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The Early Fluoride Release Pattern of an Aged Glass Ionomer Treated With Fluoride

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Abstract - *The aim of this study was to examine the pattern of early fluoride release from aged glass-ionomer cement after treatment with a 1,000 mg/L NaF solution using hourly and daily time point and to investigate whether surface profile affected release. Composite resin was used as a control. The aged glass-ionomer discs showed a 15 fold greater rate of fluoride release during 1st hour post-treatment, equivalent to the original fluoride release value of the newly mixed material. This rate of release dropped sharply to 3 fold greater than baseline during the 2nd hour. Composite resin showed negligible levels of release. No significant differences were found between smooth and rough surfaced groups.*

KEY WORDS: composite, fluoride release, glass ionomer, fluoride treatment, surface profile

INTRODUCTION

Fluoride (F) release of restorative, sealing, bonding and cementing dental materials and their alleged anti-caries effect on cavity walls or other tooth surfaces they are in contact with has been the subject of a considerable number of studies[1]. Glass-ionomer cements (GIC), either conventional or resin modified, are the usual subjects of investigation in this respect. This is due to their superior fluoride release compared to other materials where the added fluoride is less important than the intrinsic fluoride content of GIC[2].

There is general agreement about a dramatic drop in fluoride release following GIC mixing, but the rapidity of this decline in F release varies with the experimental methods used[3,4]. From in vitro and in vivo experiments it appears that fluoride release does not extend much beyond a day and is often referred to as 'burst' effect[4,5]. Although further fluoride release may continue for years, it is extremely small and most probably clinically insignificant[6]. In vitro studies have shown that the capacity to release significant amounts of fluoride can be restored by daily reloading the material with fluoride from various other sources, like a dentifrice or mouthwash, a process often referred to as 'recharging'[7,8].

GICs seem superior to other materials such as conventional and poly-acid modified composites in releasing not only intrinsic but also recharged fluoride[4,8]. The reason for the release of the latter has been partially attributed to higher GIC permeability possibly leading to greater penetration of fluoride during the recharging process[8]. Composites are known to have relatively low water sorption as a result of low permeability. GIC permeability is known to be substantial, but its long-term significance is difficult to determine because it decreases with maturation[9]. Surface profile characteristics may also be important if recharged F is partially bonded to the material surface. Use of a scanning electron microscope (SEM) shows GIC material, either sound or previously attacked by lactic acid, to have a rather rough surface[10], greatly increasing the surface area exposed to fluoride solutions. The role of surface profile in the release pattern of recharged F has not been previously reported.

Fluoride treatment can be effectively repeated several times[2]. The amount and duration of release of recharged fluoride is affected by several factors such as frequency of application[11] and the degree of fluoride concentration of the 'recharging' medium[7,8]. Fluoride treating a resin modified GIC in static solutions with a low pH dentifrice, in vitro, showed evidence of fluoride release for 2 or more days, while use of a neutral pH fluoride dentifrice resulted in no significant release beyond 24 hours[12]. In order to adequately study the pattern of fluoride release, determinations should be performed frequently, at least during the first 24 hours, especially when using a neutral fluoride medium, such as most products marketed for home use and those used in the majority of past in vitro studies. Unlike other relevant studies, which examined release every 24 hours in static solutions[7,9,11,12], Hsu et al[4] employed this model of frequent fluoride determinations in an in vitro continuous flow system. They showed the steep drop in the release of recharged fluoride occurred within the first 20 min of GIC treatment. The 24h exposure time interval also seems unrealistically long when compared to oral fluoride clearance, as salivary fluoride levels have been found to return to baseline within 2 hours after a fluoride mouth rinse[13].

Our study design employed both hourly and daily fluoride determinations in static solutions to allow comparisons with both types of past studies. The aim was to examine: (a) the initial phase of early fluoride release pattern in a

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GIC fluoride treatment model and (b) the influence of surface profile.

MATERIALS AND METHODS

A total of 24 disc specimens, 18 of Ketac-Fil and 6 of Z-250, were divided into four experimental groups of 6 specimens (I, II, III, IV). Ketac-Fil was selected as a conventional encapsulated GIC in which mechanical mixing would ensure specimen consistency and repeatability (Table 1).

