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Influence of the Curing Mode on Fluoride Ion Release of Self-adhesive Resin Luting Cements in Water or During pH-Cycling Regimen

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Clinical Relevance

Within the limited experimental conditions, the self-adhesive cements provided fluoride ion release capacity. The fluoride release for all resin cements was not uniform during pH cycling, decreasing throughout the duration of the study.

SUMMARY

This study evaluated the effects of curing modes and storage conditions on fluoride release of resin cements. In phase 1, the cumulative fluoride release rate from samples of the resin cements (Panavia F 2.0, RelyX Unicem, MaxCem, and BisCem) was quantified after 15 days storage in water (n=4). In phase 2,

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the fluoride release profiles from the same materials were analyzed during pH cycling (n=4). In this second phase, fluoride was measured at specific times (one, two, three, five, eight, and 15 days). Disk-shaped specimens were prepared (10 mm × 0.5 mm), and the materials were either light activated or allowed to autopolymerize. For both phases, the fluoride release was measured using a fluoride ion-specific electrode. The fluoride release in water was not affected by the curing mode of RelyX Unicem and Maxcem resin cements. Panavia F. 2.0 and BisCem resin cements, either light cured or autopolymerized modes, released higher amounts of fluoride in water than the other self-adhesive cements. In phase 2, the concentration of fluoride released decreased from the first day of pH cycling until the 15th day for all resin cements, for both curing modes, regardless of the storage solution used (demineralizing/remineralizing). The fluoride release rate during pH cycling by Panavia F 2.0 and MaxCem was not affected by the curing mode. The effect of the curing mode on fluoride ion release in water or during pH cycling was product dependent.

INTRODUCTION

Fluoride application to tooth structures has influenced the decline in the prevalence and severity of dental caries.[1,2] Many studies have shown that the ion is safe and effective in preventing and controlling caries development under specific dosages. Such benefits are due to its ability to inhibit the demineralization and enhance remineralization.[3-5]

A number of studies have suggested that the fluoride released from glass ionomer cements can reduce the demineralization of the surrounding dentin or enamel and favor the remineralization of lesions close to the restoration wall.[6-9] Glass ionomer cements contain calcium fluoroaluminosilicate glass, which is attacked by polyacids to release cations and fluoride ions. These ions react with polyanions to form a salt gel matrix that presents small particles of silica gel containing fluoride crystallites. The preventive and therapeutic effects of glass ionomer cements are attributed to the discharge of fluoride ions, and this releasing does not affect the physical properties of the cement.[10]

Other fluoride-containing restorative materials, such as restorative composites, resin cements, and adhesive systems, have been developed to prevent recurrent caries; however, glass ionomer cements show a fluoride-recharging ability and have demonstrated more prolonged fluoride release than these materials. Most of these resin-based materials contain different fluoride sources, and the amount of fluoride released seems to depend on the brand and other characteristics of each material.[9,11-15]

The self-adhesive resin cements were recently developed, and the main advantages of using these materials include a shorter clinical application time and technique sensitivity reduction.[16-19] In addition, self-adhesive resin cements contain different fluoride sources, and limited information is available regarding their ability to release sufficient fluoride concentrations in water or during a cariogenic challenge. Thus, the purpose of this study was to evaluate the effects of curing modes and storage conditions on fluoride release from dual-polymerizing resin cements. The research hypotheses tested were that 1) curing mode affects the amount of fluoride release in water, regardless of type of resin cement used, and 2) the fluoride release profile of resin cements is uniform during pH cycling, regardless of the resin cement and the curing mode.

METHODS AND MATERIALS

Sample Preparation

Three self-adhesive dual-polymerizing resin cements, RelyX Unicem (3M ESPE, Seefeld, Germany), MaxCem (Kerr Corp, Orange, CA, USA), and BisCem (Bisco Inc, Schuamburg, IL, USA), and one conventional dual-polymerizing cement (Panavia F 2.0, Kuraray Med, Kurashiki, Japan) were used according to the manufacturers' instructions (Table 1).

