

## RESEARCH AND EDUCATION

## Effect of firing cycle and aging on long-term chemical degradation of monolithic CAD-CAM ceramics

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### ABSTRACT

**Statement of problem.** Previous studies have shown the susceptibility of dental ceramics to degradation when subjected to certain media. However, knowledge on the effect of repeated firings and thermocycling on the ion elution of computer-aided design and computer-aided manufacturing (CAD-CAM) ceramics is lacking.

**Purpose.** The purpose of this in vitro study was to compare the effect of repeated firings on the ion elution of CAD-CAM materials before and after thermocycling.

**Material and methods.** Bar-shaped specimens were prepared from 4 different CAD-CAM materials (monolithic zirconia [Z], zirconia-reinforced lithium silicate glass-ceramic [S], lithium disilicate glass-ceramic [EX], and leucite-reinforced glass-ceramic [E]) and divided into 3 groups according to the number of repeated glaze firings (1 firing [1F], 2 firings [2F], and 4 firings [4F]). Specimens were placed into deionized water (pH 7.4) and stored at 37 °C for 168 hours. Inductively coupled plasma-optic emission spectrophotometry (ICP-OES) was used to measure the baseline values of the eluted ions in immersion. The specimens were then subjected to thermocycling. Then, surface roughness ( $R_a$ ) and ion elution values were measured. The Kruskal-Wallis and Mann-Whitney U tests were used to analyze the ion elution data before and after thermocycling, and the effect of thermocycling on ion elution was assessed by the Wilcoxon signed rank test.  $R_a$  data were analyzed with 2-way analysis of variance (ANOVA) and the Tukey honestly significant difference tests ( $\alpha=.05$ ).

**Results.** Elution of some ions varied depending on the material-firing pair before (Al, As, B, Ba, Cr, Cu, Li, Mg, Na, P, and Zn) and after (Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, K, Li, Mg, Na, P, Y, and Zn) thermocycling. Before thermocycling, all firing groups within each material showed a similar number of significantly higher eluted ions. After thermocycling, the number of significantly higher eluted ions decreased in all materials, except for EX. The effect of thermocycling on the ion elution of the 1F group of Z (Al, Be, Ca, Cd, Co, Cr, Cu, K, Li, P, Y, and Zn), S (As, Be, Cd, Co, Cr, K, P, and Y), EX (B, Cu, and P), and E (B and Ba); 2F group of Z (Al, Be, Ca, Co, Cr, Cu, K, Li, P, and Y), S (Be, Cd, Co, K, Li, and Y), EX (P), and E (P); 4F group of Z (Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, K, Li, P, and Y), S (Al, Be, Cd, Co, Cr, Li, Mg, and Y), EX (Be, Ca, Cd, Co, K, Y, and Zn), and E (Ca and P) was nonsignificant ( $P \geq .051$ ). The interaction between material and repeated firings ( $P < .001$ ) had a significant effect on  $R_a$ . For 1F groups, E showed the highest  $R_a$  ( $P \leq .003$ ), while Z had higher  $R_a$  than S ( $P = .009$ ). For 2F groups, Z had higher  $R_a$  than S ( $P = .01$ ). The differences among 4F groups were nonsignificant ( $P \geq .677$ ). An increased number of repeated firings (2F and 4F) decreased the  $R_a$  of E ( $P < .001$ ).

**Conclusions.** The effect of repeated firings and thermocycling on the chemical stability of the tested CAD-CAM materials varied. No clear trend was observed on the elution of different ions within material-firing pairs before thermocycling. However, thermocycling increased the number of significantly higher eluted ions for EX. The effect of thermocycling on the ion elution of materials varied depending on ions. Repeated firings decreased the surface roughness of E. (J Prosthet Dent 2022;■:■-■)

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## Clinical Implications

Repeated glaze firings may impair the long-term clinical success of lithium disilicate glass-ceramics because of increased chemical degradation.

With improvements in computer-aided design and computer-aided manufacturing (CAD-CAM) technologies, various esthetic restorative materials have been introduced.<sup>1,2</sup> Parallel to these developments, monolithic CAD-CAM ceramics have facilitated the fabrication of restorations in a single visit, thereby increasing patient satisfaction.<sup>3,4</sup> However, as further esthetic or functional adjustments may be required after the fabrication process, a restoration may undergo repeated firings.<sup>1,4-6</sup>

Dental ceramics are mainly referred to as insoluble or minimally soluble.<sup>7,8</sup> However, previous studies have shown the solubility of dental ceramics at oral pH.<sup>9-14</sup> Considering that chemical degradation depends on chemical and mechanical environmental factors<sup>11,13,15</sup> and the chemical diversity among current dental ceramics, specifying that these materials are inert would be misleading.<sup>16</sup> Chemical degradation is associated with the elution of alkaline ions,<sup>12,16,17</sup> which are less stable when present in the glassy phase than in the crystalline phase.<sup>9</sup> Moreover, the chemical degradation of a ceramic surface increases roughness that results in plaque accumulation, antagonist wear, and poor esthetics.<sup>12,14,17</sup>

Previous studies on the chemical degradation of dental ceramics have focused on the effect of pH values<sup>7,13,14,18,19</sup> or immersion cycles.<sup>20,21</sup> Mechanical and optical properties of CAD-CAM ceramics after repeated firings have also been investigated.<sup>1,4,5,22-27</sup> However, the authors are unaware of a study investigating the effects of repeated firings on the chemical stability of CAD-CAM ceramics. Considering that some of the ions present in commonly used CAD-CAM ceramics are listed as toxic by the Agency for Toxic Substances and Disease Registry when certain thresholds are exceeded (Table 1),<sup>28</sup> studies on the inertness of CAD-CAM ceramics may inform clinicians significantly. Therefore, the purpose of the present study was to investigate the effect of repeated firings on the ion elution and surface roughness of 4 different CAD-CAM materials before and after thermocycling. The null hypotheses were that repeated firings would not affect the ion elution before or after thermocycling, that thermocycling would not affect the ion elution of the materials, and that material type and repeated firings would not affect the surface roughness.

**Table 1.** Agency for Toxic Substances and Disease Registry minimal risk levels (MRLs), March 2021

Element	Duration	Route	MRLs
Aluminum	Intermediate	Oral	1 mg/kg/d
	Chronic		
Arsenic	Acute	Oral	0.005 mg/kg/d
	Chronic		0.0003 mg/kg/d
Barium	Intermediate	Oral	0.2 mg/kg/d
Beryllium	Chronic	Oral	0.002 mg/kg/d
Boron	Acute	Oral	0.2 mg/kg/d
Cadmium	Intermediate	Oral	0.0005 mg/kg/d
	Chronic		0.0001 mg/kg/d
Chromium	Intermediate	Oral	0.005 mg/kg/d
	Chronic		0.0009 mg/kg/d
Cobalt	Intermediate	Oral	0.01 mg/kg/d
Copper	Acute	Oral	0.01 mg/kg/d
	Intermediate		
Zinc	Intermediate	Oral	0.3 mg/kg/d
	Chronic		

