



## Comparative analysis of techniques for extrinsic characterization of CAD/CAM ceramics

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### ABSTRACT

The present study aims to evaluate surface roughness and color stability of lithium disilicate and leucite ceramics after conventional and a novel surface finish techniques. Ceramic blocks ( $n = 84$ ) of lithium disilicate and leucite were prepared and divided into 3 groups, being group 1: polished ceramic (negative control) (NC); group 2: stained (pigmented) ceramic followed by glaze (positive control) (PC) and group 3: simultaneous application of staining and glaze (experimental technique) (ET). Changes in luminosity and surface roughness were evaluated at 4 different time-points after a simulated brushing cycling test. Multivariate test results showed that there was a triple interaction effect between applied material, finish technique and time on their average roughness ( $p < 0.001$ ) and luminosity ( $p < 0.05$ ). There was no statistically significant difference in the mean roughness considering NC and ET groups for the leucite and lithium disilicate ceramics, but there was an increase in roughness mean at t1 for the PC and ET groups. Considering luminosity (materials vs. time), there was a statistically significant difference in leucite PC group. The NC and ET groups did not differ statistically from each other. There was a luminosity reduction in NC lithium disilicate ceramics for PC and NC groups. Also, analysis of color variation ( $\Delta E$ ) revealed significant differences for disilicate but not for the leucite groups. In leucite-based ceramic groups, surface roughness showed intermediate values between ET and the other groups at all times. Correlation analysis between roughness and luminosity presented significant results for leucite ( $r = 0.331$ ;  $p < 0.001$ ) and non-significant for lithium disilicate groups ( $r = 0.068$ ;  $p > 0.05$ ). Results suggested the possible application of the experimental technique (ET) for reduction of clinical time compared to conventional techniques, by the use of less ceramic firing cycles, with no prejudice in terms of surface roughness and luminosity over the studied time.

### 1. Introduction

New materials and manufacturing methodologies for ceramic restorations have been widely proposed, mainly involving CAD-CAM milling processes from monolithic ceramic blocks [1,2]. Among the applied ceramic materials for restorations are feldspar-based, leucite, lithium disilicate and zirconium oxide, as they present mechanical, biological and physical characteristics well described in the literature [3,4].

One of the great challenges of restorative dentistry is the artificial reproduction of the natural tooth aspect. The demand for all-ceramic restorations have increased due to their biocompatibility [5], excellent

esthetic properties and adequate mechanical strength to withstand the functional forces, along with their bond strength to the dental walls [6], specially of vitreous ceramics [2]. Considering their characteristics, the luminosity of ceramic restorations might be influenced by their natural color, thickness [7], surface texture [8], material composition [9], translucency [10], resin cement shades [10–13] and cementing technique [14]. Based on these features, ceramic restorations could achieve superior esthetic results by mimetizing the optical properties present in natural teeth [15].

Monolithic CAD ceramic blocks offer consistent quality manufacturing control [16], providing less material interfaces, absence

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of different flexural modulus, compatible elasticity and thermal expansion [17] and lower reported mechanical failures leading to better clinical performance when compared to conventional ceramics applying layering techniques [15,18]. Also, a substantial reduction in laboratory time that could positively influence manufacturing costs was reported [19]. Prefabricated blocks also offer a variety of shades and different levels of translucency, adding favorable esthetic properties. Also, to obtain restorations with improved esthetics, the application of extrinsic characterization of these monochromatic blocks has been suggested [20].

Concurrent with the evolution of adhesive and minimally invasive dentistry, there has been an increase in the production of restorations with reduced thickness when conservative preparations of the underlying substrate are applied [12,20,21]. These characteristics led to a stronger demand for alternative ceramic staining techniques aimed to conceal dark areas of dentin or enamel, in which the combination of hue, chroma and value would lead to a harmonic and adequate aspect of the restoration [22].

Traditional techniques for extrinsic characterization of ceramic restorations involve mainly the use of metallic oxide based pigments applied in two or more firing steps, according to guidelines established by ceramic manufacturers, resulting in additional time-consuming appointments and successive thermal stresses. To minimize these consequences, alternative techniques for ceramic extrinsic characterization have been proposed [23–26]. Some of these techniques proposed the application of three chairside stain firing steps along with the final glaze (single session), aiming reduction of clinical appointments and laboratory time along with esthetic improvements of restorations in areas of higher esthetic demand [27]. However, these alternative techniques were not longitudinally evaluated in terms of long-term color and luminosity stability, specially when subjected to daily mechanical and chemical exposures.

Therefore, the present study aims to evaluate the possible effects on surface roughness and color stability after simulated brushing cycles of leucite and lithium disilicate CAD/CAM ceramic blocks exposed to three different finishing techniques. The assumed null hypothesis was that the experimental finishing treatment would not influence the resultant ceramic surface roughness and color stability.

## 2. Materials and methods

The present research protocol was approved by the University Research Ethics Committee (Protocol SIPESQ #9224). The ceramic materials used in the present investigation are described in Table 1. Prefabricated blocks of monolithic ceramics for CAD/CAM restorations were firstly selected and subsequently divided into 6 distinct groups, totaling 84 specimens. A group of 28 ceramic specimens received only polishing (negative control). Another group of 28 specimens received

extrinsic characterization and glaze according to the manufacturer's guidelines (positive control), and another 28 specimens received an alternative extrinsic characterization technique (Table 2). Each group presenting distinct post-milling surface finishing techniques were then divided into 2 subgroups of 14 specimens each, according to their ceramic composition.

### 2.1. Specimen design and fabrication

Eighty-four specimens presenting 1 × 1 cm (width and height) and 2 mm thick were made applying a cross-sectional cutting of ceramic milling blocks for CAD/CAM (Table 1) using a cutting machine [28] (Beiping Machine Tool, Zhejiang, China) and a specific disk (Lapmaster, METS-DCUT-W04-H012 and Diamond wafering Blade Hugh concentration 4 × 0,12 × 5), under constant water cooling at 1000 rpm. Each section was standardized and measured with a digital caliper (Lee Tools, Mauá, São Paulo). Each group was then divided into three subgroups: polishing (negative control; n = 28), standard characterization technique (positive control; n = 28) and experimental characterization technique (experimental technique; n = 28) (Table 2). Then, surface preparation of all specimens was performed with sandpaper (No.400) under light manual pressure until a flat ceramic plane was obtained, establishing time zero (t0), in which all specimens had their initial surface roughness measured prior to receiving each tested finish treatment.

### 2.2. Negative control samples

After performing crystallization firing (see Table 3 for parameters) on the pre-crystallized lithium disilicate ceramics, the negative control specimens were polished with a sequence of color diamond polishers indicated for ceramic restorations (Ceram Eve Diapol H8, Germany), being blue for wear (H8DG), pink for finishing (H8Dmf) and gray for final gloss (H8D). Then, a goat fur brush (American Burs, Palhoça, Brazil) with Optrafine 0.1 μm polishing paste (Ivoclar Vivadent, Schaan Lechtenstein) was used with a handpiece (Kavo Konzept, Biberach, Germany) at 5500 rpm for manual polishing. All polishing procedures were performed by a single operator, using moderate pressure for 15 s for each applied diamond disc and brush.

