

In Vitro Study of the Effect of Three Hydrogen Peroxide Concentrations on the Corrosion Behavior and Surface Topography of Alumina-Reinforced Dental Ceramic

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Abstract

Purpose: This in vitro investigation studied the effect of three hydrogen peroxide (HP) concentrations (30%, 35%, 38% v/v) at two time intervals (1 and 2 hours) on the corrosion behavior and surface topography of a dental ceramic.

Materials and Methods: A total of 62 Vitadur Alpha discs were constructed following manufacturer instructions. Specimens were divided into four main groups (n = 8). Group 1 (control): specimens were immersed in 4% acetic acid for 18 hours at 80°C. Groups 2, 3, and 4: specimens were immersed in 30%, 35%, and 38% HP concentrations, respectively. Each of the three groups was divided into two subgroups (a and b) according to the immersion time (1 and 2 hours, respectively). Specimens of subgroup a were further immersed in 4% acetic acid for 18 hours at 80°C and were designated as subgroup c. The corrosion behavior of the ceramic specimens were tested by solution analysis using the atomic absorption method, weight loss percent, and corrosion rate. Surface topography was investigated by surface roughness (Ra) measurements and scanning electron microscopy (SEM). Results were statistically analyzed.

Results: There was a significant increase for ions leached with the increase in time of immersion for all ions at 35% and 38% HP, while at 30% HP, ions of K⁺, Al³⁺, and Si⁴⁺ did not increase significantly with time. The results also showed that at a fixed time of immersion, all ions released were dependent on the increase of HP concentration except for Al³⁺ ions (p < 0.05). The combined treatment of specimens with HP followed by acetic acid had a significant effect on the increase of ions leached (p < 0.05). The surface roughness values for all specimens increased significantly with time of immersion as well as with the increase in concentration of HP (p < 0.05). These results were confirmed with SEM.

Conclusions: The amount of released ions is directly proportional to HP concentration and time of immersion. Specimens exposed to both HP and acetic acid showed increased weight loss and a higher corrosion rate than those exposed to acetic acid only. Surface roughness values were time and HP concentration dependent.

Tooth whitening is one of the most rapidly growing areas in dentistry. In recent years, there has been an increased demand for tooth bleaching to improve the whiteness and perceived esthetic appearance of tooth tissue.^{1–3} Many bleaching techniques, ranging from over-the-counter products to sophisticated in-office bleaching systems, are now available to satisfy demand.⁴

Bleaching commonly uses hydrogen peroxide (HP), either directly or via its generation from carbamide peroxide.⁵ HP concentrations may vary enormously among bleaching systems, reaching 35% to 38% (v/v) for in-office techniques.⁶ Despite widespread debate, there is currently a trend toward employing greater concentrations of HP in tooth bleaching preparations.^{7,8}

Despite increased popularity, controversy surrounds the use of peroxide-based bleaching systems to whiten teeth. Some studies have suggested that the relatively high concentrations of peroxide used for bleaching altered the chemical structure of tooth tissues.^{9,10} In these studies, bleaching does not appear to cause macroscopic changes to the dental hard tissues; however, microscopic changes have been reported, particularly where peroxide was applied at high concentrations.^{11,12} Therefore, it was concluded that, as assessed by scanning electron microscopy (SEM), slight surface alterations and a decrease of surface microhardness and fracture toughness might occur due to bleaching of dental hard tissues.^{13–17}

It is important to consider other potential interactions that may occur in the oral cavity. The effect of such strong oxidizing agents on the properties of restorative materials should be carefully investigated.¹⁸

The literature presents controversial findings regarding the influence of bleaching agents on physical properties and surface morphology of dental materials. While most of this research assessed the effect of bleaching on direct restorative materials,^{19–21} some research has studied the effect of bleaching agents on ceramics.^{22–27} Those studies mainly investigated the effect of bleaching agents on hardness and roughness of different ceramic surfaces.

One of the earliest investigations on the effect of bleaching agents on ceramic restorative materials was performed by Turker and Biskin,²² who evaluated the effect of bleaching agents on the hardness of feldspathic porcelain among other restorative materials. They observed that 10% to 16% carbamide peroxide gels (applied for 8 hours per day for 30 days) were able to decrease surface hardness of the porcelain material tested. The same authors conducted a further study²³ to investigate the effect of some bleaching agents on the surface roughness of low-fusing porcelain (Duceram, Ducera Dental GmbH, Rosbach, Germany) among other restorative materials. They found that surface roughness values increased significantly during the first 2 weeks of application; however, Schemehorn et al,²⁴ in an SEM investigation, reported that surface roughness of feldspathic porcelain was not affected by bleaching with 6% HP gel.

