

Effect of powder/liquid ratio of glass ionomer cements on flexural and shear bond strengths to dentin

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Abstract

Aim: This study evaluated the effect of varying the powder/liquid (P/L) ratio on the shear bond to dentin (SBS) and flexural strength (FS) of glass ionomer cements (GIC)s. **Methods:** Three types of GICs (Fuji II, Fuji II LCi and Fuji IX GP) were mixed using the following P/L ratios: 20% lower than the manufacturer's ratio, manufacturer's ratio, 20% higher than the manufacturer's ratio (9 groups). SBS (MPa) was evaluated and the mode of failure checked under stereomicroscope. FS (MPa), of the specimens (25×2mm) assessed using a universal testing machine at a cross-head speed of 1 mm/min. The data were subjected to two-way ANOVA and Tukey's test for analysis ($p < 0.05$). **Results:** The highest SBS and FS (MPa) obtained for Fuji II, Fuji II LCi and Fuji IX were 6.12 ± 2.11 and 16.96 ± 2.73 ; 11.60 ± 3.19 and 49.58 ± 8.75 ; 7.39 ± 2.77 and 20.32 ± 2.09 , respectively. The interaction between materials and P/L ratios had no significant effect on the properties tested in this study. Fuji II LCi exhibited significantly higher SBS and FS than the other two GICs in all P/L ratios. **Conclusions:** No significant differences were observed between Fuji II and Fuji IX. Twenty percent variation in P/L ratio had no significant effect on SBS and FS of GICs.

Keywords: conventional glass ionomers, resin-modified glass ionomers, powder/liquid ratio, bond strength, flexural strength.

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Introduction

Glass ionomer cements (GICs) are recognized due to their remarkable advantages, such as chemical bonding ability to enamel and dentin and long-term fluoride release¹⁻². However, these cements have limited applications, especially on stress areas, which is attributed to their inferior mechanical properties²⁻⁴. New types of GICs have been introduced by the addition of resins, such as HEMA, to the conventional GIC formulation. The so-called resin-modified GIC (RMGIC) relatively possess the same shortcomings⁵⁻⁶.

Powder/liquid (P/L) ratio is one of indicative factors in altering mechanical properties of GICs; the higher the amount of powder, the higher the mechanical properties^{5,7-8}. This correlation has been explained through the particles of the powder that remain unchanged due to lower level of acid. It has been proposed that these unreacted particles appear like filler in such a way that they prevent crack propagation within the cement⁵. The powder amount of the cement has a direct correlation with compressive strength, porosity and wear resistance and an indirect correlation with solubility, setting and working time⁹⁻¹⁰. Accordingly, this is obvious that P/L ratio can significantly affect the mechanical properties of GICs and their clinical performance as well. The P/L ratio for clinical use may vary from one manufacturer to another because of several factors. Visual scaling and careless use of scaling scoop can affect this ratio adversely^{7,10-12}. The liquid drop can also vary in volume due to bottle inclination during dispensing, shape of the bottle outlet, as well as presence and size of air bubble in the bottle^{7,10}. Nevertheless, even an accurate use of the scoop and bottle leads to 2-8% of variation in P/L ratio¹³. Variation in P/L ratio (approximately 6%) in capsulated GICs has also been reported¹⁴. Therefore, the aim of this study was to evaluate the effect of different P/L ratios on the shear bond strength to dentin and flexural strength of three types of GICs.

Material and methods

In this experimental study, three types of GICs from the same manufacturer (GC corporation, Tokyo, Japan) were used (Table 1). These GICs were mixed manually with three different P/L ratios as follow:

Group 1: 20% lower than the manufacturer's instruction (subgroups: 1, 4, 7)

Group 2: Same as the manufacturer's instruction (subgroups 2, 5, 8)

Group 3: 20% higher than the manufacturer's instruction (subgroups 3, 6, 9)

Prior to mixing, the powder and liquid were weighted according to the mentioned protocol at room temperature with an accuracy of 0.001 g using a digital scale (ACULABAL-104).

Table 1 - Glass ionomer cements used in this study

Product	Lot no.	Curing method	Recommended P/L ratio
Fuji II	0502261	Self cure	2.7/1
Fuji II LC Improved	0612051	Light cure	3.2/1
Fuji IX GP	0511121	Self cure	3.6/1

Shear bond strength test

Forty-five extracted, sound (*i.e.* caries-free, restoration-free and with fully formed crowns), erupted and unerupted human third molars were used. This study was carried out according to the protocol approved by the Ethics Committee of the Iran Center for Dental Research (ICDR), Shahid Beheshti

Medical University, Tehran, Iran. Soft tissue remnants and other debris were removed from tooth surfaces and the teeth were placed in 0.5% chloramine solution for 3-4 days. Subsequently, they were washed thoroughly and kept in distilled water at 4°C for less than two months.