### 2.3. Positive control samples

#### 2.3.1. IPS e.max CAD

Lithium disilicate samples were firstly crystallized in a ceramic oven (Programat CS2 software version 3.0), without the application of stains and glaze, then shade characterization, firing and glaze was carried out. Applied materials were those recommended by the manufacturer, meaning IPS e.max Ceram Shades, Essences and Glaze.

The applied firing temperature was 403 °C/757 °F, being 6 min the required time for equipment locking. Then, an increase in temperature of 90 °C/162 °F/min was executed. The initial firing temperature was 820 °C/1508 °F for 10 s. Afterwards, there was an increase rate in temperature of 30 °C/54 °F/min, and then the second firing at 840 °C/1544 °F for 7 min was performed. The samples then received the first vacuum at 550 °C/820 °F (1022°/1508 °F) and the second vacuum at 820 °C/840 °F (1508°/1540 °F). The applied oven cooling temperature was from 700 °C/1292 °F to room temperature.

**Table 2**  
Distribution of samples in the control and test groups.

Materials	Polishing (NC)	Stain + Glaze (PC)	Experimental Technique (ET)	SAMPLES (n)
Lithium disilicate	14	14	14	42
Leucite	14	14	14	42

**Table 1**  
Description and chemical composition of the applied ceramic materials.

Material	IPS e.max CAD	IPS Empress CAD
	<sup>a</sup> Ivoclar Vivadent	<sup>a</sup> Ivoclar Vivadent
Description	Lithium disilicate-reinforced glass ceramic	Leucite-reinforced glass ceramic
Chemical composition (%)	SiO <sub>2</sub> (57–80) Li <sub>2</sub> O (11–19) K <sub>2</sub> O (0–13) ZnO (0–8) ZrO <sub>2</sub> (0–8) P <sub>2</sub> O <sub>5</sub> (0–11) Other oxides	SiO <sub>2</sub> (60–65) Al <sub>2</sub> O <sub>3</sub> (16–20) Na <sub>2</sub> O (3,5–6,5) K <sub>2</sub> O (10–14) Other oxides
Color	A2	A2
Batch	W29214	Y26515

<sup>a</sup> Source: Schaan, Liechtenstein.

**Table 3**

Description of the crystallization cycles applied in leucite and lithium disilicate ceramic groups, as recommended by the manufacturer.

B (° C)	S (min.)	t <sup>1</sup> (° C/min.)	T <sup>1</sup> (° C)	H <sup>1</sup> (min.)	t <sup>2</sup> (° C/min.)	T <sup>2</sup> (° C)	H <sup>2</sup> (min.)	V <sup>1</sup> (° C)	V <sup>2</sup> (° C)	L (° C)	L° C/min
403	6:00	90	820	0:10	30	840	7:00	550/1508	820/1544	700/1292	0

The firing parameters for staining and glaze firing were as follows: the standing temperature was 403 °C/757 °F, with 6 min as the required time for equipment locking. Afterwards, there was a gradual increase in temperature rate of 60°/108 °F/min. The initial firing temperature was 770°/1418 °F and held for 2 min. Then, the samples received the first vacuum at 450°/842 °F and the second vacuum at 769°/1416 °F.

#### 2.4. IPS empress CAD

The leucite specimens received extrinsic staining and glaze applying the following parameters: waiting temperature at 403 °C, being 6 min the required time for equipment locking. Afterwards, there was an increase in temperature rate of 100 °C/min. The initial firing temperature was 790 °C and kept for 2 min.

#### 2.5. Experimental finish technique

To finish the samples in the experimental technique, at first, a thin layer of Glaze Fluo (Ivoclar/Vivadent, Liechtenstein) was applied over their entire surface. Then, the first extrinsic characterization layer with the SDO shade (Ivoclar/Vivadent, Liechtenstein) was brushed on the surface of each sample (Syntec 000 Smile Line, São Paulo, Brazil). In this technique, the pigment is prepared along with the modeling liquid Zir-Liner (Ivoclar/Vivadent, Liechtenstein), until a dough consistency is achieved. The specimens were placed in the CS2 oven (Ivoclar, Liechtenstein) applying manual programming and fast crystallization mode. After cooling, a new layer of the same pigment diluted with ZirLiner was applied to their entire surface for the second firing step. Then, a third firing step was performed just as described above, using the same SDO pigment (Table 4).

#### 2.6. Abrasion test through simulated brushing

Abrasion test was conducted applying a brushing simulation machine with bidirectional rectilinear 12 mm-stroke cyclic movements of a tooth brush as previously described [29]. Each specimen was fixed in an acrylic plate (55 × 25 × 4mm) with cyanoacrylate at their base for stabilization. Each plate was then inserted in an acrylic bowl and fixed to the brushing machine by metal pins. Both applied toothpaste (Colgate Classic, Colgate-Palmolive, Brazil) and toothbrush (Colgate Professional, Colgate-Palmolive, Brazil) were purchased in the market.

Six grams of toothpaste were weighed on a precision scale (AG 204 Mettler/Toledo) and mixed with 6 ml of distilled water, forming a

homogeneous paste in a 1:1 ratio, which was dispensed into the acrylic vat covering each sample completely. The toothpaste and toothbrush were changed every four brushed samples from each group. The load applied at the equipment articulated arm was standardized at 200 g over the specimen surface, aiming simulation of clinical hygiene conditions, with a constant speed of 250 cycles per minute. The simulated brushing period was divided into three times: t1: 10,000 cycles (40 min sequences), which simulated approximately 1 year of brushing [30]; t2: 20,000 cycles, corresponding approximately to 2 years of brushing [28] (1 h and 20 min) and t3: 40,000 cycles, corresponding approximately to 4 years of brushing [29,30] (2 h and 40 min), totaling 70,000 cycles per sample.

#### 2.7. Color analysis with spectrophotometer

Color measurements were performed using a spectrophotometer (Vita Ease Shade Advance 4.0, Vita Zahnfabrik, Bad Säckingen, Germany), and CIE L\*a\*b\* values were recorded and analyzed, as well as color (ΔE) variations. This device presents a measurement head that utilizes 45° illumination and 0° viewing angle geometry (specular component included) for color measurements of shiny surfaces, with light provided by a pulsed xenon lamp over a 3 mm area. Measurements were performed at the most central point of each specimen, both at the beginning (t = 0) and at the end of each brushing cycle (t1, t2 and t3), and the mean value of the CIE L\* was obtained, where L represents the luminosity of the ceramic surface. The spectrophotometer was calibrated according to the manufacturer's instructions and as described in the literature [10].

