Influence of photoirradiation conditions on the dentin bond durability and interfacial characteristics of universal adhesives

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Contents

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Abstract

A recent trend in adhesives has been the utilization of universal adhesives, which can be used in self-etch, total-etch, and selective-etch modes. Universal adhesives have a high hydrophilicity even after polymerization, which may lead to poor dentin bond durability of the adhesives. These phenomena may be dependent upon the degree of polymerization of the adhesive, which is strongly influenced by the total energy per unit area at the adhesive surface. Previous study reported that a reduction in energy density caused lower dentin bonding performance in single-step self-etch adhesives, and adequate bond strength required an energy density surpassing 4,000 mJ/cm^2. However, there is no independent research on the influence of different photoirradiation conditions at constant total energy on the dentin bond durability of universal adhesives. The purpose of this study was to determine the influence of photoirradiation conditions on the dentin bond durability and interfacial characteristics of universal adhesives.

The universal adhesives used were Adhese Universal, All-Bond Universal, G-Premio Bond, and Scotchbond Universal. The universal adhesives were applied to the dentin surfaces ground with 320-grit silicon carbide paper either with or without phosphoric acid pre-etching, and photoirradiated with 100 mW/cm^2 for 40 s, 200 mW/cm^2 for 20 s, or 400mW/cm^2 for 10 s. A resin composite was bonded to the dentin to determine shear bond strength (SBS) to dentin after 24 h water storage and 30,000 thermal cycles. SBS measurements were performed using a notched-edge test as described in ISO 29022. The water contact angles of cured adhesive were measured by the sessile drop method using a contact angle measurement apparatus. Scanning electron microscopy (SEM) observations of the fracture surface after the SBS test and of resin-dentin interfaces were conducted.

Greater dentin SBSs after 24 h water storage and 30,000 thermal cycles were achieved at a light intensity of 200 and 400 mW/cm^2, regardless of pre-etching. Universal adhesives photoirradiated at 200 and 400 mW/cm^2 exhibited significantly higher water contact angles than those at 100 mW/cm^2 regardless of pre-etching. SEM observations of fracture surfaces after SBS testing showed larger numbers of voids in the specimens photoirradiated at 100 mW/cm^2 than in those at 200 and 400 mW/cm^2 regardless of pre-etching. SEM observations of the resin-dentin interface showed that the adhesive layer was considerably thinner under photoirradiation at 100 mW/cm^2 than at 200 and 400 mW/cm^2 regardless of pre-etching.

The results of this study indicate that the photoirradiation conditions affect the dentin bond durability of universal adhesives even at the same total energy. In addition, the water contact angle and thickness of universal adhesives increased with increasing of light intensity due to the enhanced polymerization of these adhesives, even though the same energy density was maintained. Sufficient light intensity might enhance the polymerization reaction of the universal adhesive, leading to greater dentin bond durability of the adhesive. On the other hand, universal adhesives photoirradiated at 100 mW/cm^2 exhibited higher hydrophilicity, leading to inferior bond durability.

Introduction

The latest adhesives require fewer and simpler application steps and have shorter application times in clinical settings (1), leading to time-saving options such as single-step self-etch adhesives (2). A recent trend in adhesives is the utilizing of universal adhesives, which can be used in self-etch, total-etch or selective-etch modes (3), and can also be used to bond to a variety of substrates (4). These simplified adhesives consist of acidic functional, hydrophilic and hydrophobic monomers, solvent, water, and fillers, which are incorporated into the adhesive to meet clinicians' demands for simpler clinical processes (5). Water plays an essential role in ensuring the ionization of acidic functional monomers, while the added organic solvents facilitate mixing between hydrophilic and hydrophobic components (6). However, the incorporation of water and solvents into the adhesive may reduce its mechanical properties, decreasing bond durability (7). Therefore, their removal from these adhesives before photoirradiation is important in order to achieve optimal bonds.

The simplified adhesives present high hydrophilicity even after polymerization, which may increase their solubility and water uptake compared with their conventional multistep equivalents (8). Long-term water leaching into adhesives might further compromise their mechanical properties, affecting their bond durability (9). These phenomena depend on the degree of polymerization of the adhesive, which relies on the energy density at the adhesive surface. This energy density, which is calculated by multiplying the light intensity by the total photoirradiation time during curing (10), varies according to clinical situations such as cavity preparation, tooth position, and existing adjacent teeth (11). The energy density decreases when the distance between the light tip and cavity wall increases (12). According to Nojiri *et al.* (13), such a reduction in energy density causes lower dentin bond strength in single-step self-etch adhesives, and adequate bond strength requires an energy density surpassing 4,000 mJ/cm^2. In addition, the observed dentin bond strength of single-step self-etch adhesives was strongly related to the interfacial characteristics of the cured adhesive, and these parameters were affected by the light intensity of the curing unit (13).

