The effect of powder/liquid ratio on microleakage of resin-modified glass-ionomer

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Abstract
Introduction: Resin modified glass-ionomer cement (RMGI) is prepared by manual mixing of powder and liquid. Different mixing ratios influence on the RMGI properties. The aim was to compare the effect of different mixing ratios on the microleakage of RMGI.

Materials & Methods: In this in vitro study, 60 Class V cavities (3x2x1.5 mm) with the gingival margin of 1 mm apical to the cement-enamel junction were prepared on the buccal and lingual surfaces of 30 sound premolars. The teeth were randomly divided into 6 groups. Group 1: The manufacturer’s recommended ratio, without conditioning; Group 2: The manufacturer’s recommended ratio with conditioning; Group 3: 20% lower than the manufacturer’s ratio without conditioning; Group 4: 20% lower than the manufacturer’s ratio with conditioning; Group 5: 20% higher than the manufacturer’s ratio without conditioning; Group 6: 20% higher than the manufacturer’s ratio with conditioning. After thermocycling, the microleakage was evaluated using silver nitrate staining. The teeth were cut into two mesial and distal halves, and the microleakage at occlusal and gingival margins was recorded based on a 0–3 scoring system under a stereomicroscope. Data were analyzed using Kruskal-Wallis and Mann-Whitney tests with significance level at P<0.05.

Results: The maximum microleakage at gingival margins was recorded for group 4, which was significantly higher than that of group 2 and 6 (P=0.043 and P=0.043, respectively). No significant differences were observed in the microleakage between occlusal and gingival margins.

Conclusion: A 20% reduction in P/L ratio of RMGI increases the gingival microleakage when surface conditioning was applied.

Keywords: Dental leakages, Glass-ionomer cement, Dental cements
Microleakage is defined as the penetration of bacteria and their toxins through teeth and restoration margins. Microleakage around restorations is a major clinical problem. It can cause hypersensitivity, discoloration, recurrent caries and pulpal problems. Most dental materials, particularly tooth-colored resin materials, exhibit various degrees of microleakage because of dimensional changes and lack of marginal integrity. Glass-ionomer was widely used as a dental restorative material, base, liner and cement. Because of the ability to release fluoride and form real chemical bonds with teeth, Glass-ionomer is recommended as a restorative material to restore cervical lesions. In addition, due to the similarity between the thermal expansion of glass-ionomer and tooth which results in their similar dimensional changes, adhesion between glass-ionomer and tooth will not be under tension. Therefore, it is expected that glass-ionomer will exhibit less marginal microleakage in standard cavities compared to other tooth-colored restorative materials. Glass-ionomer cements are preferable over composite resins for the restoration of cavities with dentinal walls, especially Cl V cavities on the root surface, due to their ability to bond to the tooth structure, micromechanical bond to dentin and enamel, fluoride release, thermal expansion coefficient similar to that of dentin and consequently less microleakage. Varieties in how to prepare the P/L ratio of glass-ionomers by clinicians increases the possibility of changes in the mechanical properties of the cement. Increasing the P/L ratio improves the properties of glass-ionomer.

Glass-ionomer systems usually require manual mixing of the powder and liquid. In addition, capsular forms of glass-ionomers are available. Furthermore, resin-modified glass-ionomers containing a resin component have also been developed. The later types exhibit better physical and mechanical properties and esthetic appearance compared to conventional glass-ionomers. Any change in the mixing ratio gives rise to changes in the properties of glass-ionomer. The P/L ratio may vary from the manufacturer recommendations based on clinicians’ opinions in different situations. Visual measurements and careless use of the measuring spoon can affect the P/L ratio. Studies have shown that an increase in P/L ratio has a direct effect on compressive properties of the cement.
Effect of P/L ratio on microleakage of RMGI

After that, the teeth were checked under the stereomicroscope (Meiji Techno Cold, Tokyo, Japan) to confirm the absence of enamel at the gingival margin. Cavity preparation was carried out with a high-speed 008 diamond fissure bur (Tees kavan Co, Ltd, Tehran, Iran) under air and water spray. A new bur was used for each of 6 cavities. After washing and drying the cavities, the teeth were randomly divided into 6 groups, and Fuji II LC (GC, Tokyo, Japan) glass-ionomer was used in different powder-to-liquid ratios to restore the cavities as follows:

Groups 1 and 2 (3.2 grams of powder and 1 gram of liquid): according to the manufacturer’s recommended ratio, without and with conditioning, respectively.

Groups 3 and 4 (2.56 grams of powder and 1 gram of liquid): 20% lower than the manufacturer’s recommended ratio, without and with conditioning, respectively.

