

## Effect of Acidic Agents on Surface Roughness of Dental Ceramics

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### ABSTRACT

**Background:** An increase in surface roughness of ceramics may decrease strength and affect the clinical success of ceramic restorations. However, little is known about the effect of acidic agents on ceramic restorations. The aim of this study was to evaluate the surface roughness of dental ceramics after being immersed in acidic agents.

**Methods:** Eighty-three ceramic disk specimens (12.0 mm in diameter and 2.0 mm in thickness) were made from four types of ceramics (VMK 95, Vitadur Alpha, IPS Empress Esthetic, and IPS e.max Ceram). Baseline data of surface roughness were recorded by profilometer. The specimens were then immersed in acidic agents (citrate buffer solution, pineapple juice and green mango juice) and deionized water (control) at 37°C for 168 hours. One group was immersed in 4% acetic acid at 80°C for 168 hours. After immersion, surface roughness was evaluated by a profilometer at intervals of 24, 96, and 168 hours. Surface characteristics of specimens were studied using scanning electron microscopy (SEM). Data were analyzed using two-way repeated ANOVA and Tukey's multiple comparisons ( $\alpha = 0.05$ ).

**Results:** For all studied ceramics, all surface roughness parameters were significantly increased after 168 hours immersion in all acidic agents ( $P < 0.05$ ). After 168 hours in 4% acetic acid, there were significant differences for all roughness parameters from other acidic agents of all evaluated ceramics. Among all studied ceramics, Vitadur Alpha showed significantly the greatest values of all surface roughness parameters after immersion in 4% acetic acid ( $P < 0.001$ ). SEM photomicrographs also presented surface destruction of ceramics in varying degrees.

**Conclusion:** Acidic agents used in this study negatively affected the surface of ceramic materials. This should be considered when restoring the eroded tooth with ceramic restorations in patients who have a high risk of erosive conditions.

**Keywords:** Aluminum oxide, Dental porcelain, Surface properties, Aluminum silicate.

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### Introduction

Dental ceramics are considered chemically inert restorative materials. However, many factors such as the composition, microstructure, chemical properties of the ceramic materials, erosive or acidic agents, exposure time, and the temperature, may influence the durability of dental ceramics.<sup>1</sup> Hence, previous studies have reported degradation of dental ceramics when exposed to aqueous solutions or acidic agents.<sup>2-9</sup> This condition results from selective releasing of alkaline ions,<sup>6-8</sup>

which are far less stable in the glassy phase than in the crystalline phase of dental ceramics.<sup>2</sup> The consequences of ceramic degradation are coarseness of the exposed surface,<sup>8-10</sup> increase in plaque accumulation<sup>2,6-8,10,11</sup> and wear to antagonist materials or teeth.<sup>2</sup> In addition, an increase in surface roughness of ceramics may decrease strength<sup>12,13</sup> and affect the clinical success and failure of ceramic restorations.<sup>14</sup>

Dental ceramics have different microstruc-

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tures, chemical compositions, and properties. Feldspathic ceramics enclose 19 weight percentage (wt%) of leucite crystals ( $K_2O \cdot Al_2O_3 \cdot 4SiO_2$ ) after incongruent melting of a mixture of glass and potassium feldspar.<sup>15</sup> Aluminous ceramics, the feldspathic-base ceramics, has increased the amounts of 40 to 50 wt% aluminum oxide crystals.<sup>16</sup> Feldspathic and aluminous ceramics are used as laminate veneers, inlays, onlays and as covering material for ceramic restorations. The high leucite ceramics contain up to 40-50 wt% leucite crystals.<sup>17</sup> Recently, fluorapatite ceramics are composed of dispersed fluorapatite crystals [ $Ca_{10}(PO_4)_6F_2$ ] in a feldspathic glassy matrix that results in a microstructure unlike that of any other commercially available dental ceramic.<sup>18</sup>

In general, the behavior of eating and chewing sour fruits, such as green mangoes, pineapples, and limes are most commonly found in tropical countries such as Australia, Cuba and also countries in southeast Asia.<sup>19-22</sup> People who frequently consume these foods often have a high incidence of dental erosion.<sup>23,24</sup> The potential erosive effect of these acidic food and beverages on enamel are affected primarily by the dissolution of apatite crystals.<sup>24,25</sup> However, little is known about their effect on ceramic restorations. The present study was conducted to evaluate changes of surface roughness and surface characteristics of 4 types of dental ceramics after being immersed in acidic agents (citrate buffer solution, green mango juice, pineapple juice, and 4% acetic acid) for 168 hours. The null hypothesis was that there would be no significant changes of surface roughness and surface characteristics of dental ceramics after immersion in acidic agents.

