Carbamide and Hydrogen Peroxide Bleaching Effect on Modern CAD/CAM Dental Ceramics: An Investigation on Ion Release and Surface Characteristics

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ABSTRACT

Objectives: This study aimed to investigate the impact of bleaching agents based on carbamide or hydrogen peroxide on dental ceramics in vitro, utilizing scanning electron microscopy (SEM) and elemental analysis via inductively coupled plasma optical emission spectroscopy (ICP-OES). Methods: CAD/CAM ceramics (IPS e.max®CAD, IPS Empress®CAD, Vitablocs® Mark II, Celtra Duo, and inCoris TZI) were treated with bleaching agents using either 10%, 20%, 30% carbamide peroxide or with 35%, and 40% hydrogen peroxide. Results: Surface elemental release was not significantly affected by the type or concentration of bleaching agent ($p>0.05$). Ion release in feldspathic ceramics was significantly higher than in other ceramic materials ($p<0.0001$). Microstructural surface changes were observed in all materials except for lithium disilicate and zirconia-reinforced lithium silicate ceramics. Conclusions: All bleaching agents tested in this study showed a similar impact within each material type tested regarding total mass loss, elemental composition, or surface structure. Clinical relevance: Lithium disilicate and zirconia-reinforced lithium silicate ceramics were the most resistant to bleaching agents. In contrast, feldspathic ceramic showed the highest ion release and surface deterioration when exposed to all bleaching agents tested.

INTRODUCTION

Bleaching, commonly referred to as tooth whitening, removes discoloration from teeth caused by internal and external factors. While internal factors are less common and are associated with non-vital teeth,[1] external factors are more common and are caused by pigment deposition on the tooth surface from tobacco, tea, coffee, red wine, carrots and oranges.[2,3] Techniques for teeth whitening have been documented in professional literature since the 19th century,[4] involving the application of bleaching agents to the external tooth surface (external bleaching) or into a pulp chamber (internal bleaching) to enhance aesthetics by oxidizing chromogens within the dentin.

Hydrogen peroxide (HP) or its precursor, carbamide peroxide (CP), is the main component in most modern teeth-whitening solutions. As it degrades, HP acts as a potent oxidizing agent, producing anions, reactive compounds, and free radicals.[5] This process can be enhanced by activators such as heat or light.[6] In contrast, CP breaks down over time and produce HP and urea, resulting in a prolonged bleaching effect compared to HP alone.[6] In addition to altering tooth color, HP also affects the surface hardness, microhardness, roughness, and surface topography of the tooth surface.[7]

External bleaching techniques are also available as over-the-counter products that patients use on their own, or for in-office bleaching treatments performed by dentists, and at-home bleaching treatments provided by dentists.[8,9] In-office bleaching may involve high concentrations of HP or CP with heat and photoactivation. In contrast, dentist-supervised bleaching utilizes a tray loaded with CP placed in the patient's mouth during a dental visit.[8,9]

While the effect of HP or CP on indirect restorative materials has undergone intensive testing,[10-13] only a limited number of studies have assessed their impact on computer-aided design/computer-aided manufacturing (CAD/CAM) restorations. In such materials, ion release from bleach-exposed areas varies depending on the material characteristics.[14,15] Given the increasing implementation of digital workflows for manufacturing all-ceramic restorations in dentistry, this study seeks to assess and illustrate the impact of bleaching agents on dental ceramics employed in CAD/CAM restorations. The null hypotheses tested were that bleaching agents of different concentrations would not affect the a) mass, elemental loss, surface structure and b) ion release from glassy matrix and polycrystalline dental CAD/CAM ceramics.

MATERIALS AND METHOD

FABRICATION OF SPECIMENS

The study specimens were typical dental ceramics employed in clinical settings employing digital workflow.[16] Ceramics blocks (10×12×15 mm^3), were used (IPS e.max®CAD, IPS Empress®CAD, Vitablocs® Mark II, Celtra Duo, and inCoris TZI) to generate specimen blocks (9×9×1 mm^3) (*Table 1*).

Table 1. Types, brands, manufacturers, and chemical compositions of CAD/CAM materials used in this study.

