RESEARCH ARTICLE

The influence of acid etching time on the bonding performance of universal resin adhesives to dentin

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Abstract

Objective: To study the impact of different phosphoric acid etching time on the bonding strength and durability between two universal resin adhesives.

Method: The study was conducted at the Oral and Maxillofacial Surgery Department of the Affiliated Stomatological Hospital of Nanjing University Medical College, China, from August to October 2020, and comprised fresh human molars without caries that were exposed to the dentin surface. They were randomly divided into 4 groups based on different phosphoric acid etching time; 0s, 5s, 10s and 15s. Each group was bonded by composite resin columns with general resin adhesives Clearfil Universal Bond and All-bond Universal to form micro-tensile bonding specimens. After 24h of storage in distilled water, micro-tensile bond strength tests were carried out directly or after thermocycled for 10,000 cycles. After fracture, the bonding interface was observed by stereomicroscope and the fracture mode was recorded. Data was analysed using SPSS 22.

Results: Of the 72 tooth specimens 18(25%) were in each of the 4 groups. Micro-tensile bond strength of Clearfil Universal Bond groups of 10s and 15s and All-bond Universal groups of 0s, 5s and 10s was higher than that of the other groups before and after thermocycling ($p<0.05$). The thermocycling treatment significantly reduced the micro-tensile bond strength of 0s, 10s and 15s groups of All-bond Universal ($p<0.01$), but had no significant effect on Clearfil Universal Bond groups ($p>0.05$). The micro-tensile bond strength of 10s and 15s group of Clearfil Universal Bond was higher than that of corresponding groups of All-bond Universal groups before and after thermocycling ($p<0.05$).

Conclusion: Etching time of dentin surface could affect the micro-tensile bond strength and durability of universal resin adhesives.

Keywords: Universal resin adhesive, Dentin, Bond strength, Etching time, Durability. (JPMA 75: S-92 [Suppl. 02]; 2025)

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Introduction

The oral adhesion system realises the adhesion between composite resin and oral tissue by micromechanical adhesion between composite resin and oral hard tissue.[1] According to the bonding strategy, the dental adhesive system can be divided into three categories: total etching (TE) adhesive, self-etching (SE) adhesive, and glass iomer (GI) adhesive.[2] SE adhesive system means that the acidic functional components are mixed with coupling agents, and tooth demineralisation and coupling occur simultaneously, eliminating independent etching steps.[3] Universal adhesives have emerged as a trend in dentistry as they can be applied in etch-rinse or self-etch modes due to their reduced application steps and flexibility. In recent years, universal adhesives have appeared on the market that can be combined with resin adhesives, various substrates that do not require surface treatment, substrates that reduce the number of treatments, or various surface moisture on enamel and dentin surfaces. Despite this exciting flexibility, several studies have suggested that many universal adhesives are less durable than two-step SE adhesives.[4,5] For enamel, this occurs in etch-rinse and SE modes, whereas dentin occurs in the SE mode. Nonetheless, the flexibility of universal adhesives is becoming increasingly popular in clinical settings, and further research is needed to better utilise them.[6] Clearfil Universal Bond (CUB) and All-Bond Universal (ABU) are two types of universal resin adhesives. CUB is a single-group spectrocuring adhesive that combines three etching techniques (total etching, self-etching and selective etching) for direct and indirect repair. It is also suitable for the surface treatment of zirconia and silica-based glass ceramics.[7] ABU is a light curing single component dental adhesive that combines etching, priming and bonding in a bottle. ABU is an ethanol/water-based dental adhesive that connects dentin to both cut and uncut enamel. As a universal adhesive, ABU is designed to be fully compatible with light-curing, self-curing and double-curing composites. ABU can be used for bonding of direct and indirect repairs, with or without phosphoric acid etch.[8] Different universal resin adhesives contain different acid monomer components, and potential of hydrogen (pH)

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values are also very different, which cause different degrees of demineralisation on the dentin surface.[9] Traditional acid etching washing resin adhesives do not contain acid monomers and require 15s of phosphoric acid etching on the dentin surface. However, there is still a lack of relevant studies on whether pre-etching is necessary for general resin adhesives containing acidic monomers.

