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ORIGINAL ARTICLE

Surface treatment of dental porcelain: CO_2 laser as an alternative to oven glaze

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Abstract This work tested continuous CO_2 laser as a surface treatment to dental porcelain and compared it to oven glaze (auto-glaze) by means of roughness and color parameters. Three commercial veneering porcelains with different crystalline content were tested: VM7, VM9, and VM13. Porcelain discs (3.5×2.0 mm, diameter × height) were sintered and had one side ground by a diamond bur (45 μm) simulating a chairside adjustment in a clinical office. Specimens (n=7) were divided into the following groups: C—control (no treatment), G—auto-glaze (oven), and L—surface continuous irradiation with CO_2 laser (Gem Laser, Coherent; λ=10.6 μm). Laser was tested in three exposure times (3, 4, or 5 min) and two irradiances (45 and 50 W/cm^2). Roughness parameters (Ra, Rz, and Rpm/Rz) were measured using a rugosimeter (Surftest 301, Mitutoyo). Color differences (ΔE) between the G and L groups were calculated (VITA Easyshade); ΔE values up to 3.3 were considered as not perceivable. A surface analysis was conducted by stereomicroscopy (Olympus SZ61) and SEM (Stereoscan 440, LEO). Crystalline content of specimens from groups C and L (50 W/cm^2, 5 min) was assessed by X-ray diffraction and then compared. Surface roughness (Ra and Rz) observed for laser-irradiated groups was similar to G for all studied porcelains. Rpm/Rz ratios were near 1.0 for all groups that indicated a sharp ridge profile for all specimens. Only one laser condition studied (50 W/cm^2, 3 min) from VM7 porcelain resulted in color difference (ΔE=3.5) to G. Specimens irradiated with 50 W/cm^2 for 5 min presented the smoother surface observed by SEM, comparable to G. X-ray diffraction data revealed an increase in leucite crystallite size for VM9 and VM13 porcelains after laser treatment. Regarding roughness, continuous CO_2 laser applied on porcelain surface was as effective as conventional oven auto-glaze.

Keywords Dental materials · Dental ceramic · Ceramic processing · Laser

Introduction

Dental porcelain as a restorative material has a prominent role in dentistry because of some important properties such as chemical and color stability, low thermal conductivity, and good biocompatibility [1, 2]. However, these materials are brittle and have very low fracture toughness (~1.0 MPa m$^{1/2}$) [3–5], which limit their applications.

The limited fracture toughness is related to the porcelain's failure mechanism. Stress concentrations around microstructural defects will eventually lead to crack propagation and catastrophic failure [3, 6, 7]. Due to the low fracture toughness of porcelains, even low stress levels concentrated around crack tip can result in crack propagation [5]. Therefore, the presence of pores and flaws within the porcelain microstructure has a significant influence on clinical lifetime of the restoration.

The existence of flaws and defects in a porcelain structure is directly related to the processing technique, the method used to produce the green body, and the firing cycle [7].

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Moreover, ceramic prostheses usually need some mechanical adjustments prior to or after cementation which increase the surface roughness and change the flaw population [8, 9]. A recent fractographic study demonstrated that a simple surface pore may act as an important stress concentrator and lead to catastrophic failure [10]. In order to reduce the number of flaws and surface roughness caused by processing or grinding, a porcelain restoration can be submitted to glazing [8, 11, 12] or polishing procedures [13–15].

Glazing consists of a firing cycle that reaches porcelain's close-to-sintering temperatures [8, 12]. During this cycle, the porcelain surface melts and the glassy phase fills small surface irregularities. The glaze cycle may be carried out with or without the application of a new glass powder (the so-called overglaze and auto-glaze, respectively). Both approaches produce a smooth surface with significantly less flaws and higher gloss level [16, 17]. The literature shows that glazing is very important from the biological standpoint, as it reduces plaque accumulation and formation of bacteria aggregates on the porcelain surface [18–20].