## MATERIAL AND METHODS

An overview of the study is presented in Figure 1, and the monolithic CAD-CAM ceramics used in the present study are given in Table 2. One hundred and thirty-two bar-shaped specimens were wet-sectioned by using a low-speed sectioning machine (Secotom 10; Struers A/S) from 4 different CAD-CAM materials: monolithic zirconia (Z) (Lava All Zirconia; 3M ESPE); zirconia-reinforced lithium silicate glass-ceramic (S) (VITA SUPRINITY; VITA Zahnfabrik); lithium disilicate glass-ceramic (EX) (IPS e.max CAD; Ivoclar AG); and leucite-reinforced glass-ceramic (E) (IPS Empress CAD; Ivoclar AG) (n=33). Considering the 20% sintering shrinkage, Z specimens were prepared larger (22.5×5×1.5 mm) than the actual specimen size (18×4×1.2 mm). All sectioned specimens were cleaned in distilled water for 5 minutes in an ultrasonic bath (Eltrosonic Ultracleaner 07-08; Eltrosonic GmbH). Specimens were then divided into 3 groups according to the number of glaze firings (n=11) (Table 3); control (1F): Z-1F (sintering followed by glaze firing), S-1F (crystallization and glaze firing), EX-1F (crystallization and glaze firing), E-1F (glaze firing); 2F: Z-2F (sintering followed by 2 glaze firings), S-2F (crystallization and glaze firing followed by 1 glaze firing), EX-2F (crystallization and glaze firing followed by 1 glaze firing), E-2F (2 glaze firings); 4F: Z-4F (sintering followed by 4 glaze firings), S-4F (crystallization and glaze firing followed by 3 glaze firings), EX-4F (crystallization and glaze firing followed by 3 glaze firings), E-4F (4 glaze firings).

Before sintering, the Z specimens were stored in a plastic cup containing zirconia coloring liquid (A3, Lava Plus High Translucency Zirconia Dyeing Liquid; 3M ESPE) for 2 minutes. The specimens were removed from

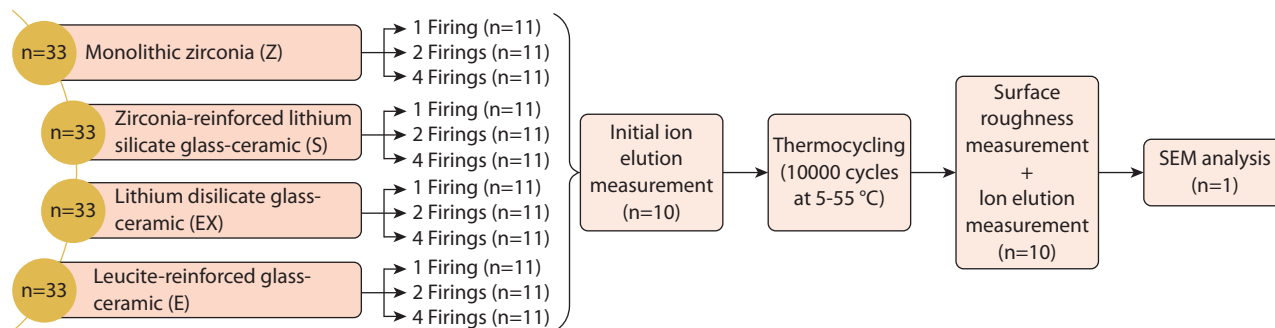


Figure 1. Overview of study. SEM, scanning electron microscope.

Table 2. Materials used

Material	Composition	Code	Manufacturer
Lava All Zirconia (zirconia)	ZrO <sub>2</sub>	Z	3M ESPE
VITA SUPRINITY (zirconia-reinforced lithium silicate glass-ceramic)	ZrO <sub>2</sub> , SiO <sub>2</sub> , Li <sub>2</sub> O, pigments	S	VITA Zahnfabrik
IPS e.max CAD (lithium disilicate glass-ceramic)	SiO <sub>2</sub> , Li <sub>2</sub> O, K <sub>2</sub> O, MgO, Al <sub>2</sub> O <sub>3</sub> , P <sub>2</sub> O <sub>5</sub> , other oxides	EX	Ivoclar AG
IPS Empress CAD (leucite-reinforced glass-ceramic)	SiO <sub>2</sub> , Al <sub>2</sub> O <sub>3</sub> , K <sub>2</sub> O, Na <sub>2</sub> O, CaO, other oxides, pigments	E	Ivoclar AG
VITA AKZENT Plus Glaze	Not provided	-	VITA Zahnfabrik
VITA AKZENT Plus Glaze LT Spray	Isobutane: 75%<80% Ethanol, ethyl alcohol: 5<10%	-	VITA Zahnfabrik
IPS e.max CAD Crystall/ Glaze Spray	Isobutane: 25%-50% Propan-2-ol: 10%<25% Nonhazardous ingredients: to 100%	-	Ivoclar AG
IPS Empress Universal Glaze Spray	Isobutane: 50%-100% Propan-2-ol: 25%-50%	-	Ivoclar AG

Al<sub>2</sub>O<sub>3</sub>, aluminum oxide; CaO, calcium oxide; K<sub>2</sub>O, potassium oxide; Li<sub>2</sub>O, lithium oxide; MgO, magnesium oxide; Na<sub>2</sub>O, sodium oxide; P<sub>2</sub>O<sub>5</sub>, phosphorus pentoxide; SiO<sub>2</sub>, silicon dioxide; ZrO<sub>2</sub>, zirconium dioxide.

the liquid, left to dry for 2 hours, and sintered in a sintering oven (inFire HTC Speed; Dentsply Sirona) at 1450 °C for 120 minutes.

Recommended glaze materials (Z: VITA AKZENT Plus Glaze; VITA Zahnfabrik, S: VITA AKZENT Plus Glaze LT Spray; VITA Zahnfabrik, EX: IPS e.max CAD Crystall/Glaze Spray; Ivoclar AG, E: IPS Empress Universal Glaze Spray; Ivoclar AG) were applied on 1 surface of the specimens to provide a dry and uniform whitish glaze layer. Spray-formed glaze materials were applied from a distance of 10 cm. Then, crystallization and glazing were performed in 1 step for S and EX, while glazing was performed for Z and E in a ceramic oven (Programat P300; Ivoclar AG). Additional glazing cycles were performed for the 2F and 4F groups, without any adjustments between firings. All specimens were then ultrasonically cleaned for 15 minutes.

The specimens were sealed into polyethylene bottles containing 20 mL of deionized water (pH 7.4). Next, the bottles were placed in an incubator (Mettler model

Table 3. Firing parameters of CAD-CAM materials used

Material	Crystallization and Glaze	
	Firing	Glaze Firing
Z	Preheating: 20 °C/min to 800 °C; preheating: 10 °C/min to 1450 °C; holding time: 120 min at 1450 °C; cooling: 15 °C/min to 800 °C and 20 °C/min to 250 °C	Standby temperature: 500 °C; closing time: 4 min; heating rate: 80 °C/min; firing temperature: 900 °C; holding time: 1 min
S	Standby temperature: 400 °C; closing time: 4 min; heating rate: 55 °C/min; firing temperature: 840 °C; holding time: 8 min; V <sub>1</sub> : 410 °C/V <sub>2</sub> : 840 °C; long-term cooling: 680 °C	Standby temperature: 400 °C; closing time: 4 min; heating rate: 80 °C/min; firing temperature: 800 °C; holding time: 1 min
EX	Standby temperature: 403 °C; closing time: 6 min; heating rate, t <sub>1</sub> : 90 °C/min/t <sub>2</sub> : 30 °C/min; firing temperature, T <sub>1</sub> : 820 °C/T <sub>2</sub> : 840 °C; holding time, H <sub>1</sub> : 10 min/H <sub>2</sub> : 7 min; V <sub>1</sub> : (1 <sub>1</sub> : 550 °C; 2 <sub>1</sub> : 820 °C)/V <sub>2</sub> : (1 <sub>1</sub> : 820 °C; 2 <sub>1</sub> : 840 °C); long-term cooling: 700 °C	Standby temperature: 403 °C; closing time: 6 min; heating rate: 60 °C/min; firing temperature: 770 °C; holding time: 1-2 min; V <sub>1</sub> : 450 °C/V <sub>2</sub> : 769 °C
E	-	Standby temperature: 403 °C; closing time: 6 min; heating rate: 100 °C/min; firing temperature: 790 °C; holding time: 1-2 min