Sixteen disks of each resin cement were prepared (total = 64 samples). The mixed base and catalyst pastes were dropped into individual cylindrical silicon molds (10.0 mm diameter × 0.5 mm depth, Aquasil Ultra LV, Dentsply Caulk, Milford, DE, USA), covered with a polyester strip and pressed with one microscope glass slide. Half of the total samples (32 discs) were light cured for 40 seconds (XL 3000, 3M ESPE, St Paul, MN, USA), and the other half were allowed to autopolymerize only. Thirty-two resin cement discs were evaluated according to the cumulative fluoride release rate in water, and thirty-two samples were prepared for fluoride release profiles during the pH-cycling regimen. All resin cement samples were stored at 37°C for 48 hours (n=4). A schematic diagram of the study design is shown in Figure 1. After curing, the disk-shaped samples were removed from the silicon molds, and the excess material was removed using

Table 1: *Compositions of the Resin Cements Used in This Study*

Resin Cement	Composition
Panavia F 2.0	Paste A: 10-MDP, silanated colloidal silica, bisphenol A polyethoxy dimethacrylate, hydrophobic and hydrophilic DMA, silanized silica filler, benzoyl peroxide, dl-camphorquinone
	Paste B: hydrophobic and hydrophilic DMA, sodium 2,4,6–triisopropyl benzene sulphinate, N,N-diethanol-p-toluidine, bisphenol A polyethoxy dimethacrylate, colloidal silica, sodium fluoride, silanized barium glass filler, silanized titanium oxide
RelyX Unicem (capsules)	Power: glass powder, silica, calcium hydroxide, self-curing initiators, pigments, light-curing initiators, substituted pyrimidine, peroxy compound
	Liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-curing initiators, light-curing initiators
MaxCem	Multifuncional DMAs, GPDM, proprietary Redox initiators and photoinitiators, barium, fluoroaluminosilicate, fumed silica (66 wt.%)
BisCem	Bis-GMA, uncured dimethacrylate monomer, phosphate acid monomer, glass filler

Abbreviations: 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol-A-diglycidylether dimethacrylate; DMA, dimethacrylates; GPDM, glyceroldimethacrylate dihydrogen phosphate; TEGDMA, triethylene glycol dimethacrylate.

600-grit SiC paper. Samples were individually fixed to orthodontic steel wires, allowing each to be suspended in each storage media.

Cumulative Fluoride Release Rate in Water

The self-cured and light-activated samples (n=4) were placed into polyethylene vials containing 2 mL of deionized water and individually stored for 15 days. The water was not renewed daily, and 1 mL of each vial was collected for the analysis of fluoride release. TISAB III (total ionic strength adjustment buffer, Orion Research Inc, Cambridge, MA, USA) was added to the collected samples at a ratio of 1:10 (buffer:sample), and the quantification of fluoride in water was carried out by an ion-selective electrode (96-06, Orion Research Inc, Cambridge, MA, USA), connected to an ion analyzer (Orion 720 A model potentiometer, Orion Research Inc, Cambridge, MA, USA), which was previously calibrated with a series of standard solutions. The concentration of fluoride released was expressed in $\mu g/cm^2$, and the data were analyzed by Kruskal-Wallis and Dunn tests ($p<0.05$).

Fluoride Release Profile During pH-Cycling Regimen

The self-cured and light-activated samples were submitted to 15 days of a pH-cycling regimen, simulating caries development (n=4).[9,20] Each day or cycle consisted of the individual immersion of the disk-shaped specimen in demineralizing solution (1.4 mM Ca, 0.9 mM P, 0.05 m acetate buffer, pH 5.0, 2 mL per specimen) for eight hours and remineralizing solution (1.5 mM Ca, 0.9 mM P, 0.1 M Tris buffer, pH 7.0, 2 mL per specimen) for 16 hours. The solutions (demineralizing and remineralizing) were renewed daily, and 1 mL of each solution was collected at the first, second, third, fifth, eighth, and 15th days for the analysis of fluoride release, which was carried out by the same methodology previously used.

