

Fluoride release, weight loss and erosive wear of modern aesthetic restoratives

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Objective In this investigation, the *in vitro* sustained fluoride release, weight loss and erosive wear of three conventional glass ionomer cements (Fuji IX, ChemFil Superior, Ketac-Silver), three resin-modified glass ionomer cements (Fuji II LC, Vitremer, Photac-Fil), a polyacid-modified resin composite (Dyract), and a resin composite control material (Z100) were compared.

Methods The amounts of fluoride released and weight changes were measured for 12 weeks using a fluoride electrode with TISAB III buffer. After 12 weeks, the specimens were recharged with fluoride using 2 mL of 1.23% APF gel. The recharged specimens were assessed for the amounts of fluoride released and weight changes over another 12 weeks. At the end of the experiment, the specimens were examined with SEM and surface profilometry.

Results All materials, with the exception of Z100, showed the highest initial fluoride release rates during the first 2 days, dropping quickly over 2 weeks and becoming largely stabilised after 5 weeks, in an exponential mode. The recharging of the specimens with APF gel caused a large increase in the amounts of fluoride released during the first 2 days only. Analyses for all cements showed strong correlations between mean weight loss and cumulative fluoride release over a 5-week period following the application of the APF gel. SEM and surface profilometry found that roughness increased from the polyacid-modified resin composite to the conventional glass ionomer cements.

Conclusions APF gel caused erosive wear of the glass ionomer cements especially, and the wear correlated well with the weight losses. To minimise surface erosion, APF gel should not be used on these cements, especially as the recharging effects are transitory.

Glass polyalkenoate (ionomer) cement is a water-based restorative material consisting of an ion-leachable glass powder and a poly(alkenoic acid) which react together to form a cement mass.¹ The original cement was developed by Wilson and Kent² and has undergone continuous development, improvement, and diversification. It now plays an important role in clinical dentistry.

Fluoride released from the cement is taken up by the tooth structure,^{3,4} and a reduced caries experience has been observed in clinical practice.⁵⁻⁸ The exhausted glass ionomer cement (GIC) can be recharged with fluoride application,^{9,10} potentially to maintain its cariostatic capability.

Despite these desirable properties, disadvantages such as early water sensitivity, poor strength and occlusal wear resistance have

limited the scope of conventional GICs in clinical application. More recently-introduced resin ionomers have less water sensitivity, and improved aesthetic and mechanical properties.¹¹ However, there are great differences between various materials in the levels of fluoride released,¹² and there is also less fluoride released when artificial¹³ and human saliva¹⁴ are used, rather than deionised water. Some materials claimed by their manufacturers to release fluoride hardly do so in measurable quantities.¹⁵ In fact, a variety of such fluoride-releasing materials are primarily resin-composite systems with some form of fluoride incorporated into the resin matrix.¹⁶ Most of these materials have a lower level of fluoride release than GICs, and their clinical effectiveness is also unknown.

Therefore, the objectives of this study were to investigate the long-term levels of fluoride released from selected GICs and resin ionomers, and the effects of acidulated phosphate fluoride (APF) gel on the levels of fluoride released and on the physical structure of the glass ionomer cements, by monitoring their weights and surface changes.

Materials and methods

Restorative materials

The details of the materials investigated in the present study are listed in Table 1. ChemFil Superior was the positive control and Z100 was the negative control.

Specimen preparation and immersion

Five specimens of each material were prepared according to the manufacturers' instructions and placed into disposable, cylindrical Teflon moulds (3.0 mm diameter × 2.7 mm height),¹⁷ and then pressed between two Mylar-covered glass slides. The resin ionomer cements were light cured from both ends of the moulds for 40 seconds using a VCL 200 visible light unit (Demetron Research Corporation, Danbury, CT, USA). They were allowed to set for about an hour. After setting, each specimen was removed from its mould, weighed by an electronic balance, then placed in a polypropylene vial with 2 mL of artificial saliva (0.05 M acetate buffer with 2.2 mM CaHPO₄ adjusted with glacial acetic acid to pH 5.0) and stored at 37°C. The solution was replaced at 6 hours, 1 day and 2 days, then weekly for 12 weeks. The amount of fluoride released was also measured at these same times for the first 5 weeks, then at 8 weeks and 12 weeks. Each specimen was dabbed dry before weighing at the times of solution replacement.

