©Operative Dentistry, 2020, **45-2,** 196-208

The Effects of Aluminablasting on Bond Durability Between Universal Adhesives and Tooth Substrate

H Ouchi • T Takamizawa • K Tsubota • A Tsujimoto
A Imai • WW Barkmeier • MA Latta • M Miyazaki

Clinical Relevance

Aluminablasting may adversely affect the dentin bond strength of universal adhesives in self-etch mode.

SUMMARY

The aim of this study was to determine the effect of aluminablasting on the bond durability of universal adhesives and adherent surface characteristics. Adhese Universal (Ivoclar Vivadent), All-Bond Universal (Bisco), Bondmer Lightless (Tokuyama Dental), G-Premio Bond (GC), and Scotchbond Universal (3M ESPE) were used in self-etch mode. The prepared bovine enamel and dentin specimens were divided into two groups based on whether they received an aluminablasting prior to application of the universal adhesives. The resin composite bonded specimens were stored in distilled water at 37°C for 24 hours, following which the shear bond strength (SBS) of half of the specimens was measured (24-hour group). The other half was subjected to 30,000 thermal cycles between 5 and 55°C before SBS measurement (TC group). Surface roughness (Ra) and surface free energy (SFE) of the adherent surfaces were also measured, and scanning electron microscopy observation and scanning electron microscopy/energy-dispersive X-ray analysis were carried out. Most of the adhesives did not show any significant differences in enamel SBS values between the two pretreatment groups, regardless of the storage condition. However, the dentin SBS values were significantly lower in specimens that underwent aluminablasting compared with those that did not, irrespective of their storage conditions. Significantly higher Ra and SFE values were observed in the enamel and dentin of specimens that underwent alu-

Hajime Ouchi, DDS, Nihon University School of Dentistry, Operative Dentistry, Tokyo, Japan

*Toshiki Takamizawa, DDS, PhD, Nihon University School of Dentistry, Operative Dentistry, Tokyo, Japan

Keishi Tsubota, DDS, PhD, Nihon University School of Dentistry, Operative Dentistry, Tokyo, Japan

Akimasa Tsujimoto, DDS, PhD, Nihon University School of Dentistry, Operative Dentistry, Tokyo, Japan

Arisa Imai, DDS, Nihon University School of Dentistry, Operative Dentistry, Tokyo, Japan

Wayne W Barkmeier, EBM, DDS, MS, Creighton University School of Dentistry, General Dentistry, Omaha, NE, USA

Mark A Latta, DMD, MS, Creighton University, General Dentistry, Omaha, NE, USA

Masashi Miyazaki, DDS, PhD, Nihon University School of Dentistry, Operative Dentistry, Tokyo, Japan

*Corresponding author: 1-8-13, Kanda-Surugadai, Chiyoda-Ku, Tokyo, 101-8310 Japan; e-mail: takamizawa.toshiki@nihon-u.ac.jp

DOI: 10.2341/18-170-L

minablasting. Although aluminablasting increased the Ra and SFE values of enamel and dentin, its effect on the SBS value was dependent on the tooth substrate. In addition to C, O, Na, Mg, P, and Ca, the element Al was detected in the enamel and dentin of samples that had undergone aluminablasting. These results suggest that although aluminablasting of the tooth surface is thought to be effective for modification of the adherent surface, it may not enhance enamel bond performance and may also adversely affect the dentin bond effectiveness of the universal adhesives.

INTRODUCTION

It is inevitable that restorations in the oral cavity will partially fracture or develop recurrent caries around their margins over time. In such cases, the surrounding sound tooth structures can be preserved by adopting a minimally invasive approach, which advocates the use of composite resins to repair defective or aged restorations.[1] The tooth cavity is typically made up of more than one material and exhibits a complicated configuration, making it desirable that the resin composites bond effectively to all of these materials. Several procedures, including phosphoric acid etching, airborne particle abrasion, and the use of different primers on the aged restorations prior to application of the adhesive, have been recommended for the achievement of durable bonds between different substrates.[2-4] However, these pretreatments require additional clinical steps, and it may be difficult to apply pretreatments on just the aged restorations or the tooth substrates of the cavity. Moreover, contamination of the tooth substrate by metal primers or silane coupling agents may negatively affect the bond strength between the substrate and resin composite.[5,6]

Universal adhesives have distinctive characteristics and versatility when compared with the previous generations of adhesive systems.[7] Different etching techniques, including self-etch, etch and rinse, or selective etching, can be used in direct composite resin restorations with universal adhesives.[8,9] Furthermore, universal adhesives have also simplified bonding procedures considerably as they contain various functional monomers that allow them to bond to surfaces other than tooth substrates.[10]

Airborne particle abrasion has been used extensively in various dental treatments, including stain removal, cavity preparation, and inner surface modification of indirect restorations.[11,12] The material and size of the abrasion particles depend on the purpose of application. Aluminablasting of indirect restorations such as metal alloy, zirconia, and cured resin composites has been reported to be a reliable method of pretreatment,[11,13-15] and the resulting surface modifications can increase mechanical interlocking and modify the adhered surface.[16] In contrast, Loomans and others[2] suggested that none of the surface treatments for restoration repair can be recommended as a universally applicable repair technique for the different types of resin composites. Moreover, the effects of aluminablasting on the bond strength and surface characteristics of the restorations are still unknown, and there is a possibility that residual alumina particles and surface modifications of the teeth may negatively affect the chemical bond with the universal adhesives by interfering with the chemical reactions between the functional monomers and the hydroxyapatite (HAp).

The aim of this study was to examine the effects of aluminablasting enamel and dentin on the bonding performance and surface characteristics when using universal adhesives in self-etch mode. This was determined by testing the bond strength and surface free energy measurements of the adherent surfaces before and after thermal cycling. The null hypotheses tested were 1) the bond performance of universal adhesives would not be influenced by aluminablasting, 2) the surface characteristics of the adherent surface would be altered by aluminablasting, and 3) there would be no difference in the effects of aluminablasting between enamel and dentin.

METHODS AND MATERIALS

Study Materials

The materials used in this study are shown in Table 1. The five universal adhesives used were 1) All Bond Universal (ABU; Bisco, Schaumburg, IL, USA) 2), Adhese Universal (ADU; Ivoclar Vivadent, Schaan, Liechtenstein), 3) Bondmer Lightless (BML; Tokuyama Dental, Tokyo, Japan), 4) G-Premio Bond (GPB; GC, Tokyo, Japan), and 5) Scotchbond Universal (SBU; 3M ESPE, St. Paul, MN, USA). Clearfil AP-X (Kuraray Noritake Dental, Tokyo, Japan) was used as a restorative material for bonding to enamel and dentin. A halogen quartz tungsten curing unit (Optilux 501, sds Kerr, Danbury, CT, USA) at an average light irradiance of 600 mW/cm^2 was used.

