ว ทันต จุฬาฯ 2558;38:21-34
CU Dent J. 2015;38:21-34

Original Article
บทวิทยาการ

Microtensile bond strength of self-adhesive resin composite to dentin

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Abstract

Objective To evaluate the effect of pretreatment and aging process, by means of water storage, on microtensile bond strength (μTBS) of a self-adhesive resin composite to dentin.

Materials and methods 72 extracted human molars were selected. μTBS was evaluated using mid-coronal dentin. According to pretreatment methods, type of materials and water storage time, teeth were randomly divided into 4 groups: control group: dentin pretreatment with Single Bond™ Universal followed by Filtek™ Z350 XT Flowable; group without pretreatment followed by Vertise® Flow™.group with pretreatment with 37.5% phosphoric acid followed by Vertise® Flow™; and group with pretreatment with 37.5% phosphoric acid and Optibond® Solo Plus™ followed by Vertise® Flow™. Each group was further randomly divided into 2 subgroups, total of 8 groups to be tested for 24 hours or 3 months. The specimens were tested for μTBS after water storage, and failure modes were recorded. Data were analyzed using two-way ANOVA and Bonferroni post hoc test ($p = 0.05$).

Results The μTBS revealed the highest in group of dentin pretreatment with 37.5% phosphoric acid and Optibond® Solo Plus™ followed by Vertise® Flow™ at 24 hours (42.63 ± 4.57 MPa) and the lowest in group without pretreatment followed by Vertise® Flow™ at 3 months (23.39 ± 3.88 MPa). Considering the effect of pretreatment, groups with pretreatment showed significantly higher μTBS than groups without pretreatment. In terms of influence of aging process, μTBS were significantly decreased by water storage.

Conclusion Dentin pretreatment and water storage had effect on μTBS of self-adhesive resin composite to dentin.

(CU Dent J. 2015;38:21-34)

Key words: *dentin; microtensile bond strength; self-adhesive resin composite; water storage*

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Introduction

The use of direct resin composite materials has become an integral part of contemporary operative dentistry. Esthetic appearance and constantly improved properties have made these materials the main choice for direct restorations.[1] However, resin composites, in common with the majority of dental materials undergo deterioration and degradation in the oral environment. Moreover, being technique-sensitive materials, failure at the tooth-restoration interface may also occur.[2] As a result, managing of failed restorations is a common problem encountered in daily practice. Presently, many of the researches and innovations in dental materials are focused on simplification of the bonding procedures to eliminate technique-sensitivity and time consumption.[3]

At the moment, resin composite materials that are able to adhere to dentin and enamel without the application of a separate phosphoric-acid etching and adhesive agent, the so-called “self-adhesive resin composite”, have been developed. One of the materials in this group is flowable resin composite, which adheres to tooth substrates, based on bonding technology. One brand incorporates glycerol phosphate dimethacrylate (GPDM) adhesive monomer that acts like a coupling agent. The bonding mechanism has been claimed to be two-fold. Firstly, chemical adhesion is speculated to occur by the phosphate functional group of the GPDM monomer united with the calcium ions within the tooth structure, however not proven. Secondly, through a micromechanical interlocking by etching process as a result of an interpenetrating network formed between the polymerized monomers and collagen fibers of dentin.[1,4]

The bonding performance of self-adhesive resin composite revealed that dentin bond strength of this material was significantly less than that of conventional flowable resin composite used in combination with other self-etch adhesives.[5-7] Self-etch adhesives have been associated with lower bonding effectiveness as compared to total-etch adhesives. These adhesives are usually used according to manufacturers’ recommendations, but some have been found to perform better using modification of application technique, such as surface pretreatment.[8-10] For this reason, further work is required to evaluate the performance regarding adhesion to dentin. Its capacity to increase bond strength with etchant or adhesive agent is still open to question. It raises the subject as how to improve or at least to maintain acceptable bond strengths overtime.

This study investigated the effects of pretreatments and artificial aging, by means of water storage, on microtensile bond strength (μTBS) of one self-adhesive resin composite to dentin. A conventional flowable resin composite in combination with 1-step self-etch adhesive was selected as control material because the first layer of self-adhesive resin composite was acting, in effect, as 1-step self-etch adhesive.[11] The null hypotheses tested was that (1) there was no significant difference in μTBS when using different pretreatments (2) there was no significant difference in μTBS when using different water storage times (24 hours vs. 3 months).

