The erosion kinetics of conventional and resin-modified glass-ionomer luting cements in acidic buffer solutions

Binnaz HAZAR-YORUC¹, Andac Barkin BAVBEK² and Mutlu ÖZCAN³

¹Yıldız Technical University, The Institute of Science, Division of Bioengineering, Davutpasa Campus, Esenler, 34210 Istanbul, Turkey

²Yüzuncu Yıl University, Faculty of Dentistry, Department of Prosthodontics, Kampüs 65080 Van, Turkey

³Dental Materials Unit, University of Zürich, Center for Dental and Oral Medicine, Clinic for Fixed and Removable Prosthodontics and Dental Materials Science, Plattenstrasse 11, CH-8032 Zürich, Switzerland

Corresponding author, Mutlu ÖZCAN; E-mail: mutluozcan@hotmail.com

This study investigated the erosion kinetics of conventional and resin-modified glass-ionomer luting cements in acidic buffer solutions as a function of time. Disc shaped specimens were prepared from conventional (Ketac-Cem: KTC) and resin-modified glass ionomer cements (Fuji Plus: FP) and immersed in three acidic buffer solutions (0.01 M) namely, acetic acid/sodium acetate (AA_B), lactic acid/sodium lactate (LA_B) and citric acid/sodium citrate (CA_B) with a constant pH of 4.1 and stored for 1, 8, 24, 48, 80, 120 and 168 h. F concentration was determined using ion-specific electrode. Si, Ca and Al concentrations were determined by atomic absorption spectroscopy. Ca, Al, Si and F solubility rates in both FP and KTC were the highest in CA_B solution. The erosion rates of both FP and KTC in all buffer solutions increased as a function of immersion time. The amount of F eluted from FP was more than that of KTC. The total amount of elements released from FP was less than KTC in all solutions.

Keywords: Acidic buffer solutions, Erosion, Glass-ionomer cement, Resin-modified glass-ionomer cement, Solubility

INTRODUCTION

Cementation is the final step after a sequence of clinical procedures and plays a significant role for the biological and mechanical durability of restorations in dentistry¹. The disintegration and erosion level of the luting cements is one of the most critical factors affecting the longevity of the restorations²⁻⁴. The solubility characteristics of luting cements under the oral conditions have been the topic of interest for a long time⁵⁻⁷.

Luting cements can be classified as water-based and polymerizing cements⁸. Among conventional water-based cements, glass-ionomer cements were reported to be relatively more stable for the oral conditions when compared to zinc phosphate and polycarboxylate luting cements⁹. The introduction of new techniques and materials, particularly to be used in adhesive and restorative dentistry, also led to the development of new dental cements providing improved adhesion. Resin-modified glass-ionomer luting cements are a combination of glass-ionomer and resin chemistries that set through an acid-base reaction of aluminosilicate glass powder and an aqueous solution of polyalkeneic acids modified with methacrylate units¹⁰. Such cements are generally considered as an intermediate material between conventional glass-ionomers and resin composite materials¹¹. Glass-ionomer cements have desirable properties to prevent recurrent caries by eluting ions for a long period of time^{12,13}. Unfortunately, there is still some confusion in the literature about solubility, disintegration and erosion characteristics of

dental cements when clinical results^{14,15} are compared with those of the laboratory ones^{5,6,9,12,16-19}. Furthermore, the results of *in vitro* studies also show variations for these cements^{9,12}. This is partially because the erosion behaviour of such cements was studied using different media. While some investigators studied the erosion behaviour of glass-ionomer cements in distilled water^{6,16,20,21}, others used organic acids^{3,18,22,23}, organic acid buffer solutions^{7,9,12,17,19} or preferred to use the actual oral environment^{4,14,15}. The organic acids such as lactic acid and acetic acid that changes the acidity of the oral environment are produced by the microorganisms as a result of carbohydrate intake to the mouth²². On the other hand, oral pH level decreases by the consumption of acidic dietary that includes citric acid and shows greater amounts of traces in various fruits and vegetables²⁴. Some studies about the erosion potential of the enamel in the acidic media emphasizes the aggressiveness of the oral conditions^{24,25}. Similar to the studies on demineralisation characteristics of the enamel, the dissolution potential of the glass-ionomer cements need to be evaluated in different acidic media that is frequently encountered in oral environment.