This study aims to evaluate the influence of photoirradiation conditions on the dentin bond durability and interfacial characteristics of universal adhesives. The null hypothesis to be tested was that different photoirradiation conditions achieving an energy density of 4,000 mJ/cm^2 would not affect the dentin bond durability or interfacial characteristics of universal adhesives.

Materials and Methods

1. Materials tested

The universal adhesives used were Adhese Universal (AU, Ivoclar Vivadent, Schaan, Lichtenstein), All-Bond Universal (AB, Bisco, Schaumburg, IL, USA), G-Premio Bond (GP, GC Corp., Tokyo, Japan), and Scotchbond Universal (SU, 3M ESPE, St. Paul, MN, USA). Ultra-Etch (Ultradent Product Inc., South Jordan, UT, USA) was used as a phosphoric acid pre-etching agent for bonding to dentin, and Clearfil AP-X (Kuraray Noritake Dental Inc., Tokyo, Japan) was used as the resin composite for the bonding procedures. The lot numbers and compositions of the materials used are listed in Table 1.

2. Specimen preparation

The shear bond strength (SBS) of the universal adhesives to dentin with and without phosphoric acid pre-etching was measured by a notched-edge test as described in International Organization for Standardization (ISO) 29022 (14). Bovine mandibular incisors were extracted from 2–3 year old cattle and stored frozen (–20°C) for up to 2 weeks. Roots were cut off using a slow-speed saw equipped with a diamond-impregnated disk (Isomet, Buehler, Lake Bluff, IL, USA) before the pulps were removed. Subsequently, each tooth pulp chamber was filled with cotton to avoid penetration of the embedding media. The labial surfaces were wet ground with 240-grit silicon carbide (SiC) paper (Struers, Cleveland, OH, USA) to create a flat dentin surface. Excess debris was eliminated by ultrasonic cleaning for 30 s in distilled water and surfaces were washed and dried using a dental three-way syringe at a distance of 5 cm above the surface at air pressure of approximately 0.37 MPa. Each tooth was then mounted in self-curing acrylic resin (Tray Resin II, Shofu Inc., Kyoto, Japan) and placed under water to limit the temperature rise caused by the exothermic polymerization of the acrylic resin. The dentin bonding surfaces were ground flat using a grinder-polisher (Ecomet 4, Buehler Inc.) and a sequence of SiC papers of grit sizes of #180 and #320, as specified by ISO 29022. These surfaces were then washed and dried using a dental three-way syringe. Some dentin surfaces were prepared by phosphoric acid etching for 15 s before adhesive application (with pre-etching), while surfaces without phosphoric acid pre-etching were also prepared (without pre-etching). These procedures were conducted under ambient conditions of 23 ± 2ºC at 50 ± 10% relative humidity.

3. SBS test

SBSs were evaluated using a Shear Bond Test Kit (Ultradent Product Inc.). Adhesives were applied to dentin surfaces according to the manufacturers' instructions before photoirradiation. Each adhesive was photoirradiated at standardized distance of 2 mm with a quartz-tungsten halogen unit (Optilux 501, Kerr, Orange, CA, USA) with various combinations of light intensity and photoirradiation time to achieve a constant energy density of 4,000 mJ/cm^2.

The quartz-tungsten halogen unit was connected to a variable voltage transformer. Light intensities of 100, 200, 400 and 600 mW/cm^2 were established using a radiometer (model 100, Kerr). Three of these were used for the adhesives, under photoirradiation conditions of 100 mW/cm^2 for 40 s (100 mW/cm^2), 200 mW/cm^2 for 20 s (200 mW/cm^2), and 400 mW/cm^2 for 10 s (400 mW/cm^2). Each condition was applied to 30 specimens from the groups described earlier. After application of the adhesive to the bonding sites, plastic moulds (Bonding Mold Insert, Ultradent Product Inc.) were clamped to the fixture (Bonding Clamp, Ultradent Product Inc.) against the dentin surfaces and filled with resin composite using a condenser. The resin composite was subsequently photoirradiated for 30 s at standardized distance of 2 mm at a light intensity of 600 mW/cm^2. The plastic mould was removed and the finished specimens were transferred to distilled water and stored at 37°C for 24 h. Each group of 30 specimens was randomly split into two groups (n = 15 per group): (1) no thermal cycling (24 h group); (2) 30,000 thermal cycles between 5 and 60ºC (TC group). Thermal cycling was conducted using a thermal shock tester (TTS-1 LM, Thomas Kagaku Corp., Tokyo, Japan). Each cycle consisted of water bath incubation for 30 s, with a transfer time of 5 s. Measurements were performed using a universal testing machine (5500R, Instron, Norwood, MA, USA) equipped with a shearing fixture (Crosshead Assembly, Ultradent Product Inc.) at a crosshead speed of 1.0 mm/min. The SBSs (MPa) were calculated by dividing the peak load at failure by the bonding area. After testing, the specimens were examined by optical microscopy (SZH-131, Olympus Corp., Tokyo, Japan) at a magnification of ×10 to determine the type of the failure. The proportions of the resin composite surface with adherent dentin and visible residues were estimated to classify the failure as adhesive failure, cohesive failure in dentin, cohesive failure in resin composite or mixed failure (a combination of adhesive and cohesive failure).