Fluoride release was also recorded in $\mu g/cm^2$. Mann-Whitney and Wilcoxon tests were used to analyze the influence of curing modes and storage media (demineralizing and remineralizing solutions) on fluoride release, respectively ($p<0.05$). The Friedman Non-Parametric Repeated Measures Comparisons analyzed the fluoride release profile during the pH-cycling regimen ($p<0.05$).

RESULTS

The results of the experimental groups and the amount of fluoride released after 15 days of storage in deionized water for each resin cement in both curing modes are displayed in Table 2. The fluoride release of RelyX Unicem and MaxCem self-adhesive resin cements was not affected by curing mode, while light activation significantly increased the fluoride releasing of Panavia F 2.0 and decreased the

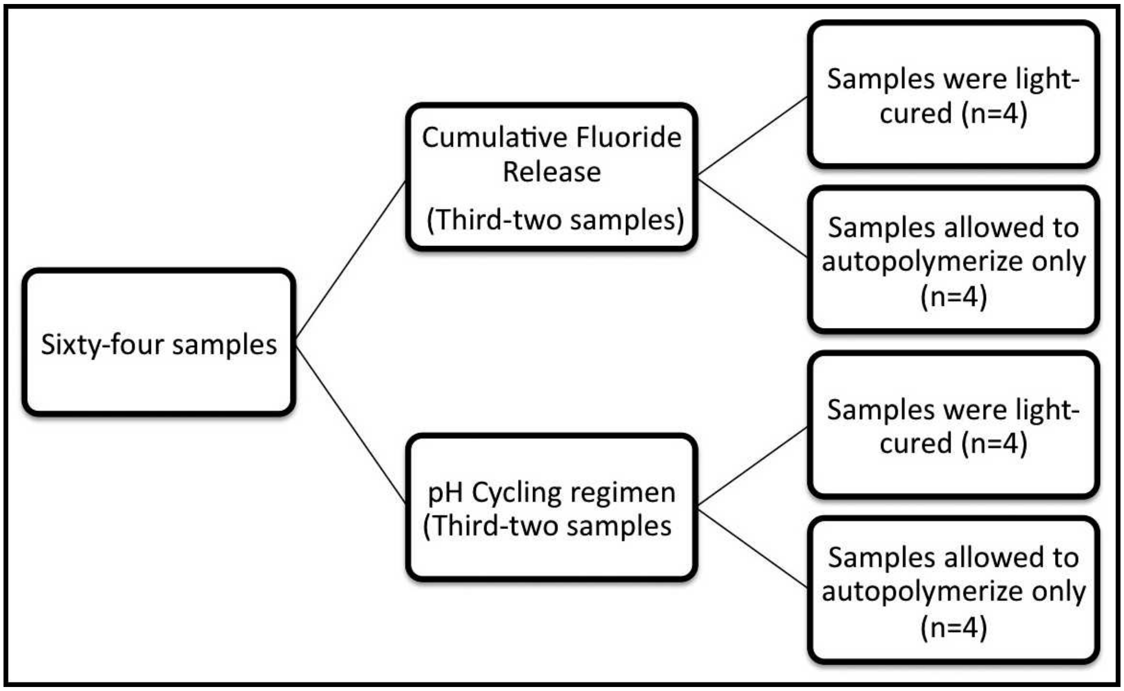

Figure 1. *Schematic diagram of study design: Four resin cements were tested in this study (Panavia 2.0, RelyX Unicem, MaxCem, and BisCem).*

releasing for BisCem self-adhesive cement. BisCem and Panavia F 2.0 released more fluoride ions in water than RelyX Unicem and MaxCem self-adhesive resin cements in both curing modes.

The fluoride release profiles during the pH-cycling regimen of the resin cements at each period are shown in Figures 2 (Panavia F 2.0, RelyX Unicem), 3 (MaxCem), and 4 (BisCem). In general, the concentration of fluoride released tended to decrease significantly from the first to the 15th day of pH cycling, regardless of the curing mode and storage solution (demineralizing/remineralizing) used.

