Dentin Moisture Effect on Microleakage of Indirect Composite Inlays luted with Three Self Adhesive Resin Cements

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Abstract

Objective: The purpose of this study was to investigate the microleakage of Class II indirect composite inlays luted with 3 different self adhesive resin cements on moist and dry dentin. Methods: Sixty caries-free extracted human third molars were mounted in plastic ring using dental stone. Class II OM, OD cavities were prepared on both proximal sides of each tooth. The teeth were randomly divided into 6 groups (n=10) according to three type of self adhesive resin cements [RelyX U100 (3M ESPE), Clearfil SA Cement (Kuraray), MaxCem Elite (Kerr)] and two dentin surfaces conditions: moist dentin (rinsed for 10 seconds, air-dried for 2 seconds from a distance of 1 cm, dentin surfaces were slightly moist and shiny appearance); dry dentin (rinsed for 10 seconds, air-dried for 10 seconds from a distance of 1 cm, dentin surfaces were dry and no shiny appearance). A thin layer of resin cement was applied on all cavity walls. The inlay was placed manually into the prepared cavity by use of finger pressure. Then removed excess cement and all restoration margins were light-polymerized (Elipar 3M) for 40 seconds for each side. Specimens were stored in water at 37 °C for 24 hours. All tooth surfaces were sealed with two layer of nail-varnish to within 1 mm from the restoration margins and they were immersed in 0.5% basic fuchsin solution for 24 hours at 37 °C. The restored teeth were then sectioned mesiodistally and dye penetration was assessed according to a five-point scale. The data were collected and statistically analyzed by Kruskal-Wallis non-parametric test and Mann-Whitney U test. Results: Microleakage in moist dentin groups are less than microleakage in dry dentin groups (p<.05). Microleakage in RelyX U100 was significantly less than Clearfil SA and MaxCem Elite in moist dentin. For dry dentin, there were no significant differences of microleakage in three resin cements (p>.05). CONCLUSIONS: The microleakage in moist dentin groups were significantly less than microleakage in dry dentin groups. RelyX U100 showed lower microleakage than Clearfil SA Cement and Maxcem Elite in moist dentin condition.

Keywords: Self adhesive resin cement, Microleakage, Moist dentin

Introduction

The clinical success of an indirect restorative procedure depends on the cementation technique which has been assisted by the development of a reliable bond between restoration and dentin tissue [1]. Recently, the use of self adhesive resin cement has increased widely because of their simplicity and replacing the sensitive multistep procedures [2,3]. They present several advantages when compared to conventional luting agents, such as favorable mechanical properties, good esthetics and appropriate adhesion to tooth structure and restoration [4,5]. Self adhesive resin cements are designed to self-etch and bond to dentin without using separate etching or priming agents, a single clinical step by self adhesive resin cement was mixed and applied directly on the dentin surface, the adhesion mechanism starts by acidic monomers simultaneously demineralized dentin through smear layer to promote a micromechanical retention to the tooth structure [6]. Chemical retention may be achieved by the reaction between phosphoric acid monomers and the hydroxyapatite of the dental hard tissue [5,6].

The use of indirect restorations has been shown to be an effective way to improve the seal on both dentin and cementum margins by reducing the shrinkage volume of intraoral polymerized materials [7]. However, success with bonding to dentin has been less reliable due to the chemical variations of tooth substrates [8,9], dimensional changes [10], adhesive properties of the materials [11] and differences in the coefficient of thermal expansion of luting materials...
and tooth restoration [12]. Poor adhesion may occur due to limited etching potential of self-etching systems, which could impair the proper infiltration of the cement into dentin [7,13-15]. The adhesive interface between dentin and the restorative material at the gingival margin is acknowledged as a problematic zone in terms of microleakage [16].

Many techniques have been used to assess microleakage. Dye penetration is one of the most common methods in detecting leakage because dyes are inexpensive, easily detectable, and can be safely used because they are nontoxic [17]. The use of 0.5% basic fuchsin dye solution has been considered as acceptable technique for this purpose [18]. The advantage of this tracer is the size of basic fuchsin dye particles. Its size is not an extremely small particle like silver nitrate. Other methods, such as the silver nitrate staining technique, or penetration of radioactive substances, are not commonly used because they are time-consuming and the handling of these materials is difficult [19].