## Materials and Methods

### *Specimen preparations*

In this study, four types of dental ceramics were selected as representatives of the various ceramic types available. The studied ceramics were VMK 95 (Lot No. 7530, VITA Zanhfabrik, Bad Säckingen, Germany) feldspathic ceramic, Vitadur Alpha (Lot No.7435, VITA Zanhfabrik) aluminous ceramic, IPS Empress Esthetic (Lot No. JM0817, Ivoclar Vivadent AG, Schaan, Liechtenstein) high leucite ceramic, IPS e.max Ceram (Lot No. 583614, Ivoclar Vivadent) fluorapatite ceramic. A total of 83 disc specimens (12 mm in diameter

and 2 mm in thickness) from each ceramic were fabricated. The sintered ceramic specimens were made by a silicone mold (Provil, Heraeus Kulzer, Wehrheim, Germany) and then, cured according to the manufacturer's instructions. For IPS Empress Esthetic, the specimens were fabricated by heat press technique. Subsequently, the specimens were polished (Phoenix 4000, Buehler GmbH, Düsseldorf, Germany) under running water using 600 and 1,200-grit silicon carbide paper (3M ESPE, St.Paul, MN, USA) and were submitted to self-glazing according to the manufacturer's instructions. The exclusion criteria of the specimens were the presence of defects, pores, or cracks on the surfaces as evaluated by visual inspection and under a stereoscope (SMZ 1500m, Nikon Instech, Kanagawa, Japan) at  $\times 40$  magnification. All of the specimens were then ultrasonically cleaned in distilled water for 10 min and dried in a stream of oil-free compressed air and kept at room temperature before testing.

### *Storage agent immersions and surface roughness measurements*

Fifty ceramic disks of each type of the studied ceramics were divided into 5 groups (N=10). Surface roughness of the specimens was measured by a profilometer (Surfcorder SE-2300, Kosaka Laboratory Ltd., Tokyo, Japan) before immersion. The cut-off value for surface roughness was 0.8 mm and the traversing distance of the stylus was 4 mm. The radius of the tracing diamond tip was 5  $\mu m$ , and the measuring force and speed were 4 mN and 0.5 m/s, respectively. The surface roughness values ( $R_a$ ,  $R_{max}$ ,  $R_z$ , and  $S_m^{26}$ ; see Table 1) of each specimen were obtained in five different positions (1.5 mm apart). Four groups were immersed in 25 mL of three acidic agents (citrate buffer solution, pH 4.99; green mango juice, pH 2.37; and pineapple juice, pH 3.65) and deionized water (control) at 37°C for 168 hours. One group was immersed in 4% acetic acid, pH 2.45 at 80°C for 168 hours (as modified from ISO 6872<sup>27</sup>). The original pH of each acidic agent was measured using a pH meter (Orion model 900A, Orion Research Inc., Boston, MA, USA). Subsequently, the specimens were rinsed with deionized water, blotted dry and subjected to surface roughness testing at intervals of 24, 96, and 168 hours.

**Table 1.** Surface roughness parameters<sup>26</sup>

Ra	The arithmetical average of surface heights
Rmax	The magnitude of the peak-to-valley height in all cut-off lengths
Rz	The average height difference between the ten highest peaks and ten lowest valleys within each cut-off length
Sm	The arithmetical average spacing between peaks at the mean line over the cut-off length

**Surface topography analysis**

Three specimens of each ceramic before immersion and three specimens from each of the acidic agents at 96 and 168 hours immersion (n = 33) were examined by a scanning electron microscopy (JSM 5800LV, JEOL, Tokyo, Japan) to evaluate the surface topography. The specimens were rinsed with distilled water for 5 minutes, dried and fixed onto an aluminum cylinder (13 mm in diameter and 10 mm in height). Consequently, the specimens were sputter-coated with a gold-palladium alloy (SPI-Module sputter, SPI Supplies, West Chester, PA, USA) and evaluated under SEM.

