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Effect of air moisture content on adhesion to dentine: a comparison of dental air/water syringe tips

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Abstract - *This study aimed to evaluate the spray pattern and air moisture content produced by single-use syringe and multiple-use syringe tips. The drying efficacy was evaluated by analyzing the spray and by detecting the presence of moisture in the air blast through the tips. Single-use tips had a more consistent spray pattern and produced a moisture-free airflow compared to the multiple-use tips. The differences were statistically significant. Adhesion to dentine between tooth preparations dried with the two tips was evaluated using a tensile test. The differences were statistically insignificant.*

KEY WORDS: Bond strength, Dentine, Moisture, Spray pattern, Syringe tips

INTRODUCTION

Today, we are in the age of adhesive dentistry where techniques allow deteriorating restorations to be repaired and debonded restorations to be replaced with little or no additional loss of tooth structure.[1] Adhesion reduces microleakage at the restoration-tooth interface. Prevention of microleakage reduces postoperative sensitivity, marginal staining, and recurrent caries, all of which may jeopardize the clinical longevity of restorative efforts.[2,3]

Dentine is an intrinsically wet tissue, due to its composition and structure.[1] This makes it a more difficult substrate for bonding than enamel, as water competes for the adhesion sites on the hard tissue, affecting adhesion.[4] The fundamental principle of adhesion to tooth substrate is based upon an exchange process by which inorganic tooth material is exchanged for synthetic resin.[5] This process involves two phases – one phase consists of removal of calcium phosphate, by which microporosities are exposed in both enamel and dentine surfaces. The second phase, termed the hybridization phase, involves infiltration and subsequent in situ polymerization of resin within the created surface microporosities.[1]

It is during this hybridization phase that dentine moisture control is important, as this is when the hydrophilic dentine surface is transformed into a hydrophobic and spongy state that allows the adhesive resin to penetrate the exposed collagen network efficiently.[6,7,8] Surface moisture control is key to obtaining optimal bonding, especially for adhesion of resin composites to dentine.[9] After conditioning with etch, the demineralized collagen network is susceptible to collapse when water is removed by drying. Collapse and subsequent shrinkage of collagen can lead to suboptimal resin infiltration.[1] At the same time, excessive moisture dilutes the primer and renders it less effective.[10] Excessive moisture also impedes effective penetration of adhesive monomers as water molecules and resin composites compete for space inside the demineralized dentine, resulting in inadequate resin-dentine infiltration.[4] Such deficiencies weaken the bond. It is fundamentally important to effective hybridization that the collagen fibril web in dentine, after being deprived of its mineral support following acid treatment, keeps its sponge-like quality, allowing interdiffusion of monomers in the subsequent priming and bonding steps.

Single-use air/water syringe tips (SU) are an alternative to multiple-use syringes (MU) to limit cross infection.[11,12] MU tips are made of metal to allow autoclave sterilization, whereas SU tips are made of plastic and are pre-sterilized. Water from MU tips when only air is required presents a problem when drying dentine.

When both air and water are activated to produce an air/water spray, an aerosol results. One of the important objectives in developing an aerosol product is to obtain the spray pattern best suited for the intended application. This study had two aims: To evaluate the spray pattern and air moisture content produced by the tips, and to compare the effectiveness of the SU and MU tips for drying dentine surfaces via measurements of resin-dentine adhesion strength.

The first null hypothesis was that there would be no difference in the spray pattern and moisture content obtained with the SU versus MU tips. The second null hypothesis was that there would be no difference in adhesion strengths between surfaces dried with the tips.

