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RESEARCH ARTICLE

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The effect of milling and postmilling procedures on the surface roughness of CAD/CAM materials

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Abstract

Purpose: The aim of this study was to evaluate the surface roughness and analyze the surface topography of five different CAD/CAM ceramics and one CAD/CAM composite resin for CEREC after milling and postmilling procedures.

Materials and Methods: Blocks of the ceramics Mark II, IPS Empress CAD, IPS e.max CAD, Suprinity and Enamic, and blocks of the composite resin Lava Ultimate were milled at CEREC MCXL. Ten flat samples of each material were obtained. The surface roughness (Ra) test was performed before and after milling, crystallization, polishing, and glaze when indicated, followed by SEM and AFM analysis. Data were submitted to one-way ANOVA with repeated measures and the Tukey HSD test ($\alpha = 0.05$).

Results: The milling step significantly increased the roughness of all the tested materials ($P < .05$). Lithium-based ceramics (IPS e.max CAD and Suprinity) were more suitable to roughness than the other tested materials ($P < .05$).

Conclusions: The polishing methods were able to reduce roughness to baseline values, except for lithium-based ceramics. Glaze reduced significantly the roughness of lithium-based ceramics without a difference from the baseline. SEM and AFM images revealed that glazed surfaces are smoother than polished surfaces.

CLINICAL SIGNIFICANCE

All hard-milling CAD/CAM materials, that is, fully sintered, should be only hand polished. The glaze step can be suppressed resulting in time saving. However, the glaze step in soft-milling lithium disilicate is imperative.

KEYWORDS
CAD/CAM, dental ceramics, surface roughness

1 | INTRODUCTION

The first CAD/CAM glass ceramic Mark II introduced in the early 90s for CEREC defined a novel method of production of all ceramic restorations. The development of hardware and software led to the improvement of machined restorations, with a long-term clinical success rates.[1] Unlike slip-casting or press techniques, the milling method is based on the selective reduction of premanufactured blocks by diamond burs to form the final shape.

The diamond burs used in the milling process of the ceramic and composite resin and blocks result in a rougher surface,[2] which may facilitate the retention of microbial film[3,4] and the consequent inflammation of the periodontal tissue when it is located close to the gingival area. In addition, rougher surfaces facilitate discoloration,[5] decrease the resistance of the

TABLE 1 Materials used in the research

Trademark	Classification	Composition (wt%)	Lot	Manufacture
Mark II	Feldspar ceramic	SiO_2 (56–64) Al_2O_3 (20–23) Na_2O (6–9) K_2O (6–8) Other oxides	#35671	Vita Zanhfabrik, Bad Säckingen, Germany
Enamic	Hybrid ceramic	Ceramic (86) SiO_2 (58–63) Al_2O_3 (20–23) Na_2O (6–11) K_2O (4–6) Other oxides Polymer (14) UDMA and TEGDMA	#45810	Vita Zanhfabrik, Bad Säckingen, Germany
Suprinity LS	Zirconia-reinforced lithium silicate glass ceramic material, ZLS	SiO_2 (56–64) Li_2O (15–21) ZrO_2 (8–12) P_2O_5 (3–8) K_2O (1–4) Al_2O_3 (1–4) Other oxides	#39921	Vita Zanhfabrik, Bad Säckingen, Germany
IPS Empress CAD	Leucite-based glass ceramics	SiO_2 (60–65) Al_2O_3 (16–20) K_2O (10–14) Na_2O (3.5–6.5) Other oxides	#T50414	Ivoclar Vivadent, Schaan, Liechtenstein
IPS e.max CAD	Lithium dissilicate glass-ceramic	SiO_2 (57–80) Li_2O (11–19) K_2O (0–13) P_2O_5 (0–11) ZrO_2 (0–8) ZnO (0–8) Other oxides	#U32267	Ivoclar Vivadent, Schaan, Liechtenstein
Lava Ultimate	Resin nanoceramic	Nanoceramic fillers (80) nonagglomerated/nonaggregated 20 (nm) silica filler, nonagglomerated/nonaggregated 4–11 nm zirconia filler, and aggregated zirconia/silica cluster filler Highly cross-linked polymeric matrix	#1505000708	3M, St. Paul, MN, USA

restoration,[6,7] and cause wear of the opposing dentition when the roughness is located in the occlusal region.[8] Consequently, a reduction in the surface roughness is mandatory; such reduction can be obtained by mechanical polishing, associated with glazing, when it is indicated.

