ORIGINAL ARTICLE

Evaluation of acidulated dentifrice influence on fluoride releasing from glass-ionomer cements

Avaliação da influência de creme dental acidulado na liberação de flúor de cimentos de ionômero de vidro

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ABSTRACT

Objectives: To evaluate the acidulated dentifrice influence on fluoride releasing from glass-ionomer cements. **Methods:** 27 specimens were constructed, divided into 3 groups: G1 (Maxion - R), G2 (Ketac Molar 3M/ESPE) and G3 (Vittremer 3M/ESPE), and subdivided into 3 subgroups (n = 3): A (exposure to acidulated fluoride dentifrice – pH 4.5 / 7h / 37 °C), B (exposure to non-acidulated fluoride dentifrice / 7h / 37 °C), C (control - exposure to artificial saliva). After exposing, the specimens were immersed into artificial saliva and the fluoride releasing measured at the following time periods: 1 to 14 days. Data were subjected to analysis of variance using the Statistical Package for Social Science (SPSS). ANOVA test was applied with level of significance of 5% to compare the means and the behavior of each material. **Results:** The exposure to acidulated dentifrice showed fluoride releasing means values (ppm/mm^2) that varied from 1 and 14 days : G1A (0.269 to 0.204); G2A (0.394 to 0.038); G3A (0.080 to 0.123). The relationship among the G1 subgroups at T1 and G3 subgroups at T1 and T14 was statistically significant. **Conclusion:** The acidulated dentifrice positively influenced on fluoride releasing of hybrid GIC but not on that of conventional GICs.

KEYWORDS

Glass-ionomer cement; Fluoride releasing; Acidulated dentifrice.

RESUMO

Objetivo: Avaliar a influência da exposição a um creme dental acidulado sobre a liberação de flúor de Cimentos de Ionômero de Vidro (CIV). **Material e Métodos:** Confeccionou-se 27 corpos de prova divididos em 3 grupos: G1 (Maxion - R), G2 (Ketac Molar 3M/ESPE) e G3 (Vittremer 3M/ESPE), subdividido em subgrupos (n = 3): A (exposição ao creme dental flúor acidulado – pH 4,5 / 7 h / 37 ºC), B (exposição ao creme dental fluoretado não acidulado / 7 h / 37 ºC) e C (controle - exposição à saliva artificial). Após exposição aos cremes dentais, os corpos de prova foram imersos em saliva artificial e aferidas a liberação de flúor nos períodos de tempo: 1 e 14 dias. Os dados obtidos foram submetidos a análise de variância, utilizando o pacote SPSS (Statiscal Package for Social Science). Aplicou-se o Teste ANOVA com 5% de probabilidade, para comparação das médias e do comportamento de cada material. **Resultados:** A exposição ao creme dental ácido apresentou valores de liberação de flúor (ppm/mm^2) que variaram, em suas médias, de 1 e 14 dias: G1A (0,269 a 0,204); G2A (0,394 a 0,038); G3A (0,080 a 0,123). Foi estatisticamente significante a relação entre os subgrupos para G1 em T1 e G3 em T1 e T14. **Conclusão:** a exposição ao creme dental acidulado influenciou positivamente a liberação de flúor do CIV híbrido testado e não impactou a liberação deste íon nos CIVs convencionais.

PALAVRAS-CHAVE

Cimento de Ionômero de Vidro; Liberação de Flúor; Creme Dental Ácido.

INTRODUCTION

Fluoride is a successfully preventive/ therapeutic agent mostly impacting on both the people's health and quality of life [1]. On the nature, fluoride is a gas; in Dentistry, fluoride prevents caries by reducing hydroxyapatite solubility, balancing the demineralization/ remineralization rate, and stabilizing the crystalline structures of the enamel which become more resistant to the acid challenges from bacteria. The rationale behind the anticaries effect of fluoride is the interference with the bacterial metabolism [2,3,5,6]. Accordingly, fluoride releasing from restorative materials may affect the development of caries lesions through mechanisms that reduce or prevent demineralization [6,7].