BE500; Mettler GmbH) for 168 hours for immersion (37 °C),<sup>12</sup> which has been shown to affect the clinical lifetime of a metal-ceramic restoration.<sup>29,30</sup> The concentration of the ions transferred (mg/kg) to the immersion solution was analyzed by using an inductively coupled plasma-optic emission spectrophotometer (ICP-OES) (SPECTRO GENESIS; SPECTRO Analytical Instruments GmbH) and static immersion test method.<sup>31</sup> The same operator (M.S.) repeated the measurements 3 times, and the mean values were recorded. The measured ions were aluminum (Al), arsenic (As), barium (Ba), beryllium (Be), boron (B), cadmium (Cd), calcium (Ca), chromium (Cr), cobalt (Co), copper (Cu), lithium (Li), magnesium (Mg), oxygen (O), phosphorus (P), potassium (K), silicon (Si), sodium (Na), titanium (Ti), yttrium (Y), zinc (Zn), and zirconium (Zr).

Specimens were then submitted to 10 000 cycles of thermocycling at 5 °C to 55 °C with a transfer time of 5 seconds and a duration time of 30 seconds (MTE-101; MOD Dental), which approximately simulated 1 year of

**Table 4.** Mean  $\pm$ standard deviation values (mg/kg) of ions before thermocycling

Material	Firing Cycles	Ions																
		Al	As	B	Ba	Be	Ca	Cd	Co	Cr	Cu	K	Li	Mg	Na	P	Y	Zn
Z	1F	0.042 $\pm$ 0.001 a	0.047 $\pm$ 0.001 b	0.053 $\pm$ 0.001 b	0.067 $\pm$ 0.001 b	0.065 $\pm$ 0.000 a	1.090 $\pm$ 0.335 a	0.001 $\pm$ 0.000 a	0.062 $\pm$ 0.001 a	0.077 $\pm$ 0.001 a	0.006 $\pm$ 0.000 ab	0.198 $\pm$ 0.030 a	0.371 $\pm$ 0.091 a	0.204 $\pm$ 0.005 a	0.737 $\pm$ 0.028 a	0.067 $\pm$ 0.01 a	0.064 $\pm$ 0.000 a	0.036 $\pm$ 0.002 a
	2F	0.044 $\pm$ 0.005 a	0.046 $\pm$ 0.001 b	0.052 $\pm$ 0.003 b	0.067 $\pm$ 0.000 b	0.065 $\pm$ 0.000 a	0.889 $\pm$ 0.150 a	0.001 $\pm$ 0.000 a	0.062 $\pm$ 0.001 a	0.076 $\pm$ 0.001 a	0.006 $\pm$ 0.000 a	0.183 $\pm$ 0.044 a	0.312 $\pm$ 0.042 a	0.207 $\pm$ 0.01 a	0.751 $\pm$ 0.053 a	0.067 $\pm$ 0.01 a	0.064 $\pm$ 0.000 a	0.036 $\pm$ 0.002 a
	4F	0.042 $\pm$ 0.001 a	0.045 $\pm$ 0.001 a	0.047 $\pm$ 0.001 a	0.065 $\pm$ 0.001 a	0.065 $\pm$ 0.000 a	1.080 $\pm$ 0.427 a	0.001 $\pm$ 0.000 a	0.062 $\pm$ 0.001 a	0.075 $\pm$ 0.002 a	0.006 $\pm$ 0.000 b	0.196 $\pm$ 0.031 a	0.310 $\pm$ 0.101 a	0.215 $\pm$ 0.003 b	0.804 $\pm$ 0.028 b	0.059 $\pm$ 0.006 a	0.064 $\pm$ 0.000 a	0.038 $\pm$ 0.005 a
S	1F	0.044 $\pm$ 0.002 a	0.045 $\pm$ 0.001 a	0.049 $\pm$ 0.002 a	0.067 $\pm$ 0.001 a	0.065 $\pm$ 0.000 a	1.298 $\pm$ 0.345 a	0.001 $\pm$ 0.000 a	0.062 $\pm$ 0.000 a	0.074 $\pm$ 0.003 a	0.006 $\pm$ 0.001 a	0.175 $\pm$ 0.047 a	1.037 $\pm$ 0.269 b	0.275 $\pm$ 0.014 b	1.048 $\pm$ 0.109 a	0.055 $\pm$ 0.009 a	0.064 $\pm$ 0.000 a	0.054 $\pm$ 0.016 a
	2F	0.043 $\pm$ 0.002 a	0.046 $\pm$ 0.000 b	0.048 $\pm$ 0.003 a	0.067 $\pm$ 0.001 a	0.065 $\pm$ 0.000 a	1.345 $\pm$ 0.422 a	0.001 $\pm$ 0.000 a	0.062 $\pm$ 0.001 a	0.076 $\pm$ 0.000 ab	0.008 $\pm$ 0.005 b	0.213 $\pm$ 0.036 a	0.896 $\pm$ 0.469 ab	0.274 $\pm$ 0.011 b	1.024 $\pm$ 0.081 a	0.045 $\pm$ 0.018 a	0.064 $\pm$ 0.000 a	0.033 $\pm$ 0.011 a
	4F	0.044 $\pm$ 0.002 a	0.046 $\pm$ 0.001 b	0.047 $\pm$ 0.003 a	0.067 $\pm$ 0.001 a	0.065 $\pm$ 0.000 a	1.493 $\pm$ 0.397 a	0.001 $\pm$ 0.000 a	0.062 $\pm$ 0.000 a	0.080 $\pm$ 0.007 b	0.006 $\pm$ 0.001 ab	0.222 $\pm$ 0.052 a	0.574 $\pm$ 0.313 a	0.062 $\pm$ 0.005 a	1.087 $\pm$ 0.137 a	0.049 $\pm$ 0.018 a	0.064 $\pm$ 0.000 a	0.060 $\pm$ 0.062 a
EX	1F	0.040 $\pm$ 0.001 a	0.046 $\pm$ 0.001 a	0.054 $\pm$ 0.002 a	0.067 $\pm$ 0.000 a	0.065 $\pm$ 0.000 a	1.489 $\pm$ 0.948 a	0.001 $\pm$ 0.000 a	0.062 $\pm$ 0.001 a	0.076 $\pm$ 0.001 b	0.006 $\pm$ 0.001 a	0.189 $\pm$ 0.033 a	0.430 $\pm$ 0.081 a	0.213 $\pm$ 0.015 a	0.783 $\pm$ 0.076 a	0.069 $\pm$ 0.01 a	0.064 $\pm$ 0.000 a	0.036 $\pm$ 0.003 a
	2F	0.040 $\pm$ 0.001 a	0.046 $\pm$ 0.001 a	0.049 $\pm$ 0.001 a	0.068 $\pm$ 0.001 a	0.065 $\pm$ 0.000 a	1.540 $\pm$ 1.046 a	0.001 $\pm$ 0.001 a	0.062 $\pm$ 0.001 a	0.071 $\pm$ 0.003 a	0.006 $\pm$ 0.000 a	0.197 $\pm$ 0.050 a	0.392 $\pm$ 0.101 a	0.210 $\pm$ 0.009 a	0.789 $\pm$ 0.04 ab	0.044 $\pm$ 0.015 b	0.064 $\pm$ 0.000 a	0.039 $\pm$ 0.004 b
	4F	0.041 $\pm$ 0.001 a	0.046 $\pm$ 0.000 a	0.049 $\pm$ 0.003 a	0.067 $\pm$ 0.000 a	0.065 $\pm$ 0.000 a	1.292 $\pm$ 0.497 a	0.001 $\pm$ 0.001 a	0.062 $\pm$ 0.001 a	0.076 $\pm$ 0.001 b	0.006 $\pm$ 0.001 a	0.200 $\pm$ 0.062 a	0.567 $\pm$ 0.081 b	0.218 $\pm$ 0.013 a	0.856 $\pm$ 0.074 b	0.058 $\pm$ 0.015 ab	0.064 $\pm$ 0.000 a	0.037 $\pm$ 0.003 ab
E	1F	0.043 $\pm$ 0.003 b	0.045 $\pm$ 0.000 a	0.002 $\pm$ 0.001 a	0.054 $\pm$ 0.001 a	0.065 $\pm$ 0.000 a	1.030 $\pm$ 0.143 a	0.001 $\pm$ 0.000 a	0.062 $\pm$ 0.001 a	0.077 $\pm$ 0.001 a	0.006 $\pm$ 0.001 a	0.188 $\pm$ 0.043 a	0.350 $\pm$ 0.079 a	0.237 $\pm$ 0.034 a	0.873 $\pm$ 0.072 a	0.059 $\pm$ 0.012 a	0.064 $\pm$ 0.001 a	0.034 $\pm$ 0.002 ab
	2F	0.040 $\pm$ 0.001 a	0.046 $\pm$ 0.000 a	0.054 $\pm$ 0.003 a	0.067 $\pm$ 0.001 b	0.065 $\pm$ 0.000 a	1.018 $\pm$ 0.139 a	0.001 $\pm$ 0.001 a	0.062 $\pm$ 0.000 a	0.076 $\pm$ 0.001 a	0.006 $\pm$ 0.000 a	0.187 $\pm$ 0.025 a	0.362 $\pm$ 0.08 a	0.241 $\pm$ 0.032 a	0.855 $\pm$ 0.094 a	0.059 $\pm$ 0.013 a	0.064 $\pm$ 0.000 a	0.030 $\pm$ 0.004 a
	4F	0.041 $\pm$ 0.001 ab	0.045 $\pm$ 0.001 ab	0.051 $\pm$ 0.001 b	0.067 $\pm$ 0.000 b	0.065 $\pm$ 0.000 a	0.943 $\pm$ 0.322 a	0.001 $\pm$ 0.001 a	0.062 $\pm$ 0.001 a	0.076 $\pm$ 0.001 a	0.006 $\pm$ 0.001 a	0.186 $\pm$ 0.045 a	0.339 $\pm$ 0.067 a	0.210 $\pm$ 0.008 a	0.800 $\pm$ 0.04 a	0.068 $\pm$ 0.012 a	0.064 $\pm$ 0.000 a	0.035 $\pm$ 0.001 b