Recently, an alternative heat treatment using microwave technology was proposed for the glazing cycle of one porcelain [21]. Microwave glazing resulted in specimens with fewer superficial defects and smoother surface when compared to specimens glazed in a conventional furnace. In addition, microwave glazing shortened the time needed to obtain smooth surfaces. Nevertheless, some limitations of that study included the absence of a negative control and the use of only one profile parameter: average roughness (Ra).

CO_2 laser irradiance has not yet been evaluated as an alternative surface treatment for dental porcelains in order to reduce the superficial roughness. Leucite-based porcelains absorb well the CO_2 laser wavelength (10.6 μm) [22] and CO_2 laser has already been tested for sintering two commercial dental porcelains [23]. The present study tested CO_2 laser as a surface treatment for veneering porcelains after a chairside adjustment simulation. The results obtained with this new methodology were compared to those achieved by the conventional auto-glaze. The following hypotheses were tested: (1) Regarding roughness, the surface of laser-irradiated porcelain specimens would be similar to those treated with conventional oven glaze, and (2) color difference (ΔE) between laser-irradiated and auto-glazed specimens would not be perceivable.

Materials and methods

Three commercial porcelains with different crystalline contents [24] and glass transition temperatures [25] were chosen for this study (Table 1).

A metallic device was used to standardize green specimens (discs) with 4.1 mm in diameter and 2.4 mm in height. Specimens were sintered (Kerampress, Kota) according to the manufacturer's instructions (firing cycles are described in Table 2). Specimens' dimensions after firing were 3.5×2.0 mm (±0.15).

After the sintering process, specimens had one of their surfaces ground and polished (Ecomet 3, Buehler) with a 45-μm diamond suspension (MetaDi Supreme, Buehler). Discs were then fitted to a cavity preparation machine (EDPC, University of Iowa) and had their ground surface roughened by a diamond bur (2134F, KG Sorensen) at high speed with water irrigation, simulating the chairside adjustment of a porcelain restoration in the clinic.

Specimens were then divided into eight experimental groups (n=7) for each commercial porcelain according to surface treatment, totaling 24 groups (Table 3).

A CO_2 laser, 10.6 μm wavelength (Gem Laser, Coherent), with a power output of 30 W was used in this study. The laser beam was focused in a copper mirror and was directed down over the specimen positioned on a refractory base. The spot size was 0.5 cm in diameter, and the irradiances tested were 45 and 50 W/cm^2. Laser incidence was continuous and the exposure times were 3, 4, or 5 min.

An assessment conducted with a thermocouple on specimens' surface determined the temperature peaks. After the period of one and a half minute from the beginning of laser exposure, specimens achieved the maximum temperatures of 550 °C (45 W/cm^2) and 710 °C (50 W/cm^2) approximately.

Roughness

Surface roughness was measured using the Surftest 301 rugosimeter (Mitutoyo) with a 0.25-mm cutoff and five repetitions (1.25 mm length). Three measurements were made near the center of each specimen; the reading direction was always perpendicular to the grooves left by the bur.

The roughness parameters measured were Ra (arithmetical average value of all absolute distances of the roughness profile), Rz (the average maximum peak to valley height), and Rpm/Rz ratio (Rpm is the mean value of leveling depths

Table 1 Porcelains used in this study

Porcelain brand name[a]	Application	Crystalline content	T_g (°C)
VM7	Over alumina frameworks	Amorphous	607
VM9	Over zirconia frameworks	Leucite (~5 %)	600
VM13	Over metallic framework	Leucite (~17 %)	562

T_g glass transition temperature

[a] Manufacturer: VITA Zahnfabrik

Table 2 Firing cycles for studied porcelains

Porcelain	Dry time (min)	Start temp (°C)	Heating rate (°C/min)	High temp (°C)	Vacuum end temperature (°C)	Hold time (min)
VM7	6	500	55	910	910	1
VM9	6	500	55	910	910	1.5
VM13	6	500	55	880	880	1
VM7 (G)	4	500	80	900	No vacuum	1
VM9 (G)	4	500	80	900	No vacuum	1
VM13 (G)	4	500	80	880	No vacuum	2

G glazing cycle

of five consecutive sampling lengths). A two-way ANOVA was applied to the groups' means followed by a post hoc Tukey test with significance for $p<0.05$.

