

Study Regarding the Behaviour of Glass-ionomer Cements in Different Acidic Solutions

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The purpose of the study is to assess and compare the effects of different antiseptic mouthrinse solutions on traditional glass-ionomer cements. Thirty samples of three traditional glass-ionomer cements used for base: Ketac Molar Easymix (3M ESPE), for restoration: Fuji IX (GC Corporation) and for sealing: Fuji Triage (GC Corporation) were included into three groups. In group 1, the samples were immersed for 14 days, twice a day one minute each, in Listerine Cool Mint, in group 2 the samples were immersed for 14 days, twice a day one minute each, in Parodontax Extra in group 3, the samples were immersed for 14 days, twice a day one minute each, in Sensodine Cool Mint. In control group the samples were immersed in artificial saliva. Within each group, sub-groups were established according the time of cement ageing (one day, seven days and fourteen days). The samples were analyzed for surface topography using a scanning electron microscope and for chemical composition using EDX detector. The antibacterial mouthrinse solutions Listerine, Parodontax, and Sensodine have an erosive effect on traditional Fuji IX, Ketac Molar and Fuji Triage glass-ionomer cements. The most affected cement by erosive action was Ketac Molar, followed by Fuji IX and Fuji Triage. All three glass-ionomer cements tested in this study proved to be more resistant to erosive action after their ageing.

Keywords: glass-ionomer cement, mouth rinse solution, EDX, SEM

Glass-ionomer cements have been widely used in dental practice, in both conservative dentistry and fixed prosthodontics. The advantages recommending them for clinical use are the following: cariostatic action ascribed to fluoride ion release, their hydrophilic nature and their capacity of adhering to the dental hard tissues [1]. The composition of these cements is varied and complex. Cement is formed following the reaction between aqueous polyacrylic acid and aluminosilicate glass [2]. This is the outcome of an acid-base type of reaction. Fully set glass-ionomer cement may be characterized as a complex where the matrix comprising calcium and aluminum polyacrylate with glass particle incorporates and embeds an unreacted core with surrounding silica gel coating [3, 4].

The property of glass-ionomer cements of preventing the emergence and progress of caries lesions adjacent to restorations as consequence of the long-term fluoride-ion release is very well-known. Previous studies have focused on determining the amount of fluoride released from cements and they insisted on the mechanisms of this mineral loss. Crisp and his collaborators [5], Kuhn and Jones [6], Kuhn and Wilson [7] assessed the mechanisms for erosion of glass-ionomer cements in distilled water. The erosive behaviour of the oral cavity is different from the one exhibited by distilled due to the presence of organic acid and electrolytes. In an attempt to simulate oral conditions, Mesu [8] compared the dissolution rate of certain cements – including of glass-ionomer cement – after immersion in an organic-acid buffer solution. Similarly, Beech and Bandyopadhyay [9] followed the behaviour of glass-ionomer cements in various organic acids or in their buffer solutions. Some studies also reported the amounts of various types of ions released by glass-ionomer cements

after immersing them into an acetate buffer solution [10]. Nonetheless, the mechanism for erosion of glass-ionomer cements is still a matter open to discussions and clarifications.

The purpose of the study is to assess and compare the effects of different antiseptic mouth rinse solutions on traditional glass-ionomer cements.

Experimental part

Material and method

For this study were chosen three traditional glass-ionomer cements used for base: Ketac Molar Easymix (3M ESPE), for restoration: Fuji IX (GC Corporation) and for sealing: Fuji Triage (GC Corporation). Thirty samples were used from each material (1.5-cm long, 7mm-wide, and 0.5-mm thick). The first series included 30 samples made from the Fuji IX cement, the second series comprised 30 samples of the Ketac Molar Easymix cement, while the third series was made of 30 samples of Fuji Triage cement. Samples were made by placing the prepared cement in contact with two transparent celluloid matrices, between two glass plates (to ensure uniformity and to prevent air bubbles). Glass-ionomer cements were prepared by following producer instructions. Subsequently, the samples were randomly included into three groups: group 1 (9 samples), group 2 (9 samples), group 3 (9 samples) and the control group (3 samples). In group 1, the samples were immersed for 14 days, twice a day one minute each, in Listerine Cool Mint (Johnson and Johnson, Maidenhead, UK; series 585330). In group 2, the samples were immersed for 14 days, twice a day one minute each, in Parodontax Extra (Glaxo Smith Kline, Brentford, UK; series 42770670), in group 3, the samples were immersed for 14 days, twice a day one minute each, in Sensodine Cool Mint