Teeth were then sectioned sagittally with a microtome to eliminate the occlusal enamel and expose the underneath dentin. Smooth surfaces were formed on the dentin surface when the specimens were lapped on 600-grit abrasive paper. Under a stereomicroscope, complete removal of the enamel and the smoothness of dentine surface were confirmed. The specimens were then rinsed off thoroughly with distilled water for at least 30 s.

Twenty-one gauge (1.6 mm) scalp vein tube (Shan Chuan, Zibo, China) with 1 mm height were overfilled with GIC and their clean ends were placed over the conditioned dentin. For the purpose of more condensation and also removing the excess material, a Mylar strip and a glass plate were slightly pressed over the specimens. The specimens were then kept in 37°C and 80% relative humidity for 15 min and were stored in distilled water for 24 ± 1 h. Specimens of Fuji II LCi were light cured (Arialex, Tehran, Iran) for 20 s. Ten specimens were prepared for each study group.

Using a scalpel, the surrounding cylindrical tube of each specimen was removed under stereomicroscope then the specimens were transferred to the microtensile tester machine (Bisco Inc., Schaumburg, IL, USA). The bond strength (MPa) was measured through the failure load divided by cross sectional area of each specimen. The surfaces were examined under stereomicroscope (x20) to assess the failure mode.

Flexural strength test

From each group, five specimens with dimensions of 2 × 2 × 25 mm were prepared using a stainless steel mold (ISO 4049)¹⁴. The molds were placed on glass plates, overfilled with GIC and then, another glass plate was used with slight pressure on the mold in order to remove the excess cement. Two Mylar strips were used to prevent adherence of specimens to the plates.

Fuji II LCi specimens were light cured in five overlapping areas from the core part to the sides and were then stored in an incubator (37°C, 80% relative humidity) for 15 min immediately after curing while self cured specimens transferred to the incubator after 3 min. Specimens were separated from the molds and stored in distilled water at 37°C for 24 ± 1 h.

Before transferring the specimens to the universal testing machine, their dimensions were measured with a digital caliper accurate to the nearest 0.01 mm and then loaded at a crosshead speed of 1 mm/min. The fracture load of specimens was recorded to be used for calculating the flexural strength (MPa) as follows:

$$\sigma = 3Fl/2bh^2$$

(l: distance between 2 jigs, b: specimen's width, h: specimen's height and σ : flexural strength).

Data were analyzed using two-way ANOVA and Tukey's test. The significant level was set at $\alpha=0.05$.

Results

Means and standard deviations of shear bond and flexural strength are shown in Table 2.

For both conventional GICs, the lowest and highest means of bond strength were observed in groups 1 and 3, respectively; for Fuji II LCi, these values belonged to groups 1 and 2, respectively.

Fuji II showed the lowest mean flexural strength in group 2 and the highest mean flexural strength was found in group 3. For Fuji IX GP, the lowest flexural strength was recorded in group 1 and the highest in group 3. The lowest and highest means of flexural strength for Fuji II LCi was observed in group 3 and 1, respectively.

Two-way ANOVA showed that the bond and flexural strengths were not influenced by the P/L ratio, but they were significantly altered by the type of GIC. In this respect, bond and flexural strengths of Fuji II LCi were significantly higher than those of the two other materials. However, no significant difference was observed between Fuji II and Fuji IX GP ($p > 0.05$).

Failure modes revealed the increase of P/L ratio resulted in more adhesive failures for Fuji II and Fuji IX GP specimens. For Fuji II LCi, adhesive failures were predominant; however, the effect of P/L ratio was different from that of the other materials. Accordingly, the lowest incidence of adhesive failures for Fuji II LCi was observed using the P/L ratio recommended by the manufacturer (group 2). Results also showed that, in general, the increase in the mechanical strength of cement will increase the bond strength.

