

Air-blowing strategies for improving the microtensile bond strength of one-step self-etch adhesives to root canal dentin

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The effect of different air-blowing strategies using a prototype of a newly developed clinically applicable warm air-blowing device on the microtensile bond strength (μ TBS) of one-step self-etch adhesives (1-SEAs) to human root-canal dentin was evaluated. Post cavities (8 mm depth, 1.5 mm diameter) were prepared and bonded with four 1-SEAs. Air-blowing was performed using normal air ($23\pm 1^\circ\text{C}$) for 10 or 20 s; warm air ($60\pm 1^\circ\text{C}$) for 10 or 20 s; or their combination for 10 s (5 s normal, 5 s warm) or 20 s (10 s normal, 10 s warm). After filling with corresponding core materials and 24-h water storage, μ TBS test was performed. For three of the 1-SEAs, combined air-blowing for 20 s significantly increased μ TBS compared to other air-blowing strategies ($p < 0.05$). This suggests that the combination of normal and warm air-blowing for 20 s can enhance solvent evaporation from 1-SEAs, thus resulting in their improved bonding performance to root-canal dentin.

Keywords: Root canal dentin, Microtensile bond strength, One-step self-etch adhesive, Warm air-blowing device, Air-blowing strategy

INTRODUCTION

The quality of bonding to root canal dentin is one of the most important factors in retention of post and cores in the roots and preventing coronal leakage¹. Currently, one-bottle self-etch adhesives (1-SEAs) are frequently used to simplify operations. However, 1-SEAs are intricate mixtures of hydrophilic and hydrophobic compounds and water; hence solvents such as ethanol or acetone must be contained to maintain miscibility^{2,3}. After application, the evaporation of solvents and water is essential, because their remnants negatively affect the polymerization of adhesives^{2,4-8}. The evaporation is facilitated by air-blowing⁹, but a complete evaporation is clinically difficult¹⁰, especially in the deeper areas of the root canal^{11,12}. Because of the remaining solvent and water and reduced light energy in the deeper areas¹³⁻¹⁶, bond strengths of self-etch adhesives in the apical regions are decreased compared with those in the coronal regions of the root canal^{14,17-20}.

Only few solvent removal methods for the root canal cavities have been suggested. The combination of paper point usage and air-blowing for the removal of excess adhesive in root canal resulted in improved bond strength to root canal dentin^{20,21}. Recently, warm air-blowing using a hair dryer was also reported to improve the bond strengths of 1-SEAs to radicular dentin²². The heat delivered by the warm air increased the kinetic energy of the solvent molecules and facilitated the

evaporation of residual solvent and water²³⁻²⁵. However, it is impossible to use a hair dryer clinically; therefore, a prototype of a clinically applicable warm air-blowing device was designed and tested in this study. Using the newly developed three-way dental syringe, the effect of normal, warm, and combined air-blowing strategies on the bond strength of 1-SEAs to root canal dentin was investigated. The null hypothesis was that air-blowing strategies would not affect the bond strength of 1-SEAs to root canal dentin.

MATERIALS AND METHODS

Specimen preparation

Ninety-six caries-free single-rooted human mandibular premolars with similar root length were collected following the ethical approval by the Ethics Committee of Tokyo Medical and Dental University, protocol 2013-022. The teeth were stored in distilled water at 4°C before usage within six months from the extraction. The crowns were removed using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) just below the cement-enamel junction. The roots were instrumented using endodontic files and the post cavities (8 mm depth, 1.5 mm diameter) were prepared using FibreKor drills (Pentron, Wallingford, CT, USA) in a low-speed handpiece under generous water cooling. After preparation, the post cavities were thoroughly rinsed with distilled water and dried with paper points. The root external surfaces

were built up with resin composite Clearfil AP-X (Kuraray Noritake Dental, Tokyo, Japan) to create grips for the bond-strength testing and to eliminate the effect of external light from the light-curing unit, which could

pass through the thin dentin walls (Fig. 1). The overview of materials used in this study, their compositions and application procedures are presented in Table 1.

