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Effects of erosive challenge on the morphology and surface properties of luting cements

Efeito do desafio erosivo na morfologia e propriedades de superfície de agentes cimentantes

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Resumo

Introdução: Poucos estudos investigaram as propriedades de superfície de cimentos após desafio erosivo. **Objetivo:** Avaliar a rugosidade da superfície (Ra), dureza Vickers (VHN) e morfologia de superfície de 4 cimentos após desafio erosivo. **Material e método:** Vinte amostras de cada cimento foram preparadas (4×2mm) e divididas em grupo experimental (desafio erosivo) e controle (saliva artificial) (n=10): Rely X U200 (U200); Rely X ARC (ARC); Ketac Cem Easy Mix (Ketac) e Fosfato de Zinco (ZnP). O desafio erosivo foi realizado com quatro ciclos erosivos diárias (90s) em bebida à base de cola e 2h em saliva artificial durante 7 dias. As leituras de Ra e VHN foram realizadas antes e após erosão. A porcentagem de perda de dureza (%VHN) foi obtida depois da erosão. A morfologia de superfície foi analisada por microscopia eletrônica de varredura (MEV). Foram utilizados testes de ANOVA, Tukey e T-Student (α=0,05). **Resultado:** Após a erosão, foi observado aumento dos valores de Ra em todos os cimentos testados, e os grupos U200 e ZNP tiveram a maior %VHN. Após imersão em saliva, apenas os grupos U200 e ZnP tiveram aumento significativo nos valores de Ra e não houve diferenças significativas entre os grupos quanto à %VHN. A análise em MEV mostrou que os grupos Ketac e ZNP apresentaram superfícies rugosas e porosas, e o grupo U200 apresentou maior degradação da matriz comparado ao grupo ARC. **Conclusão:** O desafio erosivo com bebida a base de cola afetou as propriedades de superfície de todos os cimentos.

Descritores: Erosão dentária; cimentos de resina; cimentos de ionômero de vidro; testes de dureza.

Abstract

Introduction: Few studies investigated the surface properties of luting cements after erosive challenge. **Objective:** To evaluate the surface roughness (Ra), Vickers hardness (VHN) and morphology of 4 luting cements after erosive challenge. **Material and method:** Twenty specimens of each cement were prepared (4×2mm) and divided into experimental (erosive challenge) and control (artificial saliva) groups (n=10): Rely X U200 (U200); Rely X ARC (ARC); Ketac Cem Easy Mix (Ketac) and Zinc phosphate (ZnP). The erosive challenge was performed by four daily erosive cycles (90s) in a cola drink and 2 h in artificial saliva over 7 days. Ra and VHN readings were performed before and after erosion. The percentage of hardness loss (%VHN) was obtained after erosion. The surface morphology was analyzed by scanning electron microscopy (SEM). ANOVA, Tukey and Student-T tests were used (α=0.05). **Result:** After erosion, all luting cements had increase in Ra values and U200 and ZnP groups had the highest %VHN. After saliva immersion, only U200 and ZnP groups had significant increases in Ra values and there were no significant differences among the groups in %VHN. SEM analysis showed that Ketac and ZnP groups had rough and porous surfaces, and U200 group had higher resin matrix degradation than ARC group. **Conclusion:** Erosive challenge with a cola drink affected the surface properties of all luting cements.

Descriptors: Tooth erosion; resin cements; glass ionomer cements; hardness tests.

INTRODUCTION

Dental erosion is one of the mechanisms of tooth wear defined as hard tissue loss due to the contact of dental substrate with acids in the absence of bacteria[1]. Currently, the increased consumption of soft drinks is becoming a relevant factor for the development of tooth erosion[1,2]. In addition to the dental structure, the surface of restorative materials and luting cements in the oral cavity of aging population can also be affected by erosion[3,4].

Cementation is the last stage after a sequence of clinical procedures, and it represents a significant contribution to the biological and mechanical longevity of indirect restorations, such as crowns and bridges[5]. However, the composition of the luting cements can influence its degradation in an acidic environment[6,7]. The luting cements available can be classified as water-based and resin-based cements[8-10]. The most commonly used water-based

luting agents are zinc phosphate and glass-ionomer cements[11]. The resin-based luting cements are the most commonly used materials for the cementation of indirect restorations[9,10], and they offer advantages over water-based cements, such as the ability to adhere to multiple substrates, high strength and less disintegration in the oral environment[3,10-12].