Specimen Preparation

This study used extracted mandibular bovine incisors as a substitute for human teeth. The labial surfaces of the teeth were ground using wet #240-

Table 1: *Materials Used in This Study*

Code	Adhesive (Lot No.)	Main Components	Manufacturer
ABU	All-Bond Universal (1300008503)	MDP phosphate monomer, bis-GMA, HEMA, ethanol, water, initiators	Bisco Inc., Schaumburg, IL, USA
ADU	Adhese Universal (U49302)	MDP, bis-GMA, HEMA, MCAP, D3MA, ethanol, water, initiator stabilizers, silicon dioxide	Ivoclar Vivadent Schaan, Lichtenstein
BML	Bondmer Lightless (004067)	Liquid A: phosphate monomer, bis-GMA, TEGDMA, HEMA, MTU-6, others	Tokuyama Dental Corp., Tokyo, Japan
		Liquid B: acetone, isopropanol, water, acryl borate catalyst, γ-MPTES, peroxide, others	
GPB	G-Premio Bond (4G0011)	MDP, 4-MET, MEPS, BHT, acetone, dimethacrylate resins, initiators, water	GC Corp., Tokyo, Japan
SBU	Scotchbond Universal (41256)	MDP, HEMA, dimethacrylate resins, Vitrebond copolymer, filler, ethanol, water, initiators, silane	3M ESPE Dental Products, St. Paul, MN, USA
	Resin composite Clearfil AP-X (N416713)	bis-GMA, TEGDMA, silane barium glass filler, silane silica filler, silanated colloidal silica, CQ, pigments, others	Kuraray Noritake Dental, Tokyo, Japan

Abbreviations: 4-MET, 4-methacryloxyethyl trimellitate; BHT, butylated hydroxytoluene; bis-GMA, 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy) phenyl)] propane; CQ, dl-camphorquinone; D3MA, decandiol dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; MCAP, methacrylated carboxylic acid polymer; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; MEPS, methacryloyloxyalkyl thiophosphate methylmethacrylate; MTU-6, 6-methacryloyloxyhexyl-2-thiouracil-5-carboxylate; TEGDMA, triethyleneglycol dimethacrylate; γ-MPTES, γ-methacryloyloxypropyltriethoxysilane.

Table 2: *Protocols for Bonding Procedures*

Method Code	Pretreatment for Adherent Surface
w/o	Aluminablasting was not performed.
w	Aluminablasting was performed on the adherent surface with 50 μm Al_3O_2 powder for five seconds at an angle of 45° and a distance of 10 mm with pressure of 0.25 MPa. The treated surface was rinsed with water for 15 s (three-way dental syringe) and air dried.
Adhesive Code	**Adhesive Application Protocol**
ABU	Adhesive applied to air-dried enamel or dentin surface (not desiccated for dentin) with rubbing action for 10-15 s per coat. No light cure between coats. Gentle stream of air applied over the liquid for at least 10 s. Light irradiation performed for 10 s.
ADU	Adhesive applied to the air-dried enamel or dentin surface with rubbing action for 20 s and then medium air pressure applied to surface for 5 s. Light irradiate for 10 s.
BML	Adhesive applied to the air-dried enamel or dentin surface, and after 10 s, medium air pressure applied over the liquid adhesive for 5 s. No light irradiation.
GPB	Adhesive applied to air-dried enamel or dentin surface, and after 10 s, a strong stream of air applied over the liquid adhesive for 5 s or until the adhesive no longer moved and the solvent had completely evaporated. Light irradiate for 10 s.
SBU	Adhesive applied to air-dried enamel or dentin surface with rubbing action for 20 s medium air pressure applied to surface for 5 s. Adhesive light cured for 10 s.

Abbreviations: ABU, All-Bond Universal; ADU, Adhese Universal; BML, Bondmer Lightless; GPB, G-Premio Bond; SBU, Scotchbond Universal.

grit silicon carbide (SiC) paper (Fuji Star Type DDC, Sankyo-Rikagaku, Saitama, Japan) to create flat enamel and dentin surfaces. Each tooth was then mounted in self-curing acrylic resin (Tray Resin II, Shofu, Kyoto, Japan) to expose the flattened area, and the enamel and dentin adherent surfaces were polished using a water coolant and a sequence of SiC polishing papers (up to #320 grit; Fuji Star Type DDC).

Thermal Cycling and Shear Bond Strength Tests

The experimental protocol for tooth preparation is shown in Table 2. The prepared enamel and dentin specimens were divided into two groups depending on whether they received aluminablasting before the application of the universal adhesive or not. The aluminablasting was performed with a chairside sandblaster (MicroEtcher IIA, Zest Dental Solutions, Carlsbad, CA, USA). For each group, the adhesive agents were applied in accordance with each manufacturer's instructions for self-etch mode. Following application of the adhesive, bonded resin composite cylinders were built on the tooth surfaces by placing the resin composite in plastic molds (Bonding Mold Insert, internal diameter: 2.4 mm, height: 2.4 mm, Ultradent Products, South Jordan, UT, USA) and subjecting it to light irradiation for 30 seconds. The bonded specimens were then stored in distilled water at 37°C for 24 hours, following which the shear bond strength (SBS) was measured in half of the specimens (24-hour group). The remaining half were treated with 30,000 thermal cycles between 5 and 55°C and a dwell time of 30 seconds prior to SBS measurement (thermal cycling [TC] group). The SBSs were measured using the notched edge SBS test, as described by ISO 29022.[17]

There were 40 experimental groups in total, arising from four variables: enamel or dentin, with or without aluminablasting, five different universal adhesives, and 24-hour or thermal cycling. Fifteen specimens were used for each group, as this number gives a statistical power of 0.95 with an $\alpha = 0.05$ for typical SBS values, as calculated using statistical power analysis. A total of 600 specimens were used.

The bonded specimens were loaded to failure at 1.0 mm/min with an Ultradent shearing fixture (Test Base Clamp, Ultradent Products) using a universal testing machine (Type 5500R, Instron, Canton, MA, USA), and the SBS values (MPa) were obtained. Thereafter, the bonding sites on the tooth surfaces and resin composite cylinders were observed under an optical microscope (SZH-131, Olympus, Tokyo, Japan) at a magnification of 10× to determine the bond failure type. Based on the proportion of the substrate area (adhesive–resin composite–enamel or dentin) observed in the debonded cylinders and tooth-bonding sites, the bond failures were classified into 1) adhesive failure, 2) cohesive failure in composite resin, 3) cohesive failure in tooth, or 4) mixed failure (partially adhesive and partially cohesive).

Surface Roughness Measurements

The treated surfaces with or without aluminablasting were observed under a three-dimensional laser scanning microscope (LSM; VK-8700; Keyence, Osaka, Japan) with the excitation light at a spectral maximum of 658 nm and the light intensity and amplification of the photomultiplier being kept constant during the observation period. The surface roughness (Ra) values were measured using the software (VK-Analyzer; Keyence) included with the LSM, and each region of measurement was 0.1 mm × 0.1 mm in size. The Ra was measured at three points as close to the center as possible in 10 specimens from each group, and the mean value was calculated for each group thereafter.

Surface Free Energy Measurements

The surface free energy (SFE) of treated surfaces with or without aluminablasting was determined by measuring the contact angles formed on the surfaces of three test liquids: 1-bromonaphthalene, diiodomethane, and distilled water. The SFE parameters of these liquids have been reported previously.[18,19] The contact angles were measured automatically using a contact angle meter (Drop Master DM 500, Kyowa Interface Science, Saitama, Japan) that had been connected to a charge-coupled device camera. The equilibrium contact angle (θ) was measured in 10 enamel and dentin specimens for each test liquid. Sessile drops (1.0 μL in volume) of each liquid were dispensed at 23°C ± 1°C using a micropipette, and the SFE parameters of the solids were calculated based on the fundamental concepts of wetting. The Young-Dupré equation describes the adhesion between a solid (S) and liquid (L) that are in contact (W_{SL}), the interfacial free energy between the solid and the liquid (γ_{SL}), and the SFE of the liquid and solid (γ_L and γ_S, respectively), as follows:

$$W_{SL} = \gamma_L + \gamma_S - \gamma_{SL} = \gamma_L(1 + \cos\theta).$$

The Fowkes equation can be extended using the Kitazaki-Hata approach, as follows[20]:

$$\gamma_{SL} = \gamma_L + \gamma_S - 2(\gamma_L^d \gamma_S^d)^{1/2} - 2(\gamma_L^p \gamma_S^p)^{1/2} - 2(\gamma_L^h \gamma_S^h)^{1/2}$$

$$\gamma_L = \gamma_L^d + \gamma_L^p + \gamma_L^h, \quad \gamma_S = \gamma_S^d + \gamma_S^p + \gamma_S^h,$$

where γ^d, γ^p, and γ^h are the dispersion force, polar (permanent and induced) force, and hydrogen-bonding force, respectively, and are components of the SFE (γ). θ values were determined for all three test liquids, and the surface-energy parameters of the treated surfaces were calculated according to the equations using an add-on software and the interface measurement and analysis system (FAMAS; Kyowa Interface Science). A statistical power analysis indicated that a minimum of nine samples was necessary for effective measurement of the Ra and SFE. Therefore, this experiment included a sample of 10 specimens, and post hoc power tests performed after data collection indicated that this would result in adequate statistical power.