MATERIALS AND METHODS

1. Spray Pattern Analysis

Spraying water onto black coloured paper produced the spray patterns of 20 SU and 20 MU tips, with the tip 50 mm away to mimic the clinical situation. For each specimen, the air nozzle was first activated at a set pressure of 350

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kPa for a total of 10 seconds to eliminate any fluctuations in air pressure over the initial few seconds. Then, without releasing the air nozzle, the water nozzle was activated concurrently for 3 seconds to produce an air/water spray. A camera (Nikon D3000) was set up on a tripod and an image of the spray pattern was captured immediately after each test and its area and circularity analyzed using "FIJI is Just Image J", an image processing software. The outline of each spray pattern was digitally traced three times, and the mean area and circularity calculated. All MU tips that were examined in this study have been used in the mouth during clinical examination and treatment at the University of Otago School of Dentistry, and were randomly selected from the existing pool of instruments. SU tips were obtained from the Acteon Company, Bordeaux, France.

Statistical Analysis

The spray pattern results were analyzed by calculating the means of the area, perimeter and circularity of the two groups. Data were normally distributed, and variances were homogenous. Hence, the differences in the mean were tested for statistical significance ($P < 0.05$) using the Mann-Whitney U-test (SPSS for Windows 2007). A 2-tailed Pearson Correlation test was generated to analyze the linear relationship between the quantitative variables (area, perimeter and circularity).

2. Humidity Test

A solution of anhydrous cobalt (II) chloride (0.5% weight percentage) and 96% pure ethanol (99.5% weight percentage) was prepared by the Chemistry Department, University of Otago and 5 mL was dispensed into each of 40 test tubes. Twenty SU and twenty MU tips were tested. The change in colour from deep blue to pink indicated the presence of remaining moisture in the expelled air, as shown by the reaction:

$[CoCl_4]^{2-}$ (blue) + 6 H_2O $\rightleftarrows$ $[Co(H_2O)_6]^{2+}$ (pink) + 4 Cl^-

Air and water was dispensed from each syringe tip for 15 seconds to simulate the washing of a tooth surface. The syringe was then inserted into a test tube and air alone was dispensed for 10 seconds.

Statistical Analysis

A table of positive or negative results was generated and tested for statistical significance ($P < 0.05$) using the Pearson Chi-Square test.

3. Tensile Bond Strength (TBS) Testing

Ethical approval was obtained for the use of extracted human teeth. Permanent third molar teeth, stored in phosphate buffered saline (PBS) since extraction, were selected using criteria that rejected those with caries, cracks or other defects. Teeth were sectioned along the buccal aspect of the tooth using a carborundum disc and then embedded in cold cure acrylic resin blocks (Castapress). Consistent flat dentine surfaces were produced on the buccal aspect by wet grinding at 300 rpm with 120 μm grit SiC paper (Grinding/polishing machine, Stuers TegraPol-21 & TegraForce5). Specimens were randomly allocated into two groups of twenty teeth labeled A and B. Group A were dried with the SU tips, and group B dried with the MU tips.

Forty glass fibre-reinforced composite tapered root canal posts (Rebilda Post 20) were cleaned with isopropyl alcohol. Posts for attachment to group A teeth were dried with a gentle stream of air using the SU tips, and posts for group B were dried in a similar fashion using MU tips. A silane coupling agent was applied to the coronal ends of the posts, and a gentle stream of air used to disperse it. The material was left undisturbed for 60 seconds as per manufacturer's instructions.

One 2.0 mm diameter post was bonded to the dentine of each tooth. The flat bonding surface of the tooth was aligned horizontally and checked with a spirit level. The tooth was cleaned with air and water spray for 15 seconds using the respective tips at 350 kPa pressure. Then, an air-only spray was dispensed perpendicular to the tooth surface for 10 seconds from a distance of 50 mm.

Adhesive was applied at the base of the posts (RelyX™ Unicem 2 Self-Adhesive Resin Cement). Posts were placed perpendicular to the dentine surfaces with a vertical force of 4.75 N provided by a custom-made apparatus to ensure consistent adhesive thickness. Excess cement was removed. The apparatus guided the post to create a perfectly perpendicular bond to the dentine, ensuring only axial forces were applied during the tensile test. The cement was then light-cured from three different angles in 20-second bursts. The specimens were stored in PBS for 24 hours before the tensile test. The same dental unit was used throughout the study and the same investigator carried out all preparations and tests.

