

# Vicker's microhardness and energy dispersive x-ray analysis of fluorapatite-leucite and fluorapatite ceramics cyclically immersed in acidic agents

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**Abstract:** This study evaluated the microhardness and elemental composition of various ceramics immersed in acidic agents. One hundred and five discs of fluorapatite-leucite (IPS d.SIGN) and fluorapatite ceramics (IPS e.max Ceram) were randomly divided into 7 groups of 15 each. Five groups were alternately immersed in acidic agents (citrate buffer solution, pineapple juice, green mango juice, cola soft drink and 4% acetic acid) for 30 min and deionized water for 5 min in 7 cycles at 37°C. Two groups were continuously immersed in 4% acetic acid at 80°C for 16 h and deionized water at 37°C for 245 min. Vicker's microhardness and elemental composition were recorded before and after immersion. Data were analyzed using two-way repeated ANOVA and *t*-test at a significance level 0.05. For IPS d.SIGN, there was statistically significant difference in microhardness values between before and after immersion in the green mango, cola soft drink and 4% acetic acid for 16 h ( $P < 0.05$ ). However, for IPS e.max Ceram, there was no significant difference between before and after immersion in any of the groups ( $P > 0.05$ ). EDX results of both ceramics found that the elemental percentage of Na, K, Zr, Al and Si decreased after being immersed in acidic agents. (J Oral Sci 51, 443-450, 2009)

Keywords: dental ceramics; energy dispersive x-ray;

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fluorapatite; leucite; microhardness.

## Introduction

Dental ceramics are highly esthetic materials which are increasingly used in dentistry to construct various types of restorations and prostheses such as veneers, inlays, onlays, crowns and fixed partial dentures. They fulfill the esthetic and functional demands of patients with their properties, which are superior to other tooth-colored materials (1). The new glass-ceramics (IPS d.SIGN, Ivoclar Vivadent AG, Schaan, Liechtenstein) have gained popularity for metal-ceramic restorations. IPS d.SIGN is a new type feldspathic-based ceramic containing dispersed fluorapatite ( $\text{Ca}_{10}(\text{PO}_4)_6\text{F}_2$ ) and leucite crystals ( $\text{K}_2\text{O Al}_2\text{O}_3 \cdot 4\text{SiO}_2$ ) in a feldspathic glass matrix (2). The leucite crystals ( $< 3 \mu\text{m}$ ) present in the IPS d.SIGN ceramic also contribute to the overall strength (3). Recently, new all-ceramic systems (IPS e.max, Ivoclar Vivadent AG) have been introduced into the market. IPS e.max Ceram, a veneering ceramic of this system, is feldspathic-based, having a microstructure unlike IPS d.SIGN. This glass-ceramic only consists of dispersed fluorapatite crystals in a feldspathic glass matrix, thus having a microstructure unlike that of any other commercially available dental ceramics (3). Fluorapatite crystals, 2-5  $\mu\text{m}$  in length and 300 nm in diameter with needle-like morphology, are known to be contained in natural bone and teeth. The very small crystals in dental microstructures result in very special optical properties such as translucence and opalescence, which also result in the same properties as in dental restorations (4).

Despite the outstanding esthetic quality of the ceramics,

the most serious problem of this material is the susceptibility to fracture (5,6). Dental ceramics exhibit inherent flaws or defects on the surface and body. These characteristics impair their physical properties, i.e., surface roughness, surface hardness, and strength (7,8) and influence the clinical success and failure of ceramic restorations (9). The chemical durability of dental ceramics is basically good, but it may be influenced by many factors, such as the composition and microstructure of the ceramic material, the chemical characteristics of the ceramic material, the chemical nature of the acidic agent, the exposure time and the temperature (10). Degradation of dental ceramics occurs when ceramics are exposed to aqueous solutions or acidic agents (11). This phenomenon takes place as a result of selective leaching of alkaline ions. Environmental conditions may also impair resistance to surface and bulk degradation. Variations in ceramics and processing techniques may result in materials with reduced hydrolytic stability. Alkaline metal ions are far less stable in the glass phase than in the crystalline phase (11). Some alkaline ions leached out after they were exposed to acidic solutions (12-14). Variations in pH, solution chemistry, wear and mechanical load make the oral cavity a complex environment (11). Crack propagation in ceramics is increased when ceramics are exposed to an aqueous environment (11). Consequences of ceramic degradation are coarseness of exposed surface (14,15), plaque accumulation (11-16) and wear to antagonist materials (11). Furthermore, increase in surface roughness of ceramics may decrease strength (5,6).