Scanning Electron Microscopy Observation

The aluminablasted surfaces and tooth/resin interfaces were observed using field-emission scanning electron microscopy (SEM; ERA-8800FE, Elionix, Tokyo, Japan). For ultrastructural morphological observations of the tooth-resin interfaces, the bonded specimens were embedded in epoxy resin and longitudinally sectioned using a diamond saw (Iso-

Table 3: *Influence of Aluminablasting on Enamel Bond Strength*[a]

	24-h		30,000 TC	
	w/o	With	w/o	With
ABU	27.9 (2.7)^{aA} [100%]	26.8 (4.2)^{aA} [96.1%]	27.6 (3.0)^{bA} [98.9%]	25.8 (2.0)^{aA} [92.5%]
ADU	26.8 (1.8)^{aA} [100%]	26.9 (2.0)^{aA} [100.3%]	27.5 (3.5)^{bA} [102.6%]	28.2 (3.1)^{aA} [102.5%]
BML	29.0 (3.8)^{aA} [100%]	30.8 (3.6)^{aA} [106.2%]	30.2 (4.2)^{abA} [104.1%]	28.8 (3.4)^{aA} [99.3%]
GPB	28.2 (4.6)^{aAB} [100%]	27.0 (4.1)^{aB} [95.6%]	32.9 (4.5)^{aA} [116.7%]	29.5 (3.9)^{aAB} [104.6%]
SBU	28.1 (3.3)^{aA} [100%]	27.2 (4.2)^{aA} [96.8%]	27.4 (3.2)^{bA} [97.5%]	26.6 (3.4)^{aA} [94.7%]

Abbreviations: ABU, All-Bond Universal; ADU, Adhese Universal; BML, Bondmer Lightless; GPB, G-Premio Bond; SBU, Scotchbond Universal.
[a] N=15, mean (SD) in MPa. The same lowercase letter in vertical columns indicates no difference at the 5% significance level. The same uppercase letter in horizontal rows indicates no difference at the 5% significance level. Values in parentheses indicate standard deviation.

Met 1000, Precision Sectioning Saw, Buehler, Lake Bluff, IL, USA). The sectioned surfaces were polished to a high gloss using SiC papers (Fuji Star Type DDC) followed by diamond pastes up to a particle size of 0.25 μm (DP-Paste, Struers, Ballerup, Denmark). All SEM specimens were dehydrated in ascending grades of *tert*-butyl alcohol and then transferred to a critical-point dryer (Model ID-3, Elionix) for 30 minutes. The tooth-resin interfaces of the specimens were etched (EIS-200ER, Elionix) for 40 seconds using an argon-ion beam (accelerating voltage 1.0 kV, ion current density 0.4 mA/cm^2) directed perpendicular to the polished surfaces. Finally, all SEM specimens were coated with a thin film of gold in a vacuum evaporator (Quick Coater, Type SC-701, Sanyu Denshi, Tokyo, Japan). All observations were performed under SEM at an operating voltage of 10 kV.

Energy Dispersive X-Ray Microanalysis

The elements present in the tooth surfaces with or without aluminablasting were analyzed using SEM/energy dispersive X-ray (EDX; GENESIS 2000, EDAX, Tokyo, Japan) at 20 kV and 2500× magnification. The measurements were conducted perpendicular to the prepared tooth surface, and the elemental content (wt%) of the carbon-coated (Quick Coater, Type SC-701, Sanyu Denshi) surface was measured in five specimens from each group. The measurements were carried out at three points as close to the center of the specimen as possible, and the mean value was calculated for each group thereafter. The analysis was performed using the ZAF correction method (atomic number, absorption, and fluorescence), based on standard-less correction.[21]

Statistical Analysis

As the data were normally distributed (determined by the Kolmogorov-Smirnov test), the tests used for statistical analysis included the analysis of variance (ANOVA) followed by Tukey's honestly significant difference (HSD) test at a significance level of 0.05. A three-way ANOVA along with a Tukey's HSD test (α=0.05) was used for analysis of the SBS data, followed by multiple one-way ANOVA tests to compare the adhesives. A one-way ANOVA followed by Tukey's HSD test was performed for all other variables. All statistical analyses were performed using the Sigma Plot software version 11.0 (SPSS Inc., Chicago, IL, USA).

RESULTS

Enamel Bond Strength

The effects of aluminablasting on the SBS of enamel are shown in Table 3. The three-way ANOVA test showed that the type of adhesive system significantly affected the SBS values ($p<0.001$), while variations in the storage conditions (24-hour or TC, $p=0.239$) and pretreatment methods (with or without aluminablasting, $p=0.104$) did not. The three-way interaction between the factors was not statistically significant ($p=0.728$), and the interaction between storage condition and adhesive system was the only pairwise interaction that exhibited statistical significance ($p=0.03$; storage condition and pretreatment, $p=0.277$; pretreatment and adhesive system, $p=0.386$).

In the 24-hour group, no significant differences were observed between the five universal adhesives, regardless of their aluminablasting status. In the TC group, no significant differences were observed between the universal adhesives when aluminablasting was performed. However, GPB exhibited a significantly higher SBS value compared with all other adhesives, except BML, in the absence of aluminablasting. When the enamel SBS value (24 hours without aluminablasting) was defined as 100% for each tested adhesive, SBS values in 24 hours with aluminablasting ranged from 95.6% to 106.2%,

Table 4: *Influence of Aluminablasting on Dentin Bond Strength*[a]

	24-h		30,000 TC	
	w/o	With	w/o	With
ABU	38.4 (1.3)^{aA} [100%]	25.5 (3.6)^{abB} [66.4%]	37.5 (3.8)^{aA} [97.7%]	24.5 (3.8)^{abB} [63.8%]
ADU	33.8 (3.2)^{bA} [100%]	23.3 (2.7)^{bB} [68.9%]	34.0 (3.1)^{aA} [100.5%]	19.3 (2.9)^{cC} [57.1%]
BML	36.9 (3.9)^{abA} [100%]	27.7 (4.0)^{aB} [75.1%]	34.2 (3.0)^{aA} [92.7%]	21.1 (2.3)^{bcC} [57.2%]
GPB	32.9 (3.0)^{bA} [100%]	23.1 (2.6)^{bC} [70.2%]	28.4 (2.3)^{bB} [86.3%]	26.2 (4.0)^{aBC} [79.6%]
SBU	36.2 (5.4)^{abA} [100%]	28.8 (3.8)^{aB} [79.6%]	35.2 (3.4)^{aA} [97.2%]	24.3 (3.5)^{abB} [67.1%]

Abbreviations: ABU, All-Bond Universal; ADU, Adhese Universal; BML, Bondmer Lightless; GPB, G-Premio Bond; SBU, Scotchbond Universal.
[a] N=15, mean (SD) in MPa. The same lowercase letter in vertical columns indicates no difference at the 5% significance level. The same uppercase letter in horizontal rows indicates no difference at the 5% significance level. Values in parentheses indicate standard deviation.

in TC without aluminablasting ranged from 97.5% to 116.7%, and in TC with aluminablasting ranged from 92.5% to 104.6%.