4. Water contact angle measurement

Mandibular incisors from cattle were prepared as described above (SBS tests), and adhesives were applied to the dentin surfaces with or without pre-etching according to the manufacturers' instruction before photoirradiation. Each adhesive was photoirradiated at a standardized distance of 2 mm and at 100, 200 and 400 mW/cm^2 to achieve a constant amount of light energy of 4,000 mJ/cm^2. The equilibrium water contact angle was measured by the sessile drop method under ambient conditions of 23 ± 2ºC at 50 ± 10% relative humidity using a contact angle measurement apparatus (DM 500, Kyowa Interface Science Corp., Saitama, Japan) for 10 specimens per photoirradiation condition. The apparatus was fitted with a charge-coupled device camera to enable automatic measurement. A standardized 3.0 μL drop of distilled water was placed on the cured adhesive surface and a profile image was captured after 500 ms using the apparatus. Water contact angles were then calculated by θ/2 method using the built-in interface measurement and analysis system (FAMAS, Kyowa Interface Science Corp.).

5. Scanning electron microscopy (SEM) observation of fracture surfaces

Ultrastructural observations of representative fracture sites after SBS tests using SEM (TM3000 Tabletop Microscope, Hitachi-High Technologies Corp., Tokyo, Japan) were carried out. SEM specimens of debonded specimens after SBS tests in each group (n = 3) were coated with a thin film of gold in a vacuum evaporator (Emitech SC7620 Mini Sputter Coater, Quorum Technologies Ltd., Ashford, UK). SEM observations were carried out using an operating voltage of 15 kV.

6. SEM observation of resin-dentin interfaces

Ultrastructural observations of representative resin-dentin interfaces were made with a field emission SEM (ERA 8800FE, Elionix Inc., Tokyo, Japan). Bonded specimens of each group (n = 3) were stored in distilled water at 37°C for 24 h, embedded in self-curing epoxy resin (Epon

812, Nisshin EM Corp., Tokyo, Japan), and then stored at 37°C for a further 24 h. These specimens were sectioned along the resin composite post diameter and the surfaces of the cut halves were successively polished with #180, #320, #600, #1,200, #2,000 and #4,000 SiC paper using a grinder-polisher. The surface was finally polished with a soft cloth using 1.0-μm-grit diamond paste (Struers). SEM specimens were dehydrated by immersion in ascending concentrations of aqueous tert-butanol (50% for 20 min, 75% for 20 min, 95% for 20 min, and 100% for 2 h), and were then transferred to a critical-point dryer (Model ID-3; Elionix Inc., Tokyo, Japan) for 30 min. These polished surfaces were etched for 30 s using an argon ion-beam (Type EIS-200ER, Elionix Inc.) directed perpendicularly to the surface at an accelerating voltage of 1.0 kV and an ion current density of 0.4 mA/cm^2. This treatment enhances the visibility of the layers in the interface (15). Surfaces were coated with a thin film of gold in a vacuum evaporator (Quick Coater Type SC-701, Sanyu Electron Corp., Tokyo, Japan) and were observed using a field emission SEM using an operating voltage of 10 kV.

7. Statistical analysis

Three-way ANOVA and Tukey's post hoc tests were used for analysis of SBS and water contact angle data. Fisher's exact test was used to statistically analyze the failure modes. All statistical analyses were conducted using a commercial statistical software package (SPSS Statistics Base, International Business Machines, Armonk, NY, USA) at a significance level of 0.05.

Results

1. SBSs

The SBSs of universal adhesives to dentin for all the groups are shown in Tables 2 and 3. The three-way ANOVA revealed that photoirradiation conditions had a significant influence on SBSs, unlike the adhesive type and pre-etching. The interaction between photoirradiation conditions and adhesive type was also more significant than that among others. Regardless of pre-etching, universal adhesives presented higher SBSs in the 24 h group at light intensities at 200 and 400 mW/cm^2 (24.4–30.1 MPa) than at 100 mW/cm^2 (17.2–20.2 MPa) for the same energy density. Similarly, SBSs of the TC group at light intensities at 200 and 400 mW/cm^2 (21.5–25.5 MPa) exceeded those at 100 mW/cm^2 (9.2–13.2 MPa) for the same energy density.

