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Effect of smear layer pretreatment with chemical methods on bond strength of universal adhesives

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Objective: The purpose of this study was to evaluate the effect of smear layer pretreatment with either 2.5% sodium hypochlorite solution followed by p-toluenesulfinic acid sodium salt (Accel) or 17% ethylenediaminetetraacetic acid (EDTA) solution on microtensile bond strength (μTBS) of two universal adhesives.

Materials and Methods: Forty-eight extracted third molars were used in this study. The crown of the tooth was sectioned to exposed dentin. Smear layer was created by grinding the exposed surfaces with medium grit diamond bur using a high speed aerotor under water coolant. Smear layer pretreatments were performed in different experimental groups: No treatment (G1), etching with phosphoric acid for 15 s (G2), agitation with 17%EDTA solution for 30 s (G3), and agitation with 2.5% sodium hypochlorite solution for 60 s followed by Accel application for 5 s (G4). The modified dentin surfaces were bonded with either All-Bond Universal (ABU) or Clearfil Universal Bond Quick (CUQ) and then restored with Clearfil AP-X ES-2 composite. The bonded specimens with different smear layer pretreatment and universal adhesives were subjected to μTBS test. Data were analyzed using two-way ANOVA and Duncan multiple comparison tests at the 95% significance level.

Results: Both adhesive systems and surface treatments had influence on microtensile bond strength. For ABU, the μTBS values were as followed G2>G3, G4>G1, respectively. Whereas the data for CUQ were G3,G4>G1,G2, respectively.

Conclusion: Smear layer pretreatment with 17%EDTA agitation for 30 s or 2.5% NaOCl agitation for 60 s followed by Accel application improved the μTBS for both universal adhesives.

Keywords: bond strength, smear layer, universal adhesive

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Introduction

Nowadays, direct restorative dental treatment with resin composite is very common in daily practice. The success of direct composite restoration relied on dental adhesive systems. With continued advancement of adhesive, the latest generation called universal adhesive has been launched since 2011 [1]. This adhesive system can be used in multimode as etch-and-rinse, self-etch or selective etching mode [2]. Moreover, some manufacturers claim that universal adhesive can be bonded with many restorative materials such as metal and ceramic [3]. Due to these versatilities, ease of use and promise efficiency, universal adhesive has increased in popularity.

Using universal adhesive, especially in self-etch mode, has a point of concern with a smear layer. Smear layer is a layer of debris covering the tooth surface after tooth preparation. The characteristic of the smear layer is different depending on the method of creation. For instance, bur cut smear layer, which is clinically relevant, has been reported that could compromise efficiency of self-etch adhesive [4-5]. It was reported that no difference in bonding performance was observed when the universal adhesives were used either in etch-and-rinse or self-etch mode [6]. The adhesive

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application in etch-and-rinse mode was beneficial in All-Bond Universal [6]. However, the aggressive demineralization of phosphoric acid could cause an incomplete resin infiltration and lead to the degradation of hybrid layer [7].

Smear layer pretreatment prior to application of self-etch adhesive has been reported to improve the bonding performance [8]. Sodium hypochlorite (NaOCl) and EDTA are routine irrigating solutions for endodontic treatment that target on the organic component and inorganic component of the smear layer, respectively. For NaOCl treated surface, the compromised bond strength of adhesives by the residual free radical has been reported. Therefore, it was recommended to neutralize the negative effect of NaOCl by using P-toluenesulfinic acid sodium salt (Accel) [9].

With the lack of studies in smear layer pretreatment with these solutions prior to applying the universal adhesive, it would be interesting to evaluate their effect on bonding performance. Therefore, the purpose of this study was to evaluate the effect of different smear layer pretreatments on microtensile bond strength of universal adhesives. The first null hypothesis was that there was no difference in microtensile bond strength with different methods. The second null hypothesis was that there was no difference in microtensile bond strength between two adhesive systems.

Materials and Methods

Specimens preparation

Forty-eight extracted human third molars with sound condition were collected and used within six months. The teeth were stored in 0.1% Thymol solution (M Dent, Bangkok, Thailand). The protocol of this study was approved by the Faculty of Dentistry/Faculty of Pharmacy, Mahidol University Institutional Review Board, Thailand (IRB 2021/002.0302).

Occlusal enamel of extracted tooth was removed by a model trimmer. Smear layer was then created by grinding on a dentin surface for 5 strokes with medium grit diamond bur (Jota, Rüthi, Switzerland) using high speed airotor (TwinPower Turbine, J Morita, Osaka, Japan) under copious water spray.

Experimental Design

Prepared dentin surfaces were divided into 4 groups according to 4 smear layer treatment methods: no treatment, thoroughly air dried (G1), etching with 37% phosphoric acid for 15 s, rinsed off 30 s and air dried to achieve moist dentin (G2), agitate with 17% EDTA for 30 s, rinsed off 30 s and thoroughly air dried (G3) and agitate with 2.5% NaOCl for 60 s, rinsed off 30 s, Accel application for 5 s and thoroughly air dried (G4). Then, the specimens from each smear layer pretreated surface were further subdivided into two groups for bonding with either All-Bond Universal (ABU) or Clearfil Universal Bond Quick (CUQ). The composition and instruction for use of two adhesives are shown in Table 1

Bonding and resin composite built-up

After adhesive application as indicated in Table 1, the LED light-curing unit (LED Bluephase; IvoclarVivadent, Schaan, Liechtenstein) with the power intensity of approximately 1,200 mW/cm^2 was used for curing the adhesive according to the manufacturer's recommendation. After light curing, resin composite Clearfil AP-X ES-2 (Kuraray Noritake Dental, Niigata, Japan) was placed onto the bonded dentin surface for two increments of 2 mm and cured by using the LED light-curing unit for 20 s each layer.

