
INFLUENCE OF DIFFERENT STORAGE SOLUTIONS ON FLUORIDE RELEASE FROM GLASS IONOMER CEMENTS

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ABSTRACT

The clinical importance of fluoride release rests on its anticariogenic property. Hence, there are many studies concerning fluoride release from glass ionomer cements. Even though fluoride release tests have not reached the level of clinical simulation, they represent an important parameter of analysis. A survey of literature shows that different solutions imply in distinct quantitative assessment of fluoride release from the glass ionomer cements. Therefore, the characteristics of solutions can influence the release. A distinction must be made between the results to avoid doubtful interpretations. The aim of this work is to analyze the influence of the different storage solutions on fluoride release in the in-vitro tests.

KEY WORDS: fluoride release; *in vitro* test; glass ionomer cements

INTRODUCTION

The ability of Glass Ionomer Cements (GIC's) to release fluoride has been known for some 20 years and has been a

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significant factor in their increasing use in dentistry (CHOW; VOGEL, 2001). Many investigations on fluoride release have been carried out *in vitro* (FORSTEN, 1990; DESCHEPPER et al., 1991; REZK-LEGA et al., 1991; GAO et al., 1997; GEURTSSEN, 1998; LEVALLOIS et al., 1998; VERBEECK, et al., 1998; WILLIAMS et al., 1999; GEURTSSEN, 1999; CARVALHO; CURY, 1999; BERTACCHINI et al., 1999; EL MALLAAKH; SARKAR, 1999; GAO et al., 2000; BEHREND; GEURTSSEN, 2001; WILLIAMS et al., 2002; CAVES et al., 2003; CREANOR et al., 2003; DIONYSOPOULOS et al., 2003; XU; BURGESS, 2003; SALES et al., 2003). Fluoride release is an advantageous characteristic of dental materials, because it has been demonstrated that materials releasing greater amounts of fluoride have superior caries preventive potential and are more desirable (FORSTEN, 1990; WILLIAMS et al., 2002; XU; BURGESS, 2003).

However, results from these studies can only be compared restrictively due to the differences in protocols for *in-vitro* testing, as the differences of the size, and shape of the tested specimens (BERTACCHINI et al., 1999; BEHREND; GEURTSSEN, 2001; CAVES et al., 2003; CREANOR et al., 2003; DIONYSOPOULOS et al., 2003; XU; BURGESS, 2003).

Furthermore, literature shows that the results from these studies have been expressed in different solutions that difficult the comparisons among them and indicate an underlying lack of understanding of the factors governing fluoride release (LEVALLOIS et al., 1998; VERBEECK, et al., 1998; GEURTSSEN, 1999; CAVES et al., 2003; CREANOR et al., 2003). Not only does the medium vary between studies, but also the volume used. The range in volume varied from 1 mL (DESCHEPPER et al., 1991) to 50 mL (CRANFIELD, 1982; BERTACCHINI et al. 1999; SALES et al., 2003) and the observation time has varied from 1 hour (REZK-LEGA et al., 1991) to 2 years (FORSTEN, 1990). Interestingly, the sample size ranged from 1 (BERTACCHINI et al., 1999) specimen to 25 (MOMOI; MCCABE, 1993) specimens of each material. In addition, specimen shape and size have also varied considerably. The size of the specimen used for the assessment of fluoride release has been one of the most salient differences. The dimensions of the discs varied from 3 mm diameter x 1.5 mm thickness (MONTEITH et al., 1999) to 20 mm diameter x 1.5 mm thickness (CRANFIELD, 1982), while other studies used cylinders of materials varied from 3 mm diameter x 2.7 mm height (MOMOI; MCCABE, 1993) to 6 mm diameter x 12 mm height 33 and bar of the 2 mm diameter x 2 mm height x 24 mm length (WILLIAMS et al., 1999). A review of the literature (TABLE 1) shows that although most

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TABLE 1 – In vitro studies on fluoride release protocols.