Groups 5 and 6 (3.84 grams of powder and 1 gram of liquid): 20% higher than the manufacturer’s recommended ratio, without and with conditioning, respectively. The powder and liquid were measured with a digital scale (A&D, Jinun, Japan). In groups with conditioning (2, 4 and 6), the GC dentin conditioner (10% polyacrylic acid, GC, Tokyo, Japan) was placed in the cavity using a microbrush for 10 seconds and washed with water for 10 seconds. In all the groups, the powder and liquid were mixed using a metal spatula on a glass slab for 20 seconds and immediately placed in the cavity using instrument to get material in one bulk. A slide of transparent matrix strip was placed on the restoration. Then, tip of the light-curing device (Valo, Ultradent, Tusan, USA) was placed in contact with the transparent strip, followed by light-curing for 20 seconds. The light intensity was 1000 mW/cm² as checked by a radiometer (Kerr, Dallas, USA). Subsequently, the restoration was polished with Sof-lex abrasive discs (3M ESPE, Neuss, Canada). The teeth were dipped in distilled water and then placed in a thermocycling machine (Nemo, Mashhad, Iran) for 24 hours and 500 cycles. Each cycle consisted of 30 seconds in hot water tank (55±2°C), 30 seconds in cold water tank (5±2°C) and 10 seconds to transfer from one container to another. After thermocycling, the apices of all the teeth were sealed with wax; then all the surfaces of the samples were covered with 2 layers of nail varnish except for 1 mm around the restorations margins. The samples were dipped in a solution of silver nitrate (EMSURE, Dermstadt, Germany) for 4 hours in an absolutely dark room. After that, the teeth were rinsed with water and placed in a clearing solution for 8 hours. The teeth were mounted in epoxy resin and sectioned by a diamond disc (0.3 mm) in a cutting machine (Nemo, Mashhad, Iran) in a buccolingual direction into two mesial and distal halves. Each sample was observed by a blinded observer under a stereomicroscope at ×20 (Meiji Techno Coldt, Tokyo, Japan) to determine color penetration and microleakage of the occlusal and gingival margins according to the following ranking system.

0 = no leakage
1 = up to half of the occlusal and gingival walls
2 = up to the dentinal walls, not extending to the axial wall
3 = up to the axial wall

Materials & Methods

In this experimental study, 30 human premolar teeth, extracted for orthodontic reasons, were used. The sample size was calculated based on previous studies.[10-12] The specimens were collected at most three months before the study was undertaken. The teeth were free of caries, facets and fractures. They were stored in 0.9% normal saline solution during the waiting period before the study. The teeth were cleaned using pumice powder and a low-speed handpiece, and disinfected in 0.05% chloramine-T solution (Merk, Darmstadt, Germany) at room temperature for 3-4 days. Then, 60 standard Cl V cavities (3×2×1.5 mm) were papered on the buccal and lingual surfaces. The gingival margins were placed 1 mm apical to the CEJ. All the cavities were checked under the stereomicroscope (Meiji Techno Coldt, Tokyo, Japan) to confirm the absence of enamel at the gingival margin. Cavity preparation was carried out with a high-speed 008 diamond fissure bur (Tees kavan Co, Ltd, Tehran, Iran) under air and water spray. A new bur was used for each of 6 cavities. After washing and drying the cavities, the teeth were randomly divided into 6 groups, and Fuji II LC (GC, Tokyo, Japan) glass-ionomer was used in different powder-to-liquid ratios to restore the cavities as follows:

Groups 1 and 2 (3.2 grams of powder and 1 gram of liquid): according to the manufacturer’s recommended ratio, without and with conditioning, respectively.
The greatest degree of microleakage at occlusal and gingival margins of each half of each tooth was recorded and data were analyzed by non-parametric Kruskal-Wallis and Bonferroni corrected Mann-Whitney tests with SPSS 21. The significant level was considered $P<0.05$.

**Results**

Based on the Kruskal-Wallis test, both values of occlusal and gingival microleakage between the 6 studied groups were not statistically significant. Scores of microleakage are illustrated in table 1. A significant difference was found between gingival microleakage values of groups 2, 4 and 6, (with conditioning) but no significant difference was found for occlusal margins ($P=0.025$ Vs $P=0.254$). There were no significant differences between both gingival and occlusal microleakage values of groups 1, 3 and 5 (without conditioning) ($P=0.276$ and $P=0.380$, respectively). Based on the Mann-Whitney test, no significant difference was found between two groups in gingival and occlusal surfaces except groups 2-4 and groups 4-6 in the gingival margin ($P=0.043$ and $P=0.043$) (table2). Figure 1 indicates the various microleakage values (A-D).

