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

Kazuhide YONEKURA1, Keiichi HOSAKA1, Antonin TICHY1,2, Keita TAGUCHI1, Masaomi IKEDA3, Ornicha THANATVARAKORN4, Taweesak PRASANSUTTIPORN5, Masatoshi NAKAJIMA1 and Junji TAGAMI1

1 Department of Cariology and Operative Dentistry, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University (TMDU), 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan
2 Institute of Dental Medicine, First Faculty of Medicine of the Charles University and General University Hospital in Prague, Karlovo namesti 32, Prague 121 11, Czech Republic
3 Department of Oral Prosthetic Engineering, Graduate School of Medical and Dental Sciences, Medical and Dental Science and Technology, Tokyo Medical and Dental University (TMDU), 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan
4 Faculty of Dentistry, Bangkok Thonburi University, 16/10 Taweewatana, Bangkok 10170, Thailand
5 Department of Restorative Dentistry and Periodontology, Faculty of Dentistry, and Center of Excellence in Materials Science and Technology, Chiang Mai University, T. Suthep, A. Muang, Chiang Mai 50200, Thailand
Corresponding author, Keiichi HOSAKA; E-mail: hosaka.ope@tmd.ac.jp

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 (μTBS) 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±1°C) for 10 or 20 s; warm air (60±1°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, μTBS test was performed. For three of the 1-SEAs, combined air-blowing for 20 s significantly increased μTBS 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 leakage11. 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 miscibility2,3. After application, the evaporation of solvents and water is essential, because their remnants negatively affect the polymerization of adhesives2,4-8. The evaporation is facilitated by air-blowing9, but a complete evaporation is clinically difficult10, especially in the deeper areas of the root canal11,12. 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 canal14,17-20.

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 dentin20,21. 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.

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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, γMPTS, methacrylate-modified polyalkenoic acid copolymer, HEMA, water, ethanol, filler, photo initiator  | 1. Apply adhesive for 20 s, mild air-blow for more than 5 s, light-cure for 10 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  | 1. Apply adhesive for 10 s, mild air-blow for more than 5 s, light-cure for 10 s.  | <adhesive> Light-curing <core> Dual-curing     |
|                          |                         | <core> Silanated barium glass filler, silanated silica, Bis-GMA, TEGDMA, CQ, chemical catalyst, accelerators  | 2. Apply core, light-cure for 20 s.  | <adhesive>               |
| 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  | 1. Mix equal amounts of liquid A and B.  | <adhesive> Light-curing <core> Dual-curing     |
|                          |                         | <core> Fluoro-aluminosilicate glass, UDMA, di-methacrylate, photo/chemical initiators, chemical catalyst  | 2. Apply adhesive, leave for 30 s, mild air-blow for more than 10 s, light-cure for 10 s.  | <adhesive> Light-curing <core> Dual-curing     |
|                          |                         |                                                                            | 3. Apply core, light-cure for 20 s.  | <adhesive>               |
| Estelink (EL) and Estecore | Tokuyama Dental, Tokyo, Japan | <adhesive> 3D SR-monomer, HEMA, phosphoric acid monomer, water, isopropl alcohol, acetone, Bis-GMA, TEGDMA, borate catalyst, peroxide  | 1. Mix equal amounts of liquid A and B.  | <adhesive> Light-curing <core> Dual-curing     |
|                          |                         | <core> Silica-zirconia filler, Bis-GMA, TEGDMA, Bis-MPEPP, peroxide, CQ, radical amplifier  | 2. Apply adhesive, leave for 10 s, mild/strong air-blow for 5–10 s.  | <adhesive> Light-curing <core> Dual-curing     |
|                          |                         |                                                                            | 3. Apply core, light-cure for 20 s.  | <adhesive> Light-curing <core> Dual-curing     |

10MDP, 10-methacryloyloxydecyl dihydrogen phosphate; γMPTS, γ-methacryloyxypropyl 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-methacryloyethyl trimellitic acid; UDMA, urethane dimethacrylate; 3D SR-monomer, three dimensional surface-reinforcing monomer; Bis-MPEPP, 4-methacryloyloxy 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±1°C) for 10 s (N10s) and 20 s (N20s), warm air (60±1°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±1°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 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 0.6×0.6 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 μL 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 μTBSs and standard deviations are presented in Table 2. Specimens from the coronal region exhibited significantly higher μTBSs 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±6.3Aa | 33.3±6.1Aa | 34.2±9.0Aa | 36.0±6.5Aa | 30.2±5.3Aa | 44.8±7.3Ab |
| Apical | 19.3±4.0Ab  | 21.0±6.2Ab  | 19.6±5.9Ab  | 23.3±6.8Ab  | 20.5±5.1Ab  | 37.6±5.8Ab  |
| SEO   |        |        |        |        |        |        |
| Coronal | 33.3±7.2Aa | 32.9±6.2Aa  | 33.4±12.6Aa | 31.1±5.8Aa  | 30.8±6.6Aa  | 33.8±7.5Aa  |
| Apical | 20.8±5.4Ab  | 21.6±2.2Ab  | 24.8±10.1Ab | 21.7±5.2Ab  | 20.9±5.4Ab  | 25.3±7.6Ab  |
| UC    |        |        |        |        |        |        |
| Coronal | 32.9±9.7Aa | 32.4±5.7Aa  | 33.6±8.2Aa  | 35.1±6.1Aa  | 30.3±6.2Aa  | 44.6±6.5Ab  |
| Apical | 19.6±5.0Ab  | 22.2±5.3Ab  | 22.0±6.3Ab  | 22.8±6.4Ab  | 21.1±4.8Ab  | 30.1±7.3Ab  |
| EL    |        |        |        |        |        |        |
| Coronal | 24.4±8.5Aa | 26.7±6.2Aa  | 25.3±4.8Aa  | 26.7±6.3Aa  | 25.9±5.1Ab  | 34.2±8.9Ab  |
| Apical | 15.2±4.6Ab  | 17.6±5.2Ab  | 19.5±7.6Ab  | 18.0±4.8Ab  | 17.4±4.0Ab  | 19.1±7.1Ab  |

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.

C20s strategy significantly increased the μTBS 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 μTBS 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.

**DISCUSSION**

The removal of volatile solvents and water is a significant factor for the bonding performance of 1-SEAs to root canal dentin\(^\text{20-22}\). Taguchi \textit{et al.} reported that the μTBS of 1-SEAs to radicular dentin was enhanced by warm air-blowing using a hair dryer\(^\text{22}\). 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...
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 μTBS 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 μTBSs 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 adhesives26-28), 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 μTBS 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 resin-dentin interface in the groups where the μTBS increased. 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 solvents29). 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 evaporation41).

The other air-blowing strategies, N20s, W20s, and C10s, did not exhibit any significant difference in μTBS 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 μTBS 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 attenuated17,18,21,31-34). However, apical regions exhibited significantly lower μTBS than coronal regions for all materials and air-blowing strategies. This agrees

|                | 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.
with previous studies that also revealed decreased bond strengths in the deeper regions of post cavities.\(^{15,22,23}\) The results of this study and of Taguchi et al.\(^{20}\) 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 unit\(^ {14}\) eliminated 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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