Investigations continued using different concentrations and techniques aiming to simulate both in-office and at-home bleaching circumstances. Butler et al²⁵ reported increased roughness of three porcelain formulations as a result of the application of 10% carbamide peroxide. Moraes et al²⁶ and Kamala and Annapurni²⁷ reached the same conclusion after using 35% and 16% carbamide peroxide, respectively. Dental ceramics are considered the most chemically inert restorative materials,²⁸ hence, very little concern has been directed toward investigating their corrosion behavior and ionic leach.

Corrosion of ceramics may be studied by many procedures ranging from the simplest optical observations to more sophisticated bulk analysis.²⁹ It is advisable to understand the environment where the ceramic is to be used and then to monitor some parameters and extreme conditions of this environment to reach the appropriate procedural tests to perform. Anusavice³⁰ stated that the degradation of dental ceramics in the oral environment generally occurs because of mechanical forces, chemical attacks, or a combination. Some studies showed rapid ceramic surface deterioration in the presence of acidic fluorides.^{31,32} These corrosive processes, in addition to deteriorating mechanical properties, increase the surface roughness of the restoration. This, in turn, changes the color of the ceramic restoration and causes wear of the antagonistic teeth or restorative materials.^{30,33,34} Increased surface roughness will also lead to plaque adhesion, thus changing the interaction of the clinical restoration with the biologic environment.³⁴ Reduction of chemical stability induces further elution of inorganic ions from the surface of dental ceramic materials; these may have a toxic influence on vital body structures.²⁸

The main factors influencing the stability of dental ceramics are type and composition of the corrosive solution and duration of immersion.^{35–37} Hydrochloric and acetic acids have been used for testing the chemical stability of ceramic restorations because they cause faster material degradation, offering longterm prediction of dental ceramic stability. In addition, the use of acids allows testing for situations when pH is lowered in the oral cavity. pH values in the oral cavity vary considerably. Therefore, ISO standard 68721 uses 4% acetic acid for chemical stability testing of dental ceramics.³⁸ The influence of all kinds of acidic solutions and bleaching agents, including those found in toothpastes, on the teeth and involved restorations are practically inseparable. Although much research has focused on the effect of HP on hardness or surface roughness of teeth, very little has been concerned with the effect on the corrosion of ceramic restorations.

The aim of this study was to examine the effect of three HP concentrations (30%, 35%, 38%), commonly used in inoffice and in-waiting-room procedures, due to fast whitening results, on the corrosion behavior and surface topography of Vitadur Alpha dental ceramic. The study was performed at 1 and 2 hours, typical bleaching time in moderate and darkly stained cases, respectively. In addition, the study aimed to test the combined effect of HP and acetic acid on the corrosion behavior of the selected ceramic using ISO standard 6872, claimed to be equivalent to 22 years of intraoral use.

Materials and methods

Specimen construction

A total of 62 porcelain discs were fabricated from Vitadur Alpha (Vita Zahnfabrik, Bad Sackingen, Germany). X-ray fluorescence (Philips X-Unique II, Philips, Inc., Eindhoven, The Netherlands) analysis was carried out for three specimens to determine the chemical composition of the ceramic specimens. Table 1 shows the percent of constituting elements. The discs (10 mm diameter, 2 mm thick) were fabricated using a split stainless steel mold for standardization. The porcelain powder (shade A3) was mixed with modeling liquid. The slurry was packed and condensed into the mold placed on a glass slab to ensure a smooth surface. The compacted non-sintered

 Table 1 Percentage of constituting elements as received from X-ray fluorescence test

Element oxide	Percentage
SiO ₂	62.0% to 65.0%
Al ₂ O ₃	14.0% to 15.0%
CaO	1.0% to 2.0%
K ₂ O	7.0% to 8.0%
Na ₂ O	4.0% to 5.0%
TiO ₂	≈0.01%
P_2O_3	≈0.01%
Fe ₂ O ₃	≤0.01%
MnO, MgO, ZrO ₂	Traces



Figure 1 Specimen grouping and experiments.

specimens were transferred to the furnace on a thermal cotton pad and fired according to the manufacturer's recommendations in the Vita Vacuumed oven (Vita Zahnfabrik) at 960°C. To compensate for the shrinkage firing of ceramic, defective specimens were adjusted by porcelain slurry addition and corrective firings (the weight of the specimens must be standard \pm 0.001 g). The fired discs were air cooled to room temperature. The ceramic specimens were finished with a medium-grit diamond bur (Brasseler size 016 #848-11; Brasseler USA, Savannah, GA) on both sides to remove any irregularities and to create a flat surface. The discs were auto-glazed according to the manufacturer's recommendations by firing at 940°C for 1 minute.