### Table 1. Microleakage in occlusal and gingival margins

<table>
<thead>
<tr>
<th>Type of group</th>
<th>Occlusal Mean Rank</th>
<th>gingival Mean Rank</th>
</tr>
</thead>
<tbody>
<tr>
<td>Score0</td>
<td>Score1</td>
<td>Score2</td>
</tr>
<tr>
<td>Group 1</td>
<td>4</td>
<td>6</td>
</tr>
<tr>
<td>Group 2</td>
<td>7</td>
<td>3</td>
</tr>
<tr>
<td>Group 3</td>
<td>5</td>
<td>3</td>
</tr>
<tr>
<td>Group 4</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Group 5</td>
<td>7</td>
<td>3</td>
</tr>
<tr>
<td>Group 6</td>
<td>6</td>
<td>4</td>
</tr>
</tbody>
</table>

| p-value | 0.437 | 0.072 |

### Table 2. P-value based on the mann-whitney test

<table>
<thead>
<tr>
<th>P-value</th>
<th>1-2</th>
<th>3-4</th>
<th>5-6</th>
<th>6-7</th>
<th>2-6</th>
<th>2-4</th>
<th>3-5</th>
<th>1-5</th>
<th>1-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>gingival</td>
<td>0.280</td>
<td>0.165</td>
<td>0.739</td>
<td>0.043</td>
<td>1.00</td>
<td>0.043</td>
<td>0.579</td>
<td>0.143</td>
<td>0.579</td>
</tr>
<tr>
<td>occlusal</td>
<td>0.280</td>
<td>0.796</td>
<td>0.739</td>
<td>0.315</td>
<td>0.739</td>
<td>0.190</td>
<td>0.353</td>
<td>0.280</td>
<td>0.971</td>
</tr>
</tbody>
</table>

**Figure 1:** Different values of dye penetration and microleakage score (A-D)

A: No leakage (Score0)
B: Dye penetration up to half of the occlusal and gingival walls (Score1)
C: Dye penetration up to the dentinal walls, not extending to the axial wall (Score2)
D: Dye penetration up to the axial wall (Score3)
Discussion

The first aim was to determine the effect of P/L ratio of resin-modified glass-ionomer on microleakage of Cl V restorations. The later aim of this study was to determine the effect of a cavity conditioner on microleakage.

There was no considerable difference both in gingival nor occlusal microleakage values by a 20% reduction or a 20% increase in P/L ratio without conditioning. However, gingival microleakage increased with a 20% reduction in P/L ratio when the conditioning was used. To the best of our knowledge no such study has been under taken to date, this no data comparsion can be illustrated norder with our. Never the less, previous researches have demonstrated the use of conditioner in glass ionomer cement restorations reduced the microleakage at gingival margins. 

It was demonstrated that the bond strength of RMGI to enamel is greater than to dentin. Typical values for tensile bond strengths varied from 4.90 to 11.36 MPa on enamel and from 2.52 to 5.55 MPa on dentin. Two distinct adhesion mechanisms to tooth surface were found for RMGI: a chemical bonding between calcium ions in hydroxyapatite and anions of polyalkenoic acid and a micromechanical bonding.

It is also reported that enamel (occlusal) margin of permanent teeth restored with GICs shows less microleakage than dentin (gingival) margins. The difference observed between microleakage values of enamel (occlusal) and dentin (gingival) margins may be due to the difference between the quality of the bond regarding enamel and to dentin structures.

The dentin treatment procedures are optionally performed to increase the marginal adaptation and bond strength of GICs to dentin. The cavity conditioner removes the smear layer without completely unplugging the dentin tubules. The exposed calcium ions within hydroxyapatite are available for chemical bonding with the carboxyl groups of the polyalkenoic acid. The collagen that have become exposed may provide additional micromechanical retention. Aluminum chloride in cavity conditioner is thought to stabilize the collagen matrix during demineralization, allowing better penetration of the RMGI.

The authors hypothesized that the difficulty in manipulation and placing the low consistency RMGI into cavity may adversely affect the marginal adaptation and since dentin conditioning caused more porosity in the dentinal surface, the possibility of microleakage might be increased. According to this result and justifications, further investigation should be done.

The authors hypothesized that less filler content in low viscosity resin modified glass ionomer (group 4) resulted in more microleakage value than that of high and normal viscosities of RMGI. Gupta suggested that the microleakage of nano-filled resin –modified glass ionomer was lower than that of conventional glass ionomer and resin-modified glass ionomer in gingival margin. They indicated that it was due to the higher filler loading in the nano-filled type which may result in lower polymerization shrinkage and lower thermal expansion. A polymerization reaction occurs with the HEMA and urethane dimethacrylate monomers of the resin matrix that produce additional shrinkage.