Few studies have investigated the erosive effects on the properties of luting cements[3,7], and prolonged times of exposure in erosive solution have been used. However, to simulate erosive development caused by the daily ingestion of acidic beverages, in vitro studies have used dynamic erosive challenge with daily cycles of immersion in acidic beverages and artificial saliva[13-15].

The aim of study was to investigate the in vitro effects of dynamic erosive challenge with a cola beverage on the surface properties of water and resin-based luting cements. The hypothesis tested was that there would be differences among the luting cements regarding microhardness, roughness and morphological surface characteristics, as analyzed by scanning electron microscopy (SEM), after erosive challenge.

MATERIAL AND METHOD

Specimen Preparation

Four luting cements were investigated in this study: one conventional resin-based cement – Rely X ARC (ARC) (3M/ESPE, St. Paul, MN, USA); one self-adhesive resin-based cement – Rely X U200 (U200) (3M/ESPE, St. Paul, MN, USA); one glass ionomer cement - Ketac Cem (Ketac) (3M/ESPE, St. Paul, MN, USA); and one zinc phosphate cement (ZnP) (SS White, Rio de Janeiro, Brazil). The compositions of the materials are listed in Table 1.

Twenty specimens of each luting cement were fabricated using silicone molds (4 mm diameter × 2 mm height), according to the manufacturers' instructions. After manipulation and insertion into the molds, the specimens were covered with an acetate strip (Probem Ltda, Catanduva, São Paulo, Brazil) and were pressed flat with a glass slide to compact the material. Ketac Cem was allowed to set for 5 min, and ZnP was allowed to set for 7 min. The resin-based luting cements were polymerized for 20 s with an LED curing light (1200 mW/cm^2 - Radii Cal, SDI, Bayswater, Victoria, Australia). The specimens were maintained in relative humidity for 24 h, and the baseline roughness and microhardness measurements were then obtained, as described above. The specimens of each cement were divided in two groups (n=10): erosion (erosive challenge) and control (artificial saliva).

Erosive Challenge

The specimens in the erosion group were immersed in a cola drink (Coca-Cola, SP, Brazil - pH 2.3), using individual containers (10 mL/specimen) at room temperature, for 90 seconds 4 times/day[3,16]. Subsequently, the specimens were rinsed thoroughly with deionized water and immersed in artificial saliva, at a pH of 7.0 (10 mL/block) at room temperature for 2h, between the erosive challenges[17] and overnight[13,16]. The artificial saliva was created according to Amaechi et al.[18]. This erosive challenge was repeated for 7 days. The cola drink and artificial saliva were changed after every cycle. During the acidic cycles, the samples were kept in hermetically sealed containers to prevent the loss of carbonation from the cola drink. The specimens in the control group were immersed in artificial saliva for 7 days. The artificial saliva was changed every day.

Surface Roughness Measurements

At the end of the erosive challenge, the specimens were ultrasonically washed for 10 min and dried with absorbent paper. The specimens were then fitted to the surface roughness-measuring instrument (TR200, Digimess, São Paulo, SP, Brazil). In each specimen, three successive measurements in the central area in different directions were obtained by the same examiner, and the