Determination of fluoride

One mL of the solution was mixed with 0.1 mL of TISAB III solution and the fluoride concentration was measured with a specific fluoride electrode (Orion 9609BN electrode: Orion Research Incorporated, Boston, MA, USA) and read in millivolts. Calibration of the fluoride electrode was done before each measurement session

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REFEREED PAPER

Received 20.07.98; accepted 21.05.99

© British Dental Journal 1999; 187: 265-270

using standard fluoride solutions (Orion Research Incorporated) containing 0.05, 0.1, 0.5, 1.0, 5.0, 10 and 100 ppm fluoride. The fluoride concentration was converted to ppm by the computer software *PLOT* written by the Oral Biology Unit, Faculty of Dentistry, The University of Hong Kong.

Fluoride recharge with APF gel

After 12 weeks, the specimens were recharged with fluoride using 2 mL of 1.23% APF gel (John O. Butler Company, Chicago, IL, USA) left in place for 4 minutes. The specimens were then rinsed and sprayed gently (to avoid any surface damage) with deionized water to remove any visible remnants of gel. Each specimen was placed again in a polypropylene vial with 2 mL of artificial saliva and stored at 37°C. The solutions were replaced using the same time schedule as before and the recharged specimens were assessed, also at the same time intervals as previously, for the amounts of fluoride released over another 12 weeks. Their weight changes were also recorded at the times of solution replacement. No precipitates were observed in the vials at any time.

Surface examination with scanning electron microscopy

Three test specimens were randomly selected and one new control specimen was prepared as before for each material. The specimens were mounted on aluminium stubs and sputter coated with gold, then examined using a JEOL 840A Scanning Electron Microscope (JEOL Limited, Tokyo, Japan) with an acceleration voltage of 10 kV. Photomicrographs at $\times 400$ and $\times 1000$ magnification were taken.

Surface profilometry study

One new control specimen for each material was prepared as before. Average surface roughness (R_a) values of the control and the two remaining test specimens of each material were measured with the Taysurf 10 (Rank Taylor Hobson, Leicester, UK) Surface Roughness Tester. Two graphical print-outs and digital readings of the mean roughness average R_a (along a length of about 0.8 mm each) were made across the diameter of the specimen surface, and

then another two measurements were made perpendicular to the first run. The procedures were repeated for the opposite surface of the specimen. Therefore, a total of eight measurements was made for each specimen.

Statistical analysis

The raw data were input by using *Excel 5.0* for *Windows 3.1* (Microsoft Corporation, Redmont, WA, USA). Any differences between the fluoride release from the materials for each time interval were assessed using one-way ANOVA, while any changes over time (including weight loss after APF gel application) for each material were assessed using repeated measures ANOVA, using the software *InStat 2.0* (GraphPad Software Inc, San Diego, CA, USA). Tukey-Kramer post-tests were used. The Kruskal-Wallis nonparametric ANOVA test was used to compare the surface roughness among the materials. Welch's approximate *t*-test was used to evaluate any differences in surface roughness for each material, before and after the experiment. Linear regression and correlation (r^2) were performed for cumulative fluoride release and weight changes following APF gel application.

Results

Fluoride release before APF gel application

The cumulative fluoride release from the materials before APF gel application is shown in figure 1. All materials, with the exception of Z100, showed the highest fluoride release rates during the first two days, dropping quickly over 2 weeks, and becoming largely stabilized after 5 weeks, in an exponential mode. Z100 remained stable throughout the experimental period at a level of about 0.02–0.03 ppm fluoride.

