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Effect of Powder/Liquid Ratio on Fluoride Release of Glass Ionomers

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Abstract

Objective: Evidence shows that the powder/liquid mixing ratio recommended by the manufacturers is often not respected when mixing the glass ionomer (GI) powder and liquid, yielding a GI cement with disproportionate powder/liquid ratio. Considering the confirmed effect of powder/liquid ratio on the GI properties, and more importantly, its fluoride release potential, this study aimed to assess the effect of powder/liquid ratio on fluoride release of GI cements.

Methods: Fuji II, Fuji II LC Improved and Fuji IX GI cements were used in this experimental study. Of each material, three groups with powder 20% less than recommended, the exact recommended ratio and powder 20% more than recommended, were prepared. To assess the fluoride release potential, 45 disc-shaped specimens measuring 2×4mm were prepared (5 per each group). After fabrication, the specimens were immersed in 5 mL of distilled water. The amount of fluoride released into distilled water was measured at days 1 to 7, and also at 13, 14, 15, 28, 29, 30, 58, 59, 60, 88, 89, and 90 days, using Ion Selective Electrode (ISE). After each time of measurement, distilled water was replaced. Data were analyzed using repeated measures ANOVA. Tukey's post hoc test was used for pairwise comparison of groups and powder/liquid mixing ratio. For pairwise comparison of time points, the Bonferroni adjustment was applied (p<0.05).

Results: Based on the results, although the amount of fluoride released from Fuji IX was higher than Fuji II, this difference was not statistically significant (p=0.589). The lowest fluoride release was seen in Fuji II LC and this difference was statistically significant (p<0.05). Change by 20% in the powder/liquid mixing ratio in the three GI cements had no significant effect on fluoride release (p=0.650, p=0.103, p=0.082).

Conclusion: Fluoride release from GI was time-dependent and the amount of released fluoride decreased over time. Fuji II LC resin-modified GI (RMGI) released less fluoride than Fuji II and Fuji IX. Also, 20% change in powder/liquid mixing ratio had no significant effect on fluoride release in different groups.

Key words: Fluoride, Glass ionomer cement, Ion selective electrode, Powder/liquid ratio, Release pattern

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Introduction:

Unique properties of GIs such as their bond to enamel and dentin, and their anti-caries potential due to fluoride release make them suitable for clinical application. However, they suffer shortcomings as well including short working time, relatively long setting time, brittleness, very low wear resistance and susceptibility to moisture or dehydration within the initial phase of setting (1).

To overcome water sensitivity and improve the mechanical properties of conventional GIs, a hydrophilic polymerizing resin was incorporated into the formulation of conventional GIs, yielding RMGIs. Similar to conventional GIs, RMGIs chemically bond to enamel and dentin and are more conveniently used in the clinical setting due to having longer working time (2-4). Some mechanical properties of RMGIs have improved compared to conventional GIs. For instance, RMGIs have higher flexural and tensile strengths (5) higher wear resistance, less brittleness, and optimal polishability immediately after light curing (3). Their higher bond strength compared to that of conventional GIs is among their most important advantages (6, 7). Polymerization shrinkage and toxicity due to monomer incorporation and their high water sorption are among the shortcomings of RMGIs. Water sorption of these materials is influenced by their resin component and results in their hydrolysis and softening of their matrix (8-10). Theoretically, it has been stated that by increasing the powder/liquid ratio, the mechanical and physical properties of GIs improve (9, 11, 12); because, by doing so, the concentration of reinforcing glass particles increases. This also decreases porosities in the cement mass. But, this increase shortens the mixing time and working time and decreases translucency. However, Torabzadeh et al., in 2011 increased the P/L mixing ratio and observed no significant change in flexural strength (13).

The results regarding the correlation of P/L ratio of GIs with their fluoride release potential have been controversial. Some studies have reported a reduction in fluoride release by decreasing the P/L ratio of GIs (9, 14-16). While, some other have demonstrated increased release of fluoride from GIs following decreasing the P/L ratio (17-20).