According to the implementation in clinic, before cementation, dentin surface must be cleaned by rinsing and air-dry with triple syringe. This creates an opportunity of making dentin surface being too dried. The purpose of this study was to evaluate the influence of the dentin surface condition (moist or dry) whether it has any impacted on microleakage of indirect composite restorations luted with self adhesive resin cements.

**Materials and Methods**

Sixty extracted intact human third molars were selected for this study after examination with magnifying glass (Topan Asia Autotech Co., Ltd.) and stored in 0.5% Chloramine-T (Fisher Scientific, Pittsburg, Pa) at 4°C, stored less than 6 months. The occlusal surface of teeth were cut 2.0 mm by using Isomet saw (Model Isomet 1000, Series 15, Buehler, Lake Bluff, USA) and they were mounted in plastic ring with dental stone up to 2.0 mm apical to the cementoenamel junction. Class II (OM, OD) cavities were made on both proximal side of each molar with a diamond rotary cutting instrument (204, intensive, Grancia, Switzerland). After every five preparations, the diamond rotary cutting instrument was discarded and replaced with a new one. Each standardized Class II preparations (proximal: 4 mm wide, 5 mm long, 1.5 mm deep and occlusal: 4 mm wide, 1.5 mm long, 1.5 mm deep) prepared on each side and the gingival walls were below the CEJ 1.0 mm (Figure.1). The dimensions of the cavities were verified with a periodontal probe and a digital veneer caliper (Mitutoyo, Tokyo, Japan). All preparation was examined under 20X magnification for possible cracks or fissure. The teeth were then stored in distilled water at room temperature.

| Table 1: Self adhesive resin cements used in study |
|-----------------|-----------------|-----------------|
| Resin cement    | Manufacturer    | Lot number      |
| RelyX U100      | 3M ESPE, USA    | 405969          |
| Maxcem Elite    | Kerr Corp, USA  | 3596266         |
| Clearfil SA Cement | Kuraray Co., Ltd, Japan | 0187AA |
|                  |                  |                  |
| Composition     |                  |                  |

- Methacrylated phosphoric esters, Triethylene glycol dimethacrylate (TEGDMA), dimethacrylates, acetate, initiators, stabilizers, glass fillers, silica, calcium hydroxide (filler = 72 wt%; avg. <9.5 μm)
- Glycerol dimethacrylate dihydrogen phosphate (GPDM), Methacrylate ester monomers , barium glass fillers, fluoride, colloidal silica, surface treated sodium fluoride activator, initiators, stabilizers, accelerators (filler = 66 wt%; 45 vol%; avg. 2.5 μm)

![Figure 1: Class II (OM, OD) cavities design](a) Top view (b) Lateral view

A single impression was made with a 2-phase vinyl polysiloxane material (Aquasil Ultra™ light body, putty, Dentsply, Milford, USA) and poured into type IV stone (Fuji Rock, GC, Tokyo, Japan). After the stones were fully set, stone replicas were removed from impressions and examined for integrity. The cavity outlines were drawn with red pencil. Die spacer (Pico fit™, Renfert, Hilzingen, Germany) was applied.
in proximal cavities (orange color in Figure 1). First layer was applied with silver and second layer with gold. A indirect composite inlay [Tetric®-N Ceram, A1, (Ivoclar Vivadent), Schaan, Liechtenstein] was fabricated for each cavity and polymerized with 700 mW/cm² for 40 seconds using an Elipar™ Halogen curing light (3M ESPE, USA). All margins of indirect composite inlays were finished and polished under cooling water with Sof-lex™ disks (3M ESPE, USA). Indirect composite inlays were removed from the stone replicas and cleaned by using ultrasonic cleaner (Branson 5210, Branson, Germany) in distilled water for 5 minutes.

