Original Article

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Does delayed light activation of resin composite affect dentin bond strength?

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Objective: The objective of this study was to compare the effect of immediate and delayed light activation of resin composite on the microtensile bond strength (μTBS) of self-etch adhesive and universal adhesive in self-etch mode under dynamic pulpal pressure.

Materials and Methods: Thirty dentin discs were prepared from extracted third molar without any pathological conditions. Smear layers were created by coarse diamond bur. The dynamic pulpal pressure model was set at 15 cm H_2O and connected to the tooth specimen. A two-step self-etch adhesive (Clearfil SE Bond) and two universal adhesives applied in self-etch mode (Clearfil Universal Bond Quick and G-Premio BOND) were applied to dentin surfaces according to the manufacturer's instructions. Each adhesive was divided into 2 groups: immediate light activation (IM) and delayed light activation (DL). For IM group, the resin composite was placed over the bonded dentin disc and light activated immediately while the resin composite in DL group was placed and left for 150 sec before light activation. Specimens were stored in distilled water at 37°C for 24 h and subjected to μTBS test using a universal testing machine at crosshead speed of 1 mm/min until failure occurred. The μTBS data were analyzed using two-way ANOVA and Tukey's multiple comparison tests.

Results: Two-way ANOVA showed that the effect of delayed light activation was not significant on the μTBS ($p>0.05$), but the effect of adhesives was significant ($p<0.001$). There was no significant interaction between adhesive and time ($p>0.05$). The highest bond strength was observed in Clearfil SE Bond while no statistically significant difference was detected when compared with G-Premio BOND.

Conclusion: Delayed light activation of resin composite for 150 sec does not affect dentin bond strengths of self-etch adhesive and universal adhesive in self-etch mode.

Keywords: dentin, dynamic pulpal pressure, light activation, microtensile bond strength, resin composite, self-etch adhesive

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Introduction

Currently, the development of dental adhesive materials focuses on the simplification of application procedure to reduce the step of application and technique sensitivity. Single step self-etch adhesives has been developed by incorporating etching, priming and bonding resin into a single solution [1]. However, this dental adhesive has several drawbacks, especially its high hydrophilicity makes it acts as permeable membranes, allowing water movement across the adhesive layer [2-5]. Recently, a new dental adhesive has been introduced and classified as universal or multi-mode adhesive. It has been designed under the all-in-one concept of the already existing one-step self-etch adhesives. Universal adhesives could be used in a multimode approach [1].

Bonding restorative materials to dentin is different from enamel as it has a smear layer, organic contents and presence of fluid inside the dentinal tubules [6]. Dentinal fluid is normally

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a continuous outward flow through the exposed dentin. The flow rate of dentinal fluid through dentin depends on the changing of pulpal pressure and the permeability of dentin [7, 8].

In clinical situations, the light activation of the resin composite is performed after the material is well adapted in the cavity. Nevertheless, in some clinical situations, the light activation might be delayed until proper restoration is achieved [9]. Transudation of fluids across polymerized adhesive bonded with dentin has also been observed when resin composite build-up was delayed [3, 10, 11]. It was demonstrated that delayed light activation of resin composite has negative effect on the bond strength of adhesives. However, the reported drawback was observed when the light activation of resin composite was delayed for 10 and 20 min which might not clinical relevance [3, 4]. Therefore, it is interesting to compare the effect of immediate and 150 sec delayed light activation of resin composite on the microtensile bond strength of self-etch adhesives under dynamic pulpal pressure.

The objective of this study was to compare the effect of immediate and delayed light activation of resin composite on the microtensile bond strength of self-etch adhesives under dynamic pulpal pressure. The null hypothesis tested was, there would be no difference in dentin bond strengths between immediate and delayed light activation of resin composite.