For Panavia F 2.0 and MaxCem resin cements (Figures 2 and 4, respectively), the amount of fluoride released in the demineralizing or remineralizing was not affected by the curing mode at any time. Also, the fluoride concentration in the remineralizing solutions of these resin cements was higher than in the demineralizing solutions on the first day. At the second day, the four groups of Panavia F 2.0 resin cement released similar concentrations of fluoride until the 15th day, which tended to reduce the concentration to zero.

Fluoride-releasing behavior for RelyX Unicem during the pH-cycling regimen (Figure 3) showed that the autopolymerized samples released more fluoride ions in both demineralizing and remineralizing solutions than light-activated samples at the first day. RelyX Unicem and MaxCem self-adhesive resin cements continued to release fluoride until the 15th day, and the concentration measured in the solutions varied from 0.015 to 0.005 $\mu g/cm^2$ (Figures 3 and 4, respectively). From the second day until the 15th day, the four groups of RelyX Unicem or MaxCem cements presented similar mean values of fluoride release.

The fluoride release profile during the pH-cycling regimen for BisCem self-adhesive resin cement indicated that light activation reduced the fluoride release

Table 2: *Mean (Standard Deviation) of Cumulative Amounts of Fluoride Ions Released ($\mu g/cm^2$) From Resin Cements (Light or Self-cured) Over 15 Days[a]*

	Curing Mode	
Resin Cements	**Light Activated**	**Autopolymerized**
Panavia F. 2.0	9.5 (0.4) Aa	6.5 (0.1) Bb
RelyX Unicem	1.3 (0.2) Ab	1.3 (0.1) Ac
MaxCem	3.0 (0.2) Ab	2.7 (0.2) Ac
BisCem	8.9 (0.3) Ba	13.6 (0.6) Aa

[a] *Similar letters (uppercase, row; lowercase, column) are not statistically different ($p>0.05$).*

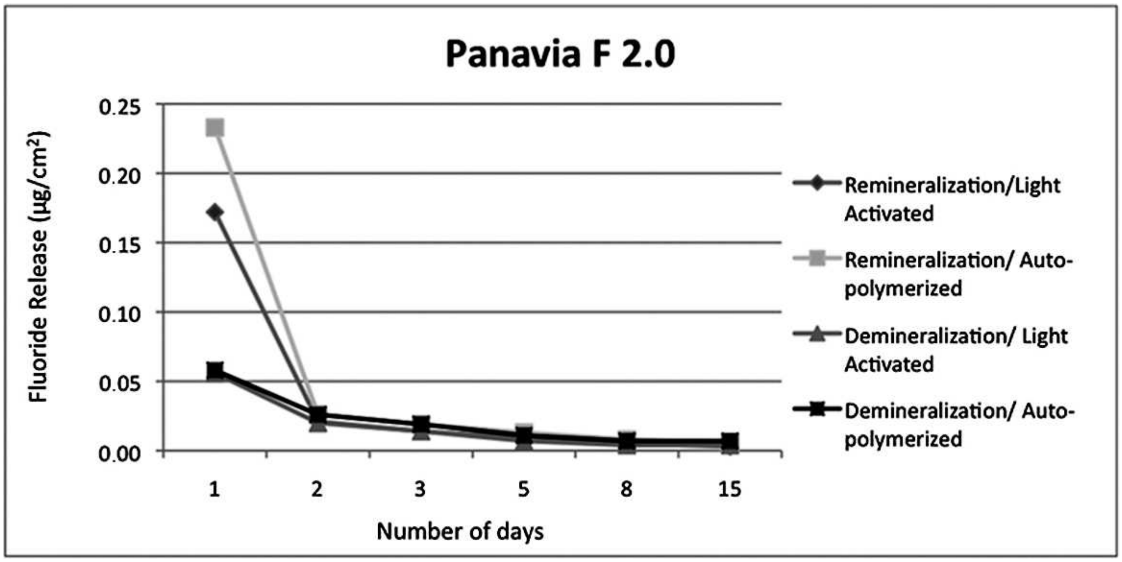

Figure 2. *Fluoride-releasing behavior for Panavia F 2.0 during the pH-cycling regimen (fluoride released [μg/cm^2] as a function of elapsed time for up to 15 days).*

