Resin-modified glass ionomer cements: fluoride release and uptake

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The aim was to study the short- and long-term fluoride release from resin-modified glass ionomer cements (GIC). The aim was also to determine the effect of fluoride treatment of 9-month-old specimens, consistency of the mix, and pH of the environment on the fluoride release. GIC test specimens were continually exposed to running water, and the fluoride release was measured periodically by storing the specimens in 5 ml deionized water for 1 week and measuring the fluoride content of the solution. After 24 h, 1 month, 9 months, and 11 months in running water four of the six resin-modified GICs released as much as or more fluoride than the auto-curing GIC tested for comparison. Fluoride treatment after 9 months also increased the fluoride release of these four brands, as was the case with the conventional GIC. At 24 h and 1 month two of the resin-modified GICs released smaller amounts of fluoride than the other materials, and the fluoride treatment used on those had no or only a minimal effect. Thin consistency of a mix resulted in higher fluoride release for one resin-modified material than a thick mix. Low pH increased the fluoride release for all materials. □ *Dental materials; dentistry, operative; dental restoration*

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Modifications of glass ionomer cement (GIC) are introduced as light-curing. This name is, however, misleading, since the GIC component still sets chemically without light. Only the setting of the 'resin' component is activated by light. In the present study the conventional GICs are called auto-curing, and the new ones are called resin-modified, in accordance with the suggestion of McLean et al. (1).

The two most important properties of the glass ionomer cements are the ability to establish a permanent seal between the tooth and the filling (2) and the fluoride release (3). To ensure a permanent anticariogenic effect, a dynamic situation is desirable. If there were only a one-way release of fluoride, there would be a danger of emptying the fluoride resources after some period of time. If, on the other hand, aged GIC material is capable of binding fluoride and releasing it again, this would ensure a permanent fluoride effect. Results of laboratory studies (4–6) suggest that there is such an ability in conventional, auto-curing GICs. Resin-modified GICs have been developed, first as lining and base materials but recently also as filling materials. Some of the lining resin-modified materials have been shown to release fluoride to the same extent as conventional GICs, whereas some products released minute amounts only (7). One resin-modified GIC filling (Fuji II LC) has been shown to release as much fluoride as the respective auto-curing material (8). Furthermore, the resin-modified GICs have been shown to inhibit caries in in vitro tests (9).

Thin mixes of auto-curing GICs have been shown to release more fluoride than thick mixes (10). In association with dual-curing brands the effect of the consistency has not been studied so far.

It may be assumed that resin-modified GICs are more acid-resistant than conventional GICs (11). Consequently, the increase in fluoride release observed at low pH with conventional GICs should be less pronounced than with resin-modified brands (12).

The aim of this study was to seek answers to the following questions: 1) Is the pattern of fluoride release and uptake by resin-modified GIC restorative materials similar to that of auto-curing GICs, and are there differences between different resin-modified GIC restorative brands? 2) Does the consistency of the mix (powder to liquid ratio) influence the fluoride release and uptake of resin-modified GICs? and 3) Will an acidic environment increase the fluoride release from resin-modified materials?

Materials and methods

The GIC products studied are shown in Table 1. Four of the cements (FII LC, E, P, and V) are claimed to be resin-modified GICs recommended for restorative purposes, whereas one is a resin-modified fissure sealant (FIII LC). Product D is called a fluoride-releasing compomer by the manufacturer. Product E was an experimental cement later marketed under the brand name Vitremer. A conventional GIC (FII A–C) was used as a 'control' for comparison. The hand-mixed restorative materials FII LC, E, and V were mixed in two different powder to liquid ratios (Table 1). The materials were handled in accordance with the manufacturers' instructions, and 2-mm-thick round discs with a diameter of 5 mm were made in a plastic mold. During setting, the bottom of the mold was placed on a glass plate, and the

Table 1. Materials examined

Product	Batch no. Powder	Batch no. Liquid	Code	Consistency, spoon/drops	Manufacturer
Resin-modified					
Fuji II LC	061113	071011	FII LC	Thick 1/2 Thin 1/4	GC
Fuji III LC	072792	072792	FIII LC	Thin 1/1	GC
EXM 3M*	156 B	156 L	E	Thick 1 tan/2 Thin 2 blue/2	3M
Photac-Fil	0003		P	Capsule (thick)	ESPE
Variglass	9206025	9206241	V	Thick 1 tan/2 Thin 2 blue/2	Dentsply
Dyract	930753		D	Capsule (thick)	Dentsply
Auto-curing ('control')					
Fuji II	930114A	930114A	FII AC	Thick 1/1	GC