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(Glaxo Smith Kline, Brentford, UK; series 4280491). In control group the samples were immersed in artificial saliva throughout the study. The artificial saliva used was AFNOR standard S90-701, with pH = 8.67 and with the following composition: NaCl 0.7g/L; KCl 1.2 g/L; Na₂HPO₄ 0.26 g/L; NaHCO₃ 1.5g/L; KSCN 0.33 g/L; urea 1.35 g/L. Within each group, sub-groups were established as follows: sub-group 1 (samples were immersed in the mouth rinse solution after being stored in artificial saliva for one day); sub-group 2 (samples were immersed in the mouth rinse solution after being stored in artificial saliva for seven days) and sub-group 3 (samples were immersed in the mouth rinse solution after being stored in artificial saliva for 14 days). After preparing, the samples were analyzed for surface topography using a scanning electron microscope Vega II LSH (Tescan, Cech Republic), and for chemical composition using EDX Quantax QX2 detector (Bruker/Roentec, Germany).

Results and discussions

SEM aspects of cement samples in the study groups are presented in figure 1. Round or polygonal pores as a result of glass particles dissolution were present in sub-group 1 (groups 1, 2 and 3, series I, II and III) and in some samples in sub-group 2 (group 2 series I, II and III, group 3 series I).

The mean values of silicon, aluminium, calcium, phosphorus and fluoride ions concentrations expressed in weight percentage (wt%) are presented in table 1.

Cement samples included in group 1, sub-group control recorded the lowest values of aluminium, silicon, calcium, phosphorus and fluoride ions concentrations, irrespective of the series, followed in ascending order by those in sub-group 1, sub-group 2 and sub-group 3. The same tendency of ions concentration variation was recorded in groups 2 and 3.

The immersion of all cement samples in Parodontax Extra antiseptic solution led to a decrease of aluminium, silicon, calcium, phosphate and fluoride ions concentration values compared to control group, irrespective of the duration of ageing time. The variation tendency of the concentration of these ions was similar for all three types of glass-ionomer cement tested. The samples aged for one day recorded the lowest concentration values, followed in ascending order by those aged for 7 days and by those aged for 14 days.