Materials and methods

Seventy-two selected non-carious and non-restored extracted human molars were collected, after informed consent was obtained under a protocol approved by the Ethics Committee of the Faculty of Dentistry, Chulalongkorn University (Study Code: HREC-DCU 2012-049). All selected teeth were debrided and stored in a 0.1% thymol solution at 4°C for up to 1 month after extraction. The roots of teeth were embedded into a dental wax, pink type (Fig. 1A) leaving the clinical crown exposed. The occlusal third of the embedded tooth was removed to expose mid-coronal dentin using low-speed cutting machine (ISOMET® 1000; Buehler Ltd, Lake Bluff, IL, USA)

(Fig. 1B, C). Teeth presented with enamel or pulp exposure were excluded when evaluated using a stereomicroscope (ML 9300; MEIJI, Saitama, Japan) at 40X magnification. Smear layer on dentin was created by grinding the surface with 180-grit silicon carbide paper in one direction under running water for 30 s.

The teeth were randomly divided into four groups, each group containing eighteen specimens as follows:

Group SF (Control group): 1-step self-etch adhesive (SBU; Single Bond™ Universal; 3M ESPE, St. Paul, MN, USA) used in combination with flowable resin composite (FZF; Filtek™ Z350 XT Flowable; 3M ESPE, St. Paul, MN, USA).

Group V: Self-adhesive flowable resin composite (VF; Vertise® Flow™; Kerr, Orange, CA, USA).

Group PV: VF was used after etching with 37.5% phosphoric acid (Kerr® Etchant; Kerr, Orange, CA, USA).

Group POV: VF was used after etching with 37.5% phosphoric acid (Kerr® Etchant) followed by 2-step total-etch adhesive (OSP; Optibond® Solo Plus™; Kerr, Orange, CA, USA).

After pretreatment according to the experimental groups, VF or FZF in shade A2 was built up following the manufacturer instruction (Table 1) to approximately 6 x 6 x 4 mm^3 using a silicone mold, onto the treated dentin surface (Fig. 1D). Each 2 mm increment was polymerized using a LED light-curing system (Demi™ Plus; Kerr, Orange, CA, USA) with 1,100 mW/cm^2 intensity. Light guide was held perpendicularly and within 1 mm superior to the silicone mold. The light output from the light-polymerizing unit was checked using a radiometer (Model 100 Optilux; Kerr, Orange, CA, USA) throughout the experiment.

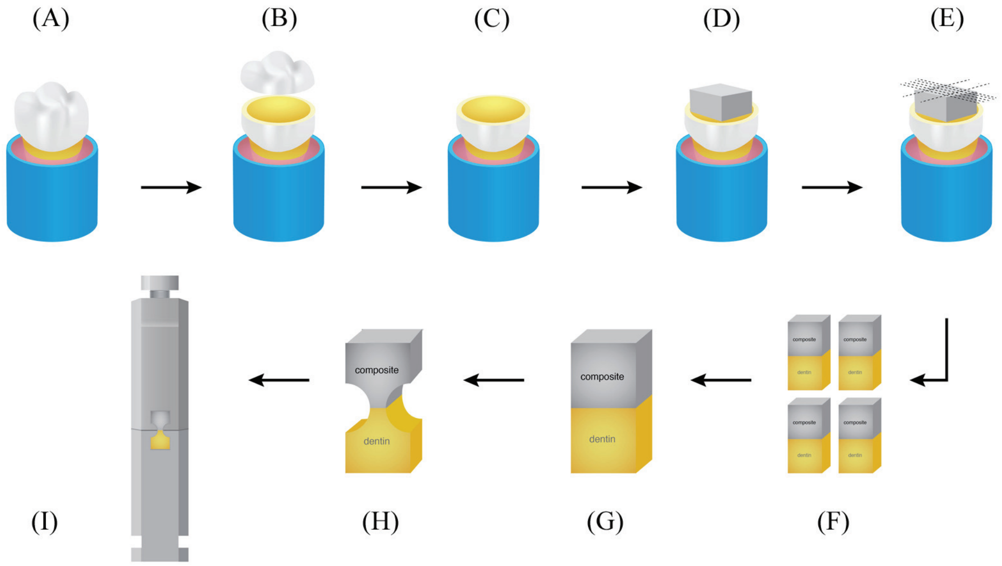

Fig. 1 Schematic diagram of specimen preparation, (A) Embedded tooth was prepared. (B) Occlusal third of crown was removed. (C) Mid-coronal dentin was exposed. (D) Composite was built up. (E) Specimen was sectioned for µTBS test. (F) Four slaps were retrieved from a tooth. (G) A rectangular slab was obtained for specimen preparation. (H) Each slap was prepared for hour-glass specimen. (I) Specimen was tested until failure.