Several studies have assessed the ability of various techniques and systems to restore the surface smoothness. Some studies have shown that mechanical polishing can restore the ceramic smoothness to a level close to that of the glazed ceramics.[9–11] However, other studies have shown that the best procedure is glazing.[12,13] In fact, the quality of ceramic or composite resin polishing methods is a controversial topic in the literature. The results concerning polishing systems and their performance are inconsistent because of the different measuring

TABLE 2 Firing procedures

Material	Stand-by temperature, °C	Closing time, min	Temperature increasing rate, °C/min	Firing temperature, °C	Holding time, min	Vacuum
Suprinity crystallization	400	4	55	840	8	Vac 1 410°C Vac 2 840°C
IPS e.max CAD crystallization	403	6	90	820	7:10	Vac 1 550°C Vac 2 820°C
Akzent Plus Glaze	500	6	80	950	1	-
IPS Empress Universal Glaze	403	6	100	790	1	-

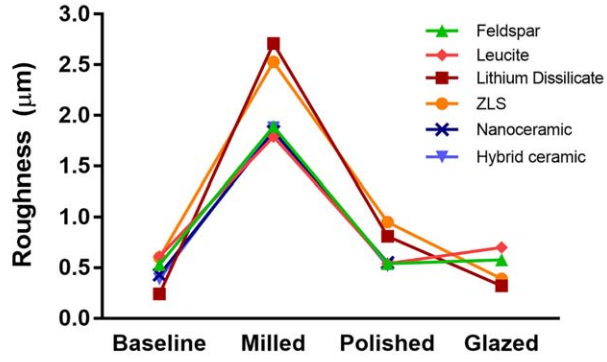
TABLE 3 Surface roughness averages (μm) and standard deviations of the materials

Mark II (Feldspar)	Baseline	0.52^{abcd} (0.10)
	Milled	1.88^{f} (0.41)
	Polished	0.54^{abcd} (0.09)
	Glazed	0.58^{bcd} (0.10)
IPS Empress CAD (Leucite)	Baseline	0.60^{bcd} (0.14)
	Milled	1.79^{f} (0.49)
	Polished	0.54^{abcd} (0.15)
	Glazed	0.70^{cde} (0.11)
IPS e.max CAD (Lithium disilicate)	Baseline	0.23^{a} (0.03)
	Milled*	2.71^{g} (0.39)
	Polished	0.81^{de} (0.11)
	Glazed	0.32^{ab} (0.10)
Lava Ultimate (Nanoceramic)	Baseline	0.43^{abc} (0.07)
	Milled	1.84^{f} (0.13)
	Polished	0.54^{abcd} (0.09)
Enamic (Hybrid ceramic)	Baseline	0.39^{abc} (0.05)
	Milled	1.88^{f} (0.26)
	Polished	0.51^{abcd} (0.09)
Suprinity (Zirconia-reinforced lithium silicate, ZLS)	Baseline	0.60^{bcd} (0.15)
	Milled*	2.52^{g} (0.29)
	Polished	0.95^{e} (0.12)
	Glazed	0.38^{abc} (0.12)

*After crystallization.
Values with different superscript letters are significantly different according to the Tukey HSD test ($P < .05$).

parameters and different combinations of polishing methods and ceramic or composite resin materials. However, there are few studies discussing the effect of milling and postmilling procedures on CAD/CAM materials.[14–16] In particular, for dental practitioners who use chair-side CAD/CAM-technologies, such as CEREC, it would be of great interest to know the roughness on the material surface due to the milling process, and the efficiency of the polishing procedures (mechanical polishing associated or not with glazing, when indicated) in restoring a smooth surface.

Therefore, the aim of this study was to evaluate the surface roughness and analyze the surface topography of five different CAD/CAM ceramics and one CAD/CAM composite resin for CEREC after milling and postmilling procedures (crystallization, polishing, and glaze, when suitable). The hypothesis tested was that mechanical polishing associated with glazing, when indicated, is important to restore the surface roughness of the materials.



SCHEME 1 Overall comparison of surface roughness of tested materials at different procedures

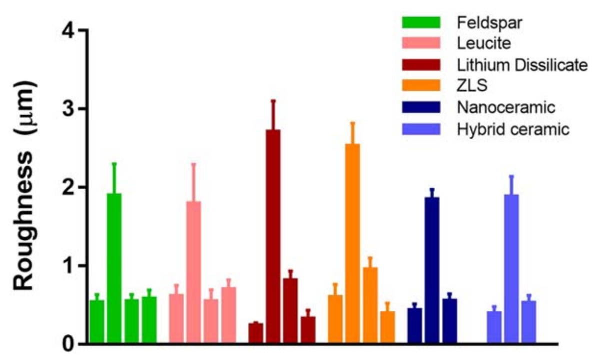
2 | METHODS AND MATERIALS

2.1 | Specimen preparation

The materials used in this study are listed in Table 1. The blocks of the materials were milled at CEREC MCXL (Sirona, Bensheim, Germany), and 10 flat samples with 0.5 cm^2 for each material were obtained. All samples were designed trough CEREC SW 4.2 software and milled with Step Bur 12 and Cylinder Pointer Bur 12 using the normal milling method.