Specimen grouping

The discs were divided into four main groups (n = 8) as follows:

Group 1 (control): Discs were immersed in 4% acetic acid for 18 hours at 80°C with continuous shaking at a constant rate, according to ISO standard 6872, to evaluate the hydrolytic resistance for dental ceramics, equivalent to 22 years in use in the oral cavity.

Groups 2, 3, and 4: Discs were immersed in 30%, 35%, and 38% HP (v/v), respectively. Each of these three groups was divided into two subgroups according to the time of immersion in the HP concentration: subgroup a specimens immersed in HP for 1 hour and subgroup b specimens immersed in HP for 2 hours at 37° C. The specimens of subgroup a were removed from the relevant HP solution and were washed for 1 minute in distilled water and left to dry at room temperature for 15 to 20 minutes. The specimens were further immersed in 4%

acetic acid for 18 hours at 80°C with continuous shaking and were designated as subgroup c for each of the three tested HP concentrations. Figure 1 shows the groups' specimens.

Chemicals and solutions

All chemicals used were British Drug Houses (BDH, Dorset, UK) reagent-grade chemicals. The required solutions of these chemicals were prepared using deionized water.

Thirty milliliters of each of the prepared chemical solutions were placed in a Pyrex-quick fit tube (30 cm long, 3 cm diameter). Ceramic specimens were weighed to the sixth decimal using a standard balance (A T20 model 2003, Mettler-Toledo, Columbus, OH) before immersion in the relative corrosive solution and after immersion in the relative studied solution to determine weight loss. The pH values of the prepared solutions were measured using a digital pH meter (Orion pH meter with a combined glass electrode, ThermoScientific, Geel, Belgium). Table 2 shows the chemical composition of studied solutions and the corresponding pH values at the beginning and at the end of each experiment. The tube was fitted to a multiwrist shaker (Lab-Line Instruments, Inc., Melrose Park, IL), which was joined to a water thermostat (mlw-16 thermostat with $\pm 1.0^{\circ}$ C accuracy). The temperature was raised gradually to avoid cracks in the ceramic discs. The tube was shaken for the required period at the selected temperature (18 hours at 80°C for groups 1, 2c, 3c, and 4c; 1 hour at 37°C for groups 2a, 3a, and 4a; 2 hours at 37°C for groups 2b, 3b, and 4b). Figure 2 shows the multi-wrist shaker and the connected test tube where the corrosion tests were performed. Due to the higher reactivity of

 Table 2
 Concentrations and the corresponding pH values of solutions used

Solution	Composition	pH value
Acetic acid	4% v/v glassial acetic acid	2.40, 2.9 after 18 hours, at the end of the experiment
30% H ₂ O ₂	60 ml of 50% H ₂ O ₂ completed to 100 ml by deionzed water	2.60, 2.67 after 1 hour, 2.77 after 2 hours
35% H ₂ O ₂	70 ml of 50% H ₂ O ₂ completed to 100 ml by deionzed water	2.30, 2.430 after 1 hour, 2.433 after 2 hours
38% H ₂ O ₂	76 ml of 50% H ₂ O ₂ completed to 100 ml by deionzed water	2.10, 2.19 after 1 hour, 2.23 after 2 hours

was used (Perken Elmer Spectrophotometer, Perken Elmer, Waltham, MA). The system consists of an atomic absorption spectrophotometer instrument and atomizer. It provides complete control over wavelength selection and range, background correction intervals, and concentration values up to 0.001 ppm.

Corrosion determination by mass loss

The specimens were weighed before and after corrosion to the sixth decimal, and the weight loss percentage was determined using the following equation:

Mass loss% = average
$$\left(\frac{\text{mass loss}}{\text{mass of specimen}}\right) \times 100.$$
 (1)

Determination of the corrosion rate

The corrosion rate was calculated using the following equation:

Corrosion rate $(\mu g cm^{-2} h^{-1}) = average \left(\frac{mass loss (\mu g)}{area(cm^2) \times time(h)}\right)$ (2)

HP, as it breaks easily into H_2O and $O\bullet$, the experiments were performed in slightly dark room. At the end of each experiment, the tube was brought to room temperature, and the ceramic discs were removed with tweezers from the corrosive solution. The discs were washed with deionized water and methanol (BDH), and left to dry at room temperature for 20 minutes to ensure the dryness of the specimens. The studied solution of each group was subjected to atomic absorption analysis.