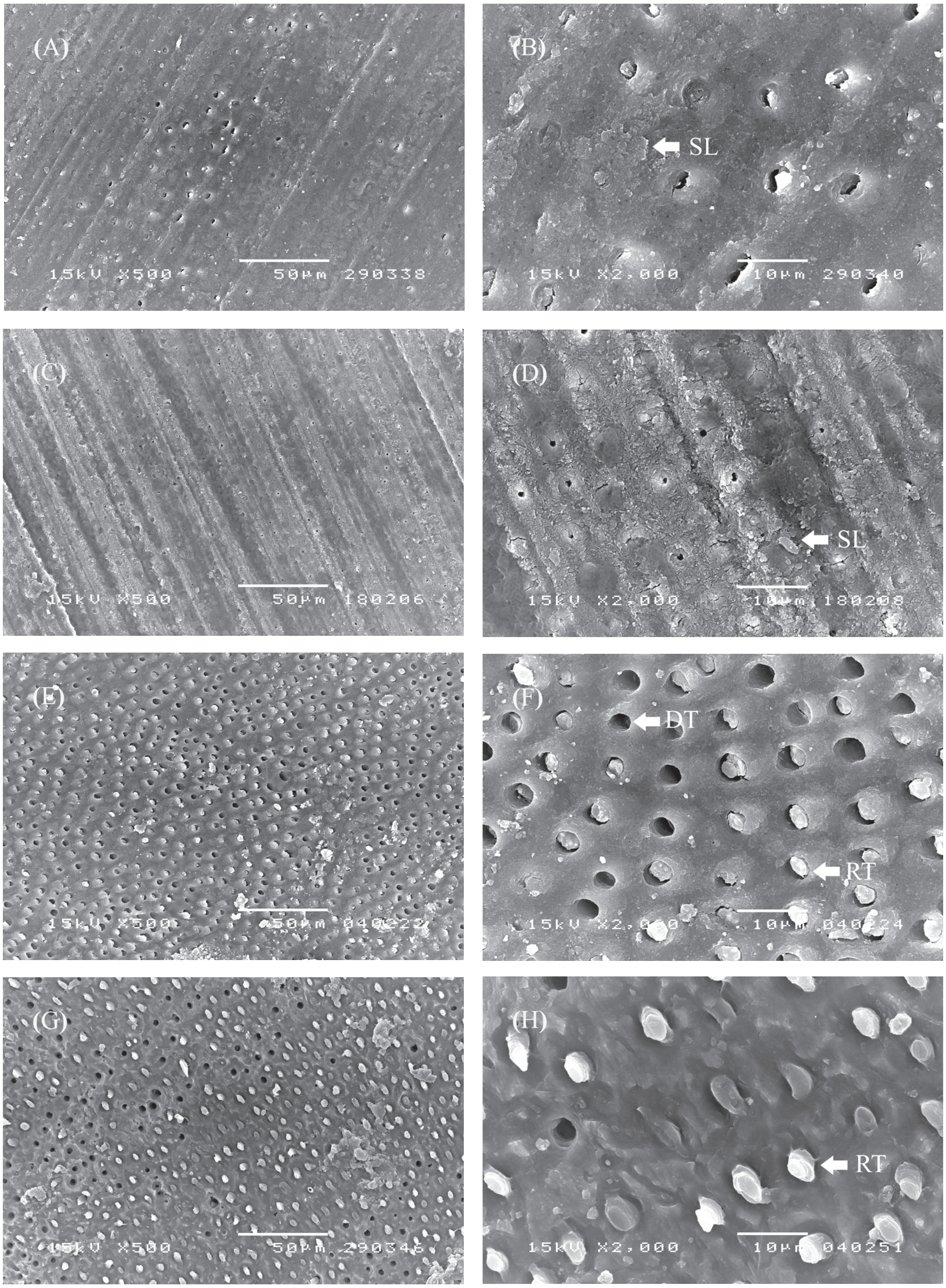

Fig. 2 Representative SEM photomicrographs of fractured surfaces of the dentin side. (A) Group SF24h at 500X. (B) Higher magnification of Fig. 2A at 2000X. (SL: smear layer). (C) Group V24h at 500X. (D) Higher magnification of Fig. 2C at 2000X (SL: smear layer). (E) Group PV24h at 500X. (F) Higher magnification of Fig. 2E at 2000X (DT: dentinal tubule; RT: resin tags). (G) Group POV24h at 500X. (H) Higher magnification of Fig. 2E at 2000X (RT: resin tags).

Table 1 Materials used in the study.