2.2 | Postmilling procedures

After milling, Mark II, IPS Empress CAD, and Enamic were polished with Finopol Diamond Polisher rubber wheels (blue, followed by pink and gray) (Finopol, Praha, Czech Republic) at 7000 rpm. Lava Ultimate was polished with Hatho Habbras Discs (yellow to green) (Hatho, Eschbach, Germany) at 7000 rpm. The specimens milled in IPS e.max CAD and Suprinity were crystallized in a ceramic oven (Programat P300, Ivoclar Vivadent, Liechtenstein) according to the temperatures recommended by the manufacturers (Table 2). After crystallization, IPS e.max CAD and Suprinity were polished with Edenta Startec Diamond Polisher rubber wheels (step 1 violet followed by step 2 green) (Edenta, Hauptstrasse, Switzerland) at 7000 rpm. All procedures for the polishing were performed by a single operator using moderate pressure for 15 seconds for each rubber wheel or disc.



SCHEME 2 Comparison of surface roughness within groups of tested materials at different procedures

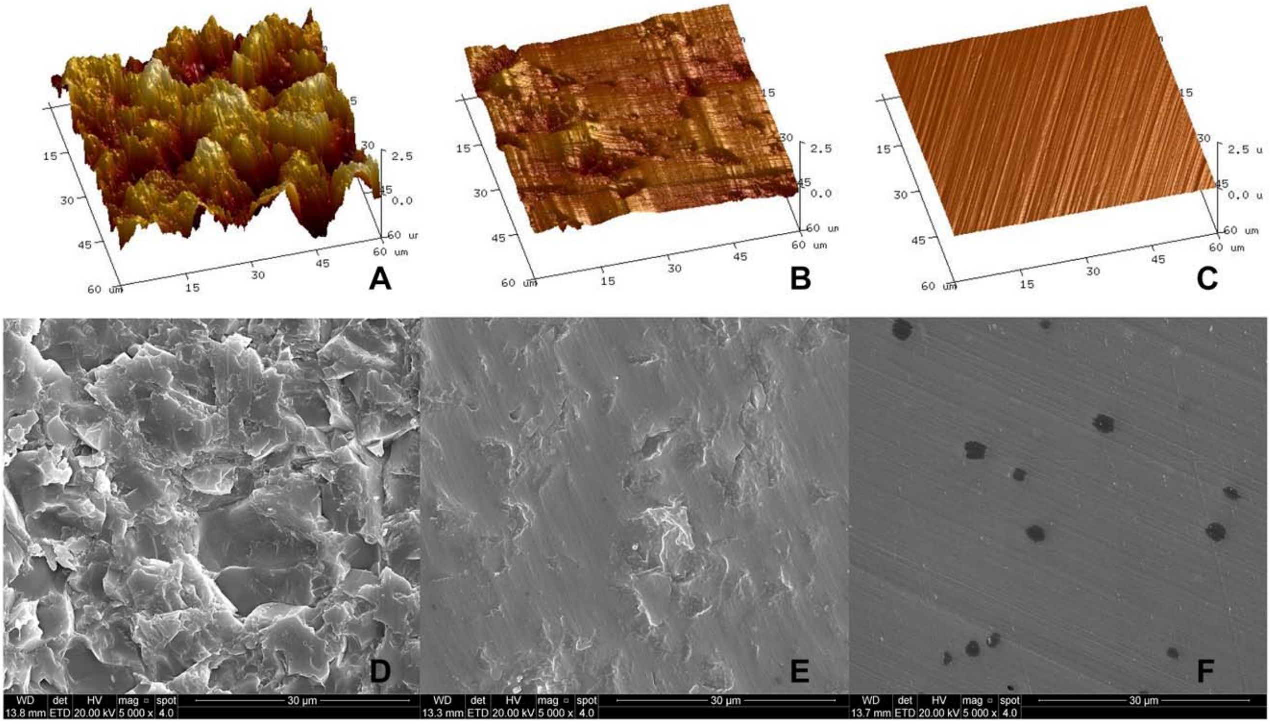


FIGURE 1 AFM and SEM of feldspar-based ceramic after milling (a;d), polishing (b;e), and glaze (c;f)

Glaze was applied according to the manufacturer's instructions and fired in a ceramic oven (CS2, Ivoclar Vivadent, Schaan, Liechtenstein) (Table 2). Mark II received a single glaze coat of 1:1 Vita Akzent Fluid and Vita Glaze AKZ 25 (Vita Zanhfabrik, Bad Säckingen, Germany). IPS Empress CAD and IPS e.max CAD received a single coat of 1:1 Empress Universal Glaze and IPS Empress Glaze Paste. Suprinity was glazed with one coat of Vita Akzent Plus Glaze Paste.