Different lowercase letters indicate significant differences in columns within each material ( $P < .05$ ).

intraoral use.<sup>31</sup> The surface roughness ( $R_a$ ) of all specimens was measured with a profilometer (Mitutoyo SurfTest SV-2100; Mitutoyo Corp) (5.5 mm transverse length, 1 mm/s stylus speed, and 0.8 mm cutoff value) after thermocycling. Three measurements were obtained from the center of each specimen at least 0.5 mm away from each other to calculate the average  $R_a$  value. Thereafter, ion elution measurements were repeated.

To examine the effect of multiple firings on surface topography, 1 specimen from each group was evaluated with scanning electron microscopy (SEM) (LEO 440; Zeiss) at  $\times 1000$  magnification. Specimens used in this analysis were not used in  $R_a$  and ion elution analysis.

The statistical analysis was performed by using a software program (IBM SPSS Statistics, v25.0; IBM Corp). The normality of the data was assessed by using the Kolmogorov-Smirnov test. For each material, ion elution values (mg/kg) were analyzed with the Kruskal-Wallis and Mann-Whitney U tests before and after thermocycling. For each material-firing pair, ion elution values (mg/kg) at the baseline and after thermocycling were compared with the Wilcoxon signed rank test.  $R_a$  values

were analyzed by 2-way analysis of variance (ANOVA) and Tukey HSD tests ( $\alpha = .05$ ).

## RESULTS

Mean values and standard deviations of ions in each material-firing group before and after thermocycling are presented in Tables 4 and 5. The number of repeated firings affected the elution of some ions before (Al, As, B, Ba, Cr, Cu, Li, Mg, Na, P, and Zn) ( $P \leq .045$ ) and after (Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, K, Li, Mg, Na, P, Y, and Zn) thermocycling ( $P \leq .041$ ).

For the effect of number of repeated firings on ion elution before thermocycling, Z-1F showed higher As, B, and Ba elution; Z-2F showed higher As, B, Ba, and Cu elution; and Z-4F showed higher Mg and Na elution ( $P \leq .04$ ). S-1F showed higher Li and Mg elution; S-2F showed higher As, Cu, and Mg elution; and S-4F showed higher As and Cr elution ( $P \leq .024$ ). EX-1F showed higher B, Cr, Li, and P elution; EX-2F showed higher Li and Zn elution; and EX-4F showed higher Cr and Na elution ( $P \leq .04$ ). E-1F showed higher Al elution; E-2F showed