The current study was planned to compare the effects of different etching time on the bonding strength and durability of two universal resin adhesives, and to provide reference for rational use of general resin adhesives in clinics.

Materials and Methods

The study was conducted at the Oral and Maxillofacial Surgery Department of the Affiliated Stomatological Hospital of Nanjing University Medical College, China, from August to October 2020, and comprised fresh human molars without caries that were exposed to the dentin surface. The materials used included 35% phosphoric acid etchant (Kuraray Company, Japan), CUB (Kuraray Company, Japan), ABU (Bisco Company, United States), light curing composite resin Z350 (3M Company, United States), self-curing dental care water and dental care powder (Shanghai Pigeon Company, China), Rhodamine B isothiocyanate (Sigma Company, United States), LED light curing lamp Elipar Deepcure (3M Company, USA), slow cutting machine Isomet 1000 (Biaylor Company, USA), laser scanning confocal microscope Ism800 (Zeiss Company, Germany), stereomicroscope sma1500 (Nikon Company, Japan), micro-tensile tester T-61010K (Bisco Company, USA), vernier caliper JEM-200CX (Shanghai Precision Instrument Company, China), 37ºC biochemical incubator SPX-80 (Shanghai Botai Experimental Equipment Co., Ltd., China), and cold and hot circulator TC-501FIII (Suzhou Weill Experimental Supplies Co., Ltd., China) (Table 1).

After approval from the ethics review committee of Nanjing University School of Stomatology, China, and with informed consent of the patients, intact human caries free molars were collected. The roots of the isolated teeth were embedded with self-setting plastic. The upper half of the line between the enamel cementum boundary of the proximal middle buccal axis angle and the proximal middle buccal tip was taken as the cutting point. The crown enamel was cut along the vertical long axis of the cutting point with a low-speed cutting machine under water until the superficial dentin. After no enamel residue was confirmed under stereomicroscope, 320 mesh and 600 mesh silicon carbide sandpapers were used to polish under running water for 30s to prepare the dentin bonding smear layer.

Table-1: Composition of universal resin adhesives.

Materials	Main components	Lot number	Manufacturer	pH
Clearfil Universal Bond	Glycidyl methacrylate, hydroxyethyl methacrylate, 10 methacryloyloxydecyl phosphate, colloidal silica, hydrophilic aliphatic dimethacrylate, silane coupling agent, DL CAMPHORQUINONE, ethanol, promoter, initiator, water.	9Q0012	Kuraray company (Japan)	2
All-bond Universal	Two-part A glycidyl methacrylate, hydroxyethyl methacrylate, 10 methacryloyloxydecyl phosphate, ethanol, initiator, water.	1600006776	Bisco company (USA)	3.2

The tooth specimens randomly divided into 4 groups based on the following different etching time: 0s (no etching; control group), 5s, 10s and 15s. Each group was randomly divided into 2 subgroups (CUB and ABU) based on different types of universal resin adhesives. After the tooth specimens were etched with 35% phosphoric acid solution at different etching times, according to the requirements of the product manual, the bonding process was carried out. The resin adhesive was applied on the surface of the dentin, repeatedly applied for 15s, gently blown at medium pressure for 5s, illuminated for 10s, and laminated with light curing composite resin Z350 for 5mm. The bonded tooth specimens were stored in distilled water at 37ºC for 24 hours, and then the bonded specimens of each group were cut perpendicular to the surface of the adhesive body along the buccal lingual direction and the mesial distal direction with a low-speed cutting machine, and cut into 1.0mm×1.0mm×8.0mm micro-tensile test piece. Excluding the samples with cracks, eight groups of specimens were randomly divided into 2 groups based on the experimental process: the pre-thermalcycling group and the post-thermalcycling group. Specimens with dentin length not less than 4mm were selected from each group. Before the cold and hot cycle, the samples of the group were immersed in distilled water and placed in a 37ºC incubator for 24h. After the cold and hot cycles, the temperature of the group was 5ºC and 55ºC, respectively, and the group was cycled 10,000 times, each time staying at high or low temperature for 30s, with an interval of 2~3s.