Dentin Bond Strength

The effects of aluminablasting on the SBS of dentin are shown in Table 4. The three-way ANOVA test showed that all of the factors examined significantly affected the SBS values ($p<0.001$). In addition, the three-way interaction between the factors was statistically significant ($p=0.002$). Although the interaction between the aluminablasting status and the adhesive system was significant ($p<0.001$), none of the other pairwise interactions were statistically significant (storage condition and pretreatment, $p=0.396$; storage condition and adhesive system, $p=0.124$).

In the 24-hour group, GPB and ADU tended to exhibit lower SBS values compared with the other adhesives, regardless of their aluminablasting status. In the TC group, the SBS values of the aluminablasting group varied with the adhesive, with ADU exhibiting a significantly lower SBS compared with the other adhesives. In contrast, no significant differences in the SBS values were observed between the adhesives, except for GPB, in the group without aluminablasting. When the dentin SBS value (24 hours without aluminablasting) was defined as 100% for each tested adhesive, 24 hour SBS values with aluminablasting ranged from 66.4% to 79.6%, in TC without aluminablasting ranged from 86.3% to 100.5%, and in TC with aluminablasting ranged from 57.2% to 79.6%. Most of the adhesives exhibited significantly lower SBS values with aluminablasting compared with the absence of aluminablasting, irrespective of their storage conditions.

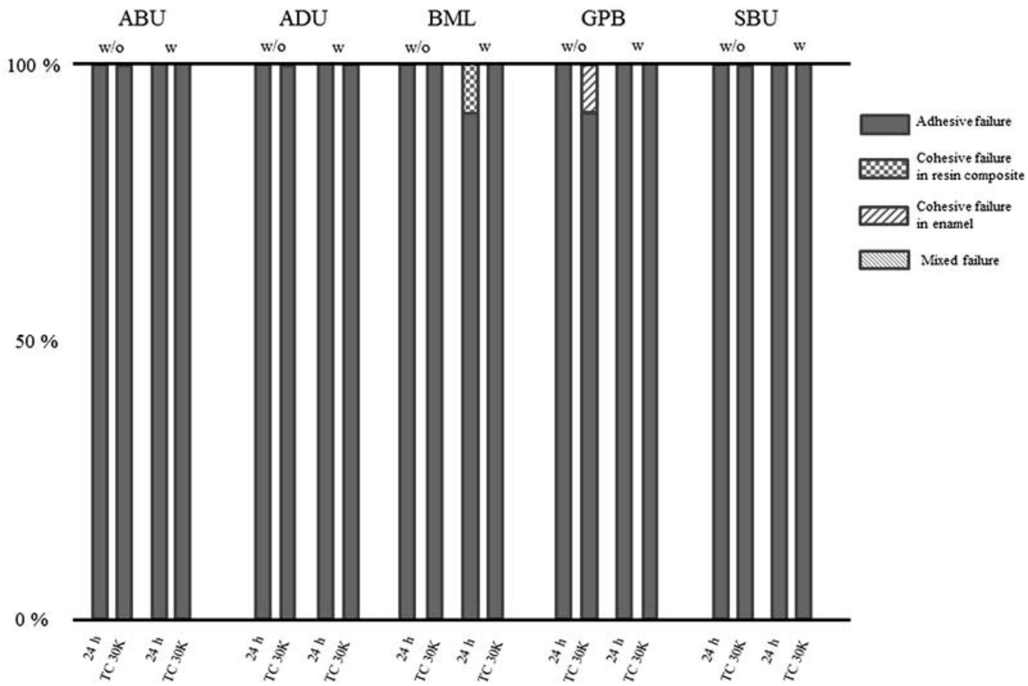
Failure Type of Debonded Specimens

The frequencies of the different failure types are shown in Figures 1 and 2. All enamel debonded specimens, except for two, exhibited adhesive failure regardless of the storage condition, aluminablasting status, and type of adhesive (Figure 1). However, the dentin debonded specimens exhibited a different trend compared with the enamel (Figure 2), with all debonded specimens that had undergone aluminablasting exhibiting adhesive failure patterns, regardless of the storage condition and type of adhesive. Conversely, adhesives in the specimens that did not undergo aluminablasting tended to mainly exhibit mixed and cohesive failures in the dentin.

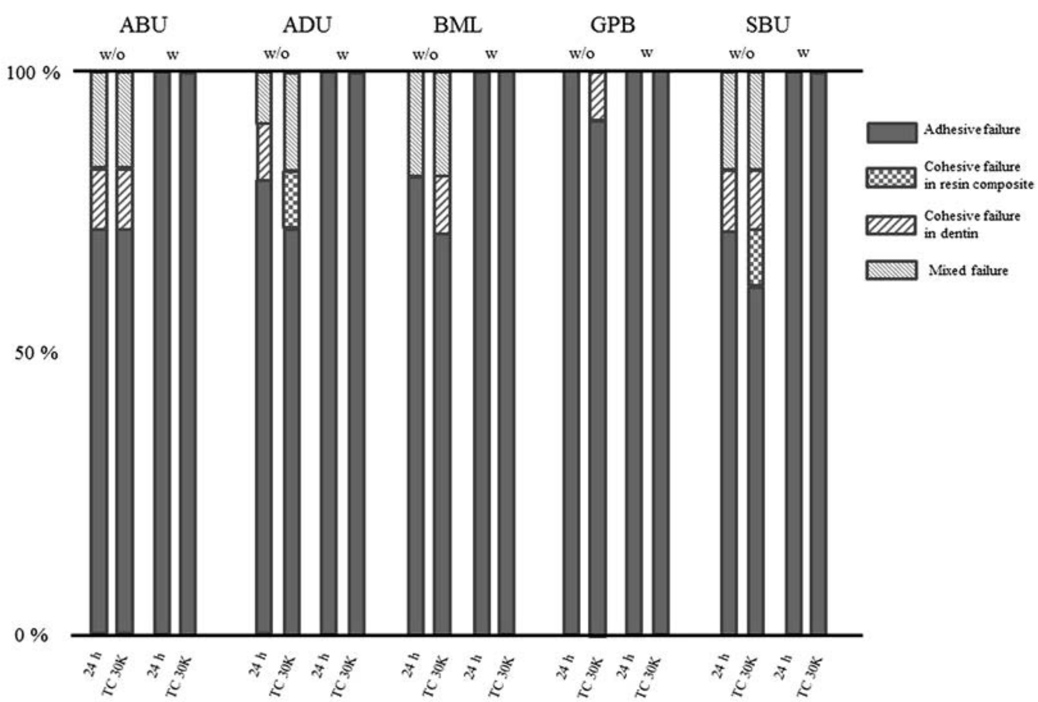
Ra and SFE

The effects of aluminablasting on the Ra and SFE of the enamel and dentin surfaces are shown in Table 5. No significant differences were observed in the Ra values of the enamel and dentin surfaces that did not undergo aluminablasting. Conversely, the Ra values of specimens with aluminablasting were significantly higher compared with the specimens without aluminablasting, regardless of the tooth substrate. In particular, aluminablasted dentin surfaces exhibited a significantly higher Ra value compared with aluminablasted enamel surfaces, and it was almost four times rougher than enamel and dentin without aluminablasting.