Materials and Methods

Thirty extracted human third molars from the patients between 18 to 30 years old were collected. This study was approved by the Institutional Ethical Review Committee (MU-DT/PY-IRB 2020/054.2710). The teeth were stored in 0.1% thymol solution and were used within 6 months.

The mid-coronal dentin discs were prepared by cutting perpendicular to the long axis of teeth using a slow speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA). The lower cut was done at 2 mm. below the cemento-enamel junction to expose the pulp chambers [12]. Dentin disc was approximately 5 mm. thickness. A pincer-type caliper was used for measuring the remaining dentin thickness (RDT) approximately 0.9 mm. from the surface to the highest pulpal horns [12, 13]. The pulp chamber was irrigated with 2.5% sodium hypochlorite solution (NaOCl) for 30 sec followed by immersion in distilled water for 30 min to neutralize the effects of NaOCl [14, 15].

Adhesives and bonding procedures

Each dentin disc was attached to a plexiglass plate (0.75 x 0.75 x 0.18 inches) that was perforated by an 18-gauge stainless steel tube using cyanoacrylate adhesive. Each specimen was connected to dynamic pulpal pressure device with an infusion pump (ISMATEC®, Wertheim, Germany). The dynamic pulpal pressure device was set up at 15 cm H_2O [16] and the fluid flow was set up at 0.36 µL/min in order to simulate intrapulpal pressure and pulpal blood flow rate [17].

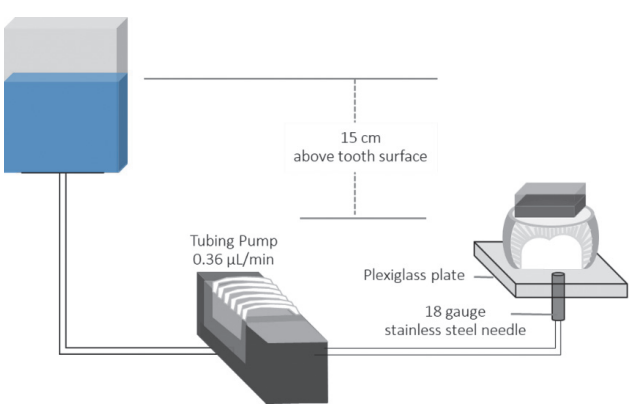

Figure 1 Schematic illustration showing the dynamic pulpal pressure device.

Occlusal dentin surfaces were prepared by coarse diamond bur (Cylindrical-shape, mean abrasive size 20 µm, Jota, Rüthi, Switzerland) using a high-speed with 5 light pressure strokes per tooth to make a uniform surface as described by Saikaew *et al*. A new bur was replaced every 5 surfaces [18]. The pressure used by the same operator was about 100 grams, which was found to be the mean pressure exerted by most clinician at the tip of the bur [19].

The dental adhesives investigated in this study were a two-step self-etch adhesive system (Clearfil SE Bond) and two universal adhesives (Clearfil Universal Bond Quick and G-Premio BOND). The chemical compositions and the instructions of adhesives used in this study are listed in Table 1.

Each adhesive was applied to the dentin surface according to the manufacturer's instruction. Light activation was performed using the LED light curing unit with the power intensity of approximately 1,200 mW/cm^2. (Bluephase G2, Ivoclar Vivadent, Schann, Liechtenstein).

Resin composite bulidups

Each adhesive group was divided into two groups based on the contact time of the resin composite with the bonded dentin surface prior to light activation. For IM group, a layer of resin composite (Clearfil AP-X ES-2), approximately 2 mm in thickness, was placed on the bonded dentin surface within 20 sec and then light activated immediately with the LED light-curing unit for 20 sec Subsequently, the additional resin composite was

Table 1 The chemical compositions and the instructions of adhesives used in the study