#### 2.8. Surface roughness analysis

A surface roughness meter (SJ 201 Mitutoyo, Kawasaki, Honshu, Japan) was used to measure the mean surface roughness (Ra, μm) of the samples. Three readings were performed on each specimen, being two at their right and left ends and one at their center. The arithmetic mean of measurements was calculated to determine the total roughness of the sample. The needle of the roughness meter traveled the surface of the specimens with a limited displacement of 250 μm, digitally recording the results (μm), with a cutoff of 0.25 until obtaining the average roughness (Ra). The roughness meter was coupled to a metal base to eliminate unwanted vibrations, ensuring the accuracy of each reading. An identification mark on the edge of each sample was performed with a diamond drill at high speed to ensure the same direction in each

**Table 4**

Description of simultaneous enamel extrinsic pigmentation cycles (alternative technique) in leucite and lithium disilicate ceramic groups.

Burn	A (° C)	S (min.)	t (° C/min)	V <sup>1</sup> (° C)	V <sup>2</sup> (° C)	H (min.)	TIME (min.)	T <sup>1</sup> (° C)	T <sup>2</sup> (° C)
1st	403	2:00	80	450	724	1:00	6	820	840
2nd	403	2:00	80	450	724	1:00	6		
3rd	403	6:00	60	450	724	1:00	13		

\*A = preheating.

\*B = hold temperature.

\*S = closing time.

\*V<sup>1</sup> = activate the vacuum.\*V<sup>2</sup> = disable vacuum.\*H/H<sup>1</sup>/H<sup>2</sup> = residence time without vacuum.\*t/t<sup>1</sup>/t<sup>2</sup> = heating rate.\*T<sup>1</sup>/T<sup>2</sup> = firing temperature.

\*L = long cooling.

\*l = cooling rate.

roughness reading (perpendicular to the scratches). After each simulated brushing period, samples were washed in running water and immediately cleaned in an ultrasonic washer for 10 min, followed by air drying. Then, a new reading of the surface roughness was performed (Ra1, Ra2 and Ra3).

2.9. Qualitative analysis of surface roughness in scanning electron microscopy (SEM) and Atomic Force Microscopy (AFM)

SEM analysis of surface roughness was performed at Ra0, Ra1, Ra2 and Ra3 on all samples. These samples were dried in a silica gel dehumidifier for 72 h and subsequently observed in a scanning electron microscope (SEM, FEI, Stem mode, Hillsboro, Oregon) at 500× magnification. Scanning probe microscopy (MFA, Dimension Icon, Bruker, Billerica, Massachusetts), cutting edge model RTESPA 6 (T: 3.75 μm, f0: 300 kHz, L: 125 μm, k: 40 N/m, W: 35 μm) and a peak force of 60 × 60 μm<sup>2</sup> were applied aiming to ensure eligibility in the same direction (perpendicular to the surface scratches). Three dimensional images were generated for each sample using NanoScope 1.40 software (Bruker, Billerica, Massachusetts). Also, a qualitative analysis of all surfaces was performed, evaluating their structural characterization in high resolution, nanopatterning and texturing, in order to detect the presence of bubbles, grooves, cracks, glaze discontinuity and depression areas. Due to the destructive characteristics of the MFA test, at each brushing cycle and qualitative analysis, one sample from each analyzed group had to be discarded. Thus, the statistical analysis was performed with 10 samples from each group.

2.10. Statistical analysis

The obtained roughness values and color change results were submitted to the Shapiro-Wilk test, to verify their normal distribution. A three-way ANOVA test was then performed, with two fixed factors and repeated measures over time, with a significance level of 5%. Correlation analysis between luminosity and surface roughness was calculated using Pearson’s correlation coefficient.

3. Results

The multivariate test results indicated a triple interaction effect between material, treatment and time on surface roughness (p < 0.001). The combination of material and time affected the average surface roughness over time (Table 5).

When analyzing the tested materials and time, results of surface roughness indicated a statistically significant difference between tested post-machining techniques for the leucite samples, namely between t1 and t2 (p < 0.01); however, there was no significant difference in roughness at t3 for the polishing group in relation to the alternative technique. The pigment and glaze group differed statistically from the other groups at the end of t3 (p < 0.05). On the other hand, lithium disilicate ceramics showed the same behavior at t0 and t3, being the

polishing group the only one differing statistically from the other groups between t0 and t3 (p < 0.05). Considering surface roughness results, at t1 and t2, all groups showed a statistically significant difference (Table 5 and Fig. 1) (p < 0.05). When comparing different time-points, it was verified that for the leucite samples there was no statistically significant difference between the polishing and alternative technique groups, but instead a significant reduction in surface roughness was observed between the analyzed times for the stain and glaze techniques (p < 0.05). Considering the lithium disilicate samples, there was no statistically significant difference for the polishing group, but there was an increase in the average roughness verified at t1 in both stain and glaze and experimental technique groups. (Table 5 and Fig. 2). Regarding luminosity results, there was also a triple interaction between material, treatment and time factors (p < 0.05). It is noteworthy that this triple interaction was only significant due to the polishing result, which varied over time mainly for the lithium disilicate-based samples. The other combinations of material and surface treatment did not present significant changes over time (Table 6 and Fig. 4). When comparing applied materials and times, there was a statistically significant difference in the leucite samples receiving the pigment and glaze technique in relation to the other groups (p < 0.01). The polishing and alternative technique groups did not differ statistically from each other (Table 6 and Fig. 3).

Observed color (Δ&Egr) variations of tested materials, surface treatments and time were analyzed and presented in Table 7. Considering clinically perceptible thresholds of color (±2.62 Δ&Egr) variations [31], in leucite samples there was a perceptible color difference among the studied techniques, and remained perceptible over time. Color (Δ&Egr) variations did not differ within each treatment over time. Stain and glaze (PC) treatment has promoted a more intense color compared to the other surface treatments, although all tested techniques failed to promote a perceptible change in color over time. Considering disilicate samples, there were significant differences in color variation between techniques over time. Stain and glaze (PC) again promoted a more intense color compared to other techniques at t0. There were no significant differences in color (Δ&Egr) variations between all tested treatments, not exceeding the proposed perceptibility thresholds for color change over time [31].

Qualitatively analysis of the scanning electron microscopy (SEM) images revealed a high-density ceramic surface without cracks. Pores were present in lithium disilicate and leucite in NC, PC and ET groups. Black spherical areas on the lithium disilicate and leucite ceramic surfaces after glazing were also observed (Figs. 5, 6, 9 and 10). Vitrified surfaces showed greater roughness compared to polished surfaces. Atomic force microscopy (AFM) revealed a rougher surface in PC and ET groups compared to NC group (Figs. 9 and 10). After milling and glaze application, a constant topography of peaks and valleys could be verified (Figs. 5, 6, 9 and 10). The polishing technique promoted smoother surfaces compared to PC and ET groups, producing unidirectional grooves by the rubber wheels which were observed in both tested ceramics. Polished samples showed a distinct surface topography, with slight elevations and fewer wave-like structures. In the glazed ceramic,

Table 5 Comparison of the obtained mean surface roughness (Ra) between treatments and materials over time (in μm).