Corrosion experiments

Three corrosion-related parameters were used to judge the corrosion behavior of Vitadur Alpha; analysis of solutions to determine the concentration of leached ions from the tested ceramic, corrosion rate, and weight loss percentage.

Solution analysis using atomic absorption method

The corrosion solutions were analyzed after the specified time intervals for the leached elements by atomic absorption technique. An analyst 100/300 Atomic Absorption apparatus



Figure 2 Test tubes with corrosive solutions and ceramic specimens, immersed into the water path thermostat. They are connected to the multi-wrist shaker.

Scanning electron microscopy

Two specimens were selected blindly from all studied groups to be examined by the same examiner. The specimens were cleaned in pure ethanol for 3 minutes, rinsed in an ultrasonic bath, dried with absorbent paper, then coated with a thin layer (200-300 nm) of gold using gold sputter apparatus (JEOL, JEC-1100, Tokyo, Japan). The surfaces were examined by SEM (Philips XL 30, Philips, Inc.), and the results were presented as photographs.

Surface roughness values (Ra values)

Three selected blank specimens were tested for surface roughness and considered as baseline. Randomly selected specimens of each group were studied using a profilometer (Surftest SJ 201, Mitutoyo Corp., Kawasaki, Japan). Ra values of these specimens were compared to baseline surface measurements obtained from blank specimens that did not receive any treatment.

Statistical analysis

The recorded values of leached ions were converted into microgram per milliliter for ease of statistical calculation. A paired *t*-test was used to determine the interaction between concentration of released ions and time of immersion within each HP group and ion release difference between the control group and subgroups of combined effects of HP and acetic acid. One-way ANOVA was used to test the interaction between ion release concentration and fixed time of immersion at the three tested HP groups. Two-way ANOVA was used to study the interaction between ions released and the combined effect of acetic acid and HP as corrosive solutions. Surface roughness measurements were analyzed between each HP group and the blank specimens immersed in distal water. Statistical results were compared between the control group and groups 2, 3, and 4c using two-way ANOVA.

Group	Subgroup	Na	К	Са	Al	Si	Total
30%	1 hr	12.85 (1.3)	3.79(1.1)	7.3 (0.3)	3.62 (1.34)	1.46 (0.45)	29.16 (2.12)
	2 hr	39.2 (0.9)	4.3866 (0.3)	9.6 (0.4)	4.03 (0.5)	1.8 (0.5)	59.12 (2.43)
	<i>t</i> -test	**	-	**	-	-	**
		1.77478 × 10 ⁻⁹	0.311	3.35749×10^{-5}	0.54	0.06	1.62528×10^{-8}
		<0.000		<0.000			<0.000
35%	1 hr	34.49 (0.87)	7.34 (1.1)	11.57 (0.4)	2.38 (0.5)	2.1 (0.4)	57.95 (1.3)
	2 hr	43.57 (0.89)	12.43 (0.73)	19.87 (0.68)	5.17 (1.0)	3.57 (0.87)	84.62 (3.322)
	<i>t</i> -test	**	**	**		**	**
	**	1.91786 × 10 ⁻⁷	6.117×10^{-5}	1.1697×10^{-7}	0.001	0.016	8.573×10^{-6}
		<0.000	<0.000	<0.000			<0.000
38%	1 hr	38.81 (1.4)	7.78 (0.44)	12,68(1.89)	2.27 (0.5)	2.19 (0.5)	63.67 (3.4)
	2 hr	69.04 (2.45)	17.14 (2.3)	23.9 (2.4)	3.9 (0.78)	3.0 (0.5)	116.98 (7.6)
	<i>t</i> -test	**	**	**	**	*	**
		1.39447 × 10 ⁻⁷	0.0003332	6.165×10^{-5}	0.005	0.05	1.417×10^{-5}
		<0.000		<0.000			<0.000

Table 3 Mean (standard deviation) and t-test results of ion leach data (µg/ml) at the two studied periods for the three HP concentrations

**Highly significant results.

Results

Corrosion tests

Analysis of ion release

The effect of immersion time on the concentration of eluted ions was analyzed in the three studied HP concentration groups. The results (Table 3) illustrate that the concentration of ions increased significantly when time increased for all ions at 35% and 38% HP, whereas at 30% HP, K⁺, Al³⁺, and Si⁴⁺increased but not significantly. In all groups, Na⁺ ions were the main leached ions, followed by Ca⁺⁺ ions, while Si⁴⁺ and Al³⁺ were feebly eluted.