Materials	Composition	Application
Vertise® Flow™ (Kerr, Orange, CA, USA)	**Resin**: GPDM and methacrylate co-monomers **Filler**: prepolymerized filler, 1 µm barium glass, nano-sized colloidal silica, nano-sized ytterbium fluoride	0.5 mm thin layer created by 20 s agitation by brush, light cure (20 s), build more restoration in increments of 2 mm or less, light cure (20 s)
Filtek™ Z350 XT Flowable (3M ESPE, St. Paul, MN, USA)	**Resin**: Bis-GMA, TEGDMA and Bis-EMA **Filler**: nano-sized non agglomerated/non-aggregated silica filler, nano-sized aggregated zirconia/silica (cluster filler, 0.1-5 µm), 0.1-5 µm ytterbium trifluoride particle	The thickness of the individual increments must not exceed 2.0 mm, light cure (20 s)
Optibond® Solo Plus™ (Kerr, Orange, CA, USA)	**Etchant**: 37.5% H_3PO_4 **Adhesive**: Bis-GMA, HEMA, GPDM, ethanol, barium aluminoborosilicate glass, fumed silica (silicon dioxide), sodium hexafluorosilicate, CQ	Acid etch (15 s), rinse (15 s) and gentle air stream (do not desiccate), apply adhesive (15 s with agitation), air dried thin (3 s), light cure (20 s)
Single Bond™ Universal (3M ESPE, St. Paul, MN, USA)	Bis-GMA, HEMA, water, ethanol, silane-treated silica, 10-MDP, 2-propenoic acid, 2-methyl-, reaction products with 1,10-decanediol and P_2O_5, copolymer of acrylic and itaconic acid, dimethylaminobenzoat (-4), CQ, (dimethylamino) ethyl methacrylate, methyl ethyl ketone, silane	Gentle bond agitation (20 s), air stream (5 s), light cure (10 s)

Abbreviation-10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; Bis-EMA: ethoxylated bisphenol A glycol dimethacrylate; Bis-GMA: bisphenol A glycol dimethacrylate; CQ: camphorquinone; GPDM: glycerol phosphate dimethacrylate; HEMA: 2-hydroxyethyl; H_3PO_4: phosphoric acid methacrylate; P_2O_5: phosphorous oxide; TEGDMA: triethyleneglycol-dimethacrylate

After the restorative procedure, each group was further randomly divided into two subgroups ($n = 9$ each) to be tested for 24 hours or 3 months after artificial aging by means of water storage in distilled water at 37°C in an incubator (Contherm 160M; Contherm Scientific Ltd., Lower hut, New Zealand) as followed: Group SF; (1) SF24h (24 hours) (2) SF3m (3 months), Group V; (3) V24h (4) V3m, Group PV; (5) PV24h (6) PV3m, Group POV; (7) POV24h, (8) POV3m.

After storage, teeth were sectioned perpendicular to the adhesive-tooth interface using low speed cutting machine (ISOMET® 1000; Buehler Ltd, Lake Bluff, IL, USA) (Fig. 1E) to obtain four rectangular sample of 1 x 4 x 8 mm-slap from each tooth (Fig. 1F). Nine teeth from each group yielded thirty-six slabs for bond strength evaluation ($n = 36$ per group). Each slab was trimmed to an hourglass-shape with standard grit, cylinder diamond bur of 1.4 mm in diameter (B1, CrossTech Diamond bur #014; CrossTech, Bangkok, Thailand) under air-water irrigation with a cross-sectional area of approximately 1 mm^2 (Fig. 1G and H). The exclusion criteria included presence of any obvious flaw or specimens debonding before testing.

The dimension of each slap was measured using a digital vernier caliper (Mitutoyo digital caliper; Mitutoyo Corp., Kanogawa, Japan). All slabs were then attached to the testing apparatus using a cyanoacrylate adhesive (Model Repair II Blue; Dentsply-Sankin, Ohtawara, Japan), and stressed to failure in tension using universal testing machine (EZ-S Shimadzu; Shimadzu AG-IS, Tokyo, Japan) at a cross-head speed of 1 mm/min (Fig. 1I). The μTBS, given in MPa, was recorded when a fracture occurred. μTBS were determined from the specimens surviving processing, with a record of the number of pretest failures.

Modes of failure of all specimens were recorded using a stereomicroscope (ML 9300; MEIJI, Saitama, Japan) at 40X magnification. Fracture mode was classified into one of four type categories as: Type I: Adhesive failure, Type II: Cohesive failure within dentin, Type III: Cohesive failure with composite, Type IV: Mixed failure. Some representative fracture surfaces were processed for scanning electron microscope (SEM) (JSM-5410LV; JEOL, Tokyo, Japan).

All data were analyzed statistically using SPSS statistics for Windows, version 17.0 (SPSS Inc., Chicago, IL, USA). The μTBS data were analyzed with two-way ANOVA and Bonferroni post hoc test ($p = 0.05$).

Results

The Shapiro-Wilk test showed a normal distribution of μTBS in all groups ($p > 0.05$). Two-way ANOVA revealed that the factor "pretreatment" and the factor "water storage time" influenced the μTBS ($p < 0.05$ for all factors). There was a significant two-factor interaction between "pretreatment" and the factor "water storage time" ($p < 0.05$) in all groups.

Mean of μTBS, standard deviations, and numbers of pretest failures are shown in Table 2. The highest μTBS was found in group POV24h (42.63 ± 4.57 MPa) and the lowest μTBS was found in group V3m (23.39 ± 3.88). Considering the effect of pretreatment, groups with pretreatment gave significantly higher μTBS than groups without pretreatment ($p < 0.05$), except when comparing between group V24h and group PV24h which showed no statistical difference ($p = 0.106$). Regarding influence of water storage time, μTBS was significantly decreased by water storage ($p < 0.05$).