Color

Color differences among specimens from different groups were verified by CIELAB system, as proposed by the *Commission Internationale de l'Éclairage* (1976) [26]. In this system, color is expressed by three coordinates: L value represents the lightness of an object; a value represents the chromatic coordinate that varies from red (+) to green (−) and b value is determined in the chromatic coordinate that varies from yellow (+) to blue (−).

The measurement of color differences was based on the L, a, and b parameters obtained from the laser treatment groups and on the same parameters obtained from G group (L_0, a_0, b_0). This color difference, also called ΔE, was determined by the following equation:

$$\Delta E = \left[(L-L_0)^2 + (a-a_0)^2 + (b-b_0)^2\right]^{1/2}.$$

For this study, differences in ΔE greater than 3.3 were considered as perceivable differences for an untrained observer [27].

Color parameters were obtained with a portable spectrophotometer (VITA Easyshade). Before measurements, all specimens were positioned over a pre-sintered 1.5-cm-thick zirconia block (VITA In-Ceram YZ for inLab) in order to standardize the background.

Table 3 Group distribution

Group	Treatment
C	Control ground with a diamond bur
G	Ground with a diamond bur followed by oven glazing
L45/3	Ground followed by continuous CO_2 laser, 45 W/cm^2, 3 min
L45/4	Ground followed by continuous CO_2 laser, 45 W/cm^2, 4 min
L45/5	Ground followed by continuous CO_2 laser, 45 W/cm^2, 5 min
L50/3	Ground followed by continuous CO_2 laser, 50 W/cm^2, 3 min
L50/4	Ground followed by continuous CO_2 laser, 50 W/cm^2, 4 min
L50/5	Ground followed by continuous CO_2 laser, 50 W/cm^2, 5 min

Stereomicroscopy and SEM

Micrographs were obtained by stereomicroscopy (Olympus SZ61) and scanning electron microscopy (Stereoscan 440, LEO). Images were analyzed and compared.

X-ray diffraction

Crystalline microstructures in groups C and L50/5 were analyzed and compared using X-ray diffraction (RU-200B, Rigaku), with Cu Kα radiation, step scan $2\theta=0.05°$, and counting time per step of 10 s.

Results

Roughness means per group are shown in Table 4. Regarding Ra parameter, ANOVA showed no significance for porcelain brand; group L45/4 showed roughness reduction when compared to control group (C). All specimens treated with CO_2 laser exhibited Ra roughness similar to that observed in

Table 4 Means and standard deviations (in micrometer) of average roughness (Ra) and average maximum peak to valley height (Rz)

Group	Ra	Rz	Rpm/Rz[a]
C	3.1 (0.5) b	10.5 (2.0) B	0.9 (0.2)
G	2.9 (0.5) ab	8.6 (1.5) A	0.9 (0.1)
L 45/3	2.5 (0.6) ab	9.4 (2.3) AB	1.0 (0.3)
L 45/4	2.4 (0.6) a	8.4 (2.3) A	0.9 (0.2)
L 45/5	2.7 (0.7) ab	9.1 (2.0) AB	0.9 (0.2)
L 50/3	2.6 (0.7) ab	8.4 (2.0) A	1.0 (0.2)
L 50/4	2.8 (0.7) ab	8.9 (2.1) AB	1.0 (0.2)
L 50/5	2.5 (0.5) ab	8.5 (2.0) A	0.9 (0.2)

The same letters in the same column correspond to statistically similar results ($p>0.05$). Rpm is the mean value of leveling depths of five consecutive sampling lengths

[a] The morphological profile of studied surfaces. A profile near 1 indicates a sharp-ridged surface

oven-glazed specimens. The ANOVA for Rz showed significance to both main effects porcelain brand (p=0.0002) and surface treatment (p=0.088), with porcelain VM13 showing a smoother surface (8.1±0.5) than VM7 (9.2±0.4) and VM9 (9.5±0.5). Interaction between factors was not significant (p=0.16). Considering a statistical analysis for the groups regardless of porcelain brand, there was a reduction in Rz for groups G, L45/4, L50/3, and L50/5 in relation to the control group (Table 3). All laser irradiances and times tested showed Rz similar to group G.