2. Failure mode analysis of the de-bonded specimens

The failure mode analysis of debonded specimens of 24 h and TC groups is shown in Table 4. Fisher's exact test revealed no significant differences in failure mode among photoirradiation conditions, adhesive type, and presence or absence of pre-etching, and the predominant failure mode in all groups was adhesive failure.

3. Water contact angles

Water contact angles with universal adhesives on dentin with and without pre-etching photoirradiated under different conditions are shown in Table 5. The three-way ANOVA revealed that photoirradiation conditions and adhesive type significantly affected the water contact angle of universal adhesives, unlike pre-etching. In addition, the interaction between photoirradiation conditions and adhesive type was more significant than those among others. Universal adhesives exhibited significantly higher water contact angle when photoirradiated at 200 and 400 mW/cm^2 than at 100 mW/cm^2. They also displayed different water contact angels depending on adhesive type.

4. SEM observations of fracture surfaces

Representative SEM images of debonded specimens of the TC group after bond strength tests are shown in Fig. 1 (the images of the 24 h group were similar). The debonded specimens showed predominantly adhesive failure, and debonding at the interface between dentin and the adhesive can be clearly observed regardless of the photoirradiation conditions and pre-etching. In the SEM images of fracture surfaces of the specimens photoirradiated at 100 mW/cm^2, large numbers of voids were observed when compared to specimens photoirradiated at 200 and 400 mW/cm^2 regardless of pre-etching.

5. SEM observations of resin-dentin interfaces

Representative SEM images of resin-dentin interfaces are shown in Fig. 2. These interfaces showed excellent adaptation regardless of the photoirradiation conditions or pre-etching. Specimens photoirradiated at 100 mW/cm^2 exhibited a thinner adhesive layer than those photoirradiated at 200 and 400 mW/cm^2.

Discussion

Regardless of pre-etching, the SBSs of universal adhesives to dentin in both the 24 h and TC groups were higher for those at 200 and 400 mW/cm^2 than for those irradiated at 100 mW/cm^2, even with the same energy density. A previous study reported that hydrophobicity of the adhesive is essential for durable bonds, and that incomplete polymerization leads to interface degradation and decreased dentin bond durability (16). Incomplete polymerization of adhesives can accelerate water degradation effects, leading to bond deterioration (17). In the results of the water contact angle with the cured adhesive under different photoirradiation conditions, universal adhesives photoirradiated at light intensities of 100 mW/cm^2 presented significantly lower water contact angles than those photoirradiated at 200 and 400 mW/cm^2, indicating their greater hydrophilicity. This higher hydrophilicity indicated the incomplete polymerization of the adhesive at 100 mW/cm^2, even at a constant energy density of 4,000 mJ/cm^2.

Previous studies reported that differences in photoirradiation conditions affect the kinetics of polymerization (18). Monomers in adhesives form into a highly crosslinked network upon polymerization, reducing the hydrophilicity of the adhesive (13). The complete polymerization of monomers present in universal adhesives is thought to be expected to lower the hydrophilicity of the adhesive. Therefore incomplete polymerization of the adhesives at 100 mW/cm^2 might be one of the reasons why weaker dentin bond durability was observed at 100 mW/cm^2 compared to those at 200 and 400 mW/cm^2, regardless of the adhesive type and pre-etching. In the SEM observations of fracture surfaces of debonded specimens at 100 mW/cm^2 in the TC group, large numbers of voids were observed, but they were not seen in observations of the specimens at 200 and 400 mW/cm^2 regardless of pre-etching. A previous study which investigated the relationship between degree of conversion of adhesive and bond strength reported that a low degree of conversion of adhesives increases permeability and induces hydrolysis of the adhesive layer (19).

Consequently, the observed large numbers of voids with the SEM images might be related to incomplete polymerization of the adhesives, indicating weak points of the specimens at 100 mW/cm^2 with lower bond durability. In addition, SEM observations of the resin-dentin interface showed that the adhesive layer was considerably thinner under photoirradiation at 100 mW/cm^2 than at 200 and 400 mW/cm^2 regardless of pre-etching. This also suggested that lower intensity photoirradiation (below 100 mW/cm^2) limits the polymerization process, creating a thicker uncured adhesive potion. This largely unpolymerized layer might be displaced upon resin composite application, leading to the thinner adhesive layer observed. A previous study reported that the thickness of universal adhesives might be around 10 µm, and insufficient thickness of the adhesive layer has been seen as contributing to lower bonding performance (20). Therefore, the inadequate thickness of the adhesive layer at 100 mW/cm^2 observed in this study might impair the bond durability of universal adhesives. The results of the SBS test and SEM observation of fracture surfaces and adhesives interfaces suggest that differences in photoirradiation conditions at constant total energy influence the bond durability of universal adhesive.