ANOVA comparing the materials over 12 weeks revealed broadly different amounts of fluoride release. Photac-Fil released the highest amount of fluoride, followed by Vitremer throughout the 12 weeks. The differences between Photac-Fil and Vitremer were statistically significant throughout this time ($P < 0.01$). The rate of fluoride release from Photac-Fil had not stabilised at

Table 1 Details of the restorative materials used in the present study

Material	Type	Manufacturer	Shade	Batch no.	Expiry date
ChemFil Superior (CS)	Hand-mixed	DeTrey/Dentsply, Konstanz, Germany	LY(4)	940316	96/11
Dyract (D)	Compule	DeTrey/Dentsply, Konstanz, Germany	B3	940375	95/08
Fuji IX (FJ IX)	Hand-mixed	GC International Corp., Tokyo, Japan	N/A	110341	97/03
Fuji II LC (FJ II)	Hand-mixed	GC International Corp, Tokyo, Japan	B3	P: 060122 L:071011	Not available
Ketac-Silver (KS)	Capsule	ESPE GmbH, Seefeld, Germany	N/A	413/02	96/03
Photac-Fil (PF)	Capsule	ESPE GmbH, Seefeld, Germany	A3.5	0013	95/10
Vitremer (V)	Hand-mixed	3M Dental Products, St Paul, MN, USA	B3	P:19940922 L:19940725	97/09 97/07
Z100 (Z)	Syringe	3M Dental Products, St Paul, MN, USA	B3	19931119	96/11

Fig. 1 Cumulative fluoride release from restorative materials

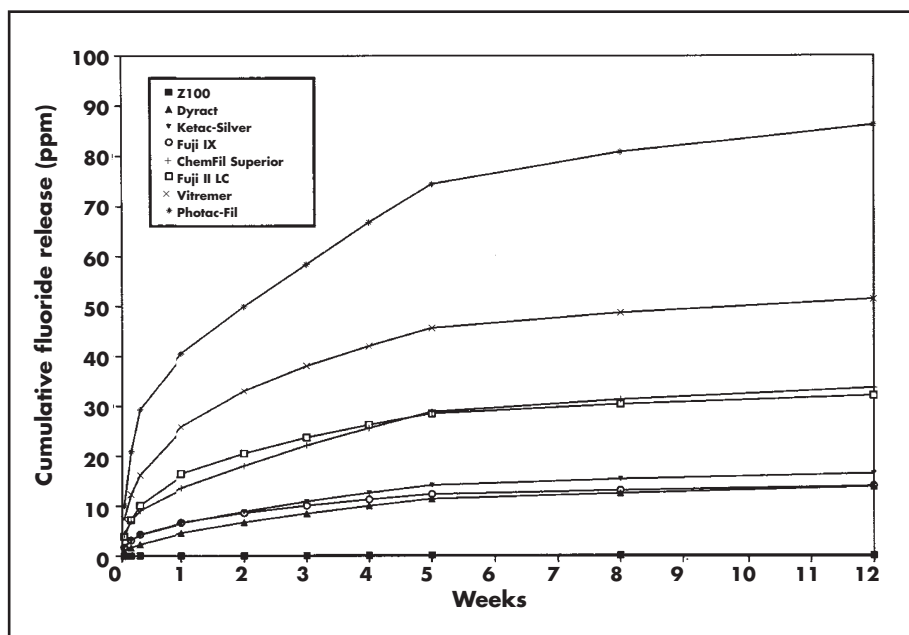
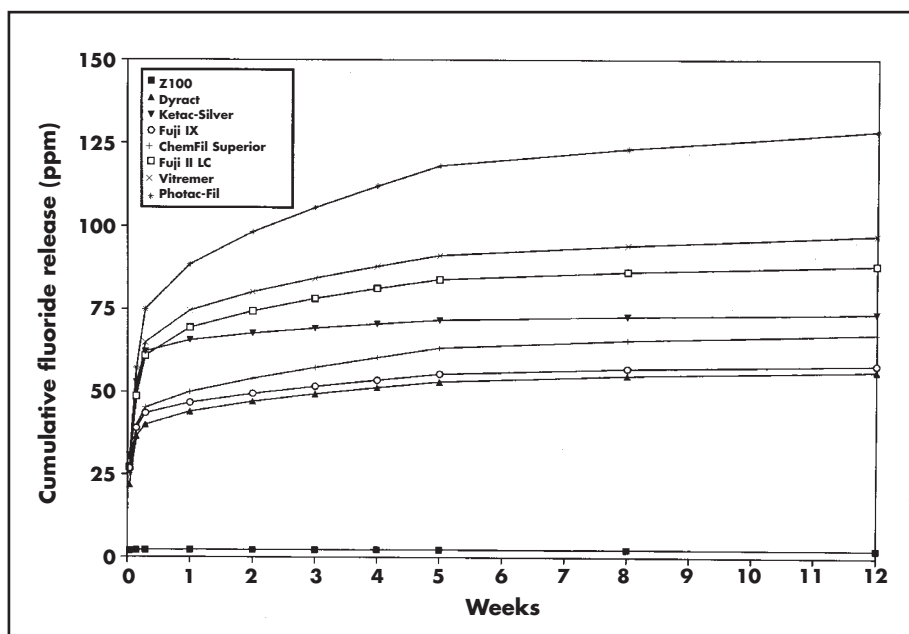