Preparation of the disc specimens

Following pilot experiments, all discs were prepared by placing the material into identical cylindrical Teflon moulds (interior diameter 5mm, height 2mm). The mould was placed between glass slides covered with an adhesive tape strip for easy removal of the material and the slides were pressed to ensure uniform, consistent discs[8]. Capsules were mixed in a mixing unit for 10sec and were allowed to harden in the mould for 30 min. They were removed from the moulds to completely cure for 24 hours under specified conditions (37^oC and 85% relative humidity)[9]. The discs produced had 31.42 mm^2 of curved surface, and a slightly larger area of flat surfaces (39.27 mm^2). The total area of 70.69 mm^2 was used for expressing fluoride release per surface unit of disc.

Group I: six Ketac-Fil discs.

The surfaces of these discs were left smooth. The specimens were stored for 30 days in a beaker of 500ml de-ionized water, renewed every week, for 'aging' and to exhaust the release of intrinsic fluoride.

Group II: six Ketac-Fil discs.

Both flat surfaces were roughened by 20 circular strokes using sandpaper (grit P800). Roughness was verified by loss of shine as observed by a stereomicroscope (10X mgn). This group was then aged in an identical manner to group I. The difference in flat surface textures between groups I and II was further verified post experimentally by examination with a Scanning Electron Microscope (SEM) equipped with an energy dispersive spectrometer.

Group III: six Keta-Fil discs.

These discs were freshly prepared at the conclusion of the 30-day aging period for groups I and II and left smooth.

Group IV: six composite resin discs (Z-250).

These were freshly prepared at the same time as group III and photo-polymerised for 40 sec on each side.

Fluoride treatment and measurement of Fluoride release

26 determinations were carried out for each group over 9 days. Three determinations were made in each of the nine 24h periods of the experiment. These were at hours 1, 2, and 22 hours later, at hour 24. There was no 22-hour fluoride release determination on the last day (fig 1). Fluoride treatment was performed twice, on days 3 and 6, in both cases after the completion of the respective 22-hour measurement. The 1^{st} fluoride-treatment was performed when the GIC specimens had aged, at that point, for 33 days (groups I, II) and on the composite discs (group IV). Group III (newly mixed Ketac-Fil discs) were not treated, as they were first allowed to approach a steady state release; this was deemed to have been reached when constant low release readings were obtained for 4 successive measurements of hourly fluoride release. This occurred after 6 days, making this group (III) 6-days aged GIC. The 2^{nd} fluoride-treatment was performed on all groups to confirm the repeatability of the results. So, at day 6, the 36-days aged GIC and the resin composite discs specimens (groups I, II and IV) were fluoride treated for the second time and the 6-days aged GIC specimens (group III) for the first time (fig 1).

Table 1. *Characteristics and preparation of the disc specimens in each group of the materials tested.*

Product	*Type of Material*	*Manufacturer, Mixing*	*Use*	*Group Preparation*
Ketac fil	Conventional glass ionomer GIC	3M-ESPE Capsulated	Test	I: 30-days aged, smooth surface II: 30-days aged, rough surface III: newly mixed,mooth surface
Z-250	Non-fluoride Resin Composite	3M-ESPE Compule	Control	IV: newly mixed, smooth surface

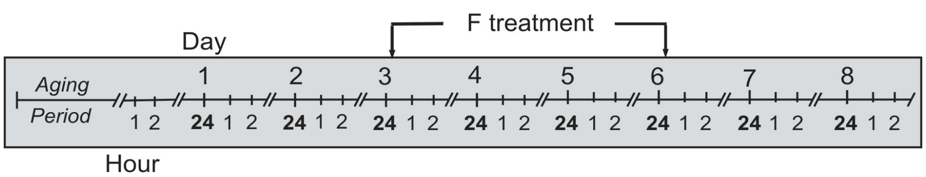


Figure 1. *Flow chart of the study. Three determinations of F-release were performed during the nine experimental days, at the completion of hours 1 and 2 (hourly) and 24 (22-hour release). There was no 22-hour release determination for day 9. There has been a 30-days aging period for groups I and II .*

Fluoride treatment was effected by immersing each disc in 1.5 ml fluoride solution (1,000 mg/L) in the form of NaF for 30sec, a condition close to clinical caries preventive procedures. Before and after the immersion the disc was rinsed with 5ml of de-ionized water to remove surface fluoride, and then stored in a polypropylene container with 1.5 ml of fresh de-ionized water. All specimens were stored in an upright position, with their curved surfaces on the container bottoms, to prevent interference with fluoride release kinetics.