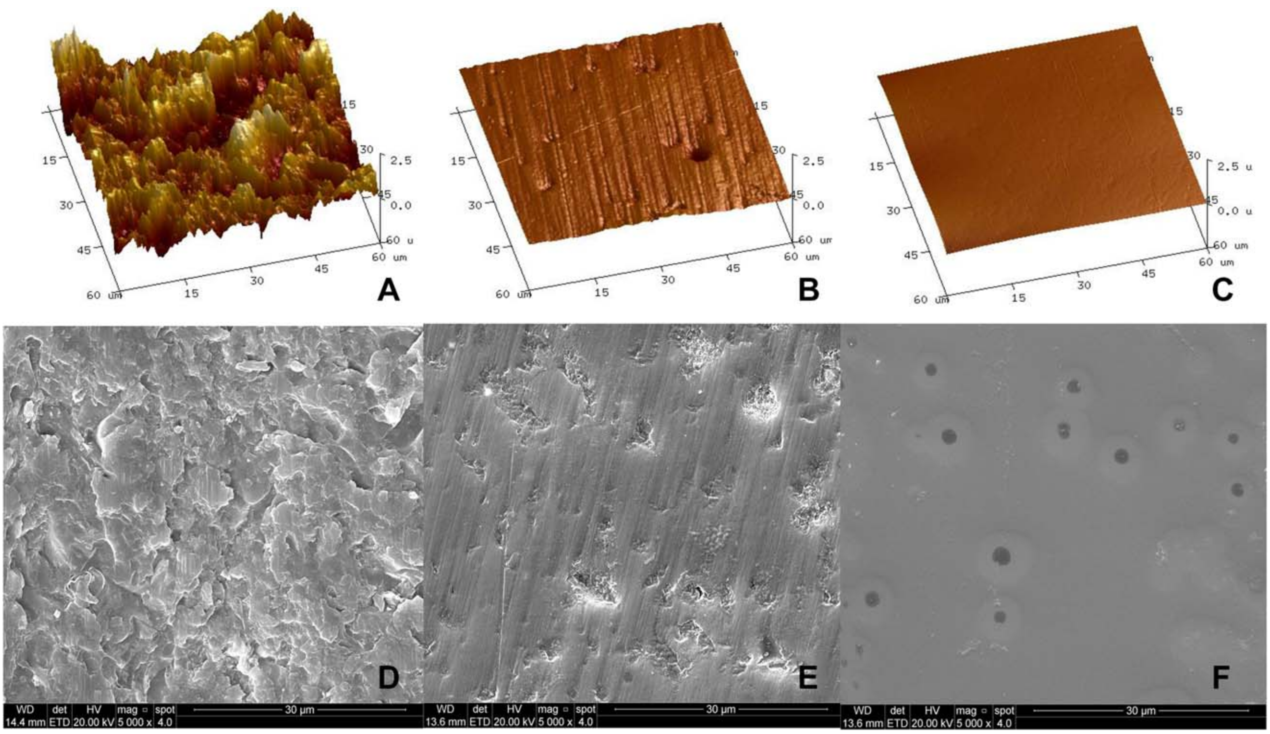


FIGURE 2 AFM and SEM of leucite-based ceramic after milling (a;d), polishing (b;e), and glaze (c;f)

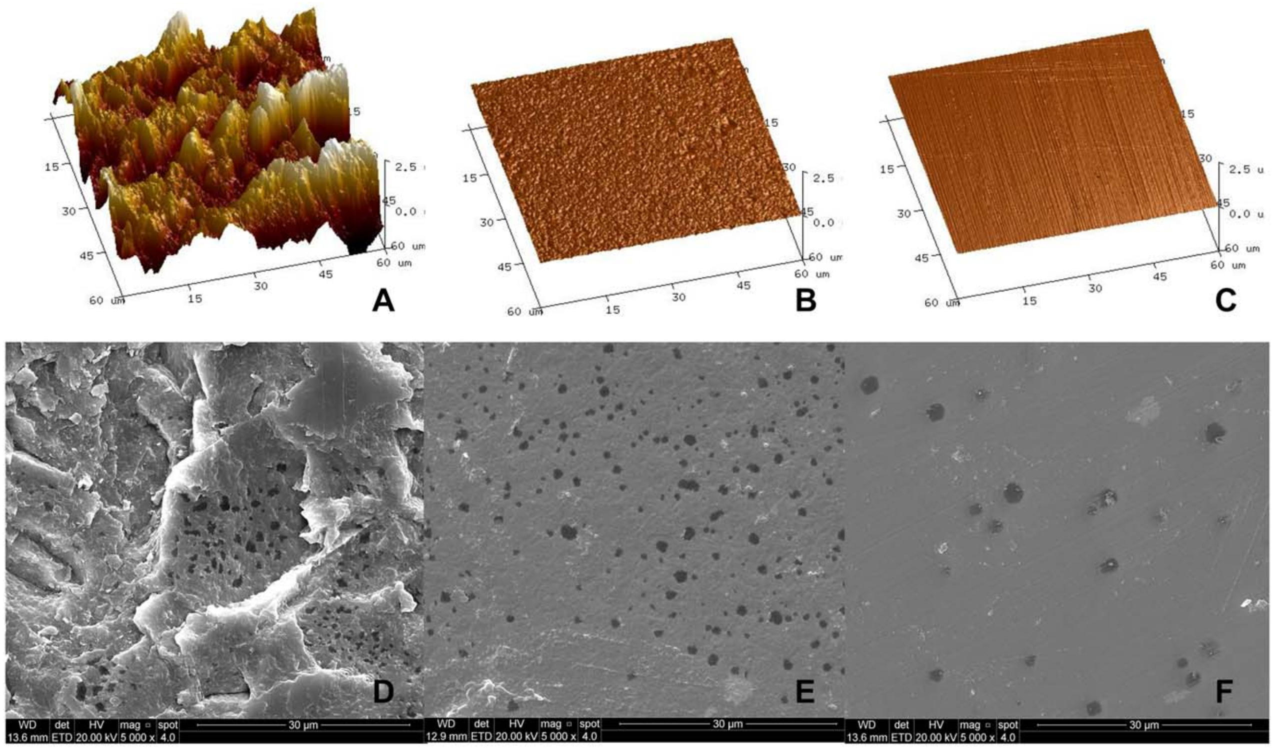


FIGURE 3 AFM and SEM of lithium disilicate-based ceramic after milling (a;d), polishing (b;e), and glaze (c;f)