Fig. 2 Cumulative fluoride release from restorative materials after APF gel application



12 weeks, and continued to decline. Fuji II LC and ChemFil Superior released similar, moderate amounts of fluoride ($P > 0.01$). Dyract, Ketac-Silver and Fuji IX also released similar, but the lowest amounts of fluoride ($P > 0.01$).

Fluoride release after APF gel application

The application of APF gel caused a large increase in the amounts of fluoride released, as shown in figure 2. The highest rates were again the first 2 days, but the rate dropped very quickly to become largely stabilised after 2 weeks, in an exponential mode. The amounts of fluoride released dropped back to the levels present before APF gel application, at about 8 weeks.

Initially, at 6 hours, Photac-Fil released about six times the amount of fluoride that was released before APF gel application. Vitremer released about nine times, but was not statistically different from Fuji II LC and ChemFil Superior until after 8 weeks. At 6 hours, Fuji II LC released about 14 times the fluoride released before APF gel application, and ChemFil Superior about 11 times. Dyract released about 16 times, and Fuji IX and Ketac-Silver released about 30 times the initial fluoride released at 6 hours. Ketac-Silver was affected the most by the

APF gel. The mean cumulative fluoride release at 12 weeks before and after APF gel application is presented in figure 3.

Relationship between weight loss and fluoride release from glass-ionomer cements

Linear regression and correlation between the weight loss and the cumulative fluoride release over a 5-week period following APF gel application were assessed for each material. The correlations were very good with coefficients of determination (r^2) ranging from 0.80 to 0.97 (Table 2).

Surface roughness before the experiment

There were differences in the surface roughness of the newly-made specimens among the restorative materials before the experiment started. There was a trend of increasing roughness from pure resin composite, to resin ionomer cements, to conventional GICs. The surfaces of Dyract (figs 4a,b) and Z100 were the smoothest. However, there was no statistically significant difference between Z100 and the resin ionomer cements ($P > 0.05$). The three conventional GICs had the roughest surfaces.

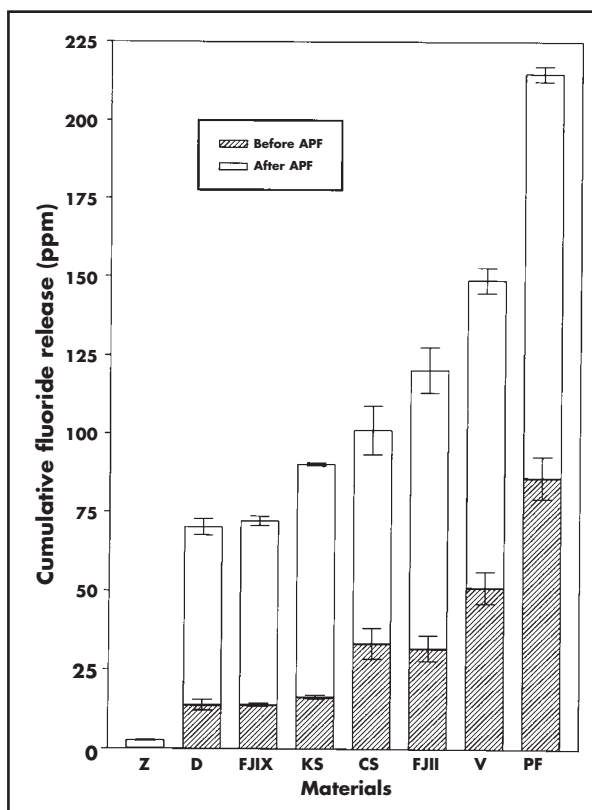


Fig. 3 Stacked bar charts to show the cumulative fluoride release at 12 weeks before and at 12 weeks after APF gel application (mean ± standard deviation)