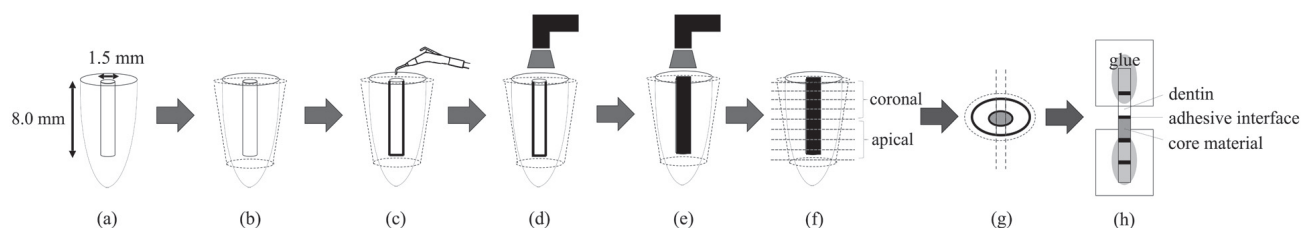


Fig. 1 Specimen preparation.

(a) The crowns of 96 lower premolars were removed and post cavities were prepared. (b) The external surfaces were built up with resin composite. (c) The cavity was dried with paper points prior to the application of adhesives. Air-blowing was performed either using with normal, warm, or combined air. (d) The adhesives (except Estelink) were light-cured for 10 s. (e) The corresponding core materials were applied and light-cured for 20 s. (f) The specimens were sectioned into 8 slabs (4 coronal, 4 apical). (g) The slabs were sectioned into $0.6 \times 0.6 \text{ mm}^2$ stick-shaped beams. (h) The μ TBS was performed.

Table 1 Overview of materials, compositions, and application protocols used in this study

Materials	Manufacturer	Composition	Application protocol	Curing mode
Scotchbond Universal (SBU) and RelyX Ultimate	3M ESPE, St. Paul, MN, USA	<adhesive> 10-MDP, γ MPTS, methacrylate-modified polyalkenoic acid copolymer, HEMA, water, ethanol, filler, photo initiator <core> Methacrylate monomer, radiopaque filler, silanated silica, CQ, chemical catalyst, accelerators	1. Apply adhesive for 20 s, mild air-blow for more than 5 s, light-cure for 10 s. 2. Apply core, light-cure for 20 s.	<adhesive> Light-curing <core> Dual-curing
Clearfil Bond SE ONE (SEO) and Clearfil DC Core Automix ONE	Kuraray Noritake Dental, Tokyo, Japan	<adhesive> 10-MDP, HEMA, water, ethanol, Bis-GMA, silica micro-filler, photo/chemical initiator <core> Silanated barium glass filler, silanated silica, Bis-GMA, TEGDMA, CQ, chemical catalyst, accelerators	1. Apply adhesive for 10 s, mild air-blow for more than 5 s, light-cure for 10 s. 2. Apply core, light-cure for 20 s.	<adhesive> Light-curing <core> Dual-curing
Unifil Core EM Self-etch Bond (UC) and Unifil Core EM	GC, Tokyo, Japan	<adhesive> 4-MET, dimethacrylate, water, acetone, silicon dioxide, photo/chemical initiator <core> Fluoro-aluminosilicate glass, UDMA, di-methacrylate, photo/chemical initiators, chemical catalyst	1. Mix equal amounts of liquid A and B. 2. Apply adhesive, leave for 30 s, mild air-blow for more than 10 s, light-cure for 10 s. 3. Apply core, light-cure for 20 s.	<adhesive> Dual-curing <core> Dual-curing
Estelink (EL) and Estecore	Tokuyama Dental, Tokyo, Japan	<adhesive> 3D SR-monomer, HEMA, phosphoric acid monomer, water, isopropyl alcohol, acetone, Bis-GMA, TEGDMA, borate catalyst, peroxide <core> Silica-zirconia filler, Bis-GMA, TEGDMA, Bis-MPEPP, peroxide, CQ, radical amplifier	1. Mix equal amounts of liquid A and B. 2. Apply adhesive, leave for 10 s, mild/strong air-blow for 5–10 s. 3. Apply core, light-cure for 20 s.	<adhesive> Chemically-curing <core> Dual-curing