2.3 | Surface roughness

Surface roughness Ra (μm) was measured using a roughness tester SL-201 (Mitutoyo, Kawasaki, Honshu, Japan) after milling, polishing, and glaze and compared to the external surface from 10 unmilled blocks of each material, as delivered by the manufacturer (baseline). Three consecutive measurements of the specimens were taken from different

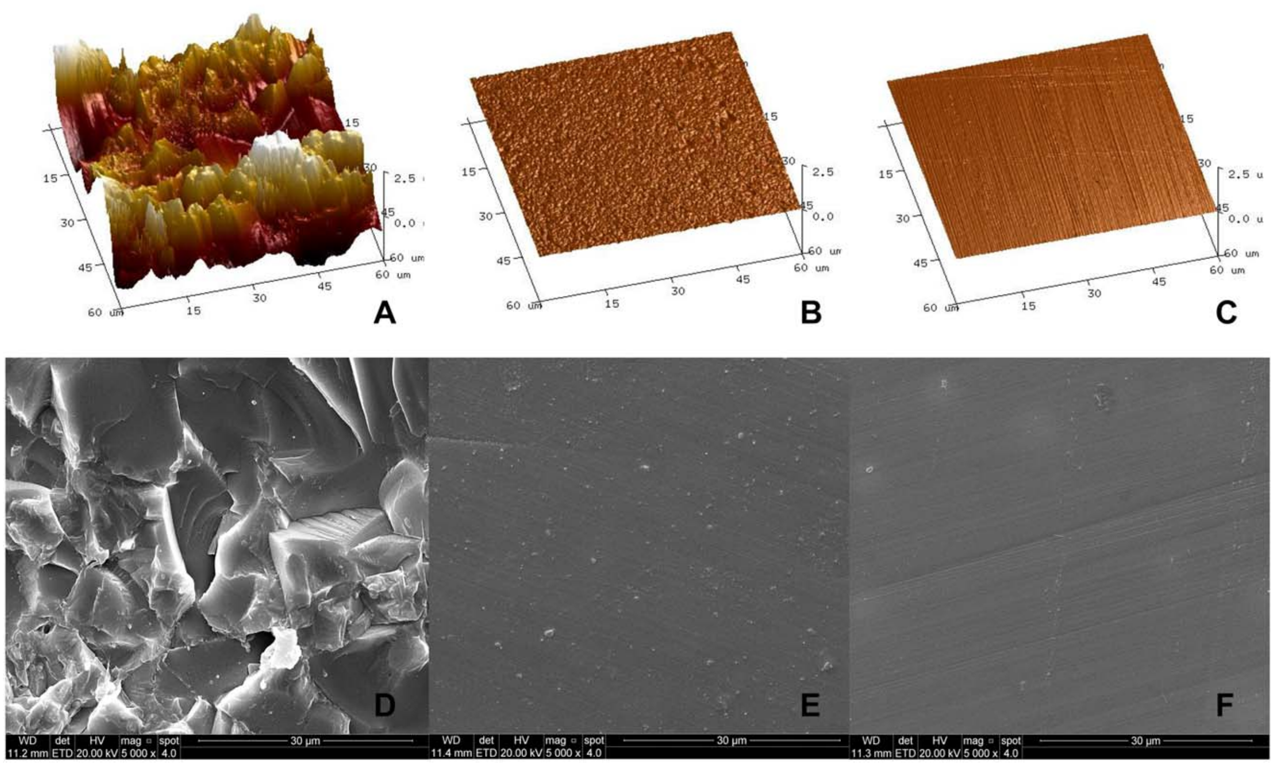


FIGURE 4 AFM and SEM of zirconia-reinforced lithium silicate after milling (a;d), polishing (b;e), and glaze (c;f)