**Statistical analysis**

The data were statistically analyzed using SPSS version 16. A two-way repeated analysis of va-

riance (ANOVA) was performed for each of the roughness parameters to assess the influence of different storage agents and ceramics on the surface roughness. Tukey's HSD multiple comparison was used in each of the parameters for comparison among the five storage agents. Within-group analysis was compared between the baseline value and each time point ( $\alpha = 0.05$ ).

**Results**

The results of two-way repeated ANOVA for each of the surface roughness parameters revealed significant differences among five storage agents and among four ceramic materials ( $P < 0.001$ ). All of the roughness parameters had no significant interactions between the type of storage agents and ceramics.

**Table 2.** Mean (standard deviation) values of roughness (Ra, Rmax, Rz, and Sm) of VITA VMK95 ceramic immersed in various storage agents at different times

Roughness parameter	Storage agent	Time (hours)			
		Before immersion	24	96	168
Ra ( $\mu\text{m}$ )	Deionized water	0.23 (0.09)	0.24 (0.08)	0.24 (0.12)	0.24 (0.07) <sup>c</sup>
	Citrate buffer	0.24 (0.04)	0.25 (0.04)	0.32 (0.04)*	0.34 (0.06) <sup>*.b</sup>
	Green mango juice	0.25 (0.05)	0.27 (0.12)	0.34 (0.08)*	0.36 (0.05) <sup>*.b</sup>
	Pineapple juice	0.24 (0.06)	0.25 (0.03)	0.26 (0.06)	0.33 (0.04) <sup>*.b</sup>
	4% Acetic acid	0.25 (0.08)	0.27 (0.09)	0.38 (0.08)*	0.46 (0.05) <sup>*.a</sup>
Rmax ( $\mu\text{m}$ )	Deionized water	5.58 (1.09)	5.59 (1.15)	5.59 (1.29)	5.60 (1.17) <sup>c</sup>
	Citrate buffer	5.63 (1.03)	6.21 (1.19)	7.85 (1.05)*	8.52 (1.02) <sup>*.b</sup>
	Green mango juice	5.59 (1.12)	7.17 (1.18)	8.21 (1.05)*	9.07 (1.03) <sup>*.b</sup>
	Pineapple juice	5.62 (1.08)	6.15 (1.44)	7.94 (1.02)*	8.26 (1.15) <sup>*.b</sup>
	4% Acetic acid	5.64 (1.07)	8.11 (1.23)	9.94 (1.08)*	10.89 (0.87) <sup>*.a</sup>
Rz ( $\mu\text{m}$ )	Deionized water	4.89 (1.21)	4.87 (0.81)	4.88 (0.93)	4.91 (1.02) <sup>c</sup>
	Citrate buffer	5.02 (1.01)	5.13 (1.12)	5.24 (0.91)	6.46 (1.03) <sup>*.b</sup>
	Green mango juice	4.91 (1.06)	4.95 (1.38)	6.03 (0.95)*	7.23 (1.06) <sup>*.b</sup>
	Pineapple juice	4.95 (0.71)	4.97 (1.02)	5.02 (1.02)	6.73 (0.98) <sup>*.b</sup>
	4% Acetic acid	5.04 (0.98)	5.89 (1.13)	7.23 (1.03)*	9.86 (1.07) <sup>*.a</sup>
Sm (mm)	Deionized water	0.25 (0.05)	0.26 (0.21)	0.26 (0.15)	0.27 (0.13) <sup>b</sup>
	Citrate buffer	0.24 (0.07)	0.27 (0.03)	0.28 (0.04)	0.29 (0.11) <sup>b</sup>
	Green mango juice	0.27 (0.09)	0.28 (0.02)	0.34 (0.05)*	0.36 (0.07) <sup>*.a</sup>
	Pineapple juice	0.27 (0.08)	0.28 (0.01)	0.29 (0.04)	0.30 (0.05) <sup>b</sup>
	4% Acetic acid	0.26 (0.05)	0.29 (0.05)	0.35 (0.05)*	0.38 (0.06) <sup>*.a</sup>

\* Indicates no significant difference between different storage times. Identical superscripted letters indicate no significant differences among five storage agents ( $P < 0.05$ ).

