and ions are contained in pulpal fluid. Therefore, using water to simulate pulpal fluid was another limitation of the present study.

Conclusion

Within the limitations of the present study, it can be concluded that in the absence of PP hardness of AEB was significantly higher than APLP, and when PP was simulated hardness of APLP was higher than that of AEB. Pulpal pressure had no effect on polymerization of AEB. On the other hand, simulation of PP positively influenced polymerization of APLP.

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