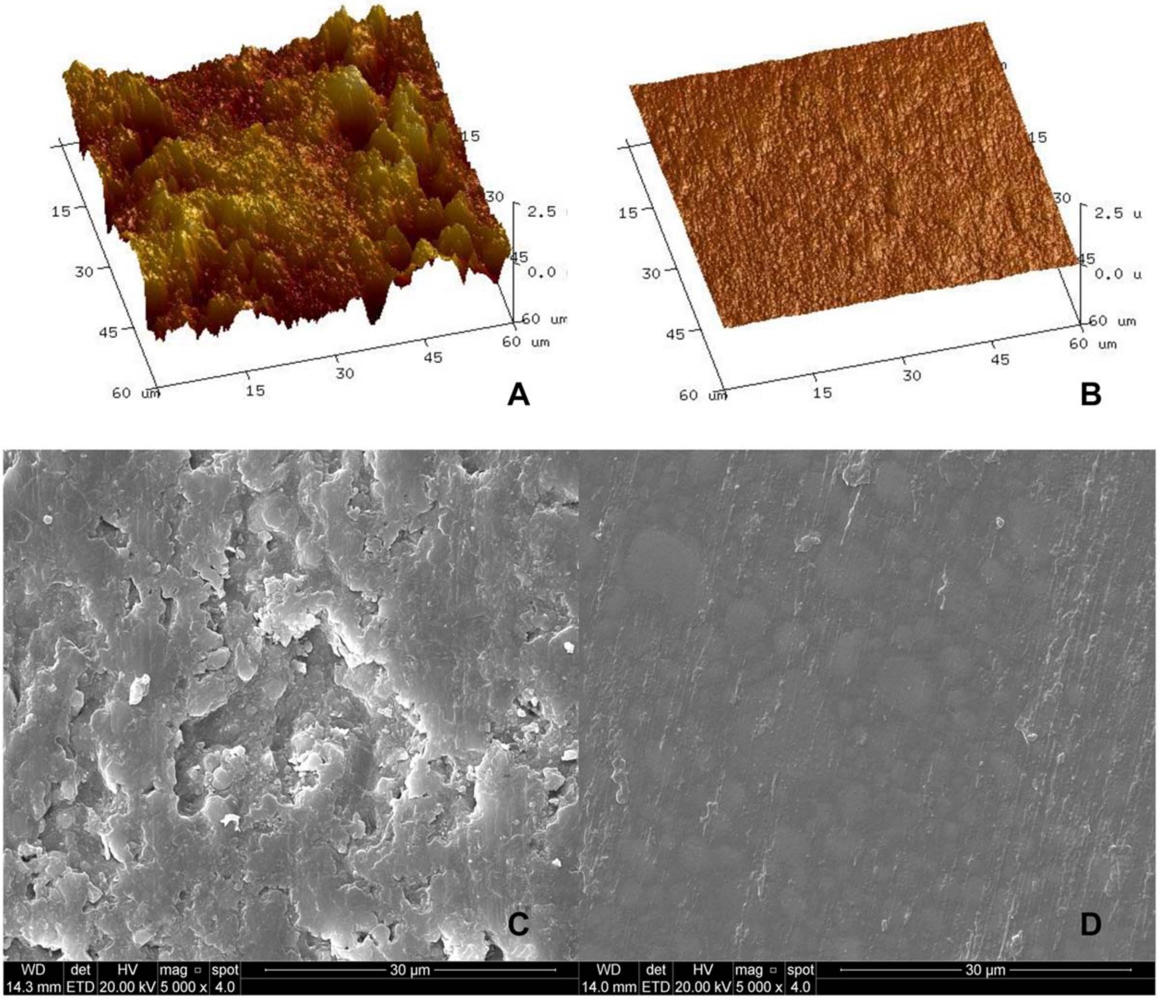


FIGURE 5 AFM and SEM of nanoceramic after milling (a;c) and polishing (b;d)

regions (one central, one right, and one left) with a cutoff of 0.25, and the arithmetic mean roughness (Ra) was obtained. The surface roughness reading was perpendicular to the scratch direction. To always ensure readability in the same direction (perpendicular to the scratches), a mark with a diamond bur and a high-speed handpiece was made on the border of each specimen.

2.4 | Statistical methods

The values of surface roughness were compared with one-way ANOVA with repeated measures and the Tukey HSD test ($\alpha = 0.05$).

2.5 | Atomic force and scanning electron microscopy

At each step, the samples were sonicated in isopropyl alcohol (99.8%) for 5 min and then air dried. A representative sample was analyzed using an atomic force microscope (AFM, Dimension Icon, Bruker, Billerica, Massachusetts) with tip model RTESPA 6 (*T*: 3,75 μm, *f0*: 300 kHz, *L*: 125 μm, *k*: 40 N/m, *W*: 35 μm) in Peak Force Tapping of 60 × 60 μm^2, without any prior preparation. The 3D images were generated for each sample using NanoScope Analysis 1.40 (Bruker, Billerica, Massachusetts). Afterward, the specimens were mounted onto aluminum stubs and then gold sputter-coated (100 seconds, 60 mA) using a sputtering device (Bal-Tec SCD 005, Leica Microsystems, Wetzlar, Hesse, Germany). The samples were observed at ×5000 magnification using a field emission scanning electron microscope (SEM, Inspect F50, FEI, Hillsboro, Oregon).