Material	ST	Time 0		Time 1		Time 2		Time 3					
		Mean	Std. Error	Mean	Std. Error	Mean	Std. Error	Mean	Std. Error				
Leucite	NC	0,472	0041	Ca	0,617	0066	Ca	0,635	0101	Ca	0,539	0085	Ba
	PC	1383	0,047	Aa	1378	0,059	Aab	1397	0,042	Aab	1136	0,077	Ab
	ET	1009	0,081	Ba	0,865	0022	Ba	0,893	0032	Ba	0,734	0043	Ba
Dissilicate	NC	0,377	0036	Ba	0,469	0049	Ca	0,363	0023	Ca	0,309	0046	Ba
	PC	0,831	0044	Ac	1400	0,059	Aa	1174	0,047	Ab	0,963	0111	Abc
	ET	0,749	0039	Ab	0,970	0072	Ba	0,712	0024	Bb	0,687	0049	Ab

Note: NC: Polishment; PC: Pigment + Glaze; ET: Experimental technique; ST: Surface Treatment. Mean roughness within the same combination, material and time, with different CAPITAL letters differ by the Bonferroni test at the level of 5% between treatments. Mean roughness within the same combination of material and treatment with different lowercase letters differ by the Bonferroni test at the level of 5% between times.

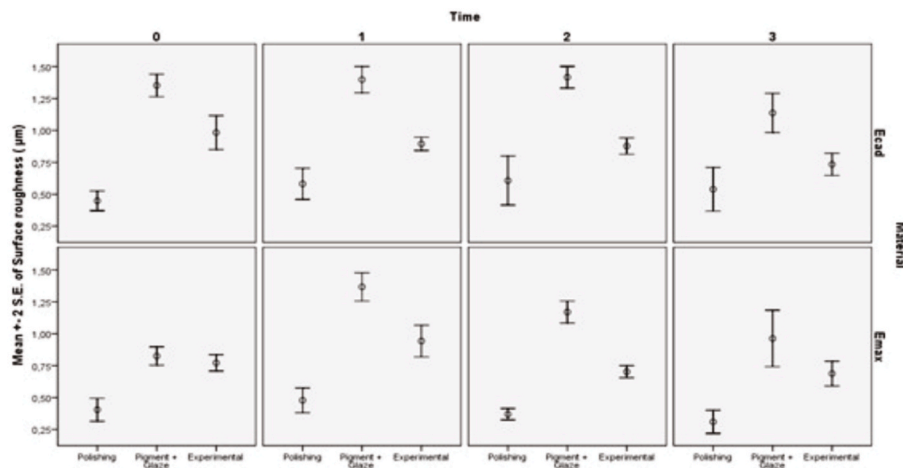


Fig. 1. Comparison of mean surface roughness between treatments, separately by material and time.

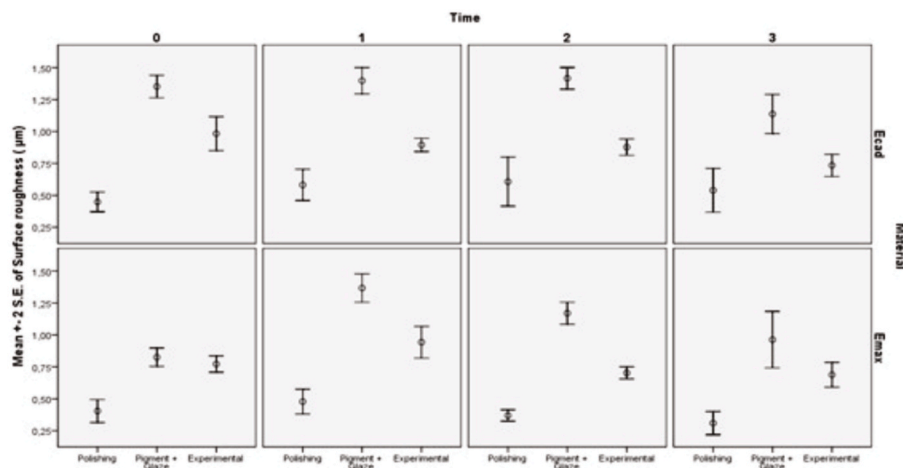


Fig. 2. Comparison of mean surface roughness over time, separately by material and treatment.

**Table 6**  
Comparison of the luminosity (CIE L\*) verified between treatments and materials over time.

Material	ST	Time 0		Time 1		Time 2		Time 3					
		Mean	Std. Error	Mean	Std. Error	Mean	Std. Error	Mean	Std. Error				
Leucite	NC	-0,255	0349	Ba	-0,100	0290	ABa	-0,100	0290	ABa	-0,100	0290	Ba
	PC	2955	0,849	Aa	2709	0,933	Aa	2709	0,933	Aa	2709	0,933	Aa
	ET	-0,763	0373	Ba	-1374	0,424	Ba	-1524	0,436	Ba	-1374	0,424	Ba
Dissilicate	NC	0,150	0307	Aa	-2770	1040	Ab	-2770	1040	Ab	-2770	1040	Ab
	PC	-0,846	1190	Aa	-1782	0,900	Aa	-1782	0,900	Aa	-1791	0,901	Aa
	ET	-1020	0,279	Aa	-1030	0,260	Aa	-1030	0,260	A	-1030	0,260	Aa

Note: NC: Polishment; PC: Pigment + Glaze; ET: Experimental technique; ST: Surface Treatment.

Mean luminosity within the same combination, material and time, with different CAPITAL letters differ by the Bonferroni test at a level of 5% between treatments. Mean luminosity within the same combination of material and treatment with different lowercase letters differ by the Bonferroni test at the 5% level between times.

the presence of greater elevations and waves could be observed. These surfaces were considered rough due to the greater number of glass layers applied to the ceramic. Representative AFM and SEM images of the tested ceramics after the application of each technique are shown in Figs. 5–10.

Correlation between roughness and luminosity was investigated and revealed a direct and significative correlation between all tested samples ( $r = 0.261$ ;  $p < 0.001$ ), suggesting that higher surface roughness levels might be related to increased luminosity. Calculated correlation analysis for each ceramic material presented significant results for leucite ( $r =$

$0.331$ ;  $p < 0.001$ ), suggesting a moderate correlation, and statistically non-significant for lithium disilicate groups ( $r = 0.068$ ;  $p > 0.05$ ), indicating that roughness was not correlated with luminosity in case of lithium disilicate ceramics.