Surface roughness after the experiment

All specimens showed increased surface roughness after their immersion and storage in artificial saliva and APF gel application. The general trend was similar despite the rearrangement of the order of increasing roughness for different materials (Table 3). The differences between the mean roughness average Ra, for the resin ionomer cements (except Photac-Fil) and Z100 were not statistically significant ($P > 0.05$). The roughness of Photac-Fil was similar to that of the conventional GIC, Fuji IX (figs 5a,b). Again, the conventional GICs formed the rougher surface group.

Discussion

Fluoride release from GICs is diffusion-limited and affected by the concentrations in both the cement matrix and the particles. During the initial acid dissolution of powder-particle surfaces, a large amount of fluoride becomes part of the reaction-product matrix. This fluoride diffuses quickly from the matrix exposed on the surface of the material and is only slowly replaced by fluoride diffusing over greater distances from the matrix below the surface, or by fluoride diffusing from the particles into the matrix for the first time.¹⁸

Kuhn and Wilson¹⁹ stated that the dissolution of GICs and silicate cements included three steps: (1) surface wash-off, (2) diffusion from the solid state, and (3) surface corrosion. Fluoride release can also occur partly by diffusion through pores and cracks.¹⁹ Causton claimed that the fluoride release would cease after a few months.²⁰ Probably, he overlooked the possibility of diffusion of fluoride ions from the solid state. All other studies support the long-term fluoride release from GIC.^{21,22} Forsten reported that fluoride was still released from 8-year old GIC specimens.⁷ Therefore, initially there is a high burst of fluoride release, and the long-term release of fluoride is at much lower rates.

The relatively small 2 ml volume of artificial saliva used in this study may be a problem if the solution is not changed frequently.

Table 2 Results of linear regression between weight loss and the cumulative fluoride release from the restorative materials for the first 5 weeks after APF gel application

Material	Slope (ppm/0.8 mg)(±SD)	r ²	r	P-value
Dyract	92 (8.7)	0.95	0.97	< 0.0001
Ketac-Silver	60 (6.6)	0.93	0.96	< 0.0001
Fuji IX	98 (20)	0.80	0.89	0.0028
ChemFil Superior	79 (6.9)	0.96	0.98	< 0.0001
Fuji II LC	120 (9.9)	0.96	0.98	< 0.0001
Vitremer	150 (23)	0.88	0.93	0.0005
Photac-Fil	140 (9.3)	0.97	0.98	< 0.0001

r² = coefficient of determination, r = correlation coefficient

Table 3 Mean roughness values (R_a) for materials before, and after storage and APF gel application

Material	Before		After	
	R _a /μm	95%CI/μm	R _a /μm	95%CI/μm
Z100	0.02	0.00–0.04	0.03	0.01–0.08
Fuji II LC	0.06	0.04–0.08	0.27	0.20–0.36
Vitremer	0.09	0.06–0.12	0.35	0.21–0.49
Dyract	0.02	0.00–0.04	0.40	0.30–0.51
Photac-Fil	0.04	0.02–0.07	0.86	0.71–1.01
Ketac-Silver	0.12	0.08–0.16	1.03	0.89–1.17
Fuji IX	0.16	0.04–0.08	0.95	0.69–1.21
ChemFil Superior	0.22	0.09–0.35	1.05	0.86–1.24

CI = confidence interval

Therefore, the solution was changed at the initial 6 hours, 1 day and 2 day periods when fluoride release was abundant. As the experiment progressed, the dwell time was longer, because the amount of fluoride release was much less. Therefore, the 2 ml volume of artificial saliva was considered acceptable for use in this study.