Fluoride release is among the most important characteristics of GIs and plays an important role in clinical application. Considering the significant effect of P/L ratio on the properties of GIs, lack of adequate information and the existing controversial studies in this regard, this study aimed to assess the effect of change in P/L ratio of GIs on the amount of fluoride released from two types of self-cure GIs and one type of RMGI cement.

Methods:

This experimental study was conducted on three types of GIs namely: Fuji II, Fuji IX and Fuji II LC. The characteristics of these GIs are presented in Table 1.

Table 1- The characteristics of the three types of GIs used

	Fuji II	**Fuji II LC (improved)**	**Fuji IX GP**
Manufacturer	GC Corporation, Tokyo, Japan	GC Corporation, Tokyo, Japan	GC Corporation, Tokyo, Japan
Color	22 Yellow-Brown	A3	A3
Serial number	0502251	0612011	0612051
Curing method	Chemically cured	Light cured	Chemically cured
Mixing time	30 seconds	20-25 seconds	25-30 seconds
Working time	1 minute and 45 seconds	3 minutes and 45 seconds	2 minutes
Setting time	5 minutes and 30 seconds	-	2 minutes and 20 seconds
Light curing time	-	20 seconds	-
A2 curing depth	-	1.8 mm	-
Suggested P/L ratio	2, 7.1	3, 2.1	3, 6.1

By changing the P/L ratio, 3 groups of 5 specimens each were prepared:

Group one had P/L ratio 20% less than the ratio recommended by the manufacturer

Group two had P/L ratio similar to what was recommended by the manufacturer

Group three had P/L ratio 20% more than the ratio recommended by the manufacturer.

The powder and liquid of GIs were weighed with Acculab AL-104 (Acculab, USA) digital scale with 0.0001g accuracy and specimens were prepared at room temperature.

The powder and liquid were then mixed according to the manufacturer's instructions (Fuji IX GP) with a plastic spatula within the instructed time period. Mixed cement was transferred to a plexyglass mold measuring 4mm in diameter and 2 mm in depth and a thin stainless steel wire was placed inside the molds in such way that one end of the wire was out of the mold. This wire was used to suspend the specimens in the container. A Mylar strip and a glass slab were placed over the specimens and slightly compressed in order to better pack the GI and allow the excess material to leak out. The specimens were rested at 37° C and 80% humidity for 15 minutes. Fuji II LC specimens were light cured for 20 seconds from the top and 20 seconds from the bottom to set. All specimens (n=45) were then transferred to screw-top containers containing 5mL of double distilled water. The specimens were kept in an incubator at 37° C during the study period. The solution containing the fabricated specimens was tested at days 1-7 and then at 13, 14, 15, 28, 29, 30, 58, 59, 60, 88, 89 and 90 days. At each time of testing, the specimens were removed from the solution and rinsed with 1mL of double distilled water; this water was then added to the 5mL of distilled water already in the container. Afterwards, the specimens were transferred to new containers containing 5mL of fresh double distilled water. To assess the amount of fluoride released into the solutions, a fluorometer (GLP22+, Crison, Spain) was used. Prior to each measurement, the device was calibrated; 2mL of each solution was diluted with 0.5 mL of TISAB III (Batch #078171, 070171, Crison, Spain) and along with the electrodes, transferred to the measurement container. To transfer the solutions, 0.5 and 1mL samplers (Labtron, Iran) with disposable tips were used.

The amount of fluoride ions in the solutions was recorded by a fluorometer in µg/mL and converted to µg/mm^2 for easier comparison with the results of other studies.

Repeated measures ANOVA with two between-subjects factors of type of material and P/L ratio and one within-subjects factor of time was applied for statistical analysis of data. Pairwise comparison of materials and P/L ratio was carried out using Tukey's HSD test. Pairwise comparison of time points was done using Bonferroni adjustment.

Results:

Table 2 shows the mean and standard deviation (SD) values for the amount of released fluoride at different time points using different P/L ratios of GIs.

Considering the significant interaction effects, analyses were performed separately for each material. Based on Table 2, Fuji II released different amounts of fluoride over time and this difference was statistically significant ($p<0.001$). Comparison of the cumulative amount of fluoride released showed that Fuji II specimens with P/L ratio less than the recommended ratio by the manufacturer released more amount of fluoride than specimens prepared as recommended by the manufacturer or those with a higher P/L ratio; although this difference was not statistically significant ($p=0.286$). Moreover, the interaction effect of time and P/L ratio on fluoride release was not significant ($p=0.257$).