#### 4. Discussion

Results of the present investigation indicated that the tested finishing techniques do influence surface roughness of both lithium disilicate and leucite ceramics. Also, based on the analyzed results, the null hypothesis

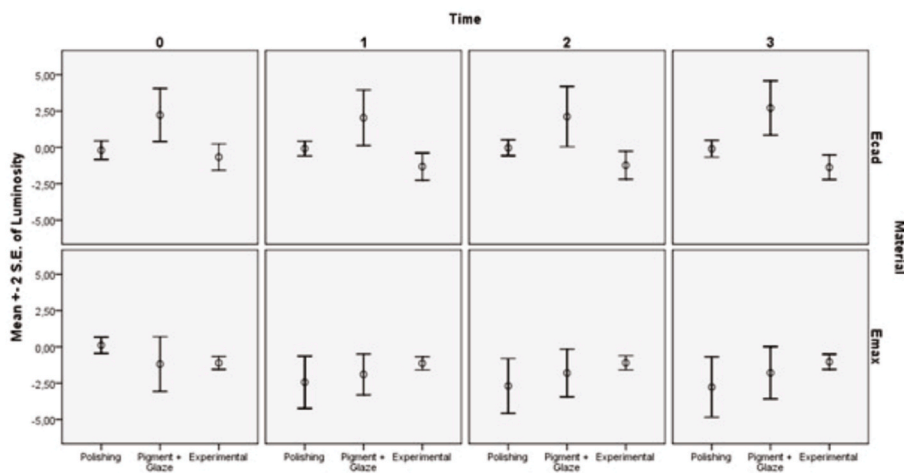


Fig. 3. Comparison of luminosity between treatments, separately by material and time.

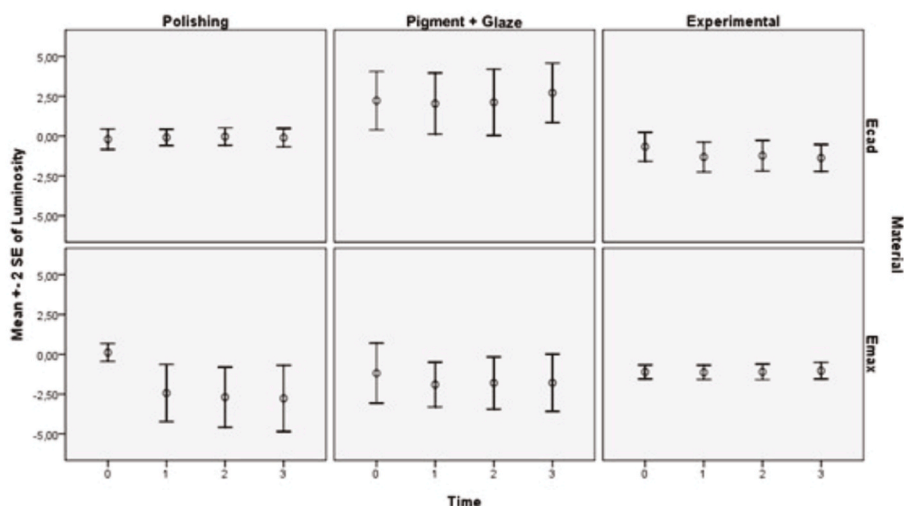


Fig. 4. Comparison of luminosity over time, separately by material and treatment.

**Table 7**  
Comparison of the color ( $\Delta E$ ) variations between treatments and materials over time.

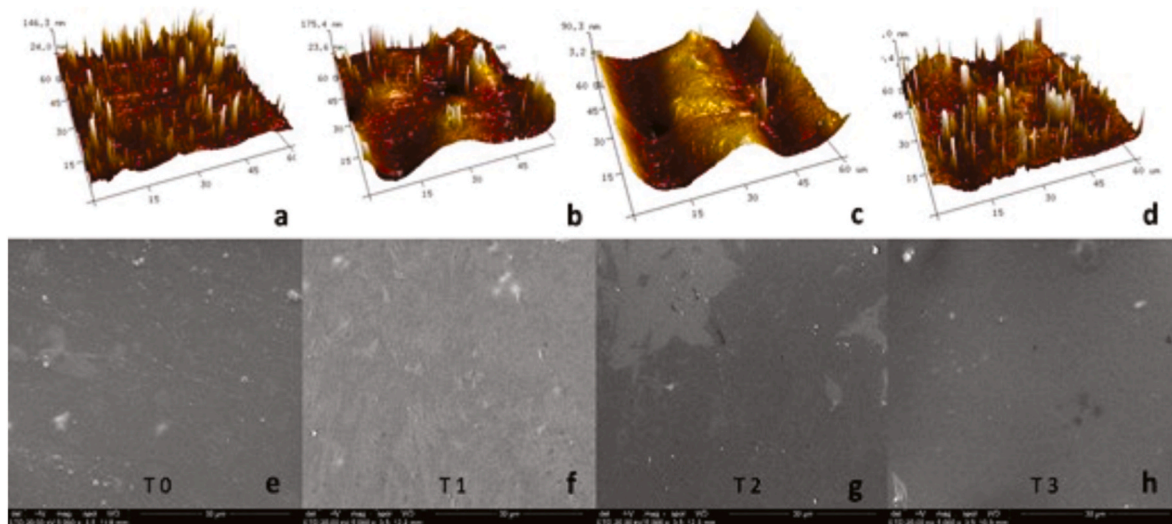
Material	ST	Time 0		Time 1		Time 2		Time 3					
		Mean	Std. Error	Mean	Std. Error	Mean	Std. Error	Mean	Std. Error				
Leucite	NC	1946	0,101	Ba	2092	0,191	Ba	2158	0,195	Ba	2118	0,209	Ba
	PC	5792	0,440	Aa	5431	0,467	Aa	5583	0,480	Aa	5391	0,481	Aa
	ET	1890	0,328	Ba	2500	0,267	Ba	2389	0,271	Ba	2413	0,306	Ba
Dissilicate	NC	2775	0,228	Ba	3483	0,836	Ba	3745	0,869	ABa	3760	0,961	ABa
	PC	5693	0,467	Aa	4914	0,468	ABa	5142	0,518	Aa	5191	0,562	Aa
	ET	2169	0,182	Ba	1958	0,143	Ba	1964	0,156	Ba	1920	0,166	Ba

Note: NC: Polishment; PC: Pigment + Glaze; ET: Experimental technique; ST: Surface Treatment.  
 $\Delta E$  Mean within the same combination, material and time, with different CAPITAL letters differ by the Bonferroni test at a level of 5% between treatments.  
 $\Delta E$  Mean within the same combination of material and treatment with different lowercase letters differ by the Bonferroni test at the 5% level between times.