**Table 3.** Mean (standard deviation) values of roughness (Ra, Rmax, Rz, and Sm) of Vitadur Alpha ceramic immersed in various storage agents at different times

Roughness parameter	Storage agent	Time (hours)			
		Before immersion	24	96	168
Ra ( $\mu\text{m}$ )	Deionized water	0.24 (0.08)	0.24 (0.09)	0.24 (0.11)	0.24 (0.07) <sup>c</sup>
	Citrate buffer	0.25 (0.07)	0.26 (0.05)	0.33 (0.05)*	0.34 (0.07) <sup>*,b</sup>
	Green mango juice	0.24 (0.08)	0.27 (0.12)	0.33 (0.11)*	0.36 (0.05) <sup>*,b</sup>
	Pineapple juice	0.25 (0.09)	0.25 (0.03)	0.26 (0.06)	0.33 (0.07) <sup>*,b</sup>
	4% Acetic acid	0.25 (0.07)	0.29 (0.08)	1.09 (0.08)*	1.45 (0.06) <sup>*,a</sup>
Rmax ( $\mu\text{m}$ )	Deionized water	5.47 (1.10)	5.48 (1.08)	5.49 (1.21)	5.49 (1.09) <sup>c</sup>
	Citrate buffer	5.46 (1.08)	6.15 (1.08)	7.72 (1.10)*	8.43 (1.03) <sup>*,b</sup>
	Green mango juice	5.50 (1.09)	7.05 (1.03)	8.19 (1.04)*	8.87 (1.04) <sup>*,b</sup>
	Pineapple juice	5.52 (1.11)	6.12 (1.26)	7.85 (1.05)*	8.35 (1.15) <sup>*,b</sup>
	4% Acetic acid	5.49 (1.06)	7.04 (1.15)	11.57 (1.04)*	14.75 (1.06) <sup>*,a</sup>
Rz ( $\mu\text{m}$ )	Deionized water	4.91 (1.13)	4.89 (0.85)	4.91 (1.01)	4.92 (1.04) <sup>c</sup>
	Citrate buffer	4.97 (0.96)	5.04 (1.12)	5.19 (0.91)	6.74 (1.02) <sup>*,b</sup>
	Green mango juice	5.01 (1.03)	5.11 (1.38)	6.12 (0.96)*	7.35 (1.05) <sup>*,b</sup>
	Pineapple juice	4.85 (0.87)	4.98 (1.01)	5.11 (1.02)	6.82 (0.87) <sup>*,b</sup>
	4% Acetic acid	5.06 (1.05)	5.92 (1.10)	9.54 (1.06)*	10.78 (1.02) <sup>*,a</sup>
Sm (mm)	Deionized water	0.26 (0.08)	0.26 (0.10)	0.27 (0.12)	0.27 (0.09) <sup>b</sup>
	Citrate buffer	0.25 (0.06)	0.26 (0.04)	0.28 (0.05)	0.30 (0.12) <sup>b</sup>
	Green mango juice	0.26 (0.08)	0.28 (0.03)	0.35 (0.07)*	0.35 (0.09) <sup>*,b</sup>
	Pineapple juice	0.27 (0.07)	0.27 (0.03)	0.29 (0.05)	0.31 (0.06) <sup>b</sup>
	4% Acetic acid	0.27 (0.06)	0.29 (0.06)	0.42 (0.05)*	0.52 (0.06) <sup>*,a</sup>

\* Indicates no significant difference between different storage times. Identical superscripted letters indicate no significant differences among five storage agents ( $P < 0.05$ ).

The surface roughness values for Ra, Rz, Rmax and Sm parameters of the studied ceramics in different storage media and storage times were shown in Tables 2-5. Baseline values (before immersion) were recorded in order to verify the initial surface roughness which found not to be any significant differences among all groups. Ra, Rz and Rmax were significantly increased after 168 h immersion in all acidic storage agents ( $P < 0.05$ ). There were significant differences for Ra, Rmax, Rz and Sm after 168 h in 4% acetic acid from other groups ( $P <$

0.001) of all of the ceramics evaluated. There was no significant difference for Sm in 4% acetic acid and green mango juice of VMK 95, Empress Esthetic, and IPS e.max Ceram. Among all studied ceramics, Vitadur Alpha showed significantly the greatest values of all surface roughness parameters after immersion in 4% acetic acid ( $P < 0.001$ ) while in other acidic agents, there was no significant difference for all surface roughness parameters among all studied ceramics.