Although the results of SBSs of universal adhesives to dentin of in both the 24 h and TC groups showed different tendencies among the different photoirradiation conditions, there were no significant differences in failure type depending on the photoirradiation conditions, storage conditions, type of adhesive, or pre-etching, and adhesive failures were observed most frequently. A previous study has stated that an increased incidence of adhesive failure in bond strength tests was desirable to provide more relevant information about the bond strength (21). Therefore, in this study, the type of fracture observed may provide evidence for the clinical relevance of the measured shear bond strengths of universal adhesives under different photoirradiation conditions.

Overall, when augmenting light intensity while maintaining the same energy density, the water contact angle and thickness of universal adhesives was increased due to the enhanced polymerization of these adhesives. Sufficient light intensity might enhance the polymerization reaction of universal adhesives, leading to greater dentin bond durability of the restorations. On the other hand, universal adhesives with photoirradiated at 100 mW/cm^2 exhibited higher hydrophilicity, leading to inferior bond durability. Therefore the null hypothesis, different photoirradiation conditions achieving an energy density of 4,000 mJ/cm^2, would not affect the dentin bond durability or interfacial characteristics of universal adhesives was rejected. The results of this study indicated that the photoirradiation conditions affect the dentin bond durability and interfacial characteristics of universal adhesives even at the same total energy.

Conclusion

1. Dentin bond durability of universal adhesives were higher for those at 200 and 400 mW/cm^2 than for those photoirradiated at 100 mW/cm^2, even with the same energy density.
2. The water contact angle with the cured universal adhesive photoirradiated at light intensities of 100 mW/cm^2 was lower than those photoirradiated at 200 and 400 mW/cm^2 regardless of pre-etching.
3. SEM observations of fracture surfaces after SBS tests showed that large numbers of voids were observed for the specimens photoirradiated at 100 mW/cm^2 than at 200 and 400 mW/cm^2 regardless of pre-etching.
4. SEM observations of the resin-dentin interface showed that the adhesive layer was considerably thinner under photoirradiation at 100 mW/cm^2 than at 200 and 400 mW/cm^2 regardless of pre-etching.

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Tables and Figures

Table 1 Materials used in this study			
Material (Lot No.)	**Type of Adhesive (Code)**	**Main components**	**Manufacturer**
Adhese Universal (165543)	Universal Adhesive (AU)	Bis-GMA, HEMA, MDP, MCAP, Decandiol dimethacrylate, Dimethacrylate, Ethanol, Water, Initiators, Stabilizers, Silicon dioxide	Ivoclar Vivadent, Schaan, Lichtenstein
All-Bond Universal (1312131)	Universal Adhesive (AB)	MDP, Bis-GMA, HEMA, Ethanol, Water, Initiator, Silanated colloidal silica	Bisco, Schaumburg, IL, USA
G-Premio Bond (541424)	Universal Adhesive (GP)	MDP, 4-MET, MEPS, Methacrylate monomer, Acetone, Water, Initiator, Silica	GC Corp., Tokyo, Japan
Scotchbond Universal (566724)	Universal Adhesive (SU)	Bis-GMA, HEMA, MDP, Vitrebond copolymer, Polythelene glycol, Water, Initiators, Silica	3M ESPE, St. Paul, MN, USA
Ultra-Etch (G019)	Pre-etching agent	35% phosphoric acid, Glycol, Cobalt aluminate blue spinel	Ultradent Products Inc., South Jordan, UT, USA
Clearfil AP-X (Shade: A2, 1312131)	Resin composite	Bis-GMA, TEGDMA, Silanated barium filler, Silanated colloidal silica, *dl*-camphorquinone, Catalysts, Accelerators, Pigments, Others	Kuraray Noritake Dental Inc., Tokyo, Japan

Bis-GMA: 2,2-bis[p-(2-hydroxy-3-methacryloxy propoxy)phenyl]propane; HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; MCAP: methacrylated carboxylic acid polymer; 4-MET: 4-methacryloyl- oxyethyl trimellitate; MEPS: methacryloyloxyalkyl thiophosphate methylmethacrylate; TEGDMA: triethylene glycol dimethacrylate.