Year	First author	Materials/Manufacturer	Characteristics of specimens			Type of storage solutions	Volume of solution	Observation time
			Nº*	Shape	Size (mm)			
1982	Cranfiel ⁷	ChemBond (Amalgamated Dental), ASPA (Amalgamated Dental), Fuji- Ionomer (GC)	----	Disc	20 x 1.5	Deionized water	50 mL	91 days
1984	Swartz ³¹	Aspa (Caulk), Fuji II (GC), Ketac (Espe), M.Q.(SS White), Chembond (Caulk), Fuji I (GC), Ketac-Cem(Espe), Fluorothin (SS White), Durelon (Premier-Espe)	6	Disc	10 x 2.25	Distilled water	3 mL	30 days
1991	Rezk-Lega ²⁹	Ketac- Cem Radiopaque (ESPE), Aqua-Cem (Dentsply)	6	Disc	8.5 x 3.5	Distilled water pH 7; Saliva; Saliva + water pH 7; Saliva + water pH 4.5; Albumen phosphate buffer pH 7	10 mL	1 hour
1990	Forsten ¹⁵	ASPA (Amalgamated Dental), Fuji II (GC), Fuji III (GC), Chemfil II (Dentsply), Ketac- Fil (Espe), Ketac-Silver (Espe), Miracle Mix (GC), Fuji II + Sybralloy (GC +Kerr), Fluor Alloy (Dentoria), Heliomolar Radiopaque (Vivadent)	-----	Disc	10.7 x 2.8	Deionized water	5 mL	2 years
1998	De Moor ¹⁰	Fuji II (GC), Fuji Cap II (GC), Miracle Mix (GC), Chemfil II (Dentsply), Chemfil Cap II (Dentsply), Chelon-Fil (Dentsply), Ketac- Fil (3M), Chelon-Silver (Dentsply), Ketac-Silver (3M), Shofu II (Shofu)	5	Discs	6 x 3	0.01 mol/l acetic acid solution, with pH =4	25 mL	140 days
1998	Levallois ²	Fuji II LC Improved (GC), Vitremer (3M), Dyract (Dentsply)	12	Discs	5 x 3	Distilled water, artificial saliva medium (NaCl 125.64 mg-1, KCL963.90 mg-1, KSCN 189.20 mg-1, KH2PO4 654.50 mg-1, CO(NH2)2 200.00 mg-1, CaCl22H2O 227.80 mg-1, Na2SO410H2O 763.20 mg-1, NH4CL 178.00 mg-1, NaHCO3 630.80mg-1)	5 mL	7 days

Year	First author	Materials/Manufacturer	Characteristics of specimens			Type of storage Solutions	Volume of solution	Observation time
			N°*	Shape	Size (mm)			
1999	Carvalho ⁴	Chelon-Fil (3M-ESPE), Dyract (Dentsply), Variglass (Dentsply) Vitremer (3M-ESPE), Tetric (Ivoclar-Vivadent)	6	Disc	8.6 x 1.6	Deionized water -Artificial saliva: Ca 1.5mM, PO4 0.9 mM; KCL 150 mM and Tris buffer 20 mM, pH 7.0, containing NaN3 0.02% -pH –cycling system- demineralizing (De) and remineralizing (Re) De- Ca 2.0mM, PO4 2.0 mM and acetate buffer 75 mM, pH 4.3, containing NaN3 0.02% Re-the same as artificial saliva	2 mL	15 days
1999	Geurtsen ²¹	Compoglass F (Vivadent), F200 (3M ESPE), Dyract AP (Dentsply), Experimental compomer	15	Disc	5 x 2	Distilled water, acid buffer I (20-mM KCL, 154- mM NaCL, 3.6- mM NaH2PO4.H2O, pH 4.2), Neutral buffer II (1.09mM CaCL2, 30mM KCL, 0.68mM KH2PO4, 0.0026mM NaF(0.05PPM), 50- mM HEPES, pH 7.0), Neutral buffer III (neutral buffer II supplemented with 40mU/mL of porcine liver esterase)	5 mL	6 days
2000	Marks ²³	Vitrebond (3M ESPE), Vitremer (3M ESPE); PhotacBond (3M ESPE); GC Lining (GC); Variglass (Dentsply); Dyract (Dentsply)	5	Cylinders	6.0 x 3.0	0.01 mol/l citric acid adjusted to 7 pH with NaOH Distilled water	25 mL	168 hour
2001	Behrend ²	Compoglass F (Vivadent), F200 (3M ESPE), Dyract AP (Dentsply), Fuji II LC (GC), Experimental polyacid-modified composite resin	12	Discs	5 x 2	Distilled water, Acidic buffer II (20-mM KCL, 154- mM NaCL, 3.6- mM NaH2PO4.H2O, pH 4.2), Neutral buffer III (1.09-mM CaCL2, 30-mM KCL, 0.68-mM KH2PO4., 0.0026 -mM NaF(0.05PPM), 50- mM HEPES, pH 7.0), Neutral buffer IV (neutral buffer III supplemented with 1.6 u/mL porcine liver esterase)	5 mL	1 years