In the aluminablasting group, the enamel specimens exhibited significantly higher total free energy (γ_S) values compared with the dentin. When comparing groups, the enamel and dentin specimens with aluminablasting exhibited significantly higher γ_S values than those of specimens without aluminablasting. The dispersion force (γ_S^d) was found to be similar in all groups, and no statistically significant differences were observed between the aluminablasting and no aluminablasting groups. However, the γ_s^p and γ_s^h values were seen to be affected by aluminablasting, with significantly higher values being observed in specimens that had undergone alumina-



1 Failure mode analysis of the de-bonded enamel specimens



2 Failure mode analysis of the de-bonded dentin specimens

Figure 1. *Failure mode analysis of the debonded enamel specimens.*

Figure 2. *Failure mode analysis of the debonded dentin specimens. ABU w, All Bond Universal with aluminablasting; ABU w/o, All Bond Universal without aluminablasting; ADU w, Adhese Universal with aluminablasting; ADU w/o, Adhese Universal without aluminablasting; BML w, Bondmer lightless with aluminablasting; BML w/o, Bondmer lightless without aluminablasting; GPB w, G-Premio with aluminablasting; GPB w/o, G-Premio without aluminablasting; SBU w, Scotchbond Universal with aluminablasting; SBU w/o, Scotchbond Universal without aluminablasting.*

Table 5: *Influence of Aluminablasting on Ra and SFE of Adherent Surface*[a]

	Enamel		Dentin	
	w/o	With	w/o	With
Ra	0.71 (0.02)^c	1.81 (0.05)^b	0.70 (0.02)^c	2.48 (0.10)^a
γ_s	58.9 (3.9)^c	76.5 (2.8)^a	50.9 (3.8)^d	68.6 (3.9)^b
γ_s^d	41.8 (0.5)^a	42.0 (0.7)^a	41.4 (0.6)^a	42.1 (0.8)^a
γ_s^p	3.7 (1.7)^b	6.7 (2.0)^a	3.5 (1.2)^b	6.2 (1.9)^a
γ_s^h	13.4 (2.2)^c	27.8 (1.2)^a	6.0 (2.4)^d	20.3 (3.5)^b

Abbreviations: Ra, surface roughness; SFE, surface free energy; γ_s, total surface free energy; γ_s^d, dispersion force; γ_s^p, polar force; γ_s^h, hydrogen bonding force.

[a] N=10, mean (SD) in MPa. The same uppercase letter in horizontal rows indicates no difference at the 5% significance level. Values in parentheses indicate standard deviation.

blasting compared with those that had not, irrespective of the tooth substrate. The enamel of specimens with aluminablasting exhibited significantly higher γ_s^h values than the dentin.

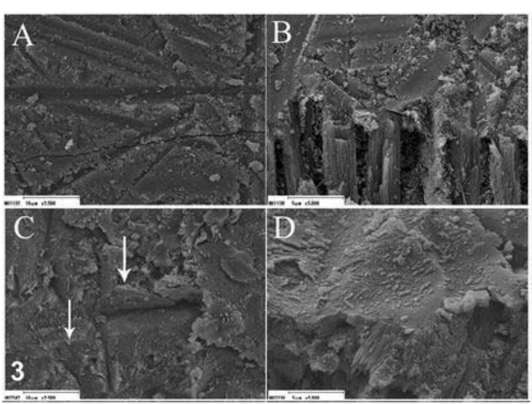
SEM Observations

SEM images of the tooth surfaces with or without aluminablasting are shown in Figures 3 and 4. Scratches from the SiC papers and smear layers were obvious in the enamel and dentin of all specimens without aluminablasting (Figures 3A and 4A). In addition to the scratches from the SiC paper, the specimens that underwent aluminablasting also exhibited impact marks from the alumina particles, regardless of the substrate, and partial removal of the smear layer, thus exposing the enamel surface. In addition, the smear layer of the dentin appeared to be compressed by aluminablasting.

Representative SEM images of the tooth-resin interfaces with or without aluminablasting are shown in Figure 5. The interface between the enamel substrate and adhesive in the specimens with aluminablasting appeared to be more irregular compared with those without aluminablasting (Figure 5A,B). A similar trend was observed in the dentin-resin interfaces (Figure 5C,D), although the irregularities in the dentin interfaces of specimens with aluminablasting were deeper than those seen in the enamel substrate. A high-density transitional layer was observed in the vicinity of the adhesive-dentin interface, regardless of the type of adhesive and the aluminablasting status.

EDX Microanalysis

Representative images of the elemental spectra for enamel and dentin surfaces of specimens with or without aluminablasting are shown in Figure 6, and their elemental compositions are shown in Table 6. The elements C, O, Na, Mg, P, and Ca were detected in the enamel and dentin specimens without aluminablasting (Figure 6A,C). In addition to C, O, Na, Mg, P, and Ca, the element Al was also detected in the enamel and dentin of samples with aluminablasting (Figure 6B,D). No significant differences in the wt% of Na, Mg, or Al were observed between the enamel and dentin of specimens with aluminablast-



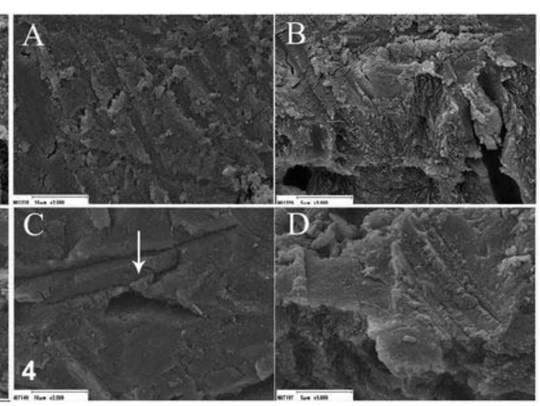
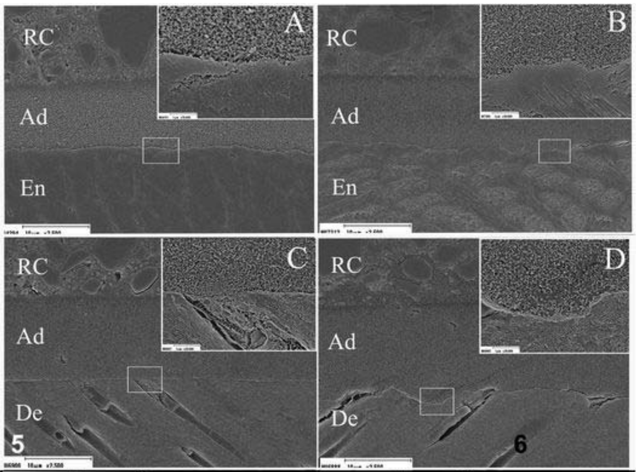


Figure 3. *Representative scanning electron micrographs of enamel and dentin adherent surfaces. Arrows indicate evidence of impact marks from alumina particles. (A): Enamel adherent surface without aluminablasting (2500×). (B): A longitudinal section of the enamel adherent surface without aluminablasting (5000×). (C): Enamel adherent surface with aluminablasting (2500×). (D): A longitudinal section of the enamel adherent surface with aluminablasting (5000×).*

Figure 4. *Representative scanning electron micrographs of enamel and dentin adherent surfaces. Arrows indicate evidence of impact marks from alumina particles. (A): Dentin adherent surface without aluminablasting (2500×). (B): A longitudinal section of the dentin adherent surface without aluminablasting (5000×). (C): Dentin adherent surface with aluminablasting (2500×). (D): A longitudinal section of the dentin adherent surface with aluminablasting (5000×).*