Fluoride analysis

The fluoride concentration was determined using an ion-selective electrode connected to an expandable Ion-Analyzer. The electrode was calibrated with a range of fluoride standards (0.01-0.1, 0.1-1.0 or 10-100 mg/L) according to the expected fluoride concentration, after the addition of 1:10 TISAB (Total Ionic Strength Adjuster Buffer)-Aluminon, used to provide constant background ionic strength, to ionize the fluoride present in the test solutions and to adjust the pH to 5.0. The electrode slope was checked daily before performing measurements and the calibration was repeated every 6th measurement. Freshly de-ionized water was also measured every day as a control. A maximum error of 0.3% was found at re-calibrations. Room temperature was 26^{o}C.

Statistical analysis

The experimental design was a Mixed Factorial with one factor *between groups* (Groups: I, II, III) and three factors *within group* (Fluoride-treatment: before-after, Day: 1, 2, 3, Hour: 1st , 2nd). Data were analyzed according to the above mentioned experimental setting in the frame of General Linear Models, using SPSS.11.5 package[14]. The comparisons of means were performed by the Bonferroni test, at significance level a=0.05.

RESULTS

According to the statistical analysis no significant differences appeared between the fluoride release values following the 1st and 2nd fluoride treatment in groups I and II. However, since group III had been treated only during the 2nd fluoride treatment, statistics of the mean values among all three Ketac-Fil groups was based on the pre-treatment vs. after 2nd fluoride treatment data. It did not include group IV data due to the appearance of many zero values (table 2).

During the 3 pre-treatment days, all but two hourly fluoride release values from the aged Ketac-Fil groups I and II, ranged between 0.24 to 0.40 μg/cm^2. Immediately after both fluoride treatments, the 1st hour fluoride release dramatically increased to between 5.23 and 6.04 μg/cm^2,

Table 2. *Mean values and SD of F released per disk surface unit in μg/cm^2 during each of the 1st and 2nd hour periods of each experimental day, both before and after F treatments.*

Day	Hour of F release	Ketac-Fil GIC groups						Z-250 group	
		I – aged, smooth		II – aged, rough		III – new, smooth		IV – new, smooth	
1	1	0.36^{bA}	0.05	0.23^{bA}	0.02	4.66^{aA}	1.51	0.02	0.00
	2	0.36^{bA}	0.06	0.27^{bA}	0.05	4.27^{aA}	1.18	0.02	0.00
2	1	0.39^{bA}	0.03	0.28^{bA}	0.07	2.20^{aA}	0.66	0.00	0.00
	2	0.24^{bA}	0.01	0.17^{bA}	0.03	1.37^{aB}	0.24	0.00	0.00
3	1	0.59^{bA}	0.21	0.28^{bA}	0.04	1.58^{aA}	0.56	0.00	0.00
	2	0.34^{bA}	0.07	0.25^{bA}	0.03	1.01^{aB}	0.16	0.00	0.00
1st F treatment		*Yes*		*Yes*		*No*		*Yes*	
4	1	5.69	1.18	6.04	0.77	1.13	0.31	0.07	0.04
	2	1.14	0.41	1.19	0.26	0.73	0.25	0.02	0.00
5	1	0.46	0.08	0.39	0.02	0.71	0.11	0.02	0.01
	2	0.44	0.08	0.28	0.02	0.72	0.19	0.03	0.00
6	1	0.43	0.06	0.33	0.06	0.67	0.24	0.02	0.01
	2	0.27	0.03	0.20	0.02	0.42	0.17	0.02	0.00
2nd F treatment		*Yes*		*Yes*		*Yes*		*Yes*	
7	1	5.23^{aA}	1.03	5.65^{aA}	0.72	3.64^{bA}	0.80	0.06	0.03
	2	1.03^{aB}	0.16	1.09^{aB}	0.32	0.99^{aB}	0.27	0.02	0.01
8	1	0.45^{aA}	0.07	0.42^{aA}	0.06	0.57^{aA}	0.19	0.02	0.00
	2	0.46^{abA}	0.05	0.38^{bA}	0.05	0.56^{aA}	0.11	0.02	0.00
9	1	0.54^{aA}	0.19	0.44^{aA}	0.03	0.58^{aA}	0.16	0.02	0.00
	2	0.27^{abB}	0.04	0.19^{bB}	0.05	0.37^{aB}	0.10	0.02	0.00

