Effect of Scrubbing Technique with Mild Self Etching Adhesives on Etching Pattern Under Scanning Electron Microscope - In Vitro Study

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Abstract

Background
To evaluate the effect of a scrubbing technique with one step self etching adhesives on etching pattern.

Materials and methods
The occlusal enamel of 40 human molars was removed in order to expose flat human dentin surface. Both the mild self etching adhesives, single bond universal and Tetric bond universal were applied in two modes, with and without scrubbing technique. Then light curing (600mW/cm²) was done for 20 s. The etching pattern was examined under scanning electron microscope.

Results
Morphological evaluation of treated dentin surfaces demonstrated that single bond universal with scrubbing showed highest etching ability, followed by Tetric bond universal with scrubbing> single bond universal without scrubbing>Tetric bond universal without scrubbing.

Conclusion
Mild self etch adhesives when applied with scrubbing technique efficiently removed smear layer with clear opening of dentinal tubules which could improve resin monomer infiltration into dentin.

Keywords
Self Etching Adhesives; Single Bond Universal; Tetric Bond Universal; Scrubbing Technique; Smear Layer; Scanning Electron Microscope

Introduction
Self etching adhesives were developed to reduce manipulation time, to make more comprehensible bonding procedure and curtail technique sensitivity [1, 2]. They provide effective bonding to enamel, dentin, as well as sclerotic dentin [3]. This system consists of acidic resin monomers, solvent, and water into one mixture and allows bonding procedure in single application [1]. The etching and rinsing steps are absent in self etch approach making them more advantageous.

In an attempt to increase the effectiveness of bonding of these materials, Miyazaki et al suggested that active adhesive application may be helpful to eliminate smear layer in order to attain micromechanical and chemical interaction with underlying dentin [4]. Prior to polymerization, the residual water and solvent from the adhesive systems should be completely removed. Because its entanglement within the hybrid layer may be detrimental to the quality of polymer within hybrid layer. So, methods such as increased application times, delayed polymerization, and scrubbing of adhesives can be advocated to improve the strength of polymer formed within the collagen fibrils and allow high bond strength values [5].

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The adhesive application using a scrubbing action augments the interaction of acid monomers with dentin substrate [2], also scrubbing action promotes fresh acidic solution to contact with tooth structure and enhance the performance of simplified etch and rinse adhesives to aid in preservation of resin dentin bond degradation [6]. Few studies stated that scrubbing technique may not result in morphological alterations of adhesive/dentin interface of one step self etching adhesives. Therefore this study was undertaken to evaluate the effect of scrubbing technique with mild self etching adhesives on etching pattern and examined under scanning electron microscope.

Materials and Methods

Specimen Selection and Preparation

Forty caries free human molars were used. The teeth were collected after the patient’s informed consent. The teeth were disinfected in 0.1% thymol solution, stored in distilled water and used within 6 months after extraction. A flat and superficial dentin was exposed on each tooth after wet grinding the occlusal enamel on #180-grit Sic paper (Figure 1). The enamel free exposed dentin further polished on wet #600-grit silicon carbide paper, under running water for 60 seconds to create a standardized smear layer. The specimens were initially divided into two groups, Single bond universal (3M ESPE, St Paul, MN, USA) and Tetric bond universal (IVOCALR VIV ADENT, Schaan, Liechtenstein) and were again sub divided based on mode of application.

Figure 1: Flat and Superficial Dentin Was Exposed On Each Tooth after Grinding the Occlusal Enamel

Composition, application mode and batch number are described in (Table 1). A single clinician performed the procedure according to manufacturer’s instructions.

Table 1: Materials Used in the Study

<table>
<thead>
<tr>
<th>Materials</th>
<th>Manufacturer</th>
<th>Composition</th>
<th>Mode of application</th>
</tr>
</thead>
<tbody>
<tr>
<td>SingleBond Universal</td>
<td>3MESPE St Paul, MN, USA</td>
<td>MDP, Dimethacrylate resins, HEMA, vitrebond Copolymer, filler, ethanol, water Initiators, Silane</td>
<td>1. Active (with scrubbing) 2. Inactive (without scrubbing)</td>
</tr>
<tr>
<td>TetricBond Universal</td>
<td>Ivoclar Vivadent</td>
<td>MDP, Methacrylated carboxylic acid, polymer, HEMA, Bis-GMA</td>
<td>1. Active (with scrubbing) 2. Inactive (without scrubbing)</td>
</tr>
</tbody>
</table>

MDP: Methacryloxydecyl DihydrogenPhosphate; HEMA-2-Hydroxyethyl Methacrylate; Bis-GMA—2, 2 Bis Hydroxyl Methacryloxy Propoxy Phenyl Propane.

