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Long-Term Effect of Nanosized Boric Acid Powder on Optical Properties of Polymer Infiltrated Ceramic CAD-CAM Material

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Abstract: The current study investigated the effect of boric acid (H_3BO_3) nanosized powder on the optical properties of a Computer-Aided Design and Computer-Aided Manufacturing (CAD-CAM) polymer infiltrated ceramic material. Specimens (n = 60), (15 × 8 × 1.5 mm^3) were fabricated from a polymer infiltrated ceramic network (PINC) (Vita Enamic, Vita Zahnfabrik, VITA-shade scale A2). Boric acid (B) nano powder was applied to Vita Enamic in half of the specimens (n = 30), while the other half was left untreated (NB) (n = 30). Aging for all specimens was performed for 5 h at 134 °C. Color coordinates (L*, a*, and b*) before and after aging were measured to calculate the color change (ΔE_{00}) and the translucency parameter (TP) within and between the B and NB groups. One-way ANOVA was used to analyze the effect of boric acid on all color parameters (α = 0.05). Only L* increased in B after aging ($p < 0.001$). L* and b* significantly changed after aging in NB ($p < 0.001$). Boric acid application affected the color change within the ceramic after aging ($p < 0.001$). The mean color change (ΔE_{00}) in B after aging was significantly smaller than the color change in NB after aging ($p < 0.001$). The color difference between B and NB increased after aging ($p < 0.001$). No significant effect of aging was found on TP of B ($p = 0.143$). The TP of NB significantly decreased after aging ($p < 0.001$). The use of boric acid provided color stability and translucency on aged tested material.

Keywords: boric acid; CAD-CAM; color stability; dental materials; nanosize powder; polymer infiltrated ceramic network; prosthodontics; Vita Enamic

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1. Introduction

Increased esthetic demands in fixed dental prostheses have become a primary goal of the dental sciences [1]. New material fabricated for restorations, such as resin nano ceramic and polymer infiltrated ceramic networks (PICN), which do not require several firings, have altered the CAD-CAM process. The blocks have several advantages, such as faster milling, high fracture resistance, and milling failure longanimity [2]. Additionally, restorations can be effortlessly polished and adjusted in chairside applications. The structural integrity can increase the clinical lifespan of the PINC restorations. However, the opacity still prevents the development of applications for anterior restorations [3]. Dental ceramics provide color and translucency similar to the natural tooth structure with acceptable esthetics [4,5]. However, dental ceramics may be fragile [1]. Boron compounds can help all-ceramic restorations achieve higher clinical success rates and minimize fracture problems. These chemicals are used to improve ceramics' mechanical characteristics [1]. Boron is a bioactive trace element [6–8] with metal and nonmetal characteristics that is commonly employed as a bactericide, fungicide, and antiseptic in the weak inorganic acid form of boric acid [9]. Different amounts of boric acid have been used in studies to test its antibacterial activity. In addition to the structure of ceramics, boron enhances dimension change behavior, offers resistance to thermal shocks, and improves electrical and mechanical characteristics, in

addition to lowering the temperature [1,10]. Most toxicity data on boron are on boric acid [11]. However, the nontoxic upper concentration of boric acid is 250 µM and amounts above 500 µM were reported to cause cytotoxicity [12]. In addition, boric acid has been reported to have antioxidant properties in specific doses (0–12.5 mM), which prevents cell damage [13].

Nanomaterials have attracted a growing interest in recent years due to their distinctive shapes and characteristics. Nanomaterials are made of nano particles, providing a large surface area and high surface energy [14–16]. Boric acid nanoparticles are naturally occurring compounds containing three elements: boron, oxygen, and hydrogen. Boric acid has various properties, including as an antiseptic, anti-fungal, fire retardant, nutritional supplement, and its use in fiberglass.

The color stability and translucency are essential factors in the long-term esthetic success of ceramic materials. The material's ability to maintain its color depends upon whether restorations mirror the natural color of the tooth [5,14,17]. However, investigation of the effect of the boric acid on optical properties is sparse. The present study aimed to investigate the effect of Boric acid nano powder on the color coordinates, color, and translucency change of PICN material after aging. The null hypothesis of the present study was that boric acid nano powder would not affect the color coordinates, color change, or the translucency of the tested PICN material after aging.