One-way ANOVA was performed to verify the existence of inter-group discrepancies or statistical differences between groups of 1-hour immersion, at the three HP concentrations, and the 2-hour immersion groups (Tables 4 and 5). The results show a significant increase in all ions released in the three studied HP concentrations, both in 1- and 2-hour immersion, except Al^{3+} release, which seems to be a time-dependent, not a concentration-dependent ion.

The influence of both HP and acetic acid on the amount of leached ions were evaluated through specimen immersion in 30%, 35%, and 38% HP for 1 hour, then immersing the specimens of each group individually in 4% acetic acid. The concentrations of the leached ions of the control group and

 Table 4
 One-way ANOVA results between groups of the three studied concentrations of HP at 1-hour immersion

Source of variation	Elements	Sum of squares	Mean square	F-ratio	Probability
HP concen- tration	Na	1952.9906	967.1365	620.035	<0.05
	К	57.8808		26.3946	<0.05
	Ca	92.5065		29.2895	<0.05
	Al	15.2878		3.4298	>0.05
	Si	3.3671		5.5149	<0.05
	Total	3494.9475		280.327	<0.05

subgroups 2c, 3c, and 4c were statistically analyzed (Table 6). All the elements released increased significantly, except for Al^{3+} at the three HP concentrations (2, 3, and 4c groups) and K^+ at 30% HP concentration (2c group), which showed a non-significant increase. These results suggest that most of the effect was due to immersion in HP, and not to the effect of acetic acid.

To confirm the previous results, ANOVA was performed (Table 7). The results showed that acetic acid influence is not a significant factor in increasing the released ions. The combined effect of both HP and acetic acid solutions was a significant factor, mainly due to previous immersion in HP.

Corrosion rate and weight loss determination

Corrosion rate is a more accurate parameter to monitor the corrosion behavior of any material. Figures 3 to 6 show that weight loss is directly proportional to the concentration of HP and time of immersion. When the corrosion rate was calculated, it indicated that corrosion reaction activities decreased with time. This is an expected behavior, especially in materials with well-known low corrodibility, such as ceramics. Corrosion rate is determined per unit time, and hence decreases with time.

Surface roughness

Surface roughness, Ra, was measured with respect to both time of immersion (1 and 2 hours) and change in concentration of HP. The results of the two-way ANOVA (Table 8) show a significant dependence and high effect of both factors on the values of surface roughness of the involved groups.

Surface roughness values were also measured with respect to acetic acid immersion (control) and the cumulative effects of both acetic acid and HP at the three concentrations. The results (Table 9) show a significant difference in Ra values between groups, indicating the deleterious effects of both solutions on the surface texture of Vitadur Alpha; however, the effect of HP concentration on surface roughness was greater than for acetic acid. Figure 7 shows the mean values of Ra in all studied groups. Surface roughness is directly proportional to the increase in HP concentration.

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Table 5 One-way ANOVA results between groups of the three studied concentrations of HP (2-hour immersion)

Source of variation HP concentration	Elements	Sum of squares	Mean square	<i>F</i> -ratio	Probability
	Na	2620.2781	1294.736	504.346	< 0.05
	К	435.4635	208.1984	131.034	< 0.05
	Са	568.0560	270.4555	119.559	< 0.05
	Al	12.3144	2.3984	3.8283	>0.05
	Si	12.5242	3.9897	10.5344	< 0.05
	Total	8699.3542	4204.618	173.913	< 0.05

Table 6 Statistical analysis of the combined effect of HP and acetic acid on leached ions concentration

lons groups	Na	к	Ca	Al	Si	Total
1						
Mean(SD) 2c	10.9(0.09)	3.09(0.7)	6.63(0.14)	2.36(0.5)	1.34(0.05)	24.34(1.3)
Mean(SD)	12.54(0.32)	3.94(0.17)	7.5(0.5)	3.09(0.9)	2.85(0.1)	29.85(1.7)
<i>t</i> -test	**	non	**	non	*	**
3c	<0.0001	0.511	<0.0001	0.19	*0.006	<0.0001
Mean (SD)	14.05(1.2)	5.3(1.4)	8.18(1.06)	2.98(1.0)	2.68(0.33)	33.16(4.7)
<i>t</i> -test	**	**	**	non	**	**
	<0.005	0.022	0.031	0.19	0.0001	0.012
4c						
Mean (SD) <i>t</i> -test	14.8(0.32) **	6.7(0.7) **	9.55(0.6) *	3.0(0.8) non	2.61(0.3) **	36.9(1.0) **
	<0.0001	< 0.0001	0.002	0.23	0.001	< 0.0001