10MDP, 10-methacryloyloxydecyl dihydrogen phosphate; γ MPTS, γ -methacryloxypropyl trimethoxysilane; HEMA, 2-hydroxyethyl methacrylate; CQ, camphorquinone; Bis-GMA, 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)phenyl]propane; TEGDMA, triethyleneglycol dimethacrylate; 4-MET, 4-methacryloxyethyl trimellitic acid; UDMA, urethane dimethacrylate; 3D SR-monomer, three dimensional surface-reinforcing monomer; Bis-MPEPP, 4-methacryloxy polyethoxyphenyl propane.

Bonding procedure

Four 1-SEAs and corresponding dual-curing resin-composite-based core materials, Scotchbond Universal (SBU) and RelyX Ultimate (3M ESPE, St. Paul, MN, USA), Clearfil Bond SE ONE (SEO) and Clearfil DC Core Automix ONE (Kuraray Noritake Dental), Unifil Core EM self-etch bond (UC) and Unifil Core EM (GC, Tokyo, Japan), and Estelink (EL) and Estecore (Tokuyama Dental, Tokyo, Japan), were used. The curing modes of 1-SEAs were light-curing for SBU and SEO, chemically-curing for EL, and dual-curing for UC. All core materials were dual-curing. Each 1-SEA was applied to the radicular dentin following the respective manufacturers' instructions. Solvent and water evaporation was performed using normal air ($23\pm 1^\circ\text{C}$) for 10 s (N10s) and 20 s (N20s), warm air ($60\pm 1^\circ\text{C}$) for 10 s (W10s) and 20 s (W20s), or their combination for 10 s (C10s, 5 s normal air+5 s warm air) and 20 s (C20s, 10 s normal air+10 s warm air). Normal air-blowing was performed from a distance of 1 cm above the orifice of the post-cavity at air pressure of approximately 3.5 kg cm^{-2} . Warm air-blowing was performed using a prototype of a warm air generator (Osada Electric, Tokyo, Japan, Fig. 2) from a distance of 5 mm above the post-cavity orifice at air pressure of approximately 0.6 kg cm^{-2} . Then, the 1-SEAs except EL were light-cured following the manufacturers' instructions using a halogen light-curing unit Optilux 501 (Demetron Kerr, Danbury, CA, USA) at 600 mWcm^{-2} . The post cavities were filled with the corresponding core materials and light-cured according to the manufacturers' instructions (Table 1, Fig. 1). The procedures were performed at a constant room temperature ($23\pm 1^\circ\text{C}$) and 60% relative humidity.

Microtensile bond strength (μTBS) testing

After 24-h water storage at 37°C , the bonded specimens were cut perpendicularly to the bonded interface into eight slabs using a low speed diamond saw under water

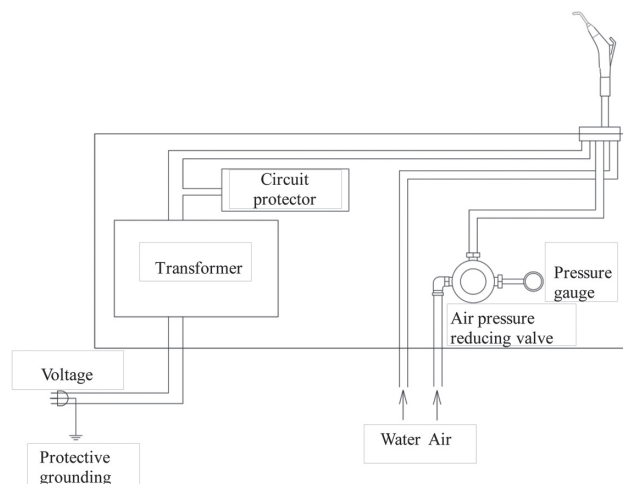


Fig. 2 Schematics of the prototype of warm air generator connected to a three-way dental syringe.