Among the restorative materials, either conventional or resin-modified (hybrid) glass ionomer cements (GICs) demonstrated greater fluoride releasing capacity. Because of the slowest GIC setting reaction, a greater active ion displacement (including fluoride) occurs at the initial phases of gelation, mainly at the first 24 h.

GICs also have the capacity of acquiring fluoride from different sources, storing and constant releasing fluoride to oral medium [13,19]. Fluoridated water, gels, varnishes, rinses, or dentifrices may contribute to this phenomenon. The pH of these vehicles seems to influence the fluoride releasing more than the concentration. For example, the topical application of 1.23% acidulated phosphate fluoride gel promoted a higher fluoride releasing from GICs than the exposure to 2% neutral NaF gel [13]. In this context, one would consider whether a low pH dentifrice caused the same GIC behavior. This study aimed to evaluate the influence of acidulated dentifrice exposure on fluoride releasing from glass ionomer cements.

MATERIALS AND METHODS

This study employed a Teflon matrix to construct 27 cylindrical samples (d = 5 mm and h = 2 mm), divided according to Chart 1.

Chart 1 - Distribution of groups and subgroups

GROUP	GIC	Setting	SUBGROUP	AMOUNT / EXPOSURE MEDIUM / pH	TIME / EXPOSURE TEMPERATURE	N
1	Maxxion R – FGM Joinville – Santa Catarina - Brazil (Conventional)	Acid-base reaction Setting time = 06 min	A	20 ml / acidulated dentifrice solution (Phormula Ativa-Recife-PE-Brazil) / pH 4.5		03
			B	20 ml / non-acidulated dentifrice solution (Phormula Ativa-Recife-PE-Brazil) / pH 10		03
			C – control	20 ml / artificial saliva solution (Phormula Ativa-Recife-PE-Brazil) / pH 7.0		03
2	Ketac Molar - 3M/ ESPE Saint Paul – Minnesota - USA (Conventional)	Acid-base reaction Setting time = = 05 min	A	20 ml / acidulated dentifrice solution (Phormula Ativa-Recife-PE-Brazil) / pH 4.5	07 consecutive hours = contact time corresponding to 20,000 toothbrushing cycles / 37 °C	03
			B	20 ml / non-acidulated dentifrice solution (Phormula Ativa-Recife-PE-Brazil) / pH 10		03
			C – control	20 ml / artificial saliva solution (Phormula Ativa-Recife-PE-Brazil) / pH 7.0		03
3	Vitremer – 3M/ ESPE Saint Paul – Minnesota - USA (Resin-modified)	Triple Setting Reaction: light-curing, self-curing, and acid-base reaction Light-curing time = 40 s.	A	20 ml / acidulated dentifrice solution (Phormula Ativa-Recife-PE-Brazil) / pH 4.5		03
			B	20 ml / non-acidulated dentifrice solution (Phormula Ativa-Recife-PE-Brazil) / pH 10		03
			C – control	20 ml / artificial saliva solution (Phormula Ativa-Recife-PE-Brazil) / pH 7.0		03

GICs were mixed on cooled glass plate following the manufacturers' instructions and inserted into the Teflon matrix with a nylon thread (to fix the sample) supported on a polyester strip and a thick glass plate. A second polyester strip and glass plate were placed over the matrix together with a constant weight of 1 kg until the material setting. A LED device (ultraLED Gnatus) was used to light-cure the resin-modified GIC (1400 mW/cm²).

Next, the samples were stored in a humidifier (40 min), had the excesses removed with scalpel blade, and weighed in analytical scale (Scout Pro-Okaus) (mean weight = 0.11 g). Then, through the nylon thread and with the aid of utility wax, the specimens were fixed on polyethylene flask lids properly identified, kept suspended taking care not to touch the flask walls, and exposed to the study solutions. After that, the specimens were washed in deionized water, dried in absorbent paper, and kept in 20 ml of artificial saliva (pH 7.0 / 37° C), daily changed. Fluoride releasing was measured at 1 (T1) and 14 (T14) days. To perform the readings, the flasks were kept at 4 °C.