The percentage distributions of failure modes were recorded using a stereomicroscope at 40X magnification. As shown in Table 3, the majority of the failures were predominantly adhesive failure between resin composite and dentin. Group V3m showed the highest adhesive failures (100%), few cohesive failures within composite and mixed failures were found, and cohesive failures within dentin were not found.

Table 2 Microtensile bond strength values, standard deviations and statistical significant after 24 hours and 3 months of water storage

Group	Mean ± SD (MPa)		PTF (n)	
	24 hours	3 months	24 hours	3 months
SF	41.15±6.27 (n=36) a,#	34.89±6.36 (n=35) x,*	0	1
V	32.30±5.62 (n=36) b,#	23.39±3.88 (n=29) y,*	0	7
PV	36.11±5.19 (n=36) b,#	28.89±5.13 (n=31) z,*	0	5
POV	42.63±4.57 (n=36) a,#	37.86±6.47 (n=35) x,*	0	1

Groups having similar letters or symbols (# or*) (letters: column; symbols: row) are not significantly different in their μTBS (two-way ANOVA and Bonferoni post hoc-tests; *p*=0.05). (Group SF=Single Bond™ Universal/ Filtek™ Z350 XT Flowable; V=Vertise® Flow™; PV=37.5% phosphoric acid/Vertise® Flow™; POV=37.5% phosphoric acid/Optibond® Solo Plus™/Vertise® Flow™; SD=standard deviation; PTF=pretest failures; n=number of specimens)

Table 3 Percentage distribution of failure mode

Group	Water storage time	Adhesive	Cohesive in resin	Cohesive in dentin	Mixed
SF	24 hours	31/36(86.1%)	3/36(8.3%)	-	2/36(5.6%)
V		33/36(91.7%)	2/36(5.6%)	-	1/36(2.8%)
PV		32/36(88.9%)	2/36(5.6%)	-	2/36(5.6%)
POV		31/36(86.1%)	4/36(11.1%)	-	1/36(2.8%)
SF	3 months	33/35(94.3%)	1/35(2.9%)	-	1/35(2.9%)
V		29/29(100%)	-	-	-
PV		30/31(96.8%)	-	-	1/31(3.2%)
POV		32/35(91.4%)	2/35(5.7%)	-	1/35(2.9%)

SEM micrographs from representatives illustrating the fractured surfaces of the dentin side of the specimens. Group SF24h; at 500X, shows adhesive failure (Fig. 2A). Higher magnification of Fig. 2A at 2000X, shows dentin surface with smear layer remnants. Dentinal tubules are covered by smear plugs (Fig. 2B). Group V24h; at 500X, shows adhesive failure (Fig. 2C). Higher magnification of Fig. 2C at 2000X, shows dentin surface with smear layer remnants. Dentinal tubules are covered by smear plugs (Fig. 2D). Group PV24h; at 500X, shows adhesive failure (Fig. 2E). Higher magnification of Fig. 2E at 2000X, smooth dentin surface is visible. Etching pattern can be observed as, smear layer have been completely dissolved. Opening dentinal tubules with or without resin tags can be observed (Fig. 2F). Group POV24h; at 500X, shows adhesive failure (Fig. 2G). Higher magnification of Fig. 2G at 2000X, shows adhesive layer on dentin surface. Remnant of resin tags into the dentinal tubules can be observed. Some opening dentinal tubules can be observed (Fig. 2H).

Discussion

The objectives of this study were to determine the μTBS of a self-adhesive resin composite to dentin when using different pretreatment methods and water storage times, compared to a conventional flowable resin composite used in combination with an adhesive agent as a control. SBU was selected as an adhesive agent in control group because the first layer of self-adhesive resin composite was acting, in effect, as 1-step self-etch adhesive.[11] In addition, it contains 10-MDP monomer that has been rated as the promising monomer for chemical bonding to hydroxyapatite.[12] In this study, the first and second null hypotheses tested were rejected. Significant differences in μTBS among groups were shown when using different pretreatments and water storage times.

The μTBS test was chosen, because several specimens could be obtained from one tooth, and many researchers considered that μTBS was the most reliable technique to investigate "true" interfacial bond strength between an adhesive material and the substrate of interest.[13-15] In addition, small bonded surface areas of approximately 1 mm^2 may provide higher bond strengths compared to conventional methods, which used a bonded area of 7-12 mm^2.[13,16] In this study, μTBS specimens were trimmed with burs at the adhesive interface to produce an hourglass appearance called "trimming technique".[15,17] Betamar *et al*[18] studied the stress distribution of the specimens in μTBS test using a finite-element analysis. The results indicated that stress distribution of trimmed specimen was better than that of a non-trimmed specimen. However, improper preparation of trimmed specimen resulted in more sensitive to flaws introduced during specimen preparation and pretest failures during trimming, therefore the preparation of a specimen must be carefully performed. The smear layer, in this experiment, was produced by 180-grit abrasive paper. Koibuchi *et al*[19] recommended 180-grit abrasive paper for dentin preparation since it simulated smears formed clinically with dental burs.