cooling (Fig. 1). The slabs from each tooth were divided into two subgroups: Four slabs from the coronal third of the root (“coronal region”) and four slabs from the middle third (“apical region”). The slabs were transversally sectioned in the central part into $0.6\times 0.6 \text{ mm}^2$ beams and the dimensions of their cross-sectional areas were checked using a digital caliper (Mitutoyo CD15, Mitutoyo, Kawasaki, Japan). Using a cyanoacrylate glue (Zapit, DVA, Anaheim, CA, USA), the beams were attached to a testing jig and subjected to a tensile load in a table-top testing machine (EZ Test Shimadzu, Kyoto, Japan) at a crosshead speed of 1 mm/min until failure. The force at failure was recorded in Newton (N) and converted to μTBS values (MPa). Because Levene's test indicated inhomogeneity of variances, the μTBS values were statistically analyzed using a Student's *t*-test with Bonferroni correction for multiple comparisons at a significance level of 0.05.

Failure mode analysis

Both the resin side and the dentin side of the fractured beams were desiccated before mounting on brass stubs and sputter-coating with gold. Four different failure modes were determined using a scanning electron microscope (SEM; JSM-IT100, JEOL, Tokyo, Japan): cohesive failure in the core material (over 70% of the area within the core material), adhesive failure (over 70% of the area within the adhesive resin or at the resin-dentin interface), cohesive failure in dentin (over 70% of the area within the dentin), and mixed failure (combination of cohesive and adhesive failure).

Mass loss during air-blowing

Approximately $15 \mu\text{L}$ of each 1-SEA, which corresponds to a coat with a saturated microbrush, were applied to a tared, flat container (diameter 9.0 cm). The baseline mass of the adhesive (rounded to the nearest 0.1 mg) was immediately recorded using an electric balance (GR-202, A&D, Tokyo, Japan). Firstly, normal or warm air-blowing was periodically applied, and the remaining mass of the adhesive was recorded every 10 s until the container stopped losing weight to measure the maximal amount of evaporable compounds. Then, the loss of mass was measured for each air-blowing strategy and divided by the previously measured maximal evaporable mass to obtain comparable percentage values among the different 1-SEAs. The experiments were performed at 5 cm distance from the container, room temperature (23°C), and 60% relative humidity. Light filters were used to assure the protection of the adhesives from external light. Ten specimens of each 1-SEA per each air-blowing strategy were used.

RESULTS

The mean μTBS s and standard deviations are presented in Table 2. Specimens from the coronal region exhibited significantly higher μTBS s than those from the apical region regardless of the material and the air-blowing strategy ($p < 0.005$, except for EL W10s $p = 0.011$). The

Table 2 Microtensile bond strength to root canal dentin: Mean±S.D. (MPa)

		N10s	N20s	W10s	W20s	C10s	C20s
SBU	Coronal	32.1±6.3 ^{Aa}	33.3±6.1 ^{Aa}	34.2±9.0 ^{Aa}	36.0±6.5 ^{Aa}	30.2±5.3 ^{Aa}	44.8±7.3 ^{Ba}
	Apical	19.3±4.0 ^{Ab}	21.0±6.2 ^{Ab}	19.6±5.9 ^{Ab}	23.3±6.8 ^{Ab}	20.5±5.1 ^{Ab}	37.6±5.8 ^{Bb}
SEO	Coronal	33.3±7.2 ^{Aa}	32.9±6.2 ^{Aa}	33.4±12.6 ^{Aa}	31.1±5.8 ^{Aa}	30.8±6.6 ^{Aa}	33.8±7.5 ^{Aa}
	Apical	20.8±5.4 ^{Ab}	21.6±6.2 ^{Ab}	24.8±10.1 ^{Ab}	21.7±5.2 ^{Ab}	20.9±5.4 ^{Ab}	25.3±7.6 ^{Ab}
UC	Coronal	32.9±9.7 ^{Aa}	32.4±5.7 ^{Aa}	33.6±8.2 ^{Aa}	35.1±6.1 ^{Aa}	30.3±6.2 ^{Aa}	44.6±6.5 ^{Ba}
	Apical	19.6±5.0 ^{Ab}	22.2±5.3 ^{Ab}	22.0±6.3 ^{Ab}	22.8±6.4 ^{Ab}	21.1±4.8 ^{Ab}	30.1±7.3 ^{Bb}
EL	Coronal	24.4±8.5 ^{Aa}	26.6±7.2 ^{Aa}	25.3±4.8 ^{Aa}	26.7±6.3 ^{Aa}	25.9±5.1 ^{Aa}	34.2±8.9 ^{Ba}
	Apical	15.2±4.6 ^{Ab}	17.6±5.2 ^{Ab}	19.5±7.6 ^{Ab}	18.0±4.8 ^{Ab}	17.4±4.0 ^{Ab}	19.1±7.1 ^{Ab}