Table 2- The mean (SD) of the amount of fluoride released from GIs with different P/L mixing ratios

	Fuji II			Fuji II LC			Fuji IX		
	A	**B**	**C**	**A**	**B**	**C**	**A**	**B**	**C**
At 1 day	29.43 (8.83)	35.58 (13.81)	31.39 (21.57)	13.52 (2.07)	13.69 (9.01)	9.56 (1.75)	34.52 (2.07)	44.40 (9.01)	81.33 (1.75)
At 2 days	25.43 (8.59)	19.70 (5.66)	14.97 (6.32)	5.29 (2.22)	12.97 (7.14)	4.66 (1.13)	18.09 (2.22)	15.63 (4.72)	14.12 (1.13)
At 3 days	13.02 (2.36)	10.87 (3.59)	7.05 (5.25)	2.90 (0.96)	7.53 (4.84)	3.24 (0.50)	5.77 (0.96)	9.24 (7.14)	5.33 (0.50)
At 4 days	5.76 (0.72)	4.52 (0.33)	3.15 (0.64)	2.40 (0.31)	3.40 (0.41)	3.30 (0.24)	3.12 (0.31)	4.84 (3.84)	6.35 (0.24)
At 5 days	7.43 (1.49)	5.97 (0.19)	4.10 (0.89)	2.60 (0.30)	3.07 (0.40)	2.97 (0.27)	4.38 (0.30)	5.07 (0.41)	7.58 (0.27)
At 6 days	6.61 (1.53)	5.51 (1.23)	5.28 (2.17)	2.55 (0.56)	3.07 (0.54)	3.06 (1.89)	4.44 (0.59)	5.07 (0.40)	7.37 (1.89)
At 7 days	6.73 (1.84)	5.55 (1.11)	5.12 (1.21)	4.77 (0.38)	3.99 (0.67)	4.42 (0.55)	6.37 (0.37)	5.56 (1.22)	5.12 (1.84)
At 13 days	14.23 (8.75)	33.68 (12.94)	32.68 (15.14)	12.55 (3.48)	9.74 (2.15)	11.01 (2.76)	22.73 (8.42)	35.56 (15.14)	18.67 (8.76)
At 14 days	4.60 (1.06)	4.91 (0.42)	4.20 (1.18)	2.07 (0.19)	2.11 (0.16)	2.09 (0.34)	5.75 (5.28)	9.25 (1.18)	5.33 (2.21)
At 15 days	2.27 (0.60)	2.09 (0.20)	1.58 (0.56)	1.74 (0.31)	1.91 (0.35)	1.11 (0.97)	2.27 (0.21)	2.09 (0.56)	1.58 (0.91)
At 28 days	46.90 (15.27)	38.31 (18.89)	31.70 (12.45)	16.34 (8.18)	11.92 (3.2)	7.83 (2.64)	18.64 (6.36)	28.96 (12.45)	23.16 (13.03)
At 29 days	2.72 (1.17)	2.17 (0.82)	2.54 (0.93)	5.11 (2.22)	3.87 (1.31)	3.09 (0.88)	4.44 (0.30)	5.07 (0.93)	7.37 (2.46)
At 30 days	2.36 (0.47)	4.56 (0.52)	1.93 (0.72)	1.56 (0.20)	1.71 (0.33)	1.42 (0.14)	1.68 (0.16)	1.89 (0.70)	2.17 (1.19)
At 58 days	130.56 (3.27)	118.34 (44.15)	79.93 (48.71)	41.65 (20.09)	40.95 (9.41)	31.40 (12.42)	52.59 (20.41)	74.75 (39.83)	60.71 (33.00)
At 59 days	1.54 (0.65)	2.55 (0.33)	1.56 (1.13)	1.53 (0.30)	1.53 (0.17)	2.37 (0.53)	0.88 (0.42)	1.54 (0.85)	3.22 (2.30)
At 60 days	1.61 (0.28)	2.73 (0.37)	2.85 (0.50)	0.72 (0.11)	0.65 (0.13)	0.56 (0.07)	1.23 (0.33)	3.12 (0.34)	1.62 (0.52)
At 88 days	44.92 (0.17)	26.87 (4.35)	34.06 (23.15)	14.78 (5.56)	21.16 (2.69)	9.86 (1.09)	24.07 (5.23)	21.68 (4.61)	16.13 (4.05)
At 89 days	1.18 (0.37)	2.29 (0.71)	1.30 (0.39)	0.74 (0.11)	0.41 (0.05)	0.75 (0.29)	1.00 (0.32)	1.30 (0.54)	1.42 (0.22)
At 90 days	0.45 (0.08)	0.33 (0.06)	0.19 (0.04)	0.24 (0.09)	0.21 (0.04)	0.17 (0.04)	0.29 (0.05)	0.28 (0.03)	0.15 (0.01)