In many countries, people frequently consume acidic

food, sour fruits and drinks resulting in a high incidence of dental erosion (17,18). The potential erosive effect of these acidic food and drinks on enamel occurs primarily by dissolution of apatite crystals (18,19). However, their effect on ceramic restorations has not been well-documented. Therefore, the present *in vitro* study was designed to evaluate changes of surface microhardness and surface elemental compositions of fluorapatite-leucite and fluorapatite ceramics after being exposed to acidic agents (pineapple juice, green mango juice, cola soft drink, citrate buffer solution and 4% acetic acid) in cyclic immersion. The hypothesis tested was that the acidic agents under investigation would lead to significantly decreased surface microhardness and dissolution of surface elemental composition of fluorapatite-leucite and fluorapatite ceramics after immersion compared to before immersion.

## Materials and Methods

### Specimen preparations

Two commercial dentin shade A3 ceramics were used: IPS d.SIGN and IPS e.max Ceram (Table 1). IPS d.SIGN and IPS e.max Ceram are used as veneering ceramics for metal-ceramic and all-ceramic restorations, respectively. One hundred and five disc specimens of each ceramic were fabricated. Briefly, the ceramic powder was mixed with deionized water and condensed into the silicone mold (Provil, Haraeus Kulzer, Wehrheim, Germany), 12.0 mm in diameter and 2.0 mm in thickness, with a condenser (Ceramosonic II, Shofu, Kyoto, Japan). The specimens were then fired according to the manufacturer's instructions (Table 2). Subsequently, the specimens were polished

Table 1 Dental ceramics used in this study

Ceramic	Type	Composition (wt %)	Manufacturer
IPS d.SIGN	Fluorapatite-leucite ceramic	SiO <sub>2</sub> 50-65, Al <sub>2</sub> O <sub>3</sub> 8-20, Na <sub>2</sub> O 4-12, K <sub>2</sub> O 7-13, CaO 0.1-0.6, P <sub>2</sub> O <sub>5</sub> 0.0-0.5, F 0.1-3.0	Ivoclar Vivadent AG, Schaan, Liechtenstein (Lot H28470)
IPS e.max Ceram	Fluorapatite ceramic	SiO <sub>2</sub> 60-65, Al <sub>2</sub> O <sub>3</sub> 8-12, Na <sub>2</sub> O 6-9, K <sub>2</sub> O 6-8, ZnO 2-3, CaO 1-3, P <sub>2</sub> O <sub>5</sub> 0.1-1, F 1-2,	Ivoclar Vivadent AG, Schaan, Liechtenstein (Lot H18984)

Table 2 Firing parameter for the ceramics of the present study

Ceramic	Type of firing	Starting temperature (°C)	Heating rate (°C/min)	Vacuum temperature (°C)	Firing temperature (°C)	Holding time (min)
IPS d.SIGN	Dentine	403	60	450-909	910	1
	Glaze	403	60	450-829	830	0
IPS e.max Ceram	Dentine	403	50	450-849	850	0
	Glaze	403	50	450-799	800	0

(model Phoenix 4000, Buehler GmbH, Dusseldorf, Germany) under running water using 600 and 1,200-grit silicon carbide paper (3M ESPE, St. Paul, MN, USA). The specimens were then cleaned in an ultrasonic cleanser for 10 min. Finally, the specimens were subjected to self-glazing according to the manufacturer's instructions (Table 2).