Tensile testing was carried out using a constant rate of extension machine fitted with a 50 N load cell (Lloyd Microtensile tester, Model T5002). Specimens were prepared for testing by attaching lengths of brass tubing (internal diameter 2.0 mm) to the posts bonded to the teeth. One end of the tubing was compressed flat and perforated with a bur. The tube was attached to the post with cyanoacrylate adhesive (Permbond). The flattened end was connected to the crosshead of the testing machine using a custom-made self-aligning grip. The grip ensured no force was applied to the specimen during the mounting procedure and provided a consistent vertical force during the test. Each specimen, mounted in an acrylic block, was secured in the lower clamp of the testing machine in a horizontal position using a spirit level. Efforts were made to ensure the specimens were secured with the post in line with the central axis of the crosshead grip.

Specimens were tested to failure at an extension rate of 0.5 mm/second. Force-extension data for each specimen were recorded using LabChart 7 for Windows 2007. Force at failure was recorded from the plotted data and manually converted to tensile stress (MPa) using the surface area of the bond.

Statistical Analysis

The tensile strength results were analyzed by calculating the means of the two groups. Data were not normally distributed and were tested for statistical significance ($P < 0.05$) using the Mann-Whitney U-test.

4. Fracture Topography and Fracture Mode

After the tensile tests were completed, the failure surfaces of all specimens were examined with a light microscope at x25 magnification and the findings confirmed under the SEM.

Specimens were coated with 10 nm of gold palladium in a Peltier-cooled high-resolution sputter coater (Emitech K575X), and examined using a field emission microscope (JEOL JSM-6700F).

Statistical Analysis

The fracture mode data were tested for statistical significance ($P < 0.05$) using the Chi-squared test.

RESULTS

1. Spray Pattern Analysis

The spray pattern analysis showed that the SU created a greater spray area and a more circular pattern compared to the MU (Table 1). These differences were statistically significant ($P < 0.001$). The MU produced a larger perimeter than the SU, although the difference was not significant ($P = 0.430$). A larger perimeter suggests that the spray pattern is more irregular in its outline.

A moderate positive relationship was found between the area and the perimeter of the spray pattern ($P = 0.02$). This suggests that the larger the spray perimeter, the larger the spray area. The same correlation could be seen between the area and the circularity of the spray pattern (Table 1). There was, however, a strong negative relationship between the circularity and perimeter of the spray pattern obtained ($P < 0.001$). This result was consistent with the fact that the more regular and circular an outline is, the smaller the perimeter ought to be. The greater the perimeter, the more irregularly-shaped the pattern is, and hence, less circular.

2. Humidity Tests

Eighteen of the twenty (90%) SU tips tested during the experiment had no moisture in the air dispensed, compared to eight out of twenty (40%) of the MU types. This difference was statistically significant ($P = 0.001$).

3. Tensile Bond Strength (TBS)

The mean tensile bond strength for teeth dried by the two different syringes is shown in Table 2. There was no statistical difference in the adhesion strengths obtained ($P = 0.883$).

Table 1. Spray Pattern Analysis: Mean area, perimeter and circularity produced by the drying protocol using the SU versus MU tips, and Pearson's Correlation showing the relationship between the three quantitative variables of the spray pattern – Area, perimeter and circularity.

Type of Syringe used	*N*	*Area (cm²)*	*Perimeter (cm)*	*Circularity**
Multiple-use	20	78.5	38.3	0.7
Single-use	20	58.8	40.1	0.5
		P < 0.001	P = 0.430	P < 0.001
	Area vs Perimeter	*Area vs Circularity*	*Circularity vs Perimeter*	
Correlation	0.37	0.43	-0.65	
	P = 0.02	P = 0.005	<0.001	

* Circularity = A measurement of how circular a shape is. A circularity value of 1.0 indicates a perfect circle. As the value approaches 0.0, it indicates an increasingly elongated polygon.