The teeth were randomly divided into 6 groups (n=10), each group containing 20 cavities, according to three types of self adhesive resin cement and two dentin surface conditions: moist dentin and dry dentin (Table 2). For moist dentin groups, the prepared cavities were rinsed with water for 10 seconds and air-dried for 2 seconds from a distance of 1 cm, the dentin surfaces were slightly moist and shiny appearance. For dry dentin groups, the prepared cavities were rinsed with water for 10 seconds and air-dried for 10 seconds from a distance of 1 cm, the dentin surfaces were dry, no shiny appearance. A thin layer of self adhesive resin cement was applied on all cavity walls. The inlay was placed manually into the prepared cavity by use of finger pressure until it was seated in the cavity. All restoration margins were light-polymerized for 5 seconds and removed excess cement after that light-polymerized for 40 seconds on each side. Specimens were stored in water at 37°C for 24 hours.

Following dye exposure, the teeth were rinsed under tap water, removed nail-varnish with a sickle (#1 Anterior Double End Nevi Scaler, Hu-Friedy, Germany) and polished with pumice then the restored teeth were sectioned mesiodistally with water-cooled, slow speed diamond saw (Isomet, Model Isomet 1000, series 15, Buehler, Lake Bluffil, USA) three 0.8 ± 0.2-mm-thick slabs from the center of the restoration were generated. The slabs were analyzed with a stereomicroscope at 45× magnification and scored for the degree of dye penetration along the dentin walls (mesial and distal gingival margins), according to the microleakage scores in Figure 2.

The present study was performed in accordance with the ISO/TS 11405:2003 standard guidelines [20].

There were 20 cavities per group. Each cavity yielded 3 slabs, so there were 3 measurements for one cavity. The microleakage of each slab was assessed for the mesial and distal margin of the restorations. Then, the highest microleakage score was selected and analyzed. A Kruskal-Wallis non-parametric analysis of variance and Mann-Whitney U test at p<0.05 were used to analyze microleakage scores.

**Results and Discussion**

**Results**

The microleakage scores of each group, means and standard deviations are shown in Table 2.

<table>
<thead>
<tr>
<th>Groups</th>
<th>Microleakage Score</th>
<th>Mean</th>
<th>SD</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0 1 2 3 4</td>
<td></td>
<td></td>
</tr>
<tr>
<td>RelyX &amp; Moist (G1)</td>
<td>3 10 4 2 1</td>
<td>1.40</td>
<td>1.05</td>
</tr>
<tr>
<td>Clearfil &amp; Moist (G2)</td>
<td>0 6 7 4 3</td>
<td>2.20</td>
<td>1.06</td>
</tr>
<tr>
<td>Maxcem &amp; Moist (G3)</td>
<td>0 4 5 6 5</td>
<td>2.60</td>
<td>1.10</td>
</tr>
<tr>
<td>RelyX &amp; Dry (G4)</td>
<td>0 4 7 7</td>
<td>2.95</td>
<td>1.10</td>
</tr>
<tr>
<td>Clearfil &amp; Dry (G5)</td>
<td>0 6 7 4 3</td>
<td>2.95</td>
<td>1.10</td>
</tr>
<tr>
<td>Maxcem &amp; Dry (G6)</td>
<td>0 2 4 4 10</td>
<td>2.53</td>
<td>1.07</td>
</tr>
</tbody>
</table>

The results of Kruskal-Wallis test (Table 3) showed that microleakage between all groups were significantly different (p<0.0001). Microleakage in moist dentin groups were significant difference (p=0.004), but microleakage in dry dentin groups showed that no significant difference (p=0.815). The Mann-Whitney U Test among moist dentin groups (Table 4) showed that microleakage in G1 significantly less than G2 (p=0.010), microleakage in G1 significantly less than G3 (p=0.002), but G2 and G3 was no significantly different (0.24). So in moist dentin condition, RelyX U100 (G1) was less microleakage than Clearfil SA cement (G2) and...
Maxcem Elite (G3). The Mann-Whitney U Test among same resin cement (Table 5) showed that microleakage in G1 significantly less than G4 (p=0.000), microleakage in G2 significantly less than G5 (p=0.035) but microleakage in G3 and G6 was no significantly different (p=0.134). So RelyX U100 was less microleakage in moist dentin than dry dentin, Clearfil SA cement was less microleakage in moist dentin than dry dentin but Maxcem Elite was no significantly different in both moist and dry dentin.