Active Application (with Scrubbing)

The adhesive was applied actively on entire dentin surface. The micro brush was scrubbed on the dentin surface under manual pressure (35g) for 15 seconds (Figure 2). Then specimens were air dried for 10 seconds at a distance of 20 cm [2, 4, 7].

Figure 2: Self Etch Adhesive Was Taken On Microbrush and Applied With Scrubbing Technique
Inactive Application (without Scrubbing)

The adhesive was only spread over entire surface without scrubbing and left undisturbed for 15-20 sec.

A light curing unit set with a light intensity of 600mW/cm² was used throughout the procedure and light cured for 20sec (Figure 3).

Figure 3: Light Curing of Specimens

Specimen Preparation for SEM Analysis

In order to evaluate the etching pattern of specimen in each group under scanning electron microscope, the specimens were immersed in 50% acetone (Figure 4) for 5 min to completely remove the applied adhesives. Specimens were subsequently dehydrated in ascending grades of ethanol( 25% for 20 min, 50% for 20 min,75% for 20 min, 95% for 30 min & 100% for 60 min) (Figure 5) and dried by immersing in hexamethyldisilazaine for 10 mins (Figure 6) and left for 24hrs on filter paper placed in a covered glass vial at room temperature (Figure 7).

Figure 4: Specimens in Acetone

Figure 5: Specimens in Ethanol

Figure 6: Specimens in Hexamethyldisilazane

Figure 7: Specimens Stored In Glass Vials for 24hrs
Teeth were mounted on aluminium stubs and coated with gold by sputter process (E1010, HITACHI, TOKYO, JAPAN) for 10 min and dentin surfaces were examined using scanning electron microscope. Based on examination of specimens under a scanning electron microscope at 1000x and 2000x magnification, each field was graded. (Table 2, 3).

Table 2: Score of Smear Layer Based on SEM Images

<table>
<thead>
<tr>
<th>SCORE</th>
<th>CRITERIA</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>No smear layer and smear plug. All dentinal tubules were cleaned and open</td>
</tr>
<tr>
<td>1</td>
<td>No smear layer, but mild smear plug. Small amount of smear plug in some dentinal tubules</td>
</tr>
<tr>
<td>2</td>
<td>No smear layer but moderate smear plug. Most of dentinal tubules had smear plug.</td>
</tr>
<tr>
<td>3</td>
<td>Moderate smear layer. Only few dentinal tubules were opened</td>
</tr>
<tr>
<td>4</td>
<td>Heavy smear layer. No opening of dentinal tubules.</td>
</tr>
</tbody>
</table>

Table 3: Scores Obtained Based on SEM Images

<table>
<thead>
<tr>
<th>Single bond universal, 3M ESPE</th>
<th>Tetric bond universal IVO-CLAR VIVADENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>With scrubbing</td>
<td>Without scrubbing</td>
</tr>
<tr>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>3</td>
<td>2</td>
</tr>
<tr>
<td>4</td>
<td>0</td>
</tr>
<tr>
<td>5</td>
<td>0</td>
</tr>
<tr>
<td>6</td>
<td>0</td>
</tr>
<tr>
<td>7</td>
<td>1</td>
</tr>
<tr>
<td>8</td>
<td>1</td>
</tr>
<tr>
<td>9</td>
<td>0</td>
</tr>
<tr>
<td>10</td>
<td>0</td>
</tr>
</tbody>
</table>

Table 4: Inter Group Comparisons: Overall Comparison of Scores between Four Groups by Kruskal Wallis Test

<table>
<thead>
<tr>
<th>Scores</th>
<th>N</th>
<th>Mean</th>
<th>Median</th>
</tr>
</thead>
<tbody>
<tr>
<td>3M Single bond with scrubbing</td>
<td>5.9</td>
<td>0</td>
<td>10</td>
</tr>
<tr>
<td>3M Single bond universal without scrubbing</td>
<td>18</td>
<td>2</td>
<td>10</td>
</tr>
<tr>
<td>Ivoclar vivadent with scrubbing</td>
<td>24</td>
<td>3</td>
<td>10</td>
</tr>
<tr>
<td>Ivoclar vivadent without scrubbing</td>
<td>34</td>
<td>4</td>
<td>10</td>
</tr>
<tr>
<td>Total</td>
<td>40</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**= Statistically highly significant (p<0.01), *= Statistically significant (p<0.05)
1. All statistically significant differences in the scores of various groups compared 2. Highest difference is present between group 1 and group 4 3. Least difference present between group 2 and group 3. The order of efficacy of various groups from best to worst as follows Group 1 > Group 2 > Group 3 > Group 4