Table 2. Mean tensile adhesion strengths for drying protocol using SU and MU tips (MPa).

Type of Syringe used	*N*	*Mean adhesion strength (SD)*
Single-use	20	5.72 (3.91)*
Multiple use	20	5.92 (4.75)*

SD = Standard deviation
*P = 0.48

Table 3. Failure modes experienced when using SU and MU tips.

Type of Syringe used	*Adhesive failure only*	*Cohesive Failure only*	*Mixed Failure*
Single-use	13	0	7
Multiple use	5	0	15

P = 0.011

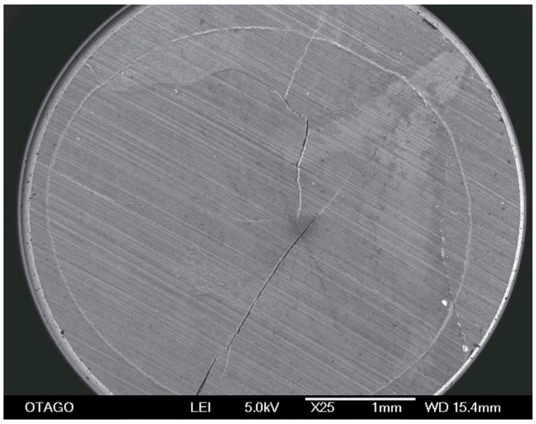

Figure 1. *Adhesive failure on a tooth surface dried using a multiple-use syringe at magnification x25.*

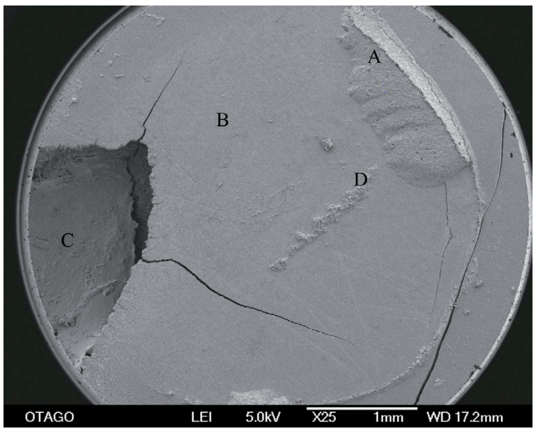

Figure 2. *Mixed mode of failure on a tooth surface dried using a single-use syringe at magnification x25.* **A** *– RelyX Adhesive,* **B** *– Dentine,* **C** *– Broken tooth structure,* **D** *– Broken post*

4. Fracture Topography and Fracture Mode (SEM)

Images of the specimens revealed pure adhesive failure (Figure 1), and a mixed mode of failure consisting of adhesive and cohesive failure of the adhesive material, failure of the post and tooth structure (Figure 2). There was a statistical difference in the mode of failure observed (P = 0.011). There was no pure cohesive failure.

DISCUSSION

There was a difference observed in the spray pattern and moisture content obtained with the SU versus MU tips, and the first null hypothesis was rejected. The two drying protocols tested in bonding a post to dentine produced adhesive strengths of similar values and the second null hypothesis was accepted.

The second part of the study focused on the adhesion between posts and dentine. The surface of the tooth was prepared with two drying protocols. To minimize variability, no etchant or primer were used. Surfaces were cleaned with a water spray for 15 seconds and dried with a continuous stream of air at 350 kPa from a set distance, avoiding desiccation. A self-adhesive resin cement with a pH of 2 was used. A review of the literature indicates that low pH self-etch adhesives have been shown to have low bond strengths, especially to dentine.[9] This may provide an explanation for the relatively low bond strengths obtained in the current study.

To ensure that the tensile test is purely tensile without shearing stresses, the prepared tooth and post surfaces must be flat. The tooth surface and the acrylic blocks were carefully trimmed and levelled. A limitation of the experiment relates to the flatness of the post surface. The posts provided may not have a perfectly flat surface as this is not the main function of a post retaining a composite core. While efforts were made to remove excess cement, this may account for some variability in adhesion strength observed in the data.

