Influence of air-powder polishing on bond strength and surface-free energy of universal adhesive systems

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The influences of air-powder polishing with glycine or sodium bicarbonate powders on shear bond strengths (SBS) and surface-free energies of universal adhesives were examined. Scotchbond Universal Adhesive (SU, 3M ESPE), G-Premio Bond (GP, GC), Adhese Universal (AU, Ivoclar Vivadent), and All-Bond Universal (AB, Bisco) were used in this study. Bovine dentin surfaces were air polished with glycine or sodium bicarbonate powders prior to the bonding procedure, and resin pastes were bonded to the dentin surface using universal adhesives. SBSs were determined after 24-h storage in distilled water at 37°C. Surface-free energy was then determined by measuring contact angles using three test liquids on dentin surfaces. Significantly lower SBSs were observed for dentin that was air-powder polished and surface-free energies were concomitantly lowered. This study indicated that air-powder polishing influences SBSs and surface-free energies. However, glycine powder produced smaller changes in these surface parameters than sodium bicarbonate.

Keywords: Air-powder polishing, Universal adhesive, Dentin bond strength, Surface-free energy

INTRODUCTION

Air-powder polishing is widely used to remove deposits from tooth surfaces and offers a convenient alternative to conventional methods using rubber cups, brush cones, and hand instruments¹. Moreover, as a contemporary approach, air-powder polishing can provide safe and efficient biofilm and stain removal². Air-powder polishing is superior to rubber-cup polishing when preparing occlusal surfaces prior to etching for sealants³. In addition, air-powder polishing of wedge-shaped lesions may enhance dentin bond strengths of resin composites⁴. Previous studies show that air-powder polishing with sodium bicarbonate powder is safe on enamel and is less abrasive than rubber-cup polishing⁵. However, decreased bond strengths with dentin were reported following air-powder polishing with sodium bicarbonate powder⁶.

Glycine powders were recently developed for air-powder polishing, and satisfactory clinical outcomes have been demonstrated in comparison with sodium bicarbonate powder⁷. Accordingly, glycine particles have lower Morse hardness indexes than dentin and have high water solubility and a non-salty taste⁸. In addition, glycine powders reportedly produced less surface damage on restorative materials than sodium bicarbonate powder⁹. However, little is known about adhesion to dentin following abrasion with air-powder polishing using glycine powder.

Dental adhesives with combined single-step functions of self-etching primers and bonding agents are increasingly popular in adhesive restorative dentistry¹⁰. Recently, multi-mode universal adhesives were introduced to reduce technical sensitivity and to shorten clinical procedure times and can be used as self-etch and

etch-and-rinse adhesives¹¹. Dentin bond strengths are influenced by smear layers that are created by cutting with either hand or rotary instruments¹². Hence, cleaning of dentin surfaces to remove smear layers is a crucial clinical step prior to bonding of restoratives¹³. Various techniques have been used to modify dentin surfaces and ensure stable bonding of universal adhesives¹⁴. Previous studies have shown that sodium bicarbonate air-powder polishing adversely affects dentin bonding of two-step and single-step self-etching adhesives. However, few studies have assessed the influence of air-powder polishing on dentin bond strengths of universal adhesives.

In the present study, we evaluated the influence of air-powder polishing on dentin bond strengths of various universal adhesive systems according to shear bond strengths (SBS) and surface-free energies of air-powder polished dentin surfaces. Specifically, we assessed differences in bond strength and surface-free energy with and without air-powder polishing.

MATERIALS AND METHODS

Adhesives used

The materials used in this study are listed in Table 1, and include the universal adhesives Scotchbond Universal Adhesive (SU, 3M ESPE, St. Paul, MN, USA), G-Premio Bond (GP, GC, Tokyo, Japan), Adhese Universal (AU, Ivoclar Vivadent, Schaan, Lichtenstein), and All-Bond Universal (AB, Bisco Dental Products, Schaumburg, IL, USA). Bond strengths were determined for the resin composite Clearfil AP-X (Kuraray Noritake Dental, Tokyo, Japan).