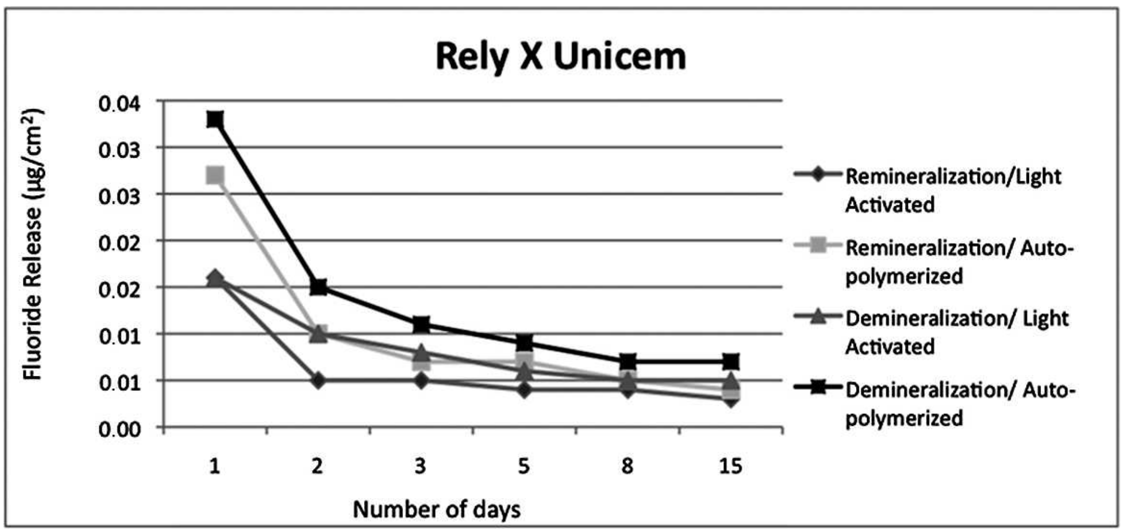

Figure 3. *Fluoride-releasing behavior for RelyX Unicem during the pH-cycling regimen (fluoride released [μg/cm^2] as a function of elapsed time for up to 15 days).*

rate in both demineralizing and remineralizing solutions (Figure 5) at the first day. A low concentration of fluoride release was observed at the 15th day for all groups of BisCem cement (Figure 5).

DISCUSSION

The first hypothesis was partially accepted since fluoride release from the resin cements, RelyX Unicem and MaxCem, was not influenced by the curing modes. These cements released the lowest amount of fluoride in water, and no differences were observed between them in either curing mode. The water did not solubilize the high amount of fluoride ions from the polymerized resin matrix, even in an autopolymerizing mode that, theoretically, increases the resin matrix permeability for fluoride release.[21] On the other hand, BisCem had its fluoride release increased with the self-curing mode, and Panavia F 2.0 presented the opposite effect. BisCem and Panavia F 2.0 cements released more fluoride ions to water than RelyX Unicem and MaxCem self-adhesive resin cements in both curing modes. The composition of each resin cement, the solubility and permeability of the resin matrix, and the source,