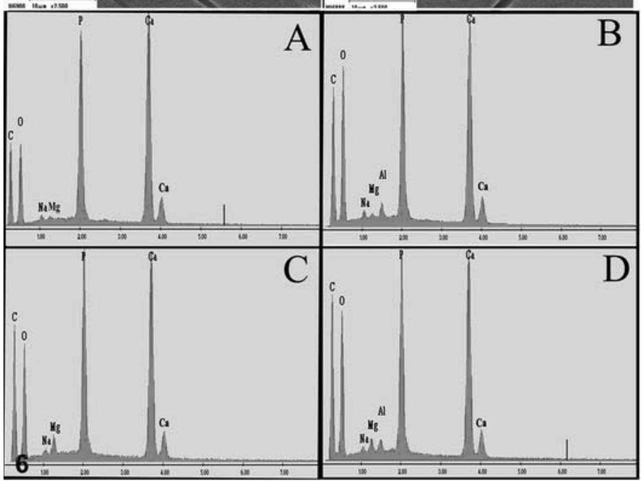


Figure 5. *Representative scanning electron micrographs of the resin-tooth interfaces of Scotchbond Universal (SBU). The visible material is indicated by abbreviations: Ad, adhesive; En: enamel; De: dentin; RC: resin composite. (A): SBU and enamel without aluminablasting at magnification (a) 2500× and (b) 20,000×. (B): SBU and enamel with aluminablasting at magnification (a) 2500× and (b) 20,000×. (C): SBU and dentin without aluminablasting at magnification (a) 2500× and (b) 20,000×. (D): SBU and dentin with aluminablasting at magnification (a) 2500× and (b) 20,000×.*

Figure 6. *Representative images of element spectra for enamel and dentin surfaces with or without aluminablasting. C, carbon; O, oxygen; Na, sodium; Mg, magnesium; Al, aluminum; P, phosphorus; Ca, calcium. (A): Enamel adherent surface without aluminablasting. (B): Enamel adherent surface with aluminablasting. (C): Dentin adherent surface without aluminablasting. (D): Dentin adherent surface with aluminablasting.*

ing. In addition, the wt% of oxygen in samples with aluminablasting was significantly higher than that of samples without aluminablasting.

DISCUSSION

Many studies have investigated optimal repair techniques with a focus on bonding to aged restorations.[2-4] However, little information about the bonding effectiveness when aluminablasting exposes sound tooth substrate during repair procedures is available. The purpose of this study was to determine the bonding effectiveness of universal adhesives to different tooth substrates after aluminablasting from several perspectives.

In the group without aluminablasting, none of the adhesives exhibited a reduction in enamel or dentin bond strength after thermal cycling. This was consistent with previous *in vitro* studies examining the bond durability of universal adhesives.[22,23] In particular, Suzuki and others[23] reported that no reduction of enamel bond strengths in self-etch mode was observed in the degradation condition groups (30,000 thermal cycles and two-year water storage) when compared with the immediate groups (24-hour water storage). These findings suggest that adhesion between the enamel substrate and universal adhesives remains stable once the bond has been established. On the other hand, although aluminablasting had a negligible effect on the bonds to enamel, dentin bond strength tests showed that aluminablasting had a negative impact in both groups (24 hour and TC), regardless of the type of universal adhesive. Therefore, the first null hypothesis that aluminablasting would not affect the bond performance of universal adhesives was rejected only for dentin.

In the group without aluminablasting, none of the adhesives exhibited a reduction in enamel and dentin bond strength after thermal cycling. This was consistent with previous *in vitro* studies examining the bond durability of universal adhesives.[22,23] In particular, Suzuki and others[23] reported that no reduction of enamel bond strengths in self-etch mode was observed in degradation condition groups (30,000 thermal cycles and two-year water storage) when compared with the immediate groups (24-hour water storage). These findings suggest that adhesion between the enamel substrate and universal adhesives remain stable once the qualitative bond has been established.

To obtain a rough and clean surface on restorations, aluminablasting is commonly used in both dental laboratories and clinics.[11,13-16] In addition, aluminablasting is used in orthodontic treatment to create roughness on the enamel surface and increase the mechanical interlocking effect for bracket bonding on occasion.[24] Patcas and others[25] reported that aluminablasting of intact enamel enhanced the surface roughness, and it could produce a rougher surface than phosphoric acid etching to enamel. In the results for changes in surface characteristics after aluminablasting, the aluminablasted enamel

Table 6: *Influence of Aluminablasting on Elemental Composition (wt%) of Adherent Surface*[a]

	Enamel		Dentin	
	w/o	With	w/o	With
Carbon	32.8 (0.8)^{bB}	32.4 (1.2)^{bB}	37.0 (1.8)^{aA}	38.2 (1.0)^{aA}
Oxygen	36.1 (0.9)^{aB}	38.0 (1.1)^{aA}	32.3 (1.8)^{bC}	37.3 (0.5)^{aAB}
Sodium	1.1 (0.3)^{eA}	1.1 (0.1)^{eA}	0.8 (0.2)^{eB}	0.8 (0.1)^{dB}
Magnesium	0.6 (0.1)^{eA}	0.6 (0.1)^{eA}	1.1 (0.2)^{eB}	1.1 (0.1)^{dB}
Aluminum	NA	0.9 (0.1)^{eA}	NA	1.0 (0.1)^{dA}
Phosphorus	11.6 (0.5)^{dA}	10.9 (0.5)^{dA}	11.3 (0.9)^{dA}	9.0 (0.2)^{cB}
Calcium	17.7 (1.0)^{cA}	16.3 (1.0)^{cA}	17.4 (2.8)^{cA}	12.7 (0.4b)^{bB}

[a] *N=10, mean (SD) in wt%. The same lowercase letter in vertical columns indicates no difference at the 5% significance level. The same uppercase letter in horizontal rows indicates no difference at the 5% significance level. Values in parentheses indicate standard deviation.*

and dentin exhibited twofold and threefold increases in Ra values, respectively, when compared with the specimens that did not undergo aluminablasting. In addition, the total SFE of the aluminablasted surfaces of the specimens was 30% higher than the surfaces of specimens that did not undergo aluminablasting. Therefore, the second null hypothesis, that the surface characteristics of the teeth would not differ irrespective of whether they had been aluminablasted or not, was rejected.

Changes in surface roughness are only to be expected, but the changes in SFE may need more explanation. The $\gamma_S{}^h$ parameter represents the water and hydroxyl components of the substrate, while $\gamma_S{}^p$ is thought to be dependent on electric and metallic interactions in addition to dipolar interactions.[18,19] The increased γ_S values of the enamel and dentin specimens that underwent aluminablasting can be attributed to the enhanced surface cleanliness and the presence of residual alumina particles, which are highly polar. This speculation was supported by SEM observations and SEM/EDX analysis, which revealed that Al was present in the enamel and dentin of only those specimens that had undergone aluminablasting. These results were consistent with a previous study examining orthodontic bracket bonding that reported the presence of residual alumina on the aluminablasted enamel.[25] Furthermore, the elemental composition of oxygen was also seen to increase in the aluminablasted surfaces when compared with the surfaces of the specimens that did not undergo aluminablasting, irrespective of the tooth substrate considered. The additional Al and O are presumably derived from the blasting alumina particles.