Different superscript letters indicate significant differences between groups ($p < 0.05$); uppercase letters in rows, lowercase letters between coronal and apical region within each adhesive group. N, normal air; W, warm air; C, combined normal and warm air; SBU, Scotchbond Universal; SEO, Clearfil Bond SE One; UC, Unifil Core EM Self-etch Bond; EL, Estelink.

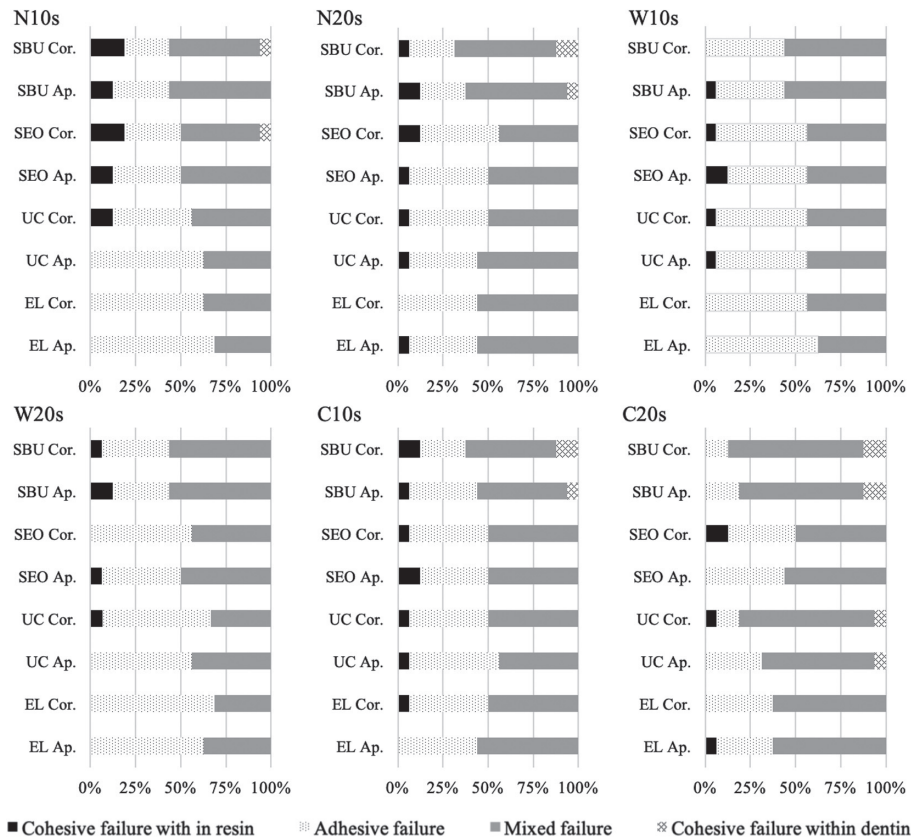


Fig. 3 Failure mode distributions. N, normal air; W, warm air; C, combined normal and warm air; SBU, Scotchbond Universal; SEO, Clearfil Bond SE One; UC, Unifil Core EM Self-etch Bond; EL, Estelink; Cor., Coronal region; Ap., Apical region.

