One-Year Evaluation of a Simplified Ethanol-Wet Bonding Technique: A Randomized Clinical Trial

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The objective of this randomized clinical trial was to evaluate the clinical performance of adhesive restorations using a three-step etch-and-rinse adhesive (TSER), a one-step self-etching adhesive (OSSE), and a simplified ethanol-wet bonding technique (EWBT) prior to the application of a composite resin in non-carious cervical lesions. Ninety-three restorations (31 for each group) were placed in 17 patients by a single operator. No cavity preparation was performed. After 6 and 12 months, the restorations were assessed by two previously trained examiners using modified Ryge criteria for retention (kappa=1.00) and marginal adaptation/staining (kappa=0.81), and the results were analyzed by Fisher’s exact and Kruskal-Wallis tests, respectively. No significant differences were observed among groups at the 6- and 12-month time points for any of the assessed criteria (p≥0.05). The intra-group analysis performed by Cochran’s test (for retention) and Wilcoxon test (for marginal adaptation/staining) revealed significant differences between the baseline/12-month time intervals in marginal adaptation in OSSE (p=0.0180) and in marginal staining in TSER (p=0.0117). The survival analysis for retention criteria performed using a log-rank test did not show significant differences (p>0.05). The restorations placed using the simplified EWBT performed equally well as the other adhesive strategies employed.

Key Words: randomized controlled trial, dentin-bonding agents, dentin.
was conducted at the Federal University of Pará, Brazil and was approved by the Ethics Committee on Investigations Involving Human Subjects at the Federal University of Pará (UFPA). This study was designed following the main guidelines of the CONSORT Group (Consolidated Standards of Reporting Trials) and was registered at the ISRCTN (International Standard Randomized Controlled Trial Number) under the number 00627732.

The retention rate (100%) of two adhesive systems at five years (23) was used as a parameter for sample size calculation. In order to detect a 20% difference among groups with 80% power and a significance level of 5%, the minimal sample size should be 31 restorations per group.

Initially, 48 volunteer patients (employees and students) of the UFPA who were interested in this research were evaluated and 17 of them, with ages ranging from 23 to 54 years old, were ultimately enrolled. They were instructed on the conditions and objective of the study and signed an informed consent form prior to participating. Eligibility criteria were: good oral hygiene, no periodontal disease and at least 3 NCCLs. Exclusion criteria were the presence of an orthodontic appliance or partially removable prosthetics, high caries risk (presence of three or more active caries lesions), heavy bruxism and patients who did not accept the conditions of the project.

Each patient received at least 3 or multiple of 3 restorations. Each adhesive system was randomly allocated to one of randomized cervical lesions until the three groups were present in the same subject and in equal amounts.

A total of 93 NCCLs (31 for each group) were restored by one experienced operator using three types of adhesive strategies: Adper Scotchbond Multi-Purpose/ SBMP (three-step etch-and-rinse adhesive - TSER; 3M ESPE, St. Paul, MN, USA); Adper Easy One/EO (one-step self-etching adhesive - OSSE; 3M ESPE, Seefeld, Germany); and simplified ethanol-wet bonding technique – EWBT (dentin saturation with ethanol solutions + hydrophobic experimental primer + step 3 of SBMP – 3M ESPE). The hydrophobic primer used in the EWBT was prepared by diluting 2 mL step 3 of SBMP (3M ESPE) in absolute ethanol corresponding to 10% mass.