Table 2: Shear bond strengths of universal adhesives to dentin with phosphoric acid pre-etching photoirradiated under different photoirradiation conditions

Code	Storage condition	Photoirradiation condition		
		100 mW/cm^2 x 40 s	200 mW/cm^2 x 20 s	400 mW/cm^2 x 10 s
AU	24 h	17.4 (5.5)^{a,A}	24.7 (4.2)^{a,b,B}	25.5 (4.4)^{a,b,B}
	TC	10.7 (4.4)^{b,,c,A}	22.5 (4.8)^{a,B}	23.9 (3.3)^{a,B}
AB	24 h	17.9 (3.2)^{a,A}	25.5 (4.1)^{a,b,B}	26.1 (4.3)^{a,b,B}
	TC	10.9 (3.2)^{b,c,A}	21.5 (4.1)^{a,B}	24.4 (4.5)^{a,B}
GP	24 h	18.5 (3.5)^{a,A}	26.5 (3.2)^{b,B}	27.2 (3.9)^{a,b,B}
	TC	9.2 (3.1)^{b,A}	22.5 (4.2)^{b,B}	23.2 (3.4)^{a,B}
SU	24 h	20.2 (3.2)^{a,A}	28.5 (3.7)^{b,B}	30.1 (4.0)^{b,B}
	TC	13.2 (3.2)^{c,A}	25.5 (3.7)^{a,b,B}	25.1 (3.0)^{a,b,B}

Unit: MPa. Values in parentheses are standard deviations. Same small letter in same individual column indicates no significant difference ($p > 0.05$). Same capital letter within individual rows indicates no significant difference ($p > 0.05$).

Table 3: Shear bond strengths of universal adhesives to dentin without phosphoric acid pre-etching photoirradiated under different photoirradiation conditions

Code	Storage condition	Photoirradiation condition		
		100 mW/cm^2 x 40 s	200 mW/cm^2 x 20 s	400 mW/cm^2 x 10 s
AU	24 h	17.2 (4.6)^{a,A}	24.5 (5.1)^{a,B}	24.4 (5.5)^{a,B}
	TC	10.8 (5.1)^{b,A}	22.4 (4.8)^{a,B}	23.2 (4.4)^{a,B}
AB	24 h	18.7 (4.1)^{a,A}	26.5 (5.1)^{a,B}	27.1 (4.5)^{a,B}
	TC	12.7 (4.1)^{b,A}	23.2 (5.1)^{a,B}	24.4 (4.5)^{a,B}
GP	24 h	17.9 (3.7)^{a,A}	25.1 (4.2)^{a,B}	26.5 (4.3)^{a,B}
	TC	11.1 (3.2)^{a,A}	23.6 (3.5)^{a,B}	24.5 (4.2)^{a,B}
SU	24 h	19.2 (4.2)^{a,A}	27.3 (4.4)^{a,B}	27.9 (3.8)^{a,B}
	TC	11.2 (3.4)^{b,A}	24.3 (4.4)^{a,B}	25.2 (3.1)^{a,B}

Unit: MPa. Values in parentheses are standard deviations. Same small letter in same individual column indicates no significant difference ($p > 0.05$). Same capital letter within individual rows indicates no significant difference ($p > 0.05$).

Table 4: Failure mode analysis of debonded specimens					
Code	**Pre-etching**	**Storage condition**	**Photoirradiation condition**		
			100 mW/cm^2 x 40 s	**200 mW/cm^2 x 20 s**	**400 mW/cm^2 x 10 s**
AU	with	24h	[13/0/0/2]	[12/0/1/2]	[13/0/0/2]
		TC	[15/0/0/0]	[15/0/0/0]	[12/1/1/1]
	without	24h	[13/1/0/1]	[14/0/0/1]	[12/1/1/1]
		TC	[15/0/0/0]	[15/0/0/0]	[15/0/0/0]
AB	with	24h	[14/0/0/1]	[15/0/0/0]	[14/0/0/1]
		TC	[15/0/0/0]	[14/0/0/1]	[15/0/0/0]
	without	24h	[14/1/0/0]	[13/0/1/1]	[12/1/1/1]
		TC	[14/1/0/0]	[14/0/0/1]	[13/2/0/0]
GP	with	24h	[15/0/0/0]	[13/0/0/2]	[15/0/0/0]
		TC	[15/0/0/0]	[15/0/0/0]	[15/0/0/0]
	without	24h	[15/0/0/0]	[14/0/0/1]	[13/1/0/1]
		TC	[13/0/0/2]	[14/0/0/1]	[12/2/0/1]
SU	with	24h	[13/0/0/2]	[12/0/1/2]	[13/0/0/2]
		TC	[15/0/0/0]	[15/0/0/0]	[12/1/1/1]
	without	24h	[13/1/0/1]	[14/0/0/1]	[12/1/1/1]
		TC	[15/0/0/0]	[15/0/0/0]	[15/0/0/0]
[ ] indicates failure mode [adhesive failure/cohesive failure in substrate/cohesive failure in resin/mixed failure].					