C20s strategy significantly increased the μ TBS of SBU ($p < 0.005$), UC ($p < 0.005$) and EL ($p < 0.02$) in the coronal region, and UC ($p < 0.03$) and SBU ($p < 0.001$) in the apical region, compared with those of other air-blowing strategies. However, C20s did not affect significantly the μ TBS of EL in the apical region and of SEO ($p > 0.05$). No

significant differences were found among the other air-blowing strategies.

The predominant failure modes were adhesive and mixed. For SBU, UC, and EL in the coronal region, the C20s strategy tended to decrease the number of adhesive failures compared with that by the other air-

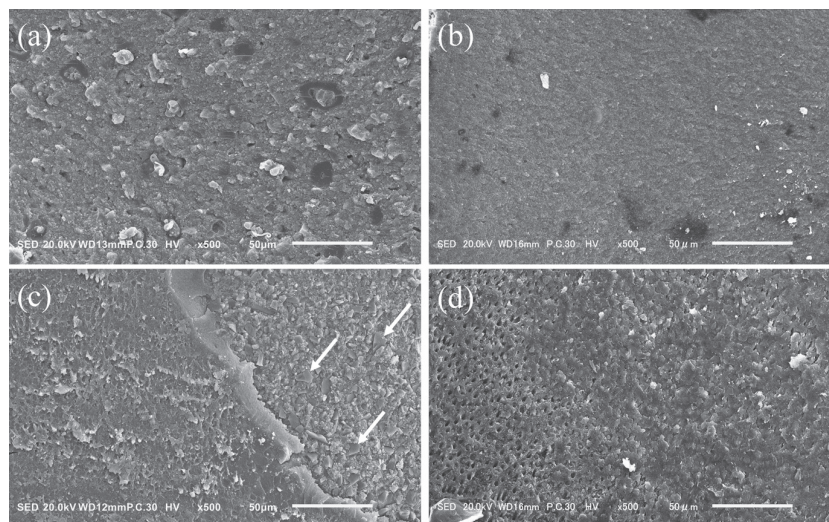


Fig. 4 Detailed SEM images. (a) RelyX Ultimate —cohesive failure within resin cement, exposed filler particles were observed. (b) Clearfil Bond SE One —adhesive failure. (c) Unifil Core EM —mixed failure, arrows point at fluoro-aluminosilicate glass filler particles of the resin cement. (d) Estelink —dentin-adhesive interfacial failure.

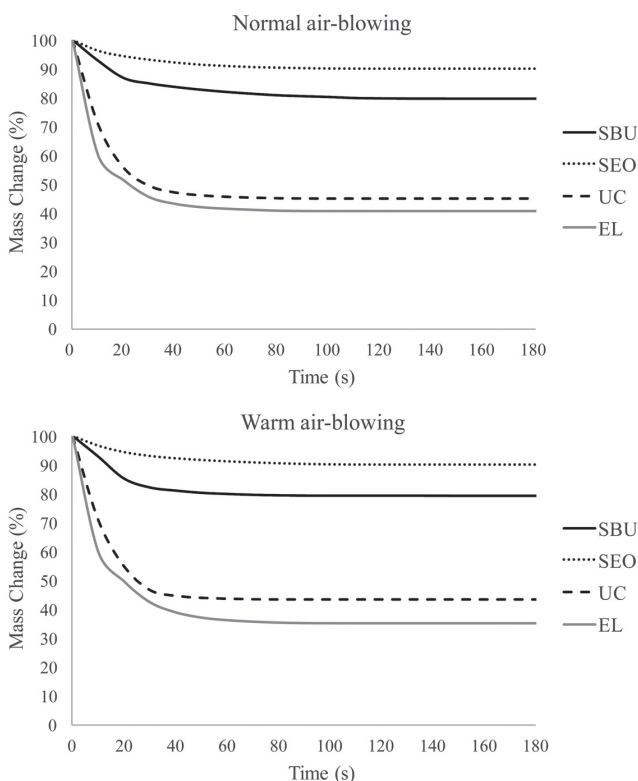


Fig. 5 Maximal loss of adhesives' mass. The loss of adhesives' mass during solvent evaporation until the container stopped the losing weight using normal air-blowing and warm air-blowing. SBU, Scotchbond Universal; SEO, Clearfil Bond SE One; UC, Unifil Core EM Self-etch Bond; EL, Estelink.

blowing strategies. Figure 3 presents the failure mode distribution in each group and Fig. 4 detailed SEM images of each material and failure mode. There were no pre-testing failures during specimen preparation for the μ TBS test.