For all groups, Rpm/Rz ratio was higher than 0.5 and near 1, indicating that surface treatment did not alter the morphological profile of the ground specimens. Low Rpm/Rz ratios (<0.5) denote a rounded surface profile, whereas high ratios (>0.5) denote a sharp one [28]. Therefore, the roughness profile of the specimens may be characterized as a sharp or peaked one.

Color differences among laser groups and the oven-glazed group (positive control) are described in Fig. 1. A perceivable color difference (ΔE<3.3) between laser and oven treatments was found only in group L 50/3 ($\Delta E \cong 3.5$) for VM7 porcelain.

Images obtained by stereomicroscopy revealed a glossier surface for specimens from group G (Fig. 2). By increasing irradiance and time in laser groups, the specimens became similar to those from the G group.

SEM images showed that specimens from groups G and L50/5 achieved a more homogeneous surface pattern compared to other groups (Fig. 2). For laser groups, the higher the laser irradiances and times tested, the smoother the surfaces became.

Diffractograms revealed a slight increase in the crystallinity of the VM9 (Fig. 3) and VM13 specimens from group L50/5 when compared to C, by measuring the peak width and peak area. All peaks in the diffractograms corresponded to leucite ($KAlSi_2O_6$). No crystalline content was found in VM7 porcelain which is amorphous.

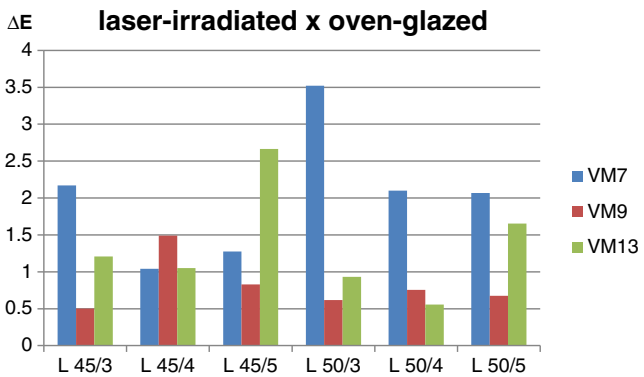

Fig. 1 Graphical representation of color difference (ΔE) between laser-irradiated and oven-glazed specimens

Discussion

The first hypothesis of this work was accepted. Laser-irradiated specimens showed Ra, Rz, and Rpm/Rz means similar to those observed in the G group.

A large number of studies that evaluated surface treatments on porcelains only measured the Ra parameter [21, 29, 30]. Considering that the coefficient of variation obtained for roughness parameters (Ra and Rz) of this study was about 20 %, it is possible to infer that more than one parameter is needed to detect small differences among different groups. Furthermore, the analysis of a higher number of roughness parameters gives more information about the surface morphology [31].

Specimens with similar Ra values may have distinct peak to valley heights on their surface profiles and these peaks may be more rounded or pointed. This fact justifies the use of Rz parameter and Rpm/Rz ratio besides Ra. The Rpm/Rz ratio could show if the oven-glazed porcelain or the laser treatment would be able to fuse the porcelain surface changing the pointed profile left by the bur in the adjustment simulation. Slight changes in surface profile were expected since there would be formation of a glassy phase, but it seemed that this glassy phase only filled the valley region, without modification of the peak shape for any of the treatments.

Though the roughness expressed in Ra and Rz showed a similarity among laser groups and G group, SEM images only indicated a similarity between G and L50/5 groups. This indicates that even adopting more than one roughness parameter, the rugosimeter should not be the only instrument used when characterizing a surface. A force atomic microscopy analysis should be conducted in the near future in order to give a more reliable surface profile and help detect minimal changes resulting from CO_2 laser treatment.