Table 3: Results of Kruskal-Wallis test

<table>
<thead>
<tr>
<th>Chi-Square</th>
<th>df</th>
<th>p-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>All Groups</td>
<td>27.459</td>
<td>5</td>
</tr>
<tr>
<td>Moist Groups</td>
<td>11.250</td>
<td>2</td>
</tr>
<tr>
<td>Dry Groups</td>
<td>0.409</td>
<td>2</td>
</tr>
</tbody>
</table>

Table 4 Results of Mann-Whitney U test among moist dentin

<table>
<thead>
<tr>
<th>Microleakage Score</th>
<th>G1:G2</th>
<th>G1:G3</th>
<th>G2:G3</th>
</tr>
</thead>
<tbody>
<tr>
<td>p-value</td>
<td>0.018</td>
<td>0.002</td>
<td>0.240</td>
</tr>
</tbody>
</table>

Table 5 Results of Mann-Whitney U test among same self adhesive resin cement

<table>
<thead>
<tr>
<th>Microleakage Score</th>
<th>G1:G4</th>
<th>G2:G5</th>
<th>G3:G6</th>
</tr>
</thead>
<tbody>
<tr>
<td>p-value</td>
<td>0.000</td>
<td>0.035</td>
<td>0.134</td>
</tr>
</tbody>
</table>

Figure 3 shows no dye penetration between RelyX U100 and dentin surface in moist condition (7×)

Figure 4 shows dye penetration between Clearfil SA cement and dentin surface in moist condition (7×)

Discussion

All materials used in this study exhibited some degree of microleakage and these finding are agree with another studies [19-20]. In this study, three-cut was made per tooth for more precise in the analysis of tracer penetration [22]. Microleakage between tooth substrate and restorative materials can be expected for all restorative polymers including resin cements. One probable explanation for this is that the adhesive bond becomes weakened by the unavoidable dimensional changes that occur when materials polymerized [23]. The initial marginal gap formation between tooth structure and a luting agent is frequently the result of the polymerization contraction of the luting agent, even if there is only a very thin layer [11]. However, polymerization contraction is only one of the parameters that has role in the mechanisms and durability of adhesion.
Therefore, a lack of marginal sealing will occur if the adhesion of luting agents to the tooth structures does not compensate for the shrinkage stress in the first stage of polymerization [23]. The shrinkage strain of RelyX Unicem showed significantly lower values than Maxcem Elite in dual-cure mode [24]. The low microleakage value in this study is correspond to the low shrinkage strain value of RelyX Unicem, which may decrease the risk of microleakage because RelyX Unicem and RelyX U100 have the same ingredient, composition. But they are in different package. The result from the current study showed that in moist dentin condition, microleakage in RelyX U100 was significantly less than Clearfil SA cement and Maxcem Elite. RelyX U100 has an organic matrix composed of multi-functional phosphoric acid methacrylates, which react with inorganic fillers that are basic in nature or with hydroxyapatite from tooth structure in setting reaction and pH increase from 1 to 6. The polymerization reaction results in extensive cross linking of cement monomers and the creation of high molecular-weight polymers [2], so moist dentin surface provide a good adaptation to the tooth surface and increase sealing ability. Clearfil SA cement contains 10-methacryloxy dihydrogen phosphate (MDP), a phosphate ester bonding agent, which can chemically bond to tooth structure, form aqueous insoluble salt complex between calcium and the relatively hydrophobic MDP [25,26]. The sealing ability is improved [27]. Maxcem Elite, which is the second generation of Maxcem. The composition of Maxcem Elite contains glycerol phosphate dimethacrylate (GPDM) monomer, and other adhesive monomers to improve wettability. GPDM is purportedly responsible in part for its self-etching and adhesive properties. A study showed the lowest interfacial strengths and highest amount of premature failures for Maxcem bonded to dentin or enamel [27], as this study showed that highest microleakage value in moist and dry dentin. To establish whether a correlation might exist between the in vitro results of this and previously conducted study [28]. The limitation of this study include that there was no evaluation of thermocycling effect and mechanical loading effect on microleakage test. The result of in vitro tests should be applied with caution to the clinical situation.

**Conclusion**

Within the limitation of this study, the following conclusions were drawn:
1. Microleakage in moist dentin groups were significantly less than microleakage in dry dentin groups.
2. RelyX U100 showed lower microleakage than Clearfil SA Cement and Maxcem Elite in moist dentin condition.
Reference