### Acidic agent exposure and microhardness measurements

Seventy ceramic discs were randomly divided into 7 groups of 10 each. Each group was subjected to microhardness measurement for baseline data (before immersion). For microhardness measurement, disc specimens were measured with a microhardness tester (Micromet II, Buehler, Lake Bluff, IL, USA) using a Vickers diamond tip under a 0.2 kg indentation load for 15 s (20). Five indentations per specimen were obtained on the top surface and the mean value of each specimen was calculated. Subsequently, the specimens were alternately immersed in 25 ml of an acidic agent for 30 min and in 25 ml of deionized water for 5 min for 7 cycles at 37°C. The same protocol was used with the different types of acidic agents included in the study (citrate buffer solution, pineapple juice, green mango juice, cola soft drink and 4% acetic acid; see Table 3). The specimens' immersion protocol simulated an individual eating acidic food, sour fruits and drinks. Total immersion time was 245 min. Two groups (4% acetic acid for 16 hours and deionized water) were continuously immersed in 4% acetic acid at 80°C for 16 h (as modified from ISO 6872 (21)) and in deionized water at 37°C for 245 min. After the immersion sequence was completed, the specimens were rinsed with deionized water, blotted dry and subjected to microhardness testing (after immersion).

### Energy dispersive x-ray (EDX) analysis

Thirty-five disc specimens were randomly divided into

7 groups of 5 each. Specimens were then dried at room temperature for 24 h before surface elemental analysis. EDX analysis was performed before and after the immersions using an EDX spectrometer (Oxford Link ISIS 300, Oxford, UK) attached to the scanning electron microscope (JSM-5800LV Scanning Microscopes, JEOL, Tokyo, Japan) under vacuum on uncoated specimens fixed on stubs. Ten areas per specimen were selected and tested on a scanning electron microscope acquired with 20 kVp, 20 mm of distance and spot size 5 nm. Each of the surface elemental compositions was calculated to a mean weight percentage (wt%) and standard deviation (S.D.). Hydrocarbon contamination which consistently showed as a carbon peak in all groups was disregarded (22).

### Statistical analysis

The data were statistically analyzed. Two-way repeated analysis of variance (ANOVA) was used to measure statistically significant differences among types of acidic agents and ceramics before and after exposure to acidic agents. A paired *t*-test was used to compare the microhardness between before and after immersion of the two types of ceramics in each erosive agent ( $\alpha = 0.05$ ).

## Results

The microhardness values of IPS d.SIGN and IPS e.max Ceram measured before and after immersion are reported in Table 4. The results of the two-way repeated ANOVA showed that the interaction between two variables (type of ceramic and type of acidic agent) was statistically significant ( $P = 0.001$ ). There were also statistically significant differences between the two types of ceramics and among the acidic agents ( $P = 0.001$  and  $P = 0.009$ , respectively). Microhardness of IPS d.SIGN and IPS e.max Ceram before immersion was not statistically different ( $P > 0.05$ ). Overall, before immersion, IPS d.SIGN groups yielded microhardness values significantly higher than the IPS e.max Ceram groups ( $P = 0.001$ ). The results of

Table 3 Acidic agents used in the study

Acidic agents	Form	Manufacturer
Citrate buffer solution pH 4.99 (0.01)	Instant	BDH Laboratory Supplies, Poole, England
100% pine apple juice pH 3.64 (0.01)	Instant	Tipco Foods, Prajuabkirikhan, Thailand
Green mango juice pH 2.39 (0.01)	Prepared from fresh green mango	—
Cola soft drink pH 2.41 (0.06)	Instant	Hadthip, Songkhla, Thailand
4% acetic acid pH 2.47 (0.01)	Dilute from 100% acetic acid	Merck KgaA, Darmstadt, Germany

Table 4 Mean microhardness (GPa) and standard deviations of IPS d.SIGN and IPS e.max Ceram immersed in acidic agents