Adhesives	Chemical compositions	pH	Application
Clearfil SE Bond (Kuraray Noritake, Niigata, Japan)	Primer: 10-MDP, HEMA, Hydrophilic dimethacrylate, Di-camphorquinone, N,N-diethanol-p-toluine, water Bond: 10-MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, di-camphorquinone, N,N-diethanol-p-toluine, silanated colloidal silica	2.0	1. Apply primer and leave for 20 s 2. Dry with mild air flow 3. Apply bonding 4. Air flow gently 5. Light cure for 10 s
Clearfil Universal Bond Quick (Kuraray Noritake, Niigata, Japan)	Bis-GMA, ethanol, HEMA, 10-MDP, hydrophilic amide monomer, colloidal silica, silane coupling agent, sodium fluoride, camphorquinone, ethanol, water	2.3	1. Apply in rubbing motion for 5 s 2. No waiting time after application 3. Dry with mild pressure air blow for 5 s. 4. Light cure for 10 s.
G-Premio BOND (GC Corporation, Tokyo, Japan)	10-MDP, 4-META, 10-methacryoyloxydecyl dihydrogen thiophosphate, methacrylate adic ester, distilled water, acetone, photo initiators, silica fine powder	1.5	1. Apply and wait for 10 s after application 2. Dry thoroughly for 5 s with maximum air pressure 3. Light cure for 10 s.

The chemical compositions and pH were obtained from references [18, 20]

placed on first layer and cured for 20 sec In DL group, the first layer of resin composite was placed and left for 150 sec before light activation. Then, the resin composite build-up was performed in the same manner as the IM group.

Microtensile bond strength test (μTBS)

After storage in 37°C water for 24 h, each bonded specimen was sectioned into beams (cross-sectional area of about 1 mm^2) with non-trimming technique using a low-speed cutting machine at a speed of 350 rpm and loading of 150 gm with constant coolant water. Central part of the resin bonded teeth was used for the μTBS test.

The beams were fixed on an experimental jig for microtensile testing with cyanoacrylate glue (Model Repair 2 Blue, Dentsply-Sankin, Otahara, Japan). The μTBS test was performed using a universal testing machine (LloydTM Testing Machine, Model LR 10K, Lloyd Instruments, FarehamHanth, UK) with cross-head speed 1.0 mm/min. The data were recorded and expressed in MPa unit.

Statistical Analysis

The bond strength data were organized and analyzed for normal distribution and homogeneity of variance using the Kolmogorov-Smirnov test and Levene's test, respectively. Two-way ANOVA and Tukey's multiple comparison tests were calculated. All analyses were performed using a statistical software system (SPSS 27.0; SPSS Inc, Chicago, IL, USA) at 95% confidence interval.

Results

In this study, no pre-test failure was observed. Two-way ANOVA revealed the statistically significant effects of adhesives (F=16.587, p=<0.001) on the μTBS, but the effect of time was not statistically significant (F=3.358, p=0.079). There was no interaction between adhesive and time (F = 0.001, p = 0.999).

Both immediate and delayed light activation of resin composite groups showed significantly lowest bond strength in the Clearfil Universal Bond Quick while no statistically significant difference was found between Clearfil SE Bond and G-Premio Bond (Table 2).

Discussion

The results of this study showed that delayed light activation of resin composite had no significant effect on dentin bond strength for all adhesive tests. Thus, the null hypothesis was not rejected. In this study, the delayed activation time of resin composite was set at 150 sec, which was expected to be sufficient for most operators to manipulate the resin composite for a single restoration. However,

Table 2 Exhibit the mean microtensile bond strength (μTBS) value ± standard deviations.