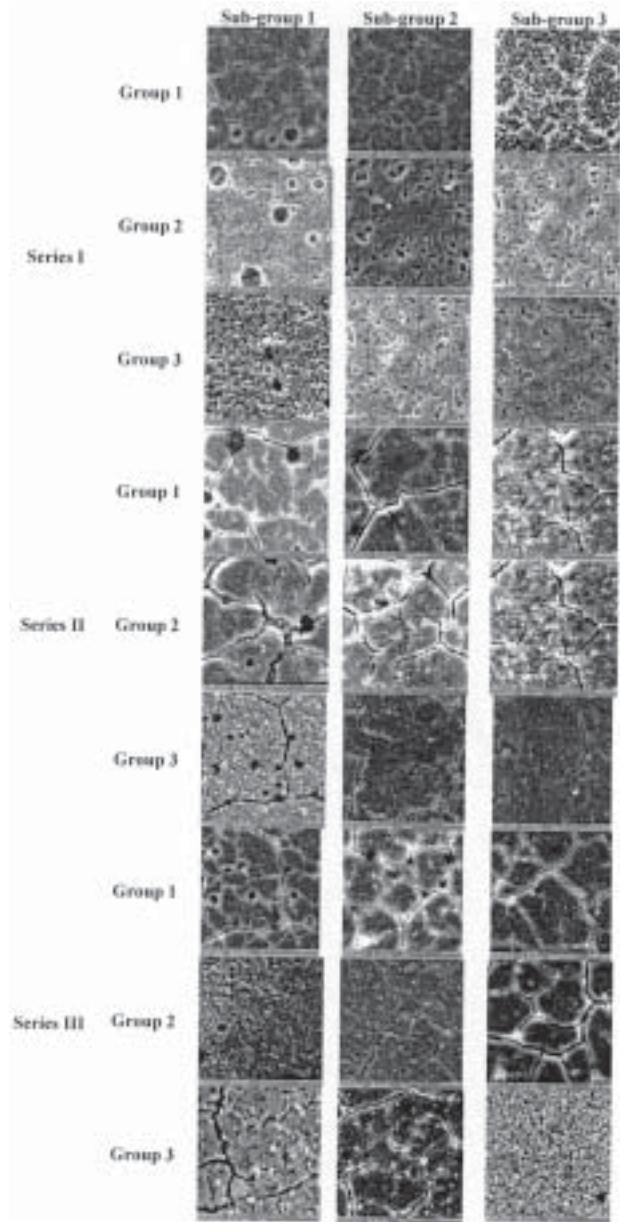


Fig. 1. SEM aspects of cement samples in study groups

Group		Series I				Series II				Series III			
		control	sub-group 1	sub-group 2	sub-group 3	control	sub-group 1	sub-group 2	sub-group 3	control	sub-group 1	sub-group 2	sub-group 3
1	Si	18.56	8.32	16.03	16.78	19.48	5.02	5.97	6.88	19.16	15.27	16.70	18.05
	Al	9.04	7.47	7.97	8.23	8.66	4.97	6.55	6.96	10.13	8.18	8.38	8.45
	Ca	9.17	0.83	1.26	1.24	10.44	0.37	5.86	7.09	3.86	0.32	0.57	0.82
	P	3.53	1.91	1.97	2.28	2.33	0.79	0.86	1.24	1.41	0.72	1.16	1.36
	F	32.44	14.54	15.91	16.68	39.54	22.07	29.54	33.87	21.27	15.52	15.94	16.97
2	Si	18.56	14.49	15.37	15.63	19.48	7.36	8.89	8.98	19.16	14.40	16.24	16.94
	Al	9.04	7.79	7.83	8.73	8.66	5.74	6.75	6.87	10.13	7.86	8.96	9.07
	Ca	9.17	3.05	3.19	4.04	10.83	9.10	10.34	10.49	3.86	2.08	2.80	3.19
	P	3.53	3.13	3.26	3.37	2.66	1.87	1.94	2.33	1.41	0.76	1.27	1.35
	F	32.44	16.14	15.35	14.06	39.54	32.27	32.41	34.08	21.27	16.42	14.87	14.99
3	Si	18.56	14.13	14.45	16.14	19.48	7.43	7.83	8.98	19.16	16.15	14.28	13.97
	Al	9.04	6.12	7.05	7.96	8.66	6.60	6.81	6.87	10.13	7.77	8.15	8.28
	Ca	9.17	3.41	3.60	3.98	10.49	9.35	10.40	10.44	3.86	2.97	3.45	3.75
	P	3.53	3.14	3.36	3.41	2.33	1.53	1.89	1.96	1.41	0.80	1.29	1.36
	F	32.44	14.56	14.84	15.03	39.54	32.39	33.88	36.88	21.27	15.29	15.93	16.14

Table 1
MEAN VALUES (wt%) OF SILICON, ALUMINIUM, CALCIUM, PHOSPHORUS AND FLUORIDE IONS CONCENTRATIONS IN CONTROL AND STUDY GROUPS

For samples immersed in Listerine solution, the same variation of aluminium, silicon, calcium, phosphorus and fluoride ions concentration was recorded for the three types of glass-ionomer cements. A decreasing tendency in the concentration of all tested ions for the samples aged for one day. Concerning the glass-ionomer cement samples aged for seven days, the ions concentrations were higher than the samples aged for one day, but lower than those of samples aged for fourteen days. Regardless of the ageing time of glass-ionomer cements, the concentration of the ions was lower than the one recorded in the control group.

The immersion of the three cements samples in Sensodine antiseptic mouth rinse solution led to a decrease in aluminium, silicon, calcium, phosphorus and fluoride ions concentration compared to control group, irrespective of the duration of ageing time. The variation of the concentration of these ions was similar for all three types of glass-ionomer cement tested. The samples aged for one day recorded the lowest concentration values, followed in ascending order by those aged for 7 days and by those aged for 14 days.

Numerous *in vitro* and *in vivo* studies have assessed the erosive potential of different beverages and foods. They have all demonstrated that the erosive potential of acidic beverages does not depend exclusively on pH values, but that it is also greatly influenced by mineral content and by titratable acidity (buffer capacity), as well as by the chelating properties of calcium ions present in foods and beverages. The oral fluid along with its components represent also a relevant biologic factor for dental erosion [11]. Saliva characteristics, in association with other factors (such as tooth structure, proximal soft tissues and erosive agents *per se*), influence the onset and the evolution of erosive lesions. The corrosive effect of saliva on materials used for restoration was recorded in previous studies [12].