** Capital index letters $^{(AB)}$ correspond to within group comparisons (1st vs 2nd hour). Small index letters $^{(a,b)}$ correspond to between groups comparisons (I vs II vs III). Means followed by different letters are statistically significantly different at a=0.05 according to Bonferroni test.*

Table 3. *Cumulative 24-hour values and SD of fluoride released per disk surface unit in μg/cm² both before and after the F treatments.*

	Ketac-Fil GIC groups						Z-250 group	
Day of F release	*I - aged, smooth*		*II - aged, rough*		*III - new, smooth*		*IV- new, smooth*	
1	4.57 *b*	0.37	3.12 *c*	0.28	41.94 *a*	9.04	0.05 *d*	0.00
2	3.74 *b*	0.58	2.63 *c*	0.31	23.1 *a*	5.38	0.02 *d*	0.01
3	4.15 *b*	0.35	2.72 *c*	0.43	17.00 *a*	4.08	0.02 *d*	0.00
1st F treatment	*Yes*		*Yes*		*No*		*Yes*	
4	10.15 *a*	1.74	11.16 *a*	1.32	11.96 *a*	3.51	0.11 *b*	0.05
5	3.90 *b*	0.72	3.28 *c*	0.19	10.01 *a*	1.78	0.06 *d*	0.01
6	3.27 *b*	0.62	2.77 *b*	0.43	7.95 *a*	2.07	0.06 *c*	0.01
2nd F treatment	*Yes*		*Yes*		*Yes*		*Yes*	
7	10.11 *ab*	1.76	9.82 *b*	1.31	13.22 *a*	2.78	0.10 *c*	0.03
8	3.70 *b*	0.60	3.11 *b*	0.50	7.68 *a*	1.47	0.06 *c*	0.00

** Comparison of groups: Means followed by different letters are statistically significant different at a=0.05 according to the Mann-Whitney test. The test was applied using the Monte Carlo simulation method[14].*

dropping to 1.03 up to 1.19 μg/cm² for the 2nd hour (table 2, fig 2). This represented a more than 15 fold increase in fluoride release within one hour after fluoride treatment, declining sharply to about 3 fold at the 2nd hour, highly statistically significant for both groups. The 1st post-treatment hour fluoride release of group III following its only fluoride-treatment at the end of day 6, was 3.64 μg/cm², significantly higher, too, than its own pre-treatment values, but significantly lower than the respective (day 7, hour 1) post-treatment values for groups I and II (fig 2).

By aggregating the 1, 2 and 22 hour periods fluoride release for each day (table 3) the graph presented in fig 3 was obtained, representing the 24 hour (cumulative calculation on daily base) fluoride release of all groups for comparisons with some previous studies[2,17]. Negligible fluoride release was observed following the fluoride treatment of the Z-250 control group IV. Its pattern was remarkably similar to that of the aged GIC, although actual values were nearly 100 times lower (tables 2, 3).

The SEM examination confirmed the observations made with the stereoscopic microscope, i.e. the smooth flat surfaces seen in group I and the rough flat surfaces seen in group II (fig. 4). The curved surfaces of the specimens of all groups were smooth. The energy dispersive spectrometry showed similar curves for Si, Al, and other elements present in lower concentrations, both in smooth and rough specimens.