*Significant results,

**highly significant results.

Table 7 Two-way without-replication ANOVA results on the effect of acetic acid and the combined effect of acetic acid and HP solutions on ion release concentration

Source of variation	SS	df	MS	F	p value	F crit
Acetic acid	1.1344	4	0.2836	0.5351	0.0712	3.2592
HP concentration + acetic acid	45.3157	4	15.1052	28.5009	0.000	3.4903
Error	6.3599	12	0.5299			
Total	52.8099					

SEM results

Photographs of the specimens representing the different groups investigated were compared to the photograph of an untreated blank Vitadur Alpha specimen (Fig 8A). After immersion in acetic acid for 18 hours, the surface shows very minor changes (Fig 8B). The surface began to respond to the increased concentration of HP by obvious manifestations such as increased depressions and irregularities (Figs 8C–E). These changes became more apparent in specimens immersed in higher concentrations and for longer durations (Figs 8F and G). Figure 8H shows the combined effect of 35% HP and acetic acid solutions. The pores are wider and look deeper due to the influence of both corrosive solutions. Magnification was increased up to $5000 \times$ to trace the pores formed in the specimens treated with both HP and acetic acid solutions (Fig 9). The irregular edges of the pores are also clearly shown.

Discussion

Corrosion studies

The chemical stability of a material represents the range of activities or gaseous partial pressure over which the material will



Figure 3 Percentage weight loss (μ g/cm²) mean, and SD, for subgroups immersed in HP.

Figure 4 Percentage weight loss (μ g/cm²) mean, and SD, in control group (0% HP + acetic acid) and other subgroups with combined effects of HP and acetic acid.

Figure 5 Corrosion rate (μ gm/cm²/hr), mean, and SD, recorded in subgroups of HP immersion.

Figure 6 Corrosion rate (μ gm/cm²/hr), mean, and SD, recorded in subgroups of combined effects of HP and acetic acid (HAC).

different studied groups with combined effects of HP and HAC

remain stable. From the practical point of view, it is advisable to test the durability of materials, especially those currently used in the aggressive oral environment. Vitadur Alpha is used as a veneering ceramic for alumina and zirconia cores in all-ceramic fixed partial dentures (FPDs) due to its excellent physical and mechanical properties.

The oral environment is subjected to variable temperatures, pH values, and cyclic stresses. The teeth and involved restora-

tions may be affected either positively or negatively by these conditions. HP is the principal ingredient in cosmetic, non-invasive bleaching procedures, considered now as the single most common esthetic treatment for handling dark or stained dentition. The bleaching treatment can be performed using an at-home technique, which employs a lower HP or carbamide peroxide concentration, or using the in-office technique, which requires highly concentrated HP, generally 35% or more,

Table 8 Relation between surface roughness, Ra, with respect to time and change in HP% using two-way ANOVA

Source of variation	SS	Df	MS	F	<i>p</i> -value	<i>F</i> crit
Time	555.031704	6	79.29024343	33.36206699	<0.001	2.285235174
Change in HP concentrations	77 <i>.</i> 967133	6	15.5934266	6.561071329	0.000210773	2.485143218
Error	83.183051	35	2.3766586			
Total	716.181888	47				

to achieve optimal and more pronounced effects in a short time. $^{\rm 39}$

In this study, the effect of high HP concentrations (30%, 35%, 38% v/v) on the chemical durability of Vitadur Alpha was studied after 1 and 2 hours of immersion. The 1-hour period was chosen to simulate the cumulative in-office clinical bleaching sessions in cases of moderate staining (three clinical visits, each 20 minutes) while the 2-hour period simulated the extreme in treating darkly stained teeth. Chemical durability was also studied, according to ISO standard 6872, equivalent to 22 years use in the oral cavity. The study aimed to test whether the specimens treated first with HP would corrode to the same extent as those not treated with HP, when immersed in acetic acid for 18 hours at 80°C. In the oral cavity, the effects of both solutions are inseparable if the patient received a bleaching protocol.