The specimen was bonded to the clamping device of the specimen with cyanoacrylate and connected to the micro-tensile testing machine through the clamp. The maximum load force (F) at the time of tensile fracture was tested, and the tensile rate was 1mm/min. The width (B) and thickness (H) at the bonding interface of the specimen were measured with an electronic vernier caliper, and the actual bonding area S=B×H was calculated. The tensile strength (MPa) was calculated according to the formula,[10] micro-tensile bond strength test (MTBS)=F/S.

The broken sections of the specimens were observed under the stereomicroscope, and the types of bonding failure were classified and counted. There were three types of adhesive failure: interface failure in which the fracture occurred between dentin and composite resin, including cohesive failure of adhesive; cohesive failure in which the fracture occurred in dentin or composite resin, respectively; and blend failure in which the fracture happened in dentin or composite resin, respectively.

After ultrasonic vibration at 37ºC, 0.2mg rhodamine B was dissolved in 1ml of two general-purpose resin adhesives, and stored in a 1ml plastic reagent bottle with a conical cap under shading. Eight tooth specimens were divided into four groups based on the following different etching time: 0s (no etching; control group), 5s, 10s and 15s. The tooth specimens of each group were randomly divided into 2 groups based on different universal resin adhesives. After etching the tooth specimens with 35% phosphoric acid solution, the bonding process was the same as before, according to the requirements of the product manual. The bonded tooth specimens were stored in distilled water at 37ºC for 24h, and then the adhesive specimens of each group were cut with a low-speed cutting machine in the direction perpendicular to the adhesive cheek tongue, and the specimens were prepared with a thickness of 2mm. Sandpaper of different surface sizes (400, 800 and 1200 mesh) were polished from coarse to fine along the direction perpendicular to the adhesive interface until the specimens were transparent.[11] The cracked sample was removed, and the sample was soaked in distilled water and placed in an incubator at 37ºC. Before the observation, the specimens were fixed on the slide, and the microscopic morphology of the bonding interface of each specimen was observed by confocal laser scanning microscope (CLSM) at 100% relative humidity. When the excitation wavelength was 514nm and the emission wavelength was 600nm, Rhodamine B showed orange red fluorescence. Zen software was used to observe the morphology of resin protrusion at the dentin bonding interface. The samples were examined at ×2000 magnifications.

Data was analysed using SPSS 22. Three-way analysis of variance (ANOVA) was used to analyse the effects of acid etching time, type of universal resin adhesive and cold-heat cycle on MTBS, and to judge the interaction of the three. The measurement data was expressed as mean±standard deviation. Single-factor ANOVA was used to compare the effects of cold-heat cycle, resin adhesive and acid etching time on bonding strength, and test the level α=0.05.

Results

Of the 72 tooth specimens 18(25%) were in each of the 4 groups. Three-factor analysis showed that there was no interaction of the three factors on the influence of acid etching time, adhesive type and cold-heat cycle on MTBS (f=2.442, p=0.064). Two-factor analysis showed that the acid etching time×the effect of cold-heat cycles on MTBS was not significant (f=2.129, p=0.096); thermalcycling × the effect of adhesive type on MTBS was significant (f=28.483, p<0.001); and aging mode × the effect of adhesive type on MTBS was significant (f=14.905, p<0.001).

Single-effect analysis of acid etching time showed that the bonding strength of 10s and 15s groups of CUB before or after thermalcycling was significantly higher than that of 0s and 5s groups (p<0.05). The bonding strength of 0s, 5s and 10s groups of ABU before or after cold-heat cycle presented higher than that of 15s group (p<0.05). The bonding strength of 0s, 10s and 15s groups of ABU decreased significantly after cold-heat cycle (p<0.01). The bonding strength of 0s, 10s and 15s groups of CUB presented higher than that of corresponding groups of ABU before or after the cold-heat cycle (p<0.05) (Table 2).

The fracture mode of the two resin adhesives under various experimental conditions was mainly interface fracture (Table 3).