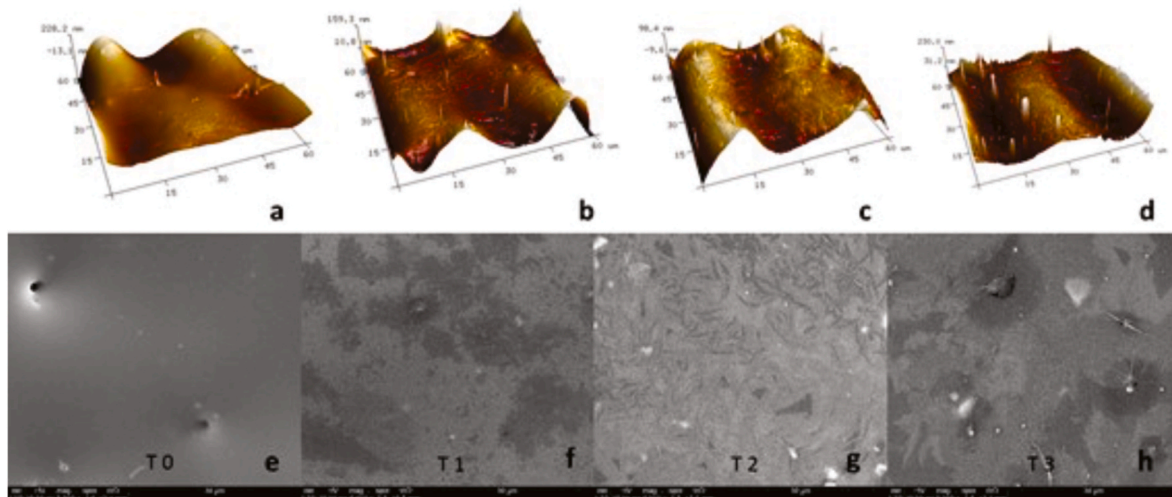
regarding comparison of treatment effects on luminosity in both materials was confirmed. However, in lithium disilicate ceramics, the polishing technique caused a statistically significant difference in surface roughness when compared to the alternative method, although both techniques differed from the application of stain and glaze. Glaze and/or stain and glaze were considered to be a mandatory step after polishing [50].

The prefabricated ceramic blocks might receive different forms of finishing surface treatment, that could be based on polishment,

conventional glazing or combined approaches. The present investigation showed that there was a significant triple-effect interaction between material, treatment and time on ceramic surface roughness, in agreement with other studies [32–36]. There were significant differences in surface roughness considering the leucite-based ceramics at t0, t1 and t2, as well as in the lithium disilicate, where the greatest increases in surface roughness were observed for the conventional stain and glaze technique. However, at t3, no differences between the stain and glaze and the experimental techniques were observed considering disilicate



**Fig. 5.** Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) of the positive control (PC) group (lithium disilicate) at initial t0 (a; e), t1 (b; f), t2 (c; g) and t3 (d; h) time-points.



**Fig. 6.** Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) of the positive control (PC) group (leucite) at initial t0 (a; e), t1 (b; f), t2 (c; g) and t3 (d; h) time-points.

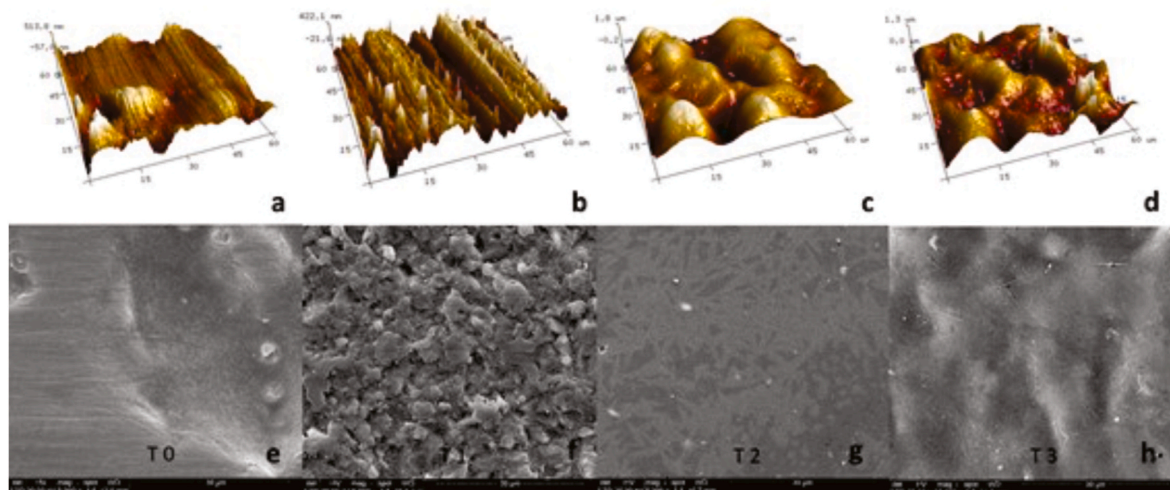
ceramics. In leucite, the experimental technique presented variations in average roughness similar to the polishing treatment.

The esthetic and functional clinical adjustments performed in ceramic restorations may affect their surface in terms of roughness and gloss [37]. In order to recover the ceramic surface smoothness and gloss after clinical adjustments, a new glaze application might be indicated. However, this process requires extra laboratory time and may result in ceramic color alterations [33]. To avoid these outcomes, finishing and polishing systems have been widely used [38]. Several studies corroborate the advantages of these polishing systems, which describe that the use of diamond bits and abrasive rubber points may also promote acceptable ceramic surface smoothness [38].

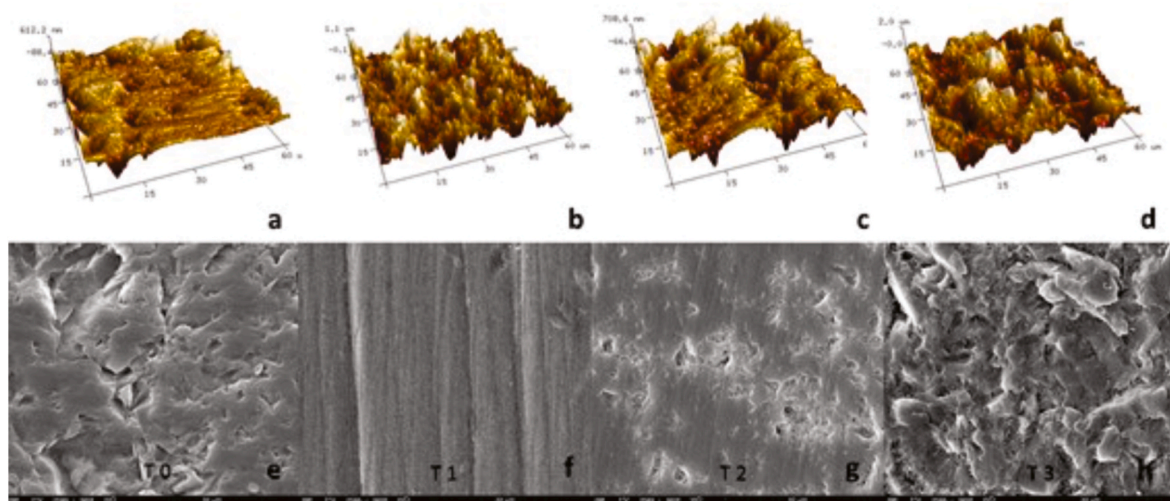
Glaze has also an effect on the ceramic surface. Studies have shown that the combination of polishing paste systems may produce even smoother surfaces or as smooth as glazing in feldspathic ceramics [38, 39]. Nevertheless, there are controversies in the literature, where some studies claim that polishing systems are less effective than conventional glaze in promoting adequate surface smoothness, both in milled and injected ceramics [32,40–42]. Also, corroborating other investigations [32,43,44], the use of a polishing paste and tips as applied here in the

control group might offer a slight improvement in gloss and surface roughness. Lower surface roughness values were observed in NC in comparison to other tested groups.