3 | RESULTS

The surface roughness (Ra) ranged from 0.23 μm of IPS e.max CAD at the baseline up to 2.71 μm when milled. A significant difference was observed between the materials and procedures according to one-way ANOVA. The CAM milling procedure resulted in a significantly increased surface roughness ($P < .05$). Polishing reduced the surface roughness to baseline standards for Mark II, IPS Empress CAD, Enamic, and Lava Ultimate, without a significant difference when glaze was indicated. Glaze significantly reduced the surface roughness of IPS e.max CAD and Suprinity ceramics after polishing (Table 3). An overall comparison between materials and procedures is presented in Scheme 1, and a comparison within groups is shown in Scheme 2.

A high-density surface with the absence of cracks was observed at all SEM images. The pores were presented at IPS e.max CAD after

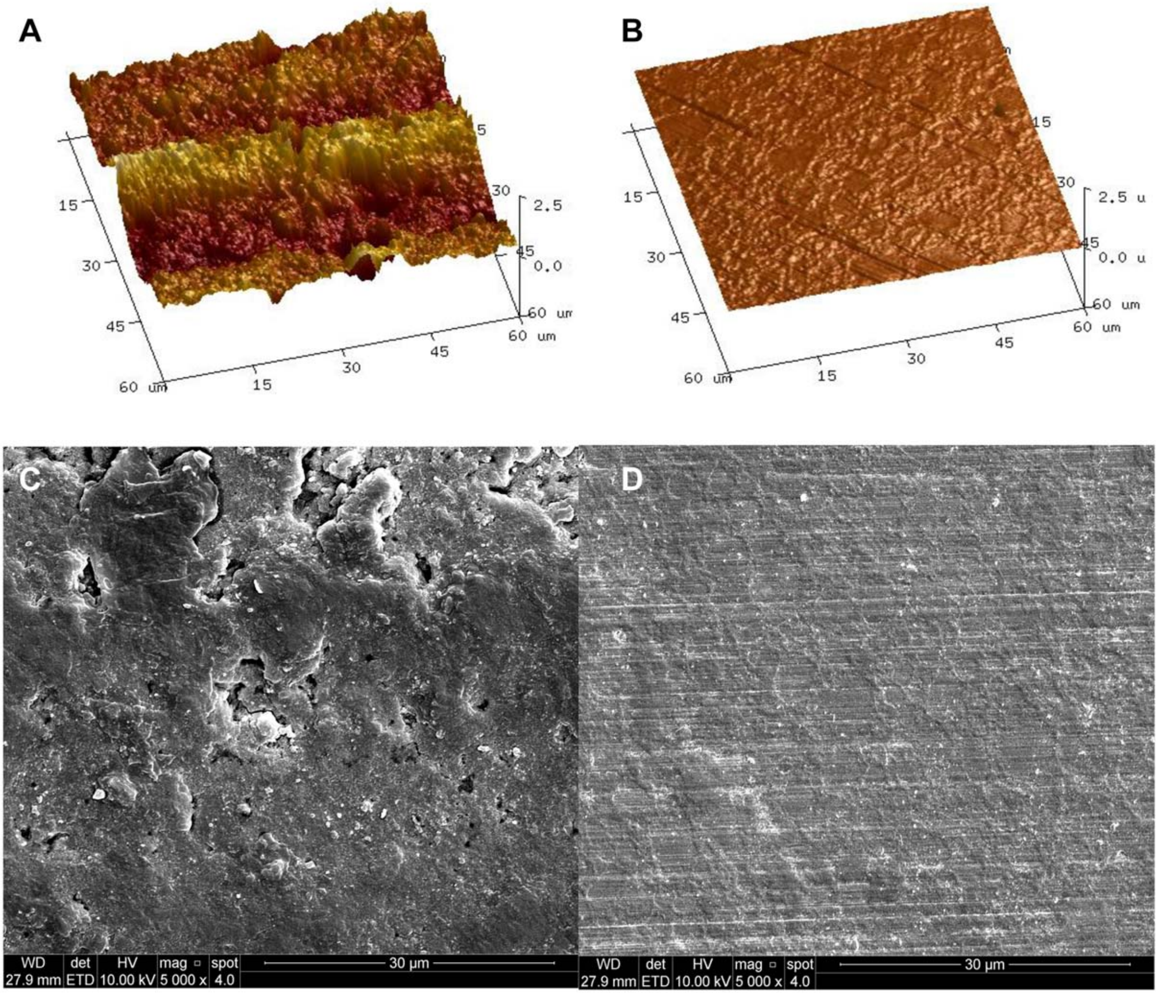


FIGURE 6 AFM and SEM of hybrid ceramic after milling (a;c) and polishing (b;d)

milling and polishing (Figure 3d,e). It is also possible to observe black spherical areas on the surface at IPS e.max CAD, Mark II, and at IPS Empress CAD after glazing (Figures 1f, 2f, and 3f). The glazed surfaces were smoother than the polished surfaces. The clusters were exposed at polished Lava Ultimate samples (Figure 5d).

From the AFM data, a rougher surface can be seen at all materials after milling, with constant topography of peaks and valleys. The polishing procedure promoted smoother surfaces in comparison with milling, and directional scratches due to rubber wheels can be seen in all materials. The polished samples presented a different topography with slight elevations and few waves. In the ceramics where the glaze step was indicated, there were absence of elevations and waves; such surfaces were considered smoother surfaces. A representative image of each material after milling obtained using AFM and SEM, polishing, and glazing when indicated are presented in Figures 1–6.