Table-2: Micro-tensile bond strength of the test groups (MPa}.

Adhesive	Etching time	Bonding strength	
		Before hot and cold cycle	After hot and cold cycle
CUB	0 s	22.45 ± 7.54^{B}	22.19 ± 6.87^{B}
	5 s	21.71 ± 7.68	21.37 ± 7.26
	10 s	29.30 ± 7.62^{AB}	29.19 ± 7.99^{AB}
	15 s	26.62 ± 6.87^{AB}	25.25 ± 6.95^{AB}
ABU	0 s	21.62 ± 5.42^{a}	15.35 ± 3.97^{ab}
	5 s	24.94 ± 7.66^{a}	24.49 ± 4.39^{a}
	10 s	25.82 ± 5.70^{a}	17.03 ± 5.01^{ab}
	15 s	16.85 ± 4.13	12.14 ± 2.56^{b}

CUB: Clearfil Universal Bond, ABU: All-bond Universal.
^{A}p<0.05, compared to 0s and 10s of CUB group before and after hot and cold heat cycle; ^{A}p<0.05, compared to 0s, 10s and 15s of ABU group before and after hot and cold heat cycle; ^{a}p<0.05, compared to 15s of ABU group before and after hot and cold heat cycle; ^{b}p<0.05, compared to ABU group before hot and cold heat cycle.

Table-3: Count of failure modes of the test groups.

Type of adhesive	Etching time	Before hot and cold cycle			After hot and cold cycle		
		Interface	Cohesion	Blend	Interface	Cohesion	Blend
CUB	0 s	29	0	0	26	3	1
	5 s	27	2	0	28	0	2
	10 s	28	0	1	29	1	0
	15 s	27	1	1	23	3	4
ABU	0 s	30	0	0	30	0	0
	5 s	26	2	2	27	1	2
	10 s	27	1	2	29	1	0
	15 s	30	0	0	30	0	0

CUB: Clearfil Universal Bond, ABU: All-bond Universal.

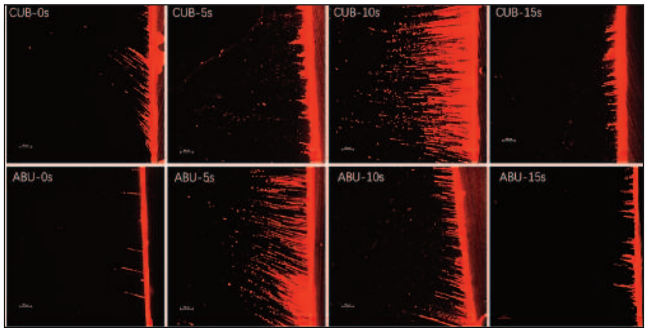

Figure: Dentin bonding interface under different etching time.
CUB: Clearfil Universal Bond, ABU: All-bond Universal.

Fluorescent image (Figure) showed that the two adhesives did not separate from the dentin surface, and both groups of adhesives had different degrees of penetration on the dentin surface. The two adhesives in 0s group could form resin protrusions in dentin tubules, and the resin protrusions formed by CUB were significantly denser than those of ABU. With the increase of acid etching time, the resin burst density formed by the two adhesives was more obvious than that of the 0s group, and the density and resin burst length of the CUB 10s group and the ABU 5s group were significantly better than those of other groups (Figure).

Discussion

Dentin surface etch 15s is the recognised gold standard for traditional acid-free monomer etch-rinsing resin adhesives. However, some studies have shown that with the prolongation of acid etching time, the amount of nano-leakage at the dentin binding interface increases, and there is a positive correlation.[12] At the same time, Sardella et al.[13] and Osorio et al.[14] have shown that the bonding strength obtained by properly reducing the acid etching time on the dentin surface is higher than that obtained by acid etching for 15s. All these studies show that excessive acid etching on dentin surface has a negative effect on its binding strength. The universal resin adhesive is an adhesive with its own acid monomer. The pH values of ABU and CUB, two general-purpose resin adhesives selected in the current study, were 3.2 and 2.3, respectively. If the conventional etching time of 15s is used to etch the dentin surface, the general acid-containing resin adhesive may produce acid etching on the dentin surface, which will affect the interface structure, binding strength and durability of the dentin.