Table 1. Adhesive compositions and instructions for use

<table>
<thead>
<tr>
<th>Material</th>
<th>Adhesive technique</th>
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</table>
| Adper Scotchbond Multi-Purpose + Filtek™ Z350 XT (3M ESPE) | 1- etching of enamel and dentin for 15 s, with 35% phosphoric acid gel and rinsing by air-water spraying for 30 s  
2- dentin blotting with absorbent paper to keep the surface visibly moist  
3- application of primer to enamel and dentin. Dry gently for 5 s  
4- application of adhesive and light curing for 10 s  
5- restoration with ≅1-mm layers of Filtek™ Z350XT |
| Adper Easy One + Filtek™ Z350 XT (3M ESPE) | 1- dentin washing and blotting with absorbent paper to keep the surface visibly moist  
2- application of Easy One adhesive for 20 s, air-drying for 5 s and light curing for 10 s  
3- restoration with ≅1-mm layers of Filtek™ Z350XT |
| EWBT: experimental “hydrophobic primer” + step 3 of Adper Scotchbond Multi-Purpose + Filtek™ Z350 XT (3M ESPE) | 1- etching of enamel and dentin for 15 s, with 35% phosphoric acid gel and rinsing by air-water spraying for 30 s  
2- dentin blotting with absorbent paper to keep the surface visibly moist  
3- application of 50% ethanol for 10 s and allowed to sit for 10 s  
4- application of 100% ethanol for 10 s and allowed to sit for 10 s  
5- application of “hydrophobic primer” to enamel and dentin. Dry gently for 5 s  
6- application of the SBMP adhesive (step 3) and light curing for 10 s  
7- restoration with ≅1-mm layers of Filtek™ Z350XT |

Table 2. Restorations distribution

<table>
<thead>
<tr>
<th>Arch</th>
<th>Adhesive system group</th>
<th>Right quadrant (45)</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Molar (7)</td>
<td>Premolar (27)</td>
</tr>
<tr>
<td>Maxilla (52)</td>
<td>TSER (18)</td>
<td>0</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>OSSE (17)</td>
<td>3</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>EWBT (17)</td>
<td>1</td>
<td>7</td>
</tr>
<tr>
<td>Mandible (41)</td>
<td>TSER (13)</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>OSSE (14)</td>
<td>0</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>EWBT (14)</td>
<td>2</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>Maxilla (52)</td>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>Mandible (41)</td>
<td>1</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>2</td>
</tr>
</tbody>
</table>

All restorations were placed under relatively dry conditions using gingival retraction cord, cotton rolls and saliva ejector. Enamel margins were not beveled and no mechanical retention was performed. The surfaces were slightly roughened with a #3118 diamond bur (K.G. Sorensen, São Paulo, SP, Brazil).

After adhesive systems application, the cavities were restored by applying at least two layers of Filtek Z350 XT (3M ESPE), light-cured for 40 s using a light-emitting diode (LED) unit (D-2000; DMC, Joinville, SC, Brazil). Restorations were polished with #3195 fine diamond burs (K.G. Sorensen) and candle flame rubber points (Dentsply/Caulk, Milford, DE, USA). Table 1 lists the materials and techniques used for each group, and the distribution of the restorations with respect to tooth type, adhesive, quadrant and arch are shown in Table 2.

Two calibrated examiners evaluated all restorations using the modified US Public Health Service guidelines (USPHS). In cases where the two examiners disagreed on a rating, both re-examined the restoration and arrived to a joint final decision. The examiners were blinded regarding the type of adhesive used in each restoration. The primary outcome was retention (kappa=1.00), with marginal adaptation/staining (kappa=0.81) as secondary outcome, both analyzed at 6 and 12 months.

The obtained data were recorded and the differences among adhesive systems at each evaluation period were analyzed using Fisher’s exact test for retention and the Kruskal-Wallis test for marginal adaptation/staining at a 5% significance level. An intra-group comparison among baseline, 6- and 12-month evaluation for each adhesive system was performed using Cochran’s test (for retention) and the Wilcoxon test (for marginal adaptation/staining) at a 5% significance level. At baseline, all restorations were scored A for all evaluation criteria. The survival analysis for retention criteria was performed using a log-rank test at a 5% significance level.

**Results**

The overall analysis revealed no significant differences (p>0.05) among groups for up to 12 months in terms of marginal adaptation/staining and retention (Table 3). The survival analysis did not detect significant differences among the groups regarding retention (p>0.05). Figure 1 shows the survival curves for adhesive systems and p values.

From the intra-group analysis that assessed the behavior of each adhesive system over time, significant differences were detected between baseline and 12 months in marginal adaptation in OSSE and marginal staining in TSER groups (Table 4).