Resin specimens were cured using a visible light curing unit (Optilux 501, Kerr, Danbury, CT, USA), and

Table 1 Materials

	Code	Lot No.	Main Components	Manufacturer
Adhesive				
Scotchbond Universal Adhesive	SU	584163	MDP, HEMA, Bis-GMA, Vitrebond copolymer, ethanol, water, initiators, filler, silane	3M ESPE (St. Paul, MN, USA)
G-Premio Bond	GP	1501211	MDP, 4-MET, MEPS, methacrylate monomer, acetone, water, initiators, filler	GC (Tokyo, Japan)
Adhese Universal	AU	T08671	MDP, HEMA, Bis-GMA, D3MA, MCAP, ethanol, water, initiator, filler	Ivoclar Vivadent (Schaan, Lichtenstein)
All-Bond Universal	AB	1300008729	MDP, HEMA, Bis-GMA, ethanol, water, initiators, silanated colloidal silica	Bisco (Schaumburg, IL, USA)
Air-polishing powder				
Air-Flow Perio	GL	031503	Glycine	Electron Medical Systems (Nyon, Switzerland)
Air-Flow Classic	SB	041502	Sodium bicarbonate	Electron Medical Systems

MDP: 10-methacryloyloxydecyl dihydrogen phosphate

HEMA: 2-hydroxyethyl methacrylate

Bis-GMA: 2,2-bis[4-(2-hydroxyl-3-methacryloyloxypropoxy)-phenyl]propane

4-MET: 4-methacryloyloxyethyl trimellitic acid

MEPS: Methacryloyloxyalkyl thiophosphate methylmethacrylate

D3MA: Hydrophobic crosslinking dimethacrylate

MCAP: Methacrylated carboxylic acid polymer

the power density (about 600 mW/cm²) of the unit was checked using a dental radiometer (Model 100, Kerr) before preparation of specimens.

Specimen preparation

Mandibular incisors were extracted from two-three-year-old cattle and were stored frozen for up to two weeks prior to use as models of human teeth. After removing the roots using a low-speed saw with a diamond-impregnated disk (IsoMet, Buehler, Lake Bluff, IL, USA), pulps were removed, and pulp chambers of each tooth were filled with cotton to avoid penetration of the embedding media. Subsequently, labial surfaces were ground on wet 240-grit silicon carbide (SiC) paper to create flat dentins. Teeth were then mounted in self-curing acrylic resin (Tray Resin II, Shofu, Kyoto, Japan), with flattened areas exposed, and were then placed under tap water to reduce the temperature rises caused by exothermic polymerization reactions of acrylic resin. Dentin surfaces were finished by grinding with wet 600-grit SiC paper, and were then dried with oil-free compressed air to remove visible water.

Bond strength measurements

Teeth were divided into three dentin treatment groups, and control specimens were bonded without air-powder

polishing prior to applying adhesive. Test specimens were air-powder polished with glycine powder (GL, Air-Flow Perio, Electron Medical Systems, Nyon, Switzerland) or sodium bicarbonate powder (SB, Air-Flow Classic, Electron Medical Systems). In these procedures, the tip of the air-polishing device (Air-Flow Master, Electron Medical Systems) was positioned approximately 5 mm above the tooth surface and polishing was performed for 10 s. Dentin surfaces were then rinsed for 10 s and gently air dried, and adhesives were applied according to respective manufacturers' instructions (Table 2). Subsequently, specimens were clamped in an Ultradent Bonding Jig (Ultradent Products, South Jordan, UT, USA) and resin composites were formed and held on dentin surfaces using plastic molds of 2.4 mm in internal diameter and 2.0 mm in height. Resin composites were condensed in molds and were cured for 20 s, and finished specimens were submerged in distilled water at 37°C for 24 h.