2. Materials and Methods

The sample size was calculated using G*Power V3.1.9.6. Levels of 95% confidence (1-α), 95% test power (1-β), f = 0.595 effect size, and a total of 48 specimens (16 in each group) were considered appropriate. Considering possible losses during fabrication and experiments, 30 specimens were included in each group [18].

Sixty disk-shaped specimens (Ø 12 mm × 1.2 mm) were sectioned and divided into two groups: boric acid nano powder-applied (B, n = 30) and no boric acid nano powder applied (NB, n = 30). In the NB group, specimens were prepared from CAD-CAM monolithic block (Vita Enamic, Vita Zahnfabrik, Bad Säckingen, Germany, Batch VITA-shade scale A2) in a cutting machine (Vari/cut VC-50, Leco Corp, Geleen, The Netherlands with a diamond-wafering blade (Isomet 15 LC, Buehler, Leinfelden-Echterdingen, Germany) under running water. For the B group, after the Vita Enimac blocks were mixed (ME 8100× multitooth Mixer), boric acid nano powder (Nanoshel, Salt Lake, USA) and ceramic resin powder were distributed at a 3% ethyl alcohol (Junub Ethanol, Shiraz, Iran) concentration. Ceramic resin powder was homogenized in a slurry in an ultrasonic bath for half an hour and swirled with a magnetic stirrer for four hours (Heidolph MR Hei-Standart, Nuremberg, Germany). The alcohol was evaporated, and the sample was taken. The slurry was passed through a 325 mesh sieve. A total of 7% moisture was added to the resulting mixture. A plexiglass mold (Farko, Tahran, Iran) was used to make the samples. The sample was pressed and the resulting tablet was dried. The firing cycle was set at half a degree per minute at 50, 75 and 110 °C for one h. The specimens were fired to the following temperatures: 550, 560, 1060, 1200, 1300, and 1450 °C, respectively, in a furnace (Programat P300, Ivoclar Vivadent AG, Schaan, Liechtenstein).

With the use of pre-wetted silicon carbide abrasive papers, the specimen surfaces were polished (Grids 600, 800, and 1200 English Abrasives Ltd., London, UK). [18] Each specimen's thickness was determined to be 0.5 mm using a digital caliper (Model Absolute Digimatic Caliper; Mitutoyo Corp, Kawasaki, Japan) [19].

The specimens were fired in a furnace according to the manufacturer's instructions (Programat P310; Ivoclar, Vivadent) at 710 °C for 12 min after the glaze powder and liquid (Initial Glaze; GC) were mixed and applied to the NB group specimen surface in a thin coating.

Before the aging process, the color was measured by using a spectroradiometer [20,21] (SpectraScan PR-704, Photo Research, Chatsworth, CA, USA) according to the CIEDE2000 color coordinates. The media between the specimen and the background was a saturated

sucrose solution with a rough refractive index of n = 1.5. The color of specimens were assessed using a D65 standard illumination at 0° and a 45° light source. [22,23] For each item, measurements were taken three times on two different backdrops at the same time of day. The CIELab values were recorded on both black (B) (L* = 2.3, a* = 0.5, b* = 2.1) and white (W) (L* = 94.6, a* = 0.2, b* = −0.8) backgrounds. On a neutral gray background, further color measurements of each item were taken and recorded, with L1, a1, and b1 standing in for the color coordinates prior to aging. The color difference between the measurements taken against the B and W backgrounds was the basis for the translucency parameter (TP) calculation [24,25].