Table 5: Water contact angles of universal adhesives with and without phosphoric acid pre-etching under different photoirradiation conditions

Code	Pre-etching	Photoirradiation condition		
		100 mW/cm² x 40 s	200 mW/cm² x 20 s	400 mW/cm² x 10 s
AU	with	39.4 (2.8)^{a,A}	48.2 (3.4)^{a,b,B}	50.1 (3.2)^{a,b,B}
	without	39.3 (2.2)^{a,A}	49.2 (3.2)^{a,b,B}	50.3 (4.4)^{a,b,B}
AB	with	42.1 (2.1)^{b,A}	52.1 (2.2)^{a,B}	53.2 (2.0)^{a,B}
	without	42.9 (3.0)^{b,A}	53.2 (2.9)^{a,B}	53.4 (2.2)^{a,B}
GP	with	37.5 (2.1)^{a,A}	47.1 (2.5)^{b,B}	48.3 (2.1)^{b,B}
	without	36.5 (2.7)^{a,A}	46.3 (2.1)^{b,B}	47.4 (1.9)^{b,B}
SU	with	40.5 (2.2)^{b,A}	49.9 (3.1)^{a,b,B}	50.6 (3.0)^{a,b,B}
	without	39.7 (2.4)^{b,A}	49.5 (2.8)^{a,b,B}	51.3 (3.1)^{a,b,B}

Unit: °. Values in parentheses are standard deviations. Same small letter in same column indicates no significant difference ($p > 0.05$). Same capital letter within same rows indicates no significant difference ($p > 0.05$).

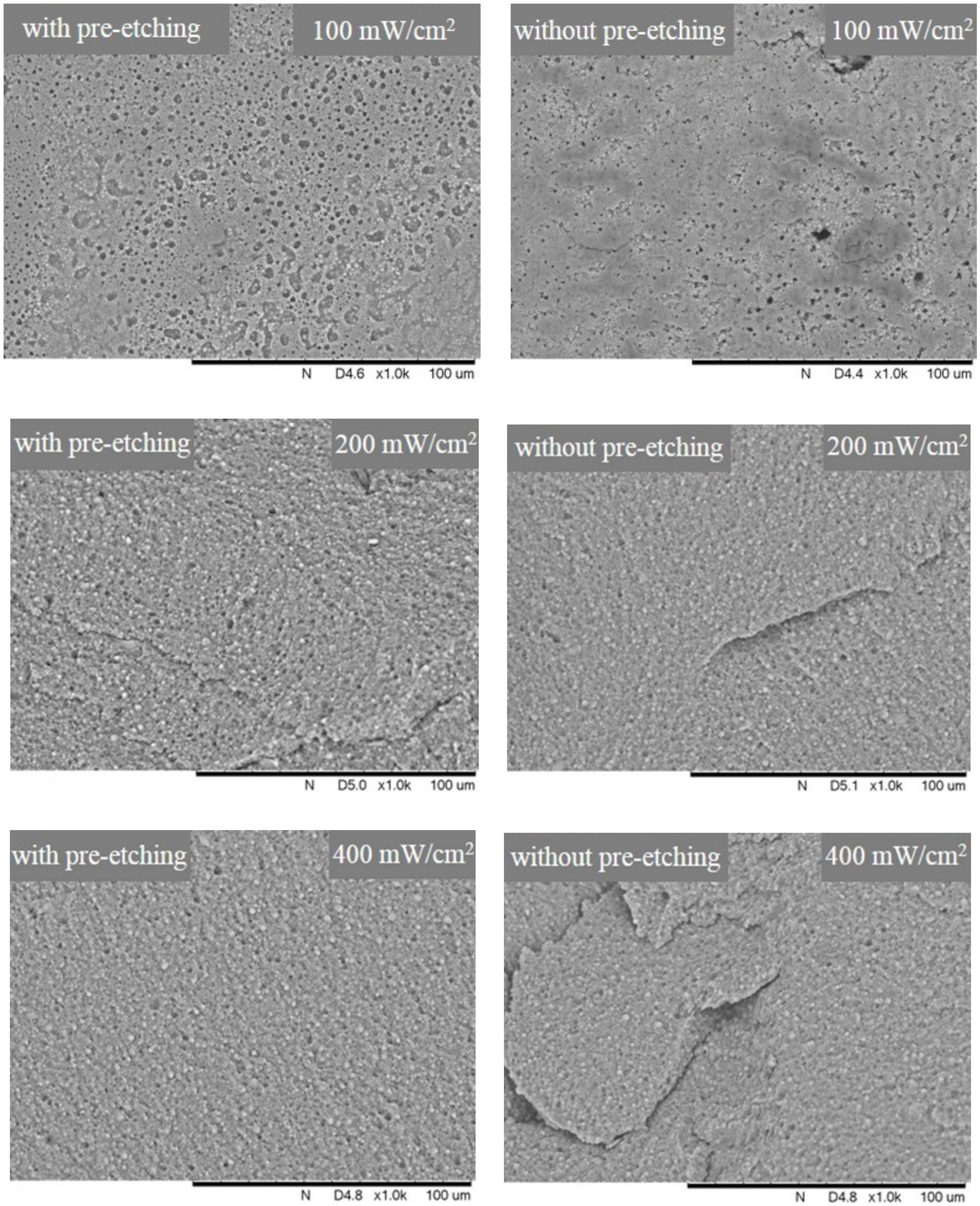

Fig. 1: Representative SEM images of debonded specimens after SBS tests at 1,000× magnification.