In fact, erosion behaviour of cements should also consider the dissolution steps of surface wash-off, diffusion in the solid state and surface corrosion²⁶. While Crisp *et al.*²⁷ reported several elements eluted from glass-ionomer cements in a lactate buffer solution, Czarnecka *et al.*¹⁸ examined the mechanism for erosion in an acidic buffer solution and Fukazawa *et al.*⁹ in organic acid buffer solutions. The erosion of glass-ionomer cements in an acetate buffer solution was controlled by the diffusion of the eluted ions through the

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cement matrix that was proportional to the square root of immersion time¹²). Also, Matsuya *et al.*²⁸) determined H⁺ ion concentration and the formation of constants for soluble complexes between the acid anions and metal cations in the set cement, controlled by the degree of the erosion of a glass-ionomer cement in organic-acid solutions. The dissolution of the cement in citric acid solutions seems to be controlled by both the diffusion and the surface reaction between the acid anion and the eluted ions⁹).

The objective of this study therefore was to investigate the erosion kinetics of conventional and resin-modified glass-ionomer luting cements with the behaviour of the eluted elements in three acidic buffer solutions as a function of time.

MATERIALS AND METHODS

Specimen preparation

Conventional (Ketac-Cem: KTC) and resin-modified glass-ionomer cements (Fuji Plus: FP) were mixed according to the manufacturers' instructions (Table 1). For each specimen, the powder was weighed on a digital balance with a measuring accuracy of 0.1 mg and the liquid was dispensed from a 1 mm tuberculin syringe calibrated to the nearest 0.1 mL. After the cement was mixed, within 30 s, it was placed into a polyethylene ring placed on a flat glass plate. A piece of dental floss was inserted into the cement and it was covered and pressed with another glass plate. The cement specimens (N=432, n=12 per group) were stored for 1 h in a humidifier (37°C, 100% relative humidity).

The cement disc specimens (diameter: 10 mm; thickness: 1 mm) were removed from the polyethylene ring. They were then suspended by the dental floss in polyethylene bottles containing 50 mL of one of the three acidic buffer solutions (0.01 M) namely, acetic acid/sodium acetate (AA_B), lactic acid/sodium lactate (LA_B) and citric acid/sodium citrate (CA_B) with a constant pH of 4.1 each in an incubator at 37°C. Bottles were stored for 1, 8, 24, 48, 80, 120 and 168 h and the immersed solutions were chemically analyzed at these time points.

Elution measurements

The released amounts of Calcium (Ca), Aluminium

(Al), Silicon (Si) and Fluoride (F) in buffer solutions were plotted in curves in relation to the square root of immersion time.

The amount of elements eluted from KTC and FP into the organic buffer solutions was well-fitted by the equation:

$$M_t = k \cdot t^n \quad (1)$$

M_t : the amount of species eluted from the cement

t : immersion time

k and n : constants

n constant was calculated from the slope (tangent α) of the line for each element in a solution. k constant was calculated from the intercept with the y axis. The values of k and n obtained from the intercept and the slope were placed respectively, in the log-log plot ($\log M = \log k + n \log t$) of Eq. 1⁹).

F concentration was determined using ion-specific electrode (Pharmacia, Biotech, LKB-NOVASPEC II, Freiburg, Germany). Si, Ca and Al concentrations were determined by atomic absorption spectroscopy (Hilger and Watts, Atomspek H1550, Margate, UK). Each measurement was repeated three times with different cement disks under the same conditions.

Statistical analysis

Statistical analysis was performed using SPSS System 15.0 for Windows (SPSS Inc., Chicago, IL, USA). Kolmogorov-Smirnov test was applied to evaluate the normal distribution of variables and homogeneity test (Levene test) was used to verify the homogeneity of variance. Both tests verified that that none of the ANOVA assumptions were violated. The means of each group were analyzed by two-way analysis of variance (ANOVA). Due to significant differences between groups, multiple comparisons were made by Tukey's adjustment test. P values less than 0.05 were considered to be statistically significant in all tests.

