The Effect of Different Drying Methods for Single Step Adhesive Systems on Microleakage of Tooth Colored Restorations

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Abstract

The purpose of this in vitro study was to evaluate microleakage of tooth colored restoratives and accompanying single step adhesive systems using two drying methods (syringe air versus "sponge" applicator blotting).

Eighty teeth were randomly assigned to four material groups. Class V cavity preparations, located half in enamel and half in cementum at the cementoenamel junction (CEJ), with a 1.0 mm enamel bevel were completed. The adhesive/composite groups included: (1) Single-Bond/Z-100 Composite, (2) Prime & Bond 2.1/Dyract AP Compomer, (3) OptiBond Solo Plus/Prodigy Composite, and (4) Scotchbond MultiPurpose/Z-100 Composite. Each material group (n=20), consisted of preparation Subgroups dried with syringe air (A), (n=10) and sponge applicators (B), (n=10). The preparations were conditioned, rinsed, and gently dried followed by placement of the primer/adhesive and restorative materials. All teeth were thermocycled, stained with methylene blue dye, invested in clear acrylic resin, and sectioned longitudinally through the center of the restoration. Readings were taken at the occlusal and gingival surface positions of each restoration section. A ratio (%) of wall length to amount of leakage along each wall was established.

One-way Analysis of Variance (ANOVA) testing revealed: (1) no significant (p<0.05) differences existed between materials at the occlusal surface position in Subgroups A and B (syringe vs. applicator drying), (2) significantly (P<0.05) greater leakage of OptiBond Solo Plus compared to Single-Bond, Prime & Bond 2.1, and Scotchbond MultiPurpose at the gingival surface position in Subgroups A and B, (3) significantly (p<0.05) greater leakage of OptiBond Solo Plus compared to Single-Bond and Scotchbond MultiPurpose, combining the occlusal/gingival surface position scores, (4) no significant difference existed between Single-Bond Subgroups A/B, OptiBond Solo Plus Subgroups A/B, Scotchbond MultiPurpose Subgroups A/B, (5) significantly (p<0.05) greater leakage of Prime & Bond 2.1 Subgroup B compared to Subgroup A, (6) no
significant (p<0.05) difference existed between material groups, except OptiBond Solo Plus (occlusal vs. gingival surface position), (7) no significant (p<0.05) difference between Single-Bond, OptiBond Solo Plus, and Scotchbond MultiPurpose comparing both Subgroups, same materials, and surface positions, and (8) no significant (p<0.05) difference existed between Prime & Bond 2.1, comparing both Subgroups, occlusal surface position.

In the present study, significantly greater leakage was revealed with OptiBond Solo Plus compared to the other material groups, especially at the gingival surface positions. Significantly greater leakage was also recorded with OptiBond Solo Plus and Prime & Bond 2.1 sponge applicator drying as compared to the same materials, syringe air drying.

**Keywords:** Microleakage, composite resin, adhesives, compomer

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**Introduction**

Restoration of Class V abrasion, erosion, caries, or abfraction dental cervical lesions is performed with several types of tooth colored restorative materials. Included among these esthetic materials: composite resin, composites (polyacid modified glass ionomer cements), and hybrid glass ionomer cements or resin-ionomers Many of the restoratives are “lesion exclusive” and are manufactured for treatment of specific dental cavity lesions and the etiologies involved. Hybrid glass ionomers and composites are designed for Class V, root lesions (exposed dentin). These materials theoretically release fluoride for patients with high caries incidence. Composite resins restore Class I-VI, anterior/posterior cavity lesions. Anterior, microfilled composite resins are for restoration of Class III, IV, and V, “esthetic” lesions yielding excellent cosmetic (polishable) clinical results. Flowable or injectable composites are advocated for Class I, II, III, and V lesions and/or repair of composite/porcelain margins. These materials are also used as liners beneath Class II direct placement posterior composites. Flowable materials have a lower filler/higher resin content for increased viscosity and possess low modulus of elasticity that compensates for flexure or abfractional forces (tensile and compressive).

Microleakage is the clinically undetectable movement of bacterial fluids, molecules, and ions at the restoration/tooth interface. Factors causing formation of marginal gaps and subsequently leakage between the cavity wall and restoration include: temperature variables, contractional forces, moisture absorption, polymerization shrinkage, inadequate moisture control, operator inability, and masticatory forces. Hygroscopic absorption (water uptake by the restorative material) and incremental insertion of restorative materials can compensate somewhat for these inadequacies; however, microgaps created at the margin cause bacterial ingress precipitating staining, secondary caries, defective restorations, sensitivity, and possible pulpal pathosis.