SEM photomicrographs of the studied ceramics,

before immersion and after 168 hours of immersion periods in different storage agents are presented in Figures 1-4. Before immersion, all of the ceramics tested showed mostly smooth surfaces, with small and little porosities. After exposure to all acidic agents and even deionized water, there were surface changes in various degrees. Immersion in 4% acetic acid of all of the ceramics tested showed the most

roughening patterns. The gradual degradation of specimen surfaces was observed; an increase of porosities corresponded with increasing immersion times. The largest defects were found when Vitadur Alpha was immersed in 4% acetic acid (Figure 2f). For IPS e.max Ceram, fluorapatite crystals could be observed before immersion, but disappeared after immersion (Figure 4).

**Table 4.** Roughness (Ra, Rmax, Rz, and Sm), mean and standard deviation values of IPS Empress Esthetic ceramic immersed in various storage agents at different times

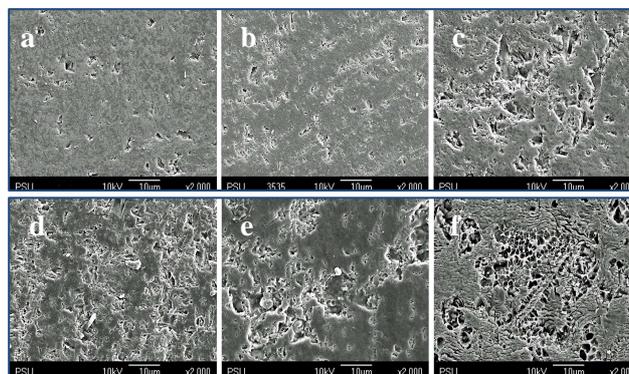
Roughness parameter	Storage agent	Time (hours)			
		Before immersion	24	96	168
Ra ( $\mu\text{m}$ )	Deionized water	0.22 (0.08)	0.23 (0.07)	0.24 (0.09)	0.24 (0.04) <sup>c</sup>
	Citrate buffer	0.23 (0.06)	0.24 (0.05)	0.31 (0.04)*	0.35 (0.07) <sup>*,b</sup>
	Green mango juice	0.24 (0.06)	0.26 (0.09)	0.32 (0.11)*	0.37 (0.08) <sup>*,b</sup>
	Pineapple juice	0.23 (0.08)	0.24 (0.05)	0.27 (0.05)	0.34 (0.06) <sup>*,b</sup>
	4% Acetic acid	0.24 (0.07)	0.27 (0.07)	0.37 (0.07)*	0.48 (0.07) <sup>*,a</sup>
Rmax ( $\mu\text{m}$ )	Deionized water	5.47 (1.02)	5.47 (1.06)	5.49 (1.18)	5.49 (0.96) <sup>c</sup>
	Citrate buffer	5.52 (0.97)	6.42 (1.07)	7.86 (1.04)*	8.52 (1.03) <sup>*,b</sup>
	Green mango juice	5.49 (1.02)	7.16 (1.03)	8.21 (0.95)*	9.07 (1.05) <sup>*,b</sup>
	Pineapple juice	5.51 (1.05)	6.39 (1.12)	7.94 (1.12)*	8.26 (1.11) <sup>*,b</sup>
	4% Acetic acid	5.53 (1.03)	7.42 (1.01)	9.85 (1.06)*	10.72 (0.85) <sup>*,a</sup>
Rz ( $\mu\text{m}$ )	Deionized water	4.82 (1.12)	4.82 (0.87)	4.83 (1.01)	4.83 (1.05) <sup>c</sup>
	Citrate buffer	4.91 (1.03)	5.03 (1.06)	5.26 (0.89)	6.25 (1.07) <sup>*,b</sup>
	Green mango juice	4.89 (1.05)	4.98 (1.17)	6.07 (0.94)*	7.05 (1.08) <sup>*,b</sup>
	Pineapple juice	4.93 (0.92)	4.97 (1.14)	5.22 (1.06)	6.33 (0.96) <sup>*,b</sup>
	4% Acetic acid	5.01 (0.96)	5.76 (1.12)	7.36 (0.92)*	9.69 (1.04) <sup>*,a</sup>
Sm (mm)	Deionized water	0.23 (0.04)	0.23 (0.11)	0.24 (0.12)	0.25 (0.12) <sup>bp</sup>
	Citrate buffer	0.24 (0.03)	0.25 (0.03)	0.28 (0.06)	0.29 (0.07) <sup>b</sup>
	Green mango juice	0.26 (0.07)	0.28 (0.02)	0.34 (0.03)*	0.36 (0.06) <sup>*,a</sup>
	Pineapple juice	0.25 (0.06)	0.27 (0.04)	0.28 (0.04)	0.30 (0.05) <sup>b</sup>
	4% Acetic acid	0.24 (0.04)	0.28 (0.03)	0.35 (0.06)*	0.37 (0.07) <sup>*,a</sup>

\* Indicates no significant difference between different storage times. Identical superscripted letters indicate no significant differences among five storage agents ( $P < 0.05$ ).