The coefficients of variation for the outcomes for both SU and MU syringe tips are relatively high but similar to those found in dentine adhesion research.[13]

Suboptimal conditions during the setting or solidification process is a common concern in restorative dentistry and all luting agents, regardless of composition and characteristics, must be protected against the effects of oral fluids prior to and during the curing process to avoid detrimental effects on the setting reactions.[14] Cohesive strength of the adhesive is important because under loading, the bond fractures due to cohesive failure of the adhesive rather than failure of the adhesive-substrate bond.[14]

An improperly installed or damaged MU syringe tip often causes syringe malfunction, resulting in a short burst of water spray from the tip when the air button is pressed. As a means of preventive maintenance, it is recommended that the two internal O-rings and the small O-ring in the syringe tip retainer assembly be replaced at least once a year.[15] As we aimed to mimic an everyday clinical situation in this research, the O-rings were not replaced by new ones for each specimen prepared.

A factor pertinent to multi-use instruments employed in dentistry is the corrosion of metallic instruments. Corrosion is defined as the destruction, or deterioration, of a material because of reaction with its environment.[16] Exposure to agents such as air and water can result in partial or complete dissolution, deterioration, or weakening of any solid substance.[17] Given that MU air/water syringes are metallic and have a high exposure to corrosive conditions, it is likely that there can be either corrosive breakdown (and subsequent pitting of the metallic surface) or contaminant build-up within the lumen of the syringe, or both. The lumina of old MU tips were examined via scanning electron microscopy (SEM) and electron dispersive spectroscopy (EDS) and were found to have more spherical protrusions and indentations compared to brand-new unused MU tips.[12] The irregularly-shaped spray patterns obtained from MU tips in our study correlates to the findings that old MU tips have been found to have irregular lumen surfaces that are a result of corrosion over time.

In conclusion, the use of SU tips allowed a more consistent and moisture-free spray pattern than the MU tips, whilst the type of tip used to dry the tooth surfaces did not affect the adhesion strength. Within the limitations of this study, SU tips are as effective as MU tips in drying dentine surfaces.

CLINICAL SIGNIFICANCE

Single-use tips are recommended as part of modern infection control procedures. Their use also allows a more consistent and moisture-free spray pattern for drying a dentinal surface. Residual traces of moisture exist when drying with multiple-use tips. This could require the operator to prolong the drying time on larger tooth surfaces, increasing the risk of desiccation and collapse of dentinal tubules before bonding. Further research is required.

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MANUFACTURER'S DETAILS

- Single-use air/water syringe tips (SU): Classic by Riskontrol, Pierre Rolland, Merignac, France
- Multiple-use air/water syringe tips (MU): Autoclavable Standard, A-dec, Newberg, OR, USA
- D3000: Nikon, Tokyo, Japan
- FIJI is Just Image J: US National Institutes of Health, Bethesda, MD, USA
- SPSS for Windows 2007: IMB, Armonk, NY, USA
- Castapress: Vertex-Dental B.V., Zeist, The Netherlands
- Grinding/polishing machine, Stuers TegraPol-21 & TegraForce5: Struers A/S, Ballerup, Denmark
- Rebilda Post 20: Voco, Cuxhaven, Germany
- Silane coupling agent: 3M ESPE, St Paul, MN, USA
- RelyX™ Unicem 2 Self-Adhesive Resin Cement: 3M ESPE, St Paul, MN, USA
- Lloyd Microtensile tester, Model T5002: J. J. Lloyd Instruments Ltd, Southampton, UK
- Permbond: Permabond Engineering Adhesives Ltd, Winchester, UK
- LabChart 7: ADInstruments Ltd, Dunedin, New Zealand
- Sputter Coater Emitech K575X: EM Technologies Ltd, Kent, England
- Field emission microscope JSM-6700F: JEOL Ltd, Tokyo, Japan

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