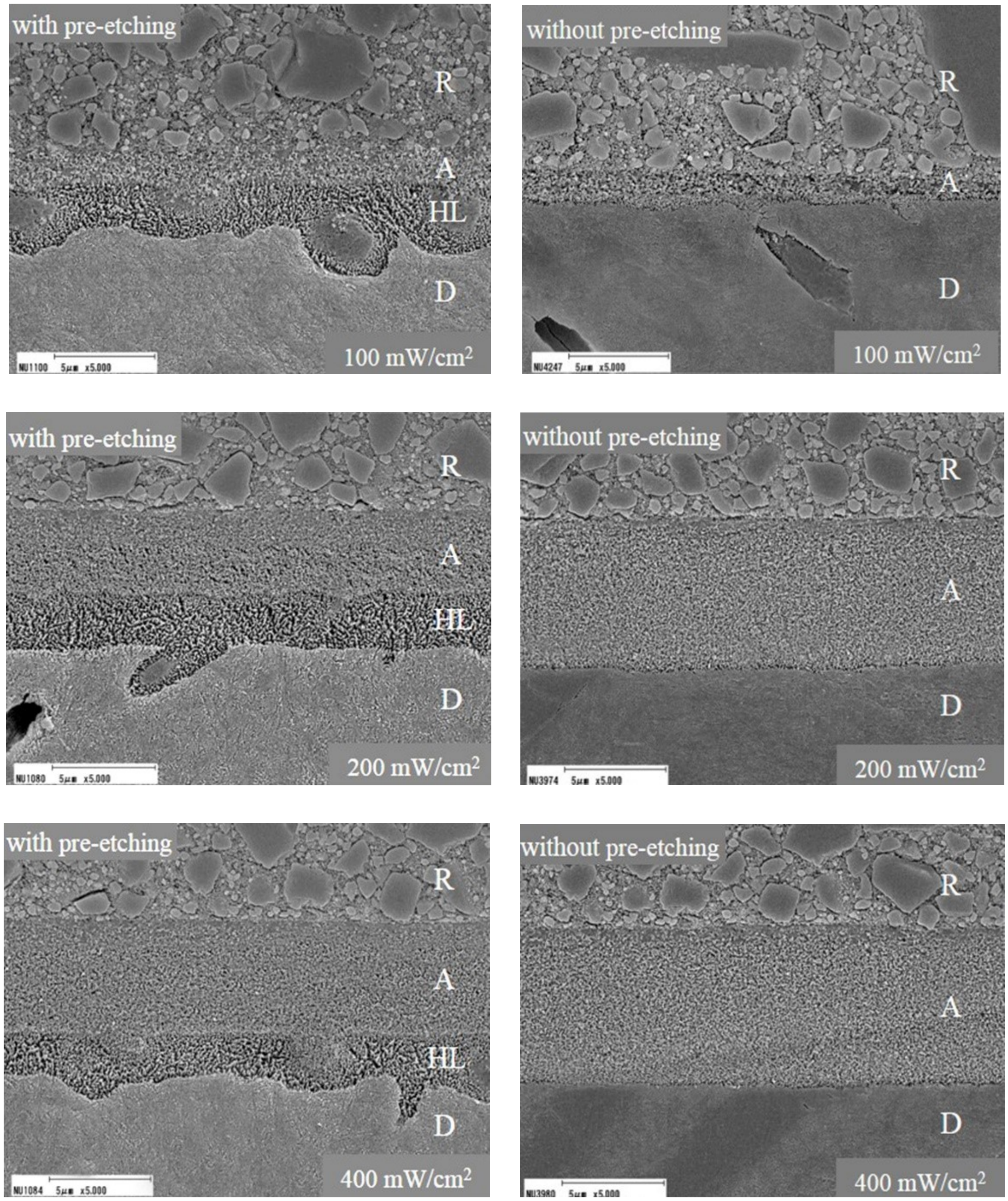

Fig. 2: Representative field-emission SEM images of the resin-dentin interface at 5,000× magnification. R: resin composite; A: adhesive layer; HL: hybrid layer; D: dentin.