Mirzaie et al. compared microleakage of nanofilled RMGI and conventional glass-ionomer in Cl V cavities, using two types of bonding agents, i.e. self-etch adhesive and self-etch primer. Since the bond strength of GI is lower than that of composite resin, self-etch adhesive systems were used to improve GI bonding to dentin. A total of 40 third molars were divided into 4 groups. On the occlusal wall, the least amount of microleakage was detected in the SE bond Fuji IX group, which was significantly different from other groups, most of the microleakages were nano glassionomer and g bond. At gingival margins, the use of Fuji ZX and SE bond decreased microleakage and this difference was significant for Fuji ZX and the highest microleakage was recorded with Fuji IX and G bond. Overall use of the SE bond compared with the G bond was more effective and was able to be effective in reducing leakage.

Mazaherí et al. studied the effect of 4 types of conditioners, 20% polyacrylic acid, 35% phosphoric acid, 12% citric acid and 17% ethylene diaminetetraacetic acid (EDTA) on the microleakage of glass-ionomer in primary teeth. The amount of microleakage in the control group (without conditioner) was significantly higher than that of the other groups. In this study, the least amount of microleakage was observed through the application of polyacrylic acid. Many studies have shown that acrylic acid is a good conditioner which increases the bond strength between dentin and glass-ionomer. This acid has little effect on the tooth structure. This means that it removes the smear layer and the contaminated layer without extremely opening the dentinal tubules. It can be concluded that the use of weak etching or a conditioner is suitable, but
it should not damage the dentinal tubes. The purpose of using a conditioner is to remove the smear layer and surface contamination of teeth, which can reduce the adhesion of cement to the tooth surface. It has been shown that concentration of the conditioner, method and application time can result in the removal of the smear layer.\[14\] This result coincides with that reported by Mirzaie et al. \[10\] In this study, no significant difference was observed in the occlusal microleakage values of all groups. The comparison between gingival microleakage values of all groups did not show a statistically significant difference as well (p=0.07), which may be considerable (Table1). The authors opinion is came to this condition that since the p-value of gingival microleakages was close to the significance level, further studies should be conducted on a larger sample size.

In the present study, thermocycling with 500 temperature cycles was used to estimate the dynamic temperature of oral cavity. Since no significant difference was observed in microleakage between 500, 1000, 2000 and 4000 cycles, thermocycling with 500 cycles was selected, which is normally used in studies.\[13\]

There are different ways to evaluate microleakage. Dye penetration is a common technique that has been applied in various studies using different dyes include methylene blue, fuchsin, silver nitrate, etc. \[20\] Silver nitrate was used in a study conducted by Déjou et al. In which they represented that silver nitrate with 50 wt% has the best penetration and reported it as the best criterion for measuring microleakage, while the use of other detectors such as fuchsin, methylene blue can affect the real amount of microleakage due to their small sizes which are smaller than bacteria. \[21\]

In this study, a 20% increase and decrease in powder-to-liquid ratios regarding the manufacturer’s ratio were examined. These three consistencies are similar to the consistency usually used in the clinic for various applications. In a study by Torabzadeh et al. these three consistencies were used to check the flexural and shear bond strengths. It was found that flexural and shear bond strengths did not change significantly by changing the powder/liquid ratio. \[7\]

Increasing the volume of reinforcing glass particles and consequently increasing the compressive strength and decreasing the working time were evident by application of manufacturers’ ratio compared to using 80% and 50% the recommended GIC powder content \[8\] So, an accurate admeasured P/L ratio should be used to consider the probable clinical problems. Therefore, the authors suggested that further studies should be carried out on other properties of resin modified glass-ionomer with different applicable consistencies.

**Conclusion**

Considering the limitation of this study and despite of the insignificant differences were observed among the study groups, a higher viscosity (a 20% increase in P/L ratio) of RMGI without conditioning, indicated the least occlusal and gingival microleakages.

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**Conflict of interest:** The authors declared no conflict of interest.

**Authors’ Contributions**

The study was designed by Samane Gharekhani. The study data were collected by Sepide Ostadi. Analysis and interpretation of data, drafting of the manuscript, and critical revision of the manuscript for important intellectual content were performed by Soraya Khafri and Samane Gharekhani. Study supervision was conducted by Samane Gharekhani and Ghazaleh Ahmadi.

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