Table 1. Compositions of the luting cements investigated in this study

Luting cement/Batch number	Type	Composition*
RelyX U200 (3 M ESPE, St. Paul, MN, USA) 506385	Self-adhesive resin-based	*Base Paste*: glass powder treated with silane, 2-propenoic acid, 2-methyl 1,10-(1-[hydroxymethyl]-1,2-ethanodlyl) ester dimethacrylate, TEGDMA, silica treated silane, glass fiber, sodium persulfate and per-3,5,5-trimethyl hexanoate t-butyl
		Catalyst Paste: glass powder treated with silane, substitute dimethacrylate, silica-treated silane, sodium p-toluene sulfonate, 1-benzyl-5-phenyl-acid barium, calcium, 1,12-dodecane dimethacrylate, calcium hydroxide and titanium dioxide
RelyX ARC (3 M ESPE, St. Paul, MN, USA) 336986	Conventional resin-based	*Paste A*: Bis-GMA, TEGDMA, zirconia silica, pigments, amines and photoinitiator system
		Paste B: Bis-GMA, TEGDMA, zirconia silica, benzoyl peroxide
Ketac Cem (3 M ESPE, St. Paul, MN, USA) 1224100111	Conventional glass-ionomer	*Powder*: Fluoroaluminosilicate glass
		Liquid: Polyacrylic acid, polybasic carboxylic acid, water
Zinc phosphate (SS White) P:0150214 L:00101114	---------	*Powder:* Zinc oxide, magnesium oxide
		Liquid: Phosphoric acid, aluminum hydroxide, oxide zinc, distilled water

*Bis-GMA: bisphenol-A-glycidyl methacrylate; TEGDMA: triethylene glycol dimethacrylate.

mean surface roughness values (Ra) were obtained and expressed in micrometers. The roughness testing was performed on baseline and 24 h after erosive challenge.

Surface Microhardness

The microhardness measurements were performed with a hardness tester (HMV II; Shimadzu Corporation, Kyoto, Japan) using a Vickers indenter (VHN) and a load of 200 g with a dwell time of 15 s. Five indentations were made in each specimen, at least 50 µm apart, and the mean VHN value was obtained. In addition, the percentage of microhardness loss (% VHN) was calculated using the following formula[14]:

$$\% = 100\ (VHN_{(F)} - VHN_{(I)})/VHN \quad (1)$$

where $VHN_{(I)}$ is the average of the initial (baseline) microhardness measurements, and $VHN_{(F)}$ is the average of the final (after erosive challenge) microhardness values.

Surface Morphology Assessment

Three representative specimens of each cement were mounted on aluminum stubs and sputtered-coated with gold in a vacuum (Balzers-SCD 050 Sputter Coater, Fürstentum, Liechtenstein, Germany). Scanning electron microscopy (SEM) was performed with a LEO 1430 scanning electron microscope (Zeiss Inc., Thornwood, NY, USA) Analyses were performed with 500×, 1500× and 2000× magnification before and after erosive challenge and artificial saliva immersion.

Statistical Analysis

Data analysis was performed with the GraphPad Instat computer program, version 2.0 (GraphPad software, CA, USA), at a level of significance of α=0.05. Because all of the variables tested satisfied the assumptions of normal distribution, one-way ANOVA and Tukey's test were performed for statistical comparisons of Ra and VHN measurements among the luting cements. The paired t-test was used to compare Ra and VHN measurements before and after erosive challenge for the same luting cement. The unpaired t-test was used to compare Ra and VHN values after erosive challenge and artificial saliva immersion for each luting cement. The surface morphology of the cements before and after erosion was analyzed by SEM.

RESULT

The results of roughness and microhardness assessments are described in Tables 2, 3, respectively. For the roughness measurements, after erosion, all of the luting cements had increases in Ra values ($p<0.05$). However, after saliva immersion, only the U200 and ZnP groups had significant increases in Ra values ($p=0.001$ and $p=0.03$,

Table 2. Surface roughness (Ra) of luting cements after erosive challenge and immersion in artificial saliva (control). Values expressed in means ± standard deviations (µm)

Roughness values (Ra)				
Luting cement	Erosion Group		Artificial Saliva Group	
	Baseline	After erosion	Baseline	After saliva
U200	0.10 ± 0.04 $^{A*, a}$	0.25± 0.07 $^{B, a**}$	0.07 ± 0.02 $^{A, a}$	0.12 ± 0.01 $^{B, a}$
ARC	0.08 ± 0.02 $^{A, a}$	0.18 ± 0.05 $^{B, a}$	0.07 ± 0.02 $^{A, a}$	0.08 ± 0.02 $^{A, a}$
Ketac	0.24 ± 0.05 $^{A, b}$	0.44 ± 0.15 $^{B, b}$	0.22 ± 0.06 $^{A, b}$	0.25 ± 0.01 $^{A, b}$
ZnP	0.71 ± 0.14 $^{A, c}$	0.92 ± 0.08 $^{B, c}$	0.54 ± 0.10 $^{A, c}$	0.67 ± 0.10 $^{B, c}$

*The same uppercase letters indicate that there was no significant difference between the initial and post-treatment values of each luting cement (paired t test, $p>0.05$).
** Same lowercase letters indicate that there were no significant differences among the luting cements at baseline or after treatment (one-way ANOVA and Tukey's test, $p>0.05$).