* Marketed as Vitremer®.

surface of the cement was covered with another glass plate. The resin-modified GICs were cured on both sides with a Visilux curing light (3M Co.) for twice the time recommended by the manufacturer. After setting, the specimens were removed from the mold, and all were protected with petroleum jelly even if the manufacturer did not recommend this kind of protection. After being stored for 24 h in tap water at room temperature (22 ± 2°C), the surface of the specimens was ground lightly against 600 grit paper. The specimens were made in triplicate. The discs were transferred and stored in a 5-l container into which non-fluoridated ($F < 0.1$ ppm) tap water was running at a speed of approximately 1 l/min.

For measuring the fluoride release each specimen was periodically transferred into 5 ml of double-distilled deionized (Milli-Q treated, Millipore) water (pH 6.1) for 1 week (7). After storage of the specimen in the deionized water the fluoride content of the water was measured with an expandable ion analyzer (EA 94, Orion Research Inc., USA), using a fluoride electrode (94-09) and a reference electrode (900100, 4 M KCl + AgCl). The ability of the materials to bind fluoride for subsequent release was tested by storing the 9-month-old specimens in 5 ml buffered fluoride solution (50 ppm F^- in 1 mM Tris-HCl, pH 7.0) for 1 week. After this period the specimens were exposed to running water for 24 h. The fluoride release in 5 ml deionized water during 1 week was then measured as described above.

To test the fluoride release in an acidic solution, 11-month-old specimens were stored for 1 week at pH 5.0 in a buffered 20 mM Na-acetate solution (5 ml). The fluoride content of this solution was then measured.

To get a crude picture of the auto-curing function of the resin-modified GICs, a test was done in which the resin-modified materials, except D, were allowed to set in darkness on the mixing slab. After 5 min the test specimen was cut with a knife to test whether it had

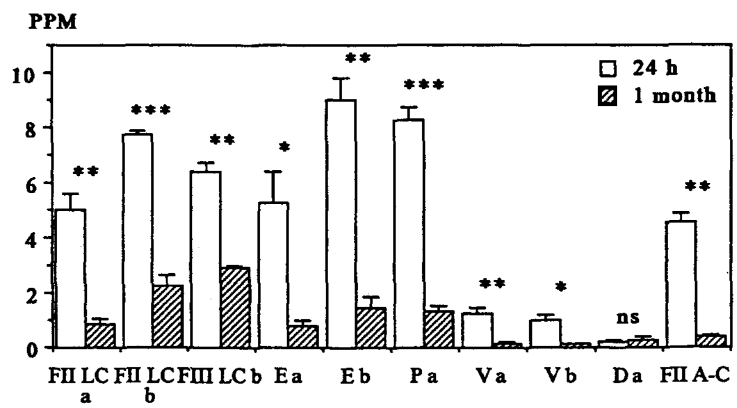

Fig. 1 Fluoride release from thick (a) and thin (b) mixes of resin-modified glass ionomer cements after 24 h (open bars) and 1 month (hatched bars). Half of the range is indicated at the top of the columns. Statistical significance: *** $p > 0.001$, ** $p < 0.01$, * $p > 0.05$. See Table 1 for explanation of the codes.