Although the study did not include any electrochemical tests, any corrosion process must include ionization and oxidationreduction chemical reactions. In the corrosion process, the initial surface reaction is mainly an acid-base reaction in which leaching ions are replaced by H⁺ ions, and the result will be an alkali-ion depleted layer overlying a silica-rich layer. The results clearly showed that HP is a strong corrosive solution, stronger than acetic acid, although the corrosive ion in both agents is H⁺. The dissociation of acetic acid is a reversible reaction, and equilibrium may be attained at which the concentration of the reactant and the products arrive to constant value. On the other hand, the dissociation of HP is a one-direction reaction, which goes to completion until it breaks down to water at the end. First, HP dissociates the free radicals, which oxidize the organic dark stain of the teeth to a much lighter one.

$$2H - O - O - H \rightarrow 2H^{+} + 2O - O - H^{-}$$
(2)

$$2O - O - H^{-} \rightarrow H2 + O2 \tag{3}$$

$$H_2O_2 \rightarrow H_2O + O$$
 (weaker free radical) (4)

 \downarrow

$$H^{\bullet} + HO^{\bullet}$$
 (stronger free radical). (5)

 H^{\bullet} and OH^{\bullet} are strong oxidizing agents and hence, strong corroding agents. The most common commercial bleaching processes used are peroxide, chlorine, and chloride, in that order.⁴¹ Hydrogen ions are small and highly reactive and can penetrate the surface layer of the ceramic, causing pores, and leading to formation of galvanic cells at the grain boundaries. Moreover, each of the multiphase microstructures of the ceramic responds differently, according to the nature of the bonds and the elements of the phase (i.e., the core phase layer is more stable than the superficial phase layer). Any two adjacent different salts in an electrolytic medium will form a galvanic cell. The more soluble ion of them will be leached first.

In this study, the results showed that the ion leach quantity increased with time and with the increase in concentration of HP; however, the order of major leached ions was not affected. $Na^+ > Ca^{2+} > K^+ > Si^{4+} > Al^{3+}$ ions, in that order, were detected at all studied concentrations. Even in acetic acid at 80°C, the same order was preserved. This means that even at the most extreme conditions of the proposed study, the silicate-



Figure 7 Surface roughness (μ m), mean, and SD, in all studied groups.

Table 9 AN	NOVA test	results of surface	roughness and th	e affecting s	solutions in all	tested groups
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Source of variation	SS	df	MS	F	<i>p</i> -value	F crit
HP concentration	487.2413194	7	69.60590277	12.28131356	<0.00001	2.359259855
Acetic acid + HP	152.5874275	4	38.14685688	6.730657772	0.0062989	2.714075804
Error	158.6935525	28	5.667626875			
Total	798.5222994	39				

rich layer was not destroyed by the corrosive solutions, offering some protection to the bulk network. Even for the specimens immersed in both HP then in acetic acid, the maximum cumulative amount of Si⁴⁺ leached was $3.1 \pm 0.5 \,\mu$ g/ml after 2 hours immersion in 38% HP, and $2.61 \pm 0.06 \,\mu$ g/ml after immersion in 38% HP for 1 hour, then 4% acetic acid for 18 hours at 80°C.

The one-way chemical decomposition of HP furnishes the solution with a higher concentration of H^+ and thus a greater extent of penetration, pitting, and then pore size increase,

giving the chance to increase the exposed surface area of the low-resistance grain boundaries phase, leading eventually to corrosion progression. This was shown clearly from the results of the solution analysis (Tables 3 and 6). When the concentration of HP increases, the amount of leached ions also increases. The results also showed that Na⁺ was the most-leached ion despite its relatively low percent of constituting elements (4% to 5%) of the ceramic. Si⁴⁺ was the least-leached ion even though it constituted the main bulk of the ceramic. This means



Figure 8 SEM photographs of different subgroups; (A) blank specimen, (B) control specimen, (C) 30% HP concentration, (D and E) 35% HP concentration, 1, 2 hours, respectively, (F and G) 38% HP concentration, 1, 2 hours, respectively, (H) specimen affected with 35% HP + acetic acid. Arrows indicate pore distribution and size. The irregularities and depressions of the surface are also recorded.



Figure 9 Magnification of the pores 2500× (left), 5000× (right) indicates the irregular boundaries and the rough surface.

that ion-elution is not proportional to the percent of the element constituents, but depends on the selective solubility of the elements and its atomic weight. Eventually Na₂O is much smaller and more soluble than SiO₄. The previous result was reported by Marko et al⁴² in a study measuring ion elution from several types of dental ceramics.