Type	Brand	Manufacturer	Composition (w%) according to manufacturers
Lithium disilicate (glassy matrix synthetic)	IPS e.max® CAD	Ivoclar Vivadent, Schaan Liechtenstein	$Li_2Si_2O_5$ SiO_2 57-80% Li_2O 11-19% K_2O 0-13% P_2O_5 0-11% ZrO_2, ZnO, colouring oxides 0-8% Al_2O_3, MgO 0-5%
Leucite-reinforced (glassy matrix synthetic)	IPS Empress® CAD	Ivoclar Vivadent	$KAlSi_2O_6$ SiO_2 60-65% Al_2O_3 16-20% K_2O 10-14 % Na_2O 3.5-6.5 % Other oxides 0.5-7%
Feldspathic ceramic (glassy matrix)	Vitablocs® Mark II	Vita Zahnfabric, Bad Säckingen, Germany	SiO_2 55-70%, Al_2O_3 20-24%, Na_2O 6-10%, K_2O 4-8% CaO, TiO < 1%
Lithium silicate zirconia-reinforced (glassy matrix synthetic)	Celtra Duo	Dentsply Sirona, Charlotte, North Carolina, USA	Lithium oxide, silicon dioxide, ZrO_2 10%
ZrO_2 tetragonal polycrystalline	inCoris TZI	Dentsply Sirona	$ZrO_2+HfO_2+Y_2O_3 \geq 99.9\%$ Y_2O_3 5.4% $HfO_2 \leq 5\%$ $Al_2O_3 \leq 0.005\%$ $Fe_2O_3 \leq 0.02\%$ Other oxides ≤ 0.2%

APPLICATION OF BLEACHING AGENTS

The specimens (N=125) were cleaned using ethanol and demineralized water (n=5 in each cleaning agent). They were then dried at 37°C in a lab oven (4 h). Prior to applying the bleaching agents, the specimens were weighed. Details on the CAD/CAM materials used in the study are provided in Table 1 and the bleaching agents with variable concentrations in Table 2. The bleaching agents were applied to all specimen surfaces following each manufacturer's instructions *(Table 3)*. Bleaching products containing 10% and 20% CP were used for 4 hours, whereas those with 30% CP, 35% HP, and 40% HP were applied for 20 minutes.

EVALUATION FOR ELEMENTAL, MASS LOSS AND SURFACE CHARACTERISTICS

The specimens were inserted in 3 mL of 0.1 mol/L HCl in polyethylene test tubes (test tubes were pre-treated with 2 mol/L HNO_3) with a volume of 10 mL according to a methodology described elsewhere.[17] Concurrently, five untreated pieces were designated as blank specimens for each type of dental ceramic, while an additional set of five untreated pieces served as controls. The blank specimens were immersed in a tube containing 3 mL of deionized water, and the control specimens were placed in 3 mL of 0.1 mol/L HCl (positive control). All tubes were then positioned in a water bath and heated to 37°C for 168 h. Following this incubation period, the specimens were retrieved, and the liquid extracts were transferred into volumetric flasks. The extracts were diluted with deionized water, reaching a final volume of 10 mL. The elemental analysis of the extracts was performed using inductively coupled plasma optical emission spectroscopy (ICP-OES) (Integra XL 2, GBC, Australia). With inductively coupled plasma optical emission spectroscopy (each dental ceramic and each bleaching agent combinations; N= 125), the analyses were performed five times for each specimen. For each specimen, the mean of the five repetitions represented the definite extract analysis results. After bleaching, the specimens were washed with distilled water, dried, and weighed again.

All reagents used were of analytical-grade quality. Deionized water was purified (SG Ultra Clear system SG Water, Nashua, USA). HCL of 35% (v/v) and 65% (v/v) HNO3 (LachNer, Neratovice, Czech Republic) were distilled in a sub-boiling distillation equipment (BSB 939 IR, Berghof, Eningen, Germany). Calibration standards were prepared from single-element stock standards (Al, As, Au, Ca, Cd, Co, Cr, Cu, Fe, La, Li, Mn, Mo, Na, Ni, Pb, Si, Ti, Y, Zn) 1±0,002 $g \cdot L^{-1}$ (SCP Science, Baie D'Urfé Canada). Weight loss was determined by measuring the difference in weight before and after leaching using a precision scale (Mettler-Toledo AG, Laboratory & Technologies, CH-8606 Greifensee, Switzerland).