Table 1 Chemical composition, pH and application of universal adhesives in this study

Adhesives	Chemical composition	pH value	Application
All-Bond Universal (ABU) Bisco, Schaumburg, USA.	10-MDP, phosphoric acid ester monomer, Bis-GMA, HEMA, ethanol, water, initiators	3.2	Applied 2 separate coats of adhesive with rubbing 15 s per coat, dried with air blow and then light-cured with the curing unit for 10 s
Clearfil Universal Bond Quick (CUQ) Kuraray Noritake Dental, Niigata, Japan.	Bis-GMA, HEMA, 10-MDP, Hydrophilic amide monomer, Ethanol, Water, Camphorquinone, colloidal silica, Silane coupling agent, sodium fluoride	2.3	Applied the adhesive with rubbing for 5 s, dried with air blow and then light-cured with the curing unit for 10 s.

10-MDP: 10-Methacryloyloxydecyl dihydrogen phosphate; Bis-GMA: bisphenol A-glycidyl methacrylate; HEMA: hydroxyethylmethacrylate

All bonded specimens were stored in distilled water at 37°C for 24 hours. After that, the central part of resin bonded teeth was subjected to microtensile bond strength test (μTBS test). Six resin-dentin beams from the central part of each bonded specimen were prepared into approximately 1.0x1.0 mm^2 with non-trimming technique using a low-speed cutting machine (Isomet, Buehler Ltd, Lake Bluff, IL, USA) at a speed of 350 rpm and loading of 150 gm with constant water coolant.

Microtensile bond strength test

For evaluation of μTBS, specimens were fixed on an experimental jig for microtensile testing using a cyanoacrylate glue (Model Repair II Blue, Dentsply, SANKIN, Tokyo, Japan) [10,11]. Then, the μTBS test was performed using a universal testing machine (LloydTM Testing Machine, Model LR 10K, Lloyd Instruments, FarehamHanth, UK) with crosshead speed 1.0 mm/min. The data was recorded and expressed into MPa. Mean value of each tooth was calculated and used in statistical analysis.

Statistical analysis

The bond strength data were organized and analyzed for normal distribution with Kolmogorov-Smirnov test. Homogeneity of variance was analyzed with Levene's test. Two-way ANOVA and Duncan 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

The μTBS values and standard deviations of all experimental groups are shown in Table 2. Two-way ANOVA revealed significant effects of adhesive (p=0.001, F=12.945) and chemical treatment (p<0.001, F=7.560). The interaction between two factors was also significant (p<0.001, F=9.895). In G2 and G3, ABU demonstrated significantly higher bond strength than those of CUQ. The highest bond strength of ABU was observed when applied with etch-and-rinse mode. In case of CUQ, pretreatment with 17%EDTA and

2.5% NaOCl with Accel demonstrated the higher bonding performance than when applied with self-etch or etch-and-rinse mode.

Discussion

According to different adhesive systems, μTBS values were different with different smear layer pretreatments. Most universal adhesives demonstrated similar bond strength when applied in either self-etch mode or etch-and-rinse mode. Except ABU, bond strength in self-etch mode was reported to be inferior to etch-and-rinse due to its weak acidity [6]. For CUQ, using this adhesive in different etching modes didn't affect μTBS significantly. This is in agreement with Ahmed that CUQ has similar bonding performance with different etching modes [12]. The bond strength of tested universal adhesives in self-etch mode were similar. This could be explained by pH of adhesive and application technique. With higher pH of ABU (Table 1), the demineralization effect might be low with the limited resin penetration. The bond strength of ABU was expected to be lower than that of CUQ. However, the double application indicated by the manufacturer could improve the etching effect of ABU [13]. Therefore, the bond strength of ABU and CUQ in self-etch mode were similar.

From the result of this study, smear layer pretreatment can improve the μTBS of universal adhesives in self-etch mode. From our pilot SEM images, the bur cut smear layer was partially removed by the chemical agents in different targets. EDTA is a chelating agent that removes inorganic minerals from the smear layer and underlying dentin [14]. On the other hand, NaOCl has proteolytic activity that dissolves organic debris [9]. From these mechanisms, both smear layer pretreatment methods may be resulting in significantly improved μTBS of both universal adhesives. For ABU, etching with phosphoric acid demonstrated the highest bond strength. However, incomplete resin infiltration to deep demineralized dentin might be expected [7]. Furthermore, acid conditioning was reported to activate endogenous collagenase enzyme in dentin called MMPs [15]. These processes could lead to the long-term degradation of the resin-dentin bond. Whereas smear layer pretreatment with chemical agents provides some benefit in improving durability of dentin bond [16,17]. Further study should be performed with the long-term storage to evaluate the effect of smear layer pretreatment with the bond durability.

Table 2 Mean microtensile bond strength among four smear layer treatment methods and two adhesive systems

	Microtensile bond strength (MPa) ± standard deviations			
	Self-etch (G1)	Etch-and-Rinse (G2)	17% EDTA (G3)	2.5% NaOCl w/ Accel (G4)
ABU	24.51 ± 2.16 [c]	39.11 ± 5.69 [a]	33.03 ± 5.18 [b]	30.99 ± 5.49 [b]
CUQ	23.16 ± 7.26 [c]	20.98 ± 3.49 [c]	31.89 ± 3.57 [b]	31.52 ± 3.91 [b]

Groups with the same superscripts are not statistically significant ($p>0.05$).

Conclusion

Within limitation of this study, smear layer pretreatment by 17% EDTA and 2.5% NaOCl with Accel could improve microtensile bond strength of universal adhesives.

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