DISCUSSION

There are a number of parameters that influence fluoride release following fluoride treatment of GICs, namely the viscosity of liquid medium i.e. water or artificial saliva[3,15], the use of a continuous fluid renewal system[4,16], the pH of the medium or the use of a pH cycling design[11], the fluoride quantity in the fluoride-treating solution[2,4] and the timing i.e. how early and how frequently fluoride is determined. Fluoride release has mostly been determined in past studies in static solutions on a 24 hour basis[6,7,9,11,17], or even less frequently[2], while one study has used some 1-hour determinations over a two year period[8]. The present study also used 1-hour fluoride determinations, for each of 9 consecutive days. Although exact comparison between these last two studies is probably best avoided due to possible batch and other minor differences in the experimental setup, the values of the present study for the same brand of material and time period are higher by a factor of 2 to 3. This is probably owed to lower kinetics due to the carboxymethylcellulose and inorganic ions content in the artificial saliva medium used by Preston et al[8].

The importance of measuring fluoride release at short time (hourly) intervals after treating the aged GIC with fluoride was underlined in the present study by the 15 fold elevated fluoride release from baseline during the 1st hour, which sharply dropped to 3 fold at the 2nd hour and subsequently to near baseline. In a continuous-flow fluoride release system, suggested as better duplicating the renewal of oral fluids[16], a study design employing even earlier fluoride determinations would be more appropriate. This was recognized by Hsu et al[4], who started determining fluoride release as early as 1 min after fluoride treatment and then every 10 min. They used such a system to test both a conventional and a resin modified GIC, with similar results. Despite the use of different brands of GIC materials, the present study showed a very similar pattern of fluoride release subsequent to fluoride treatment with that of Hsu et al[4]. The difference lay in the much, over 5 times, faster rate of release in their study as a result of continuous water renewal. This also emphasizes that the pattern of fluoride release shown by various studies is dependent on the timing of fluoride determinations.

The 1st hour release of both aged Ketac-Fil groups following fluoride treatment coupled the very 1st hour release of the newly mixed group III (fig 2). Thus, treatment with fluoride solution restored the original 'burst' fluoride release ability of GIC for one hour, supporting the similar findings of Preston et al[8]. In their menu of materials, other conventional and resin modified brands of GIC showed even better fluoride release potential than Ketac-Fil, leading them to propose that 'the choice of materials for use in the caries-prone patient should be supported by fluoride recharge/release data for the individual material'.

After the 2nd fluoride -treatment, a higher fluoride release from both 36-day aged Ketac-Fil groups was observed compared to the new 6-day aged group III (day 7, hour 1 in fig 2). The Ketac-Fil capsules were all from the same batch, and variations of specimen preparation procedures were kept to a minimum. It is difficult to explain the difference in terms of permeability since the more aged GIC specimens are expected to be less permeable, and thus less fluoride reacting, than the new GIC[9]. This finding however was in line with Preston et al[8] who found an increasing trend of release in artificial saliva following fluoride-treatment in 3 out of 4 GICs they tested over a period of two years. Therefore, aged GI restorative materials may possibly be considered as fluoride reservoirs in the case of repeated fluoride treatments and may even be superior in terms of fluoride uptake and release than recent ones.

The clinical significance of multiple use of GIC in the mouth has been demonstrated when orthodontic brackets were cemented with GIC[5] and this significantly raised salivary fluoride levels for 24 hours. Thus, a repeated fluoride treatment, i.e. a fluoride mouth rinse that can restore this level of fluoride release for short times daily may be beneficial. In contrast, the resin composite did not release any appreciable amounts of fluoride after fluoride treatment, emphatically supporting previous research findings[4,7]. If this advantage in favor of GIC is shown to occur in an intraoral or even better in a clinical experiment too, it may attract further attention in material selection for caries active paediatric patients who present multiple needs