Table 3. Surface microhardness (VHN) and percentage of VHN loss of luting cements after erosive challenge and immersion in artificial saliva (control). Values expressed in means ± standard deviations

Microhardness values (VHN)						
Luting cement	Erosion group			Artificial Saliva Group		
	Baseline	After erosion	%VHN loss	Baseline	After saliva	%VHN loss
U200	38.0 ± 6.3 ^{A*}	26.7± 5.7 ^{B}	–24.2 ^{a**}	36.1 ± 4.8 ^{A}	31.1 ± 8.0 ^{B}	–14.4 ^{a}
ARC	40.6 ± 2.6 ^{A}	32.7 ± 3.8 ^{B}	–18.6 ^{b}	41.4 ± 1.8 ^{A}	39.5 ± 0.8 ^{B}	–4.4 ^{a}
Ketac	53.5 ± 3.5 ^{A}	50.7 ± 3.4 ^{A}	–5.0 ^{c}	52.9 ± 2.4 ^{A}	52.7 ± 3.3 ^{A}	–2.3 ^{a}
ZnP	79.7 ± 1.9 ^{A}	56.7 ± 2.9 ^{B}	–28.2^{a}	79.7 ±1.2 ^{A}	70.0 ± 1.4 ^{B}	–3.0 ^{a}

*Same uppercase letters indicate that there was no significant difference the between initial and post-treatment values of each luting cement (paired-T test, $p>0.05$).
**Same lowercase letters indicate that there was no significant difference of %VHN among the luting cements (one-way ANOVA and Tukey's test, $p>0.05$).

respectively). Regardless of erosive challenge or immersion in saliva, the ZnP group showed the highest Ra values (p=0.001), following by the Ketac group (p=0.001); the U200 and ARC groups showed the lowest Ra values, without a significant difference between them (p=0.07) (Table 2).

Regarding the VHN measurements, after erosion and saliva immersion, only the Ketac group did not show decrease in VHN values (p=0.08 and p=0.07, respectively) (Table 3). The values obtained for %VHN after erosion showed that the U200 and ZnP groups had the greatest %VHN, without a significant difference between them (p=0.07), the ARC group had an intermediate %VHN, and Ketac showed the lowest %VHN (p=0.001). After artificial saliva immersion, there were no statistical differences among the groups for %VHN (p=0.07) (Table 3).

Table 4 shows the comparisons of Ra and VHN values of each luting cement between after erosion and after saliva immersion. For all of the luting cements, the Ra values were significant higher after erosion than after saliva imm ersion (p<0.05). Only the Ketac group did not show a significant difference in VHN values between the treatments (p=0.07); in the other groups, the VHN values were lower after erosion than after saliva immersion (p<0.05).

Figures 1-4 show the SEM images at baseline, after erosive challenge and after saliva immersion. The U200 group showed a higher degradation of the resin matrix than the ARC group after erosive challenge and artificial saliva immersion. The filler particles of the U200 group were dispersed and protruded in the resin matrix (Figure 1B, C) compared to the surfaces in the ARC group, which were smooth and regular (Figure 2B, C). However, there were no significant morphological changes between the treatments (Figures 1B, C and 2B, C). After erosive challenge, the Ketac and ZnP groups showed rough and porous surfaces (Figures 3B and 4B), but there was no difference in morphology between baseline and saliva immersion.