The commonly used aging methods are water storage and thermocycling.[20-24] Many studies have shown that using either or both methods as means of aging process have been well received.[20-24] In this study, water storage was selected as aging method. The methods to store specimens exposing to water, which two different strategies, direct and indirect water exposure, have been used. In this study, the indirect water exposure of the entire restored tooth was selected because it possibly simulated more closely to clinical situation. Water takes some time to diffuse from the external surface into the inner bonded interface.[25] On the contrary, direct water exposure of a small sample piece may not properly allow extrapolation of results to clinical practice.[26]

Another consideration concerning μTBS test method is how to handle the specimens that fail before being tested. Some studies included the failures with a value of zero.[27,28] Others deleted the failures from further statistical analysis.[20,29-32] In this study, the pretest failure specimens (PTF in Table 2) were not taken into consideration during interpretation, because there was a certain amount of force generating failure during specimen preparation[33], not by the effect of aging process by water storage. The pretest failures may have happened during trimming to produce hourglass specimens; hence, it was not suitable to attribute zero-value in the statistical analysis. However, when the calculation was based on the specimens surviving test, an overestimation of bonding potential must be taken into consideration.[34]

Results of the present study revealed that the two-factor interactions (pretreatment and water storage time) were all significant to μTBS ($p<0.05$). Therefore, one must take into account all involved factors including dentin pretreatment and aging process. In this study, at 24-hour water storage, μTBS of group PV24h, that 37.5% phosphoric acid was used as acid pretreatment, was increased when compared to group V24h, that VF was used alone. However this increase was not statistically significant. This result was in accordance with Juloski *et al*[35], 24-hour bonding performance of VF to dentin was better but not statistically significant after dentin pretreatment with acid etching. The increase in microporosities, produced by phosphoric acid etching, resulted in enhanced resin-interlocking and micromechanical retention. Poitevin *et al*[6], showed that dentin pretreatment with 37.5% phosphoric acid significantly improved bonding effectiveness of VF. In this study, at 3-month water storage, although group PV3m was significantly higher in μTBS when compared to group V3m, the further study should be perform using longer storage time, or using other means of aging such as thermocycling or cyclic loading, in order to determine bonding effectiveness of self-adhesive resin composite used in combination with phosphoric acid pretreatment.

In the present study, group POV, that 2-step self-etch adhesive was used as an adhesive agent, had significantly higher μTBS than group V, that VF was used alone, and group PV that pretreatment with 37.5% phosphoric acid. Moreover, even though the first layer of VF (0.5 mm thin layer created by 20-second agitation by brush) was acting, in effect, as 1-step self-etch adhesive.[11] μTBS for the group V was significantly lower than a control group (Group SF) that used 1-step self-etch adhesive (SBU) as an adhesive agent. This result is in accordance with Bektas *et al*[7], VF used with 1-step self-etch adhesive (Optibond® All-In-One) provide significantly stronger bond strength than individual usage. It is possible that although VF incorporates adhesive technology similar to that found in Optibond® products, adding other fillers may reduce the bond strength value. The previous studies showed that wetting of dentin surface was decreased due to higher viscosity of filled resin. This would decrease the penetration of monomers, thus reducing the μTBS.[36,37]

In this study, it is interesting to observe that μTBS of 1-step self-etch adhesive group (Group SF) compared to 2-step total-etch adhesive group (Group POV) at 24 hours or 3 months was not significantly different. The reason is possibly because SBU contains 10-MDP monomer, structurally, the long carbonyl chain. Yoshida *et al*[12] described that 10-MDP was rated as the promising monomer for chemical bonding to hydroxyapatite. This monomer is capable of forming ionic bonds with calcium due to the low dissolution rate of the resulting Ca-salt in its own solution. In addition, Van landuyt *et al*[38] showed that the Ca-10-MDP was one of the most hydrolytically stable salts. However, according to the material's technical profile, OSP used different acidic monomer, namely GPDM. It possesses hydrophilic acidic phosphate group and the short spacer group in the adhesive monomer.

To our knowledge, no data on chemical analysis on bonding mechanism of GPDM was available. Yoshida *et al*[12] showed that according to adhesion-decalcification concept, GPDM rather “etches” than “bonds” to hydroxyapatite compared to 10-MDP. Poitevin *et al*[6] suggested that to achieve self-adhesiveness, a relatively viscous self-adhesive flowable resin composite should contain a functional monomer that rather possesses an effective chemical bonding potential, as it cannot penetrate deeply.