The second hypothesis tested was that color parameters would not be altered by laser application. To confirm this hypothesis, the color of irradiated specimens was compared to the color of the oven-glazed specimens by means of ΔE. It was considered that color differences (ΔE) below 3.3 would not be perceivable for an untrained observer [27], although there is not a consensus in the literature about which value would represent this limit [32–34]. Based on this, the second hypothesis was accepted for VM9 and VM13. For VM7 porcelain, the hypothesis was partially accepted since group L50/3 presented a color difference in relation to G that was higher (ΔE=3.5) than the perceivable limit.

Surface temperatures achieved in irradiated specimens were different from that observed in glazed ones. Some authors have already concluded that the firing temperature as well as the number of firing cycles may have little influence on the color of ceramics [35, 36]. These statements corroborate with the results of the present study, since laser

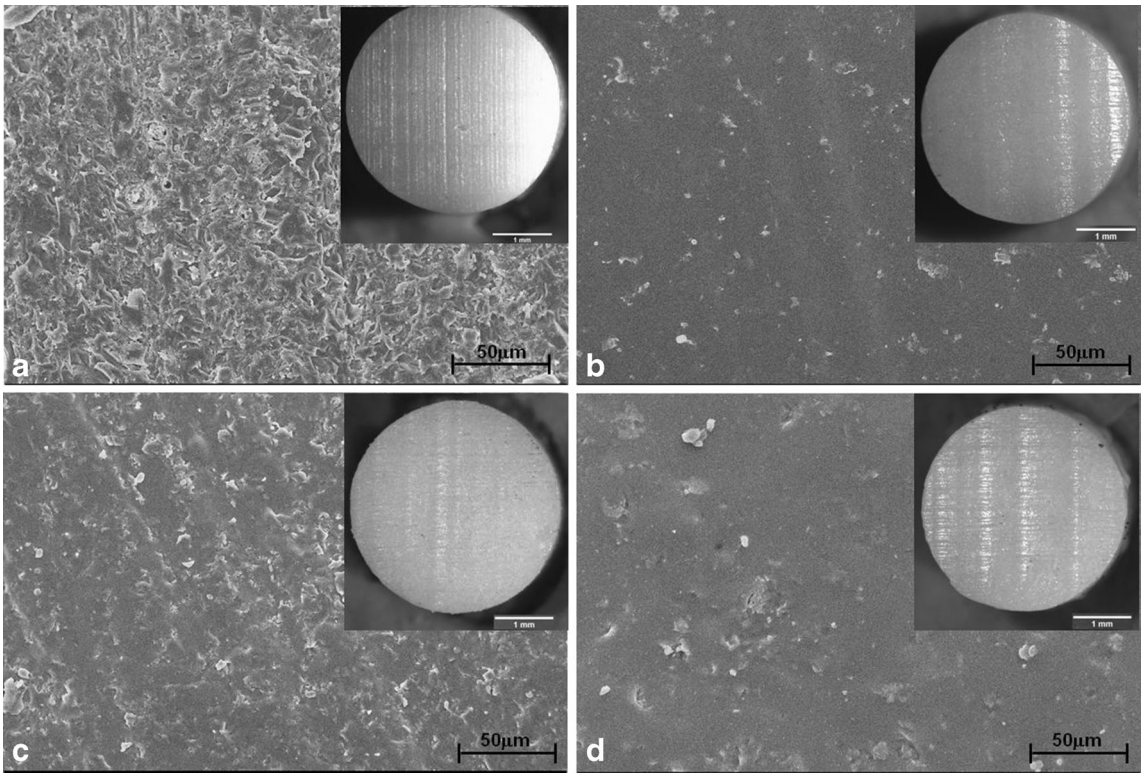

Fig. 2 Micrographs achieved by SEM (×400) and stereomicroscopy. VM13 porcelain. **a** Control group. **b** Oven-glazed specimen. **c** L 45/3. **d** L 50/5

exposure resulted in small changes in color when compared to oven glaze.