Ten specimens per group were tested in shear mode using a bond-testing notched-blade semi-circular apparatus (Ultradent Products) in a universal testing machine (Type 5500R, Instron, Norwood, MA, USA) at a crosshead speed of 1.0 mm/min. SBS values (MPa) were calculated as peak loads at failure divided by specimen surface areas.

Table 2 Application protocols of adhesives

Code	Application protocol
SU	Apply adhesive to tooth surface for 20 s. Subject to gentle stream air for 5 s until it no longer moves and the solvent has evaporated completely. Light irradiation for 10 s.
GP	Apply adhesive to tooth surface for 10 s. Subject to maximum stream air for 5 s. Light irradiation for 10 s.
AU	Apply adhesive to tooth surface for 20 s using continuous scrubbing. Subject to air until a glossy, immobile film layer develops. Light irradiation for 10 s.
AB	Rinse preparation and remove excess water. Apply two separate coats of adhesive, scrubbing the preparation with a micro-brush for 10–15 s per coat. Air dry for at least 10 s until glossy. Repeat application of adhesive and air drying. Light irradiation for 10 s.

Table 3 Surface-free energy and component values of test liquids

Liquid	Lot No.	Manufacturer	γ_L	γ_L^d	γ_L^p	γ_L^h
1-bromonaphthalene	ALH4513	Wako Pure Chemical Industries	44.8	44.6	0.2	0.0
Diiodomethane	ALL2310	Wako Pure Chemical Industries	50.8	46.8	4.0	0.0
Distilled water	—	—	72.8	29.1	1.3	42.4

Unit, mN/m

γ_L : surface-free energy, γ_L^d : dispersion force, γ_L^p : polarity force, γ_L^h : hydrogen bonding force

Fig. 1 Drop Master DM500 apparatus fitted with a charge-coupled device camera (a); automatic measurement of contact angles (b).

Following testing, all specimens were examined under an optical microscope (SZH-131, Olympus, Tokyo, Japan) at a magnification of $\times 10$ and locations of bond failure were recorded. Types of failure were determined according to percentages of substrate-free material with adhesive failure, cohesive failure in dentin, and cohesive failure in resin or adhesive.

Surface-free energy measurements

Mandibular incisors were prepared as described in the preceding section and surface-free energy was

determined by measuring surface contact angles for the three test liquids 1-bromonaphthalene, diiodomethane, and distilled water. Known surface-free energy parameters for test liquids are presented in Table 3. Surface-free energy values were determined according to contact angles in five specimens per treatment group for each dentin surface using a Drop Master DM500 instrument (Kyowa Interface Science, Saitama, Japan). The device was fitted with a charge-coupled device camera, which allowed automatic measurement of contact angles (Fig. 1). For each test liquid, equilibrium

contact angles (θ) were measured using the sessile-drop method at $23 \pm 1^\circ\text{C}$, and surface-free energy parameters of the solids were then determined based on fundamental concepts of wetting. Work of adhesion (W) between solids (S) and liquids (L) was determined using the Young-Dupré equation as follows:

$$W_{\text{SL}} = \gamma_{\text{L}} + \gamma_{\text{S}} - \gamma_{\text{SL}} = \gamma_{\text{L}} (1 + \cos\theta)$$

where γ_{SL} is the interfacial free energy between the solid and liquid, γ_{L} is the surface-free energy of the liquid, and γ_{S} is the surface-free energy of the solid. After extending the Fowkes equation, the γ_{SL} can be expressed as follows:

$$\gamma_{\text{SL}} = \gamma_{\text{L}} + \gamma_{\text{S}} - 2(\gamma_{\text{L}}^{\text{d}} \gamma_{\text{S}}^{\text{d}})^{1/2} - 2(\gamma_{\text{L}}^{\text{p}} \gamma_{\text{S}}^{\text{p}})^{1/2} - 2(\gamma_{\text{L}}^{\text{h}} \gamma_{\text{S}}^{\text{h}})^{1/2}$$