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Year	First author	Materials/Manufacturer	Characteristics of specimens			Type of storage Solutions	Volume of solution	Observation time
			N°*	Shape	Size (mm)			
2001	Gao ¹⁸	GI-1 (Shanghai Qingpu Dental), Ketac- Molar (3M ESPE), FX(Shofu), Hi-Dense(Shofu), Photac-Fill (3M ESPE), Photac-Fill Quick (3M ESPE), Hytac Aplitip (3M ESPE), Compoglass F (Ivoclar-Vivadent), Z 100 (3M ESPE)	5	Disc	3.0 x 2.7	Artificial saliva solution (0.05 m acetate buffer with 2.2 mM CaHPO4 adjusted with glacial acetic acid to pH 5.0)	2 mL	42 days
2002	Williams ³⁴	G338, LG26 (Laboratory of the Government Chemist-UK)	4	Discs	10 x 1	Distilled water	20 mL	21 days
2002	Czarnicka ⁹	G338 (Laboratory of the Government Chemist-UK)	5	Cylinders	6.0 x 12	Distilled water, Lactic acid solution (pH 2.7)	8 cm ³	42 hour
2002	Attar ¹	Ceramfil _ (PSP Belvedere), Compoglass (Vivadent), Dyract (Dentsply), Tetric (Vivadent), Valux Plus (3M)	15	Disc	6.0 x 1.5	Deionized water	4 mL	60 days
2003	Caves ⁵	Ketac-Cem (3M-ESPE), Ultra Band – Lok (Reliance) 3M-Multicure (3M Unitek)	5	Disc	4.5 x 2	Deionized water	2 mL	30 days
2003	Creanor ⁸	Fuji Ortho(GC), Ultra Band-Lok (Reliance)	5	Disc	6.0 x 3	Deionized water	2 mL	60 days
2003	Xu ³⁵	Fuji IX (GC), Miracle Mix(GC), Ketac-Molar Aplicap (3M ESPE), Ketac-Silver Aplicap (3M ESPE), Vitremer (3M ESPE), Photac-Fil Quick Aplicap (3M ESPE), Fuji II LC Improved (GC), Compoglass (Vivadent), F200 (3M ESPE), Dyract AP (Dentsply), Hytac (3M ESPE), Ariston pHc (Vivadent), Surefil (Dentsply), Solitaire (Kulzer)	5	Discs	4 x 9	Deionized water	3 mL	21 days
2003	Sales ³⁰	Dyract (Dentsply), Dyract AP (Dentsply), Compoglass F(Vivadent), Fuji II LC (GC)	6	Discs	10 x 1	Distilled water, 0.01 mol/l lactate buffer	50 mL	7 days
2003	Dionyso poulos ¹²	Ketac-Molar Aplicap (3M ESPE), Vitremer (3M ESPE), F200 (3M ESPE), Hytac (3M ESPE)	15	Discs	10 x 2.25	Deionized water	5 mL	22 days

*Number of test specimens per material and storage medium.

workers detect fluoride release into solution using a fluoride ion-specific electrode, there are many variations in the details of each experiment.