Table 2. Bleaching product brands, manufacturers and types used in the study.

Bleaching product	Manufacturer	Bleaching agent
Opalescence PF™ 10%	Ultradent, South Jordan, Utah, USA	Carbamide peroxide (10%)
Opalescence PF™ 20%	Ultradent	Carbamide peroxide (20%)
VivaStyle®	Ivoclar Vivadent, Schaan Liechtenstein	Carbamide peroxide (30%)
Perfect Bleach Office	VOCO GmbH, Cuxhavem Germany	Hydrogen peroxide (35%)
Opalescence® Boost 40%	Ultradent.	Hydrogen peroxide (40%)

Table 3. Manufacturers` instructions for the use of bleaching products.

Bleaching agent	Quantity and placement	Duration	Use	Bleaching tray
Carbamide peroxide (10%)	continuous bead of teeth whitening gel approximately 1/2 of the syringe into tray	2-4 h	At-home	Yes
Carbamide peroxide (20%)	continuous bead of teeth whitening gel approximately 1/2 of the syringe into tray	2-4 h	At-home	Yes
Carbamide peroxide (30%)	small amount toward the facial side of each tooth space in the whitening tray	20-30 min	In-office	Yes
Hydrogen peroxide (35%)	1-2 mm layer onto the labial surface of the tooth	15 min	In-office	No
Hydrogen peroxide (40%)	0.5-1.0 mm layer onto the labial surface of the tooth	20 min	In-office	No

EVALUATION OF SURFACE CHARACTERISTICS

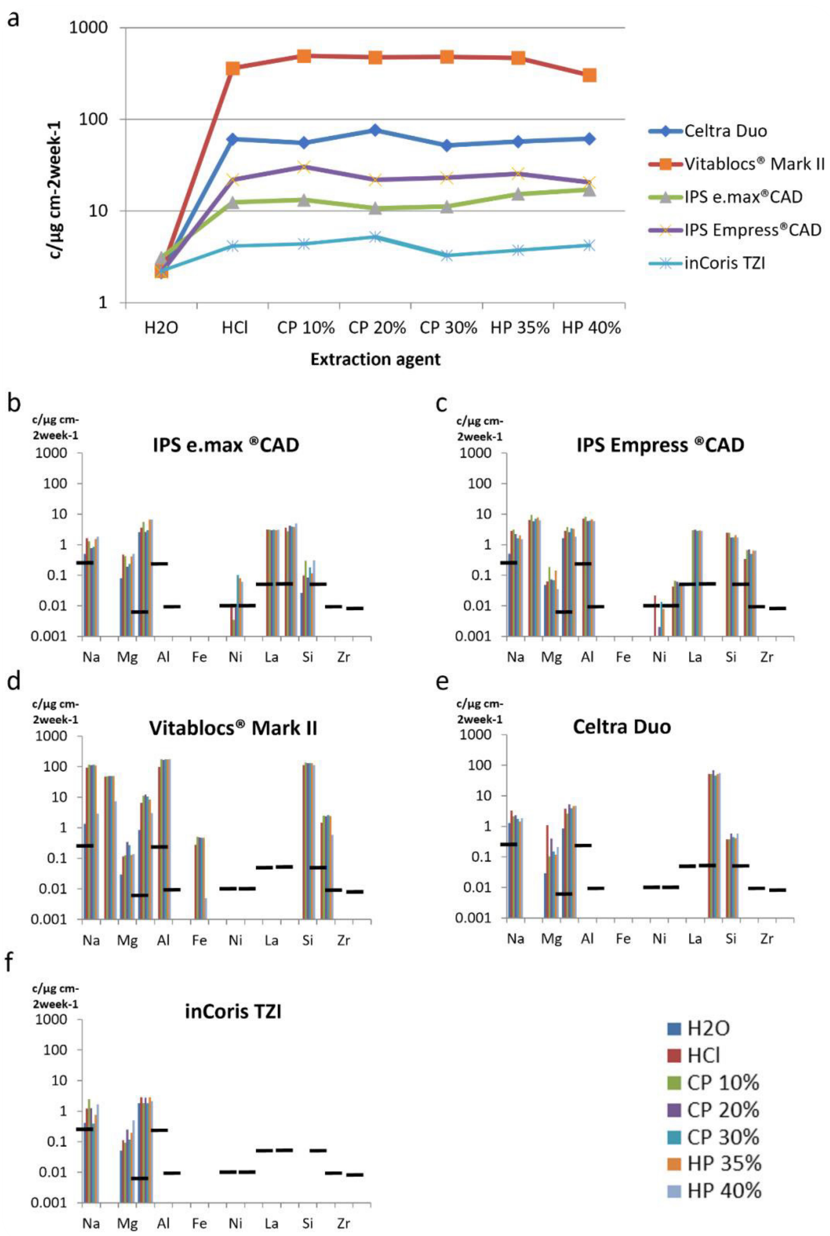
The surface of the dental ceramic specimens was examined using scanning electron microscopy (SEM) (Phenom Pro, PHENOM, Eindhoven, Netherlands) at different magnifications. The surface changes were evaluated independently by three researchers (L.V., J.S., and M.K.) and classified as follows: Score 0: no changes (surface remains intact, no visible alterations or structural damage), Score 1: minimal changes (slight surface alterations, such as minor etching, limited impact on the structural integrity), Score 2: moderate changes (noticeable surface modifications, some structural changes without severe damage), Score 3: severe structural changes (extensive and significant alterations in the ceramic structure, loss of integrity, visible cracks, or other severe structural damage).