**Table 5.** Mean  $\pm$ standard deviation values (mg/kg) of ions after thermocycling

Material	Firing Cycles	Ions																
		Al	As	B	Ba	Be	Ca	Cd	Co	Cr	Cu	K	Li	Mg	Na	P	Y	Zn
Z	1F	0.042 $\pm$ 0.001 a	0.045 $\pm$ 0.001 a	0.088 $\pm$ 0.001 a	0.064 $\pm$ 0.001 a	0.065 $\pm$ 0.000 a	1.036 $\pm$ 0.123 a	0.001 $\pm$ 0.001 a	0.062 $\pm$ 0.001 a	0.074 $\pm$ 0.003 a	0.006 $\pm$ 0.001 a	0.194 $\pm$ 0.038 a	0.372 $\pm$ 0.090 a	0.057 $\pm$ 0.002 a	0.680 $\pm$ 0.013 a	0.067 $\pm$ 0.010 a	0.064 $\pm$ 0.000 a	0.038 $\pm$ 0.010 a
	2F	0.042 $\pm$ 0.001 a	0.045 $\pm$ 0.001 a	0.088 $\pm$ 0.001 a	0.065 $\pm$ 0.001 a	0.065 $\pm$ 0.000 a	0.955 $\pm$ 0.063 a	0.001 $\pm$ 0.001 a	0.062 $\pm$ 0.001 a	0.075 $\pm$ 0.003 a	0.006 $\pm$ 0.000 a	0.171 $\pm$ 0.045 a	0.279 $\pm$ 0.138 a	0.058 $\pm$ 0.001 a	0.691 $\pm$ 0.020 a	0.065 $\pm$ 0.013 a	0.064 $\pm$ 0.000 a	0.050 $\pm$ 0.006 b
	4F	0.041 $\pm$ 0.001 a	0.045 $\pm$ 0.001 a	0.088 $\pm$ 0.001 a	0.065 $\pm$ 0.001 a	0.065 $\pm$ 0.000 a	0.986 $\pm$ 0.088 a	0.001 $\pm$ 0.000 a	0.062 $\pm$ 0.001 a	0.075 $\pm$ 0.002 a	0.006 $\pm$ 0.001 a	0.209 $\pm$ 0.027 a	0.287 $\pm$ 0.131 a	0.057 $\pm$ 0.002 a	0.699 $\pm$ 0.051 a	0.073 $\pm$ 0.020 a	0.064 $\pm$ 0.000 a	0.045 $\pm$ 0.009 ab
S	1F	0.046 $\pm$ 0.001 a	0.045 $\pm$ 0.001 a	0.085 $\pm$ 0.003 a	0.065 $\pm$ 0.001 a	0.065 $\pm$ 0.000 a	1.070 $\pm$ 0.236 a	0.001 $\pm$ 0.000 a	0.062 $\pm$ 0.001 a	0.073 $\pm$ 0.003 a	0.006 $\pm$ 0.001 a	0.186 $\pm$ 0.046 a	0.696 $\pm$ 0.130 a	0.059 $\pm$ 0.003 a	0.789 $\pm$ 0.053 a	0.062 $\pm$ 0.014 a	0.064 $\pm$ 0.000 a	0.045 $\pm$ 0.011 a
	2F	0.046 $\pm$ 0.004 ab	0.045 $\pm$ 0.001 a	0.083 $\pm$ 0.003 a	0.065 $\pm$ 0.001 a	0.065 $\pm$ 0.000 a	1.046 $\pm$ 0.119 a	0.001 $\pm$ 0.001 a	0.061 $\pm$ 0.001 a	0.074 $\pm$ 0.002 a	0.006 $\pm$ 0.000 a	0.235 $\pm$ 0.045 a	0.838 $\pm$ 0.322 a	0.061 $\pm$ 0.003 a	0.826 $\pm$ 0.081 a	0.065 $\pm$ 0.008 a	0.064 $\pm$ 0.000 a	0.083 $\pm$ 0.092 a
	4F	0.044 $\pm$ 0.003 b	0.045 $\pm$ 0.001 a	0.085 $\pm$ 0.002 a	0.064 $\pm$ 0.001 a	0.065 $\pm$ 0.000 a	1.081 $\pm$ 0.105 a	0.001 $\pm$ 0.001 a	0.062 $\pm$ 0.001 a	0.077 $\pm$ 0.007 a	0.006 $\pm$ 0.001 a	0.239 $\pm$ 0.047 a	0.574 $\pm$ 0.313 a	0.062 $\pm$ 0.005 a	0.828 $\pm$ 0.118 a	0.057 $\pm$ 0.016 a	0.064 $\pm$ 0.000 a	0.125 $\pm$ 0.169 a
EX	1F	0.044 $\pm$ 0.002 a	0.038 $\pm$ 0.006 ab	0.042 $\pm$ 0.041 a	0.058 $\pm$ 0.006 a	0.063 $\pm$ 0.002 a	0.887 $\pm$ 0.230 ab	0.005 $\pm$ 0.000 a	0.056 $\pm$ 0.001 ab	0.067 $\pm$ 0.006 ab	0.014 $\pm$ 0.007 ab	0.142 $\pm$ 0.048 a	0.043 $\pm$ 0.012 a	0.063 $\pm$ 0.006 ab	0.697 $\pm$ 0.026 ab	0.045 $\pm$ 0.029 ab	0.095 $\pm$ 0.027 a	0.073 $\pm$ 0.027 ab
	2F	0.044 $\pm$ 0.001 a	0.033 $\pm$ 0.001 a	0.006 $\pm$ 0.001 b	0.053 $\pm$ 0.001 b	0.062 $\pm$ 0.000 a	0.804 $\pm$ 0.080 b	0.005 $\pm$ 0.000 a	0.056 $\pm$ 0.001 b	0.063 $\pm$ 0.001 b	0.019 $\pm$ 0.001 a	0.363 $\pm$ 0.134 b	0.034 $\pm$ 0.017 b	0.064 $\pm$ 0.004 b	0.739 $\pm$ 0.042 a	0.035 $\pm$ 0.016 a	0.116 $\pm$ 0.000 a	0.095 $\pm$ 0.006 a
	4F	0.042 $\pm$ 0.001 a	0.045 $\pm$ 0.001 b	0.089 $\pm$ 0.001 a	0.064 $\pm$ 0.001 a	0.065 $\pm$ 0.000 b	1.050 $\pm$ 0.284 b	0.005 $\pm$ 0.000 a	0.056 $\pm$ 0.001 a	0.071 $\pm$ 0.003 a	0.005 $\pm$ 0.001 b	0.200 $\pm$ 0.033 ab	0.236 $\pm$ 0.176 b	0.057 $\pm$ 0.002 a	0.688 $\pm$ 0.027 b	0.068 $\pm$ 0.011 b	0.064 $\pm$ 0.000 b	0.038 $\pm$ 0.011 b
E	1F	0.046 $\pm$ 0.002 a	0.034 $\pm$ 0.001 a	0.003 $\pm$ 0.004 a	0.054 $\pm$ 0.001 ab	0.062 $\pm$ 0.000 a	0.815 $\pm$ 0.061 a	0.005 $\pm$ 0.000 a	0.056 $\pm$ 0.001 a	0.063 $\pm$ 0.001 a	0.019 $\pm$ 0.000 a	0.442 $\pm$ 0.122 a	0.062 $\pm$ 0.045 a	0.064 $\pm$ 0.001 a	0.742 $\pm$ 0.026 a	0.048 $\pm$ 0.020 a	0.116 $\pm$ 0.000 a	0.103 $\pm$ 0.007 a
	2F	0.045 $\pm$ 0.002 a	0.034 $\pm$ 0.001 a	0.001 $\pm$ 0.001 a	0.054 $\pm$ 0.000 b	0.062 $\pm$ 0.000 a	0.840 $\pm$ 0.129 a	0.005 $\pm$ 0.000 a	0.056 $\pm$ 0.001 a	0.064 $\pm$ 0.001 a	0.018 $\pm$ 0.001 a	0.426 $\pm$ 0.042 a	0.040 $\pm$ 0.035 a	0.065 $\pm$ 0.002 a	0.755 $\pm$ 0.023 a	0.167 $\pm$ 0.311 a	0.116 $\pm$ 0.000 a	0.090 $\pm$ 0.013 b
	4F	0.046 $\pm$ 0.002 a	0.034 $\pm$ 0.000 a	0.001 $\pm$ 0.001 a	0.053 $\pm$ 0.001 a	0.062 $\pm$ 0.000 a	0.877 $\pm$ 0.209 a	0.005 $\pm$ 0.000 a	0.056 $\pm$ 0.001 a	0.063 $\pm$ 0.001 a	0.018 $\pm$ 0.001 a	0.447 $\pm$ 0.090 a	0.067 $\pm$ 0.065 a	0.076 $\pm$ 0.034 a	0.749 $\pm$ 0.022 a	0.050 $\pm$ 0.021 a	0.116 $\pm$ 0.000 a	0.091 $\pm$ 0.014 ab