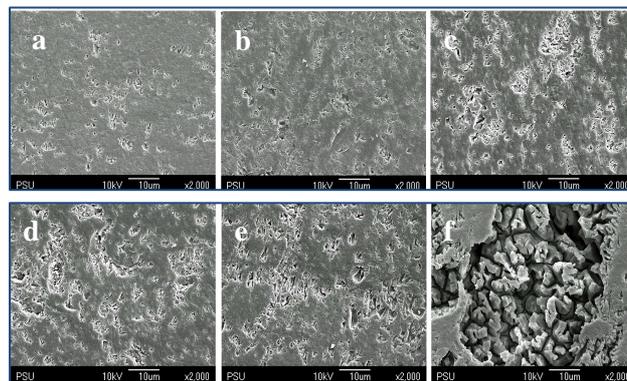
**Table 5.** Roughness (Ra, Rmax, Rz, and Sm), mean and standard deviation values of IPS e.max Ceram ceramic immersed in various storage agents at different times

Roughness parameter	Storage agent	Time (hours)			
		Before immersion	24	96	168
Ra ( $\mu\text{m}$ )	Deionized water	0.24 (0.08)	0.24 (0.07)	0.25 (0.13)	0.25 (0.06) <sup>c</sup>
	Citrate buffer	0.25 (0.06)	0.25 (0.05)	0.32 (0.05)*	0.34 (0.06) <sup>*.b</sup>
	Green mango juice	0.24 (0.08)	0.26 (0.11)	0.33 (0.09)*	0.36 (0.04) <sup>*.b</sup>
	Pineapple juice	0.24 (0.05)	0.25 (0.05)	0.30 (0.06)	0.33 (0.08) <sup>*.b</sup>
	4% Acetic acid	0.25 (0.07)	0.27 (0.08)	0.39 (0.05)*	0.46 (0.07) <sup>*.a</sup>
Rmax ( $\mu\text{m}$ )	Deionized water	5.51 (0.92)	5.51 (1.04)	5.52 (1.12)	5.52 (1.03) <sup>c</sup>
	Citrate buffer	5.49 (1.01)	6.14 (1.08)	7.58 (1.05)*	8.45 (1.06) <sup>*.b</sup>
	Green mango juice	5.48 (1.07)	7.03 (1.21)	8.13 (1.06)*	9.05 (1.01) <sup>*.b</sup>
	Pineapple juice	5.52 (1.05)	6.45 (1.35)	7.49 (0.92)*	8.37 (1.12) <sup>*.b</sup>
	4% Acetic acid	5.54 (1.17)	8.04 (1.13)	9.85 (1.08)*	10.91 (1.04) <sup>*.a</sup>
Rz ( $\mu\text{m}$ )	Deionized water	4.95 (1.12)	4.95 (0.83)	4.96 (0.97)	4.96 (1.04) <sup>c</sup>
	Citrate buffer	4.97 (1.03)	5.21 (1.12)	5.25 (0.99)	6.62 (1.03) <sup>*.b</sup>
	Green mango juice	5.01 (1.07)	5.09 (1.21)	6.05 (0.84)*	7.14 (0.96) <sup>*.b</sup>
	Pineapple juice	4.89 (0.93)	5.13 (1.02)	5.22 (1.04)	6.75 (1.04) <sup>*.b</sup>
	4% Acetic acid	5.02 (1.04)	5.86 (1.03)	7.65 (0.91)*	9.82 (1.03) <sup>*.a</sup>
Sm (mm)	Deionized water	0.25 (0.07)	0.26 (0.12)	0.26 (0.09)	0.27 (0.09) <sup>b</sup>
	Citrate buffer	0.26 (0.05)	0.27 (0.05)	0.28 (0.06)	0.29 (0.07) <sup>b</sup>
	Green mango juice	0.24 (0.06)	0.28 (0.03)	0.34 (0.05)*	0.35 (0.07) <sup>*.a</sup>
	Pineapple juice	0.26 (0.05)	0.27 (0.06)	0.29 (0.05)	0.29 (0.04) <sup>b</sup>
	4% Acetic acid	0.27 (0.07)	0.29 (0.04)	0.34 (0.06)*	0.37 (0.05) <sup>*.a</sup>