MECHANISMS OF FLUORIDE RELEASE

Fluoride release ability is attributed to chemical reactions and it has been indicated that ion diffusion process controls fluoride release from GIC's (WILLIAMS et al., 2002). In this mechanism, at least two kinetic reactions take place simultaneously (process I and II), when the fluoride ions are exchanged for other anions (such as Cl-) in the aqueous environment (VERBEECK et al., 1998). This reaction Eq (1) evidences a linear relationship with the square root of time of cement immersion (VERBEECK et al., 1998; GEURTSSEN, 1999; WILLIAMS et al., 2002), which can be represented by cumulative fluoride release $[F]_c$ of each material after immersion in water (VERBEECK et al., 1998).

$$[F]_c = \frac{[F]_I t}{t^{1/2} + t} + \beta \sqrt{t} \quad (1)$$

In Eq. (1), $[F]_I$, $t^{1/2}$ and β are constants characteristics for a given glass ionomer. The right-hand first term of equation corresponds to a short-term fluoride release ceasing after some time (with $[F]_I$ the total amount of fluoride to be released and $t^{1/2}$ its "half-life"), and it is referred to as Process I. The right-hand second term is associated with a long-term fluoride release (Process II). Thus, the Process I is most likely associated with an initial leaching of fluoride from the glass particles in the surface layer of these materials, while Process II is prolonged and with a more slowly occurring elution, which could be responsible for the long-term release of fluoride. Process II suggests a dissolution/diffusion mechanism of fluoride within the material being the rate limiting step (VERBEECK et al., 1998). The fluoride release from conventional GIC's is controlled by diffusion and/or surface reaction depending on the stability of complexes between the cations and the acid used. It is often observed that the surface reaction predominates in the acid, which forms the stable complexes. This is because the cations are extracted from the matrix of polycarboxylate salts by the acid anions. On the other hand, it is quite different for compomer and resin-modified glass ionomer. In these materials, the matrix part consisting of polymerized resins is

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more resistant to the acid attack and as a result, the surface reaction is suppressed. Thus, the diffusion of the specimens through the matrix would control the dissolution process (GAO et al., 1997; SALES et al. 2003).

Although the differences among the characteristics of the specimens and the nature of the glass ionomer cement based materials, it must be considered that *in vivo* complex parameters, such as pH fluctuation or the action of various hydrolytic enzymes in saliva, may significantly influence the behavior of a filling material in the oral cavity (BEHREND; GEURTSSEN, 2001). Consequently, data from *in vitro* studies will only allow limited conclusions for the intraoral situation. Hence, it has been reported that the amount of fluoride leaching from these materials significantly depends on the nature of the medium, as well as the product tested (GEURTSSEN, 1998).

The purpose of this survey of the literature is to analyze fluoride release in different storage solutions for *in vitro* testing. The variables in the studies' designs are often inadequately described or omitted, as well as types of solutions used and variations in studies' characteristics make direct comparisons impossible.

STORAGE SOLUTIONS

Considering that the carious lesion is recognized as a loss of mineral from a disequilibrium of remineralization/demineralization cycle of tooth substrates, the role of the fluoride ion as a potential remineralizing agent is clearly recognized (MOUNT, 1998). In order to investigate this ability of different materials, different protocols should simulate these conditions and different storage solutions were proposed.