Table 2 - Means and standard deviations for shear bond and flexural strength (MPa)

Material	Group	P/L Ratio	Shear bond strength	Flexural strength
Fuji II	Group 1	2.16/1	4.75±1.26	15.75±2.84
	Group 2	2.70/1	5.50±1.94	14.57±1.91
	Group 3	3.24/1	6.12±2.11	16.96±2.73
Fuji II LCi	Group 4	2.56/1	11.06±3.52	9.58±8.75
	Group 5	3.20/1	11.60±3.19	47.97±2.85
	Group 6	3.84/1	11.59±2.85	44.96±5.86
Fuji IX GP	Group 7	2.90/1	5.57±1.64	17.66±1.99
	Group 8	3.60/1	6.29±1.88	18.99±2.46
	Group 9	4.40/1	7.39±2.77	20.32±2.09

Discussion

Variation in the P/L ratio is an important factor in optimizing GIC properties. Nevertheless, the amount of powder used under clinical conditions is often overlooked. Billington *et al.*¹³ (1990) showed that this amount was 27% less than that recommended by the manufacturer. This finding was confirmed by Beher *et al.*⁷ (2006) who pointed out a 17% variation range. In addition, some studies showed that the more the amount of powder, the better the mechanical and physical properties^{7,15-16} and have suggested to use the

most possible powder amount the strength of GIC is required¹².

The results of the present study indicated that regardless of the variation in P/L ratio, shear bond and flexural strengths of RMGIC are higher than those of conventional GIC. This is in accordance with previous studies^{2,4-5,17}. Variation in P/L ratio of each material showed no significant difference in bond and flexural strengths, which is in contrast with other investigations^{5,10,16}. This can be explained by some differences including no report of P/L ratio variation, no exact statistical analysis¹⁶, differences in the tested materials and studied mechanical properties^{5,10,16}. As mentioned before, some researchers believe that greater powder amount can improve the mechanical properties due to the filler-like function of unreacted powder particles⁵. In fact, in a recent study, it was shown that the increase in P/L ratio of a RMGIC luting cement by 30 percent resulted in an increase in compressive, flexural and bond strengths¹⁸. However, no increase was observed when P/L ratio increased by only 20 percent, which was confirmed in the present investigation. In addition, Fonseca *et al.*¹⁹ (2010) found that a 50% alteration in P/L ratio produced a weaker RMGIC in diametral tensile strength. It should be noted that this reduction was only observed in RMGICs, and it could be attributed to a suppressed acid-base reaction, which, in turn, may lead to a compromised interaction between matrix and powder particles^{5,15,19}. Consequently, the strength of the materials does not increase.

This way, the type of material, the investigated property and also the amount of unreacted particles are critical factors to be considered. These factors may explain the difference between the present study and that of Fleming *et al.*⁹ (2003), in which the compressive strength of Chemfil was investigated and significant differences were achieved⁹. Lund *et al.*²⁰ (2007) It was also reported that the effect of P/L ratio on the diametral tensile strength of RMGIC is brand dependent. However, it is noteworthy that diametral test is only recommended for brittle materials.

Due to the vast disparity of P/L ratios and the results reported, it is very challenging to make a definite conclusion concerning the ideal P/L ratio. For instance, Mitsuhashi *et al.*¹⁵ (2003) found no significant difference in fracture strength and mechanical properties of RMGICs by reducing the amount of powder by 33%. Furthermore, a recent study indicated no significant difference in flexural strength of RMGIC after 17 and 25% decrease and 33 and 50% increase in powder level⁷.

The majority of the previous studies have considered the reduction in powder amount^{11,15-16}, whereas this study investigated both decrease and increase of powder/liquid ratio. Problems attributed to the incorporation of high powder content during mixing procedure have encouraged investigations on the decreased P/L ratio¹⁵.

This study also evaluated the effect of P/L ratio on the bonding strength; this has not been examined before, which can be due to difficult specimen preparation¹⁰.

In the present study, mixing the cement with greater powder amount was difficult, but the handling was improved due to less adhesiveness, except for Fuji II LCi in which the increase in powder amount resulted in a sticky material that

immediately lost homogeneity and made the application difficult and almost impossible.

Under clinical conditions, as the increase in powder amount did not show adverse effects on mechanical properties, it can be recommended to increase the powder amount to achieve better handling properties. In this case, the clinical longevity of GIC restorations will probably increase.

Based on the experimental design of the present study, it was verified that:

1. Twenty percent variation in P/L ratio had no significant effect on the shear bond and flexural strengths.

2. Shear bond and flexural strengths of RMGIC was significantly higher than those of conventional GICs; however, no significant difference was observed between the two conventional GICs.

3. When the powder amount is increased, adhesive failures most frequently occur between dentin and cement rather than within the cement.

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