Group A: P/L ratio 20% less than the recommended ratio by the manufacturer
Group B: P/L ratio as recommended by the manufacturer
Group C: P/Lratio 20% more than the recommended ratio by the manufacturer
All values are expressed in μg/mm^2.

Based on Table 2, Fuji IX released different amounts of fluoride over time. This difference was statistically significant ($p<0.001$). Comparison of the cumulative amount of fluoride released showed that Fuji IX specimens with P/L ratio less than the recommended ratio by the manufacturer released more fluoride than other ratios; however, this difference was not significant ($p=0.503$). Also, the interaction effect of time and P/L ratio on fluoride release was not significant for this material ($p=0.085$).

Based on Table 2, Fuji II LC released different amounts of fluoride over time and this difference was statistically significant ($p<0.001$). Comparison of the cumulative amount of fluoride released showed that Fuji II LC specimens with P/L ratio less than the recommended ratio by the manufacturer released more fluoride than other ratios; however, this difference was not significant (p=0.125). Moreover, the interaction effect of time and P/L ratio on fluoride release was not significant for this material (p=0.188).

Discussion:

This study was performed using Ion Selective Electrode (ISE); because it is among the most commonly used methods for measurement of fluoride ions present in biological environments. Theoretically, this electrode can respond to changes in the range of 100-10^{-6} M. The only important interference of this electrode is with the hydroxide ion. In the current study, in order to prevent this particular interference and other possible ionic interferences, and also for standardization of pH and ionic strength, total ionic strength adjustment buffer (TISAB) was used. Fluoride measurement is easy, accurate and fast using this method. The results obtained by this method have over 90% reproducibility (21). This value depends on the type of specimen and its method of fabrication. Evidence indicates that the P/L ratio used in the clinical setting is often less than the ratio recommended by the manufacturers and there is a higher tendency to over-use liquid than powder (11, 22). Moreover, considering the limited working time of GIs, most dentists do not mix proper amounts of powder and liquid and do not respect the manufacturers' recommended ratios (22, 23). Behr, *et al.* in 2008 reported ±7% difference from the ratio recommended by the manufacturer (11). This rate was reported to be ±27% in a study by Billington, *et al.* in 1990 (22). Based on the range of changes reported in the aforementioned two studies, in the current study we evaluated the effect of change in ratio by 20% on the amount of released fluoride. To assess fluoride release, previous studies have used continuous and static methods (24). Langenbucher in 1969 stated that although the static method is easier and cheaper, it has numerous shortcomings such as the dependence of the amount of released fluoride on the dimensions of specimens, liquid volume, position of specimens in the liquid, speed of stirring the liquid during measurement, the need to maintain a constant volume of liquid, and increased concentration of fluoride ions over time because the solution may become saturated with fluoride ions and the process of fluoride release is then ceased (25). Moreover, Tingstad and Riegelman in 1970 reported some other drawbacks such as lack of homogeneity in large amounts of solution, different methods of stirring and presence of a concentration gradient when dissolving the specimen using the static method, which are different from in vivo conditions (26). In the static method, the obtained information does not indicate details of the dissolution process. Another difference of the static method with the continuous flow is that when the fluoride concentration reaches a specific level, due to the recharge property of GIs glass particles start to re-uptake the fluoride ions from the solution (26, 27). The difference in fluoride concentration at days 14, 15, 29, 30, 59, 60, 89 and 90 in the current study also demonstrated the recharge pattern of this cement. To decrease the drawbacks of static method, since continuous method could not be used in the current study, modified static technique was used. To maintain a constant volume of the liquid, screw-top containers were used. These containers were made of plastic in order not to react with the released fluoride ions. Small 5mL containers were used and in order to prevent saturation, after each time of measurement, the liquid in the container was replaced with fresh distilled water. This replacement was done daily due to the high release of fluoride in the first week and after that every two weeks and then monthly. To measure the amount of fluoride ions released from the specimens at 15, 30, 60 and 90 days and eliminate the effect of GI recharge pattern (due to immersion of GI specimens in the solution containing fluoride ions released in previous days), the distilled water in containers (with specimens) was replaced after measurement of fluoride concentration. The recharged fluoride would then release into the solution within 24 hours after the cumulative measurement. Thus, in the next measurement time point (i.e. at 15, 30, 60 and 90 days), small amounts of recharged fluoride, now released into the distilled water, would be measured.