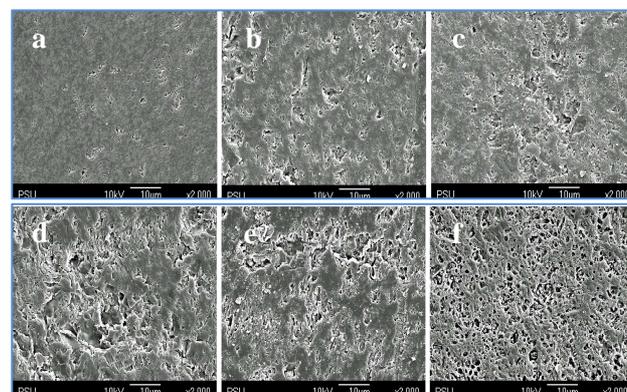
\* Indicates no significant difference between different storage times. Identical superscripted letters indicate no significant differences among five storage agents ( $P < 0.05$ ).



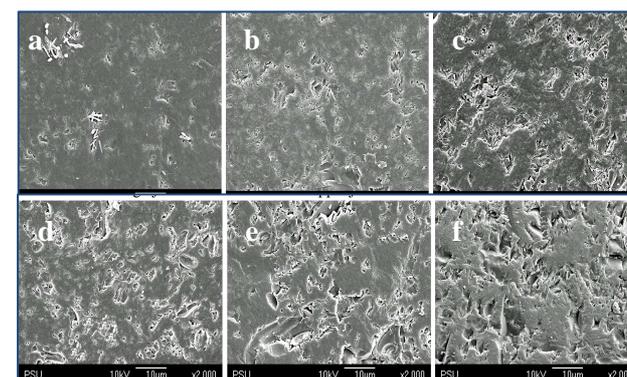
**Figure 1.** Scanning electron microscope photomicrographs of VITA VMK95 (a) before immersion, and after immersion in (b) deionized water, (c) citrate buffer solution, (d) green mango juice, (e) pineapple juice, and (f) 4% acetic acid for 168 hours. Original magnification  $\times 2000$ .



**Figure 2.** Scanning electron microscope photomicrographs of Vitadur Alpha (a) before immersion, and after immersion in (b) deionized water, (c) citrate buffer solution, (d) green mango juice, (e) pineapple juice, and (f) 4% acetic acid for 168 hours. Original magnification  $\times 2000$ .



**Figure 3.** Scanning electron microscope photomicrographs of IPS Empress Esthetic (a) before immersion, and after immersion in (b) deionized water, (c) citrate buffer solution, (d) green mango juice, (e) pineapple juice, and (f) 4% acetic acid for 168 hours. Original magnification  $\times 2000$ .



**Figure 4.** Scanning electron microscope photomicrographs of IPS e.max Ceram (a) before immersion, and after immersion in (b) deionized water, (c) citrate buffer solution, (d) green mango juice, (e) pineapple juice, and (f) 4% acetic acid for 168 hours. Original magnification  $\times 2000$ .

## Discussion

The results of the present study support rejecting the null hypothesis since the acidic agents used and even deionized water caused rough surfaces to the

ceramics tested. In oral cavity, the ceramic restorative materials would be exposed to the various temperature and acidic-base changes from food and beverages. Hence, the ceramic materials should resist or have only little changes in these environments.

Previous studies have reported a clinical service life of metal-ceramic restorations up to 20 years.<sup>28,29</sup> The speed of the degradation process was accelerated by increasing the aggressiveness of the agent compared with intraoral conditions. Therefore, a long immersion time was used in this study as an alternative for demonstrating the extensive effect of acidic solutions. The 168 hours used here is comparable to 22 years of immersion in artificial saliva at 22°C.<sup>8</sup>

The results of roughness values in the present study demonstrated that none of the ceramics evaluated were found to be chemically inert. A very low level of roughness or degradation occurred even in a neutral aqueous condition (the deionized water) as observed on SEM photomicrographs, although there was no significant difference in surface roughness. The two dominant mechanisms have been explained for this degradation process.<sup>2</sup> Firstly, the selective leaching of alkali ions, and secondly, the dissolution of the ceramic silicate network (Si-O-Si). These mechanisms are controlled by the diffusion of hydrogen ions or hydronium ions (H<sub>3</sub>O<sup>+</sup>) from an aqueous solution into the ceramic and loss of alkali ions from the ceramic surface into an aqueous solution to maintain electrical neutrality.<sup>2</sup> The results in rough surfaces are seen on SEM photomicrographs and roughness values in this study.