4 | DISCUSSION

Computer-aided manufacturing (CAM) restorations are produced by selective reduction of a block by abrasion. This study showed that the final surface roughness is influenced by milling and postmilling processes, in accordance with other studies.[14,17]

The milling process caused less surface roughness in fully crystallized/polymerized materials in comparison with partially crystallized materials (IPS e.max CAD and Suprinity). The concept of hard and soft milling explains these two distinguished groups. Hard milling refers to the milling of materials fully crystallized or polymerized from the manufacturer, namely, Mark II, IPS Empress CAD, Enamic, and Lava Ultimate. In contrast, soft milling is based on wearing of the crystalline intermediate state, lithium metasilicate phase Li_2SiO_3, with lower hardness that requires posterior controlled crystallization on a ceramic oven. Probably, the lower hardness of the IPS e.max CAD and Suprinity ceramics provides a softer surface that is more susceptible to the action of the diamond burs, causing increased surface roughness in these materials. As a result, lithium-based ceramics (IPS e.max CAD and Suprinity) were the most affected materials due to milling (2.71 and 2.52 μm, respectively).

The brittle mechanical behavior of dental ceramics could be explained by the presence of cracks and how it propagates. Extremely dense and pore-free materials are the main advantage of CAD/CAM ceramics.[18–20] CAD/CAM ceramics are different from slip-cast ceramics, where voids and cracks are expected. For the last ones, the glazing procedure becomes imperative to increase the strength of the material.[21] According to the results of surface roughness obtained in this study, the fully crystallized Mark II and IPS Empress CAD ceramics returned to baseline roughness only with diamond impregnated rubber wheel polishing, in agreement with previous studies.[14,15] When these ceramics were glazed, there was a slight increase in the surface roughness, and the same was observed in other studies.[16,17] Therefore, glaze did not improve the smoothness for Mark II and IPS Empress CAD ceramics; as a result, this step could be omitted,[15] reducing the time for the chair-side technique. Glaze would be applied only when external staining is indicated. However, partially sintered lithium-based ceramics (IPS e.max CAD and Suprinity) were not capable of achieving baseline smoothness when only performing diamond rubber wheel polishing. The glazing procedure was a very important step to decrease the surface roughness, becoming indispensable, and increasing the time required for the chair-side procedure. The glazing was not applied on Lava Ultimate and Enamic because this procedure is not indicated for these materials due to the polymeric phase. However, both materials achieved the baseline surface roughness with polishing. Next, according to the results, the tested hypothesis was partially accepted.

For the polishing procedure, materials with abrasive particles were used in the order of decreasing size to reduce the scratches produced by the diamond burs. Thus, increasingly fine scratches are formed on the material surface to the point of being imperceptible by the human eye.[22] However, the SEM images show scratches on the surfaces of Mark II (polished and glazed), IPS Empress CAD (polished), Suprinity (polished and glazed), Lava Ultimate (polished), and Enamic (polished). These scratches on the surfaces of the materials are formed by the diamond particles of the rubber wheels. SEM observations are in agreement with the observations of previous studies for Mark II polished and glazed,[15] and for IPS Empress CAD polished[15,16] and glazed[16] surfaces. In addition, pores on the surface of IPS e.max CAD after milling and polishing are observed in the SEM images. These pores occurred due to the presence of highly soluble lithium phosphate spherical crystals[23] that were removed during the milling of the intermediate phase.[24] In addition, black spherical areas in the Mark II, IPS Empress CAD, and IPS e.max CAD after glazing are observed. These black spherical areas are not pores; rather, they are the result of the glaze application. It is speculated that the black areas correspond to small rounded depressions that remained in the surface of the Mark II and IPS Empress CAD ceramics after polishing and that were filled by the glaze. These depressions can also be observed in the AFM images for Mark II and IPS Empress CAD. Because there is a greater amount of glaze in these surface depressions, the aspect is different from the rest of the surface, having a black appearance in the SEM image. For the IPS e.max CAD ceramic, the black areas correspond to the filling of the pores by the glaze.

All the materials obtained a surface roughness higher than 0.2 μm, which favors microbial retention.[3] This surface roughness is probably the result of the diamond paste not being applied after polishing with the diamond impregnated rubber wheels. Regardless of being one more procedure, studies have shown that the inclusion of a diamond polishing paste step is recommended to improve the surface smoothness.[25–27] A future study comparing the application of a diamond polishing paste should provide interesting results.

5 | CONCLUSIONS

The surface roughness (Ra) of all CAD/CAM tested materials was affected by milling. The mechanical polishing was capable of reducing the surface roughness of fully crystallized/polymerized materials to baseline values, making the glaze step dispensable. Partially crystallized materials, that is, lithium-based ceramics, were most affected by milling; such materials required glazing after mechanical polishing.

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DISCLOSURE

The authors do not have any financial interest in any of the companies whose products are included in this article.

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