Deionized and distilled water has been mostly employed mostly for fluoride release studies, although these media does not truly represent the intraoral conditions (GEURTSSEN, 1999; EL MALLAAKH; SARKAR, 1999). In the aqueous environment of the oral cavity, numerous factors like low pH due to the presence of cariogenic microorganisms or acidic food, ionic composition and strength of the saliva, or enzymatic attacks are important parameters, which might influence the quantity and quality of substances released from a restorative material. The significance of these parameters on fluoride release has been scantily investigated (GEURTSSEN, 1999; CARVALHO; CURY, 1999; BEHREND; GEURTSSEN, 2001). Therefore, fluoride might be more soluble in

the oral environment than in laboratory studies in water (WILLIAMS et al., 2002).

In attempt to offer more reliable oral conditions, artificial saliva has been proposed to these investigations (LEVALLOIS et al., 1998; GEURTSSEN, 1999; EL MALLAAKH; SARKAR, 1999; BEHREND; GEURTSSEN, 2001), considering that the active carious lesions are recognized as a remineralization/demineralization cycle from and to the tooth surfaces, the role of the fluoride ion is clearly recognized (MOUNT, 1998), and the protocols to evaluate fluoride should simulate these conditions.

When the fluoride released from different materials was compared using deionized water or artificial saliva, reversed results were found (EL MALLAAKH; ARKAR, 1999). However, it has been hypothesized that fluoride release in artificial saliva might reproduce intraoral conditions and furthermore to be the medium of choice for fluoride release studies, this situation is not correct. El Mallaakh; Sarkar (1999) showed that the values of fluoride release in water and in artificial saliva were consistently different, since more fluoride was released into deionized or distilled water than into artificial saliva. These findings show that the fluoride release is significantly influenced by the ionic strength of the medium, due to presence of cations and anions in artificial saliva, with an ionic effect on the solubility (LEVALLOIS et al., 1998; GEURTSSEN, 1999; EL MALLAAKH; SARKAR, 1999; BEHREND; GEURTSSEN, 2001).

The difference between fluoride release in distilled water and in artificial saliva is probably due to the formation of CaF₂ precipitated on the surface of the material. This insoluble layer formed a physical barrier to fluoride release (LEVALLOIS et al., 1998). Then, the reduction of fluoride release in artificial saliva in relation to fluoride release in distilled water indicates the capacity of the glass to form a protective layer (LEVALLOIS et al., 1998). Additionally, in the oral cavity, the fluoride release may be enhanced by the action of hydrolytic enzymes in saliva and pH fluctuation (EL BADRAWY; MCCOMB, 1998). This premise is supported by esterase experiments that found a corresponding activity to hydrolases in human saliva (GEURTSSEN, 1999; BEHREND; GEURTSSEN, 2001). Higher amounts of fluoride are released into neutral solutions, containing esterase than into neutral solutions without enzymes (GEURTSSEN, 1999). This effect is very likely due to an enzymatic softening of the organic matrix, which has been observed during the storage in esterase buffer (GEE DE et al., 1996).

The highest amount of fluoride release may be increased by hydrolytic enzymes in saliva and under acidic conditions, since the

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fluoride release may be influenced by the pH of the medium (GEURTSSEN, 1998; GEURTSSEN, 1999; CARVALHO; CURY, 1999; BEHREND; GEURTSSEN, 2001; CZARNECKA et al., 2002, WILLIAMS et al., 2002).

The great majority of studies have reported the fluoride release in acid solutions as acetate buffer of pH 4.3, in a phosphate buffer of pH 4.2 or a citrate-phosphate buffer of pH 4.0 (CARVALHO; CURY, 1999; GEURTSSEN, 1999). Lactic acid has also been used for the fluoride release test, since this acid is most likely to exist in the oral environment and relevant to caries initiation (NICHOLSON et al., 1999; SALES et al., 2003; CZARNECKA et al., 2002). These studies concluded that the fluoride release amount was increased under acidic conditions, which results are also dependent on the acid nature. Clinically, an acidic pH between 4.0 and 5.0 can be found on tooth or filling surfaces that are covered by a metabolic active plaque (GEURTSSEN, 1999). Therefore, intraoral fluoride release may be enhanced by plaque-associate acids, such as lactic acid (BEHREND; GEURTSSEN, 2001). It was demonstrated that GIC's and Compomers could significantly increase the pH of lactic acid by 0.5 to 1.2 units due to a buffering effect (NICHOLSON et al., 1999). The clinical consequences of these effects were increased fluoride release at low pH even if, buffering capacity is still unclear (BEHREND; GEURTSSEN, 2001).