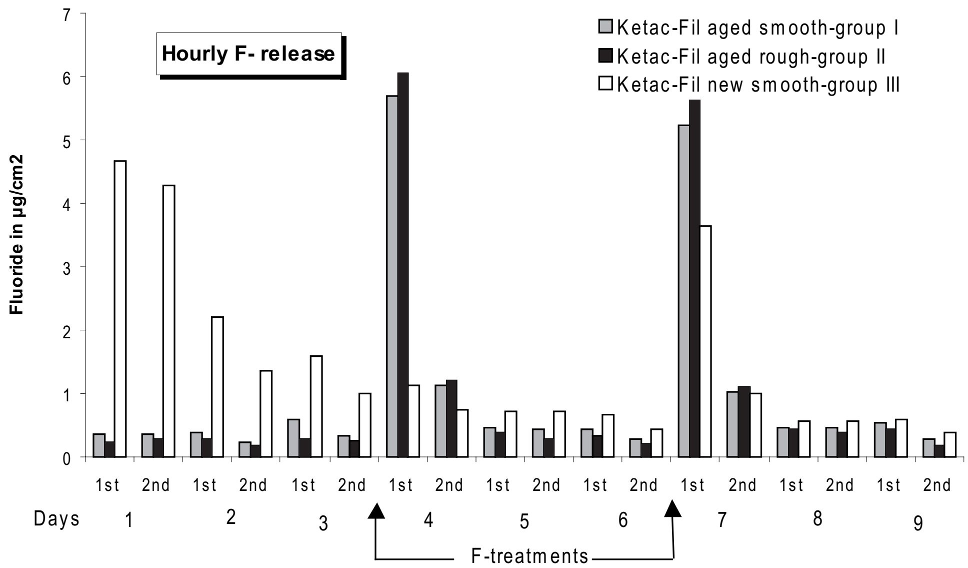


Figure 2. *The 1st and 2nd hourly F release determinations (µg/cm²) at beginning of each 9 day period. Specimens were F treated twice (groups I, II and IV) immediately after the last determination of days 3 and 6. In the case of group III, this occurred only once on day 6. Group IV is not represented due to negligible F release.*

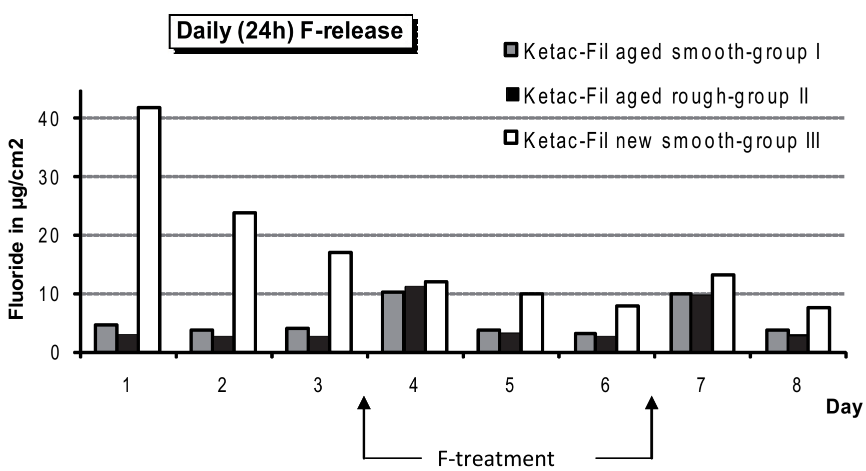
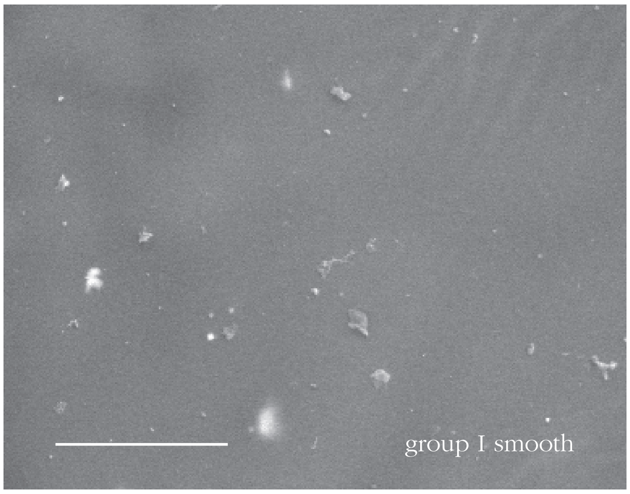


Figure 3. *Daily (24 hours) F release (µg/cm²), calculated as the total of the 3 determinations of each day. Group IV is not represented due to negligible F release.*