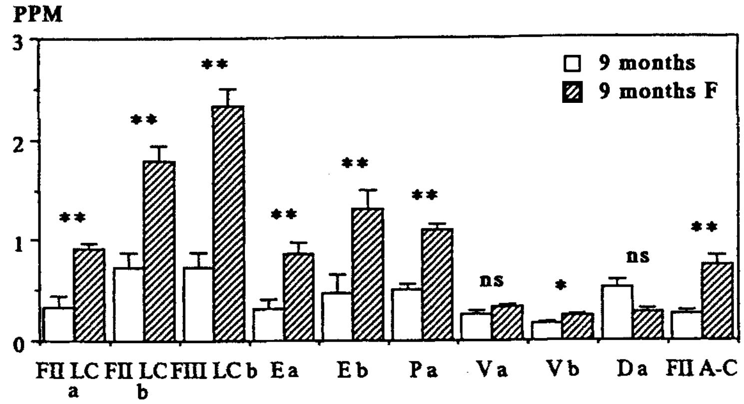

Fig. 2. Fluoride release during 1 week in deionized water from untreated glass ionomer cement specimens (open bars) and from specimens treated for 1 week with a 50-ppm fluoride solution (hatched bars) after 9 months. Specimens made from thick mixes (a) and from thin mixes (b). Half of the range is indicated at the top of the columns. Statistical significance: *** $p > 0.001$, ** $p < 0.01$, * $p > 0.05$. See Table 1 for explanation of the codes.

clinically hardened. Material D was condensed into a metal tube and put in water.

The results of the fluoride release were analyzed with a paired, two-tailed t test.

Results

The fluoride release from the different materials after 24 h and 1 month is shown in Fig. 1. The release for most materials was significantly lower after 1 month than after 24 h. Only FII LC showed significantly higher fluoride release for the thin mix than for the thick mix ($p < 0.05$) after both time periods.

The release after 24 h from thick mixes of the resin-modified products FII, E, and P and from the auto-curing product FII was higher than from products V and D.

After 9 months in running water (Fig. 2) all products studied except V and D released significantly more fluoride during 1 week after treatment with the 50 ppm fluoride solution than before the treatment.

After 11 months in running water the release in pH 5 was significantly greater than in pH 6 for all materials studied that long (Fig. 3). There was an indication of more release at pH 5 from thick than from thin mixes. The release from D and F A–C that had been only 1 month in running water did not differ significantly in the two different solutions ($p > 0.05$).

The setting test performed in darkness showed that

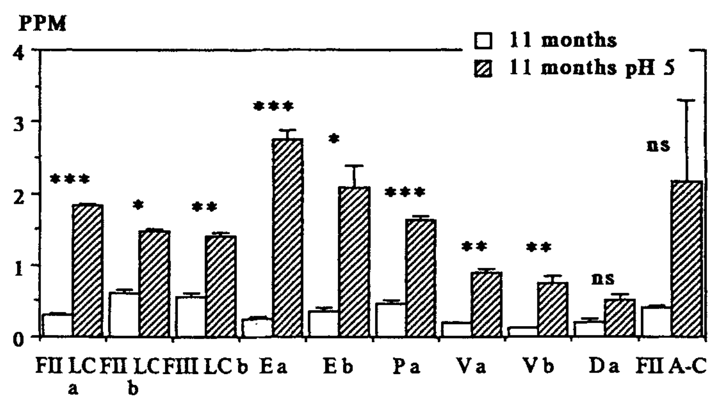

Fig. 3. Fluoride release from specimens in deionized water (open bars) and in an acidic solution (pH 5.0) (hatched bars) after 11 months in running water. Specimens made from thick mixes (a) and from thin mixes (b). Half of the range is indicated at the top of the columns. Statistical significance: *** $p > 0.001$, ** $p < 0.01$, * $p > 0.05$.

resin-modified restorative brands F II LC, E, and P were clinically hard after 5 min, whereas V and D had not hardened at all. The latter two brands were not hard even after 24 h.

Discussion

The fluoride release from and uptake by the resin-modified products FII LC, FIII LC, E, and P was higher than or the same as that of the auto-curing GIC. These materials also set in darkness and may thus be considered 'true' GICs. The fluoride release from V and D was small and was not affected by the fluoride treatment. These products do not set in darkness, and therefore they may not, in my opinion, be considered glass ionomers at all.

At 9 months brand D seemed to show more release before than after the fluoride treatment. The difference was, however, not statistically significant. The high value is most probably a result of some technical error, since it was also higher than the value at 1 month. The earlier finding that thin mixes release more fluoride than thick mixes (10) was confirmed by only one product (FII LC) in the present study.

The finding that the release of the resin-modified 'true' GICs increased in an acidic environment, as did conventional, auto-curing materials (12), was somewhat surprising. Since the resin-modified materials are claimed to be more acid-resistant (11), this would also indicate less effect of the pH than for auto-curing GICs. This finding means, however, that the question of the acid resistance of resin-modified GICs should be studied further and probably be reconsidered.

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