Adhesive	Microtensile bond strength (MPa)	
	Immediate light activation	Delayed light activation (150 sec)
Clearfil SE Bond	23.64±2.06^{Aa}	22.08±2.53^{Aa}
Clearfil Universal Bond Quick	17.87±2.39^{Ba}	16.38±1.03^{Ba}
G-Premio Bond	19.81±2.63^{Aa}	18.31±2.53^{Aa}

Different letters demonstrate statistically significant differences (p<0.05). Uppercase letter demonstrates significant difference in column. Lowercase letter demonstrates significant difference in row

this observation is in contrast with previous studies which reported a potential drop in bond strength on prolonged contact of single-step adhesives when light activation of resin composite was delayed [3, 4]. It was reported that the cured adhesive layer in single-step adhesives acted as semi-permeable membranes allowing water to infiltrate into the surface between the cured adhesive and the uncured [2-5]. Therefore, the compromised bond strength in single-step adhesives was observed. The different results between the current study and previous studies could be explained by the different delayed activation time and the adhesives used. Firstly, previous studies were performed at 10 min and 20 min delayed activation times, where more water could be permeated through the cured adhesive layer due to longer delayed activation time and resulted in lower bond strength. Secondly, the adhesives used in previous studies were all-in-one self-etch adhesive, whereas, in this study, the latest generation or universal adhesives were selected. The concentrations of hydrophilic monomer and solvent in universal adhesives were lower compared to that of the previous generation [21-24]. In addition, most universal adhesives contain 10-MDP as a functional monomer, while it has been reported that 10-MDP is relatively hydrophobic due to the long carbon chain [25].

Therefore, more hydrophobic composition of adhesive combined with shorter delayed activation time could be the reasons for similar bond strength between IM and DL observed in this study. However, it is interesting to evaluate at a longer delayed activation time in the case where a clinician needs to restore multiple restorations.

In this study, Clearfil SE Bond was used as a control and it demonstrated the highest bond strength. Clearfil SE bond is two-step self-etch adhesive and it has been recommended as the gold standard dentin-bonding agent. Self-etch primer and a hydrophobic bonding resin presented in two separate bottles demonstrated reliable results in terms of dentin adhesion durability and reducing primer's hydrophilicity [13, 26, 27].

The bond strength of G-Premio Bond was lower than Clearfil SE Bond, but the difference in bond strengths was not significant. This could be explained by the effect of HEMA, and in fact, it has been commonly incorporated in adhesive systems. Due to its low molecular weight, HEMA acts as a co-solvent, minimizing phase separation and wetting of the dentin surface. However, HEMA promotes poly-HEMA hydrogels with residual water on the adhesive interface and diffused water from dentinal tubules. These poly-HEMA hydrogels within the adhesive resin permit water movement across the adhesive layer [28, 29].

Several studies have reported that HEMA-containing adhesives exhibit higher water sorption and show an accelerated reduction in the mechanical properties of the adhesive layer during water-storage when compared with HEMA-free adhesives [29-31]. From the composition of adhesive (Table 1), G-Premio Bond is HEMA-free adhesive. Therefore, this might explain why G-Premio Bond demonstrated similar bond strength to Clearfil SE bond in this study.

Clearfil Universal Bond Quick showed the lowest μTBS, which could be explained by the shorter application time combined with the higher pH of adhesive. Clearfil Universal Bond Quick, as introduced by the manufacturer, contains a multifunctional hydrophilic amide monomer which may enhance wetting of the dentin surface when the monomer was used in short application time [32]. Therefore, the application time at 5 s was recommended from the manufacturer. In this study, the application time of Clearfil Universal Bond Quick was shorter than those of Clearfil SE bond and G-Premio Bond. Previously, there had been a report that a shorter application time led to insufficient smear layer removal and infiltration of resin monomers [18]. In addition, the pH of

Clearfil Universal Bond Quick was the highest among all the tested adhesives (Table 1). It is possible that the demineralization of the smear layer and resin penetration of Clearfil Universal Bond Quick might be insufficient and, therefore, resulting in a poor performance [33]. These might explain the lowest bond strength when Clearfil Universal Bond Quick was applied.

Conclusion

Dentin bond strengths of self-etch adhesive and universal adhesive in self-etch mode to dentin were not affected when light activation of resin composite was delayed for 150 sec.

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