In this study, CLSM technique was adopted to observe the initial morphology of the bonding interface under different etching times. CLSM combines optical microscope technology, laser scanning technology as well as computer image processing technology to directly observe the penetration of adhesive at the bonding interface.[15,16] The results showed that the acid etching on the dentin surface could improve the penetration of resin adhesive on the dentin surface. No matter how long the acid etching time was, the resin burst density formed by the penetration of universal resin adhesive on the dentin surface after acid etching was much higher than that of the experimental group without acid etching. The results showed that the mechanical bonding effect of common resin adhesive on dentin surface was better than that of self-etching resin adhesive. The interfacial observation also showed that the penetration depth of the universal resin adhesive on the dentin surface was affected by the change of etching time. Due to the composition and structure of the material itself and the design of the pH value, the two universal resin adhesives have different acid etching times to form the longest resin protrusions. ABU was in the 5s group and CBU was in the 10s group, which suggested that for the universal resin adhesive with acid, the standard 15s of acid etching and flushing mode is too long. Consistently, previous literature also indicated that shortening the acid-etching time to 5s has a good binding impact on ABU resin.[17]

The results of CLSM observation and analysis of interface structure were similar to the bonding strength and durability between the two general resin adhesives and dentin. In this study, the bonding strength of CUB 10s group and ABU 5s group before and after thermalcycling presented higher than that of other groups. This implied that the suitable etching time on the dentin surface could make the universal resin adhesive achieve good penetration effect, and had a very positive effect on the bonding strength and durability between the resin adhesive and dentin, which was in line with an earlier study.[18]

The binding strength of acid universal resin adhesive to dentin surface is affected by various acidic monomers, such as dihydrogen 10 methylpropenoxy decyl phosphate (10MDP), besides mechanical binding force caused by adhesive penetration.[19] 10MDP combines with hydroxyapatite (HAP) on the surface of dentin to form water-insoluble calcium hydroxyphosphate (ca10[po4]6 [oh]2) and obtain chemical binding.[20] The stable monomer calcium salt conjugate is conducive to improving the durability of the adhesive, and the acid etching time can directly affect the stability of the chemical bond formed between the acid monomer and the dentin surface.[21] In this study, the bonding strength of two kinds of universal resin adhesives was different after cold-heat cycles. Regardless of whether the CUB was etched or not, the etched time was long or short, after the cold-heat cycle, the combined strength of the groups was very stable and did not decrease significantly. However, except for the 5s group, the bonding strength of ABU decreased significantly in other groups. This suggested that there were differences in the chemical bonding between the two general resin adhesives and dentin, which was consistent with a previous study.[22]

Thermal fatigue method was used to simulate the temperature changes caused by diet and respiration in oral cavity, which is a common aging method and has been used in many studies.[23] The physical properties of restorations and teeth are different. When the external temperature changes, the bonding interface will generate thermal stress, resulting in interface damage.[24] Gale et al.[25] believed that 10,000 cycles of extracorporeal cold-heat circulation were equivalent to one year of normal function of bonded prosthesis in vivo. For ABU, except for the 5s group, 1-year simulated aging significantly reduced the bonding strength of the other groups, suggesting that the chemical bonding between ABU and dentin was insufficient compared to CBU, and the mechanical bond generated by good resin permeability in 5s group was needed to maintain the stability of the bonding interface. However, compared to the long clinical service life, the 10,000 cold-heat cycle tests were still too short. For CUB, longer durability test conditions are required to verify the effect of different etching time on bonding strength and durability.

The current study has some limitations. First, the study just selected two kinds of universal resin adhesives, which was not enough. In addition, the application time had an impact on the adhesive performance. Therefore, more relevant experiments should be carried out to validate the current findings.

Conclusion

The etching time of dentin surface could affect the bonding strength and durability of universal resin adhesives. The optimal etching time should be selected for each universal resin adhesive.

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