STATISTICAL ANALYSIS

The data were statistically analysed (GraphPad Prism version 8.0 for Windows, GraphPad Software, San Diego, CA, USA). Statistical analysis was performed using the Shapiro-Wilk test and two-way ANOVA with Tukey's multiple comparisons tests ($\alpha = 0.05$).

RESULTS

Figure 1a illustrates the loss in mass ($\mu g \cdot cm^{-2} \cdot week^{-1}$) for all tested dental ceramics, bleaching agents, and controls. Figure 1b-f show the loss of elements ($\mu g \cdot cm^{-2} \cdot week^{-1}$) for all tested dental ceramics, bleaching agents, and controls. Black lines on the graphs indicate detection limits.



Figures 1a-f: a) The loss in mass (µg·cm-2·week-1) for all tested CAD/CAM ceramics in all tested bleaching agents using ICP-OES, b) Lithium disilicate, c) Leucite-reinforced, d) Feldspathic, e) Lithium silicate zirconia-reinforced, f) ZrO_2 tetragonal polycrystalline. HP: Hydrogen peroxide or its precursor; CP: Carbamide peroxide.

Significantly higher elemental release in feldspathic ceramic were observed than in other materials ($p<0.0001$). Feldspathic ceramic also exhibited the highest release of elements, contrasting with zirconia oxide polycrystalline ceramic, which showed the lowest *(Table 4)*, which was in line with the mass loss values ($\mu g \cdot cm^{2} week^{-1}$) across CAD/CAM materials subjected to different bleaching agents. Among the glassy synthetic dental ceramics (lithium disilicate, leucite-reinforced, and lithium silicate zirconia-reinforced ceramics), the highest release of elements were observed in zirconia-reinforced lithium silicate and the lowest in lithium disilicate.

As regards to mass loss patterns across dental ceramics and bleaching agents, feldspathic ceramic stood out due to its high ion release *(Figure 1)*.

The type and concentration of bleaching agents had no significant effect on elemental release from CAD/CAM blocks compared to the positive control group ($p>0.05$).

Leucite-reinforced dental ceramics exhibited the most variable surface changes, depending on the bleaching agent used. In contrast, no noticeable changes were observed for lithium silicate-based dental ceramics. Table 5 provides a detailed classification of surface changes from no changes to severe structural changes across the different ceramics and bleaching agents based on SEM observations. Figure 2 supplements this with SEM images of selected specimens, visually highlighting the contrast on the surface changes on the leucite-reinforced and lithium silicate-based ceramics.