All fluoride measurements were performed in triplicate using fluoride-selective electrodes coupled to digital pH/F⁻ meter (Orion 230A, Thermo Scientific, San Jose, CA, USA), previously calibrated with a series of standard solutions with the following F⁻ concentrations: 0.4; 0.8; 1.6; 3.2; 6.4 ppm, after buffering with TISAB II (*Total Ionic Strength Adjustment Buffer*), at 1:1 ratio. To validate the analysis, the standard solutions were prepared by serial diluting a solution of 100 ppm of fluoride (Orion). After each reading the electrodes were washed in deionized water and dried in paper towel and the test solution was discarded. All this procedure was executed for the solutions to be measured.

The values (mV) were recorded in Excel sheet (Microsoft) containing the data of the standard solutions with known F⁻ concentrations to obtain the amount of fluoride released in ppm (μgF). Then, these values were divided by the surface area of the samples through the following formula: AT = LA + 2BA, where LA = lateral area, BA = base area, AT = $2.\pi.r$ (h+r), where h = height and r = radius. Thus, AT=1.099 cm². The mean of the readings obtained from the standard solutions were used to calculate the percentage of change between the amount of fluoride measured and that expected by the standards. Only calibration curves with percentage of changes up to 10% for all standards were accepted.

The obtained data were submitted to Analysis of Variance followed by Tukey test with level of significance of 5% to compare the means and behavior of each material (Statistical Package for Social Science) to verify the statistically significance differences.

RESULTS

Tables 1, 3, and 4 showed the fluoride releasing results for the glass ionomer cements Maxxion R (FGM), Ketac molar (3M/Espe), and Vitremer (3M/Espe) respectively, for the evaluation periods of 01 and 14 days, considering the subgroups according to the exposure medium (A – acidulated dentifrice solution / Phormula Ativa-Recife-PE-Brazil; B – non-acidulated dentifrice solution / Phormula Ativa-Recife-PE-Brazil; and C - artificial saliva solution / Phormula Ativa-Recife-PE-Brazil). All studied GICs exhibited the greatest releasing values at the first 24 h than those at the 14th day for all subgroups, except for subgroups A and B from group 3 (Vitremer, 3m/Espe). Table 1 evidenced the statistical difference between the evaluations at the first day. Table 2 displays the result of Tukey test to evidence which Group 1 (Maxxion R, FGM) subgroup accounted for the statistical difference at the first day. The relationships between Group 1 subgroups A with C and B with C were statistically significant. The results in Table 3 (Ketac molar - 3M/Espe) showed no statistically significant difference

among subgroups (ANOVA), so that a post-hoc test was unnecessary. Group 3 (Vitremer, 3M Espe) showed statistically significant differences (ANOVA) between subgroups at the periods of 01 and 14 days (Table 4). According to Table 5, Tukey test evidenced statistically significant difference between subgroup A with C and B with C at the first day. At 14 days, statistically significant differences occurred between subgroups A with C.

Table 1 - Mean and standard deviation of fluoride releasing (ppm/mm²) of Group 1 - Maxxion R - FGM (n = 3) after 01 and 14 days

Time	1 day	14 days
Subgroup		
A	0.269 (± 0.009)	0.204 (± 0.06)
B	0.302 (± 0.03)	0.223 (± 0.03)
C	0.473 (± 0.08)	0.311 (± 0.22)
	p = 0.005*	p = 0.063

*Statistically significant differences (ANOVA).

Table 2 - Difference among the fluoride releasing means (ppm/mm²) of Group 1 - Maxxion R - FGM (n = 3) after 01 day

Subgroups	Subgroup Comparisons	Differences between means	p value
A	B	-0.033	0.713
	C	-0.208	0.006*
B	A	0.033	0.713
	C	-0.174	0.013*
C	A	0.208	0.006*
	B	0.174	0.013*

* Statistically significant differences (Tukey test).