Group	Mean microhardness in GPa (S.D.)			
	IPS d.SIGN		IPS e.max Ceram	
	Before	After	Before	After
Deionized water	5.06 (0.26)	5.00 (0.18)	4.79 (0.12)	4.78 (0.15)
Citrate buffer solution	4.99 (0.29)	4.77 (0.31)	5.06 (0.34)	5.02 (0.43)
Pineapple juice	5.09 (0.08)	4.95 (0.23)	4.78 (0.23)	4.59 (0.14)
Green mango juice	5.09 (0.29) <sup>a</sup>	4.65 (0.23) <sup>a</sup>	4.82 (0.13)	4.49 (0.57)
Cola soft drink	5.01 (0.31) <sup>b</sup>	4.63 (0.24) <sup>b</sup>	4.79 (0.15)	4.67 (0.21)
4% acetic acid	5.06 (0.31)	4.97 (0.54)	4.83 (0.16)	4.67 (0.22)
4% acetic acid for 16 h	5.01 (0.17) <sup>c</sup>	4.48 (0.24) <sup>c</sup>	4.82 (0.17)	4.69 (0.13)

<sup>a,b,c</sup> The same superscript letters showed statistically significant difference between before and after erosive agent immersion ( $P < 0.05$ )

paired *t*-test showed statistically significant differences in microhardness values of IPS d.SIGN between before and after immersion in green mango ( $P = 0.003$ ), cola soft drink ( $P = 0.009$ ) and 4% acetic acid for 16 h ( $P = 0.001$ ). In contrast, there was no statistically significant difference in microhardness values of the IPS e.max Ceram group between before and after immersion in any of the acidic agents ( $P > 0.05$ ).

EDX results are presented in Tables 5 and 6. The C element representing hydrocarbon contamination was disregarded according to Milleding et al. (22). EDX results of IPS d.SIGN obtained before immersion revealed that oxygen had the highest weight percentage (22.34 (0.73)), followed by silicon (21.27 (1.23)). Sulphur had the lowest weight percentage (0.19 (0.04)). After immersion in all the acidic agents, the percentage of Na, K, Ca, Zr, Al and Si elements significantly decreased ( $P < 0.05$ ), while Ba, Mg, O and S elements remained unchanged or even increased slightly ( $P > 0.05$ ). Similarly, oxygen and silicon were found to be the highest and sulphur the lowest weight percentage in IPS e.max Ceram (27.94 (1.31), 23.13 (1.37) and 0.42 (0.17), respectively). Immersion in all acidic agents except the citrate buffer solution and pineapple juice caused decrease in element percentage, mainly of Na, K, Zr, Al and Si ( $P < 0.05$ ), while Ti, Zn, O and S were not affected ( $P > 0.05$ ). The pineapple juice group showed a significant decrease in sodium, silicon and aluminium ( $P < 0.05$ ). Calcium was statistically decreased in 4% acetic acid and 4% acetic acid for 16 h groups ( $P = 0.008$  and  $P = 0.002$ , respectively). In comparison to before immersion, the deionized water groups of the two types of ceramics produced similar results, except for the magnitude of the decrease in Si and Al, but with no significant difference ( $P > 0.05$ ). Nevertheless, the decrease in surface elements after immersion of IPS d.SIGN was found to be more than that of IPS e.max Ceram.

## Discussion

The present study showed that the microhardness values

of IPS d.SIGN ceramics were affected by the acidic agents used: green mango, cola soft drink and 4% acetic acid for 16 h. These effects were not found in IPS e.max Ceram. Possible explanations for these results may include the effect of pH, environmental conditions and microstructural differences of both ceramics in determining the microhardness values.

The green mango juice was the most acidic agent (pH 2.39), followed by cola soft drink (pH 2.41) and acetic acid (pH 2.47). The fruit juices tested in this study were shown to be acidic as acetic acid, which is highly erosive to ceramic. Acidic agents used in this study, including pineapple and green mango, are the favorite sour fruits in many countries. They consist of citric acid and other organic acids (17-19). Cola, one of the most consumed soft drinks, consists of carbonic and citric acid and is therefore a highly acidic drink. These acids might cause the elemental dissolution of ceramics due to its chelating effect. Acetic acid is used for chemical stability testing according to ISO standard 6872 (21). Although acetic acid is a weak organic acid, it is fairly corrosive to glass due to its chelating effect. A similar effect has been found with citric acid. According to ISO 6872 (21), the chemical stability of the ceramics is tested at a high temperature, 80°C, at which changes in microhardness are normally recordable. These changes might not be found at lower temperature, for example, at 37°C. These results clearly demonstrated the effect of environmental conditions and high temperatures on ceramic degradation.