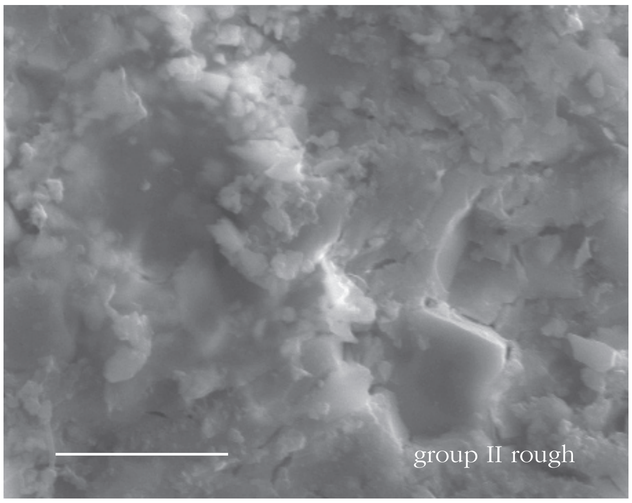


Figure 4. *Scanning electron microscopy of flat surface of smooth (group I) and rough (group II) Ketac-Fil specimens. Bars represent 10 µm.*

for restorations, since: 1) resin modified GIC behaves at least as well as conventional when fluoride treated[4,8] and its usage has been increasing in general[18]; 2) the benefit of fluoride mouth rinses, targeted as an important part of a caries preventing strategy in this population segment[19], may be enhanced by the added fluoride uptake and release of the GIC restorations.

At the first day after any fluoride treatment, the 1st hour fluoride release value was always higher than the respective 22-hour value. When the three values for each day were summated, the resulted histogram of daily (24 hour) fluoride release (fig 3) closely approached the graphs of studies employing fluoride release determinations every 24 hours[17]. In the present study, the GIC recharging potential became obscured in this manner of data presentation, because original fluoride release was only briefly – for 1 hour – restored with the fluoride treatment.

The study also addressed whether surface profile and material type influence the quantity of fluoride uptake and release. Only minor and insignificant differences were found between rough and smooth specimens of Ketac-Fil. Energy dispersive spectrometry measurements on both rough and smooth surfaced specimens showed similar curves for Si, Al and other elements, indicating low levels of contamination of the rough surfaces by exogenous debris; in other words, a smear layer effect could not be significant. The roughness observed in the SEM pictures (fig 4) resembled appearances reported by De Witte et al[20], produced by repeated neutral NaF application on GIC materials. It could not, however, have been produced by the two NaF applications in the present study, since the smooth surface specimens received identical fluoride treatment. The surface profile findings combined with the rapid loss of most reloaded fluoride within 2 hours in the present static solution study, or within 20 min in a continuous flow study[4], add support to the view that fluoride is not bound onto the surface or into the cement matrix to a significant degree[21] since it is rather quickly washed away irrespective of surface profile.

CONCLUSION

Following 30 sec treatment of a conventional, aged GIC with a 1000 mg/L Fluoride solution in vitro: 1) Fluoride release in static water was dramatically elevated for 1 hour and then declined sharply, 2) This fluoride uptake restored fluoride release to original levels, at least as effectively in aged as in new GIC, and 3) GIC surface profile was not an important factor in this fluoride release. Future in vivo or intraoral experiments could demonstrate the potential of these effects for clinical benefit.

MANUFACTURERS' DETAILS

- Ketac-Fil and Z-250: 3M-ESPE, Seefeld, Germany.
- Mixing unit: ESPE Capmix, Seefeld, Germany.
- Light polymerization unit: Heliolux Vivadent, type HL1, Shaan, Austria
- Scanning electron microscope: Jeol JSM 840A, Tokyo, Japan
- Energy dispersive spectrometer: Oxford ISIS.
- Ion-Analyzer: Orion EA 920, Orion Research Inc., Mass., USA
- Ion-selective electrode: Orion, Ionplus.