The findings of the current study suggest that aluminablasting affected the bond strength and surface characteristics of enamel and dentin substrates differently. Therefore, the third null hypothesis, that the effect of aluminablasting would not differ between enamel and dentin, was rejected. In general, larger Ra values and higher SFE are thought to be advantageous for bond performance because of enhanced wettability.[19] However, the presence of a rough surface and higher SFE by aluminablasting did not contribute to enamel and dentin bond performance; instead, the dentin specimens that had undergone aluminablasting exhibited a reduction in bond strength. Soares and others[26] investigated the effect of pretreatments on the dentin bond strength of two self-etching adhesives and reported that the aluminablasting group showed significantly lower micro tensile bond strength values than the no-treatment group (control) in both self-etch adhesives. However, they claimed that the reasons for the dentin bond strength reduction in self-etch adhesives were unclear. Integrating our laboratory results and those of the previous investigation, we may be able to explain this phenomenon. The dentin bond strength reduction after aluminablasting and the different trends observed with regard to the enamel and dentin bond strengths could be explained by the physical properties of the adherent substrates and the smear layer characteristics. Dentin substrate might suffer more damage than enamel substrate because of lower surface hardness and elastic modulus.[27] A previous study reported that air-powder polishing of dentin surfaces using sodium bicarbonate powder increased damage to the dentin substructure and reduced bond performance of two-step and single-step self-etching adhesive systems,[28] leading to the development of a prophylactic polishing powder.[29,30] In addition, since alumina particles are harder than sodium bicarbonate powder, aluminablasting may cause much more damage not only to dentin but also to an enamel substrate, such as micro cracks. The dentin smear layer is composed of disorganized organic debris with HAp minerals, while the enamel smear layer is highly porous.[31] The SEM observations showed that the dentin smear layer was compressed after aluminablasting (Figure 4C,D). This layer contains collagen fragments that can block the penetration of adhesive functional monomers, and this may be related to the fact that the intermolecular spacing of collagen (1.3 nm) is smaller than the size of the functional monomers (approximately 2 nm).[32] In the case of self-etch universal adhesives, penetration of the resin monomers into the smear layer and demineralization of the tooth surface are essential for chemical bonding with HAp. Tamura and others[33] investigated how air-powder polishing influences bonding between dentin and universal adhesives. They suggested that the presence of residual sodium bicarbonate powder on dentin surfaces leads to chemical and/or mechanical changes to collagen fibrils and prevention of adhesive penetration into dentin. It can be speculated that although a different blasting material is used in this study, the same situation might occur. Moreover, a previous study comparing the enamel and dentin bond durability of self-etch adhesives with different smear layers showed that the dentin was more susceptible to the condition of the smear layer condition than the enamel.[34]

Therefore, it can be speculated from the integrated results that the compressed dentin smear layer and embedded alumina particles may interfere with the penetration of the resin monomer and interaction with the functional monomer of universal adhesives. These may lead to lower bond strength because the dentin HAp has a higher affinity for the functional monomer than enamel.[35,36]

Changes in the elemental composition after aluminablasting showed significantly lower wt% of P and Ca in dentin surfaces than enamel surfaces and dentin surfaces without aluminablasting. Although the mineral content of dentin was lower than that of enamel, the dentin HAp has a high affinity for the functional monomer, thus creating greater nanolayering between them.[35,36] Therefore, it might be inferred that the lower concentration of P and Ca and the consumption of functional monomers by residual alumina particles in the aluminablasted dentin surfaces may lead to a weaker chemical bond compared with those in the dentin surfaces of specimens that did not undergo aluminablasting. Notably, all debonded specimens of aluminablasted dentin exhibited adhesive failure patterns, and this was in contrast to the specimens that did not undergo aluminablasting.

Regarding clinical practice, the results of this study suggest that aluminablasting should normally not be used with universal adhesives, because dentin bond performance was noticeably affected. Further, the bond strength and surface characteristic tests were performed using flat specimens in this study. However, when considering the clinical situation, the cavity when repairing a restoration is likely to have a complex configuration. It is probable that many more alumina particles may remain in a cavity than on a flat specimen and would be difficult to remove. There is a possibility that remnant alumina particles might be an inhibiting factor for immediate and long-term bond durability of dentin. In addition, although the enamel bond effectiveness did not change in response to aluminablasting, there are still reasons to avoid it when repairing aged restorations surrounded by sound enamel structures, because enamel loss after aluminablasting is much higher than after phosphoric acid etching.[25] Wendler and others[13] reported that no significant difference in Ra value was observed between aluminablasting and bur roughening. Therefore, instead of aluminablasting the tooth structure, it may be better to roughen the surface of the aged restoration with burs, avoiding damage to enamel as far as possible, and use a suitable application primer.

The results of this study suggest that aluminablasting should not be used when universal adhesives are used to make a repair restoration *in situ*. Thus, further work to determine the bonding characteristics of universal adhesives to bur-roughened aged restorations would be valuable. The flexibility of universal adhesives (used with or without phosphoric acid etching, bonding to multiple substrates), gives reason to hope that an effective and conservative protocol can be developed.

CONCLUSION

In conclusion, most universal adhesives in self-etch mode with aluminablasting exhibited lower dentin SBS values compared with the specimens without aluminablasting. Moreover, the enamel and dentin substrates exhibited similar Ra and SFE results in both groups. These results suggest that although aluminablasting of the tooth surface is thought to be effective for modification of the adherent surface, it may not enhance enamel bond performance and may also adversely affect the dentin bond effectiveness of the universal adhesives tested in this study. Therefore, when considering repair techniques using aluminablasting, we should take into account both the interaction between functional monomers and

HAp and the infiltration capability of resin monomers beyond the smear layer.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interests of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 7 October 2018)