The lowest μTBS was found in the group without pretreatment at 3 months water storage (Group V3m). Previous studies have shown that the aggressive version of simplified self-etch adhesives have been advocated to account for the suboptimal bonding performance, which may be due to 1) the stronger etching process may destabilize the collagen, leading to a decrease in bond strength[39], 2) weaker cohesive strength of the adhesive[29,40], 3) the combination of acidic hydrophilic and hydrophobic monomers into a single step compromised the polymerization of the adhesive[41], and 4) a low degree of conversion of the resin monomer that was caused by the effect of oxygen inhibition.[42]

A systematic review of Van Meerbeek *et al*[43] revealed that there were significant differences in the pooled mean bond strength, as the weighted bond strength means per adhesive class have the following ranges: 31 MPa for 3-step total etch adhesives, 29 MPa for 2-step total etch adhesives, 26 MPa for 2-step self-etch adhesives, 20 MPa for 1-step self-etch adhesives, In present study, VF had a 23.39 MPa bond strength after 3-month water storage so it can be compared to other 1-step self-etch adhesives. In this study, all bond strengths obtained were more than 20 MPa. They were, supposedly, strong enough to resist contraction forces of resin composite because it has been postulated that minimum bond strength of 17 to 20 MPa to enamel and dentin was needed to resist contraction forces of resin composite materials.[16] However, in clinical practice, the optimum bond strength of resin composite to dentin is not yet known. Bond strength was dependent not only on materials used, or storage time but also on other factors, such as, restorative technique, quality of substrate, isolation method, skills of operator and finishing technique, etc. Moreover, patient-related factors, such as oral hygiene, age, occlusal loading, and dentin quality, may be more influential than any material property.[3,44]

A factor known to promote bond degradation is long-term water exposure. The 3-month water storage applied in this experiment was regarded as a short period in comparison to life expectancy of restoration. Although, ISO/TS 11405:200345 recommendation for water storage is 6 months, several studies reported significant decreases in bond strengths in only a few months[20,21,46], since degradation throughout the dentin bonded interface occurred rapidly.[25,47] A recent study showed that μTBS significantly decreased after 3 months in water storage compared to 24 hours in all groups. Decrease in bonding effectiveness may be caused by degradation of interface components.[48] Pashley *et al*[49] found that matrix metalloproteinases (MMPs) played an important role in degradation process. These enzymes are hydrolases, which break peptide bonds by adding water. Therefore, in the presence of water, μTBS may decrease as a consequence of degradation of collagen. In addition, water infiltration decreased the mechanical properties of the polymer matrix by swelling and reducing the frictional forces between the polymer chains, a process known as “plasticization”.[50,51] Furthermore, some interface components, such as uncured monomers and break-down products of previous mechanisms, could elute and, therefore, weaken the bond overtime.[52]

Regarding failure mode, in this study, the microscopic study showed the majority of failures were adhesive failure that occurred in adhesive interface. It can be assumed that bond strength value would be representing adhesive bond strength rather than cohesive bond strength. From the SEM evaluation, the failure mode of dentin side of fractured representatives was initially classified as adhesive failure. Group SF24h (Fig. 2A and B) that used 1-step self-etch adhesive system as an adhesive agent, and group V24h (Fig. 2C and D) that VF was applied alone showed similar pattern of smear layer remnant. These SEM evaluation results agreed with Hanabusa *et al*[11], which self-adhesive resin composite was bonded to bur-cut dentin, and resin infiltrated smear layer with a maximum of a few micrometers, but resin tags were not identified since the smear plugs within the tubules were not dissolved. This kind of superficial interaction, along with the absence of resin tag formation, was typical of self-etch adhesives. Group PV24h (Fig. 2E and F) showed etched pattern that was the result from phosphoric acid etching. Group POV24h (Fig. 2G and H) showed adhesive layer that was the result from application of 2-step total-etch adhesive.

In present study, using dentin pretreatment increased the bond strength of VF. On the other hand, a limitation of this study is that only one self-adhesive resin composite was evaluated. Future investigation to improve bonding effectiveness may involve investigation of other self-adhesive resin composites and adhesive agents. The extended of water storage times may provide valuable information on longevity of material. In addition, clinical studies of self-adhesive resin composite are necessary to determine the bonding performance, and are useful for in vitro and in vivo comparison.

Conclusion

Vertise® Flow™, in combination with the use of 37.5% phosphoric acid etching and adhesive agent, Optibond® Solo Plus™, gave significantly higher μTBS than using Vertise® Flow™ alone. Regarding influence of aging process, μTBS was significantly decreased by water storage time in all groups.

Acknowledgement

This study was supported by Chulalongkorn University Graduate School Thesis Grant.