The maximum temperature achieved using the irradiance of 45 W/cm^2 (550 °C) was under the glass transition temperature (T_g) of the materials. Among the three materials used in this study, VM13 had the lowest T_g value (562 °C). This can explain why it presented lower Rz mean value than those of the other brands. A small amount of glassy phase in this material would have filled the valley region on the surface. Considering the micrographs achieved in SEM, the low temperature observed in irradiance of 45 W/cm^2 may also explain why those images revealed a rougher surface at lower irradiance. It is important to note that the temperatures registered by the thermocouple may be lower than the real temperature since a temperature loss to atmosphere may have occurred.

Another relevant question addressed in this study was if laser would change the crystalline content of the studied

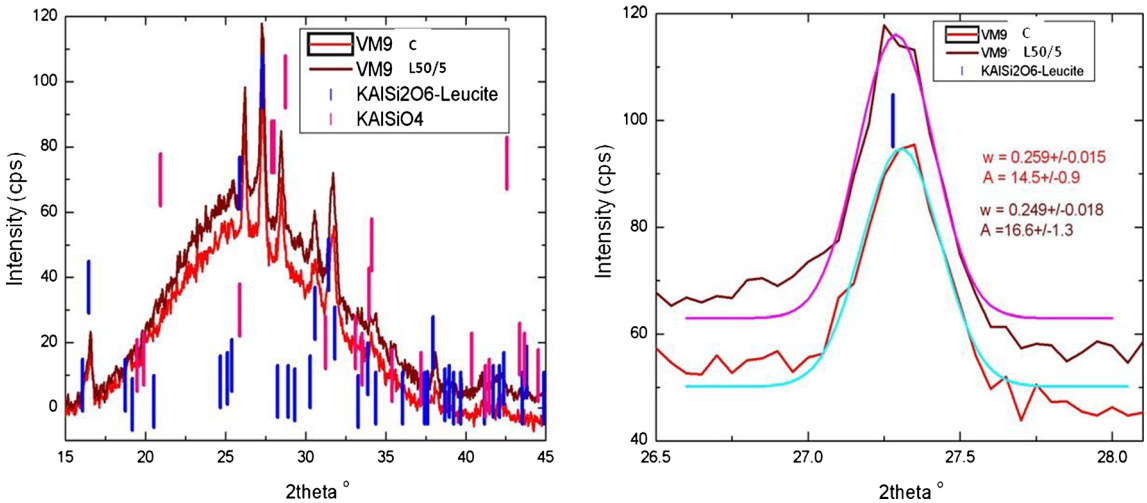

Fig. 3 Diffractograms of porcelain VM9 demonstrating the increase in crystallinity of leucite phase after laser exposition. Group L50/5 was compared to control (*C*). (*w* full width at half maximum; *A* peak area, arbitrary unit)

materials since the ceramic microstructure is largely responsible for its mechanical and optical properties. C and L50/5 groups had their microstructure assessed by X-ray diffraction. Only the higher irradiance and time were tested in order to highlight any possible difference. The diffraction results showed a slight increase in the crystallinity of the porcelains VM9 and VM13. The reduction in the full width at half maximum in the diffraction peak of irradiated groups indicated an increase in the leucite crystallite size, which is around 350 Å. A possible explanation would be the reordering of the grain boundaries due to maintenance of the specimen surface at high temperatures for some minutes. This modification in crystallite size did not result in any perceivable change in color when compared to oven-glazed group. Further studies should take into account other optical aspects like the translucency of the irradiated material by means of the contrast ratio [37]. Also, a mechanical characterization should be conducted with VM9 and VM13 porcelains in order to verify whether this crystalline change would affect the resistance of the material.

The higher speed of the process is the great advantage in relation to conventional furnace auto-glaze.

Conclusions

This study showed that CO_2 laser can be an alternative to oven glaze as a surface treatment agent after a chairside adjustment in dental veneering porcelain when considering roughness and color parameters.

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