Tooth colored restoratives, capable of mechanical and/or chemical bonding, are still subject to margin leakage. In determining resistance to formation of secondary caries, the ability of restorative materials to bond with tooth structure is a primary factor. Microleakage at the tooth/restoration margin has been significantly reduced since the introduction of acid etching by Buonocore in 1955. Dentin bonding, developed over two decades, has been successful but not as predictable as enamel etching due to organic and inorganic components of dentin.

Presence of moisture on the tooth surface serves an essential function for successful bonding to dentin. Desiccation from excessive compressed air causes deformation of the dentinal complex and unsuccessful
bonding. Current adhesive systems advocate a hydrophilic (affinity for water), slightly moist dentin surface for optimal cohesion.

In the present study two methods of dentin surface drying were performed before bonding with single step adhesive materials. Measurement of leakage at the tooth/restoration margin with dye penetration was the evaluation criterion employed.

Dental material manufacturers have advocated drying of prepared tooth surfaces following acid conditioning with (1) synthetic applicators or brushes and/or (2) compressed air from an air-water syringe. Often, these instructions are not completely specific for the type/length of surface drying and can be detrimental for successful results.

This study hoped to reveal any significant differences between material groups using two drying techniques. The author hypothesized that greater leakage would be exhibited using syringe air opposed to surface blotting with synthetic sponge applicators. It was also expected that significantly less leakage would be revealed with single step versus multiple step adhesive systems (more steps, more variables for problems to occur). An extensive review of the scientific literature located no studies involving this specific topic.

Methods and Materials
Eighty previously extracted, non carious, human molars were used in the present study. The teeth were cleaned of calculus, soft tissue, and other debris before being stored in a 1% Chloramine solution (12% active chlorine diluted in distilled water) for infection control purposes prior to usage. The teeth were randomly assigned to four material groups (n=20), with further subdivision for two drying Subgroups (n=10). The first Subgroup (A) of teeth had preparations dried by compressed air from an air-water syringe. The second Subgroup (B) of teeth had preparations dried with Mini-sponge® applicators. The four material (adhesive/composite) groups included: (1) Single-Bond™ Z-100™, (2) Prime & Bond 2.1/ Dyract®, (3) OptiBond Solo Plus/Prodigy®, and (4) Scotchbond MultiPurpose/Z-100™. The first three groups included single step adhesive components and the last material was a multiple step etchant/primer/adhesive.

Cavity Design
In all groups, Class V, non-retentive preparations, located half in enamel/half in cementum were cut on the facial and lingual surfaces at the cementoenamel junction (CEJ). The teeth were prepared with a No. 1700 carbide bur in a high-speed handpiece cooled with an air-water syringe spray. The enamel margins were beveled (1.0 mm) with a No. 257 diamond bur. All burs were discarded after preparation of each tooth group. The approximate preparation dimensions (3.0 mm x 2.0 mm x 1.5 mm in depth) were measured with a periodontal probe to maintain uniformity. A depth gauge was used to standardize preparation depth. (Figure 1) Except for additional drying methods, manufacturer instructions were strictly adhered to for all restorative materials.

Group 1
The preparations were etched for 15 seconds with Scotchbond™ 35% phosphoric acid gel and rinsed for 5 seconds with water from an air-water syringe. The preparations were gently dried using both techniques (syringe air/sponge applicator). Single-Bond primer/adhesive was applied to the surface in two consecutive coats, dried for 2-5 seconds, and polymerized for 10 seconds. Single-Bond contains 2-hydroxyethylmethacrylate (HEMA), photoinitiators, and acetone as a solvent carrier.

Z-100 composite was inserted in the preparations in two increments, each increment (approximately .75 mm) polymerized for 40 seconds. Z-100 is a hybrid, small particle, universal composite used to restore all Classes of cavity preparations.
**Group 2**
Caulk 34% Tooth Conditioner was applied to the enamel and dentin for 15 seconds and rinsed for 15 seconds with water from an air-water syringe. The preparations were gently dried using both drying techniques. Prime & Bond 2.1 single step primer/adhesive was applied to the surface for 20 seconds, air dried for 5 seconds, and polymerized for 10 seconds. Prime & Bond 2.1 contains Di and Trimethacrylate resins, dientaerythritol penta acrylate monophosphate (PENTA), nanofillers, photoinitiators, stabilizers, cetylamine hydrofluoride, and acetone. Dyragt AP composite containing UDMA resin, TCB resin, hydrofluoride, and acetone. Dyract AP is a polyacid modified composite containing UDMA resin, TCB resin, radiopaque strontium fluorosilicate glass, initiators, and stabilizers. This material is indicated for Class V adult lesions and Class I and II deciduous cavity preparations.