Figure 5 depicts the maximal mass loss for all 1-SEAs using normal or warm air-blowing. The maximal mass loss of the acetone-based 1-SEAs UC (56%) and EL (65%) was significantly higher than that of ethanol-based SBU (20%) and SEO (10%). Warm air-blowing resulted in a slightly higher maximal mass loss compared with that by normal air-blowing. Table 3 presents the relative mass loss for different air-blowing strategies. Air-blowing for 20 s exhibited more effective evaporation than that by 10 s strategies for all 1-SEAs. C20s was the most effective air-blowing strategy for SBU, UC, and EL, evaporating more than 90% of the maximal mass loss. For SEO, C20s (53.3%) was comparable to W20s and N20s (both 56.7%).

DISCUSSION

The removal of volatile solvents and water is a significant factor for the bonding performance of 1-SEAs to root canal dentin^{20,22}. Taguchi *et al.* reported that the μ TBS of 1-SEAs to radicular dentin was enhanced by warm air-blowing using a hair dryer²². Because hair dryers are not clinically applicable, we have developed an experimental warm-air-generating device, which can be connected to a dental three-way syringe. However, the pressure of warm air generated by the device is low (0.6 kg cm⁻²) compared with the pressure of normal air (3.5 kg cm⁻²). We have attempted to increase the warm air pressure but its temperature decreased, therefore, we have invented

Table 3 Relative mass loss in percentages for different air-blowing strategies: Mean±S.D.(%)

	N10s	N20s	W10S	W20s	C10s	C20s
SBU	35.1±8.9	64.0±7.4	34.7±5.6	70.2±5.0	38.5±5.5	95.7±5.2
SEO	37.9±6.5	56.7±7.0	34.5±6.8	56.7±11.6	40.2±8.4	53.3±6.7
UC	52.0±3.2	78.5±3.2	51.6±2.4	79.7±1.6	52.4±2.6	90.4±2.7
EL	62.1±1.9	75.4±2.3	61.9±2.4	77.3±3.6	62.5±2.7	99.2±2.3

N, normal air; W, warm air; C, combined normal and warm air; SBU, Scotchbond Universal; SEO, Clearfil Bond SE One; UC, Unifil Core EM Self-etch Bond; EL, Estelink.

the combination of normal and warm air-blowing. When normal air-blowing for 10 s was followed by 10 s of warm air-blowing (C20s), significantly increased μ TBS values of SBU, UC and EL in the coronal region were obtained compared with those by standard air-blowing (normal air, 10 s) and the other strategies tested. Therefore, the null hypothesis that air-blowing strategies would not affect the bond strength of 1-SEAs to root canal dentin was rejected.

The significant increase in μ TBSs using the C20s strategy can be attributed to its efficiency in solvent evaporation. Low amounts of residual solvent and water were reported to improve the polymerization of adhesives^{26–28}, and the loss-of-mass test revealed that more than 90% of the evaporable mass was removed for SBU, UC and EL. Compared with the other strategies, C20s also tended to fail less often at the resin-dentin interface in the groups where the μ TBS increased significantly. We assume that with C20s strategy normal air-blowing was effective for the initial evaporation, given its higher air-pressure, and that the warm air-blowing succeeded in the residual solvent and water removal, because of the increased kinetic energy of the heated molecules and their higher vapor pressure. On the other hand, the low pressure of warm air (0.6 kg cm⁻²) could possibly cause that some solvent remained in the apical region, resulting in a significant improvement of μ TBS of EL only in the coronal region but not in the apical region using the C20s strategy.