The results of ion elution analysis showed that when time of immersion increased, the amount of leached ions increased, for all ions at 38% HP. In the case of 35% concentration, Al^{3+} concentration did not increase significantly after 2 hours immersion in HP. While for 30% HP, K⁺, Al³⁺, and Si⁴⁺ concentrations did not increase significantly. This could lead to the assumption that even after 2 hours immersion in 30% HP. the corrosion of the ceramic is of low reactivity, and this HP concentration may not cause serious deterioration of the ceramic. Al³⁺ ions are considered as a reinforcing element and are very stable. They can immobilize an alkali ion to reduce ion exchange.43 In the case of specimens immersed in both HP and acetic acid, Al³⁺ concentration did not increase significantly. ANOVA results showed that the effect of acetic acid on the leaching ion concentration was not significant (p > 0.05) but when both HP and acetic acid affected the specimens, the results were highly significant in respect to concentration of leached ions.

The literature is devoid of studies similar to this one. Consequently, a comparison with similar results is not given; however studies on cast alloys and amalgam showed, as in this study, that ion release increases with increased HP concentration. Al-Salehi⁴⁴ reported an increase in mercury ion release from amalgam with bleaching. The increase of Ni and Pd ion release from two dental alloys was reported with increasing HP concentration from 0 to 30% (3000% increase in Ni, 1400% increase in Pd).⁴⁵

The weight loss of Vitadur Alpha increased from nearly 105 μ g/cm² to 215 μ g/cm² when the concentration of HP increased from 30% to 38%. These values are considered less than the limits of ISO standard No. 6872, which accepts loss values below 2000 μ g/cm². Corrosion rate increased with the increase in HP concentration, but decreased with time. At zero time, the rate of any chemical reaction is optimum, as the concentration of the reactants are maximum. With time, breakdown and dissociation of reactants occurs (HP breaks down to water) so the corrosion rate increased.

with time, it might lead to complete dissolution and breakdown of the ceramic, a situation that is not possible due to the low corrodibility of the ceramics as compared to metals. The corrosion rate is inversely proportional with time (eq. 2).

Surface topography

The surface roughness increased significantly with time and HP concentration. Surface roughness of ceramics increases the surface area exposed to corrosive solutions and increases the adhesion of bacterial plaque on the surface of the restoration. Ceramic texture is affected by the slightest change in the surface. Any minor release of surface ions would increase the roughness due to pore formation. The porosity of a ceramic can affect the overall corrosion if the attacking corrosive solution can penetrate through the porosity to the bulk core material.²⁹ The largest recorded Ra values were in the groups of combined effects of HP and acetic acid, due to continuous release of ions and pitting of the surface (the higher corrosion rates). SEM clearly illustrates the formation of large pores in subgroups with higher HP concentrations and in specimens affected by both solutions. Many investigations on the effect of bleaching on surface roughness for tooth-colored restorative materials indicated an increase in Ra values.⁴⁶ The surface roughness of composite, glass ionomer, and feldspathic porcelain increased after bleaching with carbamide peroxide for 8 hours a day for 30 days. The results also showed depleted surface content of SiO₂, indicating the corrosion of feldspathic porcelain and micro-filled composite.23

From the previous discussion, we may assume that the integrity of the material could be affected if the concentration of HP exceeds 35% and time of treatment exceeds 1 hour. The specimens treated with 38% HP then with acetic acid may have a diminished lifetime if the other influencing factors of the oral environment are considered.

Limitations of this study are due to the in vitro nature of the investigations, which lacked the washing effects of saliva. Repeated mechanical loading enhances stress corrosion, and fluctuation of temperature present in the oral environments may also accelerate corrosion. Further investigations are needed to identify all the side effects of bleaching, especially on different types of ceramics.

Conclusions

Within the limitation of this study, the following conclusions were drawn:

- The effect of 30%, 35%, and 38% (v/v) HP on ion release concentration exceeds that of acetic acid in the studied ceramic.
- (2) Specimens treated first with HP surpassed control group corrosion. Bleaching could impair the integrity of Vitadur Alpha.
- (3) Surface roughness increased significantly with time and concentration of HP. Time and concentration of HP should be kept concisely as possible if in-office bleaching is required.
- (4) The combined effect of HP and acetic acid increases weight loss and corrosion rate more than each solution separately. The corrosion rate decreased with time in all studied HP concentrations.

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