**Group 3**
Kerr 37.5% phosphoric acid etchant was applied to the preparations for 15 seconds and rinsed for 5 seconds with water from an air-water syringe. The preparations were gently dried using both drying techniques. OptiBond Solo Plus one step primer/adhesive was placed on the surfaces for 15 seconds, gently air dried, and polymerized for 10 seconds. OptiBond Solo Plus contains ethanol, HEMA, bisphenyl-A glycidyl methacrylate (Bis-GMA), barium glass, fumed silicon dioxide, sodium hexafluorosilicate, and preservatives. Prodigy composite was inserted in the preparations in two increments, each increment polymerized for 40 seconds. Prodigy is a hybrid, universal composite for all Classes of cavity lesions.

**Group 4**
Scotchbond 35% phosphoric acid etchant was applied to the preparations for 15 seconds and rinsed for 5 seconds. The preparations were gently dried using both drying methods. Scotchbond MultiPurpose Primer was placed and air-dried for 5 seconds followed by application of Scotchbond MultiPurpose Adhesive and polymerized for 10 seconds. Scotchbond MultiPurpose Primer/Adhesive contains HEMA, BIS-GMA, photoinitiators, and acetone. Z-100 composite was inserted and polymerized as in Group 1.

The adhesives and tooth colored materials in all groups were polymerized with a Scheinj visible curing light. The light had been monitored with a radiometer and, thus, provided adequate intensity (600mW/cm^2). All material specimens required minimal finishing with 30 fluted composite finishing burs. The restorations were polished with Sof-Lex^\textregistered aluminum oxide disks of decreasing abrasiveness (coarse-ultrafine).

**Assessment of Microleakage**
The teeth were thermocycled for 200 cycles (approximately 3.5 hours) alternating in separate water baths of (5° ± 2°) C and (58° ± 2°) C, with a dwell time of 60 seconds and transfer time of 10 seconds. Sticky wax was applied to the root apices with nail polish liberally coating the entire tooth surface to within 1.0 mm of the restoration. The teeth were immersed in a 5% methylene blue dye solution for 4 hours at room temperature. Following removal from the dye, the teeth were cleaned and rinsed thoroughly with tap water. The teeth were invested in Castin Craft® clear acrylic resin blocks and labeled. An Isomet™ precision, slow-speed diamond saw, cooled with water, sectioned each tooth longitudinally through the center of the restoration from the facial to the lingual surface. Two sections were obtained, each side of the cut yielding measurements. Two readings, at both enamel (occlusal) surface position and cementum (gingival) surface position, were recorded using 20x magnification with a Meijin binocular microscope. With an eyepiece reticle calibrated in millimeters, the length of the wall and the extent (length) of dye penetration at the tooth/
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restoration interface were measured. The degree of dye penetration (microleakage) was established as the ratio (%) of the length of dye penetration to the length of the wall. The degree of dye penetration was scored separately for each wall. An average was obtained, yielding one measurement at both surface positions for each specimen block.

Statistical Analysis
The data collected was evaluated using One-Way analysis of variance (ANOVA) testing. Fisher’s Protected Least Significant Difference post-hoc (PLSD), Scheffe F-test, and Dunnett t tests were performed measuring significance between material and surface position groups. P>0.05 (95%) was used as the significance level.

Results
Results of dye penetration/wall length are summarized in Tables 1-3. Significant differences were extrapolated from the outcome data. In Table 1, the frequency/mean percentage leakage is revealed for group differences (material and surface positions). The standard deviation and error were also recorded for each material group. ANOVA testing revealed in Subgroup A (syringe air drying): (1) there were no significant (p<0.05) differences between materials at the occlusal surface position, (2) the OptiBond Solo Plus (OBS) Group revealed significantly (p<0.05) greater leakage than the Single-Bond (SB), Prime & Bond 2.1 (PB), and Scotchbond MultiPurpose (SMP) Groups, and (3) combining the occlusal and gingival surface position scores, significantly (p<0.05) greater leakage existed in the OBS Group compared to the SB and SMP Groups. In Subgroup B (sponge applicator drying): (1) no significant (p<0.05) differences existed between materials at the occlusal surface position, (2) at the gingival surface position, significantly (p<0.05) greater leakage existed with the OBS compared to the SB and SMP Groups, and (3) combining occlusal and gingival surface position scores, the OBS Group revealed significantly (p<0.05) greater leakage than in the SB and SMP Groups.