DISCUSSION

This study examined the effects of different bleaching agents at different concentrations on mass, elemental loss and surface alterations in CAD/CAM ceramics made from glassy and polycrystalline dental ceramics. The results indicated that feldspathic dental ceramics released significantly more ions than polycrystalline ceramics, while no significant differences on surface elemental release, mass loss, or surface alterations of specimens across bleaching agents and control specimens within each material. Therefore, the first null hypothesis was partially rejected, while the second hypothesis was rejected.

The total elemental release was more significant in the lithium silicate zirconia-reinforced and leucite-reinforced dental ceramics compared to the lithium disilicate dental ceramics. In a study published by Queiroz *et al.*, an examination of leucite-reinforced material and dental ceramic with a resin matrix revealed that the lowest substance loss after in-office

Table 4. Total mass loss values (c/$\mu g\ cm^{-2} week^{-1}$) of CAD/CAM ceramics in control and bleached groups according to ICP-OES.

	Total Mass Loss						
	H_2O	HCl	CP 10%	CP 20%	CP 30%	HP 35%	HP 40%
Celtra Duo	2.14	60.97	55.54	76.19	51.96	57.31	61.45
Vitablocs® Mark II	2.22	361.43	492.08	474.80	480.01	468.53	303.82
IPS e.max® CAD	3.12	12.45	13.23	10.79	11.20	15.33	17.09
IPS Empress® CAD	2.15	22.03	30.39	21.86	23.05	25.54	20.54
inCoris TZI	2.23	4.17	4.39	4.22	2.29	3.75	4.24

Table 5. Scores given to surface changes of CAD/CAM ceramics (1000×, SEM).

	IPS e.max® CAD	IPS Empress® CAD	Vitablocs® Mark II	Celtra Duo	inCoris TZI
Carbamide peroxide (10%)	0	0	1	0	0
Carbamide peroxide (20%)	0	2	0	0	0
Carbamide peroxide (30%)	0	2	0	0	1
Hydrogen peroxide (35%)	0	1	1	0	1
Hydrogen peroxide (40%)	0	1	1	0	1

Score 0: no changes (surface remains intact, no visible alterations or structural damage), Score 1: minimal changes (slight surface alterations, such as minor etching, limited impact on the structural integrity), Score 2: moderate changes (noticeable surface modifications, some structural changes without severe damage), Score 3: severe structural changes (extensive and significant alterations in the ceramic structure, loss of integrity, visible cracks, or other severe structural damage).