$$\gamma_{\text{L}} = \gamma_{\text{Ld}} + \gamma_{\text{Lp}} + \gamma_{\text{Lh}}, \quad \gamma_{\text{S}} = \gamma_{\text{Sd}} + \gamma_{\text{Sp}} + \gamma_{\text{Sh}}$$

where $\gamma_{\text{L}}^{\text{d}}$, $\gamma_{\text{L}}^{\text{p}}$, and $\gamma_{\text{L}}^{\text{h}}$ are components of surface-free energy (γ) arising from dispersion forces, polar (permanent and induced) forces, and hydrogen-bonding forces, respectively. Surface-free energy (γ) values were determined for the three test liquids, and surface energy parameters of the treated dentin surfaces were calculated based on these equations using add-on software and an interface measurement and analysis system (FAMAS, Kyowa Interface Science).

Scanning electron microscopy (SEM)

Treated dentin surfaces and resin-dentin interfaces were observed using SEM. In these experiments, air-powder polished dentin surfaces were rinsed with distilled water followed by air blowing. Ultrastructural observations of restorative/dentin interfaces were performed after embedding bonded specimens (stored at 37°C in distilled water for 24 h) in epoxy resin (Epon 812, Nissin EM, Tokyo, Japan), and incubating them at 37°C for an additional 12 h. Embedded specimens were then sectioned through the diameter of the composite resin post, and surfaces of cut halves were polished

using a grinder-polisher (Ecomet 4/Automet 2, Buehler) with #600, #1200, and then #4000 grid SiC papers. Surfaces were finally polished on a special soft cloth using diamond paste (Buehler) with a grid size of $1.0 \mu\text{m}$. All SEM specimens were dehydrated in ascending concentrations of *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, Eiko Engineering, Tokyo, Japan) for 30 min. These surfaces were then subjected to argon-ion beam etching (Type EIS-200ER, Elionix, Tokyo, Japan) for 30 s with the ion beam (accelerating voltage, 1.0 kV; ion current density, 0.4 mA/cm^2) directed perpendicular to the polished surface. Surfaces were finally coated in a vacuum evaporator (Quick Coater Type SC-701, Sanyu Denshi, Tokyo, Japan) with a thin film of gold and were observed using a SEM instrument (ERA-8800FE, Elionix) at an accelerating voltage of 10 kV.

Statistical analysis

Data from SBS and surface-free energy tests were compared using one-way analysis of variance. Statistical analyses were performed using Sigma Plot software (Version 11.0, SPSS, Chicago, IL, USA).

RESULTS

Mean SBS to dentin ranged from 31.3 to 35.8 MPa in the control group (without air-powder polishing) and were 27.2–32.9 MPa in the GL group, and 23.9–29.4 MPa in the SB group (Table 4). Bond strengths following air-powder polishing were significantly lower under control conditions for all tested universal adhesives. However, adhesive failure predominated regardless of dentin pretreatments (Table 4).

Determinations of surface-free energies and their components (Table 5) showed decreased γ_{S} values with air-powder polishing, and were significantly lower for GL and SB groups compared with the control group. Whereas estimated $\gamma_{\text{S}}^{\text{d}}$ values remained relatively

Table 4 Influence of air-powder polishing on shear bond strengths of universal adhesive systems

Adhesive	Control	GL	SB
SU	35.6 (4.1) ^a [0/0/10]	32.7 (4.0) ^b [0/0/10]	27.5 (3.7) ^c [0/0/10]
GP	31.3 (2.1) ^b [0/0/10]	27.2 (2.8) ^c [0/0/10]	23.9 (3.5) ^d [0/0/10]
AU	35.8 (3.1) ^a [0/0/10]	30.2 (4.1) ^b [0/0/10]	26.7 (4.1) ^c [0/0/10]
AB	34.5 (3.2) ^a [0/0/10]	32.9 (3.8) ^b [0/0/10]	29.4 (3.9) ^b [0/0/10]