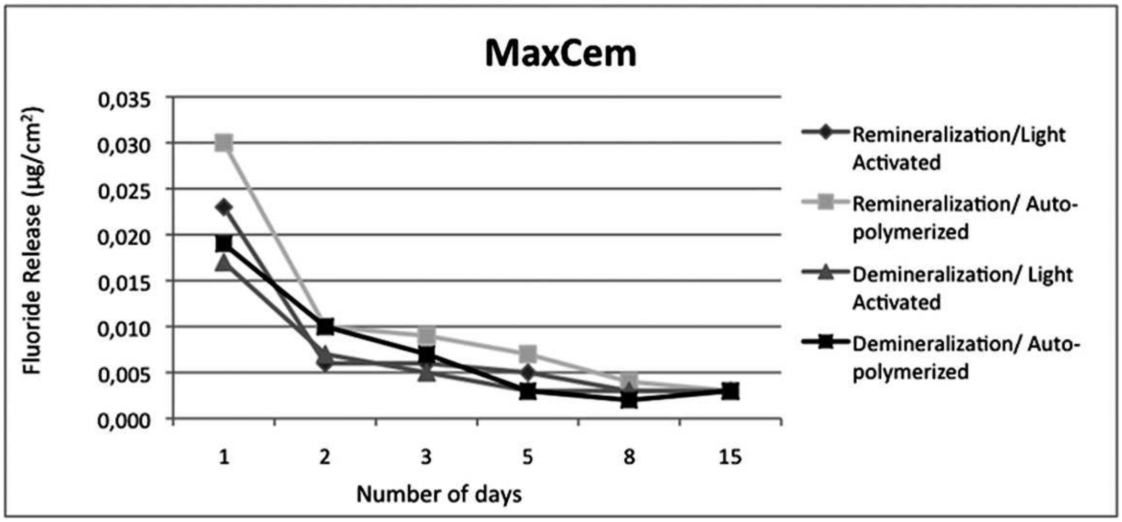

Figure 4. *Fluoride-releasing behavior for MaxCem during the pH-cycling regimen (fluoride released [μg/cm²] as a function of elapsed time for up to 15 days).*

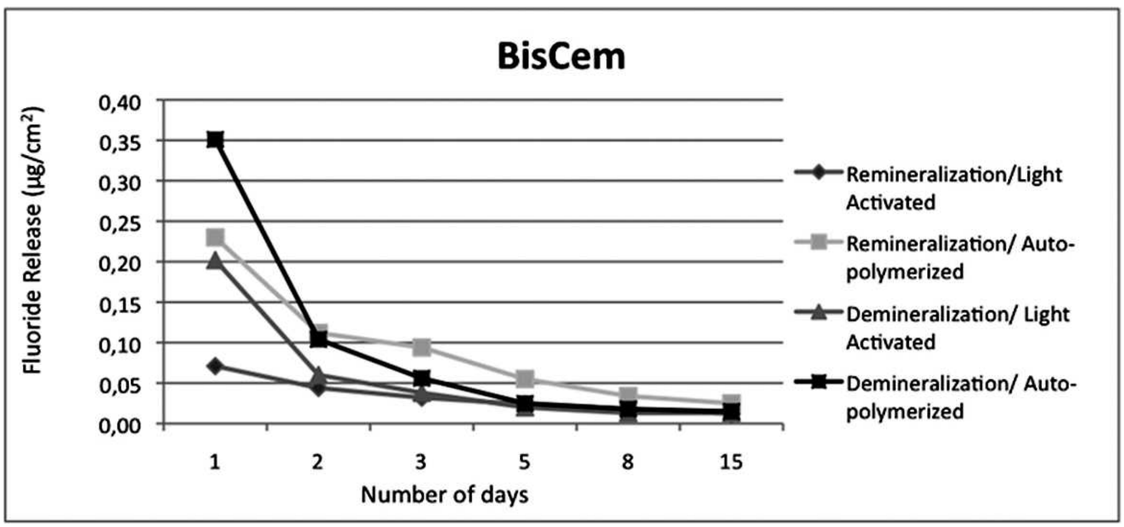

Figure 5. *Fluoride-releasing behavior for BisCem during the pH-cycling regimen (fluoride released [μg/cm²] as a function of elapsed time for up to 15 days).*

size, and concentration of the fluoride ions are important characteristics of the fluoride-releasing materials related to their ability to release the ions and, consequently, for the results of the first phase of this study.[9,14,21-23]

The fluoride source of Panavia F 2.0 is the sodium fluoride salt, which can be easily released by ionization when in contact with water or other aqueous solutions. For RelyX Unicem, the fluoride source is the glass powder, and the fluoride is available from similar acid-base reactions of glass ionomer cements.[17,24,25] MaxCem resin cement contains fluoroaluminosilicate particles that are similar to the glass powder found in RelyX Unicem. The similar fluoride source of both materials may explain the similar cumulative rate of fluoride released in water for RelyX Unicem and MaxCem (Table 2). BisCem self-adhesive resin cement contains glass fillers, which can be composed of fluoride glass. The glass fillers are presented in both base and catalyst pastes of BisCem cement and may be in relative concentration, which explains the amount of fluoride released in water.