Table 4. Surface roughness (Ra) and microhardness (VHN) measurements of luting cements after erosive challenge and artificial saliva immersion. Values expressed in means ± standard deviations

Luting cement	Roughness values (Ra) (μm)		Microhardness values (VHN)	
	After erosion	After saliva	After erosion	After saliva
U200	0.25± 0.07 [A]	0.12 ± 0.01 [B]	26.7 ± 5.7 [A]	31.1 ± 8.0 [B]
ARC	0.18 ± 0.05 [A]	0.08 ± 0.02 [B]	32.7 ± 3.8 [A]	39.5 ± 0.8 [B]
Ketac	0.44 ± 0.15 [A]	0.25 ± 0.01 [B]	50.7 ± 3.4 [A]	52.7 ± 3.3 [A]
ZnP	0.92 ± 0.08 [A]	0.67 ± 0.10 [B]	56.7 ± 2.9 [A]	70.0 ± 1.4 [B]

*Same uppercase letters indicate that there was no significant difference in Ra and VHN values between erosion and saliva treatments of each luting cement (unpaired-T test, $p>0.05$).

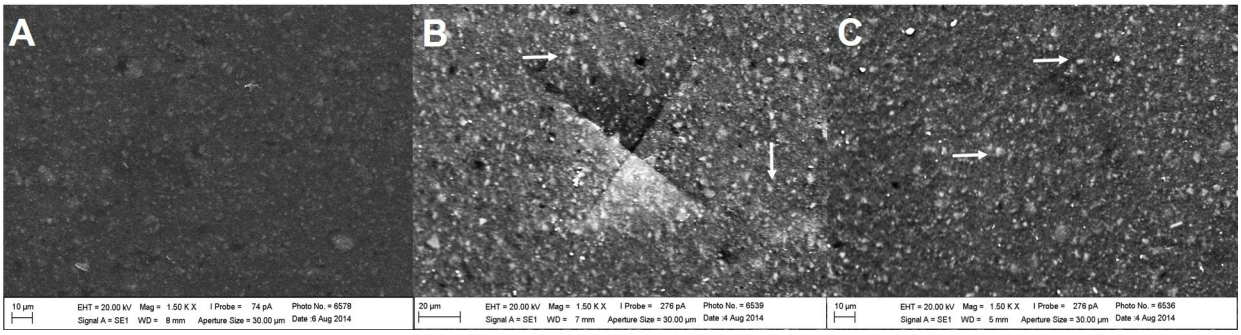

Figure 1. SEM images of surface morphology for the U200 group (1500 X): (A) baseline; (B) after erosive challenge with the indentation of Vickers diamond and (C) after saliva immersion. (→): filler particles.

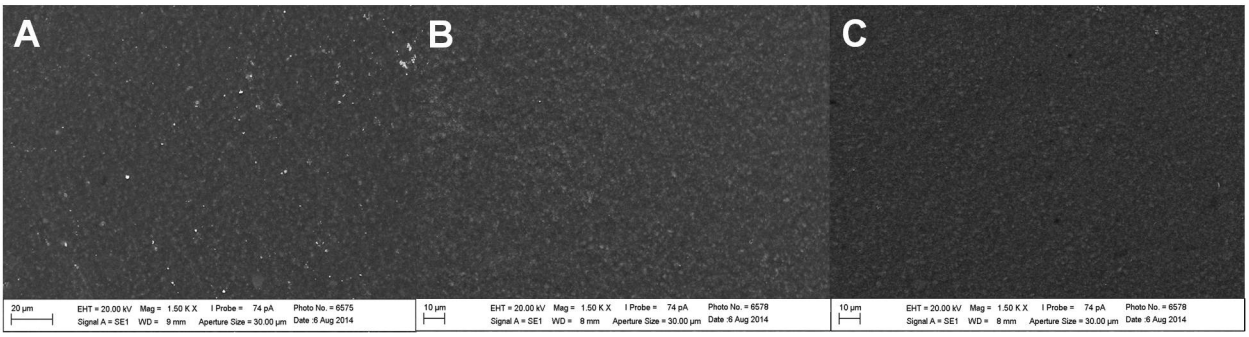

Figure 2. SEM images of surface morphology for the ARC group (1500 X): (A) baseline; (B) after erosive challenge and (C) after saliva immersion.