Although several studies have used  $\Delta E$  as a parameter to establish acceptable limits for color alterations in ceramics, there is no consensus in the literature on the minimum acceptable variation of  $\Delta E$  perceived by the human eye, which could be considered as clinically relevant. Color ( $\Delta E$ ) variations have been classified as visually perceptible when  $\Delta E < 1.0$  [42]; visually perceptible but still clinically acceptable when  $\Delta E > 1.0$  [1]; and clinically unacceptable when  $\Delta E > 3.5$  [2]. Other authors consider the color variation of an anterior restoration as satisfactory and clinically imperceptible when the color ( $\Delta E$ ) varies less than 2.62 [31]. We chose to select the L parameter (referring to luminosity) as the dimension that could accurately reflect perceptible changes in the ceramic value, along with the analysis on the ceramic color ( $\Delta E$ ) variations, which was supposed to be less than 2.62 to be considered clinically imperceptible [31]. Although the CIE  $L^*a^*b^*$  were chosen as parameters to be used in the present analysis, aging-dependent color changes can also be evaluated using CIEDE2000 parameters [45]. This classification contains three evaluation parameters: luminosity, chroma



**Fig. 7.** Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) of the negative control (NC) group (lithium disilicate) at initial t0 (a; e), t1 (b; f), t2 (c; g) and t3 (d; h) time-points.



**Fig. 8.** Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) of the negative control (NC) group (leucite) at initial t0 (a; e), t1 (b; f), t2 (c; g) and t3 (d; h) time-points.

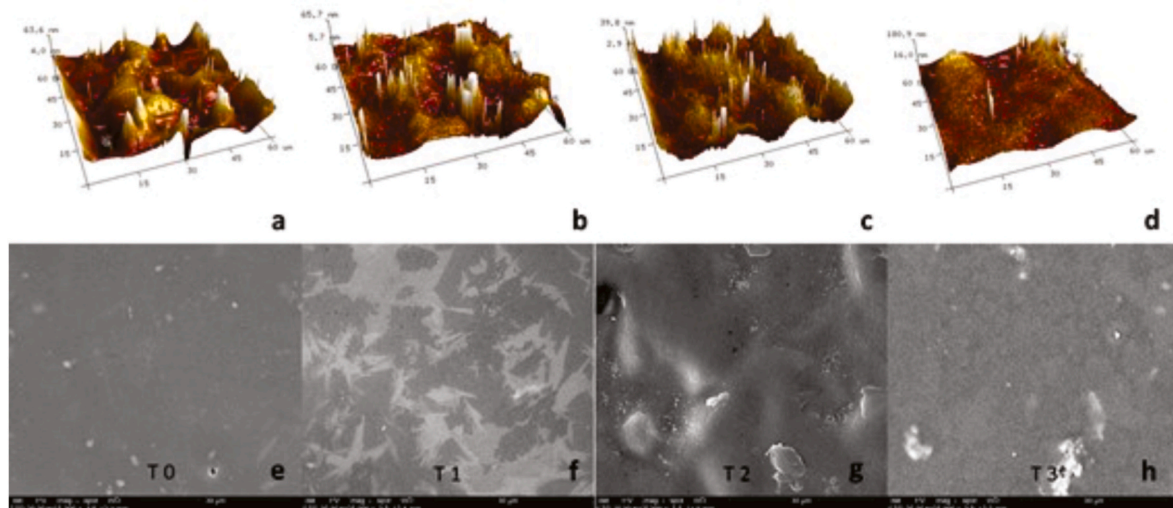
and hue, being considered the best indicator for the perception and acceptability of color differences between restorative materials and dental tissues [45]. Also, regarding the observed color ( $\Delta E$ ) variations among different analyzed groups, we believed that the type of ceramic might have influenced the observed results. A possible explanation would be that leucite is naturally more luminous than disilicate, leading to more homogeneous results in terms of color ( $\Delta E$ ) variations. Also, for this reason, we do not compare color ( $\Delta E$ ) variations among different ceramic groups, but instead compared only different techniques and times within the same material.

The possible influence of pigments present in daily diet on ceramic color has been said to be linked to the applied ceramic finishing process after milling. Surface staining rates were said to be higher for feldspathic ceramics receiving only polishing, compared to glazed ceramics [33]. These postulations corroborate the findings presented here, in which the lithium disilicate ceramic group subjected to polishing had a reduction in luminosity over time. Thus, there may be no direct relationship between roughness and luminosity, but instead there might be a direct influence of material deposition on the ceramic surface over time. Studies have shown that glaze-finished laminated ceramics showed less variation in  $\Delta E$  over time [33]. However, the same study indicated that

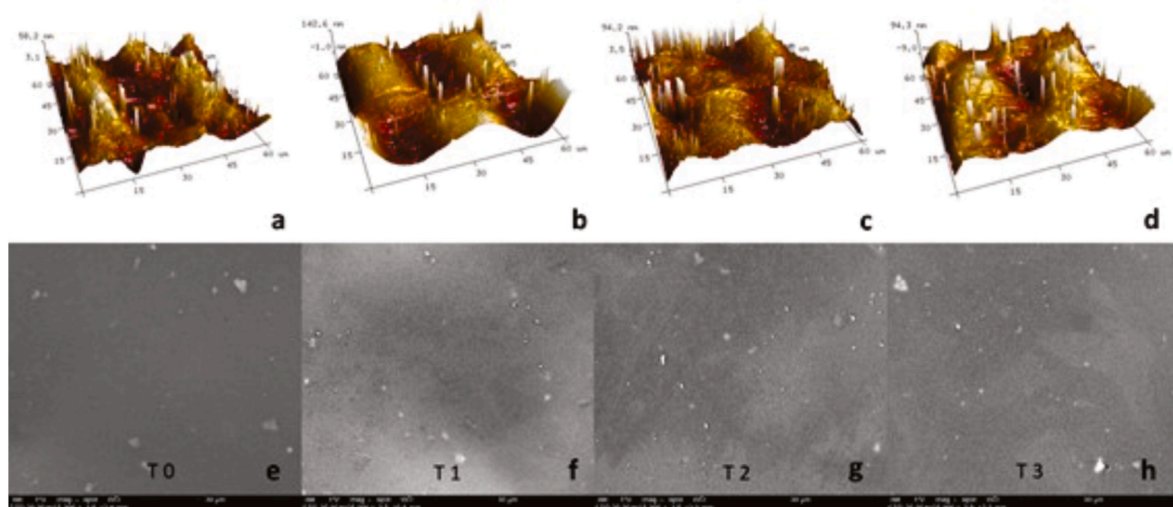
ceramic surfaces polished only with diamond burs presented the highest value of  $\Delta E$ . Other analyzed groups which received complete polishing presented intermediate  $\Delta E$  values and were statistically different from other subgroups [33].