Several studies have reported that the glass powder serves as a reservoir of fluoride.[26,27] RelyX Unicem and MaxCem self-adhesive resin cements that contain these glass particles undergo a significant reduction in fluoride release during pH cycling, but it did not tend to zero after 15 days of cycling. For Panavia F 2.0 and BisCem cements, very low concentrations of fluoride were detected at the last day of cycling, showing that releasing did not remain constant for 15 days and the fluoride source of these materials seems to be finite.[21,22] As observed in similar fluoridated resin-based studies,[9,22,28] higher ion release was observed on the initial days for all resin cements tested in this study, demonstrating that the fluoride release was not constant or uniform during the pH-cycling regimen (Figures 2-5). Since the fluoride release decreased throughout the duration of the study, regardless of the resin cement and the curing mode used, the second research hypothesis was rejected.

In this study, the storage medium (demineralizing or remineralizing solution) influenced the amount of fluoride released only for Panavia F 2.0 and MaxCem resin cements during pH cycling. At the first day, higher amounts of fluoride ions were observed in the remineralizing solution than in the demineralizing solution. These results significantly differ from the fluoride release behavior of glass ionomer cements during pH cycling, in which the fluoride release rate increases in acidic conditions (demineralizing solutions), especially in organic acids, such as acetic, lactic, and citric acid solutions.[29-31]

Yoda and others[21] demonstrated that the curing mode and storage medium influenced the amount and rate of fluoride release from fluoridated luting cements materials (Fuji II LC glass ionomer cement, GC Corp, Tokyo, Japan, and Panavia F resin cement, Kuraray Med). The authors stated that the light curing of the dual-cure resin cement was essential to enhance the mechanical properties and increase immediate bonding to tooth structures. However, when the polymerization is only chemical, an increase of fluoride release is observed since light activation enhances cross-linking density and network quality,[32,33] resulting in a reduction of resin matrix permeability for fluoride ion release.

The effects of curing modes on the degree of conversion of the same resin cements was previously evaluated by Aguiar, and the results for RelyX Unicem and BisCem self-adhesive resin cements showed that the autopolymerizing mode yielded a lower degree of conversion value than did light activation ($p<0.05$).[34] The lower degree of conversion of self-curing mode can increase the resin matrix permeability for fluoride release, which may explain the increase of fluoride from the self-cured samples.[21] In both phases of this study (first phase, cumulative fluoride release rate in water; second phase, fluoride release profile during pH cycling), the BisCem cement released a higher fluoride concentration in self-curing mode. Conversely, the RelyX Unicem was not influenced by water media, and a higher concentration of fluoride was found for self-cured samples at the first day of pH cycling. Moreover, the acidic conditions of pH cycling possibly promoted more fluoride release. Since RelyX Unicem contains ionomeric components, this resin cement may show similar fluoride-releasing behavior as glass ionomer cements, which present an increased fluoride-releasing rate in acidic conditions.[29-31]

CONCLUSION

The analysis of fluoride release from resin-based materials is complex and depends on some clinical variables that are not present in an evaluation of glass ionomer cements. Only one resin cement (BisCem) was affected by the curing mode in both storage conditions (water and pH cycling). This resin cement released a higher amount of fluoride in a self-curing mode. The storage conditions changed the fluoride release profile of Panavia F 2.0 and MaxCem resin cements. For all tested materials, the highest fluoride release was observed at initial days of the study, tending to gradually diminish with time, according to the profile of each material.

Conflict of Interest Declaration

The Authors of this manuscript certify that they have no proprietary, financial or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

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