Unit, MPa; values in parenthesis are standard deviations ($n=10$)

Values with the same superscript letters do not differ significantly ($p>0.05$)

Values in square parentheses indicate failure modes, (mixed failure/cohesive failure in dentin/adhesive failure)

Table 5 Surface-free energy and component values for dentin-surface treatments

	γ_s	γ_s^d	γ_s^p	γ_s^h
Control	53.4 (1.6)	36.4 (1.6)	0.0 (0.0)	17.0 (2.3)
GL	47.2 (2.6)	36.3 (1.9)	0.0 (0.0)	10.9 (1.7)
SB	46.7 (2.5)	38.5 (1.3)	0.0 (0.0)	8.2 (1.5)

Unit, mN/m; values in parenthesis are standard deviations ($n=5$)

Vertical lines between values indicate no significant differences ($p>0.05$)

γ_s : surface-free energy, γ_s^d : dispersion force, γ_s^p : polarity force, γ_s^h : hydrogen bonding force

Fig. 2 Representative SEM photomicrographs of treated dentin surfaces; original magnification, 2,500 $\times$; (a) control, (b) glycine, and (c) sodium bicarbonate.

constant in the range of 36.3–38.5 mN/m for all surfaces, γ_s^h values in GL and SB groups were significant lower than those in the control group.

SEM observations of treated dentin surfaces (Fig. 2) revealed scratches created by SiC paper on dentin

surfaces without air-powder polishing (control group), and smear layers over dentin tubules were observed. In contrast, smear layers were removed and dentin tubules were open in the GL group. However, after treatment with sodium bicarbonate powder, dentin surfaces were covered with smear layers and were rough.

SEM observations of resin/dentin interfaces (Fig. 3) showed excellent adaptation to the dentin surface at resin-dentin interfaces in all groups. Moreover, adhesive layers were approximately 5–10 μ m in thickness. However, resin tag formations in dental tubules were observed between dentin and adhesive in the GL group (Figs. 3c and d).

DISCUSSION

Due to their efficacy and convenience, air-powder polishing devices are increasingly used to remove biofilms, plaques, and stains from tooth surfaces⁹. Accordingly, air-powder polishing reportedly removes dental debris more effectively than curets, ultrasonic scalers, and rubber cups^{2,8}. However, some powders can cause damage to tooth surfaces during polishing. In a previous study, air-powder polishing of dentin surfaces using sodium bicarbonate powder increased damage to the tooth substructure and decreased bonding performance of two-step and single-step self-etching adhesive systems⁴, leading to the development of a prophylactic polishing powder^{15,16}. Glycine is a highly water-soluble amino acid with a lower Morse hardness index than sodium bicarbonate, and has very low toxicity, is nonallergenic, has a light sweet taste, and is used as a food flavor enhancer, indicating high suitability for intra-oral use. Additionally, glycine powder particle sizes are approximately four times smaller than those of conventional sodium bicarbonate powder. However, it remains unclear whether air-powder polishing using glycine powder affects dentin adhesion of universal adhesives.

Highly effective bonding performance of universal adhesives reflects chemical bonding capacity of functional monomers to various substrates^{17–20}. Moreover, the extent to which liquid drops wet dentin surfaces depends on chemical interactions between the liquid and dentin, and on physical considerations such as capillary action, and surface roughness²¹.

Fig. 3 Representative SEM photomicrographs of the dentin-resin interface; original magnification, 5,000 $\times$; (a) control with Scotchbond Universal Adhesive (SU), (b) control with G-Premio Bond (GP), (c) SU with glycine, (d) GP with glycine, (e) SU with sodium bicarbonate, (f) GP with sodium bicarbonate.

Investigations of air-powder polished dentin surfaces using measurements of surface-free energy may provide novel insights into bonding mechanisms between dentin and adhesive. Herein, we investigated the influence of air-powder polishing on the bonding performance of universal adhesives by measuring SBS and surface-free energies.