In Table 2, comparing Subgroups A and B and combined occlusal/gingival position scores, same materials: (1) no significant (p<0.05) differences existed between SB, Subgroups A and B, (2) significantly (p<0.02) greater leakage existed in PB, Subgroup B compared to PB, Subgroup A, (3) no significant (p<0.05) differences existed between OBS, Subgroups A/B, and (4) no significant (p<0.05) differences between SMP, Subgroups A/B.
### Table 2. Comparing Subgroups A (Syringe Air) and B (Sponge Applicators), Same Materials, Combined Occlusal/Gingival Surface Positions (n=10)

<table>
<thead>
<tr>
<th>Material</th>
<th>Mean (O+G)</th>
<th>Std. Dev.</th>
<th>Std. Error</th>
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<tr>
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<td>.18</td>
<td>.05</td>
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<tr>
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<td>.10</td>
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<td>.06</td>
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<td>SMP&lt;sup&gt;B&lt;/sup&gt;</td>
<td>.21</td>
<td>.32</td>
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O-Occusal Surface Position
G-Gingival Surface Position

A-Subgroup A (Dried by Syringe Air)
B-Subgroup B (Dried by Sponge Applicators)

SB- Single-Bond
PB- Prime & Bond 2.1
OBS- OptiBond Solo Plus
SMP- Scotchbond Multipurpose

Std. Dev.-Standard Deviation
Std. Error-Standard Error

### Table 3. Comparison of Same Materials, Occlusal vs. Gingival Surface Positions (n=10)

#### Subgroup A (Syringe Air)

<table>
<thead>
<tr>
<th>Material</th>
<th>Surface Position</th>
<th>% Mean Leakage</th>
<th>Std. Dev.</th>
<th>Std. Error</th>
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<td>.11</td>
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<td>.11</td>
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<tr>
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<td>Gingival</td>
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<td>.16</td>
</tr>
<tr>
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<td></td>
<td>Gingival</td>
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#### Subgroup B (Sponge Applicator)

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<th>Material</th>
<th>Surface Position</th>
<th>% Mean Leakage</th>
<th>Std. Dev.</th>
<th>Std. Error</th>
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<td>Gingival</td>
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<tr>
<td></td>
<td>Gingival</td>
<td>.28</td>
<td>.42</td>
<td>.13</td>
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</table>

SB- Single-Bond
PB- Prime & Bond 2.1
OBS- OptiBond Solo Plus
SMP- Scotchbond Multipurpose

Std. Dev.-Standard Deviation
Std. Error-Standard Error

%-Percent Leakage
According to Table 3, comparison of materials, occlusal versus gingival surface position, Subgroup A: (1) no significant (p<0.05) differences existed comparing SB, PB, OBS, and SMP material Groups and in Subgroup B: (1) no significant (p<0.05) differences existed for the SB, PB, and SMP material Groups and (2) significantly (p<0.002) greater leakage existed at the gingival surface position with the OBS Group.

Discussion
Stresses placed upon teeth from temperature changes, polymerization shrinkage of materials, and masticatory loading create gaps at the tooth/restoration margin. Microleakage of contaminants infiltrate through this gap causing possible disease sequela and restoration replacement.

An important consideration is selection of a dentin bonding system and its resistance to margin leakage. Effective bonding requires removal of a smear layer with acids followed by "satisfactory" rinsing and drying of the preparation for a clean, adequately moist surface before application of a primer/adhesive. The smear layer is a barrier of tooth particle/material debris following cavity preparation with a dental bur. As previously stated, the correct type/degree of surface drying promotes optimal cohesive hybridization at the tooth/restoration margin. The clinician should avoid extreme conditions of surface wetness or dehydration. The tooth surface (dentin) should not be dehydrated or desiccated following conditioning, however, excessive surface moisture results in voids.