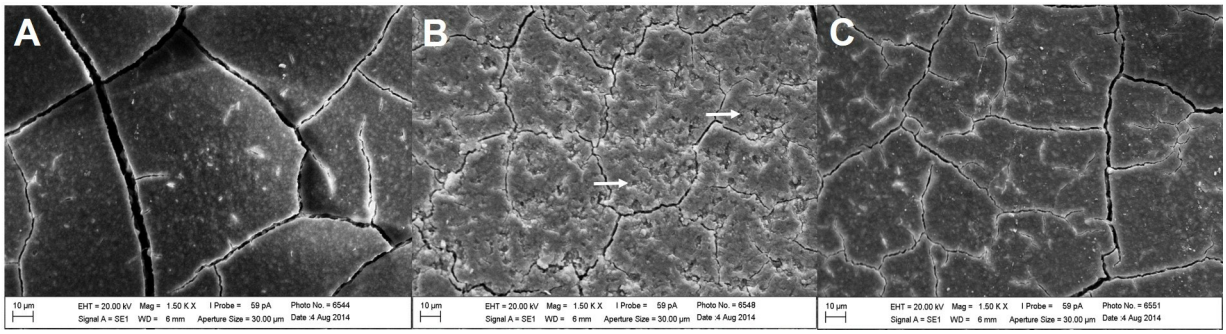

Figure 3. SEM images of surface morphology for the Ketac Cem group (1500 X): (A) baseline; (B) after erosive challenge and (C) after saliva immersion. (→): porous on surface.

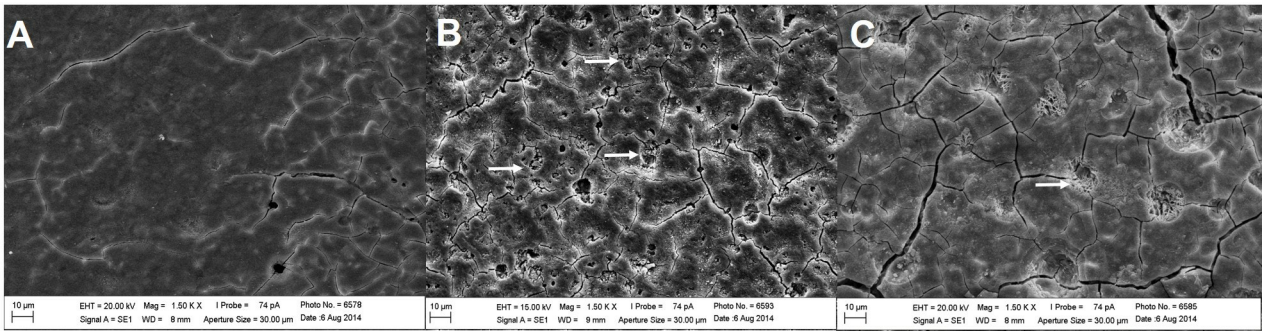

Figure 4. SEM images of surface morphology for zinc phosphate group (1500 X): (A) baseline; (B) after erosive challenge and (C) after saliva immersion. (→): porous on surface.

DISCUSSION

Some studies have shown that the erosive degradation of cements depends on the immersion time[18], the type of beverage[12,14] and the acidity of the media[18]. It is known that during consumption, a beverage contacts the tooth surface and dental materials for only a short duration before it is washed away by saliva[19]. However, previous studies have evaluated the effects of erosive solutions on luting cement properties for prolonged exposures times, and they have not included saliva in the methodology[5,6]. The present study was designed to overcome these limitations and to mimic the daily ingestion of acidic beverages. This dynamic, erosive pH-cycling model simulates the typical consumption of individuals considered to be at risk for dental erosion[20], using a beverage (Coca-Cola) that is very commonly consumed by the population and that has high erosive potential due to its low pH and fluoride/calcium concentrations[21].

After the erosive challenge, all of the cements showed increase in Ra values (Table 2). The phosphoric acid found in the cola beverage could induce the softening of dimethacrylate and Bis-GMA polymers[14] and could favor the displacement of inorganic fillers, contributing to the formation of rough surfaces. This fact could also be associated with the decrease in VHN values found after erosive challenge for resin-based luting cements (Table 3). Similarly, the acid of beverage could interfere at the ionic cross-linked polyalkenoate matrix of ionomeric cement, leading to subsequent loss of particle adherence, which could also cause increases in surface roughness. Furthermore, the Ketac group showed higher Ra values than the U200 and ARC groups (Table 2). SEM images also demonstrated higher porosity of the Ketac group surfaces (Figure 3B) than the resin-based luting cements (Figures 1B and 2B). The size of the glass filler for ionomeric cements is greater than that of the silane-treated ceramic/silica used in resin luting cements[22], so there was likely lower homogeneity between the filler and matrix the ionomeric cements, thus increasing the surface roughness.