Similar results to the present investigation were reported analyzing surface roughness obtained by different finishing approaches, meaning glaze ( $0.071 \mu\text{m}$ ), polishing systems ( $0.309 \mu\text{m}$ ) and diamond tips ( $1.279 \mu\text{m}$ ) [46]; although describing a strong correlation between color alterations and surface roughness. Our findings differed from this study possibly due to differences in applied methodologies considering polishing execution, roughness measurements and the type of applied ceramics. Also, layered ceramic methods elicit greater surface porosity compared to pressed ceramics and prefabricated blocks, which reportedly allow less surface pigmentation by extrinsic agents such as coffee, wine and green tea [47]. In the present investigation, the polishing group showed similar surface roughness and lower luminosity compared to the experimental technique in lithium disilicate ceramics. Studies have shown that color changes and consequently alterations in luminosity are directly influenced by the used polishing systems [48]. Therefore, polishing techniques may influence the color of feldspathic ceramics over time, although these changes might be considered





**Fig. 9.** Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) of the experimental technique (ET) group (lithium disilicate) at initial t0 (a; e), t1 (b; f), t2 (c; g) and t3 (d; h) time-points.



**Fig. 10.** Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM) of the experimental technique (ET) group (leucite) at initial t0 (a; e), t1 (b; f), t2 (c; g) and t3 (d; h) time-points.

acceptable in terms of visual perception.

Results presented here indicated higher roughness values in the stain and glaze groups compared to the polish and experimental technique for the tested groups. Corroborating these findings, other studies also found similar surface roughness results for lithium disilicate ceramics shortly after milling when comparing zirconia, disilicate and zirconia-reinforced silicate ceramics [49]. In addition, these same authors correlated ceramic wear to surface texture, reporting a higher ceramic and antagonist tooth wear rates associated with IPS e.max ceramics [49]. They also demonstrated that the surface roughness after clinical adjustment and chairside polishing was comparable to laboratory polishing for all tested ceramic and resin materials [49].

Restorations produced by CAD/CAM are manufactured by selective grinding of prefabricated blocks, resulting in a lower surface roughness of fully crystallized compared to partially crystallized materials. The lower hardness of lithium disilicate ceramics, due to their chemical characteristics, may provide a softer and easy-cut surface, although inducing an increased surface roughness as demonstrated previously [50]. However, in the present investigation, the leucite-based ceramic group with conventional finishing presented greater variations in

surface roughness, even though applying a hard milling process. Leucite-based ceramic with pigmentation and glaze resulted in surface roughness greater than 0.2 mm, which might eventually favor microbial adhesion according to some reports [51].

It might be relevant to point out that, although surface roughness is a factor commonly associated with changes in ceramic color, this might not be the only cause of surface staining, as ceramic pigmentation has been said to be associated with extrinsic and intrinsic factors [42]. Lithium disilicate-based ceramics show a different chemical composition, as they present crystals of different shapes in their reactive phase, making them more translucent with lower luminosity [52]. Thus, in association with specific pigment chemicals, the impregnation of extrinsic molecules on its surface might be enhanced, which may significantly change their luminosity and the final color of the restoration. This confirms the results found in the present study, since the alterations in luminosity after the simulated brushing cycles were less expressive in the IPS e.max CAD samples, whose surface roughness presented lower values, leading to the hypothesis that the ceramic molecular structure might play a more important role in color visual perception than its surface roughness.

Surface smoothness also seems to directly influence fracture strength of restorations. Surface polishing was strongly correlated with an increase in ceramic toughness and restoration fracture resistance by the elimination of minor surface cracks, being even more effective than glazing according to some studies [36,49]. Therefore, the alternative technique evaluated here might represent a promising resource, as it presented lower resultant roughness averages compared to the conventional technique for leucite samples and similar values to the conventional technique for lithium disilicate samples, while having distinct clinical advantages in terms of its application.

Another factor influencing ceramic roughness over time might be the presence of acid solutions in the oral environment. Reported *in vitro* studies verified significant changes in the Ra parameter after immersion of ceramic discs in artificial saliva, sodium fluoride and gastric fluids, inducing significant changes in surface roughness [53]. IPS e.max CAD blocks have been said to be very susceptible to dissolution in acid environments, which might induce important surface defects [54]. In the present study, there was no addition of erosive or acid substances to the applied water and fluoride toothpaste solution, so it was assumed that the verified roughness in each group was resultant solely of the combination of tooth brush bristles and the fluoride toothpaste solution on the ceramic surface.

There is also no consensus in the literature regarding the analysis of roughness as an appropriate method to assess surface degradation of a material [55]. It is important to note that the increase in surface roughness is a time-dependent process that can change significantly according to the interval of material exposure in the local environment [56]. The increase in surface roughness does not necessarily translate into greater surface degradation, but it might indicate a gradual and continuous erosion process due to the ceramic exposure to local environment agents, whether stains or acid, and the mechanical action of brushing and chewing over time.

Atomic force microscopy (AFM) images showed scratches formed by the diamond particles of the polishing agent on the leucite (polished) surfaces. These observations corroborate previous reports that analyzed leucite-based ceramics (polished and glazed) [34,50]. In addition, the presence of surface pores after ceramic milling and polishing were observed in AFM images. These pores were probably caused by the removal of highly soluble lithium phosphate spheric crystals [57] during milling of the ceramic block at the intermediate phase (lithium metasilicate) [58]. In addition, an expressive number of dark spherical areas present on the ceramic surface were not considered pores, but instead generated by the glaze process where small surface depressions were eventually sealed. Still, it is important to emphasize that the method of qualitative evaluation of AFM images is subjective, being determined by the visual analysis and interpretation of an analyst.

It should be noted that the present study presented an *in vitro* design, evaluating the effect of brushing simulation on the roughness and luminosity of each ceramic group. Although significant differences between groups were observed, it is important to consider that the presence of several factors such as specific *in vivo* oral conditions, the presence of acids in foods and beverages, fluoride compounds and the neutralizing effect of saliva might play an important role in the observed effects. Also, as the precise simulation *in vitro* of the oral environment, with all its components that might directly affect the monolithic restorations properties represents a very complex task, further *in vivo* studies should be encouraged to evaluate the long-term effects of all these oral components before conclusively recommend the clinical application of the proposed alternative finish technique.

## 5. Final considerations

Based on the findings of the present study, we may conclude that:

- 1) A similar color stability between all tested leucite groups in different periods was observed. Considering lithium disilicate ceramics, there was a reduction in luminosity in the polishing group from t1.
- 2) In leucite-based ceramics, surface roughness in the experimental technique group presented intermediate values when compared to the polishing and the pigment and glaze groups at all times. In lithium disilicate ceramics, the experimental technique group showed similar roughness to the pigment and glaze group.
- 3) The alternative technique might promote significant advantages in time and processing of ceramic restorations, by demanding less ceramic firing cycles compared to conventional techniques. Furthermore, it might provide greater surface smoothness due to the reduction in steps of ceramic staining and glaze.

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## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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