RESULTS

The type of cement ($p < 0.05$) and the organic acidic buffer solution ($p < 0.05$) significantly affected the results. The elements from each specimen were rapidly released

Table 1 The brands, abbreviations, chemical compositions, classification, manufacturers, batch numbers and mixing ratio of the luting cements used in this study. * P/L: Powder/Liquid (g/g)

Brand	Composition	Classification	Manufacturer	Batch No.	*P/L
Ketac-Cem (KTC)	P: Fluoroaluminosilicate glass L: Polyacrylic acid, polybasic carboxylic acid, water	Conventional glass-ionomer	3M ESPE, Seefeld, Germany	L352/L353	3.4/1
Fuji-Plus (FP)	P: Alumino-silicate glass L: 2-HEMA 37%, polyacrylic acid 22%, proprietary resins 10%, tartaric acid 6%, distilled water 25%	Resin-modified glass-ionomer	GC Corporation, Tokyo, Japan	P: 120491 L: 260391 C: 300391	2/1

into the organic buffer solutions in the initial stage of immersion and gradually approached saturation concentration. The total amount of elements released from FP was less than that of KTC. FP was more stable than KTC in all three buffer solutions (Table 2, Fig. 1).

Cumulative mass of Ca, Al, Si, and F eluted from KTC and FP against immersion time in AA_B, CA_B and LA_B buffer

solutions, respectively are presented in Figs. 2a–d. All of the curves showed linearity up to 168 h. The elution of Ca from FP was too small to be determined (Table 2, Fig. 2a). While the amount of Al eluted from KTC into CA_B was significantly higher than that into AA_B or LA_B (Fig. 2b), the amount of F eluted from KTC was significantly higher than that of FP (Table 2, Fig. 2d).

Table 2 The amount of Ca, Al, Si and F (mmol/L) eluted from the conventional (FP) and resin-modified

Immersion Time (h)	Buffer Solution	Ca (mmol/L)		Al (mmol/L)		Si (mmol/L)		F (mmol/L)	
		KTC	FP	KTC	FP	KTC	FP	KTC	FP
1	AA _B	0.04	0.00023	0.24	0.0014	0.15	0.0019	0.026	0.005
	CA _B	0.12	0.00013	0.20	0.0057	0.10	0.0024	0.11	0.075
	LA _B	0.0823	0.00013	0.10	0.0019	0.10	0.0010	0.012	0.075
8	AA _B	0.05	0.00026	0.28	0.0043	0.21	0.0032	0.028	0.038
	CA _B	0.23	0.00016	1.34	0.0086	0.57	0.0080	0.12	0.080
	LA _B	0.1276	0.00016	0.32	0.0043	0.14	0.0016	0.02	0.080
24	AA _B	0.07	0.00029	0.34	0.0067	0.25	0.0048	0.031	0.046
	CA _B	0.4	0.00019	2.71	0.0244	0.99	0.0100	0.13	0.084
	LA _B	0.13	0.00023	0.50	0.0095	0.19	0.0055	0.024	0.084
48	AA _B	0.11	0.00036	0.47	0.0109	0.26	0.0052	0.036	0.048
	CA _B	0.48	0.00033	3.42	0.0301	1.25	0.0120	0.13	0.101
	LA _B	0.1478	0.00026	0.63	0.0172	0.31	0.0067	0.033	0.098
84	AA _B	0.12	0.00049	0.47	0.0110	0.27	0.0071	0.037	0.054
	CA _B	0.59	0.00036	4.74	0.0435	1.32	0.0160	0.13	0.103
	LA _B	0.1562	0.00039	0.83	0.0196	0.35	0.0071	0.037	0.101
120	AA _B	0.12	0.00049	0.47	0.0124	0.27	0.0079	0.037	0.055
	CA _B	0.59	0.00039	4.86	0.0717	1.63	0.0200	0.13	0.103
	LA _B	0.17	0.00040	0.91	0.0224	0.36	0.0087	0.037	0.103
168	AA _B	0.12	0.00049	0.47	0.0124	0.27	0.0095	0.037	0.060
	CA _B	0.59	0.00040	5.11	0.0187	1.86	0.0240	0.13	0.103
	LA _B	0.183	0.00040	0.98	0.0282	0.50	0.0087	0.038	0.103

Fig. 1 The amount of Ca, Al, Si and F (mmol/L) eluted into buffer solutions (AA_B, LA_B and CA_B) from KTC and FP cements up to 168 h.

Fig. 2 Log-log plot graphs of the elements a) Ca, b) Al, c) Si and d) F eluted from KTC and FP in different acidic buffer solutions as a function of square root of the immersion time (days).