No significant difference was observed in any test for SEO when C20s was used compared with that of the other air-blowing strategies. The loss-of-mass test showed that only 10% of the SEO's mass could be evaporated, the lowest value of all the 1-SEAs. Because significantly lower portions of mass were evaporated from the ethanol-based adhesives, the previous finding that ethanol-based solutions retain significantly more water and solvent than that by acetone-based ones is confirmed²⁹. Furthermore, more solvent is retained in more hydrophilic blends, especially in ethanol-based one, because both ethanol and water can create hydrogen bonds with the monomers²⁹. It was also reported that the content of HEMA in adhesives interferes the evaporation of solvents³⁰. Despite the apparent similarity of the composition of ethanol-based 1-SEAs used in this study, the proportion of the compounds seems to be different,

because the maximal mass loss of for SBU was 20%. Therefore, we speculate that higher proportion of HEMA and water in SEO could result in higher retention of water and ethanol, which would adversely affect the polymerization and mechanical properties of the adhesive. Moreover, the 10 s application time of SEO is shorter than that of SBU (20 s), which could reportedly also affect the solvent evaporation¹.

The other air-blowing strategies, N20s, W10s, W20s, and C10s, did not exhibit any significant difference in μ TBS for any of the adhesives compared with that of the standard air-blowing strategy N10s. N20s and W20s exhibited higher mass than that by the 10 s strategies, however, the remaining solvent or water seemingly precluded any significant improvement. We assume that the heat necessary to increase the vapor pressure was missing in N20s compared with C20s. For the W20s strategy, we speculate that the pressure of warm air was too low to sufficiently remove the solvent and that this precluded any bond strength improvement. Among the 10 s strategies, there were no significant differences between normal, warm, and combined air-blowing.

A previous study of Taguchi *et al.* reported that W20s using a hair dryer increased the the μ TBS of four 1-SEAs to root canal dentin both in coronal and in apical regions, however, the difference was not significant for SEO and UC in the coronal region²². Similarly, warm air-blowing resulted in higher maximal mass loss of the adhesives²². On the other hand, the mass loss of SEO and EL were markedly higher²² compared with the results in this study. This may be explained by the higher air temperature (80±1°C) and higher air pressure obtained with the hair dryer compared with the newly developed prototype of a three-way dental syringe used in this study. We assume that the pressure and temperature of the warm air generated by the tested device were insufficient to evaporate the solvent and water residue in the apical region, thus leading to the discrepancy between the results.

Besides the effect of heat on the vapor pressure, the increased kinetic energy may stimulate polymerization. This could be beneficial especially for light-curing or dual-curing materials in the apical regions, where the light-energy is attenuated^{17,18,23,31–34}. However, apical regions exhibited significantly lower μ TBS than coronal regions for all materials and air-blowing strategies. This agrees

with previous studies that also revealed decreased bond strengths in the deeper regions of post cavities^{18,22,23}. The results of this study and of Taguchi *et al.*²² show that warm air-blowing improved the solvent evaporation and bond strength in both coronal and apical regions, but cannot eliminate the difference between them.

To improve the bond strength in apical regions, the promotion of polymerization may have desirable results. It has been reported that prolonged irradiation time^{13,14} and higher irradiance of the light-curing unit¹⁴ eliminate the difference between the coronal and apical specimens. Recently, the use of specialized accelerators (“touch-cure” systems) has been introduced in adhesive systems and core materials to promote chemical polymerization. However, despite the presence of the touch-cure system, lower bond strengths were obtained in apical regions^{14,20,35}.

CONCLUSION

It can be concluded that the bond strength of 1-SEAs to root canal dentin can be improved using the combination of normal and warm air-blowing for 10 s each. This strategy was efficient for the evaporation of solvent and water from 1-SEAs. A prototype warm air-blowing device, which can be connected to a three-way dental syringe for clinical use, was successfully tested. However, the pressure of warm air was very low (0.06 MPa) compared with that of the normal air device (0.35 MPa). Therefore, further development of this device is necessary to increase the air pressure that will improve the efficiency of the warm air-blowing and its clinical application.

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CONFLICT OF INTEREST

The authors do not have any financial interest in the companies whose materials are included in this article.

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