Compared with the control group, dentin bond strength of universal adhesives decreased significantly after air-powder polishing, suggesting that air-powder polishing influences bonding between dentin and adhesive. Subsequent SEM observations of dentin surfaces showed that smear layers remained on dentin surfaces in the SB group, suggesting that the presence of residual SB powder on dentin surfaces leads to chemical and/or mechanical changes of collagen fibrils, and prevention of adhesive penetration into dentin. Furthermore, the relatively higher pH of sodium bicarbonate (pH 8.3) may reduce the etching effect

of universal adhesives, leading to reduced bonding performance²²⁾. In contrast, SEM analyses of dentin surfaces in the GL group showed that smear layers were removed, and indicated the presence of dentin tubules. These observations suggest that bonding resins penetration demineralized dentin better in the GL group than in SB and control groups (Figs. 3c and d).

Bonding between dentin and resin composites depends on several factors, including adhesion and wetting characteristics. Previous studies report assessments of wetting of adhered surfaces according to contact angles^{23,24)}, and the surface-free energy of a solid (γ_s) reportedly reflects dispersion (γ_s^d), polarity (γ_s^p), and hydrogen-bonding (γ_s^h) forces²⁵⁾. Whereas dispersion forces represent London interactions between apolar molecules, polar (non-dispersion) forces include electrostatic, metallic, and dipolar interactions. In addition to these two parameters of γ_s , we determined hydrogen-bonding forces between water and hydroxyl

groups. However, because hydration of adhered surfaces strongly influences wettability, accurate determinations of polar interactions and hydrogen-bonding characteristics are warranted to assess interactions between adhesives and water²⁶. Thus, separate estimations of dipolar interactions and those of hydrogen-bonding interactions may provide novel insights into mechanisms of wettability and bonding characteristics of adhesives.

In the present study, γ_s^d values of dentin surfaces remained relatively constant (36.3–38.5 mN/m) compared with surface-free energy, regardless of the surface treatment. In a previous study, many surfaces had γ_s^d values that were close to 40 mN/m²⁷, and other studies attributed these values to adsorption of a so-called “atmospheric contamination layer”²⁸, particularly on chemically and/or mechanically activated surfaces²⁹. Hence, the presence of this atmospheric layer may explain similarities of γ_s^d values between the present treatment groups. γ_s^p Values reflect polar interactions that are often referred to as non-dispersion forces, and are a measure of hydrophilic interactions with hydroxyapatite. The present γ_s^p values were low for all specimens, and the smear layers were predominantly hydrophobic. Moreover, demineralized dentin surfaces were covered by collagen fibrils following removal of smear layers and demineralization of superficial dentin, leading to lower polar surface. Thus, the presence of smear layers in control and SB groups, and collagen fibrils on dentin surfaces in the GL group, may have contributed to lower γ_s^p values. In contrast with these parameters, total surface-free energies and γ_s^h values of GL and SB groups were significantly lower than those in the control group. Hence, surface-free energy may reflect surface characteristics following air-powder polishing, and decreased γ_s^h values may indicate the presence of residual glycine and sodium bicarbonate powders on dentin surfaces and tubules. Furthermore, these data indicate reduced wettability against the adhesive, regardless of the presence of smear layers.

Finally, bond strengths of GP were significantly lower than for the other adhesives, regardless of air-powder polishing. Potentially, these observations reflect the presence of HEMA in the latter adhesives, which may promote expansion of collagen fibrils leading to enhanced adhesive monomer penetration into demineralized dentin³⁰. Taken together, the present data indicate significant differences in bond strength and surface-free energy following air-powder polishing.

CONCLUSION

The results of this study indicate that air-powder polishing with either glycine or sodium bicarbonate powder influences SBS and surface-free energies. However, glycine powder produced smaller changes in these surface parameters than sodium bicarbonate. Hence, clinical use of air-powder polishing requires consideration of the types of powder and universal adhesives.

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