Table 3 - Mean and standard deviation of fluoride releasing (ppm/mm²) of Group 2 - Ketac Molar - 3M/Espe (n = 3) after 01 and 14 days

Time	1 day	14 days
Subgroup		
A	0.394 (± 0.15)	0.038(± 0.09)
B	0.311(± 0.003)	0.040(± 0.008)
C	0.116(± 0.88)	0.063 (± 0.02)
	p ͫ 0.164	p ͫ 0.190

Table 4 - Mean and standard deviation of fluoride releasing (ppm/mm²) of Group 3 - Vitremer - 3M/Espe (n = 3) after 01 and 14 days

Time	1 day	14 days
Subgroup		
A	0.080 (± 0.007)	0.123(± 0.001)
B	0.058(± 0.01)	0.094(± 0.006)
C	0.198(± 0.04)	0.083 (± 0.02)
	p ͫ 0.001*	p ͫ 0.036*

* Statistically significant differences (ANOVA).

Table 5 - Difference among the fluoride releasing means (ppm/mm²) of Group 3 - Vitremer - 3M/Espe (n = 3) after 01 and 14 days

Subgroup		Subgroup Comparisons	Differences between means	p value
01 day	A	B	0.022	0.589
		C	-0.117	0.004*
	B	A	-0.022	0.589
		C	-0.140	0.002*
	C	A	0.117	0.004*
		B	0.140	0.002*

Subgroup		Subgroup Comparisons	Differences between means	p value
14 days	A	B	0.026	0.111
		C	0.037	0.034*
	B	A	-0.026	0.111
		C	0.010	0.633
	C	A	-0.037	0.034*
		B	-0.010	0.633

* Statistically significant differences (Tukey test).

DISCUSSION

Fluoride-releasing restorative materials are increasingly necessary to maintain fluoride availability in oral medium to control dental caries therapeutically [17].

The results obtained in this study corroborates the literature by showing that all tested GICs (conventional and resin-modified) released fluoride, with higher means in control subgroups (C), at the first 24 h (T1) decreasing

at 14 days (T14), because the greater displacement due to leaching of active ions at the initial moments of the material setting. Gradually, the ions react and the fluoride releasing decreases [13].

Musa et al. [15] reported that the resin of hybrid GICs negatively influence on the fluoride releasing. However, Momoi, Mccabe [16] verified that the potential of fluoride releasing is similar for both conventional and resin-modified GICs, a phenomenon close to that seen by this study.

The constant maintenance of this therapeutic property of conventional or resin-modified GICs can be attributed to the capacity of recharging with fluoride ions, which is possible due to the deposition of extrinsic ions to GIC composition after the releasing of intrinsic fluoride to oral cavity [14]. The literature affirms that fluoride sources with low pH greatly influence on the fluoride uptake and releasing of GICs, regardless of the concentration [8,13]. Notwithstanding, the results of this study demonstrated that the acidulated dentifrice did not positively influence on fluoride releasing over time for the tested conventional CIGs. On the other hand, fluoride releasing values were higher and statistically significant after exposure to acidulated dentifrice at T14 for resin-modified GIC. This fact can be justified by the resin presence in this material (G3), resulting in smaller porosity than that of conventional GICs (G1 and G2) and, thus, in smaller fluoride releasing at the initial time periods in subgroups A and B. The low pH would increase the material dissolution leading to a higher level of fluoride availability [5], which could explain the behavior of resin-modified GIC after acidulated dentifrice exposure at T14. In the continuous preventive action against caries, detectable fluoride releasing for longer periods is better than higher fluoride releasing for shorter periods. Accordingly, fluoride uptake is necessary to maintain fluoride releasing and protect against demineralization [18-19].

The results of this study guided the behavior of GICs after acidulated dentifrice exposure, but further laboratorial and clinical studies are necessary because the literature lacks information on this issue.

CONCLUSION

The acidulated dentifrice exposure positively influence on the fluoride releasing from resin-modified GIC, but did not stimulate the fluoride releasing from conventional GICs.

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