ADDRESS FOR CORRESPONDENCE

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REFERENCES

1. Burke, F.M., Ray, N.J. and McConnell R.J. Fluoride-containing restorative materials. *Int. Dent. J.*, 2006; **56**: 33-43.
2. Dionysopoulos, P., Kotsanos, N. and Pataridou, A. Fluoride release and uptake by four new fluoride releasing restorative materials. *J. Oral. Rehabil.*, 2003; **30**:866-872.
3. El Mallakh, B.F. and Sarker, N.K. Fluoride release from glass ionomer cements in deionised water and artificial saliva. *Dent. Mater.*, 1990; **6**:118-122.
4. Hsu, H.M., Huang, G.F., Chang, H.H., Wang, Y.L., Guo, M.K. A continuous flow system for assessing fluoride release/uptake of fluoride-containing restorative materials. *Dent. Mater.*, 2004; **20**:740-749.

5. Hallgren, A., Oliveby, A. and Twetman, S. Salivary fluoride concentrations in children with glass ionomer cemented orthodontic appliances. *Caries Res.*, 1990; **24**:239-241.
6. Williams, J.A., Billington, R.W. and Pearson, G.J. A long term study of fluoride release from metal-containing conventional and resin-modified glass-ionomer cements. *J. Oral Rehabil.*, 2001; **28**:41-47.
7. Han, L., Cv, E., Li, M. et al. Effect of fluoride mouth rinse on fluoride releasing and recharging from aesthetic dental materials. *Dent. Mater.J.*, 2002; **21**: 285-295.
8. Preston, A.J., Agalamanyi, E.A., Higham, S.M. and Mair, L.H. The recharge of esthetic dental restorative materials with fluoride in vitro. Two years results. *Dent. Mater.*, 2003; **19**:32-37.
9. Dhondt, C.L., De Maeyer, E.A.P. and Verbeeck, R.M.H. Fluoride release from glass ionomer activated with fluoride solutions. *J. Dent. Res.*, 2001; **80**: 1402-1406.
10. Nomoto, R., Uchida, K., Momoi, Y. and McCabe, J.F. Erosion of water-based cements evaluated by volumetric and gravimetric methods. *Dent. Mater.*, 2003; **19**:240-244.
11. Freedman, R. and Diefenderfer, K.E. Effects of daily fluoride exposures on fluoride release by glass ionomer-based restoratives. *Operative Dent.*, 2003; **28**:178-185.
12. Olsson, C., Emilson, C.G. and Birkhed, D. An in vitro study of fluoride release from a resin-modified glass ionomer cement after exposure to toothpaste slurries of different pH. *Clin. Oral Investig.*, 2000; **4**:233-237.
13. Eakle, W.S., Featherstone, J.D., Weintraub, J.A., Shain, S.G. and Gansky, S.A. Salivary fluoride levels following application of fluoride varnish or fluoride rinse. *Community Dent. Oral Epidemiol.*, 2004; **32**:462-469.
14. Mehta C. & Patel R. (1996). *SPSS Exact Tests 7.0 for Windows.* Chicago: SPSS Inc.
15. Hayacibara MF, Ambrozano GM, Cury JA. Simultaneous release of fluoride and aluminum from dental materials in various immersion media. *Oper Dent.,* 2004; **29**:16-22.
16. Carey, C.M., Spencer, M., Gove, R.J. and Eichmiller, F.C. Fluoride release from a resin-modified glass-ionomer cement in a continuous-flow system: effect of pH. *J. Dent. Res.*, 2003; **82**:829-832.
17. Attar, N. and Turgut, M.D. Fluoride release and uptake capacities of fluoride-releasing restorative materials. *Oper. Dent.*, 2003; **28**:395-402.
18. Forss H, Widstrom E. The post-amalgam era: a selection of materials and their longevity in the primary and young permanent dentitions. *Int J Paediatr Dent.,* 2003; **13**:158-64
19. Adair SM. The role of fluoride mouthrinses in the control of dental caries: a brief review. *Pediatr Dent.,* 1998; **20**:101-104.
20. De Witte AM, De Maeyer EA, Verbeeck RM. Surface roughening of glass ionomer cements by neutral NaF solutions. *Biomaterials,* 2003; **24**:1995-2000.
21. Rothwell, M., Anstice, H.M. and Pearson, G.J. The uptake and release of fluoride by ion-leaching cements after exposure to toothpaste. *J. Dent.,* 1998; **26**:591-597.