Before immersion, the IPS e.max Ceram generally had microhardness values lower than the IPS d.SIGN. A possible explanation for this result could be the microstructural differences between these two ceramics (4). The IPS d.SIGN, a feldspathic-based ceramic, is unique and distinct from other ceramics since its microstructure consists of fluorapatite and leucite crystal phases in a feldspathic glass matrix (2), while the IPS e.max Ceram consists of only dispersed fluorapatite crystals in a feldspathic glass matrix (3). Presence of leucite particles

Table 5 Mean elemental composition (wt.%) and standard deviations (S.D.) of IPS d.SIGN immersed in acidic agents<sup>#</sup> (the C element was disregarded (22))

Element and group	Mean elemental composition in wt% (S.D.)									
	IA		IIA			IVB	IIIA	IVA	VIA	
	Na	K	Ba	Mg	Ca	Zr	Al	Si	O	S
Before	4.18 (0.18)	7.38 (0.81)	0.28 (0.07)	0.29 (0.04)	1.33 (0.18)	2.20 (0.13)	6.07 (0.17)	21.27 (1.23)	22.34 (0.73)	0.19 (0.04)
Deionized water	5.08 (0.41)	7.41 (0.26)	0.28 (0.07)	0.31 (0.05)	1.49 (0.06)	2.21 (0.16)	6.05 (0.32)	21.20 (1.21)	23.73 (1.22)	0.21 (0.08)
Citrate buffer solution	3.18* (0.31)	4.29* (1.02)	0.33 (0.04)	0.36 (0.07)	1.06* (0.26)	1.71* (0.35)	5.31* (0.46)	19.85* (2.51)	21.74 (0.98)	0.23 (0.06)
Pineapple juice	3.39* (0.32)	1.46* (0.60)	0.29 (0.20)	0.32 (0.09)	0.22* (0.09)	1.01* (0.90)	4.42* (0.56)	15.72* (2.38)	21.35 (1.45)	0.19 (0.06)
Green mango juice	3.09* (0.80)	1.66* (0.48)	0.35 (0.24)	0.32 (0.06)	0.39* (0.13)	0.86* (0.27)	4.25* (0.80)	13.46* (2.74)	20.76 (1.97)	0.20 (0.08)
Cola soft drink	3.61* (0.42)	3.45* (0.83)	0.23 (0.08)	0.39 (0.11)	0.69* (0.22)	1.51* (0.27)	4.73* (0.46)	18.44* (2.65)	21.08 (1.34)	0.21 (0.08)
4% acetic acid	3.79* (0.16)	1.86* (0.74)	0.29 (0.12)	0.32 (0.07)	0.39* (0.08)	1.17* (0.19)	4.67* (0.45)	18.24* (2.17)	21.31 (1.23)	0.20 (0.09)
4% acetic acid for 16 h	3.41* (0.27)	1.99* (0.66)	0.29 (0.12)	0.33 (0.04)	0.17* (0.14)	0.34* (0.24)	3.83* (0.59)	14.23* (2.23)	21.81 (1.33)	0.20 (0.04)

<sup>#</sup> The elemental compositions are ordered according to the division of the periodic system

\* indicates significant difference from baseline value ( $P < 0.05$ )

Table 6 Mean elemental composition (wt.%) and standard deviations (S.D.) of IPS e.max Ceram immersed in acidic agents<sup>#</sup> (the C element was disregarded (22))