REFERENCES

1. Lynch CD, Opdam NJ, Hickel R, Brunton PA, Gurgan S, Kakaboura A, Shearer AC, Vanherle G, & Wilson NHF (2014) Guidance on posterior resin composites: Academy of Operative Dentistry–European section *Journal of Dentistry* **42(4)** 377-383, https://doi.org/10.1016/j.jdent.2014.01.009.
2. Loomans BAC, Cardoso MV, Roeters FJM, Opdam NJM, De Munck J, Huysmans MC, & Van Meerbeek B (2011) Is there one optimal repair technique for all composites? *Dental Materials* **27(7)** 701-709, https://doi.org/10.1016/j.dental.2011.03.013.
3. Hickel R, Brüshaver K, & Ilie N (2013) Repair of restorations—criteria for decision making and clinical recommendations *Dental Materials* **29(1)** 28-50, https://doi.org/10.1016/j.dental.2012.07.006.
4. Özcan M (2015) Intraoral repair protocol for chipping or fracture of veneering ceramic in zirconia fixed dental prostheses *Journal of Adhesive Dentistry* **17(2)** 189-190.
5. Yoshida T, Miyazaki M, Hirohata N, & Moore BK (2005) Influence of metal conditioner contamination of the dentin surface on bond strengths of dentin adhesive systems using self-etching primers *Operative Dentistry* **30(3)** 359-367.
6. Mamanee T, Takahashi M, Nakajima M, Foxton RM, & Tagami J (2015) Initial and long-term bond strengths of one-step self-etch adhesives with silane coupling agent to enamel-dentin-composite in combined situation *Dental Materials Journal* **34(5)** 663-670, https://doi.org/10.4012/dmj.2015-050.
7. da Rosa WL, Piva E, & da Silva AF (2015) Bond strength of universal adhesives: A systematic review and meta-analysis *Journal of Dentistry* **43(7)** 765-776, https://doi.org/10.1016/j.jdent.2015.04.003.
8. Takamizawa T, Barkmeier WW, Tsujimoto A, Berry TP, Watanabe H, Erickson RL, Latta MA, & Miyazaki M (2016) Influence of different etching modes on bond strength and fatigue strength to dentin using universal adhesive systems *Dental Materials* **32(2)** e9-e21, https://doi.org/10.1016/j.dental.2015.11.005.
9. Suzuki T, Takamizawa T, Barkmeier WW, Tsujimoto A, Endo H, Erickson RL, Latta MA, & Miyazaki M (2016) Influence of etching mode on enamel bond durability of universal adhesive systems *Operative Dentistry* **41(5)** 520-530, https://doi.org/10.2341/15-347-L.
10. Tsujimoto A, Barkmeier WW, Takamizawa T, Wilwerding TM, Latta MA, & Miyazaki M (2017) Interfacial characteristics and bond durability of universal adhesives to various substrates *Operative Dentistry* **42(2)** e59-e70, https://doi.org/10.2341/15-353-L.
11. Al Jabbari YS, Zinelis S, & Eliades G (2012) Effect of sandblasting conditions on alumina retention in representative dental alloys *Dental Materials Journal* **31(2)** 249-255, https://doi.org/10.4012/dmj.2011-210.
12. Bühler J, Amato M, Weiger R, & Walter C (2016) A systematic review on the effects of air polishing devices on oral tissue *International Journal of Dental Hygiene* **14(1)** 15-28, https://doi.org/10.1111/idh.12120.
13. Wendler M, Belli R, Panzer R, Skibbe D, Petschelt A, & Lohbauer U (2016) Repair bond strength of aged resin composites after different surface and bonding treatments *Materials* **9(7)** 547-559, https://doi.org/10.3390/ma9070547.
14. Higashi M, Matsumoto M, Kawaguchi A, Miura J, Minamino T, Kabetani T, Takeshige F, Mine A, & Yatani H (2016) Bonding effectiveness of self-adhesive and conventional-type adhesive resin cements to CAD/CAM resin blocks. Part 1: Effects of sandblasting and silanization *Dental Materials Journal* **35(1)** 21-28, https://doi.org/10.4012/dmj.2015-234.
15. Yoshida K (2018) Influence of cleaning methods on resin bonding to saliva-contaminated zirconia *Journal of Esthetic and Restorative Dentistry* **30(3)** 259-264, https://doi.org/ 10.1111/jerd.12369.
16. Ersu B, Yuzugullu B, Yazici AR, & Canay S (2009) Surface roughness and bond strengths of glass-infiltrated alumina-ceramics prepared using various surface treatments *Journal of Dentistry* **37(11)** 848-856, https://doi.org/10.1016/j.jdent.2009.06.017.
17. ISO-Standards (2013) ISO 29022 2013 Dentistry-Adhesion-Notched-Edge Shear Bond Strength Test *Geneve: International Organization for Standardization* **1st edition** 1-12.
18. Imai A, Takamizawa T, Sai K, Tsujimoto A, Nojiri K, Endo H, Barkmeier WW, Latta MA, & Miyazaki M (2017) Influence of application method on surface free-energy and bond strength of universal adhesive systems to enamel *European Journal of Oral Science* **125(5)** 385-395, https://doi.org/10.1111/eos.12361.
19. Sai K, Takamizawa T, Imai A, Tsujimoto A, Ishii R, Barkmeier WW, Latta MA, & Miyazaki M (2018) Influence of application time and etching mode of universal adhesives on enamel adhesion *Journal of Adhesive Dentistry* **20(1)** 65-77, https://doi.org/ 10.3290/j.jad.a39913.
20. Hata T, Kitazato Y, & Saito T (1987) Estimation of the surface energy of polymer solids *Journal of Adhesion* **21(3-4)** 177-194.
21. Hosoya Y, Tadokoro K, Inoue T, Miyazaki M, & Tay FR (2013) Effect of SI-R20401 to remineralize artificial incipient enamel lesions in primary teeth *Journal of Oral Science* **55(4)** 301-310, https://doi.org/10.2334/josnusd.55.301.
22. Sai K, Shimamura Y, Takamizawa T, Tsujimoto A, Imai A, Endo H, Barkmeier WW, Latta MA, & Miyazaki M (2016) Influence of degradation conditions on dentin bonding durability of three universal adhesives *Journal of Dentistry* **54** 56-61, https://doi.org/10.1016/j.jdent.2016.09.004.

23. Suzuki S, Takamizawa T, Imai A, Tsujimoto A, Sai K, Takimoto M, Barkmeier WW, Latta MA, & Miyazaki M (2018) Bond durability of universal adhesive to bovine enamel using self-etch mode *Clinical Oral Investigations* **22(3)** 1113-1122, https://doi.org/10.1007/s00784-017-2196-x.

24. Baumgartner S, Koletsi D, Verma C, & Eliades T (2017) The effect of enamel sandblasting on enhancing bond strength of orthodontic brackets: A systematic review and meta-analysis *Journal of Adhesive Dentistry* **19(6)** 463-473, https://doi.org/ 10.3290/j.jad.a39279.

25. Patcas R, Zinelis S, Eliades G, & Eliades T (2015) Surface and interfacial analysis of sandblasted and acid-etched enamel for bonding orthodontic adhesives *American Journal of Orthodontics Dentofacial Orthopedics* **147(Supplement 4)** S64-S75, https://doi.org/10.1016/j.ajodo.2015.01.014.

26. Soares CJ, Castro CG, Santos Filho PCF, & de Mota AS (2007) Effect of previous treatments on bond strength to two self-etching adhesive systems to dental substrate *Journal of Adhesive Dentistry* **9(3)** 291-296.

27. Powers JM & Wataha JC (2013) *Dental Materials: Properties and Manipulation* **10th edition** Mosby, St Louis MO.

28. Frankenberger R, Lohbauer U, Tay FR, Teschner M, & Nikolaenko SA (2007) The effect of different air-polishing powders on dentin bonding *Journal of Adhesive Dentistry* **9(4)** 381-389.

29. Tada K, Kakuta K, Ogura H, & Sato S (2010) Effect of particle diameter on air polishing of dentin surfaces *Odontology* **98(1)** 31-36, https://doi.org/10.1007/s10266-009-0113-8.

30. Sahrmann P, Ronay V, Schmidlin PR, Attin T, & Paqué F (2014) Three-dimensional defect evaluation of air polishing on extracted human roots *Journal of Periodontology* **85(8)** 1107-1114, https://doi.org/10.1902/jop.2014.130629.

31. Thanatvarakorn O, Nakajima M, Prasansuttiporn T, Ichinose S, Foxton RM, & Tagami J (2014) Effect of smear layer deproteinizing on resin-dentine interface with self-etch adhesive, *Journal of Dentistry* **42(3)** 298-304, https://doi.org/10.1016/j.jdent.2013.11.026.

32. Bertassoni LE, Orgel JP, Antipova O, & Swain MV (2012) The dentin organic matrix-limitations of restorative dentistry hidden on the nanometer scale *Acta Biomaterialia* **8(7)** 2419-2433, https://doi.org/10.1016/j.actbio.2012.02.022.

33. Tamura Y, Takamizawa T, Shimamura Y, Akiba S, Yabuki C, Imai A, Tsujimoto A, Kurokawa H, & Miyazaki M (2017) Influence of air-powder polishing on bond strength and surface-free energy of universal adhesive systems *Dental Materials Journal* **36(6)** 762-769, https://doi.org/ 10.4012/dmj.2016-185.

34. Takamizawa T, Barkmeier WW, Sai K, Tsujimoto A, Imai A, Erickson RL, Latta MA, & Miyazaki M (2018) Influence of different smear layers on bond durability of self-etch adhesives *Dental Materials* **34(2)** 246-259, https://doi.org/10.1016/j.dental.2017.11.002.

35. Yoshihara K, Yoshida Y, Hayakawa S, Nagaoka N, Irie M, Ogawa T, Van Landuyt KL, Osaka A, Suzuki K, Minagi S, & Van Meerbeek B (2011) Nanolayering of phosphoric acid ester monomer on enamel and dentin *Acta Biomaterialia* **7(8)** 3187-3195, https://doi.org/10.1016/j.actbio.2011.04.026.

36. Yaguchi T (2017) Layering mechanism of MDP-Ca salt produced in demineralization of enamel and dentin apatite *Dental Materials* **33(1)** 23-32, https://doi.org/10.1016/j.dental.2016.09.037.