**Table 3. Clinical evaluation for each criterion at 6 and 12 months**

<table>
<thead>
<tr>
<th>Criteria</th>
<th>Adhesive system group</th>
<th>6 months</th>
<th>12 months</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>A</td>
<td>B</td>
<td>C</td>
</tr>
<tr>
<td>Retention</td>
<td>TSER</td>
<td>31/31</td>
<td>30/31</td>
</tr>
<tr>
<td></td>
<td>OSSE</td>
<td>30/31</td>
<td>30/31</td>
</tr>
<tr>
<td></td>
<td>EWBT</td>
<td>29/31</td>
<td>29/31</td>
</tr>
</tbody>
</table>

**Table 4. Intra-group analysis for each adhesive systems over 12 months**

<table>
<thead>
<tr>
<th>Adhesive system group</th>
<th>Retention</th>
<th>Marginal adaptation</th>
<th>Marginal staining</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>B x 6 M x 12 M</td>
<td>B x 6 M</td>
<td>B x 12 M</td>
</tr>
<tr>
<td>TSER</td>
<td>p = 1.000a</td>
<td>p = 0.317b</td>
<td>p = 0.179b</td>
</tr>
<tr>
<td>OSSE</td>
<td>p = 1.000a</td>
<td>p = 0.1088b</td>
<td>p = 0.0180b</td>
</tr>
<tr>
<td>EWBT</td>
<td>p = 1.000a</td>
<td>p = 0.317b</td>
<td>p = 0.179b</td>
</tr>
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</table>

aCochran’s test p≤0.05. bWilcoxon’s test. *Significant results.
Discussion

Retention is one of the most important criteria for evaluating the longevity of restorations. Considering this fact and according to Kubo et al. (23) and Santiago et al. (24), NCCL restorations are appropriate for evaluating the clinical performance of direct adhesive restorations.

Hydrophilicity of commercially available adhesive systems is a necessary characteristic, especially in clinical application, but it brings along some unfavorable scenario afterwards. This characteristic allows for water sorption and adhesive interface hydrolysis, which causes a significant decrease in adhesion over time (1,3,18). From a laboratory point of view, the EWBT seems to overcome part of this problem (6,8,20).

Several laboratory studies (4,5,7,21,25) demonstrated increased adhesion when dentin is saturated with progressive concentrations of ethanol solutions before the application of bonding agents. The spaces among the collagen fibrils increase, which makes hydrophobic monomer infiltration easier, reducing hydrophilicity of the interface (6,18,20).

EWBT requires the application of increasing concentrations of ethanol solutions on enamel and dentin in laboratory. In the most commonly used technique, the dentin is first saturated with 50% ethanol, followed by 70, 80, 95 and 100% solutions (4,8,9). Gradual ethanol saturation seems to be a necessary step to prevent collagen from collapsing and to replace the total amount of water within the demineralized dentin (3,6,8). However, this laboratory procedure takes a long time, which turns the technique clinically unsuitable (8). Therefore, it was necessary to adapt this technique to clinical conditions.

Different dentin saturation times are described in the literature for EWBT, varying from 20 s to 3.5 min, and the concentration of the ethanol solutions can be gradually increased or applied in a single step (5-7,18). Tay et al. (5) did not observe differences in bond strength between EWBT using one application of 100% ethanol and conventional etch-and-rinse strategy. On the other hand, Sadek et al. (9) showed poor results when a single application of 100% ethanol was used. Due to the lack of clinical studies in the literature using EWBT and considering that the main goal of this strategy is to replace the water within the demineralized dentin, the present study adjusted this step using two concentrations (50 and 100%) of ethanol solutions, which were applied for 20 s (18), reaching satisfactory laboratory results. Therefore, from now on, the strategy used in this study will be named simplified EWBT, which takes approximately 2 min, compared with approximately 1 min for the etch-and-rinse technique and 35 s for the SE protocol.

Another feature that contributes to increase the in vitro interface durability is the reduction of hybrid layer permeability and passage of water through it, achieved by EWBT. This phenomenon reduces the possibility of hydrolytic degradation and promotes long-lasting adhesion (3,21). So far, based on the clinical results of this research, it is not possible to confirm improved durability of restorations by simplified EWBT.