Element and group	Mean elemental composition in wt% (S.D.)									
	IA		IIA	IVB		IIIB	IIIA	IVA	VIA	
	Na	K	Ca	Zr	Ti	Zn	Al	Si	O	S
Before	6.41 (0.20)	3.52 (0.54)	1.38 (0.23)	1.25 (0.20)	0.47 (0.12)	0.47 (0.09)	4.38 (0.18)	23.13 (1.37)	27.94 (1.31)	0.42 (0.17)
Deionized water	7.01 (0.20)	4.47 (1.53)	1.40 (0.40)	1.28 (0.25)	0.61 (0.27)	0.48 (0.19)	4.12 (0.29)	22.48 (3.48)	28.82 (2.25)	0.45 (0.11)
Citrate buffer solution	7.23 (0.22)	4.22 (0.51)	1.41 (0.16)	1.34 (0.15)	0.66 (0.08)	0.50 (0.12)	4.20 (0.16)	22.40 (1.28)	26.20 (1.17)	0.42 (0.17)
Pineapple juice	5.88* (0.26)	3.42 (0.42)	1.25 (0.15)	1.40 (0.17)	0.64 (0.08)	0.49 (0.11)	4.02* (0.23)	21.68* (1.54)	28.04 (1.13)	0.43 (0.15)
Green mango juice	5.03* (1.23)	2.41* (0.42)	1.24 (0.17)	0.80* (0.42)	0.40 (0.07)	0.48 (0.15)	3.32* (0.23)	17.65* (1.96)	28.73 (1.14)	0.42 (0.15)
Cola soft drink	3.63* (0.58)	2.12* (0.48)	1.30 (0.42)	0.86* (0.33)	0.66 (0.12)	0.47 (0.27)	3.85* (0.32)	21.07* (4.80)	27.52 (2.22)	0.44 (0.37)
4% acetic acid	3.44* (0.16)	1.89* (0.37)	1.21* (0.16)	0.85* (0.13)	0.51 (0.09)	0.49 (0.13)	3.28* (0.41)	16.84* (1.00)	27.49 (0.78)	0.43 (0.20)
4% acetic acid for 16 h	4.29* (0.58)	0.40* (0.19)	1.19* (0.14)	0.76* (0.12)	0.49 (0.11)	0.48 (0.21)	3.77* (0.38)	13.66* (1.59)	27.20 (1.39)	0.42 (0.11)

<sup>#</sup> The elemental compositions are ordered according to the division of the periodic system

\* indicates significant difference from baseline value ( $P < 0.05$ )

in IPS d.SIGN causes ceramic degradation in a higher degree than IPS e.max Ceram. In feldspathic-based ceramics, a large thermal contraction mismatch between leucite ( $22$  to  $25 \times 10^{-6}/^{\circ}\text{C}$ ) and the glass matrix ( $8 \times 10^{-6}/^{\circ}\text{C}$ ), associated with the displacive phase transformation of leucite from cubic to tetragonal, leads to the development of tangential compressive stresses in the glass around the leucite crystal upon cooling (23-25). Such residual stresses may counteract the crack driving force, and act as crack deflectors, resulting in improved strength or even increase in hardness of the ceramic (26).

Nevertheless, leucite crystals may also appear to be accompanied by an increase in the microhardness which decreases after immersion of IPS d.SIGN in the acidic agents. The leucite particles contract more than the surrounding glass when the ceramic is cooled. In addition to the critical particle size, stress created during cooling can induce microcracks circumferential to the leucite particles (27), resulting in increased susceptibility to acid induced degradation of the ceramics (28) from acidic agents. Previous studies have documented that the size of the leucite particles in feldspathic-based ceramics increase during heat treatment within the normal ceramic firing range (29,30). This can increase the probability of microcracking (31). In contrast to fluorapatite ceramics, the fluorapatite phase particles have a needle-like morphology and possibly contribute to high chemical durability (4). However, compared to the microhardness of enamel (2.94 to 4.08 GPa) (32,33), IPS d.SIGN and IPS e.max Ceram had higher microhardness than enamel even after immersion in acidic agents.

Pinto et al. (34) investigated the effect of pH of the storage medium on slow crack growth of two types of ceramics, with and without leucite particles. They concluded that the leucite-based ceramic was more susceptible to slow crack growth in acidic conditions compared to basic and neutral conditions. In addition, fluorapatite particles are chemically durable (4). This could explain why immersion in acidic agents had a more deleterious effect on IPS d.SIGN; a fluorapatite-leucite ceramic, than IPS e.max Ceram; a fluorapatite ceramic, as shown in the present study. However, further studies are required to elaborate this effect.