Since fluoride release creates a concentration gradient around the specimens (26), the solution may become saturated and the released fluoride ions may deposit on the surface of specimens. Thus, in order to measure the released ions deposited on the specimen surface, the specimens were rinsed with 1mL of distilled water prior to measurement.

The pattern of fluoride release from the three understudy materials with different ratios of P/L indicated a fluoride release pattern similar to what was reported by Lin, *et al.* in 2008 and Luo, *et al.* in 2009 (28, 29). The highest amount of fluoride released in the first 24 hours and then the rate of fluoride release gradually decreased from day 7 to day 13. Then, after a while, the rate of fluoride release reached a plateau. Initial fluoride release burst may be due to the loss of fluoride due to its relatively weak bond following early exposure to water during polymerization. Long-term fluoride release, however, is attributed to the gradual release of fluoride ions from the cross-linked cement (28). On the other hand, comparison of diagrams in the current study, similar to that of Vermeersch, *et al.* in 2001, showed that despite a similar pattern, rate of fluoride release from different materials was not equal in different P/L ratios of GI cements (30). The results of the current study indicated that Fuji IX had the highest and Fuji II LC had the lowest fluoride release and the difference in this regard between Fuji II LC and the other two GIs was statistically significant. However, the difference between Fuji IX and Fuji II was not significant (p=0.589). The results of a study by Robertello, *et al.* in 1999 revealed that RMGIs released the same amount of fluoride as conventional GIs (31). However, Vermeersch, *et al.* in 2001 demonstrated that RMGIs released less fluoride than conventional GIs (30). Fluoride release depends on the formation of fluoridated compounds and their interaction with polyacrylic acid as well as the amount and type of resin used for the photochemical polymerization reaction (23, 29). This can explain the controversy among different studies.

Based on the results of the current study, changing the P/L ratio by 20% (compared to the ratio recommended by the manufacturer) did not cause a statistically significant change in fluoride release pattern from the materials. Muzynski, *et al.* in 1988 compared the amount of fluoride released from Fuji type 1 and Ketac-Cem and reported that the lower the P/Lratio of GIs, the higher the release of fluoride (19); this may be explained by the fact that Fuji has one-third of the P/L ratio of Ketac-Cem and the fluoride released from it was higher than that of Ketac-Cem. Similar results were reported by Perrin, *et al.* in 1994 in their one-year study (16). Our results were in accord with those of other studies indicating that decreasing the P/L ratio increased the amount of released fluoride. However, this increase was not significant. Decrease in fluoride release following an increase in P/L ratio can be primarily due to the quick formation of calcium salts and cross-links (19). On the other hand, by increasing the liquid/powder ratio, solubility of cements increases and consequently, the cement and its constituents, including the fluoride ions, dissolve faster (18-20).

Conclusion:

The results showed that the release of fluoride from GIs was time-dependent and decreased over-time. Also, the amount of fluoride released from Fuji II LC GI was less than that from Fuji II and Fuji IX and this difference was statistically significant. Change in P/L mixing ratio of GI cements by 20% had no significant effect on fluoride release in different groups.

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