When glass-ionomer cement ions arrive to the interface between cement and solution, they are released in the solution. Their diffusion in the solution is not a rate-dependent process. Starting from the idea that dissolution is controlled by the diffusion that emerges after cement setting seems to be very important (according to the diffusion law determined by Fick), that the amount of ions released in solution depend on the immersion time in solution and the diffusion coefficient. If surface concentration is maintained constant, the total amount of ions released from the cement surface unit should be linear with the square root of the immersion time in solution [13]. These aspects have also been confirmed by subsequent studies [14].

The fluoride-ion release within glass-ionomer cements has represented a research topic for numerous studies. Research reported that fluoride-ion release depends on the alteration of sample thickness: it increases as cement thickness grows [15]. On the contrary, using EDX analysis, other studies reported no alteration in the concentration gradient in glass-ionomer cements after being exposed to drinking water for two months [6]. Fukazawa (1987) demonstrated that dissolution was almost the same in all samples, regardless of their thickness [14]. The amount of ions released per surface unit did not depend upon sample surface, but it was greatly influenced by their form and their volume. Whereas diffusion controls dissolution rate, the dissolution rate should depend upon the surface of exposure in solution.

Glass-ionomer cement has a composite structure with two phases: glass particles embedded in gel matrix that contains polycarboxylic and fluoride complexes [16]. A considerable amount of COOH groups are still unreacted

in the matrix within the cement even 24 h after the setting moment [17]. This suggests that the density of cross-linking in the cement between COOH groups and the polyacids through the links with calcium and aluminum ions is rather low. Previous studies have reported that a considerable amount of these cations was extracted from glass particles in the matrix during the setting reaction. When cement dried up after setting, numerous cracks emerged on the surface. The depth of these cracks depends upon the duration of immersion in solution. However, these cracks have not been noticed when the cement had dried up prior to immersion. This comes to suggest that the solution penetrates the surface cement during immersion, while the matrix gel expands. Studies that analyzed the water absorption of glass-ionomer cements using a dye penetration test proved that after five days infiltration penetrated to a 150 μ m depth [6]. The release rate of ions from cement is controlled by diffusion in cement. It is assumed that ion transfer occurs through diffusion in the expanded matrix. Matsuya et al. (1984) reported that hydrogen ion concentration (H⁺) and that the formation of acid anions and of metallic cations in fully set cement controls cement dissolution rate in acidic organic solutions [17]. Since the occurrence of calcium or aluminum acetate complex constants is relatively low, hydrogen ion concentration is considered to control dissolution. These ions diffuse from the solution into the cement and they make exchanges with calcium or aluminium ions within the matrix. These free metallic cations diffuse outside the cement and they are released from it. For this reason, the diffusion of these ions seems to be influenced by matrix structure and by hydrogen ion concentration on the cement surface. Since the setting reaction of glass-ionomer cements is a long-time process, matrix structure can be altered as the material ages over time. The older the cement, the smaller the amount of aluminium, fluoride and silicon ions released. The amount of calcium ions released is less affected by material ageing. Studies have shown that calcium ions connect to polyacrylic acid within the first minutes of mixing [16], which could explain the lack of concordance between the amount of calcium ions released and the ageing of the material. On the other hand, it was reported that aluminium ions keep on reacting for a longer period, while fluoride ions within the matrix need more time to form insoluble complexes. Because matrix maturation makes ion diffusion difficult, the release of aluminium, fluoride and silicon ions decreases as the ageing time increases. The pH of the solution increased rapidly after the initial immersion, reason for which immersion solutions were changed every time. The immersion in antiseptic mouth rinse solutions led to an increase in the amounts of calcium and aluminium released after 24 hours of immersion. The diffusion rate of aluminium and calcium ions probably increased because of the high concentration gradient maintained by solution change.

In the present study SEM analysis showed the presence of pores following the dissolution of glass particles. Most metallic cations are extracted from glass particles during the setting process. For this reason, particles are rich in silicon and fluoride in the fully set cement, while hydrogen ions diffused in the solution dissolve them, gradually leaving pores near cement surface.

Conclusions

The antibacterial mouth rinse solutions Listerine, Parodontax Extra, and Sensodine have an erosive effect on traditional Fuji IX, Ketac Molar and Fuji Triage glass-

ionomer cements. The most affected cement by erosive action was Ketac Molar, followed by Fuji IX and Fuji Triage. All three glass-ionomer cements tested in this study proved to be more resistant to erosive action after their ageing. The cements aged for 14 days led the lowest variations of silicon, aluminium, calcium, phosphorous and fluoride ions as consequence of the contact with the three oral antiseptic solutions, followed in descending order by cement aged for seven days and for one day, respectively.

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