Organic acid buffer solutions of Ca and F in KTC and FP cements, reached their saturation points at 84th h and despite increasing immersion time, concentrations remained constant (Figs. 3a–b). The concentration of Al and Si in KTC kept increasing up to 168 h in organic buffer solutions (Fig. 3a). The same alteration was observed for Al and Si in FP but the magnitude of alteration was lower than that of KTC (Fig. 3b). Ca, Al, Si and F solubility rates in both FP and KTC were the highest in CA_B buffer solution. The amounts of eluted elements of KTC cement in LA_B solution were higher than AA_B solution (Fig. 3a). For FP cement, the amount of elements in buffer solutions depending on the solvent were as follows; Ca: $CA_B > LA_B = AA_B$, Si: $CA_B > LA_B = AA_B$, F: $CA_B > LA_B > AA_B$, Al: $CA_B > LA_B > AA_B$ (Table 2, Fig. 3b).

The dissolution of KTC and FP was proportional to the square root of immersion time. The slope (n), intercept (k) and correlation coefficient (r) values of KTC and FP cements obtained from the lines of solutions, depending on time, in organic buffer solutions, are given at Table

3. Correlation coefficient (r) values were obtained by clicking on the lines in the Excel sheet.

The solution rate of Ca, Al, Si and F from KTC cement was higher than the erosion rate of Ca, Al, Si and F from FP cement. Overall, the alteration of dissolution rates depending on the solutions were as follows:

n_{Ca} in AA_B : KTC > FP	n_{Al} in AA_B : FP > KTC
in LA_B : KTC > FP	in LA_B : KTC > FP
in CA_B : KTC > FP	in CA_B : KTC > FP
n_{Si} in AA_B : KTC = FP	n_F in AA_B : KTC > FP
in LA_B : KTC = FP	in LA_B : KTC > FP
in CA_B : KTC > FP	in CA_B : KTC > FP

DISCUSSION

The present study was undertaken to determine the erosion behaviour of conventional glass-ionomer (Ketac-Cem) and resin-modified glass-ionomer luting cements (Fuji Plus) and focused on time-dependent changes in acetic acid/sodium acetate (AA_B), lactic acid/sodium

Fig. 3 Amount of released elements in organic acidic buffer solutions. a) KTC b) FP.

Table 3 The intercept (k), value of slope (n), and correlation coefficient (r) when the amount of Ca, Al, Si, F eluted was plotted against immersion time in acidic buffer solutions

Material	Element	AA _B (mmol/L)			LA _B (mmol/L)			CA _B (mmol/L)		
		k	n	r	k	n	r	k	n	r
KTC	Ca	0.04	0.40	0.9362	0.09	0.20	0.9558	0.14	0.53	0.9458
	Al	0.24	0.20	0.9087	0.10	0.43	0.9875	0.26	0.88	0.9720
	Si	0.18	0.20	0.8540	0.06	0.30	0.9794	0.15	0.85	0.9775
	F	0.26	0.15	0.9308	0.01	0.25	0.9510	0.12	0.10	0.7822
FP	Ca	0.0002	0.15	0.9601	0.0001	0.17	0.9699	0.0001	0.20	0.9626
	Al	0.002	0.30	0.9250	0.0007	0.30	0.9909	0.008	0.30	0.9744
	Si	0.001	0.20	0.9482	0.0008	0.30	0.9514	0.002	0.25	0.9899
	F	0.02	0.05	0.8020	0.07	0.06	0.9523	0.07	0.05	0.9232

lactate (LA_B) and citric acid/sodium citrate (CA_B) buffer solutions. In this study, immersing time was set from 1 h up to 168 h to evaluate the erosion mechanism comprehensively. The results showed that the erosion of KTC and FP glass-ionomer cements in AA_B, LA_B and CA_B

buffer solutions were increased with the immersion time. Örtengren *et al.*²⁹⁾ evaluated the influence of storage time in citric-acid buffer solution of some dental composites and found that the time of storage significantly affects the solubility of the materials. Similarly, surface roughness

of resin-modified glass-ionomer, zinc phosphate and compomer specimens was affected after 15 days storage time in lactic acid solution³⁰. In addition to the immersion time, acidity of the media was postulated to have an effect on the physical properties of the materials. In another study, the tooth specimens, restored with conventional and resin modified glass-ionomer cements, were immersed into acidic drinks repeatedly for 3 times and 5 min to simulate pH cycles in a day period³¹. The specimens were maintained in artificial saliva between each cycle. According to their findings, wear property of the materials did not alter due to the immersion media. However, Da Costa³² suggested that properties of dental materials may be negatively affected by the higher acidity of a beverage than intermediate pH beverage. In fact, the amount of H⁺ ions that designate the pH of the solutions is inversely proportional to the dissolution of the cements in the media⁹.