Higher amounts of fluoride release into an acidic solution (pH 4.2) than into artificial saliva (pH 7.0) and artificial saliva with addition porcine esterase (pH 7.0) has been assessed when compomers were investigated. This phenomenon is very likely caused by a pronounced erosion of the polysalt matrix of the GIC and an enhanced hydrolytic degradation occurring at the matrix-filler interface, due to low pH (GEURTSSEN, 1998; GEURTSSEN, 1999; CARVALHO; CURY, 1999; BEHREND; GEURTSSEN, 2001). The materials' dissolution was controlled by a diffusion mechanism. The surface characteristics of each material, which were immersed in the acidic solution, were quite different from the surface before and after immersion in distilled water. The surface seemed to be roughened after immersion in the distilled water, while many pores were seen on the surface of the specimen immersed in acidic solution (SALES et al., 2003).

Czarnecka et al. (2002) suggested that acid conditions favor complexation of fluoride, which may be linked to the increased release of aluminium and the resulting formation of complexes such as AlF_2^- .

Caries process (pH cycling) and most of the data reported on fluoride release dental materials are based upon measurements made in solutions, which do not simulate the dynamics of caries development (CARVALHO; CURY, 1999). This conclusion was corroborated by observations in which the acidic conditions during the daily demineralization period probably increased the release of fluoride due to low pH. The explanation lies on the restorative material fluoride release in the localized plaque due to a pH drop as a consequence to a cariogenic challenge (CARVALHO; CURY, 1999). The increase in fluoride concentration changed the mineral saturation level favoring fluorapatite formation, reducing the effect of hydroxyapatite dissolution from enamel by the acid produced in dental plaque. When the plaque pH returned to the original level, the restorative material still released fluoride, which would enhance the remineralization of the enamel induced by salivary calcium and phosphate (NICHOLSON et al., 2000).

Intending to determine the level of fluoride release in solutions for pH –cycling (demineralizing solution – Ca 2.0 mM, PO₄ 2.0 mM, and acetate buffer 75 mM, pH 4.3, containing NaN₃ 0.02%- pH 4.3 and remineralizing solution – Ca 1.5 mM, PO₄ 0.9 mM, KCL 150 mM and Tris buffer 20 mM, pH 7.0, containing NaN₃ 0.02% – pH 7.0). Carvalho; Cury (1999) concluded that fluoride release was higher in pH – cycling than in water, and the lowest value was observed in artificial saliva, statistically significant for GIC, RM-GIC, Polyacid modified resin and composite. In this study, demineralizing and remineralizing solutions were used to reproduce a dynamic situation, because dental caries represented a process of alternating demineralization and remineralization phenomena that were a direct function of conditions that maintain a critical pH in the mouth.

CONCLUSIONS

Either distilled or deionized water do not represent the conditions in the intraoral aqueous environment. Hence, different media was tested, including artificial saliva, acidic solution and artificial saliva supplemented with esterase. It was observed a more fluoride leached into an acidic medium followed by distilled water and artificial saliva solutions. In addition, the action of hydrolytic enzymes may increase the liberation of fluoride. The association of demineralizing and remineralizing solutions would be preferable to evaluate the fluoride release of dental materials because it better

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represents the dynamic process of caries, but further evaluations are necessary for a better laboratorial performance analysis of these solutions. The survey divulgated should have given sufficient detail given to allow this to be interpreted.

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