While Wandera *et al.* have discussed the merits of using different units of fluoride measurement namely, weight of fluoride released

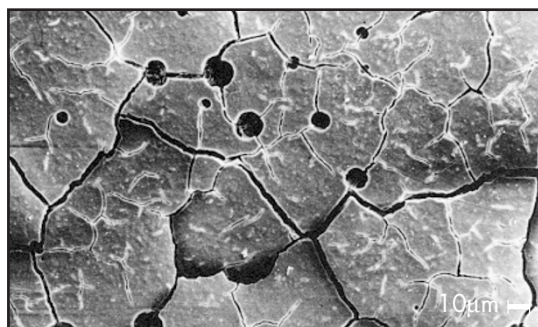


Fig. 4a SEM of Dyract before the study. The surface is smooth (the crazing is an artefact)

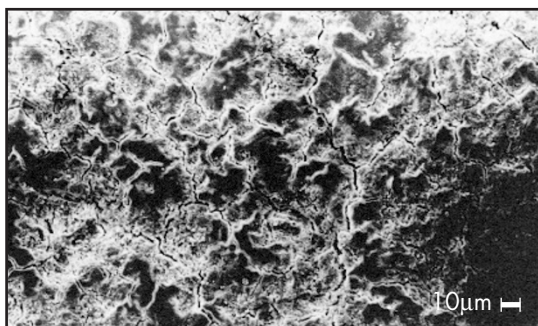


Fig. 4b SEM of Dyract after the study. Note the slightly more pronounced filler particles

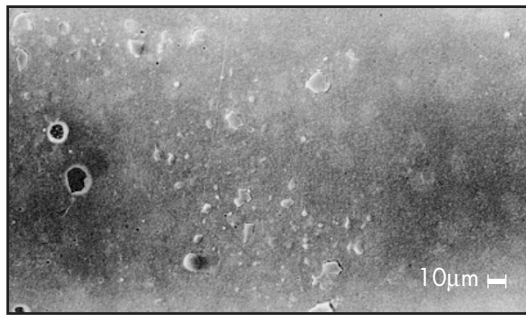


Fig. 5a SEM of Fuji IX before the study. Some surface roughness is evident

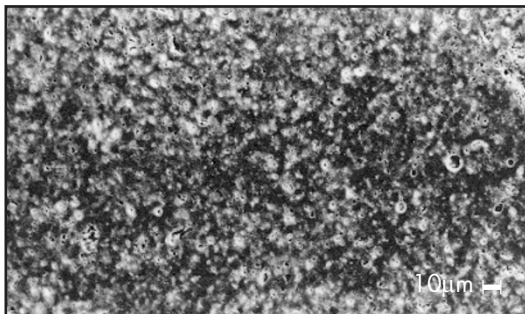


Fig. 5b SEM of Fuji IX after the study. Note the considerably roughened surface

into a volume of solution from a volume of material, weight of fluoride released from a weight of material, or weight of fluoride released from a surface area of material, no conclusion was drawn for the best unit for use.²³ To compare the present data with the results from other papers^{24–28} involving a similar range of materials, the same unit (ppm) was chosen for comparison and discussion. The treatment of data by Rothwell *et al.*²⁹ using mg/g for fluoride release versus the square root of time, assumes that fluoride release will be completely exhausted at infinity, and has gradually gained acceptance for recent publications of fluoride release. Future studies and reviews may favour the use of mg/g.

Topical application of fluoride gel is an established procedure in dentistry for the prevention of caries. Four minutes of professionally-applied APF gel in a tray is recommended for better uptake of fluoride into the enamel.³⁰ It is also recommended that patients of any age with active caries should receive this regime four times per year, plus daily use of a fluoride-containing dentifrice.³¹

In the present study, application of APF gel caused a tremendous increase in fluoride release from all materials. It is interesting to note that the conventional GICs released 30 times the amounts that were measured just before the APF gel application. Resin ionomer cements released about six times this amount, which suggested that conventional GICs were affected relatively more by the acid gel. This trend is in accord with the findings of Hotz and co-authors.³²

Taggart and Pearson pointed out that acid gels were able to penetrate into the depths of GIC,³³ and it was unlikely that the surface washing would eliminate all of the gel. Therefore, it was likely that the effects of the gel would continue until neutralised, but the damage to the cement in bulk might be extensive. This was probably reflected by the significant weight losses found during the first week after the APF gel application. Thereafter, the fluoride release profiles quickly returned to the levels of the pre-APF gel application. Thus, the action of the APF gel was short-lived and the suggestion of long-term rechargeability³⁴ is challenged in the present and in other *in vitro* studies.^{13,35}