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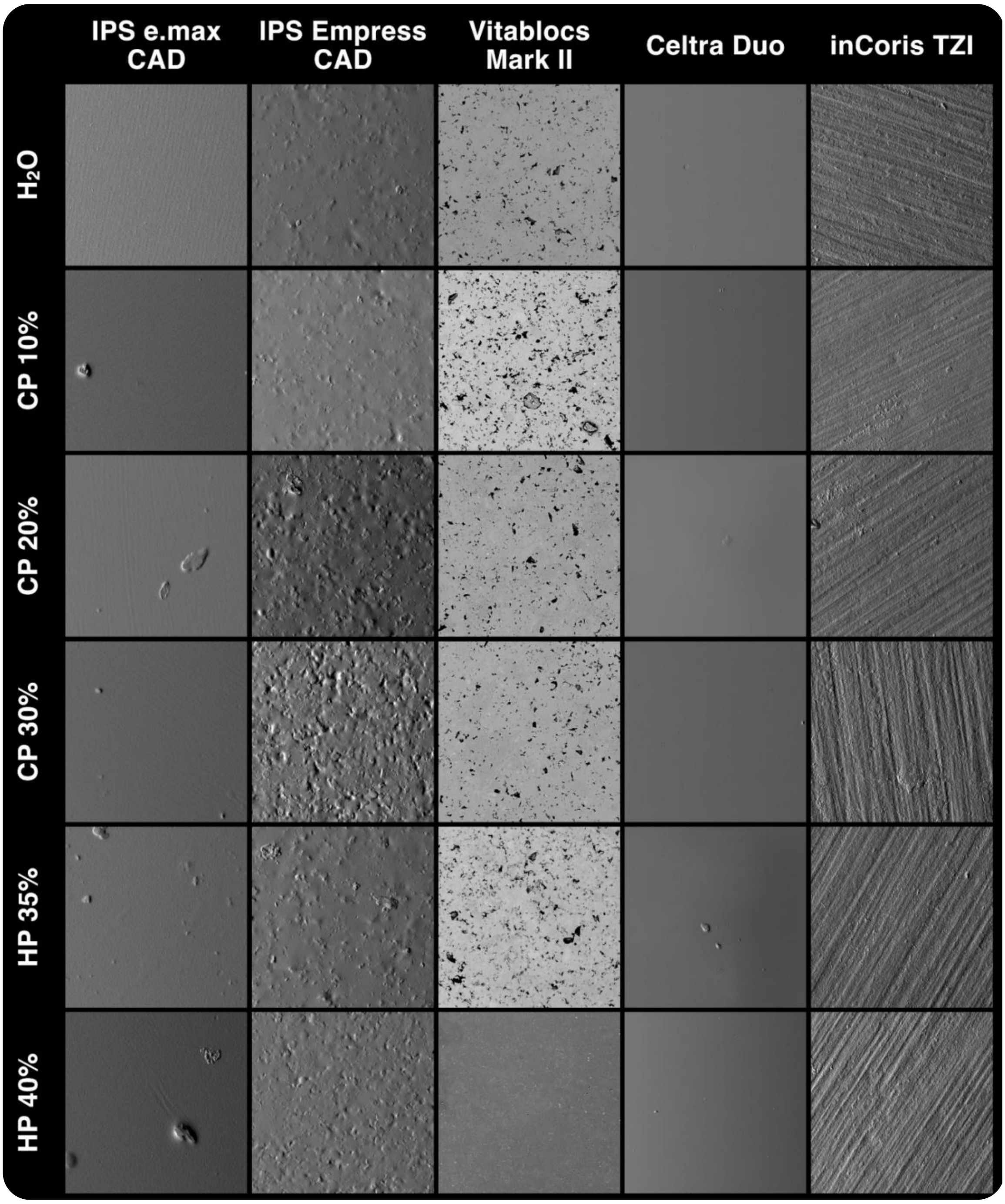

Figure 2: The surface changes for CAD/CAM ceramics tested after exposure to different bleaching agenta. HP: Hydrogen peroxide or its precursor; CP: Carbamide peroxide.

bleaching treatment with 40% HP in leucite-reinforced ceramic (IPS Empress® CAD).[18] Conversely, hybrid nanoceramics with a resin matrix (Lava Ultimate) were linked to high ion elution, indicating they should be avoided for in-office bleaching sessions with HP 40%. In our study, materials such as polycrystalline and lithium disilicate demonstrated lower weight loss after HP 40% treatment, which were not assessed in the above-mentioned study.

In contrast to our findings, Abu-Eittah *et al*. reported a direct relationship between HP concentration and immersion time.[19] Their study utilized higher concentrations of HP (30%, 35%, and 38%) combined with HCl treatment, which may have significantly increased ion leaching compared to the results in this study. Contrary to our study findings, Karaokutan *et al*. reported that a home bleaching agent increased ion elution in IPS e.max CAD, ZirCAD, and Vita In-Ceram YZ, indicating the need for modified custom tray protection during bleaching to prevent harmful effects on existing dental restorations.[20]

Characterizing ion release following HP or CP application is challenging, as the observed ion spectrum sometimes diverges from the manufacturer's composition, even with acetic acid.[17] Jakovac *et al*. found that HCl treatment of glassy dental ceramics resulted in a high elution of sodium ions, with Al^{3+} being the most prominent ion released, contradicting with the manufacturer's composition claims.[17] On the other hand, the findings of Serin-Kalay *et al*. observed ion release patterns that aligned closely with the manufacturers' composition descriptions for materials such as Vita Enamic, Vita Suprinity, and Lava Ultimate.[15] In this study selection of materials included only ceramic based CAD/CAM ceramics. Differences in material composition may explain the variation in ion release consistency observed between studies.[15]