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กำลังแรงยึดแบบดึงระดับจุลภาคของเรซินคอมโพสิตชนิดยึดติดได้ด้วยตัวเองต่อเนื้อฟัน

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บทคัดย่อ

วัตถุประสงค์ เพื่อประเมินผลของการปรับสภาพผิวเนื้อฟันและกระบวนการเสื่อมสลายด้วยวิธีการแช่น้ำต่อกำลังแรงยึดแบบดึงระดับจุลภาคของเรซินคอมโพสิตชนิดยึดติดได้ด้วยตัวเองต่อเนื้อฟัน

วัสดุและวิธีการ คัดเลือกฟันกรามแท้ที่ถูกถอนจำนวน 72 ซี่ เพื่อนำมาประเมินกำลังแรงยึดแบบดึงระดับจุลภาคของเนื้อฟันที่ระดับกึ่งกลางตัวฟัน โดยคำนึงถึงวิธีการปรับสภาพผิวเนื้อฟัน ชนิดของวัสดุ และระยะเวลาในการแช่น้ำ โดยฟันจะแบ่งเป็น 4 กลุ่ม ด้วยวิธีการสุ่ม ได้แก่ กลุ่มควบคุม ปรับสภาพเนื้อฟันด้วยซิงเกิล บอนด์ ยูนิเวอร์แซล ตามด้วยการบูรณะโดยใช้ฟิลเทค ซี 350 เอ็กซ์ที โฟลเอเบิล คอมโพสิต กลุ่มทดลองที่ไม่มีการปรับสภาพเนื้อฟัน ตามด้วยบูรณะโดยใช้เวอร์ทิส โฟล กลุ่มที่มีการปรับสภาพเนื้อฟันด้วยกรดฟอสฟอริกความเข้มข้นร้อยละ 37.5 เพียงอย่างเดียวตามด้วยการบูรณะโดยใช้เวอร์ทิส โฟล และกลุ่มที่มีการปรับสภาพเนื้อฟันด้วยกรดฟอสฟอริกความเข้มข้น ร้อยละ 37.5 ร่วมกับออพติบอนด์ โซโล พลัส ตามด้วยการบูรณะโดย เวอร์ทิส โฟล หลังจากนั้นจะทำการแบ่งแต่ละกลุ่มเป็น 2 กลุ่มย่อยด้วยวิธีการสุ่ม เพื่อแยกตามระยะเวลาการแช่น้ำที่ 24 ชั่วโมง หรือ 3 เดือน ทำการทดสอบกำลังแรงยึดแบบดึงระดับจุลภาคของชิ้นงานตัวอย่างทั้ง 8 กลุ่มหลังการแช่น้ำเสร็จสิ้น และจะมีการบันทึกชนิดของความล้มเหลว ข้อมูลที่ได้จะนำมาวิเคราะห์ทางสถิติด้วยสถิติความแปรปรวนแบบสองทางและเปรียบเทียบด้วยวิธีการของบอนเฟอโรนี ที่ระดับนัยสำคัญ 0.05

ผลการศึกษา ค่ากำลังแรงยึดแบบดึงระดับจุลภาคสูงสุดพบในกลุ่มที่มีการปรับสภาพเนื้อฟันด้วยกรดฟอสฟอริกความเข้มข้น ร้อยละ 37.5 ร่วมกับออพติบอนด์ โซโล พลัส ตามด้วยการบูรณะโดย เวอร์ทิส โฟล ที่ 24 ชั่วโมง (42.63 ± 4.57 เมกะปาสคาล) และ ค่ากำลังแรงยึดแบบดึงระดับจุลภาคต่ำสุดพบในกลุ่มที่ไม่มีการปรับสภาพเนื้อฟัน ตามด้วยบูรณะโดยใช้เวอร์ทิส โฟล ที่ 3 เดือน (23.39 ± 3.88 เมกะปาสคาล) พิจารณาถึงผลของการปรับสภาพผิวเนื้อฟันพบว่า กลุ่มที่มีการปรับสภาพผิวเนื้อฟันจะมีค่ากำลังแรงยึดแบบดึงระดับจุลภาคสูงกว่ากลุ่มที่ไม่มีการปรับสภาพผิวเนื้อฟัน และการแช่น้ำทำให้กำลังแรงยึดแบบดึงระดับจุลภาคที่ 3 เดือนมีการลดลงอย่างมีนัยสำคัญเมื่อเทียบกับกลุ่ม 24 ชั่วโมง

สรุป การปรับสภาพผิวเนื้อฟัน และการแช่น้ำมีผลต่อกำลังแรงยึดแบบดึงระดับจุลภาคของเรซินคอมโพสิตชนิดยึดติดได้ด้วยตัวเองต่อเนื้อฟัน

(ว ทันต จุฬาฯ 2558;38:21-34)

คำสำคัญ: *การแช่น้ำ; การปรับสภาพผิวเนื้อฟัน; กำลังแรงยึดแบบดึงระดับจุลภาค; เรซินคอมโพสิตชนิดยึดติดได้ด้วยตัวเองต่อเนื้อฟัน*

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