APF gel contains hydrofluoric acid and phosphoric acid.¹³ Phosphoric acid has the ability to etch glass particles.³⁶ Hydrofluoric acid

is more destructive than phosphoric acid because it can etch glass at lower temperatures.³⁷ The acidic pH (5) affects the chemical erosion of the cement by acid-etching the surface and leaching the principle matrix-forming cations (Na, Ca, Al, Sr), and will also increase its fluoride release.²⁵ El-Badrawy and co-authors¹³ suggested that phosphoric acid was capable of forming stable complexes with metal ions in the ionomer, resulting in greater surface erosion. Diaz-Arnold and co-authors²⁵ found that APF gel caused the greatest amount of fluoride release followed by neutral sodium fluoride gel. Stannous fluoride gel was similar to deionised water in its effects.

The weights of the specimens slowly and gradually decreased. The significant weight loss occurring after APF gel application found in this study suggested indirectly that the APF gel had caused surface erosion and dissolution of the specimens, releasing a great amount of fluoride. Linear regression and correlation between the weight loss and the cumulative fluoride release after APF gel application showed very good correlations for all cements (r^2 ranging from 0.80 to 0.97). Assuming that the weight loss was associated with surface erosion, then the increase in fluoride release was at the expense of the cement structure. Smith showed that the GIC surface integrity was essentially destroyed after 1 minute of phosphoric acid etching, and that individual particles dissociated from each other as the gel matrix dissolved.³³ Neuman and Garcia-Godoy also showed that glass particles were left protruding from the cement surface after APF gel application.³⁴ The specimens in the present study were placed in artificial saliva for a further 12 weeks after APF gel application and any loose particles present were probably dislodged. Therefore, the SEM examination showed very rough surfaces with voids present, and individual glass particles protruding.

El-Badrawy and McComb showed that APF gel had the most deleterious effect on all of the GICs examined.³⁹ The matrix of the resin ionomers was generally more resistant to erosion than the conventional GICs, but they concluded that the resin ionomers did not provide significant improved resistance to APF gel. By contrast, the three resin-modified glass ionomers and the polyacid-modified composite resin (Dyract) tested in this study showed more fluoride release with less weight changes. This finding supports the improved resistance by the resin-modified glass ionomers to surface erosion as proposed by Sidhu and Watson.⁴⁰

For the fresh specimens, there was a trend of increasing roughness from pure resin composite, polyacid-modified resin composite, and resin-modified glass ionomers to conventional GICs. After APF gel application, the general trend of roughness of the restorative materials tested was still valid. Uno found that there were differences in the times needed to reach a maximum level of strength for the resin-modified glass ionomers, and suggested that the reasons might be the maturation of the acid-base reaction.⁴¹ Despite the improvement in the surface resistance to erosion, stresses could build up in the glass particles-resin matrix interfaces, and early immersion into artificial saliva and subsequent APF gel application may help to propagate any cracks.

Conclusions

- The selected glass ionomer and resin ionomer cements showed the highest fluoride release rate during the first 2 days, dropping quickly over 2 weeks, and became stabilised after 5 weeks, in an exponential way.
- The 12-week cumulative fluoride release showed the following order of decreasing amounts of fluoride release; resin-modified glass ionomers, conventional GICs, polyacid-modified resin composite, and resin composite.
- Application of acidulated phosphate fluoride (APF) gel for 4 minutes caused a tremendous increase in the subsequent amounts of fluoride release during the initial 2 days, but the rate declined very rapidly to become largely stabilised after 2 weeks, again in an exponential mode.

- The application of APF gel caused a significant weight loss from the glass ionomer cement specimens during the initial first week.
- Follow-up studies using surface profilometry and scanning electron microscopy confirmed the erosive effect of APF gel on the conventional GICs especially. The general trend of increasing surface roughness was of resin composite and polyacid-modified resin composite, resin-modified glass ionomers, and conventional GICs. This trend was more obvious after APF gel application.
- There were very good correlations (r^2 ranging from 0.80 to 0.97) between the weight loss of all cements and their 5-week, post-APF gel cumulative fluoride release.

Findings in this study were presented at the 1st European Union Conference on Glass Ionomers, Warwick, UK, 1996. This study was supported by CRCG award 337/252/0005, The University of Hong Kong.

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