In an autoclave, all specimens underwent aging (Trans Getting, Systec, Linden, Germany) for 5 h at 134 ± 10 °C, which is the standard aging protocol according to ISO 13356 [26–28]. Afterwards, all specimens underwent a 15 min ultrasonic cleaning in distilled water before being dried with absorbent papers [24]. Color coordinates after aging (L2, a2, and b2) were recorded after measuring the specimens on the same gray background. The color differences after aging were calculated using the newly developed CIEDE2000 (ΔE_{00}) color difference formula [29,30].

$$\Delta E_{00} = \sqrt{\left(\frac{\Delta L}{K_L S_L}\right)^2 + \left(\frac{\Delta C}{K_C S_C}\right)^2 + \left(\frac{\Delta H}{K_H S_H}\right)^2 + R_T\left(\frac{\Delta C}{K_C S_C}\right)\left(\frac{\Delta H}{K_H S_H}\right)} \quad (1)$$

The color differences of $\Delta E_{00} \leq 0.8$ and $\Delta E_{00} \geq 1.8$ (corresponding to CIEDE2000 50:50% PT and 50:50% AT, respectively) were used to interpret the result [31].

The distribution of data was analyzed by using a Kolmogorov-Smirnov test. The Mann-Whitney U tests were used to evaluate the ΔL, Δa, Δb, ΔE_{00}, and TP values of the B group, while paired sample t-tests were used to evaluate the ΔL, Δa, Δb, ΔE_{00} and TP values of each NB group. One-way ANOVA and Tukey's HSD tests were performed to compare the ΔE_{00} values of specimens after aging. The overall TP value groups depending on aging were analyzed by using independent sample t-tests. All analyses were performed by using software (SPSS v23; IBM Corp, Armonk, NY, USA) at a significance level of (α = 0.05).

3. Results

Among the color coordinates, only the L* coordinate increased in the boric acid nanosized powder applied group after aging ($p < 0.001$), while no significant differences were found for a* and b*. L* and b* significantly changed after aging in the NB group ($p < 0.001$) (Table 1).

Table 1. Mean and standard deviation (SD) values of L*, a*, b* before and after aging.

Groups	Color Dimensions	before Aging	after Aging	*p* Value
Group B	**L***	81.45 ± 1.07	81.79 ± 0.77	<0.001
	a*	0.99 ± 0.86	1 ± 1.16	0.102
	b*	19.99 ± 0.58	18.99 ± 0.58	0.098
Group NB	**L***	80.45 ± 1.82	78.99 ± 1.46	<0.001
	a*	1 ± 1.6	1.1 ± 1.61	0.512
	b*	19 ± 1.13^{B}	17.41 ± 1.43	<0.001

U: Mann Whitney U Test; t: Independent Samples *t*-test; Mean ± standard deviation (α = 0.05).

The one-way ANOVA indicated that the boric acid application affected the color change within the ceramic after aging ($p < 0.001$). The mean color change (ΔE_{00}) in B after aging was significantly lesser than the color change in NB after aging ($p < 0.001$) (Table 2). The color difference between B and NB increased after aging ($p < 0.001$) (Table 3).

Table 2. One-way ANOVA for the effect of boric acid on ΔE_{00}.

Group	Mean ± SD	Test Statistics	*p*
Group B	0.58 ± 0.54	t = 7.811	<0.001
Group NB	1.35 ± 1.52		

The significance level was set at ($\alpha = 0.05$).

Table 3. Color differences (ΔE_{00}) between B and NB before and after aging.

	before Aging	after Aging	*p*
Color difference between Group B and Group NB	0.86	2.12	<0.001

The significance level was set at ($\alpha = 0.05$).

No significant effect of aging on the TP of A was found ($p = 0.143$), but the TP of the NB group significantly decreased after aging ($p < 0.001$) (Table 4).

Table 4. Mean and SD valus of TP values before and after aging.

TP	before Aging	after Aging	*p*
B	17.33 ± 1.2	17.92 ± 1.3	0.143
NB	16.93 ± 1.3	13.32 ± 0.97	<0.001

SD: standard deviation; TP: translucency. The significance level was set at ($\alpha = 0.05$).

4. Discussion

The null hypothesis in this investigation was abandoned since the color change of PICN material after accelerated artificial aging was significantly affected by boric acid. The colour of dental ceramics can change due to several intrinsic and extrinsic factors, such as the amount of metal oxide present, the chemical makeup of the metal oxide, and the amount of water that specific beverages absorb. [1,2,4,12,19,23] A commonly known Vita Enamic [VE] was used for tests in the presented study. According to the previous study, the lower TP values and color change were observed in the VE group because the alumina content was 8.31% by weight [2].