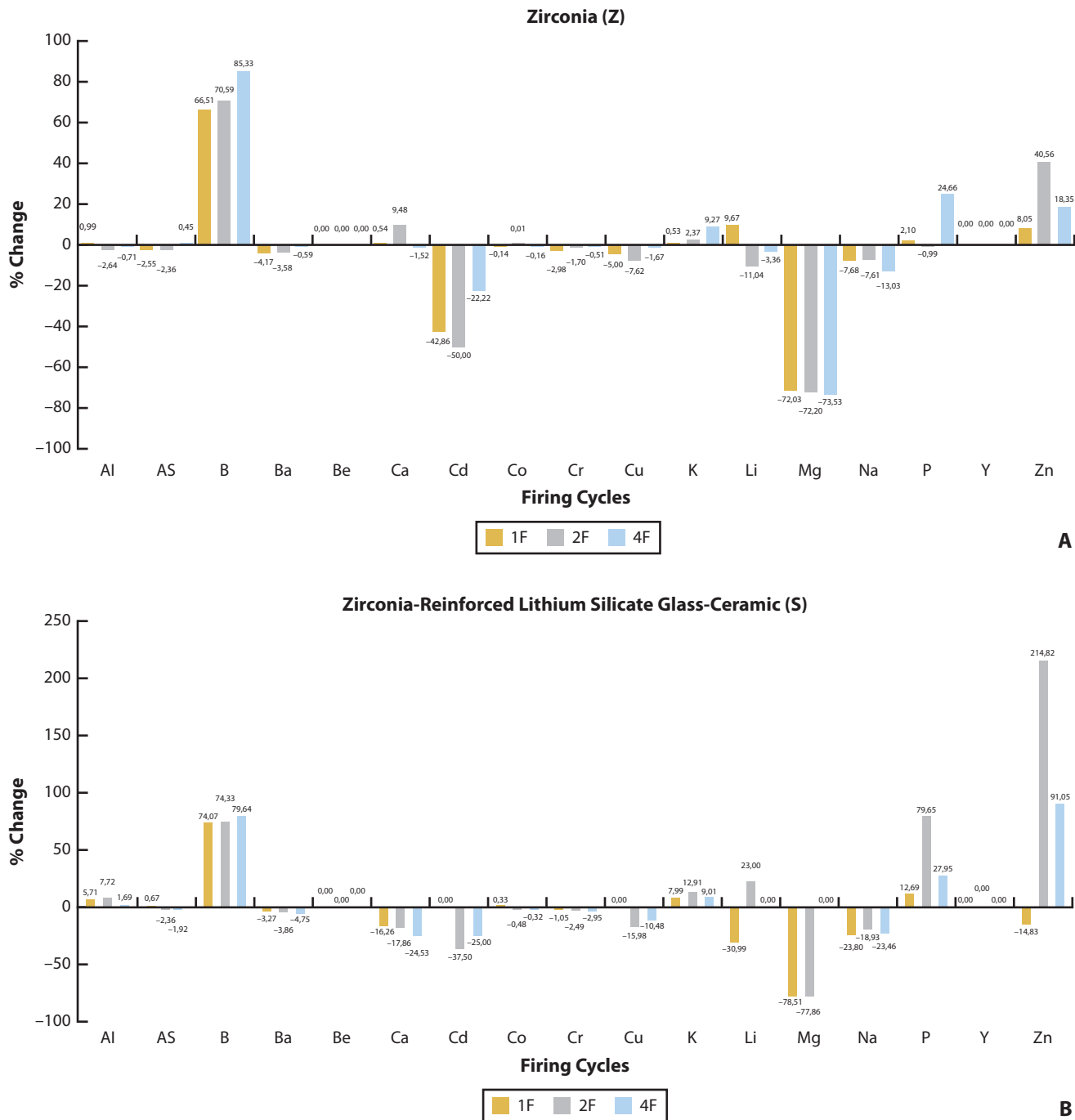
Different lowercase letters indicate significant differences between different repeated firings in columns within each material-ion pair ( $P < .05$ ).

higher As, B, and Ba elution; and E-4F showed higher B, Ba, and Zn elution ( $P \leq .018$ ) (Table 4). For the effect of number of repeated firings on ion elution after thermocycling, Z-2F showed higher Zn elution ( $P = .024$ ). S-1F showed higher Al elution ( $P = .046$ ). EX-1F showed higher B, Ba, Cd, and Y elution; S-2F showed higher Cd, Cu, K, Mg, Na, Y, and Zn elution; and S-4F showed higher As, B, Ba, Be, Ca, Co, Cr, Li, and P elution ( $P \leq .046$ ). E-1F showed higher Zn and E-2F showed higher Ba elution ( $P \leq .036$ ) (Table 5).

Significant differences were observed in the ion elution of some material-firing pairs before and after thermocycling (Fig. 2). For Al, EX, E, S-1F, and S-2F showed a significant increase ( $P \leq .046$ ). For As, all material-firing pairs other than S-1F and Z-4F showed a significant decrease ( $P \leq .041$ ). For B, Z, S-2F, S-4F, and EX-4F showed a significant increase ( $P = .005$ ). However, EX-2F, E-2F, and E-4F showed a significant decrease ( $P = .005$ ). For Ba, all material-firing cycle pairs other than E-1F and Z-4F showed a significant decrease ( $P \leq .006$ ). For Be, E, EX-1F, and EX-2F showed a significant decrease ( $P \leq .014$ ). For Ca, all material-firing cycle pairs other than 1F-Z, 2F-Z, 4F-Z, 4F-EX, and 4F-E showed a

significant decrease ( $P \leq .013$ ). For Cd, E, EX-1F, and EX-2F showed a significant increase ( $P \leq .018$ ). For Co, E, EX-1F, and EX-2F showed a significant decrease ( $P \leq .008$ ). For Cr, EX, E, and S-2F showed a significant decrease ( $P \leq .026$ ). For Cu, E and EX-2F showed a significant increase ( $P \leq .005$ ), whereas S-2F showed a significant decrease ( $P = .024$ ). For K, E, EX-2F, and S-4F showed a significant increase ( $P \leq .022$ ), whereas EX-1F showed a significant decrease ( $P = .013$ ). For Li, EX, E, and S-1F showed a significant decrease ( $P \leq .007$ ). For Mg, all material-firing pairs other than S-4F showed a significant decrease ( $P = .005$ ). For Na, every material-firing pair showed a significant decrease ( $P \leq .022$ ). For P, S-2F, S-4F, and EX-4F showed a significant increase ( $P \leq .046$ ). For Y, E, EX-1F, and EX-2F showed a significant increase ( $P \leq .014$ ). For Zn, S-1F showed a significant decrease ( $P = .007$ ), while every other material-firing pair showed a significant increase ( $P \leq .028$ ), except for Z-1F and EX-4F ( $P \geq .262$ ).

The SEM images revealed that small lines on the surface of Z-1F were replaced by small pores as the number of firings increased (Fig. 3). No visible differences were detected among the surfaces of S after a different