The usage of HCl as a strong reagent follows the declared method in ISO Standard 6872.[21] Additionally, the pH value of acetic acid (pH 2.4) was similar to the pH values of some refreshing drinks and juices.[22] Yet, surface changes in microstructure were evident in the study, particularly in feldspathic ceramic, leucite-reinforced ceramic, and, surprisingly, polycrystalline ceramic after applying bleaching agents. Moderate changes (CP 20% and 30%) were noted for leucite-reinforced dental ceramics, while for the other dental ceramics, minimal changes were observed, predominantly after 35% and 40% HP treatment. The results contrast the second hypothesis, as the microstructure changes were less prominent in glassy dental ceramics than in crystalline dental ceramics and therefore it is rejected.

One study reported that surface roughness became visible after 21 days of exposure to 10% and 35% CP on feldspathic dental ceramics.[10] According to other authors, changes in the dental ceramic surface may relate to exposure to CP home agents, leading to a reduction of silicon dioxide and potassium peroxide molecules yielding to decrease up to 4.82% and 1.89%, respectively, of the original content.[23] This aligns with our study, where minimal surface changes for feldspathic dental ceramics were demonstrated using SEM for 10% CP, 35%, and 40% HP. Another study employing SEM also reported on the impact of 10% and 16% CP on surface roughness and degradation in feldspathic dental ceramics (Vita VM7 and VM13).[24] In another study, the authors reported on the effect of 35% CP on leucite and conventional glass dental ceramic, reducing surface microhardness.[25] It was also reported that in-office bleaching (35% CP) and at-home bleaching (15% CP) significantly increased the surface roughness of overglazed ceramic restorations, highlighting the importance of protecting the restoration during bleaching.[26] However, several studies have described contradicting results where no detrimental effect was found on surface roughness, microhardness, and flexural strength of dental ceramics.[27-29]

The bleaching effect on color and microhardness is mainly CAD/CAM material-dependent, with perceptible color changes in lithium silicate zirconia-reinforced dental ceramic and imperceptible effects in lithium disilicate dental ceramic.[30] Demir *et al*. found irregular microholes in hybrid dental ceramics treated with 16% CP, advising caution when using at-home bleaching agents. Thus, ceramic polishing may be necessary in cases of accidental bleaching,[14] and laser bleaching[31] for feldspathic dental ceramics. The findings of this study also indicated that feldspathic or leucite based ceramic surfaces shoud be protected during bleaching procedures.

Although the study did not look into particular bleaching safety precautions for CAD/CAM ceramics, the findings signified that ordinary bleaching agents do not significantly harm the ceramic materials studied when used as directed by the manufacturer. Clinicians can confidently employ bleaching procedures with CAD-CAM ceramics, providing that they should still be aware of slight surface changes in some materials, like ceramics reinforced with leucite or feldspathic ceramic.

This study did not include quantitative assessments of surface roughness changes following bleaching. While SEM images provided qualitative insights, future research should use quantitative methods, such as profilometry, to accurately measure surface roughness changes. Furthermore, a limitation of this study was that no glaze was applied to the dental ceramics, which may affect the resistance of the material to bleaching agents. Finally, the *in vitro* nature of the study means that the environmental conditions did not fully replicate the complexities of the oral environment, such as temperature fluctuations, presence of saliva, and mechanical forces from mastication. These factors may impact the findings and should be addressed in future clinical studies.

CONCLUSIONS

From this study, the following could be concluded:

1. Bleaching agents based on carbamide or hydrogen peroxide at various concentrations and durations did not affect the overall ion release within each CAD/CAM tested.
2. Feldspathic dental ceramic released significantly more ions than other CAD/CAM ceramics, irrespective of the bleaching agent used.
3. Microstructural surface changes were observed in all tested materials except for lithium disilicate and zirconia-reinforced lithium silicate ceramics.

DISCLOSURE

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