Translucency decreases as light reflection rises. Porosities, additives, flaws, grain sizes, boundaries, the crystalline phase, and thickness may impact internal light scattering [11]. Furthermore, edge loss occurs during reflectance measurements when light is scattered to the edges without being entirely reflected. This can cause persistent mistakes in spectrophotometer-based color coordinates, making it difficult to determine the color of translucent specimens. The small window of the spectrophotometer used to determine the degree of translucency may cause edge loss, which affects the accuracy of the measurements [21]. Therefore, the presented study used a spectroradiometer for color and translucency determination.

Nanosized materials were divided into four categories as nano powder, nanofiber, nanomembrane, and nano block. Nano powder is the most developed technology as well as being the most mature [15,16]. As a result, nanopowder boric acid was used in the study.

An established technique for accelerated aging is autoclaving at low-temperatures [27]. The simulation of intraoral circumstances is made possible by aging, and it is possible to evaluate the potential impact of aging, particularly the application of heat, on the behavior of ceramic particles [23]. Aging by autoclave was used in the present study to investigate potential differences in the overall color of PICN after using boric acid. The CIEDE2000 (ΔE_{00}) color difference formula was used in the presented study to obtain more accurate results than CIEDELab [2]. The parametric factors of the CIEDE2000 color difference formula were set to 1. $\Delta E_{00} \leq 0.8$, considered perceptibility, and $\Delta E_{00} \geq 1.8$ were used as the acceptability threshold [27]. The present study showed promising results for color

stability, and color differences after aging for B and NB were within clinical acceptability ($\Delta E_{00} > 1.8$). The color change value for B was below the perceptibility level, $\Delta E_{00} \leq 0.58$.

The TP is defined as the color difference of the material between a white and a black background based on the thickness of the material [32]. The translucency of the materials was affected by the internal structure and composition of the material [33]. In the present study, the higher TP values were observed in Boric acid-applied specimens. Luminosity, namely, the L* value, decreases as the amount of light returning from an object decreases [34]. The boric acid applied ceramic had a higher L value than the NB ceramic. This difference may also be because the micro-structure of the boric acid, present on the ceramic surface, may help increase the amount of light returned.

Boric acid is a flame-retardant product. Flame retardants work by a physical dilution mechanism by increasing the heat capacity of the product or reducing the fuel content to a level below the lower flammability limit. On the other hand, the layers formed on the surfaces of the materials cut the contact of the material with oxygen [7,8]. The barrier for polymer chain oxidation causes the formation of a glassy protective layer that acts as a protective layer. This layer insulates the polymer, slows pyrolysis, and creates a barrier that prevents the release of additional gases for the material to burn. As the polymer burns, it produces vapor phase free radicals that improve combustion. This endothermic process cools the polymer surface and delays fire spread for a period of time. It provides flame retardancy by preventing the formation of radicals released by chain reactions from the substrates [7,8].

The results of this study suggest that boric acid may be used to improve the color stability of the tested ceramics. The limitations of this study include the limited number of test methods and the materials selected. The performance of a different amount of nano powder boric acid-ceramic powder ratios should be investigated. Maintaining the structural stability of nano powder boric acid during the production of the ceramic was a lengthy and complex process. As a result, the use of nano powder boric acid in ceramics is still in its early stages, and more research is needed. Different materials and shades might reveal different results. Due to the multifactor nature of color changes, future investigation with a more precise simulation of the oral cavity environment should be considered to evaluate the effect of these factors. Long-term randomized controlled trials are required to better evaluate the clinical performance of boric acid in all-ceramic restorations.

5. Conclusions

According to the results of this study, the application of boric acid was effective in maintaining color stability and translucency in PICN ceramic blocks. The increased translucency in the PINC ceramic blocks to which boric acid was added may provide a better color stability in terms of esthetics.

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