Results

SEM observation of treated dentin surfaces revealed different etching patterns among the groups. Single bond universal applied with scrubbing had highest etching ability, in which smear layer was completely removed, exposing collagen fibrils and dentinal tubule orifices, with peritubular dentin distinctly visible. In contrast single bond universal applied without scrubbing technique had smear layer partially remained with some dentinal tubules closed. For Tetric bond universal with scrubbing technique, intertubular dentin was observed to be slightly demineralised with some smear plugs still remaining in dentinal tubules, whereas without scrubbing technique, the smear layer was remaining with some dentinal tubules closed. All statistically significant differences in the scores of various groups compared. The mean and median score for group 1 is less and score is given as 0 and graded as best according to given criteria where as mean and median

Statistical Analysis

Statistical analysis was carried out by using SPSS version 23. The data obtained was analysed by Kruskal wallis test for overall intergroup comparison and intergroup comparison was made by Mann whitney U test. (Table 4, 5)
scores are high for group 4 indicative of less efficiency. (Table 4)

Highest difference is present between group 1 and group 4. Least difference is present between group 2 and group 3. The order of efficacy of various groups from best to worst Group 1>group 2>group 3>group 4. (Table 5) (Figure 8).

Figure 8: SEM micrographs show etching patterns of each adhesive with different application techniques under 2000 x and 1000x magnification. A, B -Single bond universal with scrubbing: smear layer was completely removed with exposure of collagen fibrils, disclosure of dentinal tubules and with peritubular dentin distinctly visible. C, D -Single bond universal without scrubbing: smear layer was left on dentin surface with occluded dentinal tubules. E, F-Tetric bond universal with scrubbing: intertubular dentin was partially demineralised with slight amount of smear layer remaining. G, H -Tetric bond universal without scrubbing: smear layer was remained with closed dentinal tubules.

**SEM Examination of Dentin Surface Morphology**

**Discussion**

One step mild self etch adhesives promote demineralization and simultaneously infiltrate into dentin and prevents formation of collagen which is unprotected. By ionization of radicals which are present in monomer, which is hydrophilic, aids in permeation into substrate. So, based on this concept, single bond universal and Tetric bond universal were used in this study [1, 8].

Incomplete polymerization due to incomplete removal of water from the primed dentin or adhesive layers, results in reduction in resin-dentin bond strength [6]. It is strenuous to evaporate water from this one step self etching adhesives. Several studies have stated that the use of air blowing, increased application times, delayed polymerization and active application with scrubbing of self etch adhesives has increased solvent evaporation from primer mixture after placement on dentin surface [5, 6]. Usually manufacturers recommend application of gentle or strong air stream. Studies have shown that viscous resinous material with entrapped air bubbles remaining on dentin surface when strong air drying is advocated and also through air syringe device variable amounts of pressure is ejected. The best way to control pressure of air which is emerged from air syringe could be getting closer or further from substrate, because the buttons of air syringe system are not sufficiently sensitive to deliver different air pressure to tooth surface [6].

On the other hand active application of adhesives using scrubbing technique may hasten solvent evaporation, which leads to impregnation of higher rate of monomers inside smear layer. The agitation of one step self adhesives through scrubbing action carries fresh acidic resin monomers to deeper part of dentin which is etched and produces aggressive demineralization, facilitating diffusion of monomers and promoting better interaction with underlying dentin and also with smear layer. This procedure increases the moieties kinetics and allow better monomer diffusion inward, while solvents are diffusing outward, shown with simplified etch and rinse adhesive systems. So, the parameters like air thinning and active application through scrubbing were considered in this study. SEM observations of treated surfaces of dentin elicited single bond universal with scrubbing was efficient for removing smear layer and demineralising dentin substrate with clear opening of dentinal tubules, where as single bond universal without scrubbing technique and applied passively, the treated dentin surface was still covered with a smear layer. Tetric bond universal
with scrubbing showed greater amount of removal of smear layer than without scrubbing. Results indicate that using scrubbing technique was more effective in both the adhesives used in study. Therefore scrubbing technique with air thinning is strongly recommended for the use of mild self etch adhesives. Studies have shown that this active application with scrubbing works well only for mild self etch adhesive systems [9]. Researchers believe that the acidic monomers in strong and moderate self etch adhesive systems are acidic enough to etch underlying substrate and also prime through smear layer and also prime through smear layer [10].

**Conclusion**

Within limitations of the study, it is concluded that application of mild self etch adhesives with scrubbing technique could efficiently remove smear layer and enhance resin monomer infiltration of dentin which can increase the eminence of resin/dentin interface.

**References**