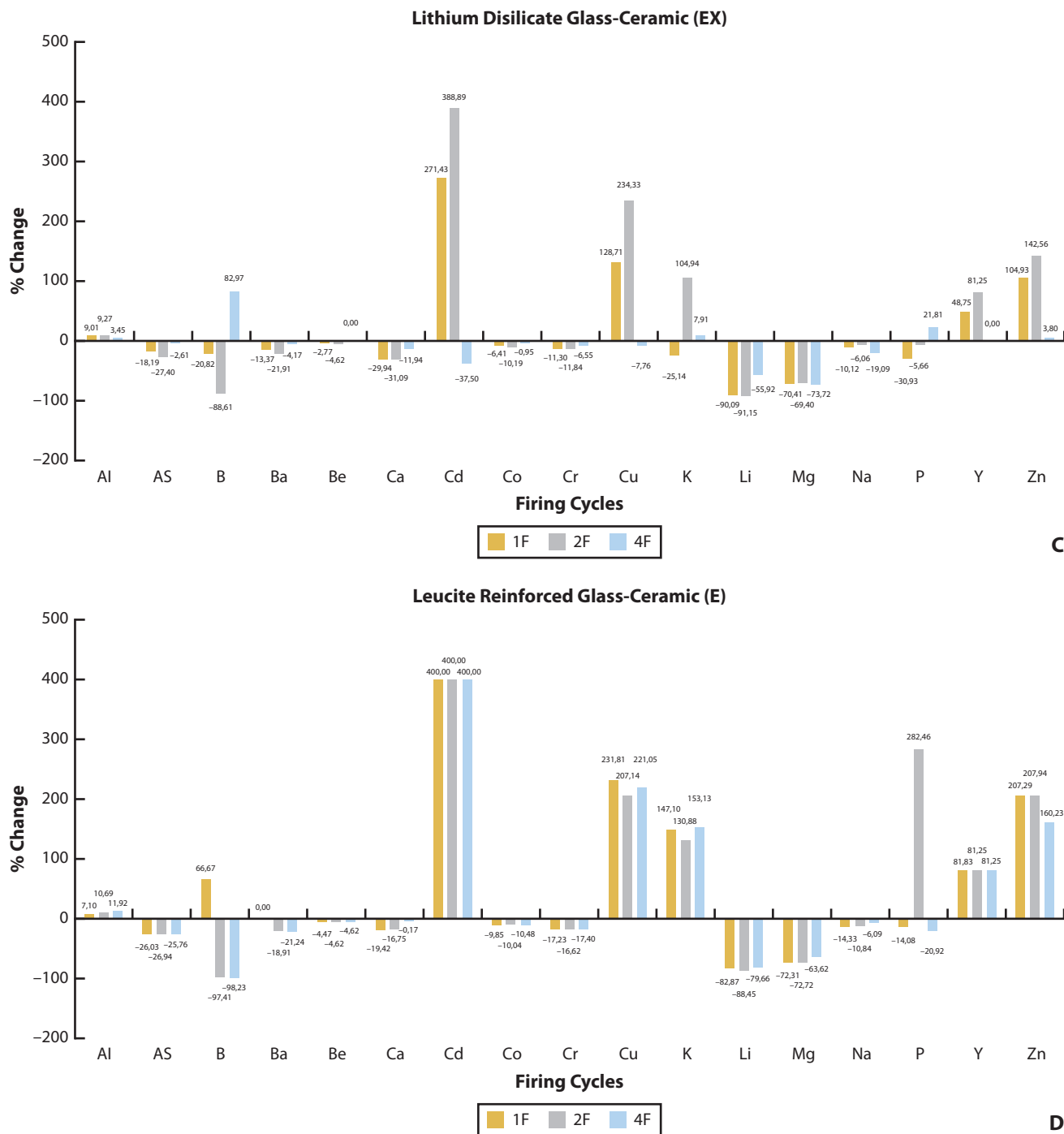


**Figure 2.** Percent change of ions after thermocycling within each firing stage. Negative value in y-axis indicates percent decrease. A, Zirconia. B, Zirconia-reinforced lithium silicate glass-ceramic.

number of repeated firings. EX-2F had multiple complex lines, which were not present in EX-1F and EX-4F. Small pores and lines were apparent on E-4F, whereas a similar topography was observed in E-1F and E-2F.

The 2-way ANOVA revealed that the type of material, number of firings, and their interaction affected the  $R_a$  ( $P < .001$ ). When clinically relevant material-firing pairs were compared, only the  $R_a$  values of E changed

significantly according to repeated firing, as E-1F presented higher values than E-2F and E-4F ( $P < .001$ ). When 1F groups of the materials were compared, E showed the highest  $R_a$  among the materials ( $P < .003$ ). In addition, Z had higher  $R_a$  than S ( $P = .009$ ), while EX showed similar values to Z ( $P = .945$ ) and S ( $P = .413$ ). When 2F groups were compared, only the difference between Z and S was significant ( $P = .01$ ) as Z showed higher  $R_a$ . No significant



**Figure 2.** (Continued). C, Lithium disilicate glass-ceramic. D, Leucite-reinforced glass-ceramic.

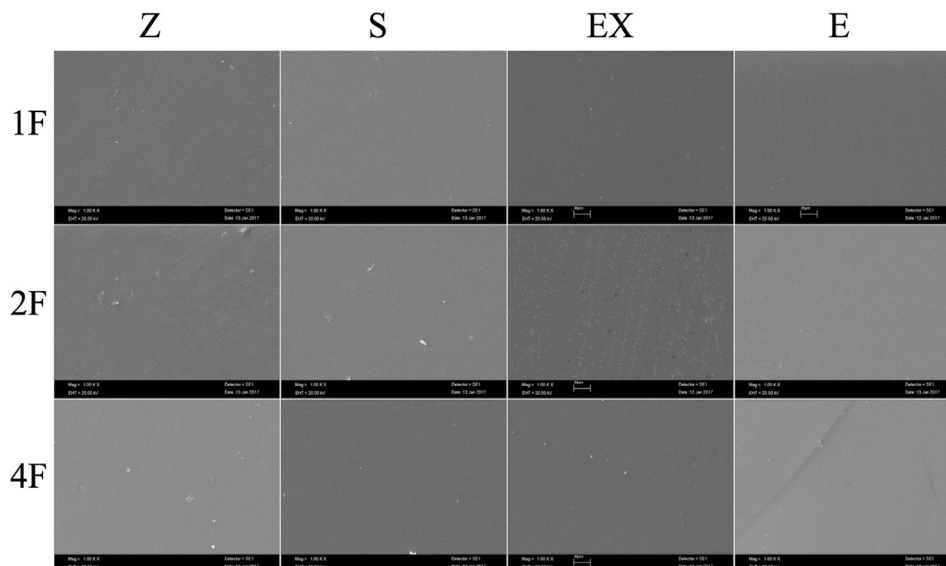
differences were observed among the 4F groups ( $P \geq .677$ ) (Table 6).

## DISCUSSION

Elution of most of the ions investigated in the present study showed significant differences within each CAD-CAM material depending on repeated firings both before and after thermocycling, and significant

differences were observed between the eluted values of each ion before and after thermocycling. In addition, the  $R_a$  of specimens was affected by both material type and repeated firing. Therefore, the null hypotheses were rejected.

Even though most CAD-CAM ceramic manufacturers recommend 1 extrinsic firing before cementation, occlusal, interproximal, or esthetic adjustments may



**Figure 3.** Representative SEM images of CAD-CAM material surfaces after different number of repeated firings (original magnification  $\times 1000$ ). CAD-CAM, computer-aided design and computer-aided manufacturing; SEM, scanning electron microscope.

require additional firings.<sup>5</sup> To the authors' knowledge, the present study was the first on the combined effect of repeated firings and thermocycling on ion elution of CAD-CAM ceramics. Before thermocycling, the number of ions released was significantly higher from each firing group within a material and was similar, ranging from 1 (E-1F) to 4 (Z-2F and EX-1F). However, after thermocycling, only the significantly higher number of ions released from EX-2F (Cd, Cu, K, Mg, Na, Y, and Zn) and EX-4F (As, B, Ba, Be, Ca, Co, Cr, Li, and P) increased.