The buffer solutions used in this study were prepared in solutions of pH: 4.1 values where the acidity could be considered relatively high. The main predisposing factor that initiates disintegration or dissolution of any luting material was attributed to oral pH caused by microorganisms or dietary acids. Organic acids such as lactic and acetic acid are produced by the bacterial activity of the oral environment with the carbohydrate intakes³³. For this reason, the acidic buffer solutions were prepared with similar pH in the present study due to the incident of these media in the oral environment.

Under acidic conditions conventional and resin-modified glass-ionomers are prone to release ions depending on their chemical composition and acidity of the media³⁴. On the other hand, the acids used for preparing the organic buffer solution alter the amount of released element in an organic buffer solution³⁵. The solubility of the elements, Ca, Al, Si and F, in KTC and FP in AA_B, LA_B, CA_B buffer solutions, was the highest in CA_B. The solubility mechanism is related with the H⁺ ions of the solutions that penetrate into the cement to replace with the metal cations^{5,7,36}. The cations diffuse through the cement outward and released from the surface. In addition to this solubility, a second reaction takes place between citric acid anions and metal cations on the cement surface in citric acid buffer solutions⁹.

The highest erosion rates as a function of time were obtained for Al in KTC and the lowest solubility rates for Ca in FP. CA_B solution reacts with metal cations such as Al⁹ and this could be one of the reasons for the eluted amount of the Al in KTC. It has to be added that the amount of Al release was followed by F element release in FP cements. Moreover, the amount of F eluted from FP was close to KTC. While the highest F release was in the 1st h of the immersion time, the total released amount between 1st and 168th h was approximately the one third of the eluted F at the initial storage time. Initial rapid F release from both conventional and resin-modified glass ionomer cement was observed previously³⁷. The eluted F amount in FP was not only associated with the solubility in the acidic media but with the silica barrier matrix being not available in the

conventional glass-ionomers that eventually facilitates the initial rapid F released from FP cement³⁷. Overall, the total amount of released elements from FP was less than those of KTC in all buffer solutions.

Fukazawa *et al.*⁹ described the amount of species eluted from the cement into the acetate buffer solution in relation to the square root of immersion time. According to the equation, when the dissolution was controlled by the diffusion of the species in the cement matrix, *n* is equal to 0.5. When *n* is equal to 1, it can be stated that the dissolution controls both the diffusion and surface reaction, and the amount of elements eluted was then proportional to the reaction time²⁶. As the erosion rates of Ca, Al and Si, in KTC in CA_B solution were in between *n*=0.5–1.00, it is highly probable that solution reactions occurred by both diffusion and surface reaction^{9,26}. Since other KTC and FP components had erosion rates of *n*<0.5 in AA_B, LA_B, CA_B solutions, solution reactions were possibly only diffusion controlled.

The solubility level of KTC cement in organic buffer solutions was found in the order of CA_B>LA_B>AA_B. For FP cement, the solubility sequence of LA_B and AA_B buffer solutions equally affected release of Ca and Si elements. However, the solubility of F and Al showed discrepancy in LA_B and AA_B buffer solutions. F was encountered as the most eluted element for FP cement in CA_B and LA_B buffer solutions. Although the glass component of KTC and FP cements resembles each other, the liquid component of FP cement was different. The liquid component of FP cement contains polyacrylic acid, water-soluble methacrylate monomer (2-hydroxyethyl methacrylate: HEMA) and some amount of catalyst. Thus, during setting reaction of FP cement (resin-modified glass-ionomer cement) goes through both acid-base reaction and resin polymerization reaction. The very slow acid-base reaction between glass powder and liquid is followed by the very rapid resin polymerization. Subsequently, shrinkage due to polymerization accompanies rapid setting reaction that may impair adhesion to dental tissues but with slow acid-base reaction, good adhesion and higher amounts of fluoride release could be achieved¹¹.

CONCLUSION

From this study, the following could be concluded:

1. Ketac-Cem (KTC, conventional glass-ionomer cement) exhibited significantly higher erosion in organic acid buffer solutions when compared to Fuji Plus (FP, resin-modified glass-ionomer-cement).
2. The erosion rates of both FP and KTC in all buffer solutions increased as a function of immersion time and citric acid/sodium citrate solution was found to be more aggressive than those of acetic acid/sodium acetate, lactic acid/sodium lactate solutions.
3. The released F amount in FP was more significant than the other elements but the cumulative F release of FP decreased after the higher burst

occurred in the 1st h.

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