EDX analysis showed that the background values and standard deviations in this study were generally small, indicating well-controlled analytical procedures. The results demonstrated that surface compositions of both ceramics were dominated by oxygen, silicon, aluminium, sodium and potassium elements. These elements are the basic components of feldspathic-based ceramics. The surface elemental compositions determined by EDX

corresponded to the information provided by the manufacturer. However, phosphorus and fluoride were not detected in this study. This may be interpreted as an effect of the specimen preparation, during which some elements may be concentrated on the surface layers or may have evaporated from the surface layers. Another plausible explanation is that there might be a reaction between ceramic and water at the elevated temperature during firing (22). Nevertheless, a number of measurements were carried out at different locations on the specimen in this study. A few elements were not detected in the EDX either due to interference from peaks of other elements or simply due to the fact that the specific element was not available at the surface (22) or due to interferences from features below the surface (35).

After immersion of both ceramics in acidic agents, as seen in EDX analysis, decrease in Si, Al, Na, K and Zr elements in both ceramics was observed, which is in accordance with previous studies (10,22). The present study showed that none of the examined ceramics were chemically inert. A very low level of degradation was observed, even in a neutral aqueous environment (the deionized water group). Even though the dental ceramics were not simple sodium-silicate glasses, the materials, nevertheless, dominated by the glass matrix and the reactions of the primary glass network former, silica, largely controlled the degradation process. It is generally believed that two dominant mechanisms are responsible for the aqueous corrosion of sodium-silicate glasses, such as dental ceramics (11). First, the selective leaching out of alkali ions and secondly, dissolution of the glass network (Si-O-Si). These mechanisms are controlled by the diffusion of hydrogen ions or hydronium ions ( $\text{H}_3\text{O}^+$ ) from an aqueous solution into the glass and loss of alkali ions from the glass surface to maintain electrical neutrality. In general, metal alkali ions from glass are much less stable in the glass matrix phase than in the crystalline phase; and thus will leach out more easily. Decrease in silicon might be due to the breakdown of the Si-O-Si bond, which impairs the entire ceramic structure. The leaching out of the more easily released elements, such as potassium and sodium, possibly created pores or channels within the glass matrix, resulting in increased diffusion of water molecules and development of internal breakage of localized Si-O-Si bond (10). However, further studies are required.

Acidic immersion caused higher level of elemental decrease in IPS d.SIGN when compared with IPS e.max Ceram. This could be the result of microstructural differences. Fluorapatite particles in IPS e.max Ceram are chemically durable (4), while leucite crystals in IPS

d. SIGN are susceptible to slow crack growth in acidic agents (34). The elemental reduction in IPS e.max Ceram did not result in microhardness degradation. However, this study evaluated only the short term effects. A long-term study on the effect of erosive agents on this ceramic is required.

The most significant finding of this experimental study is that fluorapatite-leucite ceramics could degrade in acidic agents or acidic food and drinks. However, it must be noted that there are some limitations to this study. During consumption, acidic food or drinks come in contact with ceramic restorations only briefly before they are washed away by saliva. This study did not account for the role of saliva. However, this study was designed to simulate the washing effect of saliva by cyclic specimen immersion between acidic agents and water. Moreover, the oral cavity presents a different testing environment. For example, the presence of water, temperature change, and pH level in the oral cavity may considerably affect properties of restorations. In addition, the present study evaluated only fluorapatite-leucite (IPS d.SIGN) and fluorapatite (IPS e.max Ceram) ceramics. Therefore, further studies are required to elaborate the effect on other dental ceramics.

Within the limitations of this study, it can be concluded that the microhardness values of fluorapatite-leucite ceramics were affected by acidic agents, especially green mango, cola soft drink and 4% acetic acid at 80°C for 16 h, while fluorapatite ceramics were not. The main composition of silicon, aluminium, sodium and potassium in both ceramics decreased after being immersed in acidic agents.

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