Regarding retention, although a decreasing retention rate (87.10%) was observed up to 12 months for the restorations in the OSSE group versus 100% retention for the TSER group and 93.55% for the simplified EWBT group, no significant differences could be detected among them (Fig. 1) (Table 3).

One alleged advantage of simplified SE adhesive systems is the associated reduction in the number of clinical steps and low technique sensitivity. In recent years, there has been a trend towards developing SE adhesives that required fewer steps and have decreased pH, taking to increased aggressiveness (11,14,16). However, systems with such combination of features had high hydrophilicity and short shelf lives (12). Hence, the reduced aggressiveness became preferable again. Adper Easy One, used in the present study, has pH=2.3 and is classified according to acidity as a ‘mild’ OSSE system (14). Less acidic interfaces are less hydrophilic, but ‘mild’ SE systems exhibit poor demineralization ability, which can be considered a disadvantage, especially when enamel and sclerotic dentin conditioning are considered (2,12,14). Peumans et al. (2) and Dalkilic and Omurlu (13) observed poor clinical performance for the tested OSSE systems.

Enamel acid conditioning prior to the application of mild SE adhesives is mentioned as a method for overcoming these unfavorable characteristics in some studies (12,13). However, this procedure would require an additional step and the modification of the SE approach. In the present investigation, this extra step was not performed, i.e., the adhesion was completely SE-dependent.

The intra-group analysis (Table 4) revealed that the adhesive technique of simplified EWBT group yielded satisfactory results for each evaluated criterion up to one year compared with baseline. The simplified EWBT facilitates adequate hydrophobic monomer infiltration without phase separation (3,6,22). This process takes place as the demineralized dentin is saturated by ethanol, which is a compatible solvent for the monomers. Once a fully interpenetrated hydrophobic layer is formed, the demineralized collagen matrix is better protected against the deleterious actions of water and MMPs (3,6,18,20,21). Despite the short evaluation period, the obtained results encourage testing of this new adhesive technique.

On the other hand, the OSSE group exhibited the worst marginal adaptation when baseline is compared to the
12-month period. Significant marginal degradation was observed in a short period of time (one year). Although ‘mild’ SE adhesives are less hydrophilic compared with the ‘strong’ SE adhesives (16), the absence of a hydrophobic layer, which is present in the other systems, could be partially responsible for the obtained results. The use of a hydrophobic coating in combination with SE systems has been suggested to reduce hybrid layer permeability (3,10,25). Another point to be stressed is the low conditioning capacity of the ‘mild’ SE adhesives when enamel and sclerotic dentin are the substrates. As mentioned before, the approach chosen for this study was completely SE-dependent, and separate acid etching was not performed, which may have influenced the marginal adaptation results.

Another evaluated criterion was marginal staining, which can result either from superficial imperfections (score B), which may be removed by finishing and polishing instruments, or from a deeper flaw that requires partial or total replacement of the restoration (10). After 1 year, the TSER group presented marginal staining (score B) in 25.81% of the restorations (8 out of 31), which was statistically significant compared to baseline. However, clinical inspection revealed that this staining represented only superficial imperfections.

Some of the features that can be related to marginal staining are overhangs, poor marginal adaptation and caries (1). In this study, however, none of these features could be strictly related to the observed discoloration. As can be seen in Table 4, only 2 out of 31 TSER group restorations were scored B for marginal adaptation. It is likely the thicker interface could be a reason for the high incidence of marginal staining, as the air-thinning step was not applied on the adhesive coat.

As mentioned before, a highly hydrophilic hybrid layer can facilitate interface degradation and subsequent loss of restoration. The results of this study showed that the simplified EWBT behaved similarly to the OSSE and three-step etch-and-rinse strategies, the last one being considered as the gold standard and a reference for clinical studies (3,4). Additionally, the simplified EWBT showed satisfactory individual performance for all evaluated criteria along time, which did not occur when other strategies were analyzed separated for each criteria at the evaluation periods. Therefore, subsequent evaluations should be performed to assess its potential in clinical use.

Acknowledgements
This study was supported by grant from Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq). The authors would like to thank the volunteer’s participation.

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Received December 17, 2012
Accepted May 17, 2013