Contact and non-contact methods are currently used for surface roughness measurement.<sup>30</sup> Non-contact methods use a light beam or a laser beam to obtain a surface profile. One of the disadvantages of this method is that shiny surfaces are sometimes difficult to measure due to the scattering effect of the reflected light. This can cause false values being documented.<sup>30</sup> Therefore, a contact method with a profilometer was used in the present study. Although it has been claimed that when using this contact method, the stylus tip may damage or alter the surfaces tested,<sup>26</sup> the results of this study as seen on the SEM showed that this method did not cause any damage to ceramic surfaces. There were no scratches observed on the SEM because the measuring force applied by the stylus tip was 4 mN, which was very little.

The Ra value is the most commonly used roughness parameter in both dentistry and engineering.<sup>31</sup> Nevertheless, the Ra value is limited by the two-dimensional aspect, providing only information on the average roughness height, and also giving no information at all on the surface profile.<sup>26</sup> To clearly demonstrate given this limitation, this study used

additional roughness parameters, Rmax, Rz, and Sm, including photomicrographs of SEM. Rmax and Rz represented the amplitude parameter in the vertical axis which showed how the depth of degradation was. Sm showed the average spacing between the peaks which presented how the width of alteration was. The combination of quantitative measurements and qualitative data by microscopy supports a qualitative value in three dimensions of the surface tested.<sup>30-32</sup> As seen from the roughness results of the present study along with SEM photographs, it was clearly showed that 4% acetic acid significantly caused rough surfaces to the ceramics evaluated in all dimensions ( $P < 0.05$ ). In addition, roughness measurements achieved from relatively short scans may not be representative of the whole surface. Therefore, many measuring scans are required when using a profilometer. It was shown in the present study that the roughness values of the ceramics corresponded with those of other studies.<sup>30,31,33</sup> Regarding the types of studied ceramics, Vitadur Alpha revealed the greatest degradation after immersion in 4% acetic acid. This result seemed to show that alumina crystals in Vitadur Alpha had the least durability compared with leucite crystals in IPS Empress Esthetic and VMK 95 and fluorapatite crystals in IPS e.max Ceram. Leucite and fluorapatite crystals appeared to have a comparable durability. However, further study is required to support this result.

Previous studies have documented that increasing surface roughness of ceramics may decrease strength.<sup>12,13</sup> Another study also reported that the critical mean Ra for the adhesion and colonization of bacteria on restorative materials was 0.2  $\mu\text{m}$ .<sup>34</sup> This value is equivalent to the Ra value of ceramics evaluated in this study before immersion. The increasing surface roughness of the studied ceramics may cause bacterial colonization, strength reduction of the ceramics evaluated, and would result in clinical failure of ceramic restorations.

Green mangoes and pineapples are favorite sour fruits in many Asian countries. They consist of citric acid, which has been reported as a harmful acid that can cause dental erosion.<sup>23,25</sup> This citric acid might affect a change to the surface roughness of ceramics due to its chelating effect.<sup>8</sup> This effect takes place by complex binding of citrate molecules (as chelating acids) to dissolved metal ions of ceramics in acidic agents, and results in more ion dissolution of ceramics to maintain electrical neutrality.<sup>1</sup> Acetic

acid is the acid used for chemical stability testing in accordance with ISO standard 6872.<sup>27</sup> Acetic acid is a weak organic acid, however, it is fairly corrosive to ceramics because of its chelating effect.<sup>1</sup> A similar effect has been found in citric acid.

This investigation was an *in vitro* study and the different oral conditions such as saliva,<sup>35</sup> water, the pH level, and temperature changes may affect the results. Therefore, further studies are required to elaborate the effect of acidic agents on dental ceramics *in vivo*.

### Conclusion

Within the limitations of this *in vitro* study, it can be concluded that the surface roughness of the evaluated ceramics were negatively affected by the acidic agents tested, citrate buffer solution, green mango juice, pineapple juice, and 4% acetic acid at 80°C for 168 hours. This fact should be taken into consideration when restoring the affected tooth with ceramic restorations in patients who have a high risk of erosive conditions.

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