The “hybrid layer” is created by micromechanical attachment between primer/resins and intertubular, hypermineralized dentinal collagen. Successful resin impregnation of surface dentin is necessary for success of any dentin bonding system and reduction of microleakage. Bonding systems have utilized separate acid etchants, primers, and adhesive resins. Current generation systems incorporate these ingredients in one or two step (container) components. Etchants in many systems still contain 32-37% phosphoric acid gel. Primer/adhesives consist of hydrophilic monomers (HEMA, Bis-GMA, PENTA, etc.) that sufficiently “wet” the dentin surface. Primer/adhesives are hydrophilic for the organic/inorganic portion of tooth structure and hydrophobic for bonding with the overlying restorative material. Specifically, as dentin is etched, surface demineralization occurs leaving organic collagen. Surface moisture must be maintained prior to primer/adhesive attachment preventing collapse of collagen fibers. Saturation of spaces around these fibers with primer/adhesives is essential to superior bonding. Current systems simplify the process but are technique sensitive and with few exceptions; manufacturer instructions must be strictly followed. Saliva contamination (sometimes incorrectly referred to as “moisture contamination”) controlled with a dental rubber dam must be adhered to for successful bonding. Often misinterpreted, moisture (water) is not a deterrent for successful bonding of dental materials to tooth structure. Saliva contamination (containing bacterial components) is the primary cause of pulpal symptoms and possible restoration failure.

In vitro microleakage testing of dental materials is a commonly accepted evaluation technique of margin integrity. The practice of thermocycling specimens in hot/cold baths simulates thermal stresses in the oral environment. Although every effort is made to model an in vivo setting, thermocycling does not totally equate to clinical durability. The present study adhered to procedures followed in previous in vitro microleakage studies. Laboratory studies attempt to reproduce clinical situations but do not entirely reflect variables encountered with in vivo performance.

In the present study, three single step and a multiple step adhesive were used following two dentin surface drying techniques. Some manufacturers (Single-Bond and Prime & Bond 2.1) suggest blot drying of the preparation with synthetic, absorbent materials before placement of primer/adhesive components. MultiPurpose and OptiBond Solo
Plus adhesive systems did not detail specific instructions regarding “preferred” preparation drying methods, however, all manufacturers did recommend drying the preparation thoroughly (not desiccation?).

Significantly greater leakage was demonstrated with OptiBond Solo Plus than compared to the other material groups. Also, significantly greater leakage occurred with Prime & Bond 2.1, using sponge applicators (versus syringe air). Overall, (not necessarily significant) greater leakage occurred at both the occlusal and gingival surface positions, dried with sponge applicators. Greater leakage (not necessarily significant) occurred at the gingival surface positions in all material groups, possibly due to weaker dentin bonding (opposed to enamel/dentin bonds at occlusal surface positions). Single-Bond exhibited less leakage (not necessarily significant) than the other single step adhesive systems. Single-Bond and Scotchbond MultiPurpose materials revealed comparable leakage scores. Less leakage (not necessarily significant) was observed with Subgroup A (syringe air drying), indicating more efficacious surface drying (optimal dentin moisture).

**Conclusion**

Contrary to the author’s initial hypothesis and perhaps, company information, it appears that gentle air drying with an air-water syringe leaves the dentin surface more conducive (optimum moisture content) for adhesive bonding of tooth colored restorations. One possible explanation follows that drying with syringe air allows miniscule amounts of water (expressed with the air spray) to be deposited on the dentin, rehydrating the surface collagen.

Satisfactory degrees of dentin surface moisture, primarily relies on adequate drying of tooth structure, has not been consistently defined by restoration manufacturers. Manufacturers depend on proprietary material component information for dentin bonding, allowing limited calibration for placement of esthetic filling materials. The present results are in vitro data and definite conclusions should not be drawn until in vivo studies are completed. More research is needed in the future, especially concerning dentin surface moisture and adhesive bonding.
References
27. Cardoso PE, Placido E, Franci CE, et al. Microleakage of Class V resin-based composite restora-

Manufacturers

a (Fisher Chemical, Fair Lawn, NJ)
b c f g k (3M-ESPE, St. Paul, MN)
d h (Dentsply-Caulk, Milford, DE)
e i (Kerr Co, Orange CA)
j (Sullivan-Schein Co., Melville, NY)
l (ETI, Fields Landing, CA)
m (Buehler Ltd, Evanston, IL)
n (Meiji-Labax Co, Tokyo, Japan)

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