The chemical degradation of dental ceramics in acidic media has been previously investigated.<sup>7,8,11-14,19</sup> Mil-ling et al<sup>12</sup> reported that immersion in 4% acetic acid solution at 80 °C for 18 hours increased the surface roughness of leucite ceramic, a finding that was corroborated by another study, in which organic acids were reported to negatively affect lithium disilicate and zirconia-reinforced lithium silicate ceramics.<sup>8</sup> In another study on the chemical degradation of dental ceramics, including a leucite glass-ceramic and an yttria-stabilized zirconia, oxide ceramics eluted significantly fewer ions than glass-ceramics, which was observed on a larger scale in the acidic medium.<sup>11</sup> However, other studies suggested that increasing the frequency of pH changes increased the corrosion of glass ceramics and Si ion elution compared with immersion in a constant pH environment.<sup>20,21</sup> Considering that Si, O, Ti, and Zr are the dominant elements in materials tested in the present study (Table 1), a higher amount of elution of these ions was expected. However, these elements were not detected in ICP-EOS analyses, regardless of thermocycling, possibly because these elements are present as chemical compounds formed with each other, and ion

**Table 6.** Descriptive statistics of surface roughness ( $\mu\text{m}$ ) values of different CAD-CAM ceramics and firing groups

Material	Number of Firings	Mean $\pm$ Standard Deviation
Z	1F	1.39 $\pm$ 0.29 <sup>aB</sup>
	2F	1.21 $\pm$ 0.37 <sup>aB</sup>
	4F	1.0 $\pm$ 0.44 <sup>aA</sup>
S	1F	0.60 $\pm$ 0.36 <sup>aA</sup>
	2F	0.42 $\pm$ 0.33 <sup>aA</sup>
	4F	0.58 $\pm$ 0.46 <sup>aA</sup>
EX	1F	1.09 $\pm$ 0.52 <sup>aAB</sup>
	2F	0.73 $\pm$ 0.39 <sup>aAB</sup>
	4F	0.69 $\pm$ 0.29 <sup>aA</sup>
E	1F	2.26 $\pm$ 1.0 <sup>bC</sup>
	2F	0.53 $\pm$ 0.28 <sup>aAB</sup>
	4F	0.70 $\pm$ 0.32 <sup>aA</sup>

Different superscript lowercase letters indicate significant differences among the firings within material, while different superscript uppercase letters indicate significant differences among materials within firings ( $P < .05$ ).

elution might not depend on the abundance of elements.<sup>15</sup> In addition, thermocycling was performed by using neutral-pH distilled water. However, these ions might be resistant to elution in neutral-pH liquids.

Tai et al<sup>32</sup> reported a significant effect of simulated occlusal wear by using different antagonists on the leaching of Ni and Be ions from dental alloys. In an in vivo study, Elshahawy et al<sup>15</sup> concluded that Zn elution from Type IV gold crowns and Si elution from E crowns significantly increased after 3 months of clinical service. In the present study, ion elution of Zn from E significantly increased after thermocycling regardless of the number of repeated firings, whereas Si was not present in the analyses. However, a direct relation



between the present study and that of Elshahawy et al<sup>15</sup> might be misleading considering the absence of mechanical aging in the present study. Future studies investigating the combined effect of thermomechanical aging and different repeated firings on chemical degradation of CAD-CAM ceramics are needed to increase knowledge on the clinical behavior of these materials.

Among the ions investigated in the present study, some (Al, As, B, Ba, Be, Cd, Cr, Co, Cu, and Zn) might have acute or chronic toxic effects if daily intake exceeds the thresholds stated by the Agency for Toxic Substances and Disease Registry (Table 1). The minimal risk levels of these toxic ions have been specified in a previous study on the ion elution of different esthetic materials when subjected to a home bleaching agent.<sup>7</sup> Lower elution values for Al, Ca, K, Li, Na, P, and Zn ions than the minimal risk levels were reported, whereas Cu levels were above the threshold for certain materials, including zirconia.<sup>7</sup> In the present study, As, B, Be, Cd, Cr, Co, and Zn levels exceeded the stated thresholds for each material-firing pair, regardless of aging. In addition, Cu levels were higher than the stated thresholds in EX-1F, EX-2F, and E after thermocycling. However, these results should be interpreted carefully, as the present study did not evaluate the daily release of each ion but focused on long-term ion release. Thus, future studies should focus on the daily release of ions from the tested materials to elaborate the knowledge on their toxicity after repeated firings and aging.

Even though repeated firings reduced the  $R_a$  of materials tested in the present study, only E showed a significantly lower  $R_a$  after repeated firings. This difference between E and the other materials tested may be attributed to the difference in the composition of restorative and glaze materials. Nevertheless, this result was consistent with a previous study, which reported that increased number of firings reduced the  $R_a$  of leucite-reinforced glass-ceramics.<sup>25</sup> Oğuz et al<sup>27</sup> evaluated the  $R_a$  of zirconia after repeated firings and reported similar results to those in the present study. To the authors' knowledge, no study has investigated the effect of repeated firings on the  $R_a$  of S. Therefore, the results on the  $R_a$  of S could not be compared. Meng et al<sup>1</sup> showed that repeated firing of lithium disilicate did not change the  $R_a$ , which was consistent with the results of the present study. Contrarily, in another study, repeated firings were shown to affect the  $R_a$  of lithium disilicate.<sup>6</sup> However, a staining liquid was also used in that study, which may have caused this difference. In addition, only  $R_a$  values were analyzed.

SEM images showed that the EX-4F did not display the complex multiple lines present in EX-2F, consistent with a previous study,<sup>5</sup> which showed that lithium disilicate displayed a complex mesh structure after the first and second

firings. However, Meng et al<sup>1</sup> reported that polished lithium disilicate did not show any topographical difference after a different number of repeated firings. This difference may be related to the fact that the specimens were not polished but glazed in the present study. For Z, increased number of firings resulted in a smoother surface, which substantiates the  $R_a$  findings. No significant differences were observed among the different firing groups of S and E. Considering these results, it can be speculated that the effect of multiple firings on  $R_a$  is material-dependent.

Limitations of the present study included the absence of a priori power analysis. However, significant differences were observed. The materials tested in the present study are commonly used; however, alternatives with different chemical compositions are available, and only 1 type of each material type was tested. Another limitation was that thermocycling does not completely replicate intraoral conditions as both sides of the specimens were aged and distilled water was used. In addition, other parameters that can be measured with a profilometer, such as the average value of the absolute heights of the 5 highest peaks and deepest valleys ( $R_z$ ) or root mean square value of the surface departures ( $R_q$ ), might elaborate the understanding of the effect of the tested parameters on surface roughness. Previous studies on chemical degradation have used 4% acetic acid;<sup>7,11-14,16,17,19</sup> thus, future studies should investigate the ion elution from different CAD-CAM materials after multiple firings by using 4% acetic acid. Finally, the genotoxic effects of Ni-Cr-based alloys have been reported,<sup>33</sup> and this may be implemented in future in vivo studies investigating the effect of the ions eluted from the CAD-CAM materials tested in the present study after repeated firings on neighboring tissues.

## CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. No clear trend was found on the effect of repeated firings on the ion elution of materials before thermocycling.
2. After thermocycling, ion elution increased only from EX (2F and 4F groups); therefore, EX may be more susceptible to thermocycling after repeated firings.
3. No clear trend was found on the effect of thermocycling on the ion elution across materials.
4. The SEM images supported the measured surface roughness values, which revealed varied effects of number of firings on material surfaces at different stages. Only the surface roughness of E changed depending on the number of firings and the roughness decreased.

5. Surface roughness of materials was similar when compared with each other after 4 times of firing.

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Murat Sert: Methodology, Data curation, Data interpretation, Formal analysis. Gülce Çakmak: Methodology, Investigation, Project administration, Conceptualization. Meryem Gülce Subaşı: Methodology, Investigation, Project administration, Writing - original draft. Mustafa Borga Donmez: Writing - original draft, Critical revision of article. Burak Yilmaz: Supervision, Writing - review and editing, critical revision of article.

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<https://doi.org/10.1016/j.prosdent.2022.04.028>