The results of the present study showed that ZnP cement had higher Ra values and greater %VHN loss than the glass ionomer and resin-based luting cements (Tables 2, 3). SEM images showed higher porosity and degradation of the surfaces in the ZnP group after erosion than in other groups (Figure 4B). According to Gemalmaz et al.[3] the zinc ions in phosphate cements can form a weak bond with the matrix, and the zinc can be lixiviated from it. In the composition of glass ionomer cement, there is a setting reaction between the calcium and aluminum ions of fluoroaluminosilicate glass particles and polyacrylic acid[4], which could hinder the ion lixiviation and decrease the Ra values, %VHN and the degradation process of the cement. Other studies have also demonstrated that ZnP cement underwent greater degradation in acidic solutions than glass ionomer and resin-based luting cements[7,8,12,22], but these studies used lactic acid to simulate the reduction in pH in the oral environmental. Few investigations have evaluated the effects of erosive acids on the properties of luting cements[6,7].

The glass ionomer cement tested in the present study showed the smallest %VHN, and it was the only group that did not show a decrease in VHN values after erosive challenge (Table 3). Although the study by Mckenzie et al.[7] did not evaluate surface properties, they also found no changes in the compressive and flexural strength of ionomeric luting cement after immersion in Coca-Cola. The ability of cements to resist degradation was found to vary with

the composition of the medium and not to rely simply on the pH[7]. It is likely that the reaction between the acidic beverage and the surface of the cement influenced this result. Coca-Cola contains phosphoric acid, which, although capable of chelating with the calcium in cement, forms essentially insoluble complexes in water that might protect the surface from acid degradation[7].

There was a significant difference in %VHN values between the resin-based luting cements after erosion (Table 3). The U200 group showed higher %VHN than ARC group. This result could also be visualized by SEM images because the U200 group showed greater degradation of resin matrix than the ARC group (Figures 1B and 2B). The filler particles in the U200 group were dispersed and protruded in the resin matrix (Figure 1B) compared to the surfaces in the ARC group, which were smooth and regular (Figure 2B). The self-etching luting cements had amounts of water generated during neutralization of the functional groups modified by phosphoric acid that were reused to react with acidic functional groups[9]. It is likely that the water generated during the setting reaction contributed to the erosive degradation in the U200 group, leading to greater %VHN than in the ARC group (Table 3).

After artificial saliva immersion %VHN was similar for all of the luting cements, suggesting that the acidic beverage had greater effects on surface degradation than saliva. Additionally, for all of the luting cements, the Ra values were higher and the %VHN values were lower after erosive challenge than after artificial saliva immersion, except for %VHN in the Ketac group (Table 4). Yoshida et al.[8] also reported increased degradation of luting cements in low pH environments compared to neutral conditions in distilled water.

In vitro studies are generally difficult to extrapolate to in vivo conditions, but they have the advantage that individual parameters, such as erosion time, erosive agent and pH value, can be controlled. The hypothesis tested was accepted because there were differences among the luting cements in microhardness, roughness and morphological surface characteristics after erosive challenge. These preliminary results suggested that under oral conditions in which the luting cement around the margins of the restoration is constantly being washed with fluids and possibly with erosive beverages, conventional resin luting cements might be markedly less degraded than self-etching resin and glass ionomer cements. However because the exposed cement surface (4 × 2mm) was affected by the erosive challenge, and it was greater than the clinically acceptable margin of 40 μm^3, other in vivo factors should be investigated as well to predict the degradation of luting cements caused by erosive challenge.

CONCLUSION

Erosive challenge with a cola drink affected the surface properties of all of the luting cements. However, the glass ionomer and zinc phosphate cements showed the highest values for surface roughness and the self-adhesive resin and zinc phosphate cements had the highest percentage of microhardness loss.